



THE 15TH POLISH-JAPANESE JOINT SEMINAR
ON MICRO AND NANO ANALYSIS **2026**



Book of Abstracts



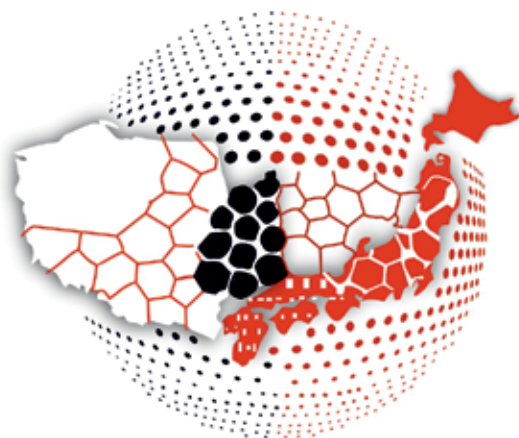
Institute of Metallurgy and Materials Science
Polish Academy of Sciences



HOKKAIDO
UNIVERSITY

September 13-16, 2026, Krynica-Zdrój, Poland

BOOK OF ABSTRACTS



THE 15TH POLISH-JAPANESE JOINT SEMINAR
ON MICRO AND NANO ANALYSIS 2026

September 13–16, 2026, Krynica-Zdrój, Poland

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Minister of Science and Higher Education
Republic of Poland



Book of Abstracts
of the 15th Polish-Japanese Joint Seminar on Micro and Nano Analysis 2026
September 13–16, 2026, Krynica-Zdrój, Poland

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Dear Friends and Colleagues,

On behalf of the organizing committee, welcome to Krynica-Zdrój for the 15th Polish-Japanese Joint Seminar on Micro and Nano Analysis (PJMICO 2026).

The story of meetings began back in the 1980s, born out of simple scientific curiosity and early contacts between Polish and Japanese researchers. What started as informal networking grew into our first official Joint Seminar in Warsaw in 1997. For nearly three decades now, we have met every two years, alternating between Poland and Japan. Our goal has never changed: to create an open, friendly space where successive generations of scientists can debate bold ideas, challenge current concepts, and build collaborations that last a lifetime.

We welcome over 100 participants for four days of intensive scientific exchange. The scientific program includes 5 Keynote Lectures, 9 Invited Lectures, 19 oral presentations, and 9 talks specifically dedicated to our younger colleagues. Additionally, our Poster Session, with 28 contributions, will serve as the hub for direct discussions and networking.

At the heart of PJMICO 2026 is microstructure analysis. The seminar will focus on how crystal structures, defects, interfaces, phase transformations, and local chemical compositions dictate material behavior. A variety of advanced tools will be presented, both experimental and computational, ranging from electron microscopy, diffraction, and spectroscopy to X-ray techniques, in-situ characterization, and modeling.

We also wanted to make sure the program combines methodological developments with practical solutions. This week, we will explore work on high-entropy alloys, magnetic and shape-memory materials, irradiation effects, additive manufacturing, nanomaterials, and biomaterials. Each session highlights how deep structural knowledge directly drives the engineering of advanced materials.

PJMICO has always been about more than just lectures, while maintaining its traditionally informal atmosphere and welcoming younger colleagues into our community. We hope you enjoy the scientific sessions, find time to connect with old and new friends, and take in the beautiful surroundings of Krynica-Zdrój.

Thank you for being here, and we wish you a productive and enjoyable seminar!

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Development of Research Topics Presented at the Polish–Japanese Joint Seminars on Micro and Nano Analysis (1997–2026)

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Keywords: electron microscopy, nanomaterials, composites, metallic alloys, irradiation effects

1. Organization of the Polish–Japanese Joint Seminars

Polish–Japanese (P–J) Joint Seminar on Materials Analysis was held at the Institute of Electron Technology (IET), Warsaw, in September 1997 as part of a joint project between IET and Kyoto Institute of Technology (KIT), authorized under the agreement of the Committee for Co-operation of Science and Technology between the Polish and Japanese Governments. The Seminar was co-organized by Prof. J. Kącki (IET) and Prof. M. Shiojiri (KIT). Thirty researchers participated, not only from IET and KIT but also a Japanese company and other research institutes and universities in Warsaw, Kraków, and Toruń. Twenty-one lectures (15 from Poland and 6 from Japan) were presented [1,2]. The abstracts were published on seminar websites, and selected papers appeared in *Electron Technology* **31** (1998).

Following the success of the first meeting, the **2nd J–P Seminar on Materials Analysis** was held at KIT in December 1998, in conjunction with the **International Symposium on Hybrid Analyses for Functional Nanostructures**. The symposium, organized by Prof. M. Shiojiri, attracted approximately 200 participants and included 36 invited lectures (3 from Poland and 22 from Japan) together with 48 poster presentations. The proceedings (260 pages, ISBN 4-87974-989-3) were published, and 30 papers appeared in special issues of the *Journal of Electron Microscopy* **48** (1999) and **49** (2000).

The **3rd P–J Seminar on Materials Analysis** was co-organized by Prof. J. Dutkiewicz, Dr. J. Morgiel (Institute of Metallurgy and Materials Science (IMIM), Polish Academy of Sciences (PAS)), Prof. T. Okabe (University of Toyama (TU)), and Prof. M. Shiojiri. It was held in Zakopane, Kraków in July 2000. Forty-eight participants (20 from Poland, 24 from Japan, 1 from the Czech Republic, and 3 from Switzerland) presented 29 lectures. During this meeting, it was decided that the Seminar would be held regularly every two years. The **4th J–P Seminar on Materials Analysis** was organized in Tateyama, Toyama in 2002 by Prof. T. Okabe, Prof. S. Ikeno (TU), and Prof. J. Dutkiewicz. It featured 45 presentations.

The **5th Polish–Japanese Symposium on Advanced Methods of Materials Characterization and Materials Analysis** was organized by Prof. J. Dutkiewicz, Prof. A. Czyrska-Filemonowicz (AGH University of Science and Technology), and Prof. K. Matsuda (TU). It was held in Niedzica in 2004, with 42 participants. The **6th J–P Joint Seminar on Materials Analysis** returned to Tateyama, Toyama in 2006. It was organized by Prof. S. Ikeno and Prof. J. Dutkiewicz and included

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18 oral presentations and 16 poster presentations, the latter being intended primarily for students. A memorial book (Photo album) was distributed, and the proceedings were published as a special issue of *Materials Transactions* **48** No. 5 (2007).

Up to this point, the Seminars had been sustained mainly through the personal efforts and financial sacrifices of the organizers and participants. They strongly wished to obtain support through the bilateral collaborative research program of the Japan Society for the Promotion of Science (JSPS) and the Polish Academy of Sciences (PAS). However, this was initially impossible because PAS funding was available only to researchers affiliated with PAS institutes. Prof. J. Kącki and Prof. M. Shiojiri engaged in persistent negotiations with PAS and its Japanese counterpart, JSPS, respectively. Their efforts were eventually successful, making it possible to apply to the JSPS–PAS bilateral collaborative research program. To reflect this achievement and to further promote Prof. Shiojiri's long-standing goal of strengthening collaboration between researchers in materials science and the life sciences, the Seminar was renamed the **Polish–Japanese Joint Seminar on Micro and Nano Analysis** [3]. This title has been used ever since. The 7th–9th, 11th, and 14th–15th Seminars subsequently received financial support through the JSPS–PAS bilateral collaborative research program.

The 7th **P-J Seminar on Micro and Nano Analysis**, celebrating the 10th Anniversary of the **Polish–Japanese Joint Seminar**, was held at IET, Warsaw, on 7–11 September 2008 and was organized by Prof. J. Kącki and Prof. S. Ikeno. Thirty-eight scientists from Poland, Japan, and Malaysia attended, and 32 lectures were presented. Thirteen papers resulting from the Seminar were published in a special issue of the *Journal of Microscopy* **236** (2009). The 8th **J-P Seminar** was organized by Prof. H. Kurata (Kyoto University) and Prof. A. Czerwiński (IET) and was held in Uji, Kyoto on 5–8 September 2010. Selected papers were published in a special issue of *Materials Transactions* **52** (2011). The 9th **P-J Seminar** was organized by Prof. J. Dutkiewicz and Prof. K. Nishimura (TU) and was held in Sieniawa in September 2012. It attracted 73 participants.

The 10th **J-P Seminar** was organized by Prof. K. Nishimura and Prof. J. Dutkiewicz, and was made possible through the dedicated efforts of Prof. Seiichi Watanabe (Hokkaido University). It was held in Sapporo in October 2014. The program included 19 oral presentations and 11 poster presentations. The 11th **P-J Seminar** was organized by Prof. S. Kret (Institute of Physics, PAS) and Prof. S. Matsumura (Kyushu University), and was held at Gniew Castle in September 2016. Fifty participants attended, presenting 32 oral and 16 poster presentations. The 12th **J-P Seminar** was organized by Prof. M. Ishimaru (Kyushu Institute of Technology) and Prof. T. Goryczka (Silesian University), and was held in Fukuoka in August 2018. It attracted 40 participants. Following the interruption caused by the COVID-19 pandemic, the 13th **P-J Seminar** was held in September 2022. It was organized by the Institute of Materials Engineering, Silesian University of Technology, together with Prof. T. Goryczka and Prof. M. Ishimaru. Sixty-seven participants from Poland, Japan, Germany, France, and Ukraine presented 55 contributions, including 40 oral presentations and 15 posters.

The 14th **P-J Seminar** was organized by Prof. K. Matsuda and Prof. W. Maziarz (IMIM, PAS) and was held in Toyama in 2024. Fifty participants (18 from Poland and 32 from Japan) attended, presenting 33 oral lectures and 16 posters during the four-day meeting. The 15th **Polish–Japanese Joint Seminar on Micro and Nano Analysis** will be held in Krynica-Zdrój, Poland, on 13–16 September 2026. It is organized by Prof. W. Maziarz and Prof. T. Shibayama (Hokkaido University) under the bilateral agreement between PAS and JSPS.

2. Evolution of Research Topics Throughout the Seminar Series

An analysis of the research topics presented at the Polish–Japanese Joint Seminars between 1997 and 2026 reveals how the scientific interests of the participants have evolved in response to advances in materials science and microscopy, as seen in Figure 1. The presentations covered a broad range of subjects; however, metallic alloys consistently constituted the largest research category

throughout the seminar series. The exception is the second J–P Seminar, held in 1998 in conjunction with the International Symposium on Hybrid Analyses for Functional Nanostructures, which was characterized by a pronounced predominance of topics focused on the advancement of microscopic research methodologies and the development of nanomaterials of the highest international standard.

Research on metallic alloys—including lightweight alloys, iron-based alloys, shape memory alloys, amorphous alloys, magnetic alloys, and related materials—accounted for the largest number of presentations at every seminar. Depending on the size of each meeting, the number of presentations in this field ranged from eight in 2006 to thirty-two in 2022. This predominance reflects not only the research interests of the participants but also the strong collaboration among universities, research institutes, and industry, as well as worldwide trends in the development of advanced structural and functional materials.

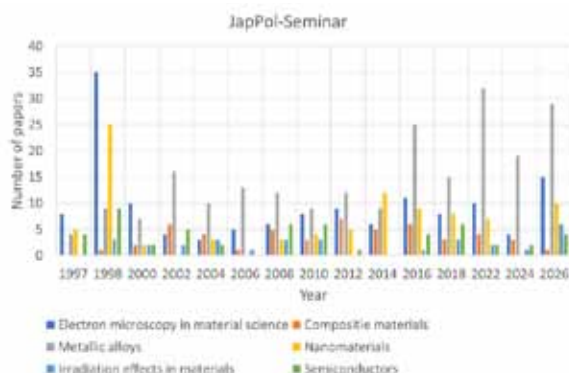


Figure 1. Evolution of research topics presented at the Polish–Japanese Joint Seminars: Number of presentations by research category.

Another major research area has been the development and application of electron microscopy techniques, particularly TEM, STEM, and SEM. Between three and fifteen presentations at each seminar were devoted to advances in microscopy and analytical methods. Many of these contributions were presented by Japanese researchers, reflecting the long-standing strength of electron microscopy research in Japan, supported by close collaboration with microscope manufacturers and by extensive studies using *in situ* observation techniques. These developments have also attracted considerable interest from Polish participants and have contributed significantly to collaborative research between the two countries.

Nanomaterials have become another important research topic over the years. Presentations have addressed nanoparticles, nanowires, bulk nanocrystalline materials, and other nanostructured materials, with the number of contributions reaching a peak of twelve in 2018. This trend reflects the rapidly growing interest in functional nanomaterials for a wide range of technological applications.

Additional research fields have included semiconductors, particularly the structural characterization of thin films, composite materials for energy-related applications, biomaterials, and irradiation effects. Research on irradiation effects was mainly conducted by Prof. H. Takahashi and his colleagues using the unique *in situ* high-voltage electron microscope installed at Hokkaido University.

Overall, the research topics presented at the Polish–Japanese Joint Seminars have evolved continuously in response to global developments in materials science and nanotechnology. At the same time, electron microscopy has remained a common foundation throughout the seminar series, providing an essential analytical tool for investigations across a wide variety of materials and contributing to the continuing scientific collaboration between Poland and Japan.

The observed distribution of research topics may also be influenced by the composition of the seminar participants, who have predominantly represented materials science, especially metallic materials research. While several attempts have been made to attract researchers from the biological and medical sciences, these have so far achieved only limited success. As the seminar series continues to evolve, it will be important to consider whether its future development should focus on strengthening its traditional research areas or on broadening its scope to embrace new interdisciplinary fields, including the life sciences. Finding an appropriate balance between these directions may be a key factor in ensuring the long-term success of the seminar series.

3. Conclusion

Organizing each seminar requires dedicated organizers who prepare proposals for the JSPS–PAS bilateral program, address numerous practical challenges, and make every effort to ensure that the meeting is held regardless of the funding outcome. This requires considerable time and effort. Thanks to a succession of excellent organizers, the P-J seminar has now reached its 15th meeting. Participants continue to attend because the seminar provides clear benefits, such as the exchange of knowledge and the promotion of collaborative research. One of the authors (MS) has experienced the termination of two bilateral seminar series: one because the participants and research themes became overly restricted, and the other because no successor organizer emerged. Perhaps the J–P Seminar has now reached a stage where it should also consider its future. In Japan, there is a saying, “*Onko Chishin* (温故知新)”: *learning from the past to discover new insights*. As the present authors are no longer as closely involved in current research as before, the most we can contribute is to provide records and materials from the past. The task of discovering new insights must be entrusted to the younger generation of researchers.

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High-Quality Thin Films Prepared by Atomic Layer Deposition II

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Keywords: AlN films, TiN films, ZrO₂/TiN heterostructures, atomic layer deposition

In the 14th Jpn-Pol Joint Seminar in 2024, we reviewed the development of high-quality nitride thin films, including AlN, GaN, and TiN, prepared by modified atomic layer deposition (ALD) techniques [1–3]. The present paper reports our subsequent investigations, focusing on (i) the low-temperature hydrogen-manipulated atomic layer epitaxy (ALE) growth of AlN films and (ii) an epitaxial antiferroelectric (AFE) ZrO₂/TiN heterostructure exhibiting a high energy storage density (UESD).

1. Low-temperature hydrogen-manipulated ALE growth of AlN films

Aluminum nitride (AlN) is an important material owing to its exceptionally high thermal conductivity, wide bandgap, and piezoelectric properties, and has been widely used in microelectronics, optoelectronics, and electromechanical systems. As previously reported, we fabricated ~30-nm-thick AlN films with good crystallinity on sapphire substrates using low-temperature ALD at 300°C combined with layer-by-layer in situ atomic layer annealing (ALA) [1]. Trimethylaluminum (TMA) and remote N₂/H₂ plasma were used as the precursor in step (i) and as the reactant in step (iii), respectively, as shown in Figure 1(a). The layer-by-layer in situ ALA process consisted of an Ar plasma treatment inserted after step (iv) as step (v), before the subsequent ALD cycle. The energy transferred from the plasma during ALA not only provided sufficient energy for crystallization but also enhanced adatom migration and ligand removal, thereby significantly improving the crystallinity of the epitaxial layer.

Here, we present a novel hydrogen-manipulated ALE method for the low-temperature epitaxial growth of AlN thin films at 300°C [4]. As illustrated in Figure 1(b), step (iii) (N₂/H₂ mixed plasma) in Fig. 1(a) was separated into step (iii) (H₂ plasma) and step (v) (N₂ plasma), with an Ar purge step (iv) inserted between them. By sequentially applying H₂ and N₂ plasmas, this approach effectively mitigates the steric hindrance caused by surface ligands. The preceding H₂ plasma removes residual precursor ligands, whereas the subsequent N₂ plasma enhances adatom migration. Comprehensive characterization, including X-ray diffraction (XRD), XRD reciprocal-space mapping, pole-figure analysis, high-resolution transmission electron microscopy, and nanobeam electron diffraction, confirms the superior crystallinity of the AlN films and establishes a well-defined epitaxial relationship with the sapphire substrate. High-angle annular dark-field scanning transmission electron microscopy reveals distinct atomic-column arrangements at the interface, indicating that the lattice mismatch is largely relaxed within the first few monolayers. These results demonstrate that the hydrogen-manipulated ALE method significantly lowers the deposition temperature required for the

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epitaxial growth of AlN. This method provides a promising route for the low-temperature epitaxial integration of AlN into semiconductor devices.

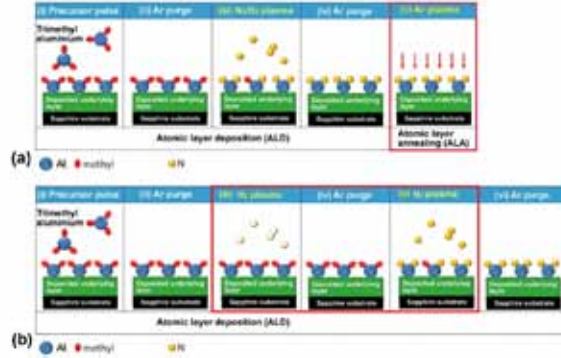


Figure 1. Schematic illustrations of the ALD processes.

- (a) ALD with atomic layer annealing (ALA),
(b) Hydrogen-manipulated atomic layer epitaxy (ALE).

2. Epitaxial AFE ZrO₂/TiN heterostructures with high U_{ESD}

In our previous presentation, we reported high-crystallinity TiN films with a record-low electrical resistivity of $8.2 \cdot 10^{-8} \Omega \cdot \text{m}$ [3]. The TiN films were prepared by ALD, in which step (i) consisted of pulsing the Ti precursor, tetrakis(dimethylamido)titanium (TDMAT, $[(\text{CH}_3)_2\text{N}]_4\text{Ti}$), step (iii) consisted of sequential N₂ plasma (40 s) and H₂ plasma (60 s) treatments, and an additional H₂ plasma treatment was introduced as step (v) of the atomic layer annealing (ALA) process (see Fig. 1). The resulting TiN films were (111)-oriented single-crystalline films with very few crystalline defects, such as dislocations and stacking faults, although a small number of twin boundaries were observed. High-resolution electron microscopy revealed that the Ti and N atomic columns remained coherently aligned across the twin interfaces, which is considered to contribute to the exceptionally low electrical resistivity. These TiN films are promising bottom electrodes for advanced quantum materials and electronic devices.

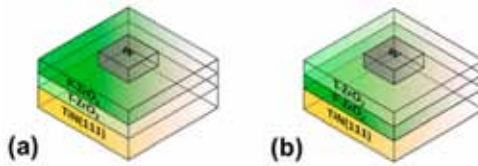


Figure 2. Schematic illustrations of the fabricated ZrO₂ capacitors.

- (a) $W/(P_{1-x}/T_x)\text{-ZrO}_2/\text{TiN}$. (b) $W/(T_x/P_{1-x})\text{-ZrO}_2/\text{TiN}$.
W: top electrode consisting of a ~30-nm-thick tungsten layer.
TiN: bottom electrode consisting of a (111)-oriented TiN film deposited on a sapphire substrate. T-ZrO₂ and P-ZrO₂: sublayers prepared by thermal ALD and plasma ALD, respectively.
x: volume fraction of each ZrO₂ sublayer.

Using these TiN films as the bottom electrodes, we fabricated two types of ZrO₂ capacitors: $W/(P_{1-x}/T_x)\text{-ZrO}_2/\text{TiN}$ and $W/(T_x/P_{1-x})\text{-ZrO}_2/\text{TiN}$ [5]. The top electrode was a ~30-nm-thick W layer

deposited by RF magnetron sputtering. The ZrO_2 layer was approximately 6 nm thick and consisted of two sublayers with different thickness ratios, represented by the volume fraction x , grown by thermal (T) and plasma (P) ALD at 300°C using tetrakis(dimethylamido)zirconium (TDMAZ, $(\text{CH}_3)_2\text{N}_4\text{Zr}$) as the precursor, with H_2O vapor and O_2 plasma as the reactants. Structural analyses revealed that the T- ZrO_2 layer grew epitaxially as a highly crystalline film with its tetragonal [110] axis, corresponding to the antiferroelectric (AFE) polar axis, normal to the (111)-oriented TiN electrode, whereas the P- ZrO_2 layer exhibited an amorphous-like structure. The maximum polarization (P_{max}) and energy storage density (U_{ESD}) extracted from the polarization–electric field (P–E) hysteresis loops increased with increasing x . Furthermore, both P_{max} and U_{ESD} were higher for $\text{W}/(\text{P}_{1-x}/\text{T}_x)\text{-ZrO}_2/\text{TiN}$ than for $\text{W}/(\text{T}_x/\text{P}_{1-x})\text{-ZrO}_2/\text{TiN}$ with the reversed stacking sequence. These trends were well correlated with the x -dependent fraction of (110)-oriented ZrO_2 relative to the total tetragonal ZrO_2 , as estimated from X-ray diffraction (XRD). The results indicate that the AFE characteristics originate primarily from the (110)-oriented ZrO_2 crystals in the T- ZrO_2 layer grown directly on the (111)-oriented TiN electrode, whereas the P- ZrO_2 overlayer serves as an effective leakage-current barrier. Finally, we fabricated a wafer-scale epitaxial AFE ($\text{P}_{0.25}/\text{T}_{0.75}$)- ZrO_2/TiN heterostructure exhibiting a state-of-the-art UESD of approximately 118.6 J/cm³, thereby demonstrating its strong potential for next-generation energy-storage applications.

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In-situ measurement of I-V properties on interconnection metals in semiconductor devices

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Keywords: in-situ TEM, semiconductor, conductor materials, electromigration

1. Introduction

As reducing a gate length from planar MOSFET (Metal-Oxide-Semiconductor Field Effect Transistor) to FinFET (Fin Field Effect Transistor), it is important to reduce a wiring metal pitch as keeping reliability of electromigration during its lifetime. Recently, prototype 2 nm generation transistors by using gate-all-around (GAA) technologies are successfully produced on 300 mm Si wafers by TSMC [1] and Rapidus [2]. It is a noteworthy point in 2 nm generation transistors. 2 nm has no relationship with previous physical parameters such as gate length, wiring metal pitch, and so on. 2 nm means a kind of process node generations. The wiring metal pitch is down to around 20 nm in the minimum center-to-center distance for beyond 2 nm process node generations. For example, grain boundaries, defects and even slight oxidation can increase resistance of wire metals. Therefore electromigration is one of critical issues to improve reliability for beyond 2 nm generation transistors. So, it is important to investigate trigger of electromigration by in-situ TEM with applying biasing to wire metals. In this study, we tried to develop operand experiments combined with in-situ TEM observation and spontaneous I-V measurements by using TEM thin specimen of wire metal thin film on single crystal Si substrate by prepared FIB (Focused Ion Beam equipment) on MEMS tip.

2. Experimental procedure

All wire metal thin film such as Al (99.999%) or Cu (99.999%) were deposited on Si(001) single crystal substrate by RF sputtering equipment under pure 1.0 Pa Ar (99.9999%) gas environments. Target and specimen (TS) distance were chose to appropriate sputtering time and its columnar grain structure. After deposited wire metals on Si(001) single crystal substrate, a cross sectional membrane with 20 μm in width and 10 μm in height was prepared by focused ion beam (FIB) equipment. After picking the membrane by glass probes, the membrane was fixed on MEMS tip by W wire metals deposition for 4 points probe measurements. Finally, operand experiments were

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done by using 4 points probe measurements sample holder (Lightning, DENSsolutions) using the membrane of wire metals on Si substrate on MEMS tip in Cs-corrected STEM/EDS (NEOARM, JEOL) operated 200 kV. Outer probes supply current and inner probes measure voltage (resistance) by constant current conditions using a voltage and current source (2450, Tektronix, KEITHLEY). All of images were taking by CMOS camera (OneView, Gatan, Inc.)

3. Results and summary

Figure 1 shows the membrane of wire metals (columnar grain structured Cu deposited by RF sputtering) on Si substrate on MEMS tip. The constant current was increased by step by step choosing a compliance voltage due to prevent a sudden voltage rise. Figure 2 shows operand observation of electromigration from (a) before biasing, (b) diffusion started by Cu to Si substrate, (c) necking occurred by progress diffusion, (d) void formed by electromigration, (e) void growth by electromigration progress and (f) finishing electromigration. We successfully observed trigger of electromigration in this study. Detailed cross section change of wire metals during biasing and microstructure evolution will be presented at the joint seminar.

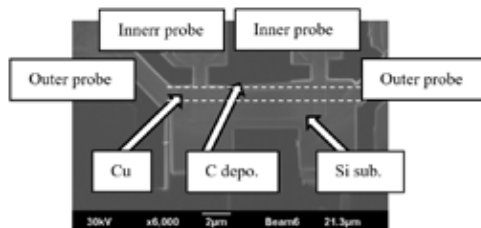


Figure 1. The membrane of wire metals.

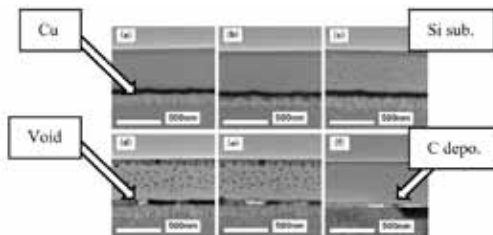


Figure 2. Video clips during operand experiments.

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Structural properties of heterostructures with nontrivial band topology towards quantum devices and applications

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Keywords: topological materials, transmission electron microscopy, molecular beam epitaxy, boron nitride, moiré superlattices

Materials exhibiting nontrivial band topology have emerged as one of the most rapidly developing research fields in condensed matter physics due to their unique electronic states protected by symmetry and topology. When integrated into low-dimensional and van der Waals heterostructures, these materials offer unprecedented opportunities for engineering quantum phenomena at the nanoscale, including topologically protected transport, spin–momentum locking, and novel excitonic states. Realizing such functionalities requires precise control over crystal structure, interfaces, defects, strain, and phase stability, making advanced structural characterization indispensable for understanding and optimizing their properties. The present work focuses on the atomic-scale structural properties of topological heterostructures and their relationship to material performance, providing knowledge essential for the development of future quantum electronic, photonic, and spintronic devices. The presented results were obtained through a multidisciplinary collaboration involving advanced transmission electron microscopy and structural characterization of van der Waals heterostructures, conducted at the Institute of Physics, Polish Academy of Sciences (IF PAN). The research combined expertise in molecular beam epitaxy (MBE) growth of transition metal dichalcogenides, metal-organic vapor phase epitaxy (MOVPE) of hexagonal boron nitride (hBN), synthesis of graphene-based materials, and comprehensive optical, structural, and nanoscale characterization carried out by collaborating teams from the University of Warsaw, and the different groups of Institute of Physics of the Polish Academy of Sciences.

A comprehensive methodology was developed to prepare high-quality cross-sectional specimens of van der Waals heterostructures, enabling precise investigation of their interfacial boundaries using transmission electron microscopy (TEM). The presented research results focused on the structural characterization, growth, and optimization of properties of two-dimensional (2D) materials and van der Waals heterostructures by molecular beam epitaxy (MBE) [1–4]. TEM investigations played a central role in determining crystalline quality, phase composition, layer uniformity, and epitaxial relationships between 2D materials and technologically relevant substrates such as GaAs and hexagonal boron nitride (hBN) [1–4,6].

The studies provided important insights into the structural and functional properties of emerging 2D materials. Investigations of MoTe_2 polytypes (2H and 1T'), boron nitride polytypes, and topological materials such as NiTe_2 and TaAs revealed fundamental relationships between crystal structure and electronic properties. High-optical-quality transition-metal dichalcogenide monolayers exhibiting narrow excitonic emission lines and excellent large-area homogeneity were obtained on micrometer-sized hBN flakes substrates, demonstrating their suitability for advanced optoelec-

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tronic applications [2,6]. The challenge of integrating 2D materials with conventional three-dimensional semiconductor platforms was addressed by growing nearly lattice-matched $\text{NiTe}_2/\text{GaAs}$ heterostructures, thereby opening new opportunities for monolithic device integration [3]. Furthermore, a novel electron-beam-assisted selective growth technique was demonstrated, enabling local control of van der Waals heterostructure formation and providing a scalable route toward device fabrication.

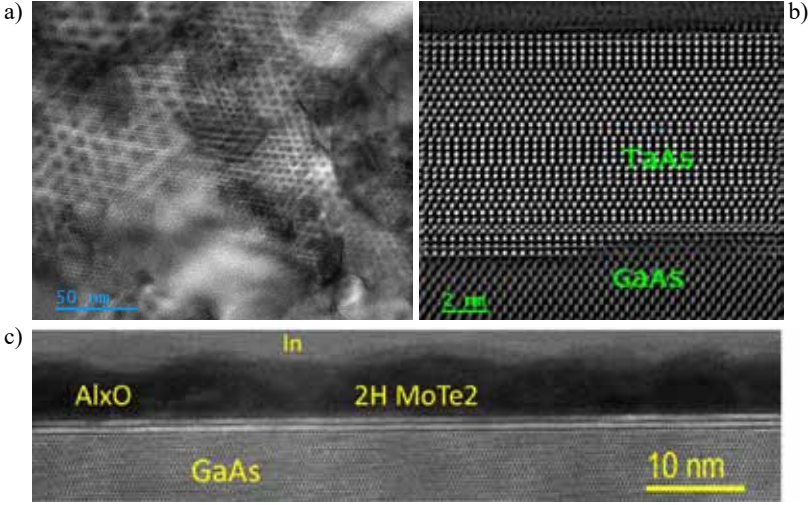


Figure 1. (a) Plan view STEM image in crystallographic contrast of Boron nitride exfoliated epilayer grown by MOVPE on sapphire [7]; (b) STEM image of cross-section of TaAs layer grown by MBE on GaAs [4] (c) 2H phase c of MoTe2 grow by MBE on GaAs(111)B substrate [1].

Significant contributions were also made to research involving both exfoliated single-crystalline hBN flakes for the growth of 2D materials, as well as epitaxial hBN layers deposited on large-area sapphire substrates [2,6]. The former approach enabled the growth of high-quality transition-metal dichalcogenide monolayers by MBE, whereas the latter provided a unique platform for investigating structural phenomena associated with polytypism, symmetry breaking, and twisted van der Waals heterostructures. By combining atomic-resolution scanning transmission electron microscopy (STEM) in specially optimized crystallographic-contrast conditions, structural modeling, and newly developed image-analysis algorithms, it was demonstrated that moiré patterns can be directly correlated with local stacking configurations and twist-angle domains, allowing nanoscale mapping of rhombohedral and hexagonal BN polytypes [7]. These structural observations were correlated with ultraviolet defect-related photoluminescence, establishing a comprehensive methodology for distinguishing BN polytypes through their local atomic environment and symmetry-dependent optical response [5]. The results significantly advanced the understanding of the complex nanostructure of epitaxial boron nitride and its influence on optical properties.

Overall, the research combines advanced electron microscopy, solid-state physics, materials engineering, optics, and nanotechnology to deepen the understanding of van der Waals materials and heterostructures. The results contribute both to fundamental knowledge of charge transport, interfacial interactions, polytypism, and topological phenomena, and to the development of next-generation electronic, optoelectronic, and photonic devices based on two-dimensional materials.

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Extraction of Demagnetization Field in Thin-foiled $\text{Nd}_2\text{Fe}_{14}\text{B}$ Crystal

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Keywords: electron microscopy, magnetic materials, magnetic domains,
grain boundary, demagnetization field

1. Introduction

Permanent magnets play a critical role in modern technologies, including electric vehicles, renewable energy systems, and advanced electronic devices. Among them, Nd–Fe–B magnets provide the highest energy product due to their large saturation magnetization and strong magnetocrystalline anisotropy. However, the coercivity of practical Nd–Fe–B magnets remains significantly lower than the theoretical anisotropy field, and understanding the origin of this discrepancy remains a major challenge in magnetism research. One of the key factors influencing coercivity is the demagnetization field, which can locally promote the nucleation of reverse magnetic domains and trigger magnetization reversal.

Despite its importance, direct experimental observation of the demagnetization field inside a magnetic specimen remains a challenge. Electron holography is a powerful transmission electron microscopy technique capable of visualizing magnetic flux distributions with nanometer-scale spatial resolution. However, electron holography measures the total magnetic flux density, which contains contributions from magnetization, stray magnetic fields outside the specimen, and demagnetization fields inside the specimen. These components are superimposed in the reconstructed phase image, making it difficult to isolate the contribution from the demagnetization field alone.

In this work, we present a methodology for extracting phase information corresponding to the internal demagnetization field in a thin magnetic specimen using electron holography [1]. By combining electron holography observation with structural characterization and numerical magnetic-field calculations, the phase component corresponding to the demagnetization field can be extracted quantitatively.

2. Methodology and Results

The method was first evaluated using an artificial bar-shaped $\text{Nd}_2\text{Fe}_{14}\text{B}$ model specimen. This simplified system provided a clear validation of the approach because the geometry and magnetization state were well defined. The extracted phase map successfully reproduced the expected demagnetization-field distribution, showing strong field concentration near specimen edges and rapid decay toward the interior. These results agreed well with predictions based on classical electromagnetism, confirming the validity of the method.

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The method was subsequently applied to a real thin-foil specimen prepared from a single-crystalline $\text{Nd}_2\text{Fe}_{14}\text{B}$ grain extracted from a commercial sintered magnet. A focused ion beam system was used to prepare a thin foil suitable for transmission electron microscopy observation. The specimen geometry, thickness variation, and crystallographic orientation were carefully characterized using transmission electron microscopy, electron diffraction, and electron energy-loss spectroscopy. In addition to the analysis of experimental data (electron holography observations), these structural parameters were also used to construct a realistic numerical model for magnetic-field calculations.

Electron holography study was performed using a 300-kV transmission electron microscope (HF-3300X, Hitachi) equipped with a double biprism system. The reconstructed phase image clearly visualized the magnetic flux distribution both inside and outside the specimen. The observed phase contours were consistent with the magnetization direction expected from the crystallographic easy axis of $\text{Nd}_2\text{Fe}_{14}\text{B}$.

A simulated phase image based on the numerical model showed excellent agreement with the experimentally observed phase distribution, demonstrating that the specimen geometry and magnetic state were accurately reproduced. Using this validated model, the contributions from magnetization and stray magnetic fields were calculated and subtracted from the total experimental phase image. As a result, a phase map corresponding to the internal demagnetization field was successfully extracted. The obtained demagnetization-field map revealed strong localization of the field near specimen edges, whereas only weak demagnetization effects were observed in the inner region of the specimen. Quantitative analysis indicated that the demagnetization field reached approximately -0.38 T near the specimen edge and decreased rapidly with increasing distance from the edge. This spatial behavior is consistent with the expected field distribution generated by magnetic surface charges.

To further evaluate the reliability of the extracted field distribution, the experimental results were compared with micromagnetic simulations based on Landau–Lifshitz–Gilbert calculations. The experimentally derived demagnetization-field map showed excellent agreement with the simulation results, demonstrating the robustness and accuracy of the proposed method.

3. Summary

This study establishes a practical experimental approach for visualizing and quantifying the internal demagnetization field in magnetic materials using electron holography. The ability to directly evaluate demagnetization fields provides new insight into coercivity mechanisms and magnetization reversal processes in permanent magnets. Beyond Nd–Fe–B systems, the proposed methodology is expected to be broadly applicable to a wide range of magnetic materials and nanostructures, offering a valuable tool for future studies aimed at designing high-performance magnetic materials with improved coercivity and stability.

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Mechanical behavior, nanostructure stability, and defect evolution mechanisms in nanocrystalline medium-entropy alloy under high-temperature irradiation

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Keywords: nanocrystalline grains, defects, mechanical properties, microstructure

To sustain extreme environments in advanced nuclear concepts, innovative structural materials with various compositional and microstructural designs have been explored for the past few decades. The current work investigates one such recently developed structural material, oxide dispersion-strengthened (ODS)-NiCoFe, which uniquely combines various design strategies, such as concentration solid-solution alloys (CSAs), high-density grain boundaries, and nano-oxide dispersion strengthening. The ODS-NiCoFe was examined following a high-temperature (580°C) Ni²⁺ irradiation up to a maximum of 63 dpa, to understand irradiation effects on the stability of the nanosized grains and oxide precipitates, mechanical behavior, and defect evolution. The enclosure of high-density grain boundaries and the ODS particles increased the hardness from 2 + 0.1 GPa (NiCoFe) to 4.3 + 0.9 GPa (ODS-NiCoFe) while irradiation hardening of 25% occurred in both NiCoFe and ODS-NiCoFe.

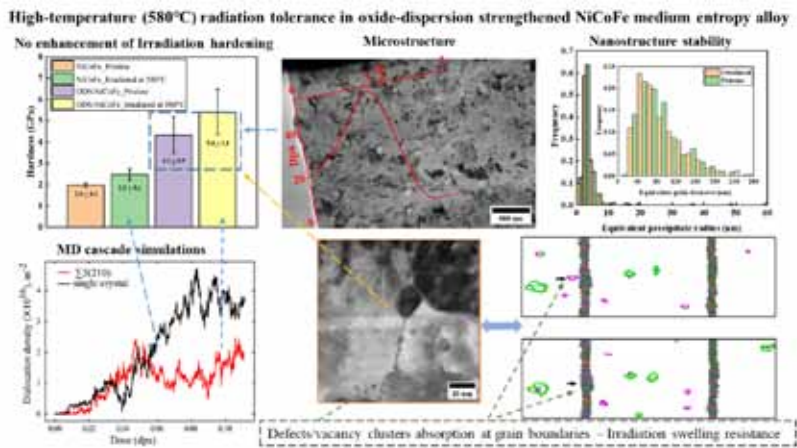


Figure 1. Description of the high temperature irradiation tolerance of newly produced HEA-ODS alloys.

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Post-irradiation transmission electron microscopy observations indicated the physical stability of nanosized precipitates and grains and the presence of irradiation-induced dislocation loops as major defect structures. However, no significant voids were noticed. Similarly, molecular dynamics cascade simulations also revealed inhibition of the growth of vacancy clusters. The simulations demonstrated the absorption of the mobile stacking fault tetrahedra containing trapped vacancy clusters at the grain boundaries and Shockley dislocation dominant chains mainly consisting of interstitial clusters that grow and move three-dimensionally in polycrystalline NiCoFe. These findings indicate high-temperature radiation tolerance of the novel ODS-NiCoFe. Thus, the current assessment presents a state-of-the-art structural material design strategy for advanced reactor applications.

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Evolution of crystal structures under tensile stress for Co-Cr-Al-Si superelastic alloys

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Keywords: shape memory alloys, biomaterials, low Young's modulus, superelasticity, neutron diffraction

1. Background

Metallic biomaterials are widely used to replace or support failed hard tissues due to their superior mechanical properties [1]. For conventional metallic biomaterials, good biocompatibility, high corrosion resistance, and strong wear resistance are generally required. Recently, it has been realized that a low Young's modulus similar to that of human bone is also important because this helps to avoid bone atrophy due to the stress shielding effect [2,3]. Stainless steels have the biggest share of the market because of their low cost, but they have inferior corrosion resistance and a high Young's modulus. Conventional CoCr alloys boast high corrosion resistance, but their Young's modulus is still high. β -type Ti alloys generally show a moderately low Young's modulus, but their wear resistance is low.

On the other hand, the use of shape memory alloys is also increasing due to their unique superelastic behavior. Ni-Ti is the most widely used system, because of its excellent superelastic behavior (up to about 7%) and good biocompatibility. However, the large amount of Ni raises concerns about allergy. There are several successful Ni-free β -type Ti shape memory alloys developed to settle this problem, but their superelastic strain is as small as about 5%.

Recently, we developed Co-Cr-Al-Si (CCAS) shape memory alloys [4]. By using $\langle 001 \rangle$ single crystals, the CCAS alloys show both a low Young's modulus of about 30 GPa and superelasticity up to 17%. Moreover, they have good corrosion and wear resistance comparable to conventional CoCr alloys, thus being promising as next-generation biomaterials. In this talk, I will briefly introduce the elastic and superelastic properties of the CCAS alloys. I will also introduce our recent experimental results using neutron diffraction measurements under tensile tests on the CCAS alloys at low temperatures.

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2. Low Young's Modulus and Superelasticity of CCAS Alloys

Using $\langle 001 \rangle$ -oriented CCAS single crystals, the mechanical properties within the elastic region at room temperature were investigated. It was found that for two different compositions, $\text{Co}_{51}\text{Cr}_{34}\text{Al}_7\text{Si}_8$ and $\text{Co}_{52}\text{Cr}_{33}\text{Al}_{6.5}\text{Si}_{8.5}$, their Young's modulus is as low as about 30 GPa; this is the same as that of human bone, which generally varies within 10–30 GPa. In Ni-free β -type Ti shape memory alloys such as Ti-Nb-Ta-Zr (TNTZ), a low Young's modulus down to about 35 GPa was reported by Tane et al. [5]. For TNTZ, because of the nature of Ti-based alloys, the elastic constants, such as bulk modulus (100 GPa or even lower), are low. Besides, for TNTZ, the elastic anisotropy factor A , being around 2.0 to 2.6, is noticeably large. As a result, a low Young's modulus of 35 GPa was obtained along the $\langle 001 \rangle$ direction [5]. However, for CCAS, the bulk modulus is as large as around 190 GPa. Thanks to the huge value of A greater than 10 (10.7 for $\text{Co}_{51}\text{Cr}_{34}\text{Al}_7\text{Si}_8$ and 12.8 for $\text{Co}_{52}\text{Cr}_{33}\text{Al}_{6.5}\text{Si}_{8.5}$), a low Young's modulus was achieved along the $\langle 001 \rangle$ direction, whereas a large Young's modulus greater than 300 GPa appears along the $\langle 111 \rangle$ direction.

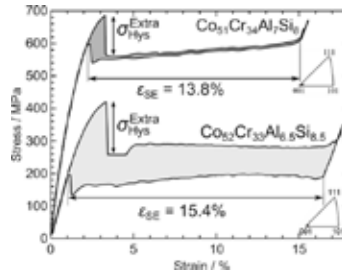


Figure 1. Superelastic properties of $\langle 001 \rangle$ -oriented Co-Cr-Al-Si single crystals at room temperature.

On the other hand, CCAS alloys also show excellent superelastic properties. As shown in Figure 1, both compositions of CCAS exhibit superelasticity with a maximum superelastic strain greater than 15% and a total recoverable strain greater than 17%. These values are among the largest in the literatures for shape memory alloys, such as 13% for Fe-Ni-Co-Al-Ta-B [6], 15% for Ni-Co-Fe-Ga [7], and 18% for Cu-Al-Ni [8]. However, within the framework of biomedical materials, this value of 17% is the largest, being over twice that of Ni-Ti [6] and over three times that of Ni-free options [9,10]. Therefore, CCAS alloys are promising for biomedical applications utilizing their superelastic properties. However, as seen in Figure 1, the current CCAS alloys show a large drop in stress upon the start of stress-induced martensitic transformation, which is denoted as $\sigma_{\text{Hys}}^{\text{Extra}}$. When $\sigma_{\text{Hys}}^{\text{Extra}}$ appears, the sample receives a large stress impact and fractures after several superelastic cycles. This is a big problem to be settled, and we are currently investigating the origin of this phenomenon to promote new alloy development in order to settle this problem in the future.

3. Superelasticity of CCAS Alloys at Low Temperatures and Neutron Diffraction Measurements

Another $\langle 001 \rangle$ -oriented CCAS single crystal was prepared and used for tensile tests at room and low temperatures. The results are shown in Figure 2(a). As the temperature decreases, both the values of critical stress σ_c and $\sigma_{\text{Hys}}^{\text{Extra}}$ decrease, which are plotted against temperature in Figure 2(b). It is especially interesting to notice that $\sigma_{\text{Hys}}^{\text{Extra}}$ becomes negligibly small at 100 K, and therefore a smaller stress impact on the sample can be expected.

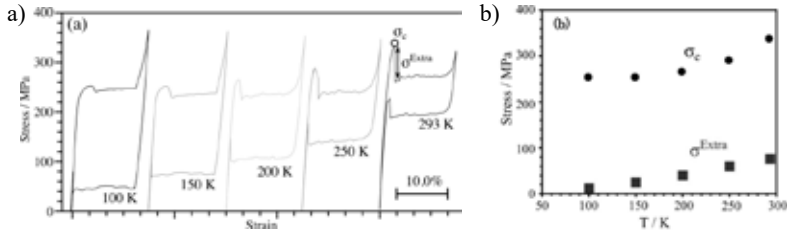


Figure 2. (a) Stress-strain curves of a (001)-oriented Co-Cr-Al-Si single crystal at low temperatures. (b) Temperature dependence of σ_c and σ_{Hys}^{Extra} .

In order to investigate why the decrease in σ_{Hys}^{Extra} occurred at low temperatures, in situ neutron diffraction measurements under tensile stress were conducted using BL19 Takumi in J-PARC. As shown in Figures 3(a) and 3(b), at 293 K, before the drop in stress, which is the start of stress-induced martensitic transformation, the peak from the B2 parent phase is seen. After the drop in stress, as expected, the peak from the L10 martensite phase appears. However, at low temperatures, a slightly different behavior was seen. Even before the small drop in stress, an extra diffraction peak was seen, as shown in Figure 3(c). After the small drop in stress, the peak from the martensite phase appears (Figure 3(d)). When the strain increases so that all the parent phase transforms to martensite, the extra diffraction peak disappears (not shown in Figure 3). During my talk, I will show other results from neutron diffraction measurements, including measurements obtained by moving the positions of the slits so that one can determine the crystal structures at different parts of the sample. By doing this, we have concluded that an intermediate phase appeared on the boundary between the parent and martensite phases only at low temperatures. Furthermore, it seems that the appearance of the intermediate phase may contribute to reducing the value of σ_{Hys}^{Extra} at low temperatures.

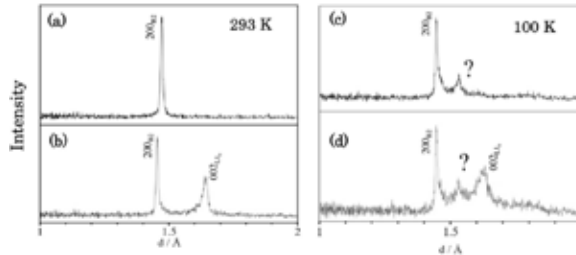


Figure 3. The neutron diffraction patterns at 293 K (a) before the drop of stress and (b) after the drop of stress obtained from (001)-oriented single crystal. Those at 100 K (c) before the drop of stress and (d) after the drop of stress.

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Correlation between structure and magnetic properties in $\text{Fe}_{81}\text{Ni}_2\text{Cu}_1\text{Si}_4\text{B}_8\text{P}_4$ ribbons

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Keywords: metallic glass, magnetic properties, crystal structure

1. Introduction

Nanocrystalline Fe-based soft magnetic alloys are among the most promising materials for modern electromagnetic devices owing to their combination of high saturation magnetisation, low coercivity, and reduced core losses. Since the development of FINEMET-type alloys, extensive research has focused on tailoring nanocrystalline microstructures to maximise magnetic performance through controlled crystallisation processes [1,2].

Among recently developed soft magnetic materials, Fe–Si–B–P–Cu alloys, known as NANO-MET-type alloys, exhibit exceptionally high saturation flux density while maintaining excellent soft magnetic characteristics [3]. Their performance originates from the formation of α -Fe nanocrystals embedded within a residual amorphous matrix. The volume fraction, size, and distribution of nanocrystals strongly influence domain-wall motion, magnetic anisotropy, and exchange coupling, which collectively determine the resulting magnetic properties [2,4].

The crystallisation behaviour of NANOMET alloys differs significantly from conventional Fe-based nanocrystalline systems because phosphorus remains preferentially in the amorphous matrix and suppresses excessive grain growth. Consequently, subtle variations in annealing conditions can substantially modify the final microstructure and magnetic response [3].

In the present work, the relationship between structural evolution and magnetic properties of melt-spun $\text{Fe}_{81}\text{Ni}_2\text{Cu}_1\text{Si}_4\text{B}_8\text{P}_4$ ribbons was investigated. Particular attention was devoted to monitoring the nanocrystallisation process by in-situ transmission electron microscopy and correlating the resulting microstructural changes with coercivity, magnetic induction, and core losses obtained after different annealing treatments.

2. Results & discussion

The as-spun $\text{Fe}_{81}\text{Ni}_2\text{Cu}_1\text{Si}_4\text{B}_8\text{P}_4$ ribbon exhibited a predominantly amorphous structure with a small fraction of primary α -Fe crystallites, as confirmed by XRD and TEM observations. Lorentz TEM imaging revealed a homogeneous magnetic-domain configuration with minimal

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domain-wall pinning, indicating that the amorphous precursor already possesses favourable soft-magnetic characteristics.

Direct observation of the crystallisation process during in-situ TEM heating provided insight into the evolution of the microstructure responsible for the alloy's magnetic behaviour. The onset of α -Fe nucleation was observed at approximately 350°C, while the temperature range between 380 and 400°C was identified as the optimum region for nanocrystalline refinement. Within this interval, a high density of α -Fe nanocrystals formed without pronounced grain coarsening. Further temperature increases promoted grain growth and the formation of Fe_3B phases.

The observed microstructural evolution directly influenced the magnetic properties. Annealing treatments that produced a homogeneous nanocrystalline structure resulted in a significant reduction of coercivity due to enhanced exchange coupling between neighbouring nanocrystals and suppression of local magnetic anisotropy. A nearly linear temperature-time relationship defining the minimum annealing conditions was identified between 340°C/20 h and 500°C/1 min, with the global coercivity minimum obtained after annealing at 400°C for 20 min.

XRD analysis of the optimally annealed ribbon revealed an average α -Fe crystallite size of approximately 31 nm. This microstructure corresponded to the best magnetic performance, demonstrating that the balance between nanocrystal formation and grain growth is a critical factor governing the soft magnetic properties of the alloy.

3. Conclusions

The present study demonstrates a clear correlation between structural evolution and magnetic properties in $\text{Fe}_{81}\text{Ni}_2\text{Cu}_1\text{Si}_4\text{B}_8\text{P}_4$ ribbons. In-situ TEM observations revealed that nanocrystallisation begins near 350°C and proceeds most effectively within the temperature range of 380–400°C, where a refined nanocrystalline microstructure is formed.

The lowest coercivity and best soft magnetic performance were obtained after annealing at 400°C for 20 min, corresponding to an average α -Fe crystallite size of approximately 31 nm. The results indicate that the development of a homogeneous nanocrystalline structure promotes magnetic softening by enhancing exchange interactions and reducing effective magnetic anisotropy.

The observed relationship between crystallisation behaviour, microstructure formation, and magnetic response confirms that precise control of nanocrystallisation is essential for optimising the performance of $\text{Fe}_{81}\text{Ni}_2\text{Cu}_1\text{Si}_4\text{B}_8\text{P}_4$ ribbons for advanced soft magnetic applications.

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Correlating crystallographic microstructure and magnetic domains in Fe-Pd alloys

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Keywords: electron holography, Lorentz microscopy, twin boundary, antiphase boundary, magnetic domain

1. Introduction

Fe-Pd alloys in the eutectoid region (in the vicinity of 62 at.% Pd) form various crystallographic microstructures consisting of ordered ferromagnetic phases [1]. For example, Fe-61.8 at.% Pd exhibits a two-phase $L1_0+L1_2$ microstructure in which the major tetragonal $L1_0$ phase forms a hierarchical polytwin structure, while the minor cubic $L1_2$ phase exists as nanometer-scale wetting layers at the structural interfaces, i.e., twin boundaries (TBs) and antiphase boundaries (APBs) of the $L1_0$ phase [2]. Conversely, Fe-62.2 at.% Pd alloy forms a two-phase $L1_2+L1_0$ microstructure consisting of predominantly isolated $L1_0$ polytwin plates embedded within an $L1_2$ matrix [3]. Given the distinct magnetic properties of the $L1_0$ and $L1_2$ phases (i.e., strong uniaxial magnetic anisotropy for the former and weak cubic magnetic anisotropy for the latter [4]), the correlation between magnetic domain phenomena and the underlying crystallographic microstructure is intriguing for tailoring magnetic properties via microstructural design. However, due to the intricate three-dimensional microstructures featuring dense internal twin layers tens of nanometers thick and structural interfaces only a few nanometers thick, structural characterization, magnetic domain observation, and the analysis of their correlations remain challenging. This study aims to investigate the effects of crystallographic microstructure on magnetic domains in Fe-Pd alloys using transmission electron microscopy (TEM) techniques.

2. Materials and Methods

Fe-61.8 at.% Pd and Fe-62.2 at.% Pd bulk alloys were fabricated, and TEM specimens were prepared by electrochemical polishing at the University of Virginia. The bulk samples were synthesized via arc melting, cold rolling, recrystallization, homogenization, water quenching, and isothermal aging [2,3]. TEM and scanning transmission electron microscopy (STEM) observations were performed using a conventional TEM (JEM-2100HCKM, JEOL Ltd.) and an aberration-corrected 300-kV microscope (JEM-ARM300F2, JEOL Ltd.). The microscopes were operated at an accelerating voltage of 200 kV to suppress radiation damage

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to the ordered structures. To observe the magnetic domains and magnetic flux distributions, Lorentz microscopy and electron holography were conducted using another TEM (HF-3300X, Hitachi High-Tech Ltd.) operated at an accelerating voltage of 300 kV, equipped with a magnetic-field-free specimen stage and a double biprism system [5]. In electron holography, both the electrostatic potential and the magnetic field induce a phase shift in the incident electrons. To separate the magnetic field contribution from the electrostatic potential contribution, a set of electron holograms (electron interference fringe images) was acquired before and after inverting the specimen [6].

3. Results and Discussions

Figure 1(a) shows a TEM image of the typical polytwin microstructure observed in the Fe-61.8 at.% Pd alloy. A magnified high-resolution high-angle annular dark-field (HAADF) STEM image of an $L1_0$ variant (Variant 1) in Figure 1(b) reveals the atomic arrangement near the TBs, observed along the $[100]$ zone axis. The HAADF-STEM image shows that the thickness of the $L1_2$ layers is only a few atomic layers; they appear as vertical bright lines (indicating Pd enrichment) at the TBs, as highlighted by red arrowheads. A bright horizontal line in Figure 1(b) represents an APB where the geometric phase of the $L1_0$ -type structural order is inverted. In comparison, the polytwin microstructure in the Fe-62.2 at.% Pd alloy, consisting of individual $L1_0$ polytwin plates dispersed in an $L1_2$ matrix, is observed along the $[111]$ zone axis (not shown). The Lorentz micrograph in Figure 1(c) visualizes magnetic domain walls, most of which are located along the TBs. These domain walls are identified as 90-degree walls, as indicated by the zig-zag contour lines observed via electron holography in Figure 1(d). The phase contour lines in Figure 1(d) represent the phase shift caused by the magnetic field and approximate the direction of the magnetic flux lines. The presence of a 180-degree wall crossing over many $L1_0$ twin plates is also observed, as shown in Figures 1(c) and (d). Notably, the APBs shown in Figure 1(c) exhibit negligible magnetic contrast, suggesting they may not serve as pinning sites for magnetic domain walls. The TEM results are discussed in relation to the effects of the crystallographic microstructure on magnetic domain configurations.

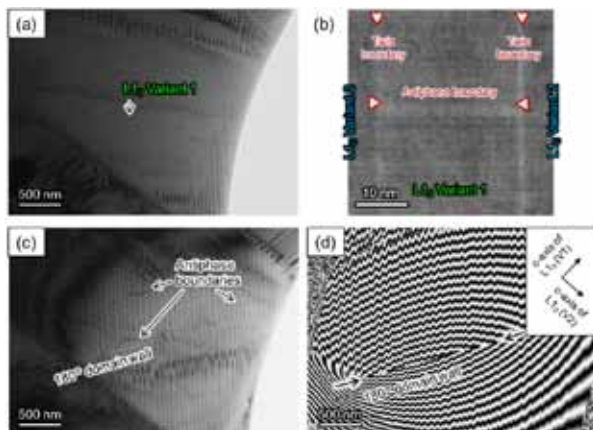


Figure 1. (a) TEM image of the polytwin microstructure in the Fe-61.8 at.% Pd alloy.
 (b) HAADF-STEM image obtained around the intersection of TBs and an APB.
 (c) Lorentz micrograph of magnetic domain walls.
 (d) Magnetic phase contour map observed by electron holography.

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Helpi Optics – A New Player in Electron Microscopy and...

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Helpi Optics is a Polish company providing advanced microscopy and imaging solutions for research and industrial applications. Our portfolio covers a broad range of techniques, including optical microscopy, confocal microscopy, atomic force microscopy and electron microscopy.

During this presentation, we will introduce CIQTEK, a new electron microscopy brand entering the Polish market, and present its portfolio of electron microscopy solutions. The portfolio covers a wide range of applications and system configurations, from tungsten-filament scanning electron microscopes (SEM) for routine imaging and materials characterization, through high-resolution field-emission SEM (FE-SEM) systems, to advanced dual-beam FIB-SEM instruments. We will also briefly introduce CIQTEK's transmission electron microscopy (TEM) solutions.

The presentation will highlight the key capabilities and application areas of the different systems and discuss how electron microscopy can be complemented by optical microscopy and AFM to provide a more comprehensive approach to sample characterization.

Our aim is to introduce CIQTEK to the microscopy community in Poland and demonstrate how its portfolio can address a broad range of imaging and characterization requirements, from routine microscopy to advanced research applications.

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STEM-ELNES mapping via final-state orbital selection

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Keywords: STEM, EELS, ELNES, orbital

Understanding chemical bonding at the atomic scale is essential for further advances in materials science. Electron energy-loss spectroscopy (EELS), when combined with (scanning) transmission electron microscopy ((S)TEM), provides a powerful approach for probing local electronic structures with atomic resolution. To date, atomic-resolution elemental mapping and electronic-state analysis have been successfully demonstrated, including the visualization of variations in valence and chemical bonding. These developments have greatly contributed to elucidating the relationship between local atomic structure and material properties.

As a next step, direct visualization of electronic orbitals has become a major goal in the field of electron microscopy. This interest arises because the energy-loss near-edge structure (ELNES) in core-loss EELS reflects the unoccupied projected density of states (PDOS) of the excited atom and therefore contains detailed information on orbital symmetry and chemical bonding. In particular, theoretical studies have predicted that s–p excitation ELNES mapping can exhibit anisotropic contrast corresponding to the azimuthal distribution of the final-state single-particle orbitals [1,2]. However, the physical origin and interpretation of the observed anisotropic contrast remain controversial, since complex electron scattering processes in crystals can also influence atomic-scale image formation. In recent years, improvements in instrumental stability and detector sensitivity have enabled experimental studies on orbital-sensitive ELNES mapping to emerge [3–6], increasing the importance of correctly interpreting experimentally obtained images. In this study, we report experimental anisotropic atomic contrast and its theoretical interpretation in STEM-ELNES-based imaging of metal oxides, including SrTiO₃ and Rutile TiO₂.

In the present research, crushed crystals were observed using spherical aberration-corrected STEM (JEOL, JEM-ARM200F, operated at 60 and 200 kV. The probe convergence semi-angle α was 23.3 mrad. The EELS collection semi-angle was 54.8 mrad which was almost twice as large as the probe convergence angle. An ultra-high-quality dark reference spectrum estimated from the average of 20,000 dark spectra was subtracted to randomize dark noise [7], and the original gain correction for corresponding hardware binning was applied. The SI data were measured from a wide area. Then, in order to improve the signal-to-noise ratio, many crystallographically equivalent regions were accumulated using a nonrigid registration drift correction with the SMARTALIGN software package (HREM Research) [8]. Figure 1 shows results obtained from SrTiO₃ observed along [110] direction. Whereas oxygen atoms in O *K*-edge mapping constructed using wide energy window (Fig.1 (e)) appear nearly isotropic, ELNES mapping with selective excitation of specific final-state orbitals (Figs. 1(f) and (g)) exhibits pronounced anisotropic contrast at the atomic scale. Based on the STEM-EELS theory formulated by D.A. Muller [9], we investigated the theoretical interpretation of STEM-ELNES imaging. Our analysis revealed that STEM-ELNES does not directly visualize the electronic density of states itself, but rather reflects anisotropy corresponding to the orientation of

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the transition dipole moment determined by the initial and final states. In particular, for $s \rightarrow p$ orbital excitations, the direction of the transition dipole moment coincides with that of the final-state p orbital, allowing the anisotropy of the electron orbital to be experimentally captured.

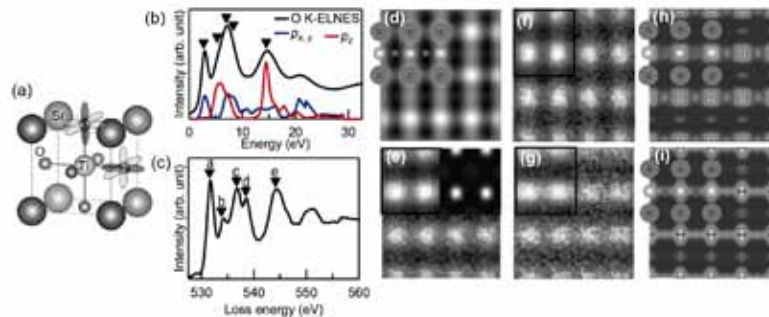


Figure 1. (a) Structural model of SrTiO₃. The O 2p orbitals can be classified into two groups: the orbitals distributed in the Sr-O plane, hybridized with the Ti 3d orbitals with t_{2g} symmetry, and the orbitals distributed in the direction of the Ti-O-Ti bond, hybridized with Ti 3d orbitals with e_g symmetry. (b) Simulated O K-ELNES and oxygen p PDOS for O1 sites. (c) Experimental O K-ELNES. (d) HAADF image along the [110] axis. (e) Oxygen elemental map using an energy window of 26 eV from the threshold of the O K-edge. O-ELNES mappings using (f) peak a, (g) peak b. Charge density maps for the unoccupied state of the (h) π^* -bond (3–4 eV) and (i) σ^* -bond (5–6 eV).

In this presentation, we discuss the contrast in STEM-ELNES mapping and present a theoretical analysis of how this contrast is related to the final-state electronic orbitals.

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Amorphous High Entropy Alloys

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Keywords: amorphous alloys, medium and high entropy alloys, mechanical alloying

High and medium entropy alloys (HEAs, MEAs) are relatively new class of materials elaborated at the beginning of XXI century. The first equimolar CoCrFeMnNi alloy, so called Cantor alloy, described in 2004, revealed simple structure of single phase solid solution instead of complex phases expected. Nowadays, many multicomponent alloys consisting of various elements are studied, resulting in a wide range of their structures, properties and applications. The entropy-based definition, separates low ($S < R$, where S is the total configurational molar entropy in an ideal solid solution and R is the gas constant), medium ($R < S < 1.5R$) and high ($S > 1.5R$) entropy alloys. For further characterization of these alloys the following parameters, besides entropy ΔS , are used: enthalpy of mixing ΔH , Zhang parameter Ω (combining entropy, enthalpy and melting temperature) and δ parameter related to the differences in atomic radii. Additionally, valence electron concentration (VEC) value is considered. However, in some cases, the alloys which fulfil all HEAs parameters, simultaneously undergo amorphization during the solid state processing. This work is devoted to the studies of amorphous high and medium entropy alloys manufactured by mechanical alloying. Literature based examples of different multicomponent alloys currently explored and studied, and finally, the results of own experiments on CoCuFeNiX system are discussed.

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What We Don't Want to See:

How Local Thermal History Creates Microstructural and Mechanical Heterogeneity in Additive Manufacturing

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Keywords: additive manufacturing, laser engineered net shaping (LENS), thermal history, microstructure, mechanical properties

Additively manufactured components are frequently described and analysed as homogeneous materials characterized by a single set of mechanical properties. In reality, however, each location within a component experiences a different sequence of rapid heating, melting, solidification, cooling, and repeated reheating. Consequently, the same nominal material may not be the same material everywhere within the manufactured part. This work investigates how local thermal history affects the microstructure, mechanical response, deformation, and residual stress distribution in metallic components produced by Directed Energy Deposition. Particular attention is given to the possibility of reconstructing the thermal history of selected locations within a deposited component and correlating it with locally measured material properties.

Temperature evolution during deposition was monitored using thermocouples, pyrometers, and a custom-developed infrared imaging system. The experiments revealed substantial challenges associated with temperature acquisition, including rapidly changing thermal gradients, limited spatial and temporal resolution, uncertain emissivity, restricted access to the melt pool, and the large volume of data generated during the process. For this reason, experimental observations were combined with numerical simulations of heat transfer and mechanical response. The model was validated using temperature measurements and substrate displacement recorded by Digital Image Correlation. The results demonstrate that apparently uniform additively manufactured components may contain regions with distinctly different thermal histories and, consequently, different microstructures, mechanical properties, and residual stress states. Such variations may occur not only close to the substrate or the free surface but also between locations separated by relatively short distances within the same component.

This approach uses thermal monitoring to study how material forms at different locations in the component. Reconstructing the thermal history of each location can help predict local properties, identify critical regions, and better control material heterogeneity during additive manufacturing.

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Role of inorganic fillers in stabilizing EPDM elastomer under ion irradiation

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Keywords: elastomers, ion irradiation, oxidative degradation

Nuclear power plants (NPPs) play a crucial role in supplying low-carbon, reliable and affordable electrical energy worldwide. Electrical cables are vital components of nuclear power plant (NPP) instrumentation and control systems. Cable insulation is made from various polymeric-based compounds. The long-term exposure of the cable insulation to the standard operational environmental conditions of NPP such as radiation, heat and humidity, cause aging and degradation of their functional properties. From the total cable structure, insulation experiences the most severe degradation of mechanical and electrical properties. Under exposure to high temperature and gamma-radiation, the dominant mechanisms are chain scission, cross-linking of polymer backbone and formation of free radicals with occurrence of oxygen. The improvement in the oxidation stability of macromolecular structures can be achieved by several approaches: addition of antioxidants, crosslinking, inclusion of proper nanohybrids.

The primary objective of this study was to develop an elastomer based on ethylene-propylene–diene rubber (EPDM) containing inorganic fillers to enhance its resistance to ionizing radiation. The EPDM material is intended for use as cable insulation in radiation-exposed environments such as nuclear power plants, where ionizing radiation is one of the main factors responsible for insulation degradation. The mechanism of radiation interaction depends on the macromolecular structure of the elastomer. Ionizing radiation can induce chain scission of macromolecules, resulting in the formation of shorter radical-terminated fragments. Subsequent radical recombination leads to crosslinking, manifested as increased hardness, brittleness, and shrinkage of the material. To simulate the effects of ionizing radiation on cable insulation, the material was irradiated with He⁺ ion beams. Ion beam irradiation modifies the chemical and physical properties of elastomeric materials in a manner similar to gamma radiation, with inelastic ion–electron interactions providing a suitable analogue for studying radiation effects. Consequently, ion irradiation allows the simulation of radiation damage typically observed under nuclear reactor conditions.

Characterization of the modified materials was performed using scanning electron microscopy (SEM), transmission electron microscopy (TEM), Raman spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and atomic force microscopy (AFM). These analyses enabled the assessment of radiation-induced changes in the structural and chemical properties of the EPDM-based

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insulation. The results provide valuable insights into the mechanisms of radiation interaction with elastomeric materials and contribute to the development of improved cable insulation systems for long-term operation in radiation-exposed environments, particularly in nuclear power applications.

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AFM-in-SEM *in-situ* Correlative Microscopy with LiteScope: Technology and Applications

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Keywords: AFM-in-SEM, correlative microscopy, conductive AFM, SSRM, failure analysis, battery characterization

1. Introduction

Atomic force microscopy (AFM) and scanning electron microscopy (SEM) are complementary techniques: SEM provides fast, wide-field 2D imaging and elemental analysis via energy-dispersive X-ray spectroscopy (EDS), while AFM delivers sub-nanometer surface topography and quantitative local measurements of electrical, mechanical, and magnetic properties. Used independently, each technique requires dedicated sample transfer and re-navigation to the region of interest (ROI), introducing positional uncertainty and risk of sample contamination or damage.

LiteScope (NenoVision, Brno, Czech Republic) is a compact AFM module designed to operate inside the vacuum chamber of any SEM or FIB-SEM system, enabling true in-situ correlative analysis on the identical sample area without stage repositioning. The system integrates a miniaturised AFM scanner, the NenoBox signal controller, and NenoView software, which synchronises AFM and SEM data acquisition in real time. The key enabling concept is Correlative Probe and Electron Microscopy (CPEM): simultaneous AFM and SEM imaging on the same location, producing spatially registered multimodal datasets from a single scan.

2. Technology

LiteScope supports a broad range of AFM measurement modes including conductive AFM (C-AFM), scanning spreading resistance microscopy (SSRM), magnetic force microscopy (MFM), piezoresponse force microscopy (PFM), Kelvin probe force microscopy (KPFM), electrostatic force microscopy (EFM), scanning tunnelling microscopy (STM), phase imaging, and force-distance (F-z) spectroscopy. The instrument is compatible with the majority of SEM or FIB-SEM platforms and can operate at full 52° stage tilt, keeping the sample in the FIB-SEM coincidence point. This eliminates stage movements between successive delayering and characterisation steps, enabling streamlined layer-by-layer 3D analysis workflows.

A critical workflow advantage is the SEM-guided tip navigation: after the ROI is identified in the SEM overview image, the AFM probe is driven to that location with nanometric precision while keeping exceptional control over the tip. One-click automatic tip retraction and return further accelerates iterative FIB-AFM cycles.

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3. Application Examples

The added value of AFM-in-SEM correlative microscopy is illustrated across four application fields. In each case, the key benefit is the ability to acquire complementary structural and functional data from exactly the same location – something that cannot be reliably achieved when the two instruments are used separately.

3.1. Semiconductor Failure Analysis

Modern semiconductor failure analysis demands localisation of defects within increasingly dense interconnect stacks, where SEM alone cannot distinguish electrically active from inactive structures. By combining plasma FIB (PFIB) delayering with in-situ C-AFM, each exposed layer can be mapped for conductivity immediately after milling – without moving the sample or breaking vacuum. The result is a layer-resolved 3D electrical map that identifies open or leaking contacts directly within their structural context. In-situ SSRM extends this capability to dopant profiling, resolving p/n junction geometry and doping gradients at the nanometre scale on the precise cross-section defined by FIB – a workflow that is practically impossible with ex-situ AFM.

3.2. Battery Research

Understanding battery degradation requires connecting microstructural changes to local electrical performance. AFM-in-SEM uniquely enables this by mapping the conductivity of individual cathode particles on the same cross-section imaged by SEM and EDS. Cycling-induced particle cracking and contact loss – invisible in morphological images – become directly visible in C-AFM current maps, with chemical identity confirmed simultaneously by EDS. This correlation between structure, composition, and local electrical behaviour in a single session provides a uniquely complete picture of degradation at the single-particle level.

3.3. Materials Science and Life Science

In materials science, SEM navigation allows the AFM probe to be directed precisely to a selected particle, grain boundary, or phase, turning a time-consuming search into a targeted measurement. This is demonstrated for roughness characterisation of complex microparticles and for mechanical phase mapping of polymer composites, where local stiffness is correlated directly with SEM-visible microstructure. In life science, fragile structures – protein nanowires, drug-carrier particles, or cells on scaffolds – are first identified by SEM before any AFM contact is made, minimising sample damage and maximising the probability of measuring the intended feature.

4. Conclusions

In-situ AFM-in-SEM correlative microscopy substantially reduces analysis time, eliminates sample transfer artefacts, and provides multimodal information inaccessible by either technique alone. The combination of SEM-guided navigation with quantitative AFM modes – C-AFM, SSRM, and F-z spectroscopy – addresses demanding characterisation challenges in semiconductor failure analysis, energy storage research, materials science, and life science. The presented use cases demonstrate that the LiteScope platform is a versatile tool for nanoscale characterisation wherever spatial correlation between morphology and local physical properties is required.

Tackling the challenges of novel materials analysis: Recent conceptual and technological advances in TEM

Reza Zamani
Thermo Fisher Scientific

Keywords:

Nowadays a great host of novel materials is being developed to address the current challenges we face in our society. The interest in applications such as green energy production, CO₂ capturing, catalysis, and advanced electronics has skyrocketed in recent years. However, understanding the functionality of these materials is still challenging and often requires reliable characterization methodology which preserves the material's integrity while its properties are examined. In this context, transmission electron microscopy (TEM) offers a wide range of new capabilities for studying the novel materials at the atomic scale.

At Thermo Fisher Scientific we continuously develop innovative solutions, in order to (i) expand the capabilities to wider ranges of information, e.g. magnetic/electrical properties, phononics and plasmonics, chemical composition, and structures at the atomic level, (ii) expand the aforementioned capabilities to a wide range of specimens from robust to highly electron-beam-sensitive, e.g. polymers, zeolites and MOFs, and on the other hand, (iii) avail TEM to a broader group of researchers, by providing fully-integrated easy-to-use reproducible platforms with imbedded automated features. In this contribution, we present the most recent advances aimed at achieving these goals. In particular, we focus on newly developed fully-integrated EELS solutions as well as other concepts and technologies for preserving specimen integrity. These are meant to provide the user-friendly all-in-one TEM platform the scientists need in order to explore the properties of novel materials at their native state.

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Radiation-induced metastable ordered phase in $\delta\text{-Sc}_4\text{Hf}_3\text{O}_{12}$ and its stability

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Keywords: fluorite structural derivatives, order-to-disorder phase transformation, ion irradiation

1. Introduction

Because of their thermal stability, chemical durability, and ability to incorporate minor actinides into their lattice sites, crystalline ceramics are a promising alternative to borosilicate glass for immobilizing high-level radioactive waste [1]. These materials are exposed to harsh radiation environments, and therefore knowledge of irradiation-induced structural changes is important for predicting the fate of the immobilization materials. Due to their excellent radiation resistance, fluorite-structured oxide ceramics are effective materials for the disposal of high-level radioactive waste. Their electrical and thermal properties can be controlled by substituting partial cation sites with other ions. When the cation's valence differs, vacancies are introduced into the anion sites to achieve electrical neutrality. Thus, the irradiation behavior of fluorite structural derivatives, where atoms are arranged regularly on cation and anion sublattices, has been extensively studied. Pyrochlores, in which atomic ordering occurs in both the cation and anion sublattices, are known to undergo an order-to-disorder phase transformation due to the accumulation of damage induced by irradiation. However, recent studies using neutron diffraction have demonstrated that a weberite-type phase emerges as a short-range ordered state [2]: the disordering process is more complex than previously thought.

There are several fluorite structural derivatives, β -, γ -, and δ -type phases with ordered oxygen vacancies and disordered cation sublattice in the $\text{Sc}_2\text{O}_3\text{-HfO}_2$ and $\text{Sc}_2\text{O}_3\text{-ZrO}_2$ pseudobinary systems. These compounds are the ideal playground to understand the radiation effects on the anion sublattice in systems where the cation sublattice can be kept almost perfectly disordered. In this study, we investigated the order-to-disorder phase transformation in ion irradiated $\delta\text{-Sc}_4\text{Hf}_3\text{O}_{12}$ using transmission electron microscopy [3–5].

2. Experimental

Polycrystalline $\delta\text{-Sc}_4\text{Hf}_3\text{O}_{12}$ pellets were irradiated with swift-heavy ions at room temperature using IRRSUD beamline at GANIL (Grand Accélérateur National d'Ions Lourds) in Caen, France. Under this condition, a significant damage region was formed from the surface to a depth of $\sim 8\text{ }\mu\text{m}$. To examine radiation-induced structural changes, the pellets were fabricated into cross-sectional TEM specimens using a focused ion beam (FIB) with a Thermo Fisher Scientific FIB-SEM Scios 2, or via Ar ion milling with a Gatan PIPS Model 691. We observed the TEM samples using

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a JEOL JEM-3000F and a JEM-F200, which operated at 300 and 200 kV, respectively. Imaging plates with high sensitivity, good linearity, and a wide dynamic range for electron intensities were used as recording media to detect weak signals.

3. Results and discussion

Figure 1 shows the selected-area electron diffraction patterns of δ - $\text{Sc}_4\text{Hf}_3\text{O}_{12}$ irradiated with 92 MeV Xe ions to a fluence of 10^{14} cm^{-2} at room temperature. In addition to the fundamental lattice spots, the superlattice reflections due to the ordering of oxygen vacancies were observed in Fig. 1(a), which was taken from the low-damage region. This pattern was consistent with the (001) reciprocal lattice plane of the rhombohedral δ -type structure. In contrast, Fig. 1(b), which was taken from the high-damage region, showed spotty diffuse scattering at a different position to that of the superlattice reflections of the δ -phase. This pattern corresponds to the (111) reciprocal lattice plane of the cubic bixbyite (C-type rare earth) structure. This suggests that the long-range ordered δ -phase transforms into the short-range ordered bixbyite phase upon damage accumulation. It was confirmed that the bixbyite microdomains are dispersed in a disordered cubic fluorite matrix. No compositional change was observed between the δ and the bixbyite phases, suggesting that the structural change observed here is due to a congruent phase transformation. The stability of the bixbyite phase under thermal annealing and electron beam irradiation [5] will also be reported in the seminar.

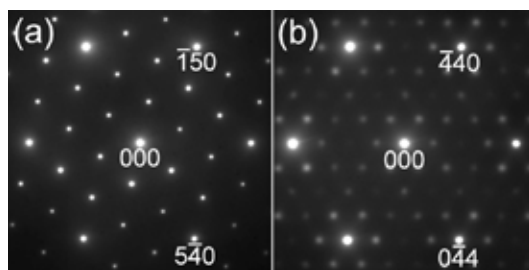


Figure 1. Selected-area electron diffraction patterns of δ - $\text{Sc}_4\text{Hf}_3\text{O}_{12}$ irradiated with swift heavy ions, taken from low-damage (a) and high-damage (b) regions.

In addition to the fundamental lattice reflections, superlattice reflections due to oxygen vacancy ordering are present in (a).

In (b), diffuse spots appear instead of the superlattice reflections.

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Effect of prolonged natural aging on clustering and precipitation behavior in an Al-Mg-Si alloy with excess Mg

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Keywords: natural aging, precipitation, cluster, (S)TEM

This study investigates the influence of natural aging (NA) on hardness evolution and clustering behavior in Al-Mg-Si alloy with ex Mg, using hardness measurements and scanning transmission electron microscopy (STEM). Hardness increases during NA, confirming that early-stage clusters are the primary source of strengthening. STEM observations reveal the formation of a square-ordered structure after 1 month NA and more numerous clusters in 1 year NA, demonstrating continued atomic ordering at room temperature. Transmission electron microscopy (TEM) analysis of peak-aged artificial aging (AA) conditions (only AA, 1 month NA+AA and 1 year NA+AA) identifies two types of elongated precipitates aligned along the $[001]_{Al}$ direction. The results show that NA strongly influences the precipitation behavior of the alloy.

1. Introduction

Al-Mg-Si (6xxx series) alloys represent an important group of heat-treatable materials. They are extensively applied in industry due to their favorable combination of properties, such as a high strength to weight ratio, good formability, corrosion resistance, and weldability [1]. The strength of these alloys is mainly developed through AA carried out at an appropriate temperature (typically between 150 and 200°C) for a suitable duration, following solution heat treatment and rapid quenching to produce a supersaturated solid solution (SSSS), during which many solute clusters and nano precipitates form. These precipitates are the main contributors to the strengthening of 6000-series alloys.

In practical manufacturing environments, components or sheets are frequently stored at room temperature (RT) for various logistical or intermediate processing steps prior to the application of AA. During this storage period, NA inevitably occurs as a result of solute atom diffusion even at ambient temperatures. This diffusion leads to the gradual formation and evolution of solute clusters, including early-stage atomic aggregates within the aluminum matrix [2]. The development of these solute clusters during NA alters the precipitation transformation pathway, thereby modifying the initial microstructural state of the alloy before AA. In this study, Al-Mg-Si alloy with excess

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Mg (ex. Mg) alloys (Mg + Si $\approx$ 2 mass%) were investigated. The clustering behavior during NA was systematically examined over different NA durations, and its influence on the subsequent AA response was evaluated. Microstructural evolution, including cluster development and precipitate formation, was characterized using STEM and correlated with hardness evolution. Through this combined approach, this study aims to elucidate how NA-induced clustering contributes to the negative effect of natural aging on precipitation behavior and strength at the peak-aged condition.

2. Experimental procedure

The as-cast ingots were first homogenized at 575°C for 36 ks and then rapidly quenched in ice water. The homogenized materials were then hot-rolled at 400 °C to a thickness of 10 mm, followed by cold-rolling to a final sheet thickness of 1.5 mm. The sheets were cut into 10 mm long specimens and solution heat-treated at 575°C for 3.6 ks, immediately followed by ice-water quenching. Next, samples were stored at 293 K for various times (after as. Q up to 1 year NA) and then AA was conducted at 200°C, the sample were directly aged to make comparison. Micro Vickers-Hardness measurement was measured using Mitutoyo HM-101 (loading of 0.98 N and holding time of 15 s). Small pieces with mass 25 mg and diameter 3 mm was used for DSC measurement with heating rate 10°C/min and TEM sample is cold rolled to 0.2 mm thick, then solid solution and aging treatment, after that polished to 0.08 mm thick mechanically and punched out a disc sheet with a diameter of about 3 mm, then use twin-jet for make samples (twin-jet polishing solution is nitric acid: methanol = 1:3).

Table 1. Chemical composition of the Al-Mg-Si alloy (mass%).

	Mg	Si	Al	Mg ₂ Si	Mg + Si	Ex Mg
Ex. Mg (wt.%)	1.58	0.56	Bal.	1.54	2.1	0.5

3. Results

Figure 1 presents STEM observations used to support the hardness results and to examine the precipitation microstructure in more detail. All images were taken at the peak-hardness condition with the electron beam aligned parallel to the [001] Al direction. Representative micrographs of the ex Mg alloy are shown for two conditions: direct ageing without NA and ageing after 1 month NA while the quantitative analysis of precipitate number density and length (shown in the plots) includes all NA conditions investigated. In all samples, needle-shaped and cross section precipitates were observed as the dominant hardening features. The sample aged without NA shows a high density of fine precipitates, consistent with its rapid hardening response. After 1 month NA, the precipitate population becomes noticeably coarser and less numerous, with many precipitates appearing longer and the length distribution becoming longer (typically on the order of ~10–50 nm) explaining the lower peak hardness after NA. This behavior corresponds to the well-known “negative effect of NA” as widely reported in the literature [3]. Especially, with sample 1 year NA and AA, can show the recovery in peak hardness, meanwhile the number density become more numerous and length become shorter, this trend agrees with the quantitative results: NA reduces precipitate number density and promotes increased precipitate length in 1 months and become recovery after 1 year of NA. STEM observation after 1 month of NA reveal the presence of square cluster. These clusters are relatively ineffective as strengthening, contributing to the reduced peak hardness observed at this stage. After 1 year of Na, these features evolve 3 β'' eyes which act as effective precursors for subsequent precipitation during artificial ageing. The formation of eyes cluster promotes a higher density of fine strengthening precipitates, thereby directly contributing to the recovery of peak hardness (this aspect is discussed in detail in the presentation).

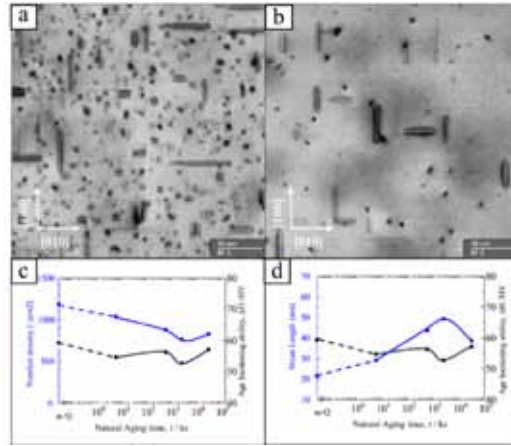


Figure 1. BF-STEM image in the sample ex Mg with (a) only AA and (b) after 1 month NA+AA. Corresponding with (c) the relationship between hardness and number density and (d) the relationship between hardness and length contribution.

4. Conclusion

In this study, the influence of NA on precipitation behavior and hardness in Al-Mg-Si alloys was systematically investigated, with particular focus on the well-known negative effect of NA and recovery with longer time NA when apply AA. The main conclusions are summarized as follows:

The reduction in peak hardness after 1 month of NA is associated with a less efficient precipitate state, characterized by a decrease in precipitate number density and the formation of longer, coarser precipitates. In contrast, after 1 year of NA, the peak hardness is largely recovered, accompanied by an increase in precipitate number density and a refinement in precipitate length.

STEM observations reveal that, after 1 month of NA, a higher fraction of square-shaped is present. These clusters are relatively ineffective for strengthening and contribute to the observed hardness loss. With prolonged NA (1 year), β'' eyes like structure was observed which act as effective precursors for subsequent precipitation during artificial aaging, thereby enabling the recovery of peak hardness.

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Shape memory effect driven by shallow energy wells

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Keywords: Fe-based shape memory alloys, molecular dynamics, deformation front, compatibility condition, Gibbs energy

1. Introduction

Iron-based shape memory alloys (Fe-based SMAs) are a promising class of smart materials for aerospace and space applications, offering low cost and large reversible strains driven by martensitic phase transformations and twinning-detwinning mechanisms. These properties make them highly suitable for compact, hydraulic-free actuators and heavy-load couplers, as well as morphing structures like adaptive aircraft wing profiles. Furthermore, Fe-based SMAs exhibit superior thermal stability compared to traditional NiTi alloys, opening doors for use in high-temperature environments such as jet engines and exhaust systems. However, space qualification requires overcoming functional degradation in superelasticity caused by dislocation activity, martensite variant cross-blockage, and residual deformations.

2. Methods

Large reversible strains in SMA single crystals are achieved through optimized chemical compositions, heat treatments, and prescribed boundary conditions – such as applied loads and kinematic constraints – which collectively dictate martensitic phase transformations. Cyclic shape recovery efficiency is governed by microstructural evolution within the moving deformation front. To identify the mechanisms of lattice reconstruction in this region, representative SMAs undergoing two complementary transformation paths – body-centered cubic (BCC) to face-centered cubic (FCC), and FCC to BCC/BCT – are studied under axial loading. The analysis is conducted for Fe–34Mn–15Al–7.5Ni (at. %) [1] and Fe–28Ni–17Co–11.5Al–2.5Ti (at. %) [2], which serve as prime examples of novel SMAs belonging to the first and second categories, respectively. To investigate the complex phenomena underlying transformation front formation and propagation, molecular dynamics (MD) simulations were performed. The interatomic interactions are described using a modified embedded-atom method (MEAM) potential developed by Sharifi and Wick (2025) [3]. This approach enables the total energy of the atomic system to be expressed as a function of atomic distances and angles, which is crucial for SMA modeling. The MD simulations are conducted using the Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) open-source software package [4].

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3. Results and Conclusions

Investigations reveal that uniaxial loading along a high-symmetry direction, combined with a uniform stress state of the opposite sign in the perpendicular plane, leads to the direct transformation of austenite (γ) into martensite (α'). Such conditions arise after exceeding the elastic range, when deformation proceeds at a constant volume. Specifically, compression along the four-fold axis $\langle 001 \rangle_\gamma$ and simultaneous tension in the plane $\{001\}_\gamma$ induces BCT martensite in Fe-28Ni-17Co-11.5Al-2.5Ti alloy through shear $\langle 011 \rangle_\gamma \{011\}_\gamma$, leaving a characteristic trace on the sample surface at an angle of 45° . Similar loading defined by the three-fold symmetry axis $\langle 111 \rangle_\gamma$ also generates the phase transformation, but it requires higher Gibbs energy; consequently, the cyclic strains are significantly lower than those in the $\langle 001 \rangle_\gamma$ orientation. Numerical simulations demonstrate that this orientation-dependent transformation behavior can be exploited to capture stable martensite within the $\langle 111 \rangle_\gamma$ crystal to act as a starting interface for transformation in the adjacent $\langle 001 \rangle_\gamma$ grain of the bicrystal. This minimizes dislocation accumulation and variant interactions, yielding stable and enhanced reversible strains within the $\langle 001 \rangle_\gamma$ region.

The results obtained for Fe-28Ni-17Co-11.5Al-2.5Ti SMA are used to explain the formation of a complex deformation front in an Fe-28Ni-17Co-11.5Al-2.5Ta-0.5B (at %) SMA single crystal subjected to compression along the $\langle 001 \rangle_\gamma$ direction. Experiments conducted at the temperature of 77 K demonstrate that the martensitic transformation interface consists of a combination of martensite variants, specifically, a target variant paired with a compliant variant orientation [5]. Under compressive loading, the crystal lattice selects shear directions that minimize system strain energy during phase transformation. Through this self-accommodation mechanism, the material utilizes the compliant variant to establish geometric compatibility between the austenite and the final martensite phase. Mathematical analysis of this lattice adjustment confirms that achieving high recoverable strain in the targeted phase requires a low-energy solution under applied stress.

MD simulations demonstrate that when the external loading condition applied to Fe-28Ni-17Co-11.5Al-2.5Ti SMA is switched from axial compression to tension, the deep Gibbs free energy wells governing the forward $\gamma \rightarrow \alpha'$ transformation transition into shallow energy minima. This fundamental alteration of the free energy landscape drastically lowers the thermodynamic barrier for phase boundary migration. Consequently, it minimizes lattice friction and dislocation accumulation, which directly enhances both strain reversibility and functional superelasticity during tensile cycles. This transformation path asymmetry can similarly be exploited in the Fe-34Mn-15Al-7.5Ni system; however, the directional dependency is entirely inverted. For this Fe-Mn-Al-Ni alloy, axial compression serves as the favorable loading direction, smoothly facilitating the reverse $\alpha \rightarrow \gamma'$ structural transformation.

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Capabilities of Data Layout Redesign and Host-Device Memory Trade-offs in the Effectiveness of the Cellular Automata Static Recrystallization Model GPU Parallelisation

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Keywords: static recrystallization, cellular automata, parallel computing, GPU computing, CUDA

1. Introduction

Static recrystallization is the microstructural evolution process in which the stored deformation energy introduced during plastic deformation is released by the nucleation and subsequent growth of new, strain-free grains. Given its complex kinetics, static recrystallization is often studied using computational methods, among which cellular automata (CA) have proven particularly effective. In a CA model, the material is represented as a two- or three-dimensional lattice of cells, each carrying information such as stored energy, crystallographic orientation, or phase. At each simulation time step, the state of each CA cell is updated according to transition rules that replicate recovery, nucleation, and recrystallization phenomena, based on the cell's current state and its neighbours' states.

However, due to the discrete nature of this approach, achieving a physically valid representation of microstructural evolution makes the simulation computationally expensive, which limits its industrial applications. A CA space consisting of several hundred CA cells along each edge already contains tens of millions of cells, and each must be updated at every time step. Earlier authors work on this issue focused on speeding up the CPU implementation [1]. Data structures were simplified, redundant calculations were removed, and the way CA cells and their neighbours were stored in memory was reorganised to reduce CPU overhead. These changes brought a significant speedup, but remained limited by the sequential nature of CPU execution.

This means that the same computation can, in principle, be carried out simultaneously for every CA cell, making the simulation a natural candidate for GPU parallelisation. On a GPU, thousands of cells can be updated concurrently instead of sequentially, offering a further speedup beyond what CPU optimisation can provide [2].

This work presents the redesign of the simulation data structures and memory layout for GPU execution, discusses the resulting implementation challenges, and summarises the remaining limitations and directions for further development.

2. GPU parallelization

The original CA SRX code stored the simulation grid as a four-level array of defined Cell objects. Each CA cell stored its own state, along with pointers to its neighboring cells, in an array

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of structure representations. This representation was restructured into a structure-of-arrays layout [3,4]. Simulation parameters that remain constant throughout a time step, including phase, boundary flag, and initial stored energy, are stored in dedicated static arrays. Data fields that are updated at every step, such as id, stored energy, and recrystallised fraction, are held in a double buffer, with one array per field for each buffer half. CA cells are addressed through a single linear index computed from their three-dimensional position, and the physics functions access the buffer arrays through a lightweight structure of pointers, usable on both CPU and GPU [4].

Advancing one simulation time step no longer requires copying the entire grid, as the original implementation did. At the beginning of each time step, only the dynamic fields arrays are copied from the read buffer to the write buffer. Such an operation addresses only the per-cell fields that change during a step, rather than entire cell objects and their neighbour pointers. As a result, each simulation phase (namely, recovery, nucleation, and recrystallisation) updates its own values in the write buffer. The two buffer halves are swapped in constant time at the end of each step. Although this reorganization alone does not significantly improve code performance, it removes the pointer indirection and per-object overhead that would otherwise make the same physics unsuitable for GPU kernels. It also allows the same per-cell functions to be compiled for both host and device execution.

2.1. Architecture

Each phase of a simulation and the results statistics part were originally executed on the CPU as a sequential traversal of the full CA space, updating one CA cell at a time before moving to the next phase (Figure 1a). Thus, the implementation was restructured around a different architecture, built on the same physical model and the same sequence of phases, but executing each phase differently. In the GPU model, one kernel is launched per phase from the host, and within each kernel, every CA cell is updated in parallel by a GPU thread, using the structure-of-arrays layout described in the previous section [5].

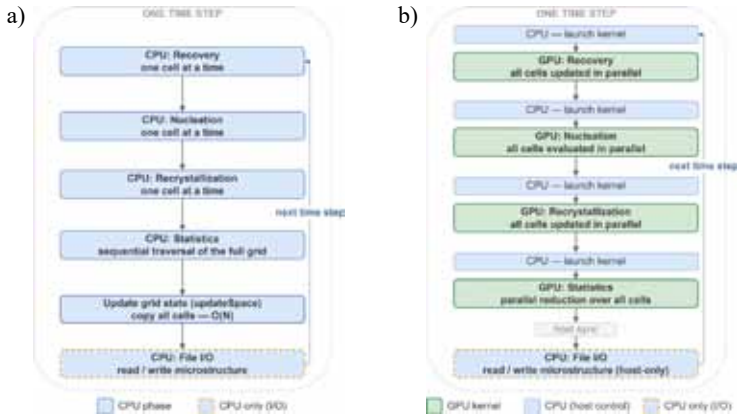


Figure 1. Architecture of one simulation time step. (a) Baseline, all phases run on the CPU and traverse the full grid sequentially, one cell at a time. (b) Implemented GPU code where the host launches one kernel per phase, and all CA cells are updated in parallel inside every kernel.

The kernels for each phase are launched sequentially on the same stream, since consecutive phases can write to the same per-cell fields, such as stored energy; running them in order

preserves this dependency without an explicit synchronization step [6]. The result statistics phase is executed as a single GPU kernel that computes aggregate quantities, such as total stored energy and the recrystallised fraction, along with a hash-based histogram of grain sizes. The CPU remains responsible for coordinating kernel launches, accumulating results into the time series written to disk, and handling file I/O itself, since none of this benefit from running on the device. A synchronisation point brings host and device into a consistent state whenever the CPU needs the current values (Figure 1b).

2.2. Memory model and remaining challenges

GPU parallelisation of a CA requires a clear policy for where space data reside and when they are moved between the host and the device. Three approaches are available: ordinary host memory, explicitly allocated device memory for the GPU, and unified memory, which exposes the same pointer to both the CPU and the GPU and relies on the driver to migrate pages on demand. Unified memory was chosen as the starting point because it simplifies integration: initialisation, file I/O, and occasional host-side access can use the same buffers as the GPU kernels. This solution comes with a cost, since the recrystallization phase performs many irregular reads of neighbor cells, and the dynamic fields are copied in full from the read buffer to the write buffer at every time step before the phase kernels run, so driver managed page migration and per-step synchronisation currently account for a large share of the runtime [6]. The natural next iteration keeps the space resident on the device for the entire simulation loop and uses explicit transfers only for loading the initial state and exporting results, removing host involvement from the hot loop while preserving the same double-buffer semantics and physics [7].

The same logic applies to two other parts of the implementation. CA cells do not keep a pre-computed list of their neighbors, so each phase recomputes neighbor positions on the fly, an access pattern that is being tuned as more of the pipeline moves to the device. The hashing strategy used for the grain-size histogram follows the same path, with its size and collision behaviour adjusted as the grid resolution used in testing increases.

3. Summary

This work presented a redesign of a cellular automata model for static recrystallization to run on the GPU. The CA space is now stored in a structure of arrays layout with flat indexing and a double buffer, and every phase of the simulation: recovery, nucleation, recrystallization, and result statistics including the grain size histogram, runs as a single-pass GPU kernel integrated into the simulation loop, with every CA cell updated in parallel within each phase. The CPU retains only the parts of the workflow that do not benefit from running on the device, namely coordinating kernel launches, accumulating results into the time series written to disk, and reading and writing the microstructure to file.

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Development of soft magnetic FeCoNi-based high-entropy alloys with single fcc phase and enhanced magnetic properties

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Keywords: high-entropy alloys, soft magnetic materials, single phase fcc structure

1. Introduction

Although extensive research has been conducted on soft magnetic materials (SMMs), a vast number of potential alloys particularly within the compositional space of high-entropy alloys (HEAs) remains unexplored. Among these, alloys based on the FeCoNi system are of significant interest [1]. Common alloying additions include Al and Si whose influence on phase formation and magnetic properties has been studied in numerous publications [2,3]. Nevertheless, significant region of the FeCoNiSiAl compositions have been yet to be systematically examined. The present study focuses on the design and development of a series of previously unreported soft magnetic FeCoNiSiAl HEAs exhibiting ultra-low coercivity and a single-phase fcc structure. Candidate compositions were selected using thermodynamic calculations and subsequently synthesized by suction casting. The proposed approach aims to expand the range of FeCoNi-based soft magnetic HEAs and provide new insights into the relationship between composition, phase stability, and magnetic performance.

2. Results and summary

The subject of this study was a series of suction-cast high-entropy alloys from the Fe–Co–Ni–Si–Al system. The alloy compositions were designed according to the methodology described in [4]. First, a base composition (S1) was selected. Subsequently, the contents of Fe, Co, and Ni were individually increased to obtain alloys S2–S4 while maintaining a predominantly single-phase microstructure. In this way, the influence of each of these elements on the properties and microstructure of the base S1 alloy could be systematically evaluated. In addition, all selected compositions were required to satisfy the thermodynamic criteria (Table 1) associated with the formation of stable single-phase high-entropy alloys.

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Table 1. Calculated phase stability parameters for the investigated alloys.

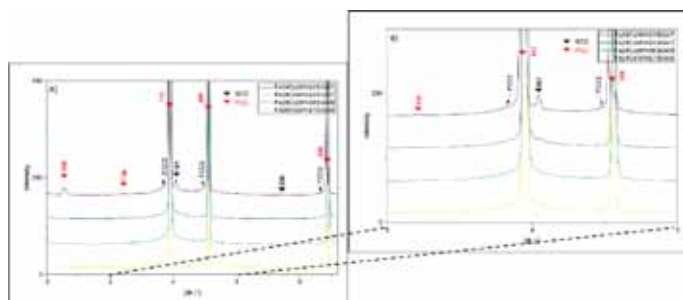
Name	Alloy	$\Delta_{\text{mix}}H$ kJ/mol	$\Delta_{\text{mix}}S$ kJ/mol	T K	Ω	δ %	VEC	$\Delta\chi$	ρ_t g/cm ³
S1	Fe ₂₉ Co ₃₅ Ni ₂₇ Si ₃ Al ₆	-6.95	11.26	1716.2	2.78	3.80	8.47	0.07	7.75
	Fe range		Co range		Ni range		Si range		Al range
	23-39		25-40		23-39		2-5		4-8
S2	Fe ₂₆ Co ₂₉ Ni ₃₆ Si ₃ Al ₆			S3	Fe ₂₆ Co ₄₀ Ni ₂₄ Si ₃ Al ₇			S4	Fe ₃₉ Co ₂₉ Ni ₂₂ Si ₃ Al ₇

To verify the chemical composition of the alloys after casting, a series of EDS measurements was performed for each sample. The results, presented in Table 2, demonstrate good control of the casting process and reveal only minor deviations from the nominal compositions.

Table 2. Chemical composition of the suction-cast alloys determined by EDS.

S1			S2			S3			S4		
Target %at		Measurement	Target %at		Measurement	Target %at		Measurement	Target %at		Measurement
Fe	29	28.88±0.33	Fe	26	25.72±0.4	Fe	26	25.91±0.43	Fe	39	38.66±0.42
Co	35	34.96±0.25	Co	29	29.46±0.6	Co	40	40.4±0.6	Co	29	29.54±0.04
Ni	27	27.37±0.39	Ni	36	36±0.55	Ni	24	24.39±0.2	Ni	22	22.08±0.36
Si	3	3.25±0.19	Si	3	3.2±0.28	Si	3	3.42±0.2	Si	3	3.28±0.23
Al	6	5.52±0.34	Al	6	5.6±0.13	Al	7	5.86±0.23	Al	7	6.42±0.11

The phase structure was analysed using a synchrotron diffraction, Figure 1. Alloy S2 was found to be single fcc structure, whereas alloys S1, S3 and S4 showed the formation of a small volume of bcc phase in addition to the fcc phase. The lattice parameter of the main fcc phase in S4 is higher than in other alloys, and there is also a trace of a second fcc phase.

**Figure 1.** a) Synchrotron X-ray diffraction patterns of alloys S1–S4. b) Enlarged view of the selected diffraction peaks highlighting the presence of minor secondary phases.

The magnetic properties of all samples were subsequently investigated. Alloy S2 exhibited the narrowest hysteresis loop, corresponding to a coercivity of 1.19 Oe. However, its saturation magnetization was relatively low, reaching only 120 emu g⁻¹. Similar magnetic behavior was observed for alloys S1 and S3, which exhibited coercivities of 1.48 and 1.76 Oe, respectively, and saturation magnetizations close to 130 emu g⁻¹. In contrast, alloy S4 showed the highest saturation magnetization among all investigated compositions, although its coercivity was approximately seven times higher than that of the other alloys. Regarding thermal stability, all alloys except S4 exhibited Curie temperatures

exceeding 1073 K, whereas the Curie temperature of S4 was below 973 K. The measured hysteresis loops and thermomagnetic curves used to determine the Curie temperatures are presented in Figure 2.

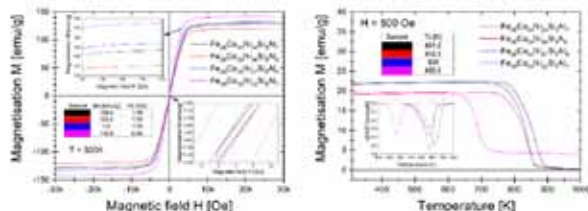


Figure 2. a) Magnetic hysteresis loops and b) thermomagnetic curves of alloys S1–S4 used to determine the Curie temperatures.

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Orbital Filtering in Rotated Lattice Junctions

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Keywords: orbital filtering, mirror symmetry breaking, tight-binding model,
orbital angular momentum, quantum transport

1. Introduction

The orbital angular momentum of electrons is an internal quantum degree of freedom that, unlike spin, takes integer values and supports higher multipole moments such as quadrupoles and hexapoles. Orbitronics aims to exploit this richness for information processing, potentially enabling low-power devices free from magnetic fields or strong spin-orbit coupling. A key challenge is understanding how orbital angular momentum is generated and filtered at material interfaces. Recent theoretical work by D’Onofrio, Cuoco et al. [1] established that orbital mixing terms arising from broken mirror symmetry — parameterized by the matrix χ_{xy} — are the essential microscopic ingredient for orbital filtering in a one-dimensional junction model. This work motivates the present study, which extends that framework to fully two-dimensional rotated lattice junctions.

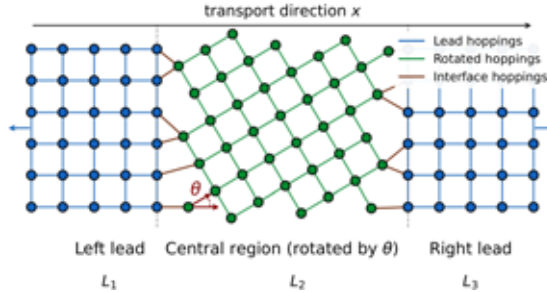


Figure 1. Schematic of the two-dimensional junction with rotated scattering region.

We simulate electron transport in two-dimensional square lattice junctions where some region of the lattice is rotated by an angle θ with respect to the rest, Figure 1. The rotation breaks mirror symmetry at the interface and induces an effective orbital mixing potential in the central scattering region. As the rotation angle is varied, different superlattice geometries emerge, each producing

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a distinct periodic orbital potential experienced by transmitted electrons. Tight-binding calculations using the Kwant library reveal that orbital filtering – the selective transmission of electrons carrying non-zero orbital angular momentum $L_z^2 \neq 0$ – occurs for most rotation angles. However, the filtering disappears at specific angles, where L_z^2 returns to zero after transmission. The resulting phase diagram as a function of θ exhibits a striking fractal-like structure, robust across system sizes and boundary conditions, Figure 2.

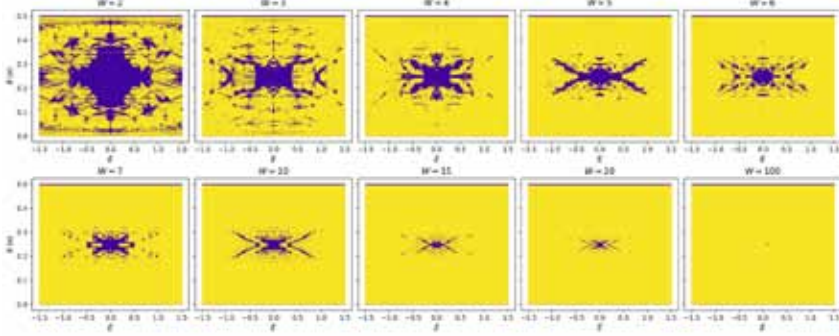


Figure 2. Orbital filtering phase diagram as a function of rotation angle θ , showing the yellow regions of filtering ($L_z^2 \neq 0$) and purple regions of non-filtering ($L_z^2 = 0$) in the two-dimensional rotated square lattice junction.

2. Results and Discussion

The fractal structure of the filtering phase diagram is the central result of this work. Figure 2 shows the transmitted orbital polarization as a function of rotation angle θ , revealing a self-similar pattern of filtering and non-filtering regions across multiple scales. This behaviour is qualitatively reproduced by a multiorbital generalization of the Aubry–André–Harper (AAH) model, in which both the hopping and on-site terms are periodically modulated by the orbital mixing matrix. The rotation-induced change in lattice geometry is captured in this one-dimensional framework through the modulation frequency β , which plays the role of the twist angle. The fractal structure persists for both rational and irrational values of β , directly mirroring the 2D results.

To isolate the underlying mechanism, we study a minimal one-dimensional model consisting of two regions of different orbital mixing strengths. An electron entering with no orbital angular momentum ($L_z^2 = 0$, quadrupole state) acquires orbital polarization upon passing through the first region ($L_z^2 \neq 0$, dipole state). Whether it recovers its original quadrupole state or retains its orbital polarization after the second region determines the filtering outcome. This two-region model serves as a piecewise approximation of the continuous AAH modulation and provides a transparent picture of the quadrupole–dipole–quadrupole conversion mechanism at the heart of the filtering phenomenon. The conditions on orbital mixing coefficients, region length, and relative phase that control this conversion are currently under investigation.

These results demonstrate that orbital filtering in rotated lattice junctions is a robust and geometrically tunable phenomenon. The fractal structure of the phase diagram reflects the interplay between the non-Abelian orbital mixing matrix and the geometry-dependent periodic potential, a combination with no analogue in purely scalar or spin systems. The minimal two-region model and its AAH generalization provide the theoretical foundation for understanding and engineering orbital filtering in realistic material platforms, including transition metal dichalcogenides and oxide interfaces.

2.1. Conclusions

We have shown that rotating one half of a two-dimensional lattice with respect to the other produces a fractal phase diagram of orbital filtering as a function of twist angle. The effect is governed by the non-Abelian orbital mixing matrix χ_{xy} induced by mirror symmetry breaking, and can be understood through a hierarchy of effective models: the two-region minimal model and its multiorbital AAH generalization. This work establishes a new theoretical framework for geometry-controlled orbital filtering, with direct implications for the design of orbitronic devices exploiting orbital angular momentum without magnetic fields or strong spin-orbit coupling.

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Effect of high temperature annealing on phase stability, microstructure and mechanical properties of CoFeCrAl HEA

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Keywords: HEA, microstructure, diffraction, phase stability, hardness

1. General

High-entropy alloys (HEAs) are a class of materials composed of multiple principal elements, typically in near-equiatomic ratios, resulting in high configurational entropy and unique microstructural features. HEAs constitute a unique class of materials due to their exceptionally high compositional flexibility, which enables the optimization of mechanical, corrosion-resistance, and functional properties [1]. Among them, CoFeAlCr alloys are considered promising candidates for demanding industrial and functional applications such as aerospace, surface engineering, additive manufacturing etc. [2] due to higher strength, hardness and oxidation resistance vs conventional alloys. Moreover, they can combine excellent mechanical properties with good soft magnetic properties, which are required, for example, in electric motors and many other applications [3].

In this work, melt-spun CoCrFeAl HEA ribbons were thoroughly investigated. The study included time-dependent experiments, synchrotron X-ray diffraction analyses for crystal structure determination and refinement, scanning electron microscopy (SEM) examinations, and hardness measurements. Particular attention was paid to high-temperature annealing, as previous studies have shown that at lower annealing temperatures, especially in the range of 1073–1173 K, precipitates form predominantly within the grain interiors, whereas precipitation along grain boundaries is limited. High-temperature annealing was therefore employed to promote the formation of the fcc phase at grain boundaries. This microstructural modification is expected to improve the material's plasticity, since the fcc phase generally exhibits greater ductility than the bcc phase.

2. Experimental

CoCrFeAl alloy was fabricated by induction melting under an argon atmosphere using high-purity elements (>99.9%). Subsequently, the alloy ingot was processed by the melt-spinning

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method, in which the molten alloy was ejected onto a rotating copper wheel at a speed of 25 m/s under a pressure of 0.25 MPa.

The obtained ribbons were heat-treated at temperature of 1473 K for different durations (0.5–24 h) followed by air-cooling. The crystal structure and texture were investigated using high-energy X-ray diffraction at DESY (Hamburg) on beamline P07B (87.1 keV, $\lambda = 0.0142342$ nm). For phase analysis, diffraction patterns were collected in continuous mode using a MAR345 2D Image Plate detector. Phase structure was performed by rotating the samples by 180° around the ω axis during X-ray exposure, whereas texture measurements were conducted using a 5° sample rotation. For both phase and texture measurements, the samples were prepared in a “sandwich” configuration consisting of more than 10 ribbon pieces to ensure reliable grain and variant statistics. The obtained 2D Debye–Scherrer diffraction rings were subsequently integrated using Fit2D software and presented as plots of relative intensity versus 2θ angle (Fig. 1).

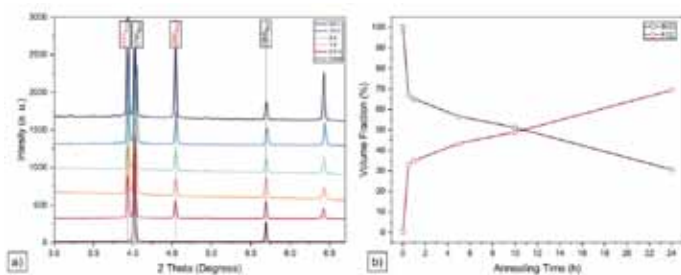


Figure 1. (a) High-energy X-ray diffraction patterns and (b) volume fractions of the fcc and bcc phases as a function of annealing time for the as-spun and annealed CoCrFeAl ribbons.

The microstructure of the material was characterized using the Electron Backscatter Diffraction (EBSD) technique. The analysis enabled the evaluation of grain orientation, grain size, and the presence of the fcc phase. In the next step, hardness measurements were also performed using a Struers testing machine. The applied load during the Vickers hardness test was limited to 25 g in order to avoid penetration through the ribbons and the acquisition of misleading results.

3. Results

Figure 1a presents high-energy X-ray diffraction patterns obtained for the as-spun and annealed CoCrFeAl ribbons. The as-spun ribbon exhibits a single-phase structure identified as a bcc phase. After annealing at 1473 K, a second face-centered cubic (fcc) phase appears. Moreover, elongation of annealing time leads to a gradual increase in the fcc phase fraction, which reaches approximately 70 vol.% after the longest annealing time (Fig. 1b). These results suggest that annealing promotes phase transformation and significantly affects the phase composition of the alloy.

Figure 2 shows the EBSD phase map of the CoCrFeAl ribbon annealed at 1473 K for 0.5 h. The microstructure is composed of two clearly distinguishable phases: the bcc phase (blue), which forms the matrix, and the fcc phase (red), located predominantly along the grain boundaries. The fcc phase creates a nearly continuous network surrounding the bcc grains, indicating that grain boundaries act as preferred sites for nucleation and growth of the fcc phase during annealing. However, fine fcc precipitates are also visible inside the grain interiors. Their presence suggests that the phase transformation is not limited only to grain boundary regions but also occurs locally inside the grains. With increasing annealing time, the volume fraction of the fcc phase increases significantly and eventually becomes comparable to, or even exceeds, that of the bcc phase. This microstructural

evolution, particularly the growth of the fcc phase, has a pronounced effect on the mechanical properties of the alloy. Hardness measurements reveal a gradual decrease in hardness with increasing fcc phase content. In addition, tensile tests indicate improved ductility and plastic deformation capability, which can be attributed to the higher intrinsic ductility of the fcc phase compared with the bcc phase. These results demonstrate a clear relationship between phase evolution and the mechanical response of the alloy during annealing.

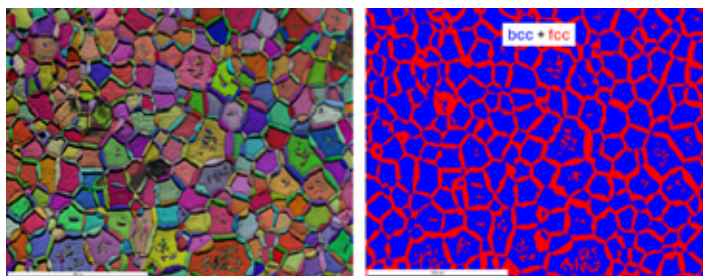


Figure 2. EBSD phase map of CoCrFeAl ribbon after annealing at 1473K/0.5h.

4. Conclusions

This study investigated the phase evolution, microstructure, and hardness of melt-spun CoCrFeAl high-entropy alloy ribbons annealed at 1473 K for different durations. The as-spun alloy exhibited a single-phase bcc structure, whereas annealing promoted the formation of a secondary fcc phase. Synchrotron X-ray diffraction and EBSD analyses revealed that the fcc phase nucleated predominantly at grain boundaries and progressively increased in volume fraction with increasing annealing time. As annealing proceeded, the fcc phase developed into a nearly continuous network surrounding the bcc grains, although fine precipitates were also observed within the grain interiors. The evolution of the phase constitution was accompanied by significant microstructural changes, highlighting the strong influence of annealing on the alloy's characteristics. These results demonstrate that heat treatment is an effective approach for tailoring the phase composition and microstructure of CoCrFeAl high-entropy alloys and, consequently, for controlling their mechanical properties.

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Irregular Eutectic Solidification in Micro- and Nanoparticles

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Keywords: in-situ transmission electron microscopy, alloy nanoparticles, solidification

1. Introduction and methods

The development of modern technologies in nanomedicine, catalysis, and microelectronics requires the creation of materials with precisely tailored properties that directly depend on their internal structure. Of particular interest are alloys belonging to the class of irregular eutectics, such as the Au–Ge and Au–Ge systems. In these systems, the combination of a metallic phase with the semiconductor germanium, which exhibits faceted growth behavior, enables the formation of an exceptionally wide variety of phases and microstructures. This work is dedicated to revealing the fundamental mechanisms governing the formation of such structures under conditions of rapid cooling and significant undercooling, which are crucial for understanding nonequilibrium crystallization at the micro- and nanoscale.

Au–Ge and Al–Ge bilayer thin films were prepared by DC magnetron sputtering (PRIMS 032, PREVAC). Upon heating, the films underwent dewetting and eutectic melting, leading to the formation of isolated micro- and nanoparticles. The resulting particles were subsequently analyzed using scanning and transmission electron microscopy techniques. The crystallization pathways and phase transformations of individual Au–Ge nanoparticles were investigated by in situ heating experiments in a Cs-corrected FEI Titan G2 60–300 equipped with a MEMS-based double-tilt heating holder. Morphology was monitored using HAADF-STEM imaging. Structural characterization was performed by high-resolution TEM, selected-area electron diffraction (SAED), convergent-beam electron diffraction (CBED), and Fourier transform (FT) analysis. Composition was analyzed using energy-dispersive X-ray spectroscopy (EDX) and electron energy-loss spectroscopy (EELS). Diffraction patterns and FT analyses were indexed using the Java Electron Microscopy Software (JEMS). For the Al–Ge system, the morphology of crystallized microparticles was examined using a Zeiss Merlin field-emission scanning electron microscope operated in secondary electron (SE) and backscattered electron (BSE) imaging modes. Cross-sectional TEM lamellae prepared by focused ion beam (FIB) milling were subsequently analyzed in the FEI Titan G2 60–300 using the same characterization methods as for the Au–Ge nanoparticles.

2. Results

2.1. Au–Ge

In situ TEM investigations of Au–Ge nanoparticles revealed a non-classical crystallization pathway [1]. The liquid-to-solid transformation was found to take place well below the nominal

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eutectic temperature, occurring over a temperature range and proceeding through a two-step mechanism involving successive phase formation. This process resulted in the transient coexistence of up to four distinct phases within individual hypo-eutectic (~25 at.% Ge) nanoparticles (Fig. 1). The coexistence of multiple phases in highly undercooled liquid creates a pathway for the formation of metastable structures and complex morphologies that may be tailored by nanoscale confinement and thermal processing conditions.

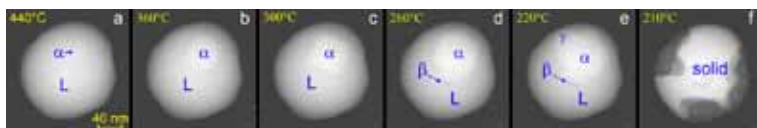


Figure 1. HAADF-STEM images of the same Au–Ge nanoparticle of hypo-eutectic composition at different temperatures during cooling from the two-phase state (a–f). The temperature is specified in the upper-left corner of each image. The scale bar is the same for all images in the row. L, α , β and γ represent liquid, fcc Au, Ge, and hcp Au phases, respectively.

For the composition exhibiting the highest number of coexisting phases, the dependence of morphology on particle size was studied. It was found that particles below 80 nm form Janus structures, whereas larger (80–500 nm) particles exhibit a metastable core–shell morphology [2]. Interestingly, nanoparticles consistently showed a high prevalence of hcp Au-rich domains, suggesting that this phase may be energetically favoured under strong spatial confinement, in contrast to the expected bulk fcc structure (Fig. 2). The temperature stability of hcp Au phase in Au–Ge nanoparticles of different size, chemical and phase mixing patterns will be discussed.

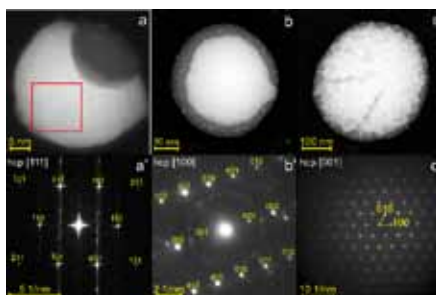


Figure 2. HAADF-STEM images of representative Au–Ge nanoparticles with (a) Janus, (b,c) core–shell morphologies. (a') FFT obtained from the region indicated by the red square in (a), indexed to hcp Au along the [111] zone axis. (b', c') SAED patterns indexed to hcp Au along the [100] and [001] zone axes, respectively.

2.2. Al–Ge

The complex crystallization pathway and the unusual phase coexistence observed in Au–Ge nanoparticles highlighted the rich microstructural evolution that can occur in irregular eutectic systems. Particularly intriguing was the morphology that developed during solidification, motivating further investigation of the Al–Ge system, where selective removal of one phase enables direct visualization of the three-dimensional solidification microstructure (Fig. 3). SEM and TEM analyses revealed that all solidified Al–Ge microparticles developed a fully interconnected irregular Ge network embedded within an Al matrix despite significant differences in wetting behavior on various

substrates. Quantitative analysis showed that the characteristic ligament thickness was independent of substrate type and contact angle, but increased with particle size and post-solidification annealing. Upon further heating, the network progressively coarsened and eventually transformed into an equilibrium Janus morphology [3].

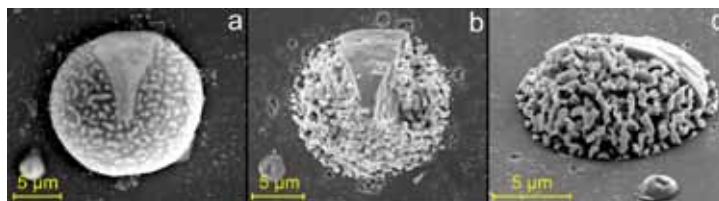


Figure 3. SEM images of Al–Ge microparticle on Si (a–c) substrate. Image (a) shows the particle before etching, whereas images (b,c) show the same particle after chemical etching. Image (c) was acquired at tilt angle of 53.

3. Discussion and conclusion

The results reveal that irregular eutectic systems exhibit complex solidification pathways and microstructure evolution that cannot be fully described by equilibrium models. By combining in situ and analytical electron microscopy, it was possible to directly observe phase formation, phase separation, and compositional changes, providing new insight into the mechanisms governing crystallization in irregular eutectic alloys.

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Effect of strain rate on the deformation behaviour of magnetron sputtered CuMoTaWV

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Keywords: HEA, RHEA, strain-rate sensitivity, magnetron sputtering, nanoindentation

High Entropy Alloys (HEAs) have attracted significant attention due to their unique combination of properties, often unattainable in conventional alloys. These materials combine high hardness and strength with good ductility, resistance to high temperature, radiation, and corrosion [1]. Among HEAs, Refractory High Entropy Alloys (RHEAs) have emerged as a promising class of advanced high-temperature materials [2], due to their exceptional mechanical stability and strength even at temperatures as high as 1600°C. This work, from a nanomechanics point of view investigated particularly interesting material [3] – nanocrystalline coating prepared using single targeted DC-magnetron sputtering of refractory based (MoTaWV) alloy with addition of component (Cu) that promotes the formation of FCC ductile phase.

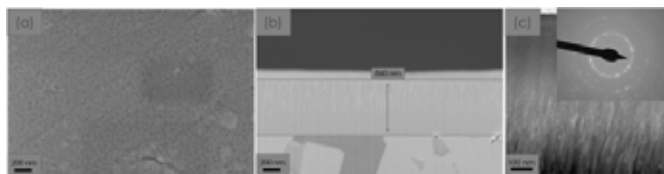


Figure 1. SEM images of CuMoTaWV (a) surface, (b) cross section, (c) cross section bright field TEM with corresponding SAED pattern.

To achieve objective, behavior of novel HEA under various loads and different loading rates were investigated with nanoindentation and compression of micropillars in situ Scanning Electron Microscopy (SEM) using an Alemnis nano-indenter. Experimental data were correlated with captured videos and images to extend the analysis. Microstructural changes induced by compressive stresses were investigated using Transmission Electron Microscopy with TKD analysis to enhance knowledge of the behavior of the material after loading. Changes in microstructure after loading were examined for various strain rates under nanoindentation indents and in cross section of the micropillars after compression tests. The imprints formed during nanoindentation were subsequently examined using atomic force microscope (AFM) (figure 2.) to enable detailed analyze features

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such as pile-up and sink-in. The material deformation occurring around the contact region – pile-up (deformation downward with respect to the indented surface plane) and sink-in (deformation upward) are correlated with material properties.

Strain rate sensitivity (SRS) describes how variations in the strain rate affect material characteristics, including hardness. In nanoindentation testing, the value of SRS_{ind} can be calculated using the following equation:

$$SRS_{ind} = \frac{\partial \ln H}{\partial \ln \dot{\epsilon}_{ind}} \quad (1)$$

where H is the hardness and $\dot{\epsilon}_{ind}$ is the indentation strain rate.

$$\dot{\epsilon}_{ind} = \frac{\dot{P}}{2P} \quad (2)$$

where P is the maximum applied load and $\dot{P}$ is the load rate. In order to determine SRS_{ind} we have plotted $\ln H$ and $\ln \dot{\epsilon}_{ind}$ and performed linear fit. SRS_{ind} is then equal to the slope of the fitted linear fit.

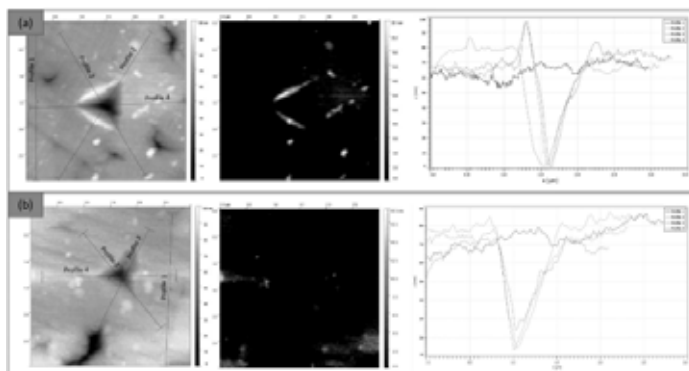


Figure 2. Pile-up pattern investigation with the use of an AFM: AFM image with the profiles indicated and cross-sections through the imprint for determination of the pile-up height (each profile is averaged over 10 pixels in width of a line) at different loading rates (a) 0.1 mN/min, (b) 1 mN/min.

This work evaluated the influence of strain rate on the deformation mechanisms of RHEAs based materials with addition of FCC former to BCC-based material, which will help with the evaluation of the influence of the different effects on the mechanics of HEAs. The objective of the work is to bring wider understanding of mechanical properties and underlying rules affecting the behavior of HEAs.

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Effect of Al and Cr additions on chemical and phase composition of carbides in SiMo ductile cast iron

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The type SiMo ductile cast irons (DCI) intended for high temperature (HT) applications are often alloyed with Cr in order to enhance their hardness and wear properties, what is achieved primarily by the precipitation of carbides. Al addition is aimed at improving the HT corrosion resistance and thermal stability. In the present work, SiMo5-1 (5 wt. % of Si, 1 wt. % Mo) alloyed additionally with Cr and Al ~2.4 wt. % each of them, was subjected to complex microstructure investigation with light, scanning and transmission electron microscopy (LM, SEM, TEM). The results showed that since in the standard SiMo5-1 the formation of type M_6C carbides and pearlite takes place, in case of the alloyed one mainly fine eutectoid precipitates have appeared. The Cr caused growth of an unidentified phase also within the solid state similar to cementite, often ascribed in the literature as “Cr-rich carbides”. Further investigation with a use of selected area electron diffractions (TEM/SAED) allowed to identify it as M_5C_2 , however the Cr caused a change in the dimension of the unit cell what conformed it to cementite. Despite the almost identical morphology, M_5C_2 contained different alloying additions what was determined precisely by energy dispersive spectroscopy in scanning transmission mode (STEM/EDS), differentiating it from any other carbide types. The obtained results allowed to determine how the addition of Cr affects the overall properties and application potential of the SiMo family alloys.

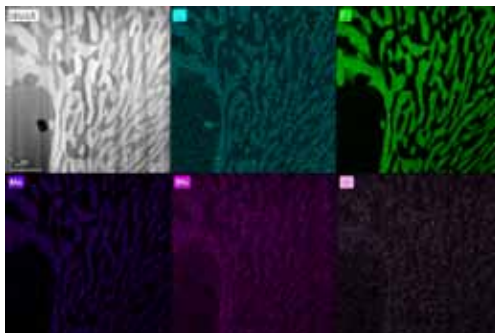


Figure 1. STEM/EDS images of Cr-rich carbides in SiMo5-1 DCI alloyed with Al and Cr.

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Mechanical and structural properties of fine-grained magnesium and magnesium alloys from 4K to 298K

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Keywords: polycrystalline magnesium, AZ-series alloys, fine grain size, work-hardening, deformation microstructure

1. Introduction

The relationship between grain size and deformation mechanisms is one of the central aspects governing the mechanical behaviour of polycrystalline materials. In conventional metallic systems, grain refinement increases the resistance to plastic flow through the Hall-Petch effect, where grain boundaries act as effective barriers to dislocation motion [1]. However, when the grain size is reduced into the ultrafine and submicron regimes, the increasing contribution of grain-boundary-mediated deformation may alter the balance between strengthening and plasticity, leading to deviations from classical Hall-Petch behaviour [2-3]. These effects are particularly pronounced in magnesium and its alloys due to their hexagonal close-packed crystal structure, limited number of independent slip systems, and strong sensitivity of deformation mechanisms to grain size and temperature. Consequently, the transition between intragranular plasticity, dominated by dislocation slip and mechanical twinning, and intergranular deformation processes becomes a critical issue in understanding the mechanical response of both polycrystalline pure Mg and technologically important AZ-series magnesium alloys.

Although the influence of grain size on the mechanical behaviour of pure Mg and its alloys has been extensively studied, most investigations have been performed at room or elevated temperatures, while studies at cryogenic temperatures remain limited. Furthermore, current understanding of grain-boundary-mediated deformation in Mg systems remains incomplete, particularly regarding the existence of a critical grain size for the activation of grain boundary sliding (GBS), its dependence on alloy chemistry, and its sensitivity to temperature. In this context, the present work aims to establish the relationship between grain size, temperature, and deformation mechanisms in both commercially pure Mg and fine-grained AZ-series alloys over a temperature range between 298K and 4K. Particular attention is devoted to identifying the conditions for the transition between intragranular and intergranular deformation, determining whether a critical grain size for GBS activation exists in both pure Mg and AZ alloys, and examining how alloying and

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temperature influence the activation or suppression of grain-boundary-mediated plasticity. The study further addresses the evolution of deformation-induced microstructures, including dislocation substructures, twinning activity, and grain-boundary processes, in order to establish the mechanisms governing strength, work hardening, and ductility in fine-grained magnesium materials under ambient and cryogenic conditions.

2. Experimental

Commercially pure Mg and AZ-series magnesium alloys were investigated in the as-extruded condition. Commercially pure Mg with a purity of 99.95% was processed by hot extrusion at temperatures between 373K and 543K, using an extrusion ratio of 22, in order to obtain polycrystalline microstructures with different grain sizes. The AZ-series alloys, including AZ31, AZ61, and AZ91, were produced by rapid solidification using melt spinning, followed by plastic consolidation through cold compaction and hot extrusion. The nominal compositions of the AZ alloys corresponded to Mg-Al-Zn systems with increasing Al content from AZ31 to AZ91. Mechanical properties were examined under uniaxial tension and compression between 298K and 4K. Tests were performed at a constant initial strain rate of $2.8 \cdot 10^{-4} \text{ s}^{-1}$, with liquid nitrogen and liquid helium used to maintain the cryogenic temperatures. Strain rate sensitivity was evaluated at room and low temperatures by changing the strain rate between $2.8 \cdot 10^{-4} \text{ s}^{-1}$ and $2.8 \cdot 10^{-5} \text{ s}^{-1}$. The initial and deformed microstructures were characterized using electron backscatter diffraction, transmission Kikuchi diffraction, scanning electron microscopy, and transmission electron microscopy. These techniques were used to determine grain size, crystallographic texture, twin activity, grain-boundary characteristics, and deformation-induced dislocation structures.

3. Results and discussion

The results demonstrate that the deformation behaviour of Mg-based materials is governed by a complex interplay between grain size, alloy chemistry, and temperature. In commercially pure Mg, grain refinement below $\sim 3 \text{ }\mu\text{m}$ at 298K promotes grain-boundary-mediated deformation, leading to deviations from conventional Hall–Petch strengthening. In contrast, deformation at 78K and 4K is dominated by dislocation slip and intensive $\{10\bar{1}2\}$ twinning. TEM observations reveal that room-temperature deformation produces a relatively low dislocation density due to pronounced dynamic recovery.

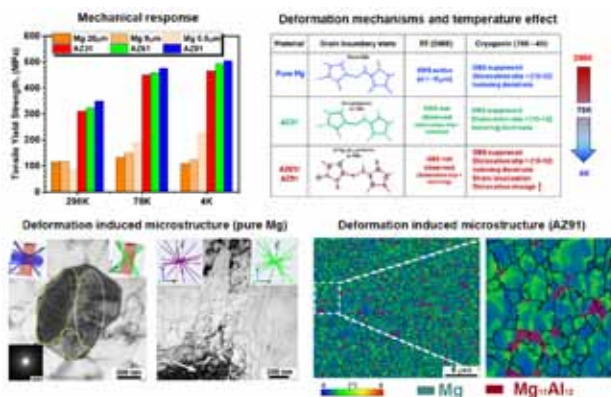


Figure 1. Schematic illustration of grain-boundary behaviour and dominant deformation mechanisms in pure Mg, AZ31, AZ61 and AZ91 alloys between 4K and 298K [2–4].

Primary grains develop sub-boundaries and low-misorientation sub-grains containing isolated dislocations, loops, and debris, while bowed dislocations undergo cross-slip onto non-coplanar planes. In contrast, cryogenic deformation results in substantially higher dislocation densities, characterized by rectilinear dislocations, extensive cross-slip segments, and longer twins subdivided into dislocation-filled sub-grains. Enhanced dislocation interactions generate jogs, entanglements, loops, and debris, indicating more effective dislocation storage and increased work hardening at low temperatures. A fundamentally different behaviour was observed in AZ-series alloys. Despite their ultrafine grain size, AZ31 alloys did not exhibit pronounced grain boundary sliding, likely due to Zn segregation at grain boundaries, which stabilized the interfaces and suppressed intergranular deformation. Similarly, AZ61 and AZ91 alloys with grain sizes of ~700–800 nm also showed no significant grain boundary sliding. In these alloys, grain boundaries were stabilized by β -Mg₁₇Al₁₂ particles formed during rapid solidification and hot extrusion, thereby preserving dislocation-controlled plasticity. Lowering the deformation temperature significantly increased strength and work hardening in all investigated materials. However, cryogenic deformation also promoted strain localization and reduced tensile ductility, particularly in AZ91, owing to its higher fraction of intermetallic particles.

4. Conclusions

- Grain refinement strongly influences the deformation behaviour of Mg-based materials; however, the effect depends critically on alloy chemistry, temperature, and grain-boundary stability.
- In commercially pure Mg, reducing the grain size below ~3 μ m at 298K promotes grain-boundary-mediated deformation and inverse Hall–Petch behaviour, whereas deformation at cryogenic temperatures is dominated by dislocation slip and twinning.
- In contrast, ultrafine-grained AZ31, AZ61, and AZ91 alloys with grain sizes of ~600–800 nm do not exhibit the same grain-boundary-mediated deformation behaviour despite their sub-micron grain size. In AZ31, Zn segregation at grain boundaries suppresses grain-boundary sliding, while in AZ61 and AZ91 the grain boundaries are stabilized by β -Mg₁₇Al₁₂ precipitates, inhibiting intergranular deformation and preserving dislocation-controlled plasticity.
- The deformed microstructure of Mg-based materials is strongly controlled by grain size, alloy chemistry, and temperature. Room-temperature deformation promotes dynamic recovery and relatively low dislocation densities, whereas cryogenic deformation suppresses recovery, leading to intense dislocation storage, extensive twinning, and enhanced work hardening. In AZ-series alloys, stabilized grain boundaries suppress grain boundary sliding and preserve dislocation-controlled plasticity even at ultrafine grain sizes.

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Microstructural Characterization of a Friction Stir Welded Quasicrystalline Aluminium Alloy

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Keywords: friction stir welding, aluminium alloy, quasicrystals, ebsd, hardness

Advanced lightweight aluminium alloys are attracting considerable attention for future transportation applications owing to their high specific strength, excellent corrosion resistance and good recyclability. Recently developed quasicrystalline (QC)-reinforced aluminium alloys exhibit significantly improved mechanical properties compared with conventional casting alloys, making them promising candidates for lightweight structural components [1]. However, their industrial implementation requires joining technologies capable of preserving their unique microstructure and mechanical properties. Since conventional fusion welding involves melting and solidification, it may alter the distribution of strengthening phases and reduce joint strength. Friction stir welding (FSW), as a solid-state joining process, provides an attractive alternative by producing high-quality joints without melting the material [2,3].

This work presents the first microstructural observations of friction stir welded joints produced in a quasicrystalline aluminium alloy within the REFORMS project. The objective was to evaluate the influence of the FSW process on the microstructure and hardness distribution across the welded joint. Butt joints were produced using optimized welding parameters, and the welded cross-sections were examined using scanning electron microscopy (SEM). Vickers hardness measurements (HV1) were performed across the entire weld to evaluate local variations in mechanical properties.

Figure 1 compares the microstructures of a commercial DIN 226 alloy and the QC1 alloy in three characteristic regions of the welded joint: the base material (BM), the thermo-mechanically affected zone (TMAZ) and the stir zone (SZ). Although both alloys exhibit microstructural refinement after friction stir welding, the effect is considerably more pronounced in the QC1 alloy. The coarse as-cast morphology of the QC1 alloy is transformed into a fine and homogeneous particle distribution within the stir zone as a result of intense plastic deformation and dynamic recrystallization during FSW. No macroscopic welding defects such as voids, tunnel defects or lack of bonding were observed, indicating stable material flow throughout the joining process.

Hardness measurements performed across the welded joint demonstrated that the quasicrystalline alloy retained its superior hardness after FSW. The stir zone reached hardness values of approximately 180 HV1, while the commercial DIN 226 alloy exhibited values close to 100 HV1, corresponding to a difference of more than 70%. The combination of a refined microstructure and high hardness confirms the suitability of friction stir welding for joining quasicrystalline aluminium alloys and highlights their potential for future lightweight structural applications in the automotive, aerospace and transportation industries.

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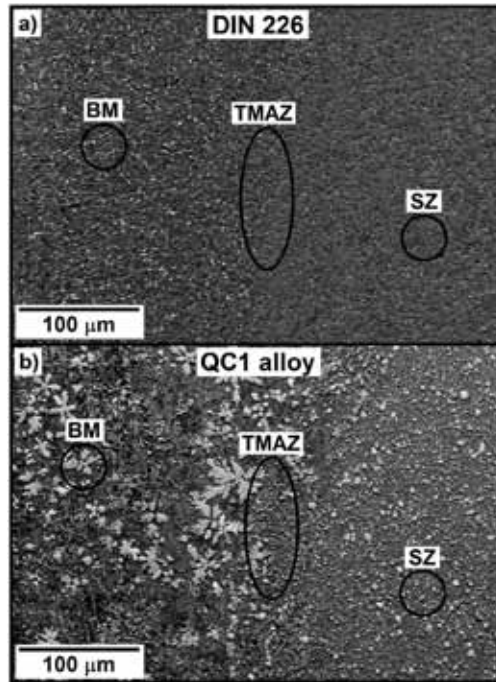


Figure 1. SEM images showing the microstructures of the commercial DIN 226 alloy (a) and the quasicrystalline aluminium QC1 alloy (b) after friction stir welding. Representative regions corresponding to the base material (BM), thermo-mechanically affected zone (TMAZ) and stir zone (SZ) are indicated.

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Microstructural Evolution, Texture Development and Mechanical Response of a Biodegradable Mg–4Ag Alloy Processed by High-Pressure Torsion

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Keywords: biodegradable magnesium alloys, High-Pressure Torsion (HPT),
Severe Plastic Deformation (SPD), crystallographic texture,
strain hardening exhaustion

Magnesium–silver alloys are promising biodegradable biomaterials because they combine the favourable mechanical compatibility and biocompatibility of magnesium with the intrinsic antibacterial properties of silver, making them attractive candidates for temporary orthopaedic implants [1,2]. However, their broader clinical application remains limited by insufficient mechanical strength and structural stability during service [1]. High-pressure torsion (HPT), one of the most effective severe plastic deformation (SPD) techniques [3], enables extreme grain refinement and the formation of ultrafine-grained microstructures, which may substantially enhance the mechanical performance of magnesium-based biomaterials [4,5]. Therefore, the present study investigates the influence of HPT processing on the microstructural evolution, crystallographic texture, and mechanical properties of a biodegradable Mg–4 wt.% Ag alloy.

The alloy was processed by HPT to different deformation levels corresponding to $N = 0.5$, 2, and 10 revolutions. The evolution of crystal structure and texture was characterized by X-ray diffraction (XRD), while mechanical performance was evaluated by microhardness and tensile testing. XRD analysis (Fig. 1a) revealed progressive peak broadening with increasing HPT strain, indicating significant grain refinement accompanied by an increasing density of lattice defects. Line-profile analysis confirmed a continuous reduction in crystallite size together with an increase in lattice distortion as the imposed strain increased. These results demonstrate the effectiveness of HPT in producing a highly refined microstructure through the accumulation of severe plastic strain.

Simultaneously, pronounced changes in crystallographic texture were observed (Fig 1b). The weak texture of the as-cast material gradually evolved into a characteristic HPT-induced shear texture dominated by a rotated $\langle 002 \rangle$ fibre component. The texture development is attributed to the

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accumulation of large shear strains during HPT, which activate multiple deformation mechanisms characteristic of magnesium alloys, including basal and non-basal slip, deformation twinning, dislocation multiplication, grain subdivision, and recovery processes. As deformation progressed, the initially heterogeneous orientation distribution was replaced by a well-developed shear texture. After 10 HPT revolutions, however, the texture became noticeably more diffuse and less intense, suggesting a gradual reduction in crystallographic anisotropy associated with continued grain refinement and microstructural homogenization at very high strains.

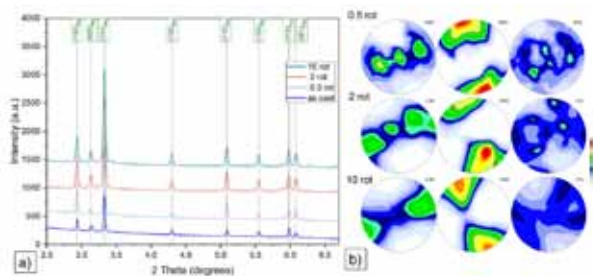


Figure 1. (a) X-ray diffraction pattern of initial and HPT-processed samples along with (b) the evolution of crystallographic texture for different numbers of HPT rotations.

Mechanical characterization demonstrated a substantial increase in microhardness from 61 HV in the as-cast condition to 119 HV after 10 HPT revolutions. This nearly twofold increase confirms the strong strengthening effect associated with grain refinement and defect accumulation induced by severe plastic deformation. However, the increase in local hardness was not accompanied by a corresponding improvement in global tensile performance. Although the ultimate tensile strength (UTS) increased after the initial deformation stage ($N = 0.5$), further HPT processing led to a gradual reduction in tensile strength and the occurrence of premature brittle-like fracture after 10 revolutions. This apparent discrepancy between hardness and tensile behaviour is characteristic of highly deformed ultrafine-grained materials and suggests that excessive strain reduces the strain-hardening capacity available during tensile deformation. As a consequence, plastic strain becomes increasingly localized, promoting premature crack initiation and early failure. The deterioration in tensile performance is therefore attributed to the combined effects of limited work-hardening capability of the ultrafine-grained structure, residual deformation defects, and deformation-induced texture.

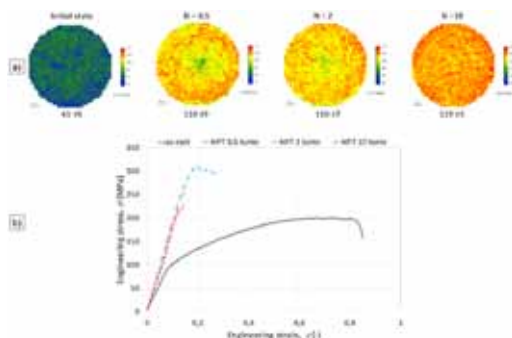


Figure 1. (a) HV0.05 mapping (0.25×0.25 mm), (b) engineering tensile stress–strain curves of Mg–4 wt.% Ag alloy in different states.

The obtained results demonstrate that HPT processing is highly effective in tailoring the microstructure and strengthening the Mg–4 wt.% Ag alloy; however, the highest imposed strains do not necessarily lead to the most favourable tensile properties. The study highlights the critical role of optimizing the degree of severe plastic deformation in order to achieve an appropriate balance between hardness, strength, and fracture resistance. These findings contribute to a better understanding of the relationships between microstructural refinement, texture evolution, and mechanical behaviour in HPT-processed Mg–Ag alloys and support the development of advanced biodegradable magnesium-based biomaterials for orthopaedic applications.

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Advanced instruments for micro- and nano- analysis. Elionix ENT-5 Nanoindenter. Table-top SEM Semplor Nanos. Ultrasonic Fatigue Testing Machine Shimadzu USF-2000A

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Shim-Pol [1] has been providing solutions and instruments for mechanical and chemical analysis for 40 years and is the official distributor of Shimadzu, Elionix and Semplor high end equipment manufacturers.

Shimadzu [2] has been providing solutions and equipment for testing mechanical properties for 109 years. The company was established 151 years ago in Japan as a family business providing various types of products. Shimadzu's portfolio has changed over time. The company became one of the largest producers of scientific equipment in the world. Research in nano- and micro- scale, as well as chemical analysis constitutes an important part of the company's portfolio. One of the most advanced instrument is Ultrasonic Fatigue Testing Machine, allowing users to obtain critical data from VHCF testing within days.

Elionix [3] is Japanese company that has been developing and manufacturing cutting-edge products since 1975. Its main products include electron beam lithography system and nanoindentation tester. The nanoindentation tester introduced here is a type of ultra-low-load hardness tester capable of measuring the hardness and elastic modulus of ultra-thin coatings less than 1 micrometer thick and materials in microscopic areas with high data reproducibility.

Examples of measurable materials include DLC, metal, insulating films, and UV-curable and thermosetting resins.

Semplor [4] is Dutch company manufacturing one of the most reliable table-top scanning electron microscope NANOSTM which connects excellent usability with low maintenance costs.

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Why there is no invariant plane strain in martensitic transformations?

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Keywords: martensitic transformation, twinning, habit plane, TEM, orientation relationship

The conflict between atomic-scale mechanisms and the high-index “habit planes” in martensitic transformations has lasted nearly one century. This fundamental problem is still plaguing metallurgists. The crux of this conflict lies in the incompatibility between atomic scale lattice transformation and the experimentally measured “habit planes” that have high indices and have been used as the cornerstone of a premise of “invariant plane strain” in martensitic transformations. The concept of “invariant plane strain” was also based on experimentally observed “line distortion” and surface tilt on a well-polished specimen surface after martensitic transformation. These experimental observations, along with the fast kinetics of martensite growth, have led to the premise that in martensitic transformation, an invariant plane shear must occur on the austenite-martensite interface. However, the concepts of “habit plane” and “invariant plane strain” in martensitic transformation face insurmountable challenges. In this work, we present atomistic simulations of fcc-to-bcc phase transition and high resolution transmission electron microscopy observations of interfacial structure between martensite and austenite. We show that line distortion is only a result of lattice correspondence and cannot be used to draw a definitive conclusion of an invariant plane shear. As such, the premise of invariant plane strain in martensitic transformation is untenable and should be abandoned. A new mechanism for martensitic transformation, lattice correspondence analysis, variant selection and strain accommodation will be presented and discussed. This mechanism explains well the origin of high index habit planes measured in the early studies and why a large scatter was observed between the experimentally measured habit planes and the predictions by phenomenological theory.

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Confined Nanocrystal Growth via Twisted Epitaxy in 2D MoS₂–Metal Trilayers

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Keywords: 2D materials, epitaxy, twisted epitaxy, nanoconfined crystal growth

Twisted epitaxy is a recently discovered form of confined crystal growth in which the crystallographic orientation of a metallic phase is governed simultaneously by two enclosing, rotationally misaligned, van der Waals (vdW) substrates [1]. In contrast to conventional epitaxy, where the growing crystal interacts with a single substrate, twisted epitaxy emerges under nanoscale confinement, where competing epitaxial interactions from two crystalline interfaces determine the final orientation of the confined material, Fig. 1. This phenomenon was first demonstrated in the Au–MoS₂ system, where initially randomly oriented Au nanoparticles (NPs) underwent a spontaneous reorientation during annealing when encapsulated between two mechanically exfoliated MoS₂ layers possessing a predefined mutual twist angle [1]. Such behaviour extends the classical concept of epitaxy beyond established paradigms including vdW epitaxy, quasi-vdW epitaxy, confined epitaxy and remote epitaxy, highlighting the rich opportunities offered by twisted vdW heterostructures for directing matter at the nanoscale [2].

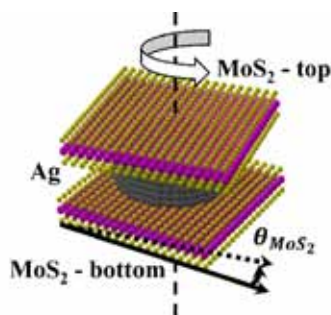


Figure 1. Schematic illustration of a typical MoS₂–Ag–MoS₂ trilayer heterostructure with a predetermined misalignment angle (θ_{MoS_2}) between the top and bottom MoS₂ substrates.

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From the perspective of nanocrystalline growth, twisted MoS₂ heterostructures provide a unique confined environment in which crystal nucleation, growth and orientation selection occur within an atomically thin nanoreactor. The simultaneous interaction of a growing nanocrystal with two crystallographically distinct interfaces introduces a new degree of freedom for controlling nanoscale assembly. Understanding such confined growth processes is of fundamental importance because metallic nanostructures integrated with two-dimensional (2D) materials constitute essential building blocks for future electronic, plasmonic, photonic, catalytic and quantum devices. Moreover, confinement-induced growth phenomena may enable access to metastable structures, unusual orientation relationships and emergent properties that are inaccessible under conventional thin-film growth conditions.

A key question is whether twisted epitaxy represents a universal mechanism of confined nanocrystalline growth in metal–transition metal dichalcogenide (TMD) systems or whether it is specific to Au–MoS₂. Previous studies have demonstrated that several metals, including Ag, Cu, Pd, Pt, Pb, Al and Zn, exhibit epitaxial relationships when deposited on MoS₂ surfaces. However, their growth behaviour under simultaneous confinement by two misoriented vdW interfaces has remained unexplored [3]. Silver constitutes an ideal model system for investigating the generality of this phenomenon because it possesses the same face-centred cubic (fcc) crystal structure and close-packed (111) surface symmetry as Au, together with comparable lattice parameters and similar interfacial chemistry with MoS₂. Furthermore, Ag exhibits high surface diffusivity, favourable wetting behaviour and weak chemical interaction with chalcogenide surfaces, all of which are expected to strongly influence confined nanocrystalline growth.

Here, we investigate the growth and structural evolution of Ag nanocrystals confined between two twisted MoS₂ layers forming a vdW heterostructure. Ag was deposited onto exfoliated MoS₂ and subsequently encapsulated by a second MoS₂ layer with a controlled twist angle. Thermal annealing promotes nanoparticle nucleation, coarsening and crystallographic reorientation within the confined interlayer space. High-resolution transmission electron microscopy (HRTEM) and four-dimensional scanning transmission electron microscopy (4D-STEM) were employed to determine the orientation relationships between Ag nanocrystals and the surrounding MoS₂ layers with sub-nanometre spatial resolution.

Our observations reveal that twisted epitaxy indeed occurs in the Ag–MoS₂ system, demonstrating that the phenomenon is not unique to Au. The orientation of confined Ag nanocrystals exhibits a systematic dependence on the relative rotation between the two MoS₂ layers, closely resembling the behaviour previously reported for Au. Nevertheless, several important differences emerge, including variations in orientation selection, the stability of intermediate states and the evolution of nanocrystal morphology during annealing. These findings indicate that while geometrical confinement and competing epitaxial interactions constitute the underlying driving force, material-specific factors such as interfacial energetics, diffusion kinetics and metal–substrate interactions play a critical role in determining the final growth behaviour.

The present study broadens the scope of twisted epitaxy and establishes confined nanocrystalline growth within twisted vdW heterostructures as a general route for engineering the structure and orientation of metallic nanomaterials. Such an approach opens new opportunities for designing nanoscale architectures with tailored structural, optical and electronic functionalities.

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Structural insights into the crystallographic phases of MnTe – canonical altermagnet

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Keywords: MnTe, altermagnet, STEM

Altermagnetism has recently emerged as a new class of magnetic order, combining compensated magnetization magnetic moments, but keeping electronic bands spin-polarised [1,2]. Additionally, due to high Néel temperatures, the altermagnetic materials attract a lot of attention as candidates for perspective applications in spintronic devices. Among them, MnTe is considered a canonical system, yet it has been almost exclusively studied in its NiAs-type structure. MnTe can occur in several crystalline polytypes: α , β , γ and δ phases ascribed to hexagonal NiAs type (nickeline), hexagonal wurtzite, cubic zinc blende and cubic rock-salt structure, respectively. Whether the wurtzite (β) phase is altermagnetic remains unresolved, and to date it has been reported only as small admixture of secondary crystalline phase regions in nonhomogeneous films.

Here, we demonstrate the epitaxial stabilization of almost phase-pure wurtzite β -MnTe thin films grown by molecular beam epitaxy (MBE) on GaAs(111)B substrates [3]. By slight modification of the growth conditions, we achieve controlled switching between multiphase layers and nearly single-phase wurtzite MnTe, providing direct control over the crystallographic phase (Figure 1). High-resolution X-ray diffraction confirms the formation of a structurally well-defined wurtzite phase with precise lattice parameters ($a = 4.472 \pm 0.004$ Å, $c = 7.3420 \pm 0.0005$ Å) and good crystalline quality. The negligible amount of secondary phases in optimized sample (Figure 1b) evidences the successful stabilization of this previously elusive polymorph. Moreover, SQUID magnetometry provides its first quantitative magnetic characterization: an antiferromagnetic transition is identified at a Néel temperature $T_N = 58 \pm 2$ K, with a large negative Curie-Weiss temperature ($\theta_{CW} \approx -420$ K) confirming strong antiferromagnetic exchange, and an effective spin close to the high-spin Mn^{2+} configuration ($S \approx 2.6$). Below T_N , an anisotropic, field-linear excess magnetization emerges, consistent with the lower, uniaxial symmetry of the wurtzite structure. These results

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provide the first experimental magnetic benchmark for testing the altermagnetic scenarios proposed theoretically for wurtzite MnTe. By analogy to altermagnetic wurtzite MnSe [4], this cannot be excluded, but there is no definite proof for that yet, either from theory or from experiment.

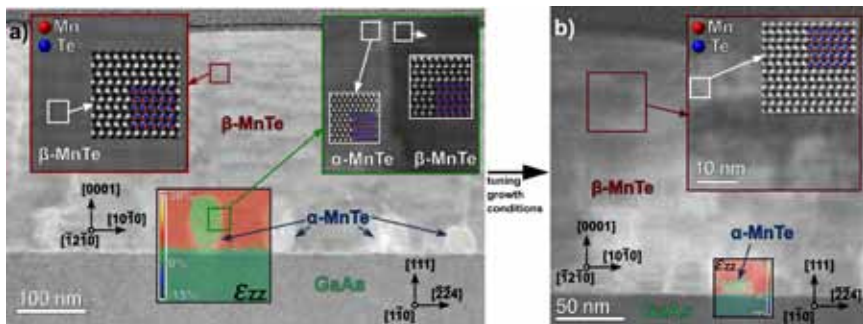


Figure 1. Cross-sectional STEM images of wurtzite MnTe layers grown on GaAs(111)B [3].

- a) Multiphase sample: the β -MnTe matrix (with superimposed Mn/Te atomic model) is locally interrupted by α -MnTe incursions originating at the GaAs/MnTe interface; the GPA ϵ_{zz} strain map (color-coded inset) reveals localized strain along growth [0001] direction.
- b) Optimized sample with almost pure-phase β -MnTe layer with only sparse, nanoscale α -MnTe incursions confined to the interface region.

Transmission electron microscopy (TEM) and scanning TEM (STEM), combined with geometric phase analysis (GPA), reveal the nanoscale origin of this phase selectivity (Figure 1). The residual α -MnTe inclusions are not randomly distributed through the layer, but are rooted at the GaAs(111)B/MnTe interface and extend along the [0001] growth direction (Figure 1a). GPA strain maps show that these inclusions are accompanied by localized strain fields and misfit dislocations at the phase boundaries. In the optimized sample, α -MnTe is barely detectable by X-ray diffraction and appears in TEM only as sparse, nanoscale inclusions confined to the interface (Figure 1b), showing that phase purification of β -MnTe proceeds mainly through suppression of interfacial α -MnTe nucleation. In both samples, structural defects have been identified: mostly stacking faults and inclusions of zinc-blende γ -MnTe phase within β -MnTe matrix. These results establish wurtzite MnTe as a new epitaxial material platform and open a pathway toward experimental exploration of altermagnetic and multiferroic properties in non-centrosymmetric systems.

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In-situ STEM observation of tungsten oxide nanoparticles heated to 1000°C

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Keywords: in-situ observation, electron irradiation, MEMS-heating, tungsten oxide, defects

1. Introduction

Tungsten oxide (WO_3) has attracted much attention due to its promising optical, electronic, and catalytic properties. Oxygen vacancies can be easily formed in WO_3 to form non-stoichiometric tungsten oxide phase (WO_{3-x}) by vacuum heating [1] and ion irradiation [2]. The introduction of oxygen vacancies can change their properties, thus the understanding of the defect structure is important. Atomic structures of WO_{3-x} were observed by STEM in some studies. For instance, oxygen vacancies on the surface induces surface reconstruction and generates a structure of 5-fold symmetry pentagonal W columns [3]. Another study conducts in-situ STEM observation of WO_3 at 500°C under H_2 atmosphere, and the planar defects are observed at the atomic scale [4]. However, in-situ atomic-scale observation under higher temperatures of 500°C and above are missing. In this study, we performed in-situ STEM observation of WO_3 nanoparticles under vacuum at temperatures up to 1000°C. Atomic scale observations were also performed at temperatures above 500°C.

2. Experimental procedure

WO_3 nanoparticles (Sigma Aldrich, purity N.A.) were dispersed in isopropanol and a few drops of the suspension were placed on a MEMS chip and a carbon-film supported grid. The in-situ observation of the MEMS chip was performed by Titan3 G2 with an acceleration voltage of 300 kV using a MEMS heating holder (Thermo Fisher Scientific, Nano Ex-i/v). Heating rate was set to 50°C/min, and the STEM observation was performed at room temperature, 500, 700, 900 and 1000°C. The HAADF- and ABF-STEM images were obtained, and some of the images were taken by drift corrected frame integration (DCFI, 20 frames). The ex-situ observation of samples was also performed for the WO_3 particles heated under vacuum at 500°C and 700°C. The in-situ observation was also performed by high voltage electron microscope (HVEM, JEM-ARM1300) with an acceleration voltage of 1250 kV using an electric heating holder. Heating experiment was performed up to 500°C.

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3. Results and discussions

WO₃ powder has a monoclinic structure, and the atomic scale observation was performed along the [001] zone axis. Figure 1 shows the STEM images of in-situ heating experiment at 500°C. The in-situ observation of the WO₃ particles showed the formation of W nanoparticles in the edge region at 500°C and 700°C. These kinds of W nanoparticles were not observed for the ex-situ heated samples. This suggests that the electron irradiation would cause the formation of W nanoparticles. Formation of oxygen vacancies and the diffusion of W ions at high temperature could result in the growth of W nanoparticles. The formation of planar defects was also observed at 500°C for in-situ and ex-situ heating. The atomic structure of this planar defect was a vacancy surrounded by six tungsten atom columns, which was also observed in the previous report [4].

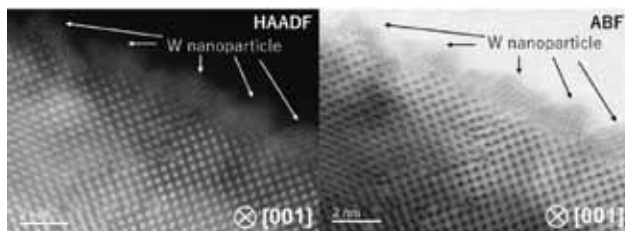


Figure 1. HAADF- and ABF-STEM images of WO₃ nanoparticles at 500°C along the [001] zone axis.

Both images were taken by DCFI with 20 frames integration.

Although the particles morphology remained almost unchanged by the observation at low magnification up to 700°C, the atomic scale observation at 700°C caused the partial sample loss in some edge regions due to the irradiation damage. Further in-situ heating up to 900°C at low magnification caused the sublimation around the severely damaged area (observed area in the atomic scale). However, the less damaged area did not cause the sublimation at 900°C. This result suggests that the irradiation damage can enhance the sublimation rate of tungsten oxide.

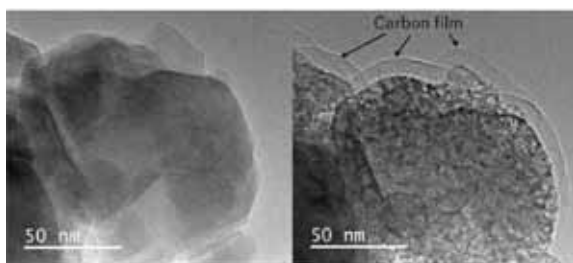


Figure 2. TEM images of WO₃ nanoparticles. (left) before heating, (right) after in-situ heating at 500°C. The in-situ observation was performed using HVEM with an acceleration voltage of 1250 kV.

In-situ heating experiment was also performed by using HVEM at 500°C. In this experiment, carbon contaminations were generated during the in-situ heating experiment as shown in Figure 2. However, tungsten carbide nanoparticles were observed at the interface between carbon layer and WO₃. Thus, electron irradiation of WO₃ with carbon film at 500°C can generate tungsten carbide. Also, as shown in Figure 2, porous oxide structure was observed after in-situ observation, and the

formation of the porous structures was thought to be caused by the high-voltage electron irradiation occurs in the HVEM.

4. Summary

In-situ observation of WO_3 particles at 500°C showed the formation of W nanoparticles in the edge regions. In-situ observation with carbon film at 500°C can form tungsten carbide at the interface between oxide particle and carbon film. The formation of W particles is thought to be caused by the formation of oxygen vacancies followed by the diffusion of W ions at the high temperature. The less damaged area by electron irradiation did not cause the sublimation at 900°C , but the severely damaged area by electron irradiation resulted in the partial sublimation in the range between 700 – 900°C . This suggests that the irradiation damage can enhance the sublimation rate of tungsten oxide.

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In Situ LC-TEM Investigation of Chitosan and Chitosan-TiN Nanocomposite Coating Formation during Electrophoretic Deposition

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Keywords: Liquid-cell TEM, electrophoretic deposition, in situ microscopy

This study presents a real-time investigation of electrophoretic deposition (EPD) processes in pure chitosan solution and chitosan-TiN nanocomposite suspension using liquid-cell transmission electron microscopy (LC-TEM). By combining electrochemical control with in situ micro- and nanoscale imaging, the dynamic evolution of polymeric and composite coatings was directly visualized. The obtained results provide new insight into the deposition mechanisms of chitosan and TiN/chitosan coatings, contributing to a better understanding of the EPD of chitosan-based coatings and demonstrating the potential of LC-TEM in this area.

1. Background and objective

Electrophoretic deposition has emerged as an effective strategy for the fabrication of various polymeric, ceramic and composite coatings, particularly for biomedical [1] and microdevice-related applications [2]. Among the naturally derived polymers, chitosan has attracted considerable attention due to its biocompatibility, film-forming ability, and sensitivity to external electrical stimuli, which make it especially suitable for electrically driven assembly processes. In acidic aqueous media, protonated chitosan molecules migrate under an electric field toward the cathode, where local pH changes induced by electrochemical reactions lead to deprotonation and the formation of an insoluble coating [3]. Although the general deposition mechanism has been previously proposed, the nanoscale processes governing coating nucleation, particle interactions, and structural evolution during EPD remain insufficiently understood.

Incorporation of inorganic nanoparticles (NPs) into chitosan matrices further expands the functionality of deposited coatings. In particular, ceramic particles such as TiN are promising additives for the development of mechanically reinforced and wear resistant nanocomposite systems [4]. In addition, they exhibit potential antimicrobial performance [5]. Previous studies demonstrated that the chemical composition and pH of the dispersed system, current conditions, and nanoparticle interactions strongly influence the EPD mechanism and kinetics and the final coating morphology. However, conventional ex situ characterization methods are unable to capture the dynamic phenomena occurring directly in the dispersed system in the vicinity of the electrode during deposition.

Recent advances in LC-TEM have enabled the real-time visualization of electrochemically driven processes with nanoscale spatial resolution, including metal nucleation, dendritic growth, and nanoparticle assembly under applied electric fields [6]. Despite the growing importance of

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biopolymer-based micro- and nanostructures, in situ LC-TEM studies of EPD of polymers remain unexplored.

Therefore, the present work aims to directly investigate and compare the dynamic formation pathways of pure chitosan coatings and chitosan-TiN nanocomposites using in situ LC-TEM. Particular attention is devoted to the deposit formation under electrophoretic conditions, which provides new insight into the mechanisms governing coating formation at the micro- and nanoscale.

2. Materials and methods

Chitosan solutions with an initial concentration of 0.5 g/L were prepared using deionized water with 1 vol.% acetic acid as the dispersing medium. Subsequently, the solutions were diluted with deionized water or ethanol (99.8%) to obtain final chitosan concentrations of 0.25 g/L in both aqueous and water-ethanol (50:50 vol.%) systems. For the preparation of the chitosan-TiN suspension, 8 mL of an aqueous TiN NPs suspension (1 g/L) was introduced into the water-ethanol chitosan solution containing 0.25 g/L of chitosan and mixed to obtain a stable dispersed system.

Real-time observations of EPD processes were carried out using a JEM-ARM200F NEOARM transmission electron microscope (JEOL) equipped with a METRO counting camera (Gatan) and Poseidon Select in situ electrochemical liquid-cell system (Protochips). Electrochemistry e-chip sets, top – ECT-45PT and bottom – EPB-55BNF and EPB-55DNF, were used for experiments. Platinum working electrodes integrated within the e-chip were used to apply negative electrical potentials during deposition experiments. LC-TEM experiments were performed under controlled chronoamperometric conditions in the voltage range from -10 to 10 V. Additional experiments were conducted to evaluate the influence of solvent composition and electron beam intensity on dispersed system stability, particle dynamics, and deposition behaviour during in situ observations.

3. Results

In an aqueous chitosan solution introduced into the e-chip liquid cell equipped with an additional 50 nm spacer (EPB-55BNF), exposure to a high-intensity electron beam (above $42\text{ e}^-/\text{px/s}$ registered on a METRO camera), the presence of spherical chitosan particles with sizes of approximately 10 nm was observed, followed by their agglomeration and formation of chain-like aggregates. Real-time LC-TEM observations provided direct insight into the behaviour of aqueous chitosan solutions under an applied electric potential difference. The experiments confirmed the migration of positively charged chitosan molecule clouds toward the cathode as well as their accumulation and bubbles formation in the solution at voltages starting from -5 V. During some stages of the process, highly concentrated chitosan clouds flowed over the electrode surface, obscuring the observation area and limiting the visualization of the deposition process. At a higher applied voltage of -10 V, the rapid accumulation of chitosan molecules near the electrode resulted in the formation of highly branched dendritic structures growing directly from the Pt cathode edge (Fig. 1a). Despite the successful visualization of the deposition, the formation of stable, compact coatings from pure aqueous solutions was impossible, most probably due to water electrolysis and strong electron-beam interactions. In the case of dendritic structures, electron beam irradiation caused progressive degradation of the deposits.

The addition of ethanol to the dispersing medium significantly altered the deposition behaviour. Under applied negative potentials starting from -7.5 V, chitosan clouds accumulated around the cathode, while chronoamperometric experiments performed at -10 V resulted in the formation of dense deposits. After completion of the deposition process (300 s), a coating with a thickness exceeding 500 nm was observed on the electrode surface (Fig. 1b). Prolonged exposure of the coating to the electron beam resulted in condensation of chitosan near the electrode, followed by rolling and partial detachment of the coating. In contrast to purely aqueous systems, the ethanol-containing solutions exhibited improved resistance to electron beam irradiation and reduced bubble generation during in situ observations.

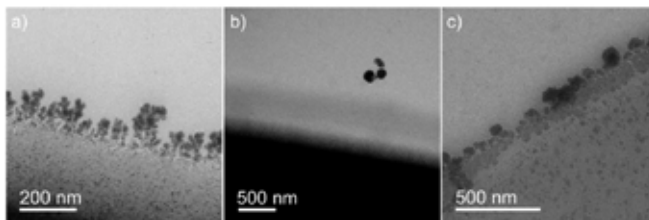


Figure 1. Chitosan deposited on Pt electrode from aqueous (a), aqueous-ethanol solution (b), and chitosan-TiN coating (c).

The deposition of coatings from chitosan-TiN suspensions occurred during experiments performed at voltages of -7.5 V and -10 V for 300 s (Fig. 1c). The deposition process itself remained difficult to visualize due to the high local concentration of chitosan molecules within the observation window. Under prolonged exposure to the electron beam, degradation of the chitosan matrix of the coating was observed, leading to the accumulation of ceramic TiN NPs at the electrode surface.

4. Conclusions

The study demonstrated the feasibility of real-time observation of the EPD process of pure chitosan and chitosan-TiN using in situ LC-TEM. The experiments provided direct insight into the migration, accumulation, and deposition of chitosan and TiN NPs under applied electric fields, revealing dynamic phenomena inaccessible to conventional ex situ characterization methods. The obtained results confirmed the strong influence of the composition of the dispersing phase in the dispersed systems and electron beam interactions on the deposition stability and deposit formation.

At the same time, several experimental challenges associated with LC-TEM investigations of polymeric systems were highlighted. The limited visibility caused by high local concentrations of chitosan molecules, electron beam-induced effects, liquid flow conditions, and the specific geometry of the electrode significantly influenced the observations. In particular, the deposition process could only be monitored at the thin edge of the electrode, while access to other deposition planes remained restricted.

Nevertheless, the work demonstrates the strong potential of LC-TEM as a powerful platform for investigating electrochemically driven assembly mechanisms in polymeric and nanocomposite polymer-based dispersed systems, opening new possibilities for the development of advanced functional micro- and nanostructured materials.

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New approaches for investigating beam-sensitive materials by TEM/STEM

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Since 1949, JEOL's legacy has been one of the most remarkable innovations in the development of instruments used to advance scientific research and technology. JEOL has more than 75 years of expertise in the field of electron.

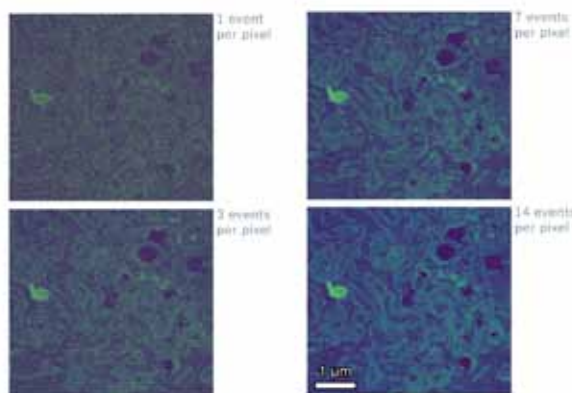


Figure 1. TEMPO images of human macrophage cells recorded with the beam blanker triggering after various event numbers. Contrast represents scattering-rate values. Data credit: turboTEM.

Beam-sensitive materials – including 2D crystals, metal–organic frameworks, zeolites, battery components, soft matter, and biological specimens – often undergo rapid structural and chemical alteration under electron irradiation, making conventional TEM/STEM characterization dose-limited. This work presents a practical framework for preserving both structural and chemical information under ultra-low-dose conditions by shifting from global beam reduction to precise dose delivery and information-efficient acquisition. After reviewing key damage mechanisms and the limits of standard parameter tuning (accelerating voltage and probe current), we outline a road-map built around four complementary strategies: (i) dose efficiency, extracting more information per electron through high-efficiency STEM detection modes (e.g., OBF/SAAF), optimized EDS geometry and solid angle, and frame-integration approaches that maintain resolution at reduced dose; (ii) dose control in time, using electrostatic dose modulation and pulsed operation to deliver

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electrons only when useful, while preserving alignment and resolution; (iii) dose control in space, minimizing unnecessary exposure via true area scanning, ROI-only acquisition, and spatial “dose painting” masks; and (iv) event-based acquisition, replacing fixed dwell time with event-driven imaging (TEMPO) to stop acquisition once sufficient information is collected (Fig. 1). We also highlight the emerging synergy between dose control and AI-based denoising to push imaging toward extreme low-dose regimes. Together, these approaches reposition dose from a limiting factor to a controllable parameter for robust TEM/STEM analysis of fragile materials.

In situ Extreme Micromechanics – Recent Innovations and Prospects

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Keywords: micropillar compression, micromechanical testing, in-situ, nanoindentation, smallscale ultra high strain rate testing

In situ SEM micro- and nanomechanical testing is an indispensable technique for materials design as well as for fundamental mechanics research. Many new protocols and testing geometries beyond traditional nanoindentation now enable the study of microstructure–property relationships, material intrinsic behaviour including orientation-dependence and plasticity, fracture dynamics, or the performance of novel micro-3D-printed metamaterials, to name but a few.



Figure 1. Alemnis Standard Assembly (ASA) configuration for ultra high strain rate testing and examples of typical test modes.

Thanks to its versatility, in situ SEM-based micromechanics is contributing to numerous scientific domains, including thin films and coatings, metallurgy, glasses and ceramics, semiconductors, biomechanics, or architected materials. Performing micromechanical tests in situ in a SEM offers two important advantages: (1) unmatched control, stability, and positioning accuracy, and (2) the possibility to perform unique correlative experiments based on, for example, the combination of mechanical data with direct imaging or EBSD measurements.

An increasingly important branch of micromechanical testing can be found in the simulation of real-world, extreme operation conditions, such as high temperatures in engines, cryogenic temperatures in hydrogen storage, dynamic loading under shock or impact, high frequency cyclic fatigue, or a combination thereof. Progress in the understanding of material behaviour at such conditions is clearly linked to the availability of laboratory equipment that can perform reliable tests under such conditions.

We present the most recent developments in instrumentation for in situ extreme mechanics testing at the micro and nanoscales. In focus is a testing platform capable of strain rate dependent

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testing over the range from 0.0001 s^{-1} up to $100'000 \text{ s}^{-1}$ (9 orders of magnitude) with simultaneous high-speed actuation and sensing capabilities with nanometer and micronewton resolution, respectively. Furthermore, the challenges and solutions to performing extreme micromechanics over the temperature range from -150°C to 1150°C , and the inherent advantages of using small volumes of sample material will be discussed. Finally, we present examples of such extreme micromechanical in situ tests and discuss future research directions in the field of extreme micromechanics.

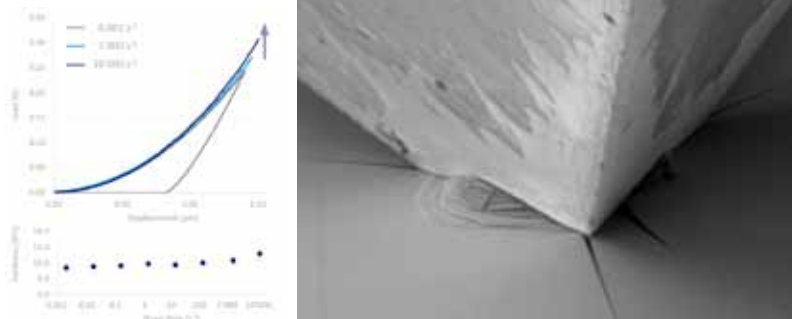


Figure 2. Example of nanoindentation data over 8 orders of magnitude of strain rate in fused silica.

Manufacturing and Unique Physicochemical Properties of Zr-based Bulk Metallic Glasses

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Keywords: bulk metallic glass, Zr-based alloys, amorphous structure, thermal stability, mechanical properties, biocompatibility

1. Introduction

Bulk metallic glasses (BMGs) represent a class of advanced structural and functional materials characterized by a fully or partially disordered atomic structure, obtained through rapid solidification of molten multicomponent alloys at cooling rates sufficient to bypass crystallization. Compared to conventional crystalline biomedical materials such as 316L stainless steel, Ti-6Al-4V and Co-Cr alloys, which suffer from limitations including ion release, allergenic reactions, high stiffness mismatch with bone, and wear-related failures, Zr-based BMGs offer an attractive combination of high strength, low elastic modulus, excellent corrosion resistance and potential biocompatibility. Their thermoplastic processability in the supercooled liquid region additionally enables shaping methods analogous to those used for polymers, opening a path toward additive manufacturing of complex, biomedical-grade components without the drawbacks typical of conventional metal 3D printing (high energy input, porosity, residual stresses). This work presents the manufacturing route and a comprehensive characterization of the microstructural, thermal, mechanical and biomedical properties of Zr-based BMGs, aiming to evaluate their suitability for advanced structural and biomedical applications.

2. Materials and methods

Zr-based BMG with nominal composition $\text{Zr}_{40}\text{Ti}_{15}\text{Cu}_{10}\text{Ni}_{10}\text{Be}_{25}$, $\text{Zr}_{50}\text{Ti}_5\text{Cu}_{10}\text{Ni}_{10}\text{Be}_{25}$, and $\text{Zr}_{40}\text{Ti}_{15}\text{Cu}_{10}\text{Ni}_5\text{Si}_5\text{Be}_{25}$ (at. %) [1,2] alloys were produced from high purity elements by arc-melting under a protective argon atmosphere, followed by suction casting into rod-shaped samples of 3 mm in diameter and approximately 20 mm length. Samples were prepared for testing by wire electrical discharge machining and subsequent surface oxide removal. Structural and compositional analysis was carried out using optical microscopy, scanning electron microscopy with energy dispersive spectroscopy, conventional and high-resolution synchrotron X-ray diffraction, atomic force microscopy and confocal microscopy. Thermal behaviour was assessed by non-isothermal and isothermal differential scanning calorimetry and thermomechanical analysis. Mechanical response was evaluated through nanoindentation and scratch tests, while biomedical performance was investigated via potentiodynamic corrosion tests in simulated body fluid, MTT cell-viability assays on hFOB1.19 osteoblast and IMR-90 fibroblast cell lines, and biofilm-formation assays. Thermoplastic behaviour was studied as uniaxial thermoplastic compression/joining tests.

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3. Results and discussion

Microstructural investigations by optical microscopy and SEM revealed a heterogeneous morphology in all Zr-based BMGs, consisting of an amorphous matrix embedding secondary crystalline phases. Area EDS analysis confirmed that the overall chemical composition of the cast rods matched the nominal alloy stoichiometry, while phase-resolved EDS mapping identified the nature of the observed precipitates: in Si-free alloys, bright regions correspond to Zr segregations and dark regions to Cu- and Ni-rich phases, whereas in the Si-containing alloy, Si partitions into dark $\text{Zr}_3\text{Ti}_2\text{Si}_3$ -type phases and bright Zr_3Si_2 - and $\text{Zr}_3\text{Cu}_4\text{Si}_2$ -type phases (Fig. 1). These findings indicate that even under controlled arc-melting and suction-casting conditions, complete suppression of crystallization in Zr-Ti-Cu-Ni-Si-Be systems is difficult to achieve, and that alloying additions such as Si actively influence the type and distribution of secondary phases formed during solidification.

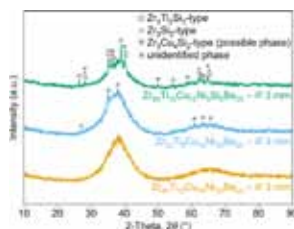


Figure 1. XRD diffraction pattern for the Zr-Ti-Cu-Ni-(Si)-Be alloys.

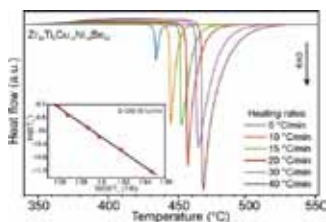


Figure 2. DSC curves together with Kissinger plot for the primary crystallization of the $\text{Zr}_{50}\text{Ti}_5\text{Cu}_{10}\text{Ni}_{10}\text{Be}_{25}$ bulk metallic glass.

Non-isothermal DSC scans confirmed the thermodynamically metastable nature of the manufactured $\text{Zr}_{40}\text{Ti}_{15}\text{Cu}_{10}\text{Ni}_{10}\text{Be}_{25}$ BMG, with crystallization occurring at elevated temperatures and overlapping exothermic peaks complicating direct interpretation of the thermograms. To resolve this, a deconvolution procedure combining a Gaussian function was applied, significantly improving peak fitting accuracy and enabling reliable extraction of characteristic temperatures (T_g , T_x) across heating rates from 5 to 40 °C/min. Kissinger-plot analysis of crystallization activation energies showed the $\text{Zr}_{50}\text{Ti}_5\text{Cu}_{10}\text{Ni}_{10}\text{Be}_{25}$ alloy exhibits the activation energy (249 ± 6 kJ/mol) for the first crystallization event. Faster heating rates were shown to permit reaching higher temperatures before the onset of significant crystallization, a practical consideration for thermoplastic processing protocols [3].

Mechanical property mapping across the cross-section of the manufactured rods confirmed spatial homogeneity, indicating that the casting process produces mechanically uniform material throughout the sample volume. Nanoindentation combined with K-means clustering allowed the discrimination of individual phases, showing that Zr-rich crystalline phases consistently exhibit higher elastic modulus (E_{IT}) and hardness (H_{IT}) than the surrounding amorphous matrix in all three Be-containing alloys, while in the Si-containing alloy the $\text{Zr}_3\text{Ti}_2\text{Si}_3$ -type phase reached the highest

values overall ($E_{IT} = 200 \pm 10$ GPa, $H_{IT} = 15.4 \pm 1.0$ GPa). At the macroscopic scale, elasticity index, resilience criterion and plastic deformation resistance were used to benchmark the alloys against conventional biomedical materials, and scratch testing demonstrated that all manufactured Zr-based BMGs outperform 316L stainless steel and Ti-6Al-4V in hardness, elastic recovery and wear resistance. Comparative analysis of scratch depths further showed that hard crystalline precipitates reduce elastic recovery of the scratch track but simultaneously limit residual scratch depth, indicating a trade-off between amorphous-phase elasticity and crystalline-phase hardness in determining overall wear performance.

Potentiodynamic corrosion testing performed in simulated body fluid (SBF), replicating physiological conditions, was used to evaluate the electrochemical stability of the manufactured BMGs in an environment representative of the intended biomedical application. The corrosion resistance observed across the tested compositions is consistent with the formation of a protective passive layer, a mechanism further corroborated by the subsequent cytotoxicity results, supporting the suitability of Zr-based BMGs for implantable applications where long-term stability in bodily fluids is required.

Cytotoxicity was assessed using MTT colorimetric assays on two clinically relevant human cell lines, the hFOB1.19 osteoblast line (Fig. 3) and the IMR-90 fibroblast line to evaluate both bone-forming and soft-tissue compatibility [1]. Osteoblast proliferation tests showed that cell viability and growth were maintained in the presence of the tested BMGs, indicating that the crystalline precipitations identified in the microstructural analysis do not exert an obvious negative impact on cytocompatibility.

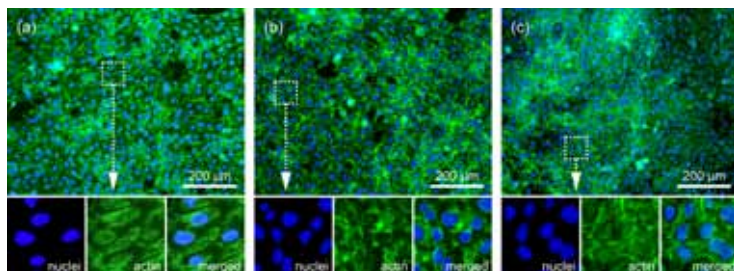


Figure 3. Fluorescence microscopy images of hFOB 1.19 osteoblasts cultured for 72 h on the (a) $Zr_{40}Ti_{15}Cu_{10}Ni_{10}Be_{25}$, (b) $Zr_{50}Ti_5Cu_{10}Ni_{10}Be_{25}$, and (c) $Zr_{40}Ti_{15}Cu_{10}Ni_5Si_5Be_{25}$ [1].

4. Conclusion

Three Zr-based bulk metallic glass alloys, $Zr_{40}Ti_{15}Cu_{10}Ni_{10}Be_{25}$, $Zr_{50}Ti_5Cu_{10}Ni_{10}Be_{25}$, and $Zr_{40}Ti_{15}Cu_{10}Ni_5Si_5Be_{25}$ (at. %) alloys, were fabricated by arc melting and suction casting and evaluated for prospective biomedical applications. Microstructural analysis revealed amorphous matrices containing composition-dependent crystalline precipitates. Thermal analysis confirmed their metastable character; the activation energy of the primary crystallization event for $Zr_{50}Ti_5Cu_{10}Ni_{10}Be_{25}$ was 249 ± 6 kJ/mol. Nanoindentation showed that Zr-rich crystalline phases had higher hardness and elastic modulus than the amorphous matrix. All alloys exhibited superior hardness, elastic recovery, and scratch resistance compared with 316L stainless steel and Ti-6Al-4V. Corrosion testing in simulated body fluid indicated effective passivation, while MTT assays using osteoblasts and fibroblasts showed no evident cytotoxic effects. The investigated alloys are therefore promising candidates for thermoplastically formed biomedical components and implantable devices.

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Dispersion state and properties of materials, from aluminum alloy to foods

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Keywords: high energy small-angle X-ray scattering (SAXS), nanostructure, slow operando

1. Introduction

For characterizing and correlating nanostructure to properties, it is important to get average feature of the structure as well as the detail of individual ones. Small-angle X-ray and Neutron Scattering (SAXS and SANS) are good tools for getting statistical representative values of size and volume fraction of constituent phase in multi-phase system, relatively easy way. Sample thickness for transmission mode-SAXS is often thought to be a few ten micro-meters, while using high energy source such as Mo-K α line, even 1 mm thick aluminium alloys can be measured. Therefore, we can start measurements of those alloys just after solution treatment without any further sample preparation process and continue to trace aging process by in-situ method. Another advantage to use laboratory SAXS with high energy source instead of large facility, we can keep measurements as long as we want. It fits to slow reaction such as room temperature (or lower temperature than zero degree) aging of aluminium alloys and some food making (or degrading) process, therefore, we call it “slow operando”. Here in this talk, we will show the importance of characterising dispersion state of multi-phase system using aluminium alloys, steels, thin films and some foods using laboratory high-energy SAXS and USAXS.

2. Experimental

Two SAXS system with Mo-source (Rigaku NanoViewer and NanoPix) are located in the same room with furnace, rolling machine, micro-Vickers and Differential Scanning Calorimeter to suppress the dead time from solution treatment to aging characterization. Pseudo Bonse-Hart type USAXS (Rigaku) with Mo-target is also located. Using those system, aging process of Al-Zn-Mg-(Cu) and Al-Cu-Mg with 1mm thick plate at and below room temperature were characterized.

Higher transmission power is also useful to characterize nanostructure of food. We can measure 5 mm thick cheese by sealing two cover glasses with diameter of 10 mm. This size of samples well-reflects state of real food. Using same cell, we can measure liquid sample, therefore, curdling (solidifying) state of milk to cheese can be studied.

Non-destructive measurements can applied distribution state of granular type thin film consists of Co particle with diameter below 5 nm embedded in ceramic matrix. In this case, we discuss the 3D array of Co particle by putting the sample at an incline of 40 degree from the normal sample position (perpendicular to incident X-ray beam).

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3. Results

Figure 1 shows SAXS profiles of Al-3.9Cu-1.5Mg of in-situ measurements at room temperature [1]. Since the measurements started in 10 min after solid solution treatment, evolution of Cu-enriched clustering can be clearly observed (Fig.1a). Because we start from thick samples, we can discuss the effects of cold rolling after solid solution treatments. Although the cold rolling enhances the clustering at the beginning, it suppresses the amount of clustering (Fig.1b). Observed difference give visible effects of the microstructural difference even after artificial aging at 190°C. Volume fraction and number density evaluated from SAXS can explain well about change of Vickers hardness by simple Orowan's mechanism.

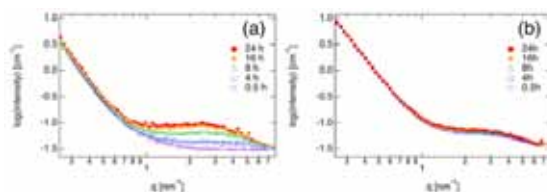


Figure 1. *In-situ* SAXS profiles of Al-3.9Cu-1.5Mg. (a) natural aging after solid solution treatment. (b) natural aging after cold rolling.

Natural aging sometimes makes us confuse when we storage and carry the sample to large facilities. Therefore, we check the aging process at lower temperature of Al-Zn-Mg-(Cu) [2]. Although clustering process of Al-6Zn-0.75Mg alloy proceeds even at -10°C (not shown here), that of Al-4.5Zn-2Mg-2.5Cu almost stops at 0°C and stops perfectly at -10°C after 72 hours as shown in Figure 2.

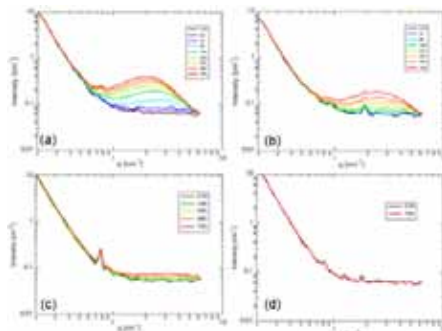


Figure 2. Time evolution of SAXS of Al-4.5Zn-2Mg-2.5Cu during aging at (a) room temperature, (b) 10°C , (c) 0°C and (d) -10°C .

Importance of characterizing nanostructure is same with food making process. Figure 3 shows nanostructure of cheese in the very early stage (just after forming pressed curd) [3]. It is well known that Ca cluster with diameter of 4 nm exists in milk. During curdling process, however, it is not clear how they change. *In situ* SAXS clearly shows that they form aggregates with about 6–7 nm in the ripening time from 1 to 5 days after making curd. The relationship between those nanostructures and texture of cheese is also discussed in this talk together with 3D-SAXS measurements of Co-based thin granular films.

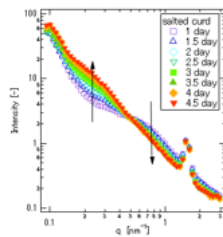


Figure 3. SAXS profiles of salt curd during the initial stage of production. “Day 0” is defined as the preparation of green curd. SAXS profiles were continuously measured every hour over a period of 4.5 days. The profiles for representative time of them are shown here.

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Overcoming EBSD Limitations Step by Step

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Keywords: EBSD, diffraction, phase analysis, microscopy

1. Introduction

Electron backscatter diffraction (EBSD) is one of the most widely used techniques for crystallographic microstructure analysis in the scanning electron microscope. In routine applications, EBSD provides rapid access to phase distribution, crystal orientation, grain boundaries, local misorientation and texture. Its strength comes from the combination of relatively fast data acquisition, sub-micrometre spatial resolution and direct coupling between the measured diffraction signal and the local crystallographic structure of the material. For this reason, EBSD has become a standard tool in the characterization of metals, ceramics, minerals and functional materials.

Despite this success, the standard EBSD workflow is still based on several simplifying assumptions. Kikuchi bands are usually expected to appear with normal contrast, each diffraction pattern is assigned to one dominant phase and orientation, and most indexing procedures reduce the measured diffraction image to a set of detected band positions. These assumptions are very effective for conventional orientation mapping, but they also define the practical limits of EBSD. When the experimental pattern does not follow the expected contrast, when more than one crystal contributes to the diffraction signal, or when the relevant information is contained in subtle intensity variations, standard indexing may fail or provide an incomplete interpretation.

The aim of this contribution is to show how these limitations can be overcome step by step by treating EBSD patterns as quantitative diffraction images rather than only as geometrical band maps. The presentation will focus on two recent developments: the analysis of inverted-contrast Kikuchi diffraction patterns and the separation of overlapping EBSD signals using experiment-simulation residuals. Both approaches are based on the same general idea: an apparent indexing failure often does not mean that crystallographic information is absent. Instead, the information may be present in a form that is incompatible with the assumptions of the standard analysis workflow. By changing the physical model used for the interpretation of the pattern, it becomes possible to recover information that would otherwise remain hidden.

2. Inverted contrast Kikuchi diffraction patterns

In conventional EBSD and TKD analysis, Kikuchi bands are usually interpreted as bright features with respect to the local background. However, under specific experimental conditions the contrast of the bands can be inverted. In such cases, Kikuchi bands appear darker than the surrounding background. This effect can be caused by anomalous absorption, nanoscale topography,

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low-tilt backscattering geometries or increased sample thickness in transmission Kikuchi diffraction. These conditions change the balance between diffraction, absorption and inelastic scattering, so that the intensity distribution in the measured Kikuchi pattern no longer corresponds to the assumptions used in standard EBSD indexing.

From the point of view of conventional data analysis, inverted contrast is problematic because the expected relation between lattice planes and band intensities is reversed. A pattern may contain well-defined crystallographic features and still remain non-indexed, simply because the indexing algorithm expects bright bands where the experiment shows dark ones. This is especially important for non-standard geometries and for samples where local thickness or topography cannot be fully controlled. In such cases, rejecting inverted patterns as failed measurements would unnecessarily reduce the usable area of the EBSD or TKD map.

To overcome this limitation, contrast-inverted physics-based master patterns can be used for orientation determination. Instead of comparing an experimental pattern only with a normal-contrast reference, the same crystallographic phase can be represented by both normal and inverted master patterns. The correct orientation is then obtained by selecting the best correlation between the experimental image and the appropriate contrast variant. In this way, the analysis is adapted to the actual physics of the diffraction image rather than forcing the image into the conventional normal-contrast model.

This strategy was demonstrated for EBSD of self-assembled Au nanostructures on Ge, EBSD from a non-tilted Ni sample and TKD of duplex stainless steel[1]. These examples represent different practical situations in which inverted contrast can appear. In the Au/Ge case, nanoscale topography and the geometry of the gold nanostructures strongly influenced the detected diffraction signal. In the low-tilt EBSD case, the conventional 70 degrees sample tilt was replaced by a non-standard geometry, leading to a different path length of the outgoing electrons. In the TKD case, the increasing local foil thickness caused a transition from normal-contrast to inverted-contrast diffraction patterns. In each case, the use of inverted master patterns significantly improved the interpretation of the experimental data. The results show that inverted contrast is not only an experimental complication, but also a useful diffraction signal when the physical model used for indexing is adapted accordingly.

3. Residual analysis of overlapping EBSD patterns

A second important limitation of conventional EBSD concerns overlapping diffraction signals. In routine EBSD mapping, each measured point is usually assigned to a single phase and orientation. This single-solution approach is convenient for visualization and data processing, but it is not always consistent with the physical origin of the EBSD signal. In real microstructures, the electron interaction volume may contain contributions from more than one crystal. This is especially important close to grain boundaries, phase boundaries, thin lamellae, nanoscale precipitates or complex multiphase regions. In such cases, the measured EBSD pattern is not produced by one crystal only, but is a superposition of several Kikuchi signals.

The overlap of diffraction patterns is one of the reasons why the physical resolution of EBSD can differ from the apparent resolution shown in an indexed map. Conventional indexing normally identifies only the strongest contribution, while weaker signals are ignored, treated as noise or lead to misindexing. As a result, minor phases or narrow microstructural features may be missed even though they contribute to the measured diffraction image. This problem is particularly relevant in steels, multiphase alloys and thin film systems, where small crystallographic regions can be located inside the interaction volume of a single EBSD measurement point.

Experiment-simulation residual analysis provides a direct way to address this problem. First, the dominant Kikuchi signal is identified using dynamic template matching. The corresponding simulated pattern is then optimized to match the experimental pattern as closely as possible. This

optimization may include intensity scaling, background correction, blurring and local gain adjustment, so that the simulated image represents the dominant experimental contribution in a realistic way. After subtraction of this fitted simulation from the experimental image, the remaining residual can reveal weaker diffraction contributions hidden below the dominant signal. The residual image can then be indexed again, and the procedure can be repeated iteratively.

In this way, a single EBSD pattern can be interpreted as a combination of multiple crystallographic signals rather than being forced into one solution only. This changes the role of the residual image. In a conventional comparison between experiment and simulation, the residual is often treated mainly as an error signal. In the present approach, however, the residual becomes a source of additional crystallographic information. If the strongest contribution is removed correctly, the remaining image can contain physically meaningful Kikuchi features from a second phase, a neighbouring grain or a weakly diffracting microstructural component.

This approach was applied to several materials systems, including pearlitic steel, two-phase bronze, dual-phase steel, tool steel and a CuPt thin film on a SrTiO₃ substrate[2]. In these examples, residual analysis improved the detection of minor or hidden phases such as cementite lamellae, retained austenite, carbides, secondary BCC phases and twin-related domains[2]. In the case of dual-phase steel, the method helped to identify retained austenite located close to ferrite-martensite boundaries. In other examples, residual indexing made it possible to separate overlapping phase contributions that were not resolved by the first indexing step. These results demonstrate that residual analysis can reveal microstructural information that is physically present in the EBSD pattern but inaccessible to a standard one-pattern-one-solution workflow.maps.

4. Step-by-step extension of EBSD analysis

The two main examples discussed here are part of a broader development in EBSD analysis. Earlier work showed that full-pattern and simulation-based methods can be used to map local lattice parameter ratios and tetragonality in martensitic steels[3,4]. Similar ideas enabled orientation analysis of quasicrystals using approximant structures or experimental Kikuchi reference patterns[5,6]. Full-pattern matching also made it possible to resolve the chirality of quartz by exploiting dynamical diffraction effects that are not available in conventional centrosymmetric indexing workflows[7].

Together, these examples show that many apparent EBSD limitations are not fundamental limitations of the diffraction signal itself, but limitations of the interpretation model. By including more realistic diffraction physics and by using the full intensity distribution of the Kikuchi pattern, EBSD can be extended to cases that are difficult or impossible for standard indexing. This includes inverted contrast, alternative measurement geometries, overlapping patterns, hidden minor phases, local lattice distortions, quasicrystal orientation analysis and chirality mapping.

The central conclusion is that EBSD should increasingly be treated as quantitative diffraction imaging in the SEM. Conventional EBSD remains essential for fast and robust mapping, but advanced pattern matching and residual-based workflows provide additional tools for complex materials and non-standard experimental conditions. In this sense, the limitations of EBSD can be overcome step by step by adapting the analysis workflow to the physical information already present in the measured diffraction patterns.

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The way to Ni-Mn-Ga jelly-like martensitic structures: ab initio study

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Keywords: NiMnGa. Ferromagnetic shape memory alloy, ab initio methods, phase transformation

1. Introduction

Among ferromagnetic shape memory alloys, compositions based on the Ni₂MnGa Heusler-type alloy are receiving significant scientific attention, as their 10M and 14M modulated martensites exhibit giant magnetic-field-induced reversible strain reaching up to 12 % [1] under low applied stresses falling below 0.1 MPa [2]. The martensitic transformation from the austenite phase with an L21 cubic structure is induced either by cooling below the martensitic transformation temperature or by the application of mechanical load. However, the final martensitic structure and the resulting ferromagnetic shape memory effect strongly depend on the exact chemical composition [3]. For certain compositions (for example Ni₅₀Mn_{28.2}Ga_{22.8}), the martensitic transformation austenite ↔ 10M martensite is followed by intermartensitic transformation sequence 10M ↔ 14M ↔ NM. Due to that, the superelastic strain of such compositions reach up to 10% [4,5]. Modulated martensitic structures can be interpreted as assemblies of nanoscale twins of NM martensite within the adaptive martensite concept [6]. Their detwinning or rearrangement of these layers can lead to NM formation. Nevertheless, there are differences in orthogonal lattice parameters and internal angles between observed structure and nanotwinned 10M, 14M, and NM martensitic structures [7,8]. Thus, the continuous atomic-plane shuffling along (110) planes leading to so-called jelly-like structure provides a more consistent description. In this work, we integrate the adaptive martensite framework, represented by DFT studies on generalized planar fault energy (GPFE) of NM martensites with varying Mn content and different local atomic and magnetic arrangements, with physically observed shuffle/switching mechanism to describe both the origin of structural modulations and intermartensitic transformations in non-stoichiometric Ni–Mn–Ga alloys.

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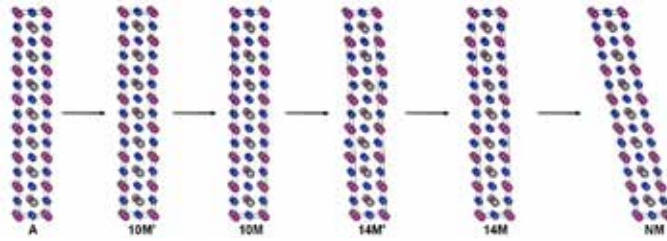


Figure 1. The transformation sequence covering both martensitic and intermartensitic transformations of Ni-Mn-Ga ferromagnetic shape memory alloys. Ni atoms are blue, Mn atoms are purple and Ga atoms are grey.

2. Methods

To determine the phases and their crystal structures, high-energy synchrotron radiation (87.1 keV) conducted on the P07B beamline at DESY, Hamburg, Germany, was employed [9]. Diffraction patterns were collected in transmission mode to probe the entire sample volume in continuous rotation mode around the ω -axis from -10° to $+10^\circ$. The scattered intensity was recorded using a 2D detector located at a distance of 1230 mm from the sample.

For in situ temperature-dependent studies, a Peltier cell was used to vary the sample temperature between 273 K and 343 K. Lower temperatures were achieved using a cold-finger cryostat consisting of a copper rod cooled with liquid nitrogen.

Presented calculations were performed using the spin-polarized DFT method implemented in the Vienna Ab Initio Simulation Package (VASP) [10,11]. The monoclinic supercells contained 32 atoms on 8 (101) planes were used. Up to two Ga atoms were replaced by Mn to reach Mn-excess of 6.25 at. %. During the simulation of twin reorientation, the lower four atomic planes were kept static, while there was a displacement cumulatively applied to the upper four planes creating planar faults.

3. Results and discussion

Our results show that all observed non-stoichiometric Ni-Mn-Ga samples undergo the same intermartensitic transformation sequence: austenite $\leftrightarrow$ 10M' $\leftrightarrow$ 10M $\leftrightarrow$ 14M' $\leftrightarrow$ 14M $\leftrightarrow$ NM with many intermediate and locally disordered structures observed during the transformation. Such observations indicate that the transformation is governed by gradual shuffling or switching of (110) planes composing various two-, three- or five-planes-high stacking units, hence it can be called the *jelly-like* behavior of the modulated lattice. There are two key factors supporting our hypothesis. First one is the interplanar distance of both (220) and (110) planes, which remains constant throughout the transformation (Figure 2a). Secondly, the proposed reversible intermartensitic transformation mechanism requires very low energy allowing the structure to be rearranged easily. The mechanism cannot be treated as twinning due to lack of mirror symmetry between most of the stacking units, as well as there is no homogeneous defined across (110) planes.

To understand various effects leading to minimal stress needed to rearrange abovementioned stacking units, GPFE curves of Mn-rich NM martensite provided us valuable data. As is depicted in Figure 2b, the height of the energy barriers during structural rearrangement with constant interplanar distance between (110) planes generally increases with rising Mn content when Mn-excess atoms occupying Ga sublattice sites exhibit antiparallel magnetic moment orientations, representing the ground-state magnetic configuration. For parallel magnetic moment orientation, the barriers decrease or even disappear for high Mn concentrations. This

configuration, however, has higher total energy and thus it also has lower thermodynamic stability. The impact of Mn presence in Ga sublattice is even more evident, if Mn-excess atoms are located within or near the moving plane [12]. The increased energy barriers observed in the ground-state AFM configuration are attributed to strong antiferromagnetic interactions between Mn-excess atoms and regular Mn atoms, which hinder twin propagation and consequently increase the twinning stress. The height of energy barriers during lattice reorientation also correlates with tetragonal ratio, as it ranges from 1.21 for 6.25 % Mn-excess and completely ferromagnetic state, to 1.3 for the same composition with all excess-Mn atoms' magnetic moments oriented antiparallely [12]. It should be denoted that PBE method generally overestimates c/a parameter. Concluding our results, the reorientation of a structure composed of various stacking units can be easier, if the displacement originating from c/a ratio is as small as possible. This applies on large FM regions, while antiparallely oriented magnetic moments of Mn in Ga sublattice, especially in the nearest vicinity of shuffling plane acts as the stabilizers of structure, helping with formation of two-plane-high stacking units due to their extraordinary stability.

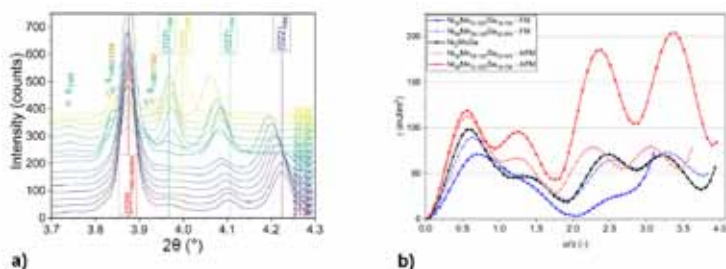


Figure 2. (a) Enlarged view of the selected region of the diffraction patterns highlighting the invariant position of the reflection corresponding to the (220) crystallographic plane. (b) The comparison of Ni-Mn-Ga GPFE curves in dependence on Mn content and excess-Mn magnetic order.

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Micromechanical Characterization of Intermetallic Phases in Zn Alloys: From EBSD Orientation Mapping to Small-Scale Deformation

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Keywords: zinc alloys, EBSD, micromechanics, micro-pillar compression, nanoindentation

1. Introduction

Zinc-based intermetallic phases appear in a wide range of technologically relevant material systems from galvanized steel coatings and Zn-Al-Mg protective layers to emerging bioresorbable Zn alloys for temporary implant applications. In both contexts, the mechanical behaviour of individual intermetallic phases governs critical performance aspects: coating integrity and adhesion on one hand, and in vivo degradation rate, structural support, and biocompatibility on the other. Despite their practical importance, the intrinsic mechanical properties of these phases at the micro-structural level, and their dependence on crystal structure and orientation, remain largely unexplored. We present the first systematic study combining electron backscatter diffraction (EBSD) with site-specific nanoindentation and micropillar compression to evaluate hardness, elastic modulus, and plasticity of Zn-based intermetallic phases as a function of crystallographic orientation. EBSD mapping enables precise identification and orientation determination of individual phase grains prior to micromechanical testing, while focused ion beam (FIB) milling allows targeted micropillar fabrication within selected grains.

2. Materials & Sample Preparation

A binary Zn-8Cu (wt.%) alloy was prepared by induction melting and cast under controlled conditions, followed by slow solidification to promote equilibrium phase formation. To ensure a well-defined and homogeneous intermetallic microstructure, the as-cast material was subjected to annealing at 400°C for 7 days, followed by slow furnace cooling. Bulk specimens were sectioned and metallographically prepared through standard grinding and polishing steps, with a final colloidal Al₂O₃ suspension polish to achieve a surface quality suitable for EBSD analysis. Crystallographic characterization was performed on a ThermoFisher Scientific Helios 5 dual-beam SEM equipped with an Oxford Instruments Symmetry 3 EBSD detector, using AztecCrystal software for large-area automated acquisition. EBSD maps were collected across the entire sample surface of approximately 15×10 mm, providing statistically representative orientation data for all constituent phases. From the resulting orientation dataset, individual CuZn₄ grains were carefully selected to populate the full inverse pole figure (IPF) triangle – spanning the complete orientation space from

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$\langle 0001 \rangle$ to $\langle 01-10 \rangle$ to $\langle -12-10 \rangle$ – ensuring comprehensive crystallographic coverage for subsequent micromechanical testing (Fig. 1). Micropillars were machined by Xe plasma focused ion beam (Xe-FIB) milling at orientations of interest identified by EBSD. In-situ nanoindentation was performed inside the SEM chamber using a Femtotools FT-NMT04 nanoindenter, equipped interchangeably with a cube corner tip for hardness and modulus measurements and a flat punch tip for uniaxial micropillar compression.

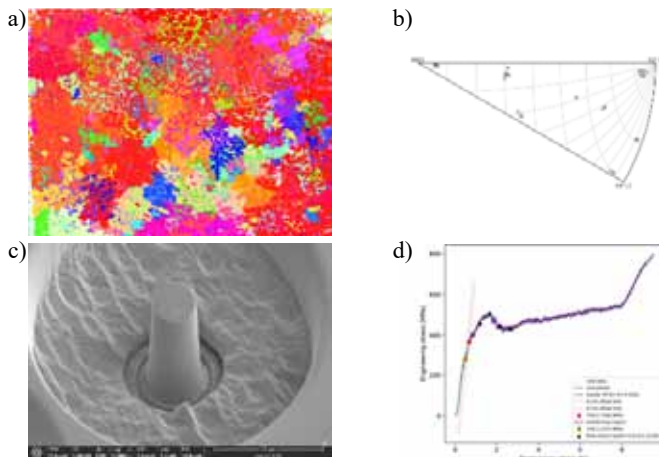


Figure 1. Overview of the experimental workflow applied to the CuZn₄ intermetallic phase in the Zn-8Cu alloy. (a) Large-area EBSD IPF map revealing the grain orientation distribution across the sample surface. (b) IPF triangle showing the crystallographic orientations of grains selected for micropillar compression, spanning the full orientation space from $\langle 0001 \rangle$ to $\langle 01-10 \rangle$ to $\langle -12-10 \rangle$. (c) SEM image of a representative Xe-FIB machined micropillar (diameter $\sim 4 \mu\text{m}$, height $\sim 12 \mu\text{m}$) with the surrounding annular trench, prior to flat punch compression. (d) Representative engineering stress-strain curve obtained from micropillar compression, showing a Young's modulus of $\sim 97.4 \text{ GPa}$, well-defined yield points at 0.1% and 0.2% offset ($YS_{0.1} = 275 \text{ MPa}$, $YS_{0.2} = 368 \text{ MPa}$), a pronounced strain hardening region, and a flow stress approaching $\sim 800 \text{ MPa}$ at $\sim 8\%$ engineering strain demonstrating unexpected but significant plasticity in this intermetallic phase.

3. Results

Nanoindentation performed on the CuZn₄ intermetallic phase with indentation sites selected according to EBSD-determined orientations reveals a clear orientation dependence of both hardness and elastic modulus, with values ranging from 2006 to 2591 MPa and 113 to 123 GPa, respectively. It should be noted that measurements were carried out using a cube corner indenter geometry, which, while well-suited for hardness determination and minimizing the indentation size effect, is not optimal for elastic modulus extraction due to deviations from the ideal Berkovich contact mechanics assumed in the Oliver-Pharr framework; the reported modulus values should therefore be interpreted with appropriate caution. Nevertheless, the orientation-dependent trend in stiffness is consistent and meaningful, further confirming that the mechanical response of CuZn₄ is inherently anisotropic. Complementary micropillar compression experiments, conducted on FIB-machined pillars with a diameter of approximately $4 \mu\text{m}$ and height of $12 \mu\text{m}$, reveal a striking orientation dependence of the yield strength, which varies from 350 to 1000 MPa across differently oriented grains. A representative example of the full experimental workflow – from EBSD grain selection through micropillar geometry to the resulting stress-strain response is presented in Figure 1.

Remarkably, despite its intermetallic character, CuZn4 exhibits pronounced strain hardening and measurable macroscopic plasticity behaviour not commonly expected in such phases and with significant implications for the mechanical integrity of Zn-based microstructures. These findings are interpreted in terms of differences in active slip systems, atomic bonding anisotropy, and structural packing density as a function of loading direction relative to the crystal lattice.

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NanoMEGAS enhanced 4D STEM solutions: latest developments combining precession with high throughput hybrid pixel cameras

Philippe Ayasse

Precession Electron Diffraction – More Information from Every Scan Point

Precession Electron Diffraction (PED) is a technique that significantly expands the capabilities of electron diffraction in TEM/STEM. Instead of analyzing diffraction with a stationary conventional beam, the electron beam performs a controlled precessional movement around the optical axis. Precession mainly reduces dynamical diffraction effects and increases the diffraction patterns resolution, enhancing significantly the patterns quality towards reliable crystallographic analysis and materials properties studies.

Precession enhanced 4D-STEM (4D-SPED). However, precession itself is only part of the story. With NanoMEGAS robust applications, precession is synchronized with beam scanning, allowing series of PED pattern to be acquired for each point of the scanned area. This direct spatial correlation between the probe position and the corresponding diffraction pattern is particularly important for high-resolution **orientation, phase, strain, electric field** and **ePDF** (for amorphous materials characterization) mapping. In addition, the NanoMEGAS acquisition system is now **compatible** with numerous of **high-end detectors**, for enhanced data quality and high-speed 4D-SPED.

Latest developments, offer a high speed precession enhanced 4D-STEM, up to **20,000 fps**, and a **sparse sampling** acquisition for 4× to 10× faster acquisition and subsampling scan, suitable for beam sensitive materials.

The resulting datasets can be used for:

- Crystallographic orientation mapping,
- Phase identification and mapping,
- Grain boundary and defect analysis,
- Strain mapping,
- Electric Field mapping,
- ePair Distribution Function (ePDF) for amorphous materials characterisation,
- Nanocrystal analysis,
- 3D electron diffraction (3DED) and crystal structure determination.

It is precisely the combination of precession + controlled beam scanning + high-end detectors + synchronized data acquisition that transforms a series of diffraction patterns into a spatially resolved dataset, from which crystallographic and other material properties can be mapped.

Precession is therefore not simply about moving the electron beam. It is a way of obtaining more reliable crystallographic information from every point of the sample, while providing a powerful opportunity to expand the capabilities of both new and existing TEM systems.

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TEM investigation of NiMo/C coatings developed for Hydrogen Evolution Reaction

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Keywords: NiMo/C, HER, magnetron sputtering, in-situ TEM

1. Introduction

The electrolysis of water may back the H₂ storage sites intended for green transport especially in places lacking in ground sources of this gas. The catalytic properties of platinum electrodes allows to lower the over-potential of this process and diminish the overall production costs. The high price of this metal even as used only in the form of coating is still hindering the large scale production of the H₂ using this rout. The catalytic properties of the platinum are to some extent matched by those of nickel, which in a way is not a cheap material by itself, especially as both acid environment and raised temperature contribute to relatively fast corrosion of such electrodes. A solution in this case might be sought by applying the this material in the form of coating. The magnetron deposition technique belonging to physical vapour deposition (PVD) group secures both high deposition rates and excellent adhesion of most metallic coatings. However, using this technique for obtaining nickel coatings requires alloying of the nickel magnetron cathodes with up to ~15 at. % Mo, Ti, or Cr, cancelling its ferromagnetic properties. Magnetron sputtering of such cathodes in the mixture of argon and methane (CH₄) or acetylene (C₂H₂) allows – at higher concentration of reactive gases – to obtain metallic nano-rods in carbon shells [1]. The preliminary experiments performed using paramagnetic Fe₆₉Cr₁₈Ni₁₁Mo₂ (316L) steel indeed allowed to produce of a series of microstructures, starting from columnar, nano-crystalline layers for those deposited at high Ar and low C₂H₂ content, through amorphous and finally amorphous metallic nano-rods immersed in amorphous carbon shells for those reactively sputtered with increased ratio of carbon carrying gass [2]. The *in-situ* heating experiments helped to establish that such coatings starts to crystallize already at 400°C producing predominantly the α-Fe phase, while presence of a lesser amounts of graphite and nickel-iron-carbon rich particles was also documented [3]. The following experiments with coatings obtained from cathodes of much higher Ni content (i.e. Ni-20 at. % Mo spattered in Ar and C₂H₂) take form of amorphous metallic nano-rods, which diameter decrease with increasing C content [4]. The *in-situ* heating up to 550°C

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caused precipitation of eqiaxed crystallites of α -Ni(C) phase separated by graphite strands, while Mo was redistributed in-between them.

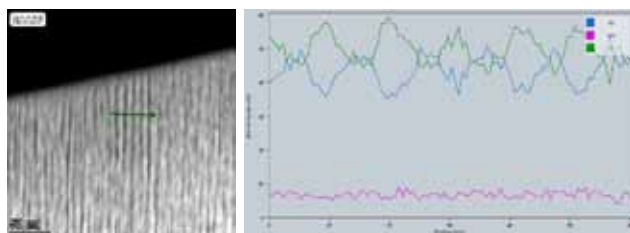


Figure 1. STEM/HAADF image of cross-section microstructure of NiMo coatings having 74 at. % C and accompanying TEM/EDS profile presenting changes in chemical composition across the nano-rods.

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Structural and morphological characterization of Ni–Mn–Ga powders produced from melt-spun ribbons

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Keywords: Ni–Mn–Ga powders, SEM, TEM, particle size distribution, microstructure

1. Introduction

Ni–Mn–Ga alloys belong to the group of ferromagnetic shape memory alloys, in which the martensitic transformation can be induced not only by conventional stimuli, such as changes in temperature or stress, but also by an external magnetic field. This phenomenon is related to the ability of martensite variants to reorient in a magnetic field, leading to magnetic-field-induced strain. Large values of this effect have been observed in Ni–Mn–Ga single crystals [1]. However, in polycrystalline materials, this effect is significantly reduced. Mainly, it is due to grain boundaries, random crystallographic orientations, and the restricted motion of twin boundaries. An additional limitation is their pronounced brittleness [2].

In the present work, the alloy's brittleness was considered an advantageous feature for powder production. It was assumed that mechanical fragmentation would enable the production of Ni–Mn–Ga powder with a particle size distribution dependent on the milling time. This study aimed to characterise the morphological and structural changes that occur during vibratory milling of ribbons.

2. Experimental procedure

The investigations were carried out on a Ni–Mn–Ga alloy, with a nominal chemical composition of Ni 51.5 at.%, Mn 27.5 at.% and Ga 21.0 at.%. The starting material in the form of a bulk alloy was produced by electromagnetic levitation melting. Then, the alloy was subjected to rapid solidification realized by melt-spinning. Due to the brittleness of the material, mainly flakes with a width of approximately 6 mm and a length of up to several centimetres were obtained instead of continuous ribbons. These flakes were used as the starting material for powder production by vibratory milling. The milling time was the variable process parameter. A separate batch of material was prepared for each planned milling. Process was carried out for 15 min, 30 min, 1 h, 3 h and 4 h.

After each processing stage, the material was analysed in terms of morphology, chemical and phase composition as well as particle size. Morphological observations and chemical composition analysis were performed using a JSM-7100F TTL LV scanning electron microscope equipped with an SEM/EDS system. The phase structure was determined by X-ray diffraction using an X'Pert-PRO

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diffractometer. Additional microstructural observations were carried out using a JEOL 3100 transmission electron microscope. The average particle size of the powder was determined by laser diffraction using an Analysette 22 NeXT particle size analyser (Fritsch).

3. Results

Figure 1 shows examples of SEM images observed for the ribbon and the powder after vibratory milling for 30 min and 4 h. The as-spun ribbon exhibits a typical microstructure of a rapidly solidified material. It consists of a fine-grained layer near the free surface and elongated columnar grains observed in the fracture cross-section. This morphology indicates directional crystallisation associated with rapid heat extraction during cooling (Figure 1a). After 30 minutes of milling, the material was fragmented into irregular, sharp-edged particles with a wide size distribution. Their sharp-edged morphology, local cracks and cleavage-like features indicate brittle fragmentation of the ribbon. It indicates that fragmentation of the starting material is the dominant mechanism at the initial stage of milling. Extending the milling time to 4 h leads to further powder refinement and an increased fraction of finer grains. The powder consists mainly of grains with a diameter of approximately 10 μm ; however, larger fragments with sizes up to about 40 μm are still visible. The observed morphology indicated intensified mechanical interactions during prolonged milling, accompanied by simultaneous fragmentation and agglomeration.

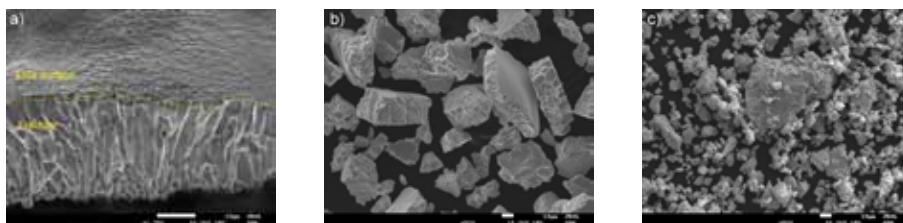


Figure 1. SEM images of the Ni-Mn-Ga alloy: cross-section of the melt-spun ribbon (a), powder milled for 30 min (b), and 4 h (c).

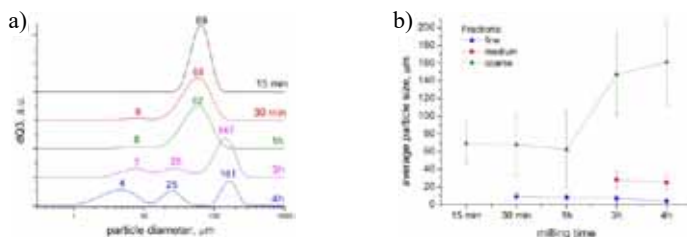


Figure 2. Particle size analysis of Ni-Mn-Ga powders after milling for 15 min, 30 min, 1 h, 3 h and 4 h: derivative curves of the particle size distribution (a); average particle sizes of the separated fine, medium and coarse fractions (b).

The SEM observations were complemented by a quantitative particle-size distribution analysis using laser diffraction. The results are presented in Figure 2. The derivative curves of the particle distribution shown in Figure 2a confirm the progressive refinement of the material with increasing milling time. They also indicate a change in the distribution character from unimodal to multimodal. After 15 min of milling, the particle size distribution was unimodal, with the dominant fraction averaging approximately 69 μm . Extending the milling time to 30 min and 1 h resulted in the appearance of an additional fine fraction, with average sizes of approximately 9 and 8 μm , respectively, while the main fraction of about 68–62 μm remained. Further extension of the milling time to 3 and 4 h resulted in a

clear change in the particle distribution. It becomes trimodal, consisting of fine, medium, and coarse fractions. For the powder milled for 3 h, the average sizes of these fractions were 7, 28, and 147 μm , whereas after 4 h they were 4, 25, and 161 μm , respectively. The average particle sizes of the individual fractions as a function of milling time are summarised in Figure 2b, which provides a clearer view of the direction of the process changes. For the fine and medium fractions, a decrease in the average particle size with increasing milling time was observed, confirming the progressive fragmentation of the material. In contrast, the coarse fraction showed the opposite trend, with its average size increasing with longer milling times. This may indicate that, under intensive mechanical interactions, further fragmentation was accompanied by agglomeration of finer particles or by the presence of larger fragments.

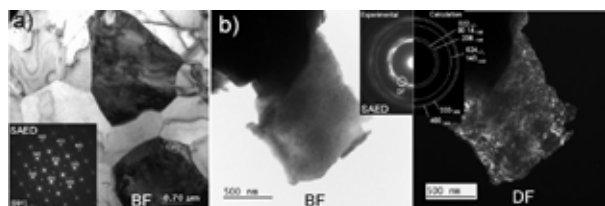


Figure 3. TEM images with corresponding SAED patterns of the Ni–Mn–Ga alloy: initial ribbon (a), powder after 30 min of milling (b).

Vibratory milling not only refined particles but also induced significant structural and phase changes, as confirmed by the TEM observations and SAED analysis shown in Figure 3. For the as-spun ribbon, the bright-field image together with the diffraction pattern recorded along the $[001]$ $L2_1$ zone axis confirms the presence of a well-crystallised, highly ordered $L2_1$ phase (Figure 3a). Observations support the results of laser diffraction, indicating that particle refinement began even after a short milling time. Representative TEM images of the powder milled for 30 min are shown in Figure 3b. The dark-field image reveals crystalline nanograins, proving that a short milling time results in nanocrystallisation of the material. The analysis of electron diffraction pattern showed the coexistence of reflections from the $L2_1$ phase and 14M martensite. This confirms the initiation of the $L2_1 \rightarrow 14\text{M}$ transformation induced by mechanical stresses generated during vibratory milling.

4. Summary

The results showed that vibratory milling of Ni–Mn–Ga alloy ribbons yields a powder with particles/grains in the micrometre size. With increasing milling time, progressive refinement of the material and a shift in the particle size distribution from unimodal to multimodal were observed. The analysis of the separated fractions revealed a decrease in the average particle size of the fine and medium fractions, accompanied by the presence of a coarse fraction. It proved the simultaneous occurrence of fragmentation and agglomeration during prolonged mechanical treatment. TEM observations confirmed that milling not only results in particle refinement down to the micro- and nanoscale but also induces structural and phase changes. The presence of crystalline nanograins and the mechanically induced transformation of the highly ordered $L2_1$ parent phase into 14M martensite were confirmed.

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Effect of doping (W, Cr, Zr) on the structure and mechanical properties TiBy coatings

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Keywords: coating, TiB₂, PVD

1. General

TiB₂-based coatings have been intensively developed due to their physical and mechanical properties, including excellent thermal stability and high hardness with good abrasion and corrosion resistance, which appear to be the most beneficial in industrial application. The main disadvantage is the low resistance to brittle fracture. This parameter can be changed by doping (W, Ni, Cr, Zr), deposition non-stoichiometric coatings TiB_x or deposition multilayer nanometric coatings.

The aim of this study was to analyze the effect of doping TiBy coating by W, Cr and Zr on the microstructure and mechanical properties. The structural characterization of Ti_{1-x}M_xBy (M=W, Cr, Zr) coatings was carried out using X-ray diffraction, X-ray photoelectron spectroscopy, transmission electron microscopy and atomic force microscopy. To investigate the mechanical properties, nano-scratch and—hardness tests (NST,NHT) were performed, and fracture toughness of the substrate layer systems was determined. There are two factors that influence on properties of doping coatings : first changing the preferred orientation of the coating from (102)(111) to (100)—this increases hardness and wear resistance; second reducing crystallite size and column width increases ductility and fracture toughness by blocking or changing the direction of cracks. Both factors occur simultaneously in the coatings depend on the elements (W, Cr, Zr) content.

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Structure and corrosion resistance of CoCrFeNiNb high entropy alloys produced by the rapid solidification

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Keywords: high entropy alloys, copper die casting, neutron diffraction,
electron microscopy, electrochemical measurements

1. Introduction

The high entropy alloys (HEAs) represent a new research direction in material engineering. Unlike the traditional approach to alloy design, its development requires a multi-element composition at concentrations between 5 and 35 at.%. As a result, these alloys exhibit significantly higher configurational entropy than conventional alloys, which contributes to improved solid solubility of the constituent elements, thus inhibiting the formation of intermetallic phases. Although these alloys are mainly multicomponent materials, they can crystallize as single- or dual-phase alloys. HEAs can exhibit exceptional properties, such as great mechanical properties at elevated temperatures, high fracture toughness, and superior corrosion resistance. They are characterized by great potential for application in many fields, such as aerospace, marine, and chemical industries [1–6].

The method often applied is to introduce refractory elements, such as Mo, Nb, or W [7]. Their addition, as a result of larger atomic radii, causes an increase in lattice distortion, resulting in the strengthening of the solid solution, and, in larger quantities, contributes to the precipitation of hard intermetallic phases, such as the Laves phase for CoCrFeNiNb_x [8,9]. The synergistic effect of solid solution and precipitation strengthening leads to a significant improvement in mechanical properties [10]. In the case of the CoCrFeNiNb_{0.45} alloy, an exceptional compressive strength was obtained, exceeding 2550 MPa, while simultaneously retaining 27.9% of the fracture deformation, which is attributed to its nanolamellar eutectic microstructure [11]. Because of the larger atomic radius, niobium increases lattice distortion in larger amounts causing destabilization of the solid solution phase resulting in the formation of intermetallic phases. The addition of niobium also affects the corrosion behaviour of these alloys, contributing to the formation of a passive layer consisting of niobium oxides on their surface [12,13].

The purpose of this work is to investigate the selected properties of the CoCrFeNiNb_x alloy ($x = 0.25, 0.45, 0.65$ and 1.0) prepared from the liquid state with two different cooling rates obtained after slow cooling of the ingots and a high cooling rate during the copper mould casting. The effects of the different niobium content and the cooling rate on the structure, and corrosion resistance were analyzed.

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2. Materials and methods

The studied HEAs were prepared in the form of ingots and plates, using high purity elements (99.99 wt.%). The chemical compositions of the alloys are listed in Table 1. Alloys in the form of ingots were obtained by induction melting method with the use of a NG-40 induction generator, under an argon atmosphere. The ingots were solidified with a low cooling rate using ceramic crucibles. Plates with 1 mm thicknesses were prepared by remelting ingots and pressure casting using a copper mould [5].

Table 1. Nominal chemical compositions of the studied high entropy alloys [at.%].

Alloy	Co	Cr	Fe	Ni	Nb
CoCrFeNiNb _{0.25}	23.53	23.53	23.53	23.53	5.88
CoCrFeNiNb _{0.45}	22.47	22.47	22.47	22.47	10.12
CoCrFeNiNb _{0.65}	21.505	21.505	21.505	21.505	13.98
CoCrFeNiNb	25.00	25.00	25.00	25.00	25.00

The phase composition of the alloys was analysed using the PEARL neutron diffractometer at the TU Delft Reactor Institute, The Netherlands. The samples were packed in a vanadium container. The Ge monochromator was used, which selected neutrons with a wavelength of $\lambda = 0.167$ nm, covering the angular range between $10^\circ \leq 2\theta \leq 160^\circ$. Data analysis was carried out using the Rietveld refinement method in the FullProf suite. The microstructure of ingots and plates of different thicknesses was analysed using the Zeiss Supra 35 high-resolution scanning electron microscope (SEM), in the back-scattered electron (BSE) detection mode. Image analysis was performed using Image J software. Chemical element maps were obtained using energy-dispersive X-ray spectroscopy (EDX). Transmission electron microscopy (TEM) observations were performed using S/TEM TITAN 80–300 FEI microscope at voltage of 300 kV. The lamellas for TEM observations were prepared with the use of focused ion beam milling.

To evaluate the corrosion resistance of the CoCrFeNiNb alloys, electrochemical measurements were performed in an environment of a 3.5% sodium chloride solution at a temperature of 25°C. The Autolab 302N potentiostat equipped with a three-electrode measuring system was used for the study. The instrument was controlled with NOVA 1.11 software. A saturated calomel electrode (SCE) was used as a reference electrode, platinum wire was used as a counter electrode, and the material was tested as a working electrode. Changes in the open-circuit potential (E_{OCP}) were recorded before polarization and impedance tests. Electrochemical impedance spectroscopy measurements were performed at open circuit potentials. Impedance data was collected in the frequency range of 10^{-2} – 10^5 Hz, using a perturbation signal with an AC amplitude of 5 mV. Polarization measurements were conducted in the range of –400 to 400 mV, with a scan rate of 1 mV/s. The corrosion potential (E_{corr}) and the corrosion current density (j_{corr}) were also determined using the Tafel extrapolation method.

3. Results and conclusions

The CoCrFeNiNb_x ($x = 0.25, 0.45, 0.65$ and 1.0) alloys are characterized by a double-phase structure, consisting of FCC solution and Co₂Nb-type Laves phase. An increase in the niobium content caused the evolution of the microstructure from hypoeutectic, in the case of the CoCrFeNiNb_{0.25} and CoCrFeNiNb_{0.45} alloys, to hypereutectic for the CoCrFeNiNb_{0.65} alloy [14]. The Rietveld refinement of the neutron diffraction pattern for the CoCrFeNiNb_{0.65} sample in a plate form carried out using FullProf is shown in Figure 1. The phase fraction obtained for the two identified phases is 48.57% for the Fe-FCC solid solution and 51.43% for the Co₂Nb structure.

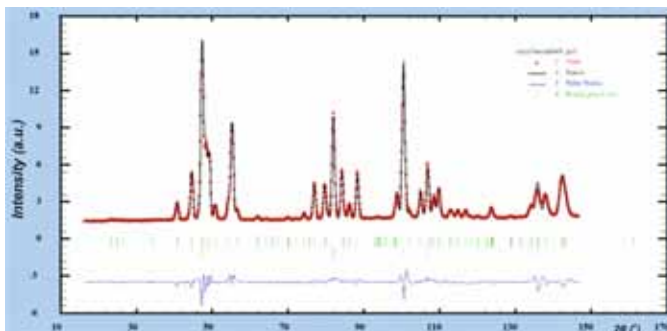


Figure 1. Rietveld refinement of neutron diffraction pattern for CoCrFeNiNb_{0.65} plate at room temperature.

Increasing the cooling rate contributed to the substantial refinement of the microstructure – allowing to obtain ultrafine eutectic structures in the case of the rapidly solidified CoCrFeNiNb_{0.65} alloy. TEM images confirmed the formation of lamellae with an average thickness of less than 100 nm which was observed for the CoCrFeNiNb_{0.65} plate.

Potentiodynamic measurements in 3.5% NaCl solution revealed a beneficial effect of increasing the niobium content on the corrosion resistance of alloys in the as-cast state. A more stable and compact passivation layer is formed with increasing niobium content for as-cast alloys in a 3.5% NaCl solution. The samples solidified with a higher cooling rate exhibited a lower susceptibility to corrosion in the 3.5% NaCl solution. The results of the EIS study indicated that a higher content of niobium contributed to the formation of a more stable and compact passivation layer.

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Oxidation behaviour of nickel/aluminium-based Al-Cr-Fe-Ni high entropy alloys

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Keywords: high entropy alloys, high-temperature oxidation, corrosion resistance

1. Introduction

High entropy alloys (HEAs) have been the subject of scientific interest since their conception in 2004 [1] due to their unique properties. However, in high-temperature oxidizing conditions, some high entropy systems, e.g., $\text{Al}_x\text{CoCrFeNi}$, grow multi-layer scales [2]. Fortunately, such a HEA system with increased nickel content, i.e. $\text{AlCoCrFeNi}_{2.1}$ (mole ratio), has proven capable of selective Al_2O_3 formation [3]. Additionally, a four-element Al-Cr-Fe-Ni high entropy alloy has also demonstrated promising oxidation properties [4]. However, it should be noted that the aforementioned studies were performed for relatively short durations under isothermal conditions. The goal of this work is to investigate nickel and aluminium-rich four-element Al-Cr-Fe-Ni high entropy alloys and determine the element ratios that allow for the growth of a primarily Al_2O_3 scale over a long duration in oxidizing conditions with cyclic temperature changes.

2. Materials and methods

High entropy alloys with nominal compositions $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$, $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$ and $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ (at%) were synthesized via induction melting from high-purity Al, Cr, Fe and Ni elements (> 99.9 at%). The ingots were remelted five times to ensure homogeneity. Samples with ~15mm diameters and ~1.5mm thickness were then cut from the ingots. These samples were subsequently ground with sandpaper of #320 – 1000 gradation, after which they were polished with diamond pastes (9, 3 and 1 μm grain size). Finally, the samples were purified in ethanol in an ultrasonic bath for 10 min. The prepared samples were then subjected to cyclic oxidation for over 600 h: T = room temperature – 1000°C, 1 cycle = 2 h heating and 10 min cooling. Sample weight changes were systematically registered using an analytical scale. Microstructure, chemical and phase compositions of the alloys before and after the oxidation process were identified by means of scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM-EDS), and X-ray diffraction (XRD) studies. SEM-EDS was also used to investigate oxidized sample cross-sections, which were achieved by first mounting the post-oxidation samples in thermosetting resin, and then cutting the obtained mounts in half. The now-exposed cross-sections were subsequently polished with the above-mentioned diamond pastes.

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3. Results and discussion

SEM-EDS and XRD studies on the samples before oxidation are illustrated in Figure 1.

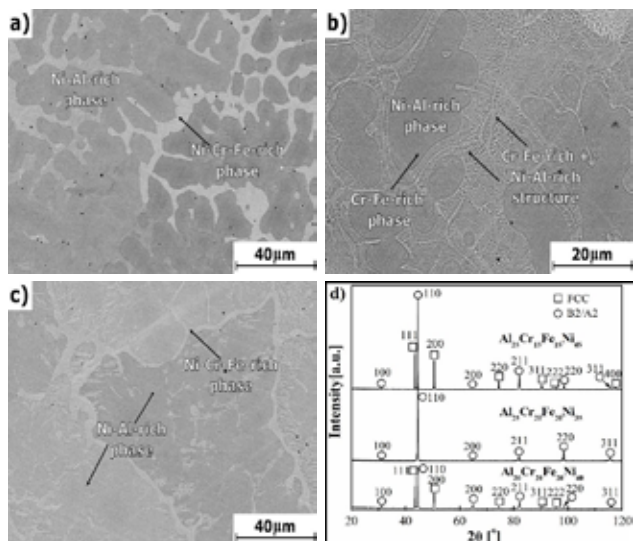


Figure 1. SEM surface images with chemical composition information of a) $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$, b) $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$, c) $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$, and d) XRD patterns obtained from the initial high entropy alloys.

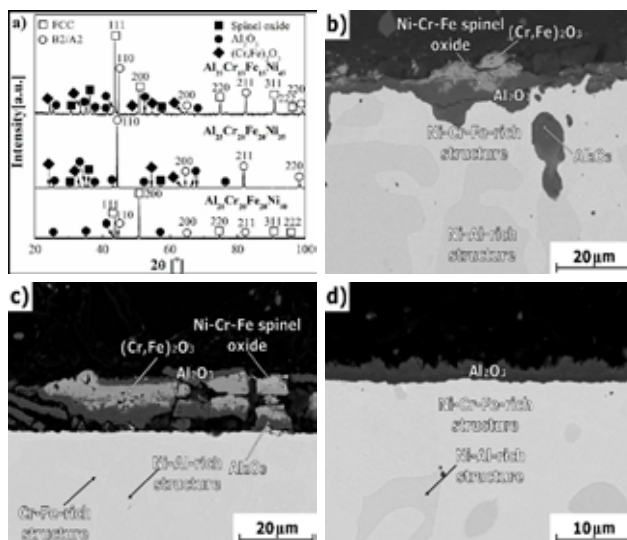


Figure 1. XRD patterns obtained from $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$, $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$, and $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ high entropy alloys, and SEM cross-section images of b) $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$, c) $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$, and d) $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ after oxidation.

From the combined microstructure, chemical and phase composition analysis it follows that the $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$ and $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ alloys are built of Ni-Al-rich ordered BCC (B2) and Ni-Cr-

-Fe-rich FCC structures, whereas $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$ consists of Cr-Fe-rich disordered BCC (A2) and Ni-Al-rich B2 phases that have very similar lattice constants, and thus, overlapping peaks. These phase interpretations are in accordance with those of similar Al-Cr-Fe-Ni HEAs [5,6].

The XRD and SEM-EDS investigation results after the oxidation process are shown in Figure 2.

These studies indicate that the materials were able to maintain their initial phase compositions, however, the phase adjacent to the scale/substrate interface of $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$ and $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ is the Ni-Cr-Fe-rich FCC phase, whereas it is the Cr-Fe-rich A2 structure in the case of the $\text{Al}_{25}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{35}$ alloy. As for the scales formed during oxidation, only $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ proved capable of maintaining a single Al_2O_3 layer over the long oxidation duration. Additional Ni-Cr-Fe spinel oxide and iron-doped Cr_2O_3 phases formed on the remaining materials. In the case of $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$, internal Al oxidation can also be observed. This is all reflected in the cyclic oxidation kinetics obtained from the above-mentioned systematic weight measurements, where $\text{Al}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Ni}_{40}$ demonstrates the lowest overall mass gain and $\text{Al}_{25}\text{Cr}_{15}\text{Fe}_{15}\text{Ni}_{45}$ the highest.

4. Conclusions

From all the performed studies it can be concluded that higher Al content in Ni-rich Al-Cr-Fe-Ni high entropy alloys does not necessarily facilitate more selective alumina formation. It can be additionally stated that excessive Ni concentration (45 at%) also does not benefit the oxidation properties. In fact, the high entropy alloy with the 20%Al/40%Ni ratio was the composition that demonstrated the capability to maintain its Al_2O_3 scale for a prolonged duration and while being subjected to cyclic temperature changes.

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From Nanoscale Analysis to Representative 3D Volumes: Multiscale Characterization Using Xe Plasma FIB-SEM

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Keywords:

Complex materials systems exhibit structural, crystallographic, and chemical features across a broad range of length scales, requiring different characterization approaches depending on the scientific question and material class. While nanoscale investigations are often necessary to reveal local defects, interfaces, or phase transformations, representative characterization of heterogeneous materials requires analysis of substantially larger volumes. Recent advances in Xe plasma FIB-SEM technology enable these complementary workflows to be performed within a single platform, providing a versatile approach to multiscale materials characterization. Based on Xe Plasma FIB-SEM workflows supporting TEM preparation, advanced analytical techniques, and large-volume tomography.

For nanoscale investigations, Xe plasma FIB enables site-specific preparation of high-quality TEM lamellae with reduced risk of ion implantation artifacts. The integration of the 4D-STEM detector directly within the FIB-SEM platform provides initial crystallographic and phase insights immediately after lamella preparation, enabling rapid assessment of specimen quality and identification of regions of interest prior to detailed TEM characterization. Together with efficient subsurface cross-sectioning, these capabilities support targeted investigations of buried interfaces, defects, and microstructural heterogeneities.

For heterogeneous materials, the high milling rate of Xe plasma FIB enables automated serial sectioning and acquisition of representative three-dimensional datasets. Depending on the material system and characterization objective, 3D FIB-SEM tomography can be performed together with integrated collection of complementary analytical data, such as EDS, EBSD, and/or ToF-SIMS, enabling correlative characterization of morphology, elemental distribution, crystallography, and chemical composition in three dimensions.

The presentation will demonstrate the application of these workflows to nanocrystalline, metallic, and battery materials. Examples include multimodal 3D FIB-SEM tomography with integrated EDS and EBSD data acquisition for three-dimensional characterization of precipitates in laser-melted alloys, and multimodal battery characterization using 3D FIB-SEM tomography with integrated 3D EDS and 3D ToF-SIMS mapping to investigate

electrode structure, chemistry, porosity, and degradation. Together, these examples demonstrate how Xe plasma FIB-SEM enables material-specific characterization workflows spanning nanoscale TEM specimen preparation and initial 4D-STEM analysis to representative-volume multimodal tomography within a single instrument platform.

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Biography

Tomáš Šamořil is Product Marketing Manager for Materials Science at Tescan, specializing in electron microscopy and advanced materials analysis. His work focuses on analytical FIB-SEM and integrated ToF-SIMS workflows, with particular experience in battery research and complex material characterization.

Molecular dynamics simulation of the damage layer formed during FIB processing

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Keywords: FIB, molecular dynamics, cascade damage, sputtering, amorphization

1. Introduction

While samples with a thickness of around 200 nm are typically the limit for observation with standard TEMs and STEMs, advancements in aberration correction technology have led to a trend towards lower acceleration voltages. Consequently, there is now a growing demand for techniques that can prepare samples with thicknesses of tens of nanometres or less. FIB processing is undoubtedly the mainstream method for sample preparation today. Thanks to innovations such as automation and gentle processing, the quality of the resulting samples has improved significantly. However, as TEM resolution continues to improve, further advancements in ion milling technology, as exemplified by FIB processing, are becoming indispensable. Nevertheless, there are physical limitations to this.

'Gentle' thin-sectioning of a material ultimately involves removing its atoms one by one from the surface. For instance, mechanical polishing creates brittle micro-fractures on the sample surface and removes atoms in blocks; therefore, it cannot be considered a gentle process. Ideally, the energy required to remove a single atom from the sample surface would lie between the sublimation energy of the substance (several eV) and the energy required to form an atomic vacancy on the surface (approximately 1 eV). In ideal ion milling, thinning is achieved by transferring energy in the above-mentioned range to the target atoms on the sample surface in the form of momentum. On the surface of, or within, a solid, some of the energy imparted to the target atom is dissipated to the surrounding atoms in the form of lattice vibrations. Therefore, sputtering or ejection requires more energy than is needed to create a point defect (or pair). Nevertheless, the maximum energy required to remove a single atom from the sample surface by sputtering is about 10 eV. Conversely, the energy imparted to a silicon atom by a 30 keV gallium ion is approximately 25 keV, around 2,500 times the threshold energy required for ejection. The ion-induced thinning process that we commonly use does not involve ejecting surface atoms one by one; rather, it is a phenomenon whereby atoms near the surface are stripped away, causing significant damage to the interior of the crystal.

Simulations based on the binary collision approximation (BCA), such as SRIM code [1], are used in discussions of sputtering rates and damage layer thickness resulting from FIB processing. It is known that these simulations can provide reasonably accurate evaluations of sputtering rates for various materials. However, SRIM does not account for the crystallinity of materials in its calculations and can only provide indirect estimates of damage layer thickness based on defect distribution and the range of the injected ions. By contrast, although the resolvable size is limited to tens of nanometres, molecular dynamics (MD) simulations enable the tracking of the temporal

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evolution of cascade damage at the atomic scale, providing a means of directly evaluating damage layer thickness. In this study, we performed MD simulations of the process from the formation to the accumulation of initial damage during FIB processing using Si as the target material. We evaluated the relationship between the acceleration energy of Ga ions and the damage layer, as well as the distribution of the penetration depth of Ga ions. We then compared these results with those from SRIM simulations.

2. Simulation method

Molecular dynamics calculations were performed using GPU-parallelised LAMMPS [Ref. 2]. The interatomic potential of silicon was the Tersoff potential, as defined by Kumagai et al. [ref. 3]. Interactions between Si-Ga and Ga-Ga were substituted with the Morse potential. The ZBL potential [ref. 3] was smoothly coupled for short-range interatomic interactions. Approximately 640,000 silicon atoms were placed in a supercell to create a slab model with a (001) surface in the z-direction. Ga ions with acceleration energies ranging from 1 keV to 15 keV were irradiated onto the (001) surface at angles between 3° and 10° in order to induce surface sputtering and internal cascade damage. Once the damaged region had sufficiently relaxed, the Ga ion irradiation process was repeated, resulting in a total of 1000 Ga ions being irradiated. The time series of the number of sputtered atoms was measured and the thickness of the damaged layer and the Ga distribution near the surface were determined.

3. Result

First, we investigated a single gallium ion irradiation event. When a Si (001) surface was irradiated with high-energy Ga ions, some of the Si atoms near the surface detached into the vacuum (a process known as sputtering). Furthermore, Ga or primary knock-on Si atoms that penetrated the crystal's interior induced a chain reaction of displacements, forming so-called cascade damage. This region of cascade damage instantly melted (due to the high temperature) and then cooled instantly, resulting in a change in atomic structure from crystalline to amorphous. As shown in Figure 1, isolated point defects were also generated around the cascade damage region. The cascade damage region formed deeper within the crystal as both the acceleration voltage and the angle of incidence of the Ga ions increased.

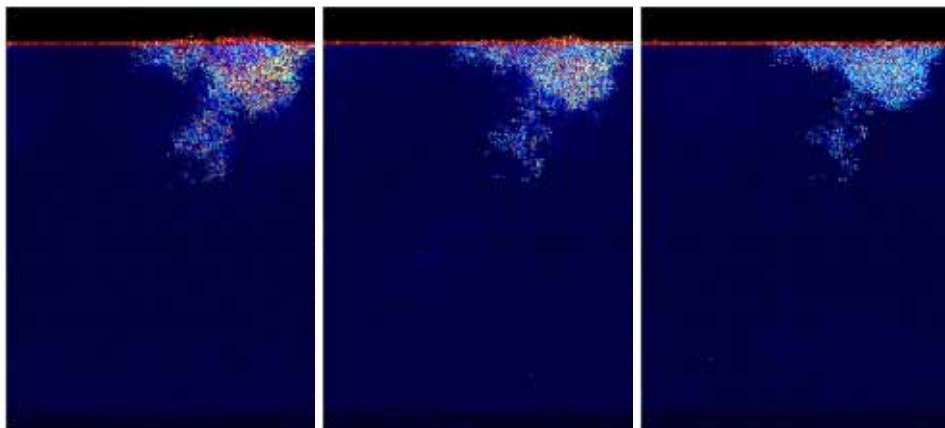


Figure 1. Time series of MD simulations of cascade damage formed near the Si (001) surface following 10 kV Ga ion irradiation.

Next, Ga ions were irradiated sequentially and images taken after 1000 irradiations are shown in Figure 2, where the acceleration voltage was 10 kV and the ion incidence angle was 10° . The area near the surface was completely amorphous and the formation of the damage layer by FIB processing could be reproduced in molecular dynamics simulations. Furthermore, several defect clusters were observed to form at depths greater than the damage layer. Detailed analysis revealed that these clusters were caused by channelling, which allowed Ga ions or primary knock-on Si atoms to penetrate deeper into the material. This deep channelling occurred regardless of the acceleration voltage and accounted for only a few percent of the total irradiated ions.

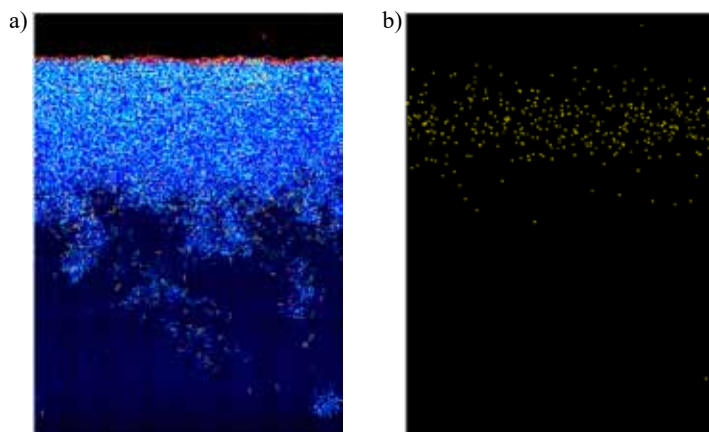


Figure 2. Damage layer near the Si (001) surface following 1,000 Ga ion irradiations (a), and distribution of the implanted Ga ions (b).

Similar calculations were repeated with varying acceleration voltages and Ga ion incidence angles to investigate the relationship between the Si atom sputtering rate, the damage layer thickness and these processing parameters. These results will be presented on the day.

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Composition, size distribution and UV-Vis absorbance of titanium oxide particles produced via a single-step, zero-waste pulsed laser ablation in liquid method

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Keywords: titanium dioxide, particle size measurement, pulsed laser ablation in liquid, microstructure, absorbance

Titanium dioxide in form of micro and nanoparticles is intensively researched due to photocatalytic properties making it attractive for different applications, starting with white pigment in paints of self cleaning effect, sunscreen creams with UV filter, pharmaceutical and food products, to water purification or removal of heavy metals [1,2]. The photocatalytic activity of titania is limited to light primarily in the UV range (below 400 nm), and as a result, it is ineffective under visible light (~400–700 nm) [3]. This is a significant limitation, as UV light is only a small fraction of natural sunlight. For this reason, research is being conducted to modify photocatalytic activity of TiO_2 which can be achieved through doping, phase composition and size control.

This paper presents the results of obtaining titanium oxide suspensions in water using titanium as target and an ytterbium fiber laser operating in pulsed mode with a wavelength of 1064 nm, and power density of $6 \cdot 10^{12} \text{ W/cm}^2$ to $1.3 \cdot 10^{13} \text{ W/cm}^2$. The suspensions and powders obtained from them after sedimentation were subjected to microstructural analysis, including chemical composition analysis by EDS, phase composition analysis by XRD and electron diffraction as well, and microstructural observations using electron scanning microscopy and transmission and high resolution transmission electron microscopy methods. Absorbance/transmittance spectra were also taken in attenuated total reflection mode by Fourier Transform IR Spectroscopy.

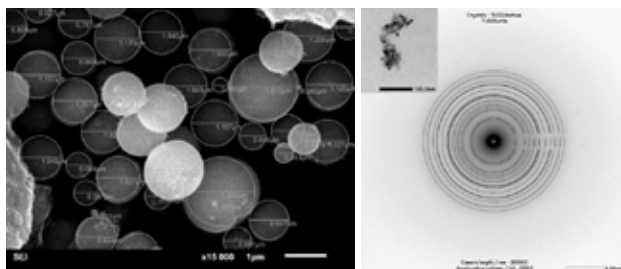


Figure 1. a). An example of secondary electron image of titania particles obtained by PLAL with superimposed circles for diameter measurement, b) Electron diffraction pattern recorded from an area of an agglomerate (image in the top-left corner), with superimposed theoretical diffraction rings corresponding to anatase and rutile.

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It has been demonstrated that pulsed laser ablation of titanium in water yields spherical, high-purity titanium oxides micro- and nanoparticle (Figure 1).

Phase identification based on by electron diffraction patterns recorded from samples taken from suspensions on mesh with a membrane, revealed the presence of TiO_2 in anatase and rutile forms (Figure 2). However, XRD analysis of the sediments also revealed the presence of a third allotropic form of TiO_2 – namely, brookite – as well as titanium monoxide (TiO). As shown in Figure 2, rutile was identified in all samples and it was the dominant phase. In contrast, brookite and anatase, occurred in varying quantities and at specific power densities. Differences in phase composition resulted in changes in absorbance spectra (not shown).

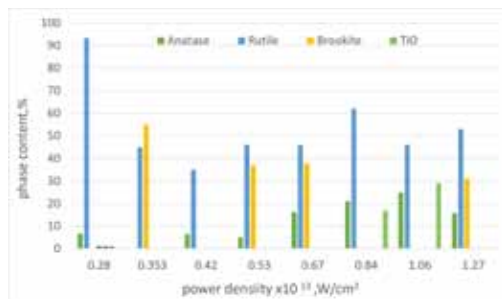


Figure 2. Phase composition (by XRD) of deposits obtained from suspensions produced via the PLAL method at various power densities.

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Phase transformations in metal nanoparticles with native oxide shell

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Keywords: melting, crystallization, EELS, STEM, nanoparticles

1. Introduction

Liquid metal nanoparticles are a unique class of materials that combine the properties of traditional metals with the flexibility of liquids. Unlike solid particles, these metallic droplets can flow, change shape, and heal their own surfaces while maintaining high electrical and thermal conductivity. These characteristics make them highly promising for the next generation of flexible electronics, soft robotics, and advanced energy storage [1].

To date, research has primarily focused on gallium and its alloys because they remain liquid at or near room temperature [2]. However, the range of metallic nanosystems that can operate in liquid states at relatively low temperatures could be greatly expanded by utilizing low-melting point metal and eutectic alloy nanoparticles, stabilizing their supercooled liquid states at the nanoscale. At the same time, most metals oxidize quickly in ambient conditions, causing the particles to naturally develop a thin native oxide shell. This shell creates a core-shell architecture where the liquid metal core is trapped inside a solid skin, significantly influencing the material's structural and phase behavior. While this native oxide shell is often viewed simply as a passive barrier, it plays a critical role in maintaining this liquid phase stability under confinement.

Currently, a significant gap in knowledge exists regarding the formation and thermal stability of the liquid phase in metal nanoparticles enclosed by native oxide shells. Bridging this gap and expanding the variety of available near-room-temperature liquid metal systems is essential for moving these materials from laboratory discoveries to functional technological components. Specifically, utilizing the interplay between the oxide shell interface and nanoscale size effects to keep metals in a supercooled liquid state remains a frontier in materials science. Consequently, this research centers on investigating the phase stability and thermal behavior of the liquid core within these oxide-encapsulated metal nanoparticles.

To address these fundamental questions, a model tin-based ($\text{Sn}@\text{SnOx}$) system was investigated in this study. Tin provides an ideal baseline for isolating these nanoscale phenomena, as bulk Sn features a well-defined melting point of 232°C and readily forms a stable, highly reproducible native oxide shell under ambient conditions.

2. Materials and Methods

The $\text{Sn}@\text{SnOx}$ nanoparticles were formed via a two-step process. First, a 16 nm-thick Sn film was deposited under high vacuum onto an amorphous silicon nitride (Si_3N_4) substrate of a nanochip

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(WildfireHB GT, DENSsolutions), followed by melting. This procedure led to the formation of an island film composed of well-separated nanoparticles. Further experimental details are provided elsewhere [3]. In the second step, the sample was exposed to ambient air, inducing spontaneous oxidation of the Sn nanoparticles and resulting in the formation of Sn@SnO_x core-shell structures. The samples were then transferred to a transmission electron microscope for *in situ* heating experiments.

A probe Cs-corrected Titan³ G2 60–300 electron microscope (Thermo Fisher Scientific, USA) operated at an acceleration voltage of 200 kV was used for the TEM studies. The microscope was equipped with a double-tilt MEMS-based heating holder (Wildfire D6, DENSsolutions), a Gatan Image Filter (model 963), and a Gatan DigiscanTM II scan generator.

The *in situ* experiments were performed in STEM mode over a temperature range of 20–360°C, using heating and cooling rates of 0.2 and 1°C/s. Particle images were acquired using a high-angle annular dark-field (HAADF) detector with a collection angle of 50–200 mrad. Low-loss EELS spectra were recorded with a dispersion of 0.05 eV/channel, a dwell time of 500 ms, and a beam current of ~45 pA. The convergence and collection semi-angles were 14 and 33 mrad.

EELS acquisition during heating-cooling cycles was automated using DigitalMicrograph scripting. At each temperature, the script sequentially acquired and summed three spectra from the center of a selected nanoparticle, subsequently applying drift correction via a cross-correlation algorithm. Prior to EELS acquisition, the electron beam was defocused by 71 nm, resulting in a beam diameter of approximately 2 nm at the sample plane. This approach effectively reduced the electron dose, thereby minimizing radiation damage to the nanoparticles. The EELS data were analyzed using custom Python code based on the open-source HyperSpy library. The energy of the Sn volume plasmon peak was determined as a function of temperature over the entire thermal cycle. The Sn plasmon peak and the zero-loss peak were fitted using the Drude model and a Gaussian function, respectively.

3. Results and Discussion

Figure 1a shows a representative STEM image of the island film composed of Sn@SnO_x nanoparticles supported on a Si₃N₄ substrate. The particle sizes in this region range from approximately 6 to 40 nm, although larger nanoparticles were occasionally observed in other areas of the film. The high-resolution HAADF-STEM image of an individual nanoparticle (Figure 1b) clearly reveals a core-shell morphology. The particle core is crystalline, exhibiting an interplanar spacing characteristic of metallic Sn, whereas the shell is predominantly amorphous, which is typical for oxidized Sn nanoparticles.

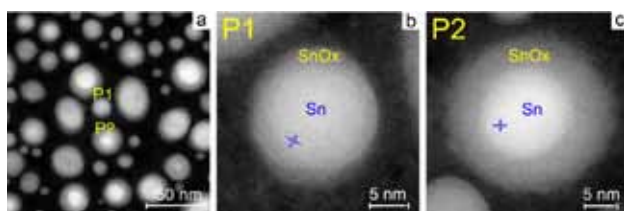


Figure 1. HAADF-STEM images of Sn@SnO_x nanoparticles on a Si₃N₄ substrate. The measured interplanar spacing in the nanoparticle core, 0.27 nm (b) and 0.19 nm (c) correspond to tetragonal β-Sn.

Representative low-loss EELS spectra acquired from the nanoparticle core, the oxide shell, and the Si₃N₄ substrate are shown in Figure 2a. Notably, only the Sn core exhibits a strong and narrow plasmon peak, whereas the oxide shell and the substrate display broader and less intense features. The plasmon energy of metallic Sn is approximately 14 eV, which is significantly lower than the corresponding peaks of SnO_x (~21 eV) and Si₃N₄ (~22 eV). This pronounced spectral

separation demonstrates that low-loss EELS provides a reliable means of distinguishing metallic Sn from its oxides within individual nanoparticles.

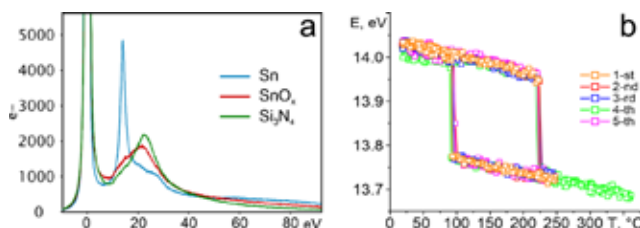


Figure 2. (a) Low-loss electron energy loss spectra acquired from the Sn core, the SiO_x shell, and the Si₃N₄ substrate. (b) Volume plasmon energy of the Sn core as a function of temperature during five consecutive thermal cycles.

Figure 2b presents the temperature dependence of the Sn volume plasmon peak energy for a Sn@SnO_x nanoparticle with a core size of approximately 25 nm and an oxide shell thickness of ~3 nm. The phase transitions of the Sn core are manifested as abrupt shifts in the plasmon peak energy, reflecting the volume change associated with melting and solidification. Melting occurred at approximately 222°C, whereas crystallization was observed at about 94°C. These transition temperatures are consistent with reported values for Sn nanoparticles of comparable size supported on amorphous substrates [4].

Thus, Sn nanoparticles confined within oxide shell exhibits a phase transformation behavior similar to that of bare Sn nanoparticles, namely melting and pronounced supercooling upon crystallization. The melting–crystallization temperature hysteresis was reproducible and independent of both the heating rate and the applied temperature range. Specifically, thermal cycles 1–4 were performed at a rate of 1°C/s, whereas cycle 5 was conducted at 0.2°C/s. The temperature range was 20–250°C for all cycles except cycle 4, in which an extended range of 20–360°C was applied.

These results indicate that Sn nanoparticles with a native oxide shell remain structurally stable during repeated solid–liquid phase transformations, with no evidence of shell cracking or inward oxidation. No measurable influence of superheating or heating rate on the magnitude of supercooling during crystallization was observed. The variation in the measured melting and crystallization temperatures did not exceed 7°C, which was taken as the experimental precision.

The possibilities for studying the effect of size on the melting and crystallization behavior of Sn@SnO_x nanoparticles using the low-dose valence EELS technique will be discussed.

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Thermomechanical aspects of dissimilar AA5083–AA7075 friction stir welding

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Keywords: friction stir welding, dissimilar aluminium alloys, tool design, microstructure, properties

1. Introduction

Friction stir welding (FSW) is a solid-state joining process that enables the fabrication of high-quality joints while avoiding the melting-related defects characteristic of conventional fusion welding. This technology is particularly attractive for joining high-strength aluminum alloys that are difficult or impossible to weld using traditional methods [1,2].

The combination of AA5083-H111 and AA7075-T651 aluminum alloys is particularly attractive for lightweight structural applications, as it combines the excellent corrosion resistance of AA5083 with the very high mechanical strength of AA7075. However, the significant differences in their physical and metallurgical characteristics, particularly the limited fusion weldability of AA7075, make the production of sound dissimilar joints a considerable technological challenge.

The quality and performance of friction stir welded joints are governed by thermomechanical conditions generated during welding. Material flow, heat generation, strain rate and dynamic recrystallization determine the evolution of the weld microstructure and consequently its mechanical and corrosion properties. Understanding the relationships between thermomechanical conditions and microstructural development is therefore essential for optimization of the FSW process.

The present study investigates the thermomechanical aspects of friction stir welding of dissimilar AA5083-H111 and AA7075-T651 aluminum alloys. The influence of local temperature and strain rate distributions on material flow, microstructural evolution, and the resulting mechanical properties of the welded joints was examined in detail.

2. Experimental procedure

Dissimilar butt joints of 6 mm thick AA5083-H111 and AA7075-T651 aluminum alloys were produced by friction stir welding. The alloys were arranged in two different configurations with respect to the advancing and retreating sides of the weld to evaluate the influence of material positioning on weld formation. Three tool pin geometries, namely threaded taper, Whorl and Triflute, were applied using a convex scrolled shoulder.

The welds were characterized using light and scanning electron microscopy combined with EBSD analysis to evaluate grain structure and crystallographic texture. Synchrotron X-ray

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diffraction was employed for phase identification and quantitative analysis of phase distribution within the stir zone. Mechanical performance was evaluated by hardness mapping and tensile testing.

To better understand the governing mechanisms, numerical simulations were performed to determine temperature and strain rate distributions around the rotating tool.

3. Results and discussion

The obtained results demonstrated that the thermomechanical conditions generated during friction stir welding strongly influenced material flow and the quality of dissimilar joints. Both alloy arrangement and processing conditions affected the mixing behavior within the stir zone. More intensive material mixing was observed when AA5083 was located on the advancing side and AA7075 on the retreating side. Increasing the tool rotational speed enhanced plastic flow and promoted material mixing; however, excessive heat input also increased the tendency for tunnel defects, wormholes and surface imperfections.

Numerical modelling revealed considerable differences in temperature and strain rate distributions generated during welding using different tools, as presented in Figure 1. Since grain refinement in friction stir welding depends primarily on these two thermomechanical parameters, the Zener–Hollomon parameter was used to interpret the experimentally observed microstructures. The Triflute pin generated the highest Z value owing to the combination of relatively lower peak temperatures and higher strain rates, providing the most favorable conditions for dynamic recrystallization.

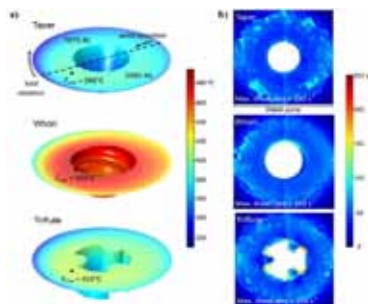


Figure 1. Numerical model for each tool pin geometry:
a) interface temperature distribution with maximum temperature location;
b) mid-thickness strain rate distribution around the pin.

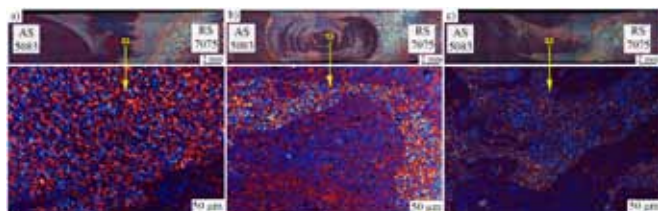


Figure 2. Microstructure of 5083–7075 joints produced using tools with different pin geometries: a) tapered threaded; b) Whorl; c) Triflute. LM: polarized light.

Material mixing during welding was influenced by the tool geometry, leading to the formation of a banded structure, as shown in Figure 2. Microstructural analysis confirmed alternating

AA5083, AA7075, and mixed regions. The morphology and distribution of these bands depended on the thermomechanical conditions established during welding. The Triflute pin produced the finest grain structure among all investigated conditions, while the Whorl geometry promoted more symmetrical material mixing throughout the stir zone.

Synchrotron X-ray diffraction identified the presence of $\text{Mg}_{17}\text{Si}_2$, $\text{Al}_7\text{Cu}_2\text{Fe}$, Al_6Mn , Mg_2Si and $\text{Al}_{12}\text{Mg}_{17}$ phases within the stir zone. Their distribution varied depending on local thermomechanical conditions. Higher temperatures promoted dissolution and coarsening of strengthening precipitates, whereas lower heat input limited phase dissolution. EBSD analysis further demonstrated that the stir zone exhibited a relatively weak crystallographic texture composed of recrystallization and shear texture components, reflecting the complex deformation history during friction stir welding.

The evolution of the weld microstructure was directly reflected in the mechanical performance of the joints. The highest tensile strength and weld efficiency, exceeding 100%, were obtained for defect-free welds produced using the Triflute pin. Fracture occurred in the softer AA5083 base material rather than in the weld, confirming that the joint strength exceeded that of the weaker alloy. Hardness distributions were closely related to the local microstructural heterogeneity developed during welding.

4. Conclusions

The performed investigations demonstrated that thermomechanical conditions play a decisive role in the formation and performance of dissimilar AA5083–AA7075 friction stir welds. Variations in local temperature and strain rate influenced material flow, dynamic recrystallization, grain refinement and phase evolution within the stir zone. These microstructural changes determined both the mechanical properties and corrosion resistance of the joints. The combined experimental and numerical approach provided a comprehensive understanding of the relationships between processing conditions, thermomechanical phenomena and weld performance, offering valuable guidelines for the optimization of friction stir welding of dissimilar aluminum alloys intended for lightweight structural applications.

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Microstructural Evolution of WTaCrV Refractory High-Entropy Alloy and Pure Tungsten Under High-Temperature Helium Irradiation

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Keywords: irradiation defects, nuclear materials, high-entropy alloys, helium irradiation

The development of advanced nuclear energy systems requires structural materials capable of maintaining their mechanical integrity and microstructural stability under extreme operating conditions. In future fusion reactors, plasma-facing materials (PFMs) will be exposed to temperatures exceeding 1000 K, intense neutron irradiation, and continuous bombardment by hydrogen and helium ions originating from the fusion plasma. Under these conditions, the generation and evolution of irradiation-induced defects govern the microstructural evolution of the material and ultimately determine its long-term performance. Therefore, enhancing the resistance of structural materials to irradiation-induced defect accumulation remains one of the major challenges in the development of plasma-facing materials for future fusion reactors.

Refractory high-entropy alloys (RHEAs) have recently emerged as promising candidates for plasma-facing applications owing to their exceptional irradiation tolerance. The severe lattice distortions inherent to these alloys modify the migration and recombination kinetics of irradiation-induced point defects, thereby suppressing defect accumulation and enhancing irradiation resistance. Among the recently developed RHEAs, the equimolar WTaCrV alloy is particularly attractive because it combines excellent mechanical properties with a composition consisting exclusively of low-activation elements, making it well suited for fusion applications. Although previous studies have demonstrated its superior resistance to irradiation-induced hardening and defect accumulation following room-temperature irradiation, its irradiation response under elevated-temperature conditions representative of fusion reactors remains largely unexplored.

In the present work, irradiation-induced defect formation and evolution in the WTaCrV refractory high-entropy alloy were investigated in comparison with pure tungsten. Bulk samples were fabricated by vacuum arc melting and subsequently homogenized to obtain a chemically homogeneous single-phase body-centred cubic (BCC) solid solution. To simulate the operating conditions of future fusion reactors, the materials were irradiated with He ions at temperatures up to 1273 K. The resulting irradiation microstructures were characterized by transmission electron microscopy (TEM).

TEM observations revealed pronounced differences in the irradiation response of the investigated materials. Pure tungsten exhibited numerous large dislocation loops distributed throughout

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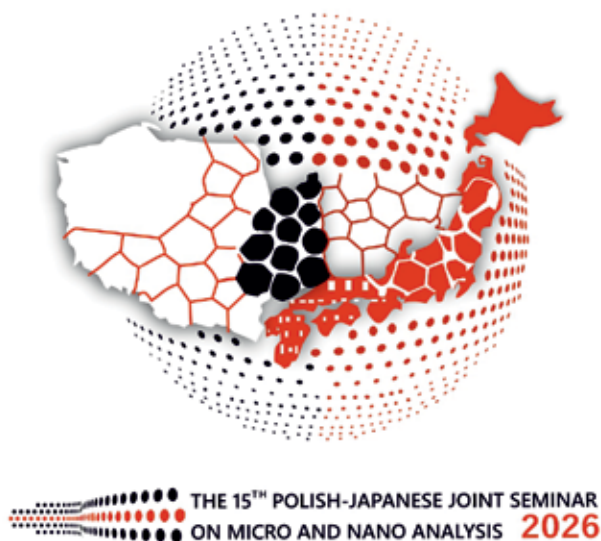


the irradiated region, indicating extensive defect migration and clustering. In contrast, the WTaCrV alloy contained only fine nanometric dislocation loops, predominantly localized near the peak damage region, demonstrating a significant suppression of defect accumulation and irradiation-induced hardening.

To further investigate defect evolution under irradiation, complementary in-situ TEM ion irradiation experiments were performed at the JANNuS-Orsay facility using 2 MeV W ions at 1073 K. The observations revealed fundamentally different defect evolution mechanisms in the two materials. In pure tungsten, the high mobility of irradiation-induced point defects promoted the rapid growth and coalescence of large dislocation loops. Conversely, the reduced defect mobility in the WTaCrV alloy resulted in the formation of a high density of stable nanoscale defects while effectively suppressing their coarsening into extended defect structures. These findings demonstrate the superior irradiation resistance of the WTaCrV refractory high-entropy alloy under elevated-temperature irradiation conditions and further support its potential as a candidate plasma-facing material for future fusion reactors.

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Microscopic Investigation of the Hidden Structures in the Oxidized SnTe Nanowires

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Keywords: nanowires, IV-VI, semiconductors, TEM, Kirkendall effect

Over the past years, topological crystalline insulators (TCI) have been excessively studied due to their unusual electronic properties, resulting from crystalline symmetry and spin-orbit coupling. In the nontrivial topological phase, in addition to the inverted semiconductor band structure, the Dirac-like metallic surface states are observed. This was first predicted theoretically [1] and subsequently confirmed experimentally in IV-VI compounds and their substitutional solid solutions [2].

The electronic properties of TCIs are typically studied by photoelectron spectroscopy, scanning tunnelling spectroscopy, or magnetotransport measurements. The last one is especially important for nanowires. In the case of nanowires, one of the key factors is their high surface-to-volume ratio. Moreover, the edges of the nanowire provide a platform for studying the associated hinge states (higher-order topological insulator states).

Here, we present the results of a study of unconventional subsurface decomposition in SnTe nanowires caused by oxidation. The nanowires were grown using molecular beam epitaxy and physical vapor deposition in two modes: with or without the assistance of gold as a catalyst. All the samples were stored in ambient conditions under normal air exposure for 3 days. For TEM analysis, the nanowires were transferred mechanically onto the Cu grid with a holey carbon film for in-plane observations. As for the perpendicular cross-section, the lamellas were prepared using FIB and lift-off procedure.

Energy-dispersive X-ray spectroscopy (EDS) reveals complex processes and the resulting outward migration of tin. HR-TEM images show specific features in the amorphous oxide skin, such as filaments and precipitates. These have the same crystal structure as the interior of the nanowire, but exhibit varying twists and tilts relative to the original nanowire structure. Observations indicate that there is a limiting twist angle that it can reach.

We identify a two-step oxidation mechanism based on the Cabrera-Mott model [3] and the Kirkendall effect [4]. The first stage involves the formation of tin oxides, which generate an electric field on the surface, and also the solid-solid interface with the nanowire. This process activates and subsequently facilitates the diffusion of cations from the interior toward the surface. The second stage is based on Kirkendall effect, which describes uneven inter-diffusion on the solid-solid interface, particularly significant in the case of vacancy-rich SnTe. The filaments observed in the amorphous

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oxide skin are the fingerprints of the Kirkendall effect and serve as the transport channel for the travelling cations.

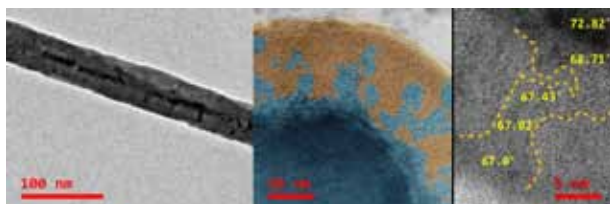


Figure 1. (left) HR-TEM image of the MBE-grown nanowire with filaments and the almost wholly degraded core visible. (middle) The cross-section of the PVD-grown nanowire with crystalline phase marked with blue, and amorphous one in orange. (right) HR-TEM image of the cross-section of the PVD-grown nanowire. The close-up shows a twisted filament marked with orange dotted lines. The values were obtained by measuring the angles between the same spots on the FFT pattern from those areas.

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Entrapment and Imaging of Enzymes in Metal-Organic Frameworks

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Keywords: MOFs, enzymes, imaging, biocomposites, biocatalysis

Enzymes are a brilliant example of how nature has evolved highly efficient biological catalysts. Currently, synthetic analogues of enzymes are still far from achieving the catalytic efficiency and multifunctionality of the enzymes. Although enzymes are highly efficient catalysts, they are fragile, difficult to recycle and sensitive to adverse conditions, such as high temperatures, too high or too low pH values, toxic solvents, or even metal sources that could disrupt enzyme folding. In addition, the process of enzyme purification is resource-intensive, requiring significant energy, labour and time, thereby adding to the overall cost of production.

To tackle enzyme fragility, a protection matrix is needed. Among various solid supports, metal-organic frameworks (MOFs) are excellent candidates because of their high crystallinity, structural rigidity, and extraordinary porosity, which allow the enzyme protection, the catalytic substrate transport across a MOF matrix, and the chemical tunability of the MOF/enzyme interface. It is also possible to synthesize MOFs under enzyme-compatible conditions and efficiently recycle them. Up to date, mainly purified enzymes have been combined with MOFs, while the crude enzyme extract, which contains enzymes along with cellular components, has not been explored as an enzyme source. The utilization of crude enzyme extracts could offer the advantage of retaining the structure and the activity of delicate enzymes that tend to deteriorate during the purification process. Another major limitation in enzyme entrapment research is the lack of methods for precisely imaging the distribution of enzymes and proteins entrapped in different solid materials.

To address both the fragility of enzymes, their deactivation during purification, and incompatibility with MOF precursors during in situ entrapment, we directly use crude enzyme extracts obtained from cell lysis instead of pure enzymes by entrapping them in MOFs structures, as well as the development of enzyme-compatible MOF syntheses that take into account compatibility of metal salts with enzymes. We focus on three enzyme types with varying sensitivities: aldoxime dehydratase, imine reductase and lipase. We evaluate the effects of different metal sources (Al, Fe, Co, Ni, Cu, Zn), their oxidation state and counterions, as well as MOF synthesis parameters on the enzyme stability and activity during their entrapment in the MOF structures. Based on these findings, we optimize protocols for enzyme entrapment in Fe-MIL-88A, Fe-MIL-100, Zn-MOF-74,

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Zn-ZIF-8 and develop a fast-aqueous room temperature synthesis of Al-MIL-53. Furthermore, a new methodology of visualizing enzymes in a MOF matrix using TEM imaging has been developed to improve our understanding of enzyme distribution within a MOF scaffold. Investigation of the biocatalytic performance of the enzyme@MOF biocomposites suggests that enzyme entrapment in MOFs using crude enzyme extracts can effectively maintain enzyme activity and stability in various catalytic reactions.

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Effect of cyclic heat treatment on microstructure and martensitic transformation in melt-spun Cu-Al-Ni shape memory alloy ribbons

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Keywords: Cu-Al-Ni shape memory alloy, melt spun ribbons, martensitic transformation, microstructure, mechanical properties

1. Introduction

Copper-based shape-memory alloys with good mechanical properties can be effectively produced as rapidly solidified ribbons obtained by melt spinning on a rotating wheel [1–4]. This processing route, when combined with appropriately designed cyclic heat treatment, makes it possible to control grain size and tailor the temperatures of martensitic and reverse transformations. Recent studies indicate that cyclic heat treatment leading to abnormal grain growth can provide a relatively simple and cost-effective pathway for producing single crystals of shape-memory alloys [5–8]. Because shape-memory effects, magnetic shape memory, and superelasticity are highly anisotropic, the ability to obtain single crystals without complex processing is particularly attractive for practical applications. However, no research has yet addressed the use of cyclic heat treatment in rapidly solidified materials, which - due to their small dimensions and unique functional properties such as shape memory, superelasticity, and the elastocaloric effect – are promising candidates for miniaturized functional devices. Therefore, the present work aims to develop a cyclic heat-treatment procedure for rapidly solidified copper-based shape-memory alloy ribbons and to determine its influence on phase-transformation behaviour, crystalline structure, microstructure, and mechanical properties.

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2. Results and summary

The subject of the study was a Cu-11.85Al-4.0Ni-3.0Mn (wt. %) melt spun ribbon with a multiphase structure composed of D03 austenite and two martensites 2H and 18R with an average grain size of 3 μm . Figure 1 shows the light microscopy (LM) and scanning electron microscopy (SEM) microstructures, elemental distribution maps, and the chemical composition of the as-cast alloy.

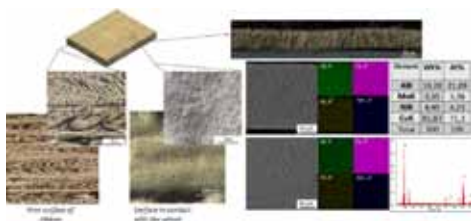


Figure 1. Set of LM and SEM microstructures, elemental distribution maps and EDS chemical composition analysis results of the as-cast ribbon.

The developed cyclic heat treatment, consisting of four alternating annealing cycles at temperatures of 500 and 900°C for 30 min, led to the formation of a martensitic structure composed of 2H and 18R martensites and to a grain growth up to 100 μm . SEM microstructures of as cast and after cyclic heat treatment samples are presented in Figure 2.

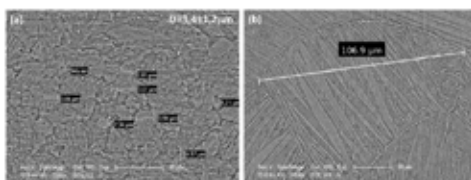


Figure 2. Set of SEM microstructures of the ribbons (a) in the as-cast state and (b) after cyclic heat treatment.

This led to more than a thirty-fold increase in grain size caused a shift in the temperature range of the martensitic and reverse transformations, as determined by DSC analysis (Fig. 3). A shift in characteristic temperatures toward higher values, a broadening of the transformation peak widths, and a slight increase in the latent heat were observed.

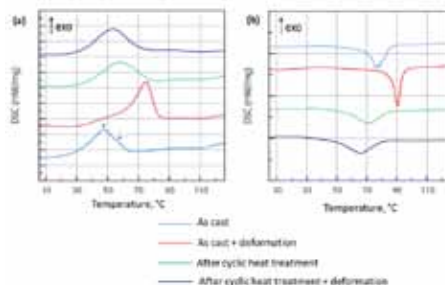


Figure 3. DSC curves after cooling a) and heating b) cycles of ribbons in different conditions.

Detailed microstructural studies performed using TEM confirmed the crystalline structure of the as cast and cyclic heat treated ribbons, and allowed for the description of the morphology of 18R and 2H martensite and ordered D03 austenite. TEM microstructure of martensites in as cast sample is presented in Figure 4.

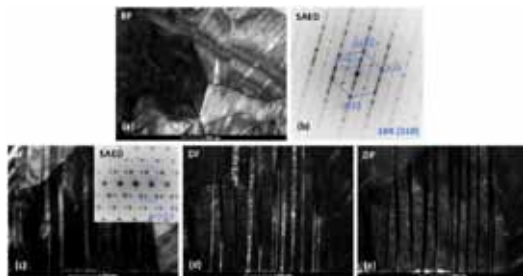


Figure 4. TEM microstructures of two types of martensites present in the as-cast strip and the corresponding electron diffraction patterns. (a) BF pattern of 18R martensite and its corresponding (b) electron diffraction pattern, (c) BF pattern of 2H martensite, and (d) and (e) DF images made from reflections from the twin and the matrix.

Tensile tests were conducted on the ribbons at room temperature (stability range of the multiphase D03+2H+18R structure and the 2H+18R martensitic structure for the ribbons after cyclic heat treatment) and at 90°C (stability range of the austenite in all ribbons) in the as-cast and cyclic heat treated states. This allowed for the determination of the critical stress range, strength range, and strain range until the ribbon rupture. These data will be used for further work to determine the superelastic properties and the elastocaloric effect.

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Microstructure and properties of cold-sprayed Ni20Cr coatings

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Keywords: cold spraying, Ni20Cr coating, microhardness, flexural strength, wear resistance

Thermal spraying processes allow for the production of thick protective coatings, which is important for components operating under conditions of intense abrasive wear [1–4]. However, high temperatures during certain processes can cause excessive oxidation and evaporation of materials. This leads to changes in phase composition, microstructure, and properties of the coatings, thus limiting their application. The cold spray technique for powder materials eliminates unfavourable processes and deposits temperature-sensitive coatings. In this process, powder particles are deposited on the substrate by impact at temperatures below the alloy melting point. This results in severe plastic deformation, which enables the formation of coatings with a defined phase composition [3, 4]. Nickel-chromium alloys are commonly used as corrosion-resistant and oxidation-resistant coatings [6–9]. The literature lacks data on the behaviour of these coatings under severe wear conditions. Therefore, this paper presents a comprehensive characterisation of these materials, which contributes to their application in improving the properties of steel components operating under severe wear conditions. Analysing the microstructural, mechanical and tribological properties of coating-substrate systems can reveal zones of potential material weakening induced by external loads. This study explored the relationships between the microstructure of the obtained Ni20Cr coatings, their mechanical properties, and the wear characteristics of the coating-substrate systems.

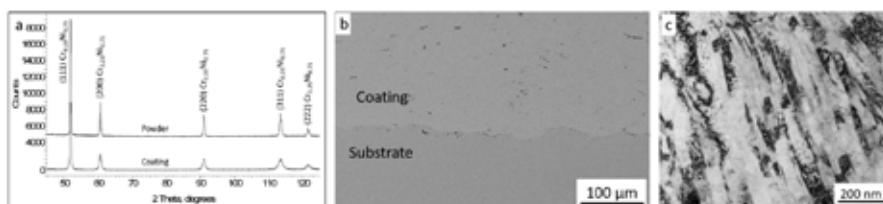


Figure 1. X-ray patterns of the powder and coating (a), SEM cross-section at the coating-substrate interface (b), and TEM microstructure of the coating (c).

Ni20Cr coatings were produced on a steel substrate using cold spraying, a cutting-edge thermal spraying technique. Both the feedstock powder and the resulting coatings were characterised. The coatings exhibited the phase present in the feedstock powders, i.e. Cr0.25Ni0.75 (Fig. 1a).

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The phase composition, micro- and nanoscale microstructures, mechanical properties, adhesion, friction coefficient and wear resistance of the coatings were thoroughly examined. It has been shown that cold spraying creates new possibilities for forming dense nanostructured Ni20Cr coatings with good adhesion to a steel substrate (Fig. 1b and c) and significantly higher microhardness than the steel substrate. The coating had a compact structure with low porosity, below 1% vol.

During three-point bending, a crack appeared on the coating surface and propagated through the coating in the direction of the applied force. As the force increased, the crack propagated through the coating to the substrate and then along the coating-substrate interface. The substrate was not damaged. The coating friction coefficient was slightly lower at higher loads, but decreased significantly with increasing test temperature. Based on the analysis of the coating wear paths during operation in a sliding pair with a rotating Si3N4 ball, the wear index of the Ni20Cr coatings was determined. It increased with both increasing load and increasing test temperature. The coatings exhibited both adhesive and oxidative wear during operation in the tested sliding combinations. The investigations showed that these coatings can be successfully used to protect steel components operating under dry friction conditions, as evidenced by low wear index values.

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The dual role of sluggish diffusion in determining the mechanical integrity and microstructure of FeNiCo/Copper joints brazed with a medium-entropy Ag-Cu-Ni-Pd-Zn alloy

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Keywords: Brazing, Kovar(FeNiCo), Sluggish diffusion, Interfacial microstructure,
Medium-entropy alloy filler metal

The advancement of high-power electronic systems and aerospace modules operating in extreme thermomechanical environments mandates highly reliable FeNiCo(Kovar)/Copper joints. Traditional low-temperature Ag-Cu-Zn braze fillers suffer significant degradation under cyclic thermal gradients, leading to creep, interfacial delamination, and vacuum leakage [2]. To address these challenges, this study investigates the application of a novel medium-entropy alloy (MEA), Ag₂₀Cu₃₅Ni₅Pd₅Zn₃₅ (HEA2), designed to mitigate these issues through the sluggish diffusion effect. Owing to their departure from the single-principal-element paradigm, HEA/MEA systems possess high configurational entropy and heterogeneous diffusion energy barriers, which can effectively retard the excessive formation and growth of brittle intermetallic compounds (IMCs) at the braze/substrate interfaces [1,3]. The five-component HEA2 alloy was selected based on rigorous thermodynamic criteria (configurational entropy of 11.28 J/mol·K and VEC of 11.25) to ensure the formation of a homogeneous face centered cubic (FCC) solid solution. Differential Scanning Calorimetry (DSC) analysis confirmed the absence of solid-state phase transformations up to the solidus temperature of 676.1°C, indicating phenomenal structural compactness. Brazing of FeNiCo/HEA2/Copper components was performed at 675°C for 20 minutes under a 3 N load in an argon atmosphere.

Microstructural investigations (SEM/EDS) of the brazed joints revealed a profound duality in diffusional processes at the interfaces. At the FeNiCo/HEA2 boundary, selective diffusion and reactive barrier formation occurred with remarkable precision (Fig.1). Nickel rapidly diffused from the Kovar substrate into the braze, while iron and cobalt remained intact as a stable structural skeleton. Concurrently, zinc from the liquid braze diffused backwards, inducing the crystallization of an exceptionally thin (1.66 μm), continuous, and coherent intermetallic layer. This layer, combined with the inherent sluggish diffusion within the HEA2 alloy, immediately blocked further mass exchange. This generated a shallow diffusion zone (3.85–4.67 μm) with an extremely flat stress gradient (Fig.1b).

Conversely, the Cu/HEA2 interface exhibited a severely detrimental manifestation of the sluggish diffusion effect. The brazing process initiated extremely aggressive physical dissolution of the copper substrate into the liquid braze. However, drastic variations in diffusion barriers within the distorted five-component MEA lattice completely blocked the efficient transport of the detaching

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Cu atoms deeper into the weld volume (Fig.1c). This massive atomic concentration caused copper to accumulate immediately at the interface, forming an atypically expanded, two-stage diffusion zone reaching 9 μm in width, with local copper concentrations cascading to 65.7 at.%. This severe compositional shift drove the massive crystallization of highly brittle and hard Cu-Zn phases.

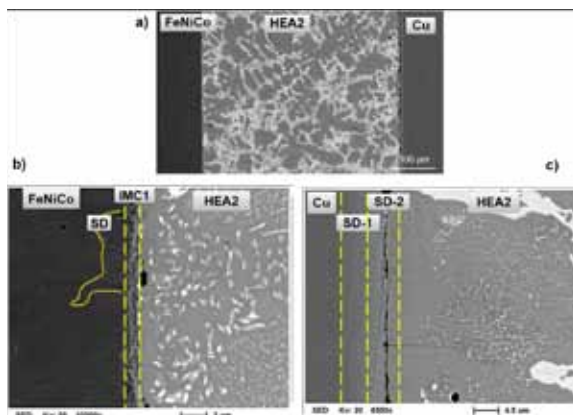


Figure 1. Cross-sectional microstructure of the joint obtained at 675°C for 20 min for: a) the FeNiCo/HEA2/Cu system, b) magnified view of the FeNiCo/HEA2 reaction zone, c) magnified view of the Cu/HEA2 reaction zone. SD – diffusion zone, IMC1 – intermetallic layer.

During cooling, the inability of these trapped brittle phases to accommodate thermal shrinkage generated linear decohesive cracks running parallel to the interface and forced the negative segregation of silver into interdendritic spaces (reaching 49.0 at. %). The mechanisms across both interfaces distinctly highlight the dual nature of sluggish diffusion in HEA-based brazing [3]. On the Kovar side, sluggish diffusion synergizes with the low solubility of Fe/Co to create a stable, kinetic barrier preventing joint degradation [1]. However, on the Copper side, the identical sluggish diffusion mechanism prevents the dissipation of dissolved Cu atoms, triggering severe „dissolution-induced embrittlement”.

The study concludes that while the sluggish diffusion of the HEA2 alloy successfully creates a stable, flexible barrier at the iron-based alloy interface, it has a detrimental effect at the copper interface. Overcoming these diffusion barriers to prevent Cu-induced embrittlement requires engineering changes, such as extending isothermal holding times or increasing brazing temperatures.

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Electron diffraction study of electron beam–induced crystalline phases in amorphous HfO_2

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Keywords: HfO_2 , electron beam irradiation, crystallization, electron diffraction, ferroelectric phase

1. Introduction

Low-power and highly integrated ferroelectric field-effect transistors (FeFETs) have attracted considerable attention as next-generation non-volatile memory devices. FeFETs store information using the polarization states of ferroelectric materials, offering high-speed operation, low power consumption, and non-volatility. Among the various HfO_2 polymorphs, the metastable orthorhombic (o) phase with the space group $\text{Pca}2_1$ exhibits ferroelectricity even in ultrathin films, making it a promising gate dielectric material for FeFET applications [1]. Crystalline HfO_2 is generally obtained through crystallization of amorphous films; however, conventional thermal annealing predominantly produces the thermodynamically stable monoclinic (m) phase, making stabilization of the ferroelectric phase a critical challenge [2]. In this study, crystallization of amorphous HfO_2 was attempted using electron-beam irradiation as a non-thermal approach.

2. Experimental Methods

Amorphous HfO_2 thin films with a thickness of approximately 30 nm were prepared by radio-frequency (RF) sputtering. The films were irradiated with a 200 kV electron beam for 10 min under low-flux ($1.28 \cdot 10^{22} \text{ m}^{-2} \text{ s}^{-1}$) and high-flux ($1.31 \cdot 10^{23} \text{ m}^{-2} \text{ s}^{-1}$) conditions. Structural characterization was carried out using transmission electron microscopy (TEM).

3. Results and Discussion

Bright-field TEM observations revealed that a wider crystallized region formed under the high-flux condition than under the low-flux condition. In contrast, the actual electron-beam irradiation area was smaller under the high-flux condition because the beam diameter had to be reduced. Consequently, an inverse correlation was observed between the irradiation area and the resulting crystallized region. These results suggest that defect formation induced by electron knock-on effects, as well as local temperature increases associated with higher electron flux, influence the crystallization behavior.

Selected-area electron diffraction (SAED) analysis revealed that only halo rings were observed in the unirradiated region, whereas Debye–Scherrer rings appeared in the irradiated region, indicating the formation of nanocrystals induced by electron-beam irradiation. Figure 1 shows the

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experimental electron diffraction pattern obtained from the low-flux specimen. For comparison, the XRD profile and simulated diffraction pattern were overlaid. Weak reflections were observed near the transmitted beam position, where no reflections are expected for the cubic or tetragonal phases. This suggests the formation of both monoclinic and orthorhombic phases. The lower panels of Fig. 1 show simulations for (a) $P12_1/c1$ (m), (b) $Pca2_1$ (o), (c) $Pbca$ (o), and (d) $Pbcm$ (o), all of which reasonably well reproduced the experimental results.

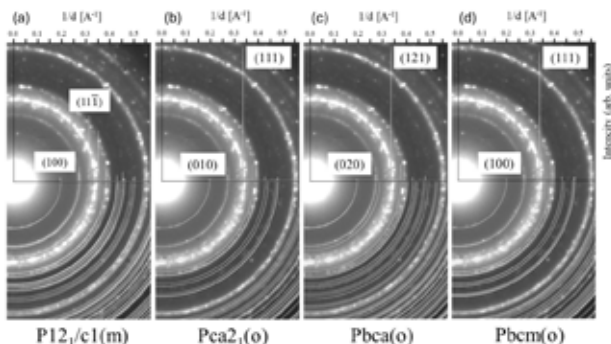


Figure 1. Comparison of selected-area electron diffraction (SAED) patterns acquired under low-flux conditions with simulated diffraction patterns and X-ray diffraction (XRD) profiles for the $P12_1/c1$ (m), $Pca2_1$ (o), $Pbca$ (o), and $Pbcm$ (o) space groups.

The reflection at the $(11\bar{1})_m$ position in the electron diffraction pattern originates exclusively from the monoclinic phase. The corresponding graphs show the simulated XRD intensities. In the $P12_1/c1$ (m) structure (Fig. 1(a)), the strongest intensity appears at the $(11\bar{1})_m$ reflection; however, the corresponding reflection was weak in the experimental diffraction pattern. This suggests that the monoclinic phase was present in small amounts and that other phases coexisted. Indeed, the strongest diffraction rings were consistent with the orthorhombic phases belonging to the $Pca2_1$, $Pbca$, and $Pbcm$ space groups.

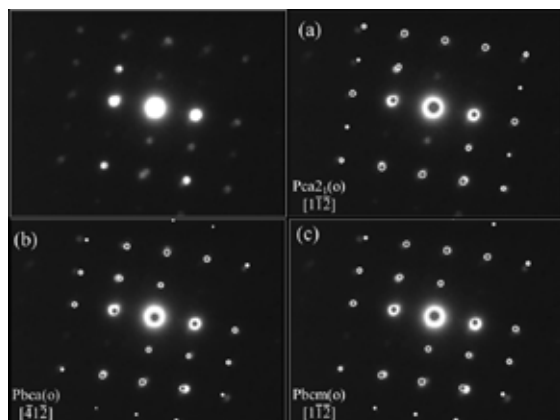


Figure 2. Indexing of nanobeam electron diffraction (NBD) patterns: (a) $Pca2_1$ (o) phase with the $[112]$ incident direction, (b) $Pbca$ (o) phase with the $[112]$ incident direction, and (c) $Pbcm$ (o) phase with the $[112]$ incident direction.

Figure 2 shows the zone-axis nanobeam electron diffraction patterns obtained using an electron beam with a diameter of approximately 5 nm, together with the corresponding simulation results. Among the acquired nanobeam electron diffraction patterns, those exhibiting only fundamental reflections were observed much less frequently than those containing superlattice reflections. This result suggests that the formation fractions of the tetragonal and cubic phases, which exhibit only fundamental reflections, were significantly lower than those of the monoclinic and orthorhombic phases. In particular, the ferroelectric orthorhombic phase with the $Pca2_1$ (o) space group was frequently observed. These results suggest that electron-beam irradiation may serve as an effective approach for promoting the formation of the ferroelectric phase in HfO_2 .

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AI-Driven Design and Experimental Validation of Al–Cu–Fe–Ni–Ti Alloys

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Keywords: computational alloy design, microstructure, DSC measurements,
mechanical properties, magnetic properties

Multicomponent alloys (HEAs/MEAs) constitute a rapidly developing class of advanced materials distinguished by their multi-component compositions and high configurational entropy, which together contribute to exceptional mechanical strength, thermal stability, and corrosion resistance. Their unique properties make them highly attractive for demanding engineering applications in aerospace, energy, transportation, and advanced manufacturing. Despite their considerable potential, the discovery and optimization of HEAs/MEAs remain challenging due to the vast compositional design space and the limited availability of reliable thermodynamic and experimental data. Systems containing only five principal elements can generate millions of possible compositions. Moreover, configurational entropy alone is often insufficient for accurately predicting phase stability or resulting microstructures, which significantly complicates rational alloy design and limits the efficiency of conventional trial-and-error approaches.

To address these limitations, this work presents a modular, uncertainty-aware computational framework designed to accelerate the discovery and evaluation of novel HEAs/MEAs using machine-learning-assisted methodologies. The proposed approach combines physics-informed descriptors, probabilistic modelling, and domain-specific design heuristics to enable efficient exploration of high-dimensional compositional spaces while explicitly accounting for predictive uncertainty. Rather than relying solely on deterministic predictions, the framework evaluates the prediction confidence, thereby supporting more reliable candidate selection and reducing the risk of experimentally infeasible compositions. The workflow is organized into multiple filtering and ranking stages that progressively reduce the compositional space based on thermodynamic, structural, and uncertainty-based criteria. The complete trimming process applied to the example Al–Cu–Fe–Ni–Ti system is shown in Figure 1. Starting from approximately 2.4 million possible compositions generated at a 5% compositional resolution, the framework efficiently reduced the candidate pool to only 91 high-confidence alloys. Each stage introduces increasingly restrictive constraints, including uncertainty-aware thermodynamic thresholds and physically motivated screening criteria. This strategy enables practical and computationally feasible candidate enumeration without opaque black-box optimization procedures.

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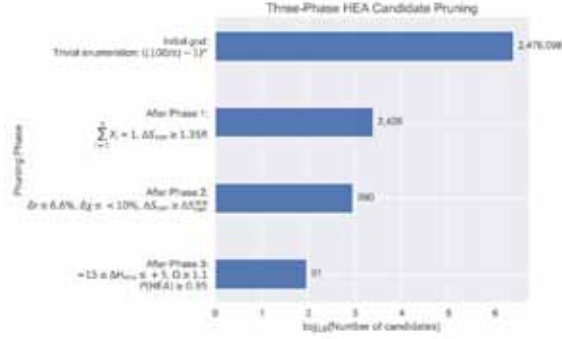


Figure 1. Three-phase pruning of Al-Cu-Fe-Ni-Ti candidates using configurational entropy, geometric/chemical constraints, and uncertainty-aware thermodynamic filtering. Initial pool: 2.4 M compositions. Final set: 91 viable candidates [1].

To further support human-in-the-loop decision-making in the final stages of alloy selection, we introduce a radar-plot-based visualization methodology for rapid, multi-criteria assessment of HEA candidates. As shown in Figure 2, the proposed visualization provides an interpretable summary of six key thermodynamic and physical metrics for each composition. This compact representation enables intuitive comparison of candidate alloys, highlights trade-offs between competing properties, and facilitates identification of compositions that simultaneously satisfy multiple performance objectives. By integrating predictive uncertainty directly into the visualization process, the method also improves transparency and trustworthiness of the decision-making workflow. The radar-based approach, therefore, serves as an effective bridge between automated machine-learning screening and expert-driven materials design. Moreover, the final set of 91 filtered compositions was additionally analysed using crystal structure prediction methods to demonstrate how the generated outputs can support subsequent stages of materials characterization and optimization. The predicted phase distribution is presented in Figure 3. It shows that the vast majority of the selected candidates are likely to form face-centered cubic (FCC) phases.

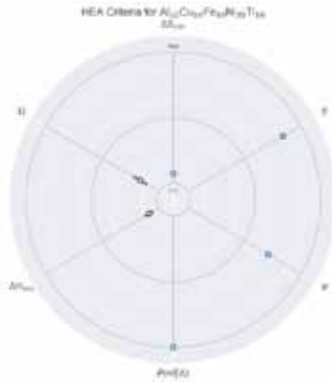


Figure 2. Radar plot of six normalized HEA criteria. Deterministic values (e.g., ΔS_{con} , δ_r , δ_c , $P(\text{HEA})$) are shown as blue-outlined circles. Probabilistic criteria (ΔH_{mix} and Ω) are visualized using purple vertical lines indicating $\pm 2\sigma$ uncertainty bands, with white markers denoting the predicted mean values [1].

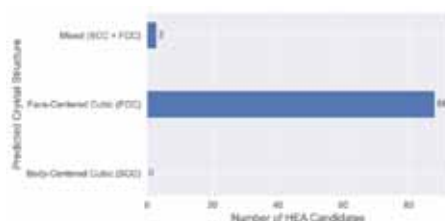


Figure 3. Crystal structure distribution of the 91 retained Al-Cu-Fe-Ni-Ti HEA candidates. FCC dominance reflects the known phase behavior of Ni- and Al-rich HEAs [1].

To experimentally validate the proposed framework, selected alloys from the Al-Cu-Fe-Ni-Ti system were synthesized by the induction melting technique. The resulting materials were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and differential scanning calorimetry (DSC) to investigate their crystal structures, microstructure, and thermal behaviour. Additionally, hardness and magnetic measurements were performed to assess the functional applicability of the synthesized alloys and to compare the experimental observations with computational predictions. The obtained results confirmed the reliability of the proposed computational pipeline and demonstrated good agreement between the predicted and experimentally observed material characteristics. To further investigate the stability and tunability of these properties, the synthesized alloys were annealed and subsequently water-quenched, after which structural characterization and property measurements were repeated to determine the impact of heat treatment on their structural, thermal, and functional properties.

Overall, the presented framework demonstrates that uncertainty-aware machine learning combined with physics-guided design principles can significantly accelerate the discovery and optimization of advanced HEAs/MEAs. The proposed methodology enables fast, scalable, interpretable, and experimentally validated exploration of large compositional spaces while maintaining transparency and practical usability for materials scientists. The agreement between computational predictions and experimental observations confirmed the reliability and practical applicability of the proposed computational pipeline. Consequently, this approach provides a robust foundation for next-generation computational materials design and the rapid development of advanced engineering alloys with tailored functional properties.

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Electron microscopy study of plasma induced sub surface changes in $\text{Al}_2\text{O}_3/\text{Cu}$ composites

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Keywords: copper matrix composites, $\text{Al}_2\text{O}_3/\text{Cu}$, plasma degradation,
microstructure of sub-surface characterization

1. Introduction

The copper matrix composites reinforced with oxide particles are considered promising candidates for plasma torch electrode applications due to their enhanced thermal stability and resistance to degradation under extreme operating conditions. Conventional plasma torch electrodes are commonly manufactured from copper because of its excellent thermal and electrical conductivity [1,2]. However, exposure to plasma, especially in reactive and active gas atmospheres, leads to intensive thermal loading, erosion, and corrosion processes that significantly reduce electrode lifetime [3]. These degradation mechanisms are not limited to the direct arc attachment region but may also occur in areas exposed to the surrounding plasma and reactive plasma species. Therefore, $\text{Al}_2\text{O}_3/\text{Cu}$ composites are being investigated as advanced electrode materials with improved resistance to plasma-related degradation compared to pure copper. Particular attention is focused on the influence of plasma on the microstructure of sub-surface layer evolution of the composite material. Understanding how plasma operating conditions affect the microstructural stability of CuMCs is essential for optimizing their performance and extending the service life of plasma torch electrodes.

This study aims to examine commercial copper matrix composite reinforced with Al_2O_3 particles, fabricated by reaction milling, in as-received condition and after exposure to N_2 , CO_2 and air plasmas for 1h. The microstructure was characterized using scanning electron microscopy (SEM), transmission and scanning transmission electron microscopy (TEM, STEM). The chemical composition was determined using energy dispersive X-ray spectroscopy (EDS) in SEM and STEM.

2. Methods

Copper matrix composite reinforced by Al_2O_3 particles fabricated by reaction milling (C3/11-M) followed by extrusion was delivered by CEP – Compound Extrusion Products GmbH, Germany, in the form of round bar with diameter of 25 mm. The bar was cut into samples of approximately 5 mm thickness. The plasma tests were performed using a high-frequency spark-discharge plasma torch in the test facility BaCon at TU Freiberg, Germany. The samples were treated in a N_2 , CO_2

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or air plasma atmosphere while resting on a rotating table. Each test lasted for 1 hour. The gap between the plasma nozzle and the sample was 5 mm. The microstructure was examined using SEM Nova NanoSEM 450 of FEI equipped with EDS, as well as by TEM and STEM using the JEM-ARM200F NEOARMeX microscope of JEOL equipped with a annular dark field STEM detector (STEM ADF), EDS dual silicon drift detector JEOL Centurio XXXL and Gatan METRO camera for direct electron detection. Samples for TEM analysis were prepared using SEM with Xe plasma focused ion beam (PFIB/SEM) Helios 5 PFIB CXe of Thermo Fisher.

3. Results and discussion

The microstructure of the C3/11-M composite is free from pores and contains Al_2O_3 particles distributed in bands arranged parallel to the extrusion direction (Figure 1a, b). The volume fraction of Al_2O_3 is 3.6%. The STEM-EDS analysis (Figure 1c–f) shows that the diameter of Al_2O_3 particles ranges from several nanometres to a few tenths of a micrometre. Smaller, oval-shaped particles are located mainly within the grains, whereas larger, polyhedral particles are found predominantly at the grain boundaries. Particles occur both individually and in agglomerates. In areas with more dense distribution of Al_2O_3 particles, the grains are smaller than in those with fewer particles, so the presence of oxides inhibits grain growth along the boundaries. Additionally, a small content of Ti was detected in STEM-EDS analysis, which was most likely dissolved in the Cu matrix.

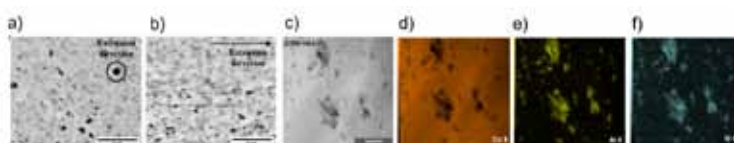


Figure 1. Microstructure of C3/11-M composite in as-received condition: SEM images of a) cross and b) longitudinal sections as well as c) STEM image and d–f) corresponding EDS maps of Cu, Al and O distribution.

The SEM analysis (Figure 2) of cross sections shows that after plasma exposure the surface scale is forming. After air plasma testing, an oxide layer is thick and spalling, more extensively than in other plasma forming gases. The STEM analysis (Figure 2) shows in more details the differences in sub-surface microstructure changes caused by different plasma types.

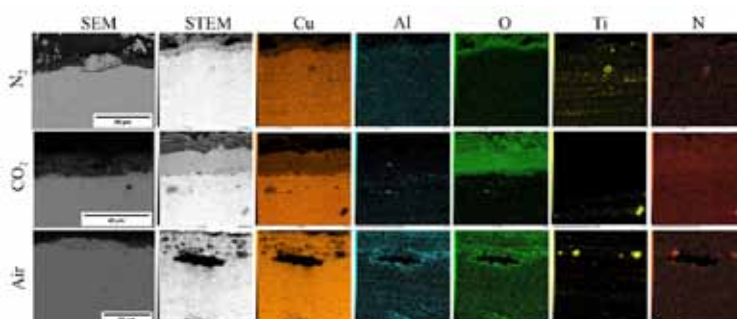


Figure 2. Microstructure of sub-surface of C3/11-M composite after N_2 , CO_2 and air plasma exposure for 1 h.

After N_2 plasma testing below the thin copper oxide scale, fine voids are present. The formation of the Al-rich film resulting from the decomposition of Al_2O_3 oxide, followed by the zone of refined grain containing fine TiN particles is observed.

The reactive CO_2 plasma exposure led to the formation of the copper oxide scale divided into three zones with different morphology. Under scale, the grain growth accompanied by Al_2O_3 decomposition or coarsening occurred. In the layer below, the very fine grains with fine aluminum and titanium oxides and coarse TiN particles are present.

The most reactive air plasma led to thick oxide formation, which was readily spalled from the surface. Under the scale, the Al_2O_3 film is formed in which the large voids elongated in a direction parallel to the surface are present. Below, a zone with refined grain and coarse TiN particles is observed.

4. Conclusion

Exposure to all plasma environments resulted in the formation of an oxide scale. The thickest and most prone-to-spallation oxide layer was observed after air plasma treatment. SEM analysis did not reveal significant differences in the overall microstructure after exposure to different plasma-forming gases. However, STEM-EDS investigations demonstrated that the oxide scale characteristics and subsurface microstructural evolution strongly depend on the type of plasma-forming gas. In all examined samples, the formation of Ti-rich particles and the decomposition/coarsening of Al_2O_3 beneath the oxide scale were observed, accompanied by the formation of voids in the subsurface region. The most distinct microstructural changes occurred after CO_2 plasma exposure, where in layer directly beneath the oxide scale coarsening of grains and oxides was observed. In contrast, exposure to the other plasma gases promoted grain refining in the subsurface region.

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Short-range order in the order-to-disorder phase transformation of ion irradiated $\beta\text{-Sc}_2\text{Hf}_7\text{O}_{17}$

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Keywords: order-to-disorder, short range order, irradiation

1. Introduction

Temperature changes over the past 50 years have made it clear that global warming is a worldwide problem. To address this issue, achieving carbon neutrality by 2050 has been proposed. To achieve this goal, the Japanese government plans to increase the share of nuclear power generation as an alternative to thermal power plants. However, the disposal of high-level radioactive waste remains a major challenge. Geological disposal is a promising method, and vitrified waste forms are expected to be used, but they crystallize at high temperatures and minor actinides can dissolve into groundwater. Thus, the development of new solidified materials is required [1]. Crystalline ceramics have high thermal and chemical stability and can incorporate minor actinides into their lattice, making them attractive candidates as nuclear waste forms. Among them, fluorite-type ceramics such as uranium and zirconium dioxides show excellent radiation resistance and are noteworthy for nuclear applications.

In fluorite-type structures composed of cations with different valences, oxygen vacancies are introduced to satisfy electrical neutrality. When these cations and oxygen vacancies are regularly arranged, new ordered phases, so-called fluorite structural derivatives are formed. These ceramics exhibit diverse properties and undergo structural changes under irradiation, leading to active research on their radiation behavior [2]. The $\text{Sc}_2\text{O}_3\text{-HfO}_2$ compounds contains several fluorite structural derivatives, making it suitable for such studies. Previous studies revealed that the rhombohedral ordered $\beta\text{-Sc}_2\text{Hf}_7\text{O}_{17}$ and $\gamma\text{-Sc}_2\text{Hf}_5\text{O}_{13}$ phases irradiated with 600 keV ions transformed into a disordered cubic fluorite phase with damage accumulation. In addition, it was found that short-range ordered phase is formed during the disordering processes [3]. However, identifying short-range ordered structures is difficult due to the narrow thickness of the damaged region. In this study, we performed MeV ion irradiation to prepare wide damage region, and investigated the short-range ordered structure of the β phase using transmission electron microscopy (TEM).

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2. Experimental

Polycrystalline β - $\text{Sc}_2\text{Hf}_7\text{O}_{17}$ pellets prepared by sintering were irradiated under the conditions, as listed in Table 1. The specimens were characterized by TEM.

Table 1. Irradiation condition.

Specimen	Ion	Energy	Fluence	Temperature
β - $\text{Sc}_2\text{Hf}_7\text{O}_{17}$	Pb^{+28}	93.6 MeV	$5 \cdot 10^{11}$ ions/cm ²	RT
			$1 \cdot 10^{12}$ ions/cm ²	
			$5 \cdot 10^{12}$ ions/cm ²	

3. Results and Discussion

Figure 1 shows electron diffraction patterns obtained from the damaged regions of β - $\text{Sc}_2\text{Hf}_7\text{O}_{17}$ irradiated to (a) $5 \cdot 10^{11}$, (b) $1 \cdot 10^{12}$, and (c) $5 \cdot 10^{12}$ ions/cm². The electron beam was incidental along the [110] zone axis of the fluorite structure. The superlattice reflections due to the β -type structure are present in Figs. 1(a) and 1(b). In contrast, superlattice reflections disappear in the specimen irradiated to $5 \cdot 10^{12}$ ions/cm² (Fig. 1(c)). This suggests that ordered β -phase transforms to the disordered fluorite phase. It should be noted that diffuse scattering appears in Fig. 1(c), indicating that the damaged regions are in a short-range ordered state. Furthermore, diffuse spots or intensity maxima are present in the β -phase irradiated with $5 \cdot 10^{12}$ ions/cm² in Fig. 1(c) as marked by circle.

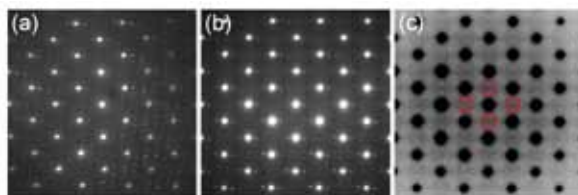


Figure 1. Electron diffraction patterns of β - $\text{Sc}_2\text{Hf}_7\text{O}_{17}$ irradiated to fluence of (a) $5 \cdot 10^{11}$, (b) $1 \cdot 10^{12}$, (c) $5 \cdot 10^{12}$ ions/cm².

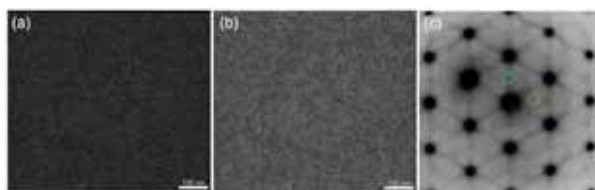


Figure 2. Dark-field TEM images of the sample irradiated to $5 \cdot 10^{12}$ ions/cm² taken from a nearly identical area using a diffuse spot and linear diffuse scattering in the [111] zone-axis.

To examine the origin of the diffuse scattering, dark-field TEM observation was performed. Figures 2(a) and (b) show dark field TEM images taken from a nearly identical area using linear diffuse scattering and a diffuse spot in the electron diffraction pattern of Fig. 2(c). The electron beam was incidental along the [111] zone axis. Weak bright spots on the order of several nanometers were observed. It suggests the presence of microdomains with a higher degree of ordering within the fluorite-type disordered matrix. The microdomains in Fig. 2(a) appear smaller and more distinct

than those in Fig. 2(b). This suggests that the microdomain structures may differ between the two images. Based on the intensity maxima in diffuse scattering, we constructed the structural model of the metastable phase.

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Microstructure evolution and mechanical properties in biomedical Ti-66Zr-3Fe (at.%) alloy subjected to high pressure torsion deformation

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Keywords: biomedical alloy, high pressure torsion, phase transformations, mechanical properties

Ti- and Zr-based alloys, due to their excellent mechanical, physical and biological properties, are increasingly used as biomaterials [1,2]. Although these alloys have been used for medical purposes for over 60 years, there are still a number of problems associated with their use in this field. For example, high-strength α or $\alpha + \beta$ titanium alloys are characterized by too high Young's modulus [3,4], while β titanium alloys have a low Young's modulus, but are characterized by low strength and fatigue properties [5]. Therefore, obtaining biomedical alloys with high strength, good plasticity and low Young's modulus is a challenge for scientists. These alloys may also contain a metastable ω phase, which can appear either by heat treatment of the metastable β phase or as a result of martensitic $\alpha \rightarrow \omega$ or $\beta \rightarrow \omega$ phase transformations under hydrostatic or quasi-hydrostatic high pressure [6,7]. The application of shear stress, like in high pressure torsion (HPT) treatment, provides an additional driving force for these martensitic transformations and makes them much easier. The effect of the ω phase on mechanical properties is ambiguous. On the one hand, it leads to the material brittleness and increases the Young's modulus [8], but on the other hand, ω phase increases the strength and biocompatibility of the alloys [9,10].

The work was based on the assumption that using phase transformations induced by severe plastic deformation (SPD) followed by low-temperature annealing can lead to the optimization of mechanical properties of new biomedical nanocrystalline Zr-Ti-Fe alloys with high strength and low Young's modulus. For this purpose, the influence of SPD performed by high pressure torsion (HPT) on the microstructure, phase transformations, microhardness and Young's modulus of a novel alloy Ti-66Zr-3Fe (at.%), that can be considered as a candidate for biomaterials [11]. Before HPT, the as-cast Ti-66Zr-3Fe alloy was subjected to annealing at different temperatures (600, 800 and 920°C) to obtain a microstructure with a combination of various phase compositions, containing the $\alpha(\text{Ti,Zr})$ and $\beta(\text{Ti,Zr})$ solid solutions, as well as $\beta(\text{Zr})$ and $\text{Fe}(\text{ZrTi})_3$ precipitates (Fig.1). Analysis of the microstructure by XRD, SEM, EBSD and TEM methods showed that the HPT process led to a significant refinement of the microstructure, partial dissolution of

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the second phase precipitates, as well as the appearance of a high-pressure ω -phase, especially in the sample pre-annealed at 600°C. The strong grain refinement of the microstructure and the appearance of the ω_{HPT} -phase resulted in a significant increase in hardness in this sample. In the samples annealed at 800 and 930°C, an additional athermal ω -phase of approximately 200 nm in size was detected. Therefore, HPT treatment did not lead to a significant increase in hardness here, although the hardness values even in the initial annealed samples exceeded the hardness obtained after HPT in the sample pre-annealed at 600°C. The positive effect of HPT deformation on the mechanical properties was observed using the three-point bending method. It was found that the strength and plasticity of the deformed samples are twice those of the initial state, despite the presence of a predominantly brittle ω_{HPT} phase.

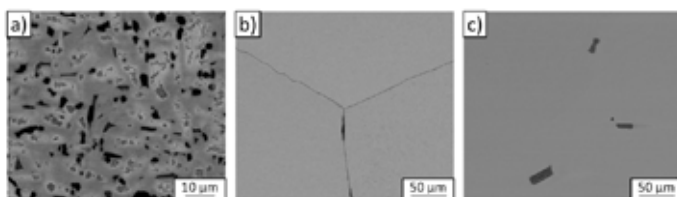


Figure 1. SEM images of Ti-66Zr-3Fe (at.%) alloy after annealing at 600°C (a), 800°C (b) and 930°C (c).

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Influence of heat treatment on microstructure and superelastic behaviour in FeNiCoAlTi melt-spun ribbons

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Keywords: high entropy alloys, superelastic behaviour, heat treatment, mechanical properties

Fe-based shape memory alloys (Fe-SMAs) are promising smart materials exhibiting the shape memory effect and superelasticity associated with thermoelastic martensitic transformations. Compared with conventional Ni–Ti and Ni–Mn-based alloys, Fe–SMAs offer significant advantages, including lower material and processing costs, high strength, excellent formability, and superior resistance to fracture during cyclic deformation. Among them, Fe–Ni–Co–Al alloy are particularly attractive due to their precipitation-strengthened microstructures, which enable stable superelastic behavior. Fe–Ni–Co–Al alloys can achieve exceptionally large recoverable strains of up to 18% for single crystal [1]. Furthermore, this alloy system demonstrate excellent potential for thermomechanical optimization and compositional modification toward high-entropy alloy concepts, resulting in an exceptional combination of superelastic functionality, ductility, and ultra-high strength, with yield strengths reaching 1.6–2.0 GPa. These unique characteristics make Fe-based shape memory alloys highly promising candidates for advanced engineering applications [2–4].

The objective of this study is to optimize the heat treatment conditions (temperature 473–1273 K and time 5–60 min of FeNiCoAlTi melt-spun ribbons. The annealing conditions are intended to stabilize the fcc and bcc phases, respectively, thereby promoting the formation of an appropriate matrix microstructure and coherent precipitates required for enhanced functional performance. To induce thermoelastic martensitic transformation in Fe-based alloys, as well as to engineer an optimal combination of strength and ductility in high-configurational-entropy alloys, it is essential to introduce uniformly distributed, coherent nanoscale precipitates. Such precipitates play a critical role in stabilizing the desired microstructure, promoting reversible martensitic transformation, and enhancing the mechanical performance of these advanced alloy systems.

To achieve the above mentioned goals, polycrystals in a form of melt-spun ribbons with modified composition of Fe–Ni–Co–Al–Ti were fabricated by melt-spinning technique, respectively. The materials were subjected to annealing at wide temperature range 473–1273 K to induce the formation of bcc-phase precipitates – their size and amount. Detailed micro-structural characterization was performed using various methods such as scanning and transmission electron microscopy, high-energy synchrotron X-ray diffraction measurements combined with Rietveld refinement. These analyses allowed the determination of the precipitate size, degree of coherence and volume fraction. In addition, the mechanical properties were evaluated by tensile testing combined with in

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situ scanning electron microscopy (SEM) observations.

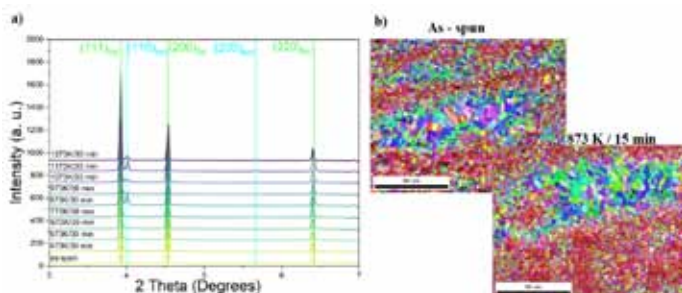


Figure 1. a) synchrotron X-ray analysis and b) example of microstructure as-spun and after heat treatment for FeNiCoAlTi alloys.

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***In situ* TEM observation of Fe-Ga phase transformation in cylindrical ZnO/Fe-Ga core-shell nanostructures**

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Keywords: *in situ* Transmission Electron Microscopy, phase transformation,
ZnO/FeGa core-shell nanowires

1. Introduction

In this study the thermal behaviour and phase changes of hybrid ZnO/Fe-Ga core-shell nanowires were investigated. These structures consist of a single-crystal, semiconducting ZnO core wrapped in a polycrystalline, magnetostrictive Fe-Ga alloy shell. They serve as an effective model for studying how multi-phase metallic materials transform under thermal conditions. Prior research indicates that the unique „feather-like” surface morphology of the shell is heavily driven by the crystal structure of the underlying core, with individual shell grains growing as single crystals tilted at a 45° angle to the main axis [1].

2. Heating experiments

For the *in situ* heating experiments, a Protochips Fusion AX system equipped with a silicon carbide MEMS E-chip was utilized inside a double-tilt TEM holder. The nanowires were separated from their sapphire growth substrate using ultrasonication in ethanol, and the resulting solution was drop-casted onto the holey-carbon-coated membrane of the chip. This allowed the nanowires to bridge the open holes to facilitate clean imaging. Due to the fabrication method, some nanowires exhibited asymmetric shell coverage, consequently, only nanowires with a shell were selected for tracking, an example of which is shown at room temperature in Figure 1a. The initial experiment involved ramping the temperature up to 400°C within seconds, which caused rapid and irreversible changes in the sample’s structure, quantifiable only by STEM-EDX, shown in Fig. 1c. Under these conditions, iron and gallium separated and the core lost its structural integrity.

Afterwards, the approach to heating was changed and the samples were slowly heated from room temperature up to 400°C at a constant rate of approximately 100°C per hour. Observations showed that distinct structural changes begin to appear in both the core and the shell at temperatures above 350°C. Interestingly, the sample support was found to influence the behaviour of the nanowires: those bridging the open vacuum holes showed physical movement during heating, while those resting flat on the carbon film remained relatively stable. Further experiments are

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planned to better separate these thermal effects from potential electron beam damage during prolonged observations and quantify thermally induced structural changes within both the core and the shell of nanowires.

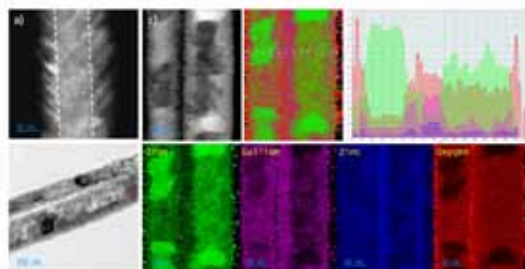


Figure 1. a) STEM image of the core-shell nanowire before heating, shell grains tilted at a 45° angle to the main nanowire axis are visible, core of the nanowire is shown with dashed lines b) Bright-field transmission electron microscopy image of nanowires heated to 400°C, structural changes were ‘frozen’ by immediate temperature drop to 25°C c) acquisition region and EDS elemental maps, as well as line scan of the nanowires showing elemental composition across their width.

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Is thinner better?

The influence of layer thickness on additively manufactured maraging and tool steels

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Keywords: additive manufacturing, laser engineered net shaping (LENS), maraging steel M300, hot-work tool steel AISI H13

Layer thickness is one of the key process parameters in laser-directed energy deposition technologies, significantly affecting manufacturing efficiency, microstructure evolution, and mechanical performance of fabricated components. In this study, the influence of layer thickness on the properties of components produced by the Laser Engineered Net Shaping (LENS) process was investigated for two steel grades: maraging steel M300 and hot-work tool steel AISI H13.

For each material, specimens were manufactured using three different layer thicknesses. The fabricated samples were subsequently divided into two groups: one retained in the as-built condition and the other subjected to heat treatment appropriate for the respective steel grade. Porosity analysis, metallographic characterization, hardness measurements, and tensile tests under static loading conditions were performed to evaluate the influence of processing parameters and post-processing treatment.

The results revealed significant differences in microstructure and mechanical performance associated with layer thickness and heat treatment. The study identified the layer thickness providing the most favorable balance between mechanical properties and manufacturing efficiency, expressed in terms of production time. Furthermore, the effect of post-process heat treatment on the performance of additively manufactured M300 and H13 steels was assessed, providing practical guidelines for process optimization in directed energy deposition manufacturing.

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Numerical and Experimental Investigation of Thermal History and Deformation in Directed Energy Deposition

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Keywords: numerical modelling, additive manufacturing, thermomechanical analysis

Additive manufacturing (AM) involves layer-by-layer material deposition until a complete part is formed. Usually, high power density energy source (like laser) is used. The process is characterized by thermal gradients and a complex thermal history, which significantly influence the resulting microstructure and mechanical properties. Optimized component properties can be achieved by controlling the thermal history through careful selection of process parameters and conditions. Inappropriate process parameters can result in high residual stresses within the component. These stresses may induce deformation, and when a critical threshold is exceeded, they can lead to fracture. To support faster and more efficient process optimization, a numerical model of the additive manufacturing process was developed. The model was validated through thermo-mechanical analysis by comparing experimentally measured temperatures and substrate deformation with numerical simulations. Temperature was monitored using both a pyrometer integrated with the laser head, which measured the melt pool temperature, and a thermocouple welded within the additively manufactured component. Substrate deformation was measured using the Digital Image Correlation (DIC) technique. The results demonstrated good agreement between simulations and experimental observations. The study also highlights the challenges associated with the validation of temperature fields, displacement measurements, and residual stress evaluation.

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Tracking Every Layer: In-Situ Monitoring of Thermal History During Additive Manufacturing of Thin-Walled Structures

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Keywords: additive manufacturing, direct energy deposition, infrared camera, thermal history

Additive manufacturing (AM) is an advanced manufacturing method in which a component is produced by repeated deposition of material layers [1]. This process is characterized by complex and dynamically changing thermal conditions that significantly impact the microstructure and, consequently, the properties of the manufactured components. For this reason, monitoring the thermal history during the additive manufacturing is crucial for better understanding the relationships among process parameters, microstructure, and material properties [2–4].

In this work, to study the thermal history during additive manufacturing using the DED technique, a test setup was built consisting of three infrared (IR) cameras with different temperature ranges (i.e., 0–250°C, 200–1500°C, and 900–2450°C) and two pyrometers. Thin-walled specimens were manufactured from AISI 304 stainless steel and 18Ni-300 maraging steel using various process parameter combinations. The acquired thermographic data enabled the reconstruction of thermal histories at selected locations within the deposited walls, including temperature evolution during successive layer deposition and repeated reheating cycles. Furthermore, microhardness measurements were performed to evaluate the influence of thermal history on the resulting material properties.

The obtained results demonstrate that the developed experimental setup, equipped with three infrared cameras, enables detailed analysis of the thermal history of components additively manufactured by Directed Energy Deposition (DED), potentially allowing the identification of relationships between the thermal cycles experienced during the process and the resulting material properties.

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Microstructural Investigation of Martensite Tetragonality in Steel via EBSD

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Keywords: steel, martensite, martensite tetragonality, EBSD, pattern matching indexing

Martensite in steels forms through a diffusionless, athermal transformation of austenite, involving coordinated atomic displacements within the crystal lattice [1]. Owing to the extremely fine morphology of martensitic regions, frequently below the micrometer scale, and their significant internal lattice distortion, advanced characterization methods are required to resolve local crystallographic heterogeneities [1]. From a crystallographic perspective, martensite can be described as a tetragonally distorted form of ferrite, with the degree of distortion characterized by the c/a lattice parameter ratio and strongly influenced by carbon concentration. This distortion reflects the transition from a body-centered cubic (BCC) lattice to a body-centered tetragonal (BCT) structure [2]. In general, martensitic tetragonality is associated with c/a values exceeding unity and increases with the carbon content dissolved in martensite. The tetragonal distortion arises from carbon atoms occupying interstitial sites within the iron lattice, leading to anisotropic strain and altering the lattice geometry. Previous investigations have reported an approximately linear correlation between carbon concentration and the c/a ratio, indicating that higher carbon levels promote stronger tetragonal distortion [2]. Nevertheless, several aspects of this phenomenon remain insufficiently understood, including the threshold carbon concentration necessary for tetragonality development, the origin of unusually high or low c/a values, and the spatial distribution of tetragonality within complex microstructures.

Reliable assessment of local tetragonality requires techniques capable of high spatial resolution analysis. According to the literature, X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) are among the most commonly applied methods for this purpose. In XRD analysis, the evaluation of tetragonality is hindered by the overlap of martensitic reflections with α -Fe diffraction peaks, particularly for low c/a ratios. Typically, tetragonality is identified by splitting the $\{200\}$ reflection into $(200)/(020)$ and (002) peaks [3], enabling determination of lattice parameters by Rietveld refinement. Although XRD provides valuable average structural information, its spatial resolution is insufficient for detailed local characterization. Consequently, EBSD has emerged as an important complementary technique for investigating martensitic tetragonality. Due to its substantially higher spatial resolution, reaching approximately 100 nm, EBSD enables the analysis of local crystallographic variations and spatial heterogeneity within martensitic microstructures [4].

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The present work investigates martensite tetragonality in steel using EBSD-based crystallographic analysis. Particular attention is devoted to the determination of local lattice parameters, phase distributions, and variations in the c/a ratio associated with changes in carbon supersaturation. The study demonstrates the capability of pattern-matching EBSD to quantify martensitic tetragonality and reveals significant crystallographic heterogeneity within the investigated microstructures. These findings contribute to a deeper understanding of the relationship between carbon distribution and lattice distortion in martensitic steels, providing new insights into the mechanisms governing martensite tetragonality.

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Structural characterization of magnetic halides confined within carbon nanotubes with the use of Transmission Electron Microscopy

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Keywords: encapsulated nanomaterials, magnetic halides, crystal structure, low-dimensional materials

1. Introduction

1.1. Reaching one dimension with the use of carbon nanotubes as hosts in the encapsulation process

Owing to their cylindrical shape, carbon nanotubes allow encapsulation of various materials inside their hollow cavities. For this reason, they can serve as “nanotemplates” for crystal growth, enabling to obtain extremely narrow, nearly one-dimensional nanowires. Due to extremely small diameters (often below 2 nm) a limited amount of filling atoms can fit within the cross-section of the nanotube, prompting interesting structural diversity of the encapsulated nanomaterials [1]. These often display structures and stoichiometries significantly different from their bulk counterparts [2]. In the case of magnetic materials, such extreme spatial confinement also strongly influences their magnetic behaviour. For instance, confined ferromagnetic nanowires have been shown to exhibit large coercivities arising from pronounced shape anisotropy [3]. Beyond the enhancement of conventional magnetic properties, theoretical studies suggest that carbon nanotubes may provide a unique platform for stabilizing unconventional magnetic states. This effect arises from the cooperation between spatial confinement and host-guest charge transfer. A notable example is encapsulated CrSBr, for which DFT calculations indicate stabilization of ferromagnetism within the 1D limit, persisting up to 50 K [4].

Low-dimensional transition metal halides, particularly those with layered structures, constitute an intriguing class of materials. They have attracted increasing attention due to their diverse magnetic properties, which are highly sensitive to structural variations. For example, bulk NiCl₂ consists of weakly bonded van der Waals layers and displays antiferromagnetic interlayer coupling alongside ferromagnetic intralayer ordering [5]. Moreover, many binary halides of reduced dimension are predicted to have elevated intermediate Curie temperature to the values accessible in the liquid

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nitrogen [6]. Consequently, these materials constitute interesting candidates for encapsulation and exploration of their properties in the 1D limit.

1.2. Transmission Electron Microscopy as a comprehensive tool for structural characterization of the materials encapsulated within carbon nanotubes

The versatility of TEM and its wide range of analytical techniques make it ideally suited for the comprehensive characterization of nanomaterials within carbon nanotubes. It is possible to easily evaluate parameters such as sample purity, filling efficiency and structural homogeneity but also perform detailed structural investigation of the material. Imaging modes exploiting Z-contrast, such as HAADF-STEM, allow for a fast and straightforward assessment of sample quality and morphology, given a substantial difference between the masses of carbon atoms of the nanotubes and metal atoms of the confined halide. In addition, analytical techniques such as energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS) enable verification of the elemental composition and, consequently, the stoichiometry of the investigated nanomaterials. Beyond elemental analysis, EELS provides access to a range of additional information, including chemical bonding or oxidation states. High-resolution imaging in both TEM and STEM modes, complemented by electron diffraction (ED), provides a comprehensive toolkit for structural analysis of the encapsulated nanomaterials. In addition, ED is generally less destructive than atomic-resolution imaging, as diffraction patterns can be acquired using a more distributed electron beam and lower electron dose. This makes ED particularly valuable in the studies of beam-sensitive materials (such as carbon nanotubes) and allows structural information to be collected from larger sample volumes, yielding statistically more representative results. Furthermore, the accelerating voltage can be adjusted to balance imaging performance and sample preservation.

2. Structural analysis of encapsulated transition metal halides based on TEM

Selected case studies of transition metal halides encapsulated within carbon nanotubes, with particular emphasis on NiCl_2 confined within single-walled carbon nanotubes (SWCNTs), will be presented to demonstrate the complementary capabilities of TEM imaging, diffraction and spectroscopic techniques. The structural investigation of $\text{NiCl}_2@\text{SWCNTs}$ based on different TEM results is presented in the Figure 1 [7].

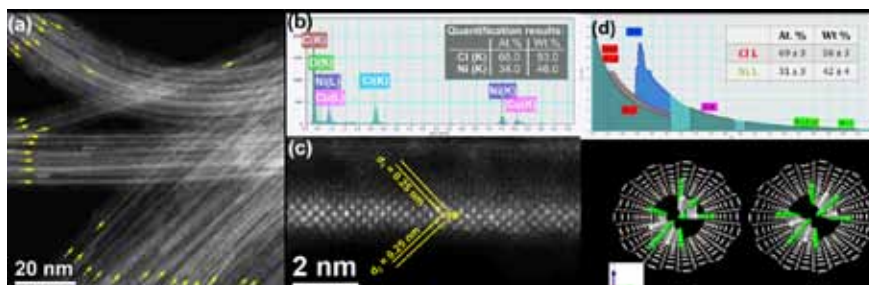


Figure 1. Structural analysis of the NiCl_2 encapsulated within SWCNTs: a) medium-magnification STEM-HAADF image showing the loading efficiency of the SWCNTs; b) EDS spectrum from the sample; c) high-resolution STEM image of the encapsulated NiCl_2 structure with structural parameters measured; d) EELS spectrum from the sample; e) structural models of the encapsulated NiCl_2 – initial (left) and DFT-optimized (right) – excerpt from reference [7].

The analysis of the sample based on TEM results enabled the determination of a high loading efficiency of SWCNTs (Figure 1a), as well as confirmation of the material's purity and stoichiometry through EDS and EELS experiments (Figures 1c and 1e, respectively). High-resolution STEM imaging further allowed for identification of the crystal structure of the chloride phase as CdCl_2 -type (space group R-3mH). On this basis, a structural model was constructed and subsequently optimized using DFT calculations.

Taken together, these results provide a comprehensive structural description of the system, forming a solid foundation for further investigations of its magnetic properties. This study highlights TEM as a powerful and versatile tool for the characterization of such hybrid nanostructures, offering comprehensive and detailed insight into their structure, composition and quality.

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Influence of Chemical Composition and Synthesis Parameters on the Structure and Magnetic Properties of Ferrite Nanoparticles

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Keywords: magnetic nanoparticle, magnetic properties, crystal structure, ferrites, SMC

1. Introduction

Soft Magnetic Composites (SMCs) are advanced materials composed of electrically insulated ferromagnetic particles bonded with a dielectric binder, enabling reduced eddy current losses and efficient operation at elevated frequencies. Among the various magnetic materials considered for SMC applications, ferrites are particularly attractive due to their high electrical resistivity, good chemical stability, low magnetic losses, and tunable magnetic properties [1–3]. The performance of ferrite nanoparticles strongly depends on their chemical composition, crystal structure, particle size, morphology, and magnetic anisotropy, all of which can be tailored through synthesis conditions and compositional engineering. Recent advances in nanoparticle synthesis have demonstrated that high-temperature thermal decomposition enables precise control over ferrite nanostructures, resulting in highly crystalline particles with well-defined morphologies and narrow size distributions. Furthermore, the introduction of secondary ferrite phases into core–shell architectures provides an effective route to modify magnetic interactions and relaxation processes [4,5]. Such structural modifications can significantly influence saturation magnetisation, coercivity, blocking temperature, and thermal stability of magnetic moments, which are critical parameters for SMC applications.

The present study investigates the influence of synthesis modifications and ferrite composition on the structural and magnetic properties of ferrite nanoparticles synthesised by high-temperature thermal decomposition. Particular attention is paid to the effect of shell composition in Fe_3O_4 -based core–shell nanostructures, where magnetite nanoparticles were modified with ferrite shells. The objective was to establish structure–property relationships that can guide the design of ferrite nanomaterials for advanced soft magnetic composite applications.

2. Results & discussion

X-ray diffraction analysis confirmed the successful synthesis of phase-pure ferrite nanostructures. The diffraction patterns revealed the characteristic spinel structure for all investigated materials, indicating that the thermal decomposition method enabled effective crystallisation of both the magnetite core and ferrite shell phases. Structural characterisation further demonstrated that chemical modification through shell formation did not introduce secondary impurity phases, confirming the effectiveness of the synthesis route. Electron microscopy observations revealed distinct

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morphological differences associated with ferrite composition. While the magnetite nanoparticles maintained a relatively uniform morphology, the introduction of ferrite shells resulted in visible changes in particle dimensions and contrast, confirming the formation of core-shell architectures. The ability to control particle morphology through compositional modification is particularly important for SMC applications because particle size and shape directly affect magnetic interactions and magnetisation reversal mechanisms.

Magnetic measurements revealed that both the ferrite composition and the synthesis conditions strongly influence the magnetic behaviour of ferrite nanoparticles. Variations in chemical composition led to significant changes in magnetisation, coercivity, and magnetic anisotropy, demonstrating that compositional engineering is an effective strategy for tailoring magnetic performance. Depending on the selected ferrite system, the nanoparticles exhibited either soft magnetic characteristics, desirable for soft magnetic composite applications, or enhanced magnetic anisotropy associated with increased magnetic hardness. Temperature-dependent hysteresis measurements confirmed that modifications of ferrite composition affect magnetisation reversal processes and thermal stability of magnetic moments. Nanoparticles with lower magnetic anisotropy maintained soft magnetic behaviour over a broad temperature range. In contrast, compositions characterised by higher magnetocrystalline anisotropy exhibited increased coercivity and stronger temperature dependence of magnetic properties. Additional information regarding magnetic relaxation processes was obtained from zero-field-cooled and field-cooled measurements. The observed changes in blocking temperature indicate that both chemical composition and synthesis-induced structural modifications influence the thermal stability of magnetic moments. These results suggest that controlled synthesis enables tuning of superparamagnetic relaxation processes by adjusting particle size, crystallinity, morphology, and interparticle interactions. Furthermore, the degree of FC-ZFC irreversibility varied considerably among the investigated ferrite systems, reflecting differences in magnetic anisotropy and magnetic coupling effects. Materials with stronger anisotropies exhibited suppressed thermal relaxation and enhanced magnetic stability, while softer magnetic ferrites showed more pronounced superparamagnetic behaviour.

3. Conclusions

Ferrite nanoparticles synthesised under different conditions exhibited significant variations in their structural and magnetic properties, confirming the strong influence of both chemical composition and synthesis parameters. Structural modifications affected particle morphology, crystallinity, magnetic anisotropy, and magnetisation reversal mechanisms, resulting in distinct magnetic behaviours. Depending on the ferrite composition, the nanoparticles exhibited either soft magnetic characteristics with low coercivity or enhanced magnetic stability associated with higher anisotropy. Temperature-dependent magnetic measurements demonstrated that synthesis-induced structural changes influence magnetic relaxation processes and the thermal stability of magnetic moments, offering additional opportunities to tailor magnetic performance. Overall, the results show that combining controlled synthesis conditions with compositional engineering is an effective strategy for designing ferrite nanoparticles with tunable magnetic properties. Such materials are promising candidates for advanced soft magnetic composite applications that require optimised magnetic behaviour and low magnetic losses.

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Delayed decomposition of a supersaturated solid solution of an Al-Mg-Zn alloy modified with Ga and Cu additions during artificial aging

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Keywords: aluminium alloys, artificial aging, Ga-addition, TEM

In our previous studies, we found that the addition of Ga had a beneficial effect on precipitation phenomena in an artificially aged Al-2.2Mg-2Zn alloy (at.%) [1–3]. As shown, Ga can retard the decomposition of a supersaturated solution at lower aging temperatures and accelerate hardening at higher temperatures [1,2]. In the present study, the effects of combined Ga and Cu additions on the hardness and microstructure of an artificially aged Al-Mg-Zn alloy at 393 K (120°C) and 473 K (200°C) were investigated. The obtained results were compared with those for the commercial 7075 aluminium alloy.

The Al-2.2Mg-2Zn-1Ga alloy was modified by the addition of 0.5 at.% Cu and is hereafter referred to as GaCu. After casting, the alloy was homogenized at 773 K (500°C) for 24 h and solution-treated by water quenching. Subsequently, the alloy was aged at 393 K (120°C) and 473 K (200°C). The resulting aging curves are shown in Fig. 1a,b. For comparison, the aging curves obtained for the Al-2.2Mg-2Zn alloy (designated as Al-Mg-Zn), the Al-2.2Mg-2Zn-1Ga alloy (designated as Ga), and the commercial 7075 alloy are also included.

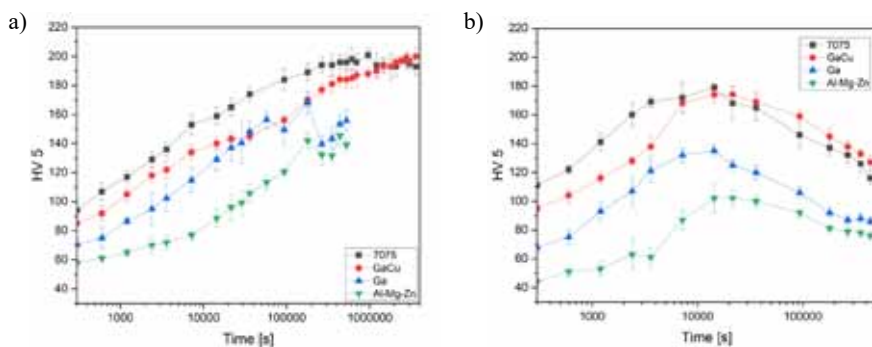


Figure 1. Aging curves obtained for the investigated alloys at two temperatures: (a) 393 K (120°C) and (b) 473 K (200°C).

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Results showed that, compared with the other investigated alloys, including the commercial 7075 alloy, modification of the base alloy with Ga and Cu resulted in delayed decomposition of the supersaturated solid solution at both lower and higher aging temperatures. At 393 K (120°C), the GaCu alloy did not reach a hardness plateau even after 1000 h of aging. At 473 K (200°C), the maximum hardness, comparable to that of the commercial 7075 alloy, was achieved after 6 h of aging, whereas the hardness peak in the other investigated alloys was attained after only 4 h.

A comparison of the microstructures of the GaCu and 7075 alloys aged at 473 K (200°C) for the same period (122 h) revealed differences in the types of precipitates formed and showed that the precipitates in the GaCu alloy were significantly finer (Fig. 2a,b). Detailed studies of the observed precipitates were performed using selected area electron diffraction patterns (SAEDP) and high-resolution images combined with fast Fourier transform (FFT) analysis. We also found that the mechanism of solid-solution decomposition in the alloy containing both Ga and Cu differs from that observed in the Ga-modified alloy. These results will be discussed in relation to differential scanning calorimetry measurements and the results obtained for samples naturally aged at room temperature.

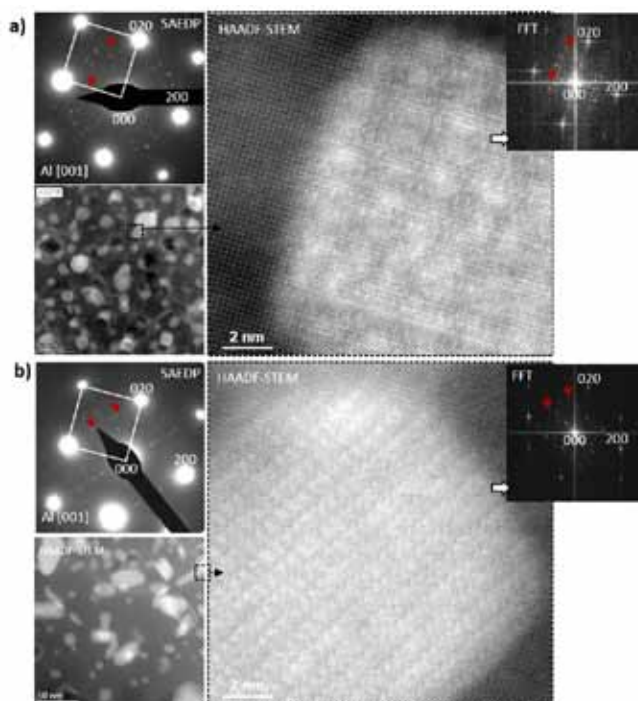


Figure 2. TEM micrographs, including SAEDPs and HAADF-STEM images, of (a) the GaCu alloy and (b) the 7075 alloy after artificial aging at 473 K (200°C) for 122 h, showing differences in the microstructure and diffraction patterns.

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SEM Analysis of Superheater Tubes after 10 Years of Service in a Biomass-Fired Boiler

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Keywords: hot corrosion, stress corrosion cracking, SA213 TP310HCbN austenitic steel, inconel 625

1. Introduction

The global transition toward renewable energy sources has led to a significant increase in the combustion of biomass for heat and power generation. While biomass offers substantial environmental benefits, its combustion presents unique and severe challenges for boiler components, particularly superheater tubes [1]. Unlike conventional coal-fired or gas-fired units, biomass-fired boilers operate under exceptionally aggressive conditions that accelerate material degradation and significantly reduce component service life [2].

The aggressive nature of biomass combustion environments stems primarily from the chemical composition of biomass fuels. Biomass typically contains high concentrations of alkali metals (primarily potassium and sodium) and chlorine, combined with relatively low sulfur content compared to fossil fuels [1,3]. During combustion, these elements are released into the flue gas, forming alkali chlorides such as KCl and NaCl, which condense on the relatively cooler surfaces of superheater tubes [2,4]. The deposited chlorides initiate a series of corrosion mechanisms, including active oxidation (the so-called „chlorine cycle”), where chlorine acts as a catalyst to continuously remove protective oxides and accelerate metal wastage [1,4]. Additionally, the presence of low-melting-point salt mixtures, such as KCl-ZnCl₂ and PbCl₂-KCl, can form molten phases at superheater operating temperatures, leading to rapid hot corrosion through oxide dissolution and fluxing mechanisms [5]. Furthermore, biomass combustion generates high concentrations of water vapor (10–20%), which can enhance the transport of aggressive species through oxide scales [6,7]. In contrast, coal combustion primarily produces sulfates, which are less corrosive at typical superheater temperatures, while gas combustion generates significantly lower levels of corrosive species [2].

To mitigate these corrosion challenges, various material solutions have been investigated, ranging from low-alloyed ferritic steels (e.g., 13CrMo44, HCM12A) used at lower temperatures, through austenitic stainless steels (e.g., TP347H, 304L, 310S, Sanicro 28) that offer intermediate corrosion resistance, to nickel-based alloys (e.g., Inconel 625, Hastelloy C-2000) that provide superior protection at higher temperatures [1,5,7]. However, the relationship between alloy composition and corrosion resistance is complex. In straw-fired boilers, high-chromium steels exhibit rapid attack through selective chromium depletion and grain boundary corrosion, with optimal corrosion resistance achieved at intermediate chromium contents of approximately 12–18% [1]. Modern approaches increasingly employ protective coatings, such as FeCrAl weld overlays and nickel-based overlay coatings, which combine a corrosion-resistant surface layer

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with a mechanically robust substrate [7]. These coating systems can form protective oxide scales (Cr_2O_3 , Al_2O_3) that provide barriers against chlorine ingress, although their performance depends critically on maintaining a stable „healing layer” beneath the scale [7]. Despite these advancements, the combination of elevated metal temperatures (typically 500–600°C) and the highly corrosive flue gas environment often results in breakaway corrosion, limiting steam temperatures in biomass boilers to approximately 540°C and thus reducing overall electrical efficiency compared to fossil-fired units [1,7].

This work presents the findings of a post-exposure microstructural investigation into the failure of a superheater tube fabricated from SA213 TP310HCbN austenitic steel with an Inconel 625 weld overlay, following 10 years of service in a biomass-fired boiler.

2. Results and summary

The study, primarily utilizing scanning electron microscopy (SEM), EDS and WDS X-ray microanalysis, was conducted to identify the principal material degradation mechanisms responsible for the tube’s failure. The analysis revealed a complex, synergistic degradation process initiated by an aggressive flue gas environment, characterized by the presence of sulfur (S), chlorine (Cl), potassium (K), sodium (Na), zinc (Zn), and phosphorus (P). At elevated operating temperatures, these species formed low-melting-point compounds and eutectics (e.g., sulfates and chlorides), which attacked and destroyed the protective chromium oxide (Cr_2O_3) scale on the Inconel 625 overlay, leading to severe hot corrosion. This attack enabled the inward diffusion of corrosive elements, initiating intergranular and interdendritic cracking within the overlay (Fig. 1a). The failure mode was identified as stress corrosion cracking (SCC), which initiated in the degraded Inconel 625 overlay and subsequently propagated into the underlying austenitic steel (Fig 1b). The propagation was accelerated by the sensitized state of the TP310HCbN steel, resulting from chromium carbide precipitation, and the increasing stress intensity factor at the crack tip. It is concluded that the synergistic action of sulfur and chlorine from the biomass combustion environment, coupled with the elevated service temperature and resulting microstructural degradation, rendered the tube susceptible to SCC, leading to a loss of integrity after a decade of service.

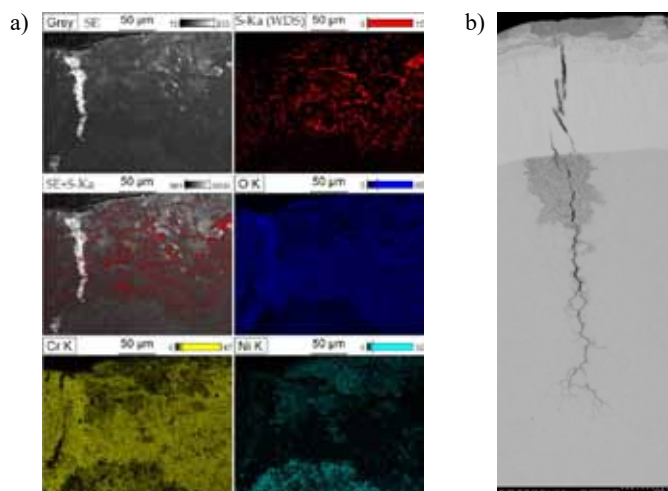


Figure 1. a) elemental mapping in the near-surface zone, EDS/WDS; b) BE image of cross-section of superheater tube wall.

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SEM/EDS Investigation of Gradient Silicide–Aluminide Coatings on Ti-6Al-4V: Microstructure, Nanoprecipitates and Self-Healing Effects

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Keywords: Ti-6Al-4V, silicide-aluminide coatings, cyclic oxidation, SEM/EDS, self-healing

1. Introduction

Titanium alloy Ti-6Al-4V is widely used in aerospace applications due to its excellent strength-to-weight ratio and corrosion resistance. However, its operational temperature is limited to approximately 350–600°C because of the formation of non-protective, porous TiO₂ scales. To overcome this limitation, gradient silicide-aluminide coatings were produced on Ti-6Al-4V substrates using a novel slurry cementation method.

The slurry consisted of Al and Si powders, sodium silicate (water glass) as an inorganic binder, and a non-halide activator mixture (NaF, KCl, NaCl). Coatings were formed in argon atmosphere at 900–1100°C for 2–6 h. This process yielded a multilayer gradient structure with increasing aluminium content toward the surface, combined with beneficial silicon additions.

2. Experimental Methods

Detailed microstructural characterization was performed using field emission scanning electron microscopy (FE-SEM, Hitachi SU-70) equipped with energy dispersive X-ray spectroscopy (EDS, Thermo Scientific NORAN System 7). Cross-sections were prepared by mechanical polishing followed by low-angle (7°) argon ion milling. EDS point analyses and elemental mapping were conducted at 15–20 kV to identify phase compositions and elemental distributions. Phase identification was supported by X-ray diffraction (XRD). Cyclic oxidation tests were carried out at 800°C for 30 cycles of 22 h each (total 660 h) in static air, with mass change monitoring after each cycle.

3. Results and Discussion

3.1. As-deposited coating microstructure

FE-SEM/EDS investigations revealed a consistent four-layer architecture (Figure 1): Layer 1 (near substrate): Ti₃Al; Layer 2: TiAl; Layer 3: TiAl₂; Layer 4 (outer): TiAl₃, Ti₃Si₃ (or Ti₅Si₄) precipitates were observed – “wool-type” in lower-temperature coatings and spherical in 1100°C coatings. Total coating thickness ranged from 35–60 μm depending on process parameters.

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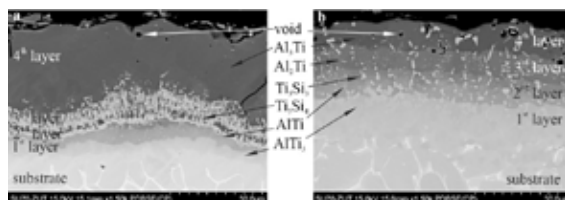


Figure 1. Microstructures (BSE-SEM cross-section) of as-deposited Al-Si coating on Ti-6Al-4V manufacturing in argon atmosphere, the sample number (a) 2 (1000°C/2 h), (b) 8 (1100°C/6 h) [1].

3.2. Microstructural evolution after cyclic oxidation

After 660 h at 800°C, the gradient multilayer structure was largely preserved, with moderate thickening, especially of the Ti_3Al layer (Figure 2). A continuous, adherent $\alpha\text{-Al}_2\text{O}_3$ scale formed on the surface, confirmed by EDS and XRD. Kirkendall voids in the outer TiAl_3 layer contained aluminium oxide.

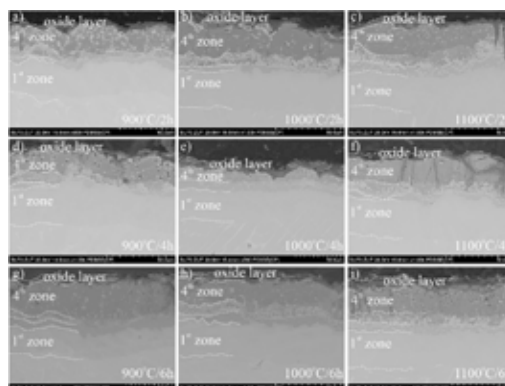


Figure 2. Cross-section microstructure of silicide-aluminide coatings after cyclic oxidising: (a) No 1, (b) No 2, (c) No 3, (d) No 4, (e) No 5, (f) No 6, (g) No 7, (h) No 8, (i) No 9 [1].

Minor perpendicular microcracks in the brittle outer layer (caused by thermal shock and CTE mismatch) were self-healed by infilling with Al_2O_3 (and minor SiO_2), as clearly visible in high-magnification SEM images and EDS maps (Figure 3).

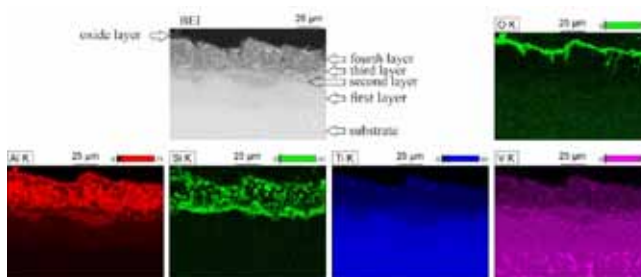


Figure 3. Elemental mapping in cross section in coating No 1 (manufactured at 900°C, 2 h) after cyclic oxidation at 800°C [1].

Long-term annealing during cyclic oxidation at 800°C for 660 h caused a significant reduction of silicon content in the outer TiAl_3 layer (from up to 7 at.% in the as-deposited state to ~1 at.%). This decrease was most pronounced in coatings produced at 900–1000°C and resulted from the precipitation of fine Ti_5Si_3 particles (200–300 nm long, ~50 nm wide), clearly visible in high-magnification SEM images (Figure 4). These nanoprecipitates contribute to dispersion strengthening, inhibit dislocation climb, and effectively impede inward oxygen diffusion, thereby suppressing the formation of non-protective TiO_2 .

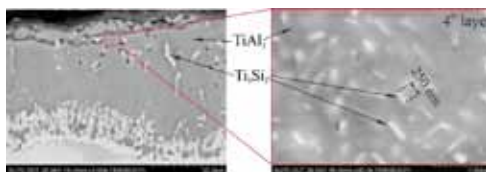


Figure 4. Microstructures (BSE) of Al-Si coating (No. 6) on Ti-6Al-4V manufacturing at 1000°C for 4 h, after cyclic oxidation at 800°C for 660 h [1].

Mass gain for coated samples was only 0.7–1.7 $\text{mg}\cdot\text{cm}^{-2}$ versus ~143 $\text{mg}\cdot\text{cm}^{-2}$ for uncoated alloy, demonstrating superior oxidation resistance.

4. Conclusions

Comprehensive FE-SEM/EDS micro- and nano-analysis demonstrated that slurry silicide-aluminide coatings on Ti-6Al-4V form a stable gradient structure that provides excellent cyclic oxidation resistance at 800°C for at least 660 h. The protective mechanism relies on the formation of a continuous $\alpha\text{-Al}_2\text{O}_3$ scale, self-healing of microcracks, and beneficial effects of Ti_5Si_3 nanoprecipitates. These findings highlight the coatings' potential for high-temperature aerospace applications and underscore the critical role of advanced SEM/EDS characterization in understanding protection mechanisms at the micro- and nano-scale.

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Composition-Dependent Phase Constitution and Microstructural Evolution in W-Ta-Cr-V High-Entropy Alloy Thin Films

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Keywords: compositionally graded thin films, high-entropy alloys, fusion materials

W-Ta-Cr-V high-entropy alloys are promising candidates for fusion-related applications due to their body-centered cubic structure, chemical complexity, and potential stability under demanding conditions. However, the influence of non-equimolar composition on phase constitution and microstructural development remains insufficiently understood.

In this work, a compositionally graded W-Ta-Cr-V thin film was investigated as a combinatorial platform for studying composition–microstructure relationships. The film was deposited by four-source magnetron sputtering, generating a continuous variation in W, Ta, Cr, and V concentrations across a single wafer. This approach enables multiple local chemistries and microstructural states to be analyzed within one material system.

The material was characterized using SEM-EDS, XRD, and TEM investigations of FIB-prepared lamellae. SEM-EDS analysis confirmed a continuous compositional gradient and revealed pronounced morphology changes related to local alloy chemistry, ranging from smooth nanocrystalline regions to textured and columnar microstructures. XRD measurements indicated a predominant BCC structure with a strong preferred (110) orientation, while peak broadening in selected regions suggests increased lattice distortion and/or reduced crystallite size. TEM observations provided further insight into local grain structure, crystallographic features, and possible nanoscale phase constitution across selected regions of the compositional gradient.

These results demonstrate that local composition strongly affects crystallization behavior and microstructural evolution in W-Ta-Cr-V high-entropy alloy thin films. The investigated material provides a basis for selecting representative compositions for future helium ion irradiation, nanoindentation, TEM, and micropillar compression studies aimed at understanding composition-dependent irradiation response.

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From Microstructure to Structural Integrity: SEM–EBSD Based Assessment of Creep–Fatigue Damage in Ferritic Heat-Resistant Steel

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Keywords: EBSD, creep, fatigue, heat-resistant steel

1. Introduction

Power plant components operating under flexible service conditions are increasingly exposed to creep–fatigue interaction (CFI) resulting from simultaneous cyclic thermal and mechanical loading at elevated temperatures [1,2]. The combined action of creep and fatigue accelerates material degradation through progressive accumulation of plastic strain, dislocation rearrangement, microstructural instability and eventual crack initiation, significantly reducing the remaining service life of critical components [3].

Conventional diagnostic techniques are generally capable of detecting degradation only after the appearance of macroscopic damage. Consequently, there is an increasing demand for advanced microstructural characterization methods capable of identifying the earliest stages of degradation before crack formation [4].

Among these techniques, Scanning Electron Microscopy (SEM) combined with Electron Backscatter Diffraction (EBSD) offers exceptional capability for quantitative assessment of local deformation. EBSD enables crystallographic characterization of strain accumulation through parameters such as Kernel Average Misorientation (KAM), Grain Reference Orientation Deviation (GROD) and Geometrically Necessary Dislocation density (GND), which directly reflect lattice distortion and dislocation evolution. When combined with complementary high-resolution techniques, EBSD provides a powerful tool for understanding degradation mechanisms across multiple length scales. The aim of this work was to evaluate the applicability of SEM–EBSD as a quantitative diagnostic methodology for creep–fatigue damage assessment in ferritic heat-resistant steel. Particular attention was devoted to identifying EBSD parameters that can serve as reliable indicators of degradation progression and support the future development of practical lifetime assessment procedures for power plant components.

2. Experimental Procedure

The investigations were performed on ferritic heat-resistant steel 14MoV6-3 removed from a long-term operated steam pipeline. Specimens representing the most degraded material condition

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were subjected to stress-controlled creep–fatigue tests performed at 540°C using a 5 s tensile hold time, reproducing thermo-mechanical loading conditions occurring during power plant operation. To investigate the evolution of degradation, tests were interrupted at three characteristic fractions of fatigue life: 0.3Nf, 0.6Nf and Nf.

Microstructural characterization was carried out using Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD) as the principal analytical techniques. EBSD measurements were performed using a field-emission scanning electron microscope equipped with an Oxford Symmetry EBSD detector employing a 0.20 μm step size. Quantitative analysis included: Kernel Average Misorientation (KAM), Grain Reference Orientation Deviation (GROD), eometrically Necessary Dislocation density (GND), Low-Angle Grain Boundaries (LAGB, $2\div15^\circ$), High-Angle Grain Boundaries (HAGB, $>15^\circ$).

To validate the EBSD observations and provide a comprehensive description of deformation mechanisms, the microstructural analysis was complemented by Transmission Electron Microscopy (TEM) and synchrotron X-ray diffraction. TEM enabled direct observation of dislocation arrangements and carbide evolution at the nanoscale, whereas synchrotron diffraction provided quantitative measurements of bulk dislocation density and lattice microstrain. The combination of these techniques enabled characterization of creep–fatigue degradation from the atomic scale to the engineering scale.

3. Results

The evolution of EBSD strain parameters clearly demonstrates the progressive accumulation of deformation during creep–fatigue loading (Figs. 1–2). At 0.3Nf, KAM maps reveal only isolated regions of increased local misorientation located mainly near grain boundaries. With increasing fatigue life (0.6Nf), the deformation becomes more uniformly distributed throughout the microstructure, indicating the development of subgrain structures. At the final stage (Nf), extensive regions of high KAM values confirm severe strain localization preceding failure.

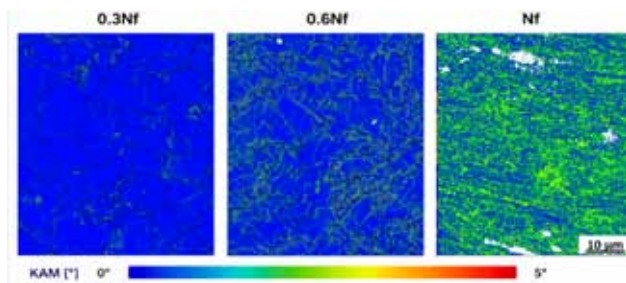


Figure 1. Representative EBSD KAM maps at different fatigue life fractions (0.3 Nf, 0.6 Nf and Nf), illustrating the gradual increase in local lattice misorientation with increasing material degradation.

Quantitative analysis shows a systematic increase of all investigated EBSD parameters. KAM increases from 0.22° to 0.63° , GROD from 3.45° to 12.34° , while GND density rises from $1.47 \cdot 10^{14} \text{ m}^{-2}$ to $6.71 \cdot 10^{14} \text{ m}^{-2}$. Simultaneously, the fraction of LAGB increases from 18.1% to 69.4%, accompanied by a decrease in HAGB from 81.9% to 30.6%, indicating progressive subgrain formation and dislocation rearrangement.

The proposed degradation model (Fig. 3) summarizes the observed microstructural evolution from the initial accumulation of dislocations, through subgrain development, to intensive strain localization immediately before fracture. Among the investigated parameters, KAM exhibited the

highest sensitivity to damage evolution, indicating its strong potential as a quantitative diagnostic parameter for creep–fatigue assessment.

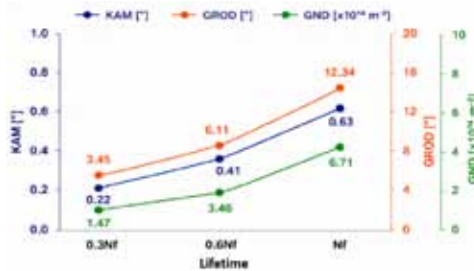


Figure 2. Evolution of the average KAM, GROD and GND values as a function of fatigue life fraction, demonstrating the progressive accumulation of plastic deformation during creep–fatigue loading.

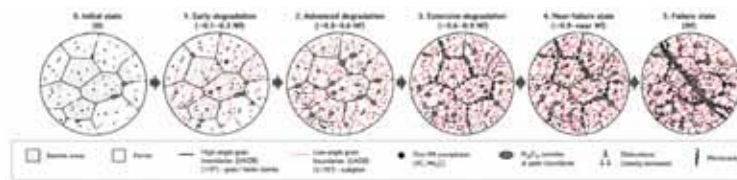


Figure 3. Schematic illustration of the microstructural evolution during creep–fatigue degradation, showing the progressive formation of LAGBs, carbide precipitation, dislocation accumulation and microcrack initiation up to failure.

4. Conclusions

- 1) EBSD parameters (KAM, GROD and GND) increased systematically with creep–fatigue degradation, indicating progressive accumulation of plastic strain and dislocation density.
- 2) Microstructural observations revealed the gradual development of LAGBs, carbide precipitation at grain boundaries and microcrack initiation prior to failure.
- 3) The combined SEM–EBSD approach provides a sensitive tool for assessing the degradation state of ferritic heat-resistant steels under creep–fatigue conditions.
- 4) The proposed degradation model may support the development of engineering guidelines for residual life assessment of high-temperature power plant components.

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Electron microscopy and X-ray diffraction characterization of mechanically alloyed and SPS-consolidated Ni–Co–Mn–In–Fe–Cr Heusler alloys

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Keywords: Electron microscopy, in situ X-ray diffraction, Ni–Co–Mn–In–Fe–Cr Heusler alloys,
spark plasma sintering (SPS), magnetic shape memory alloys

1. General

Ni–Mn-based magnetic shape memory alloys (MSMAs) are functional Heusler materials exhibiting coupled magnetic and structural transformations, which enable magnetic-field-induced strain, magnetocaloric response, and shape memory effects. Due to their fast response and magnetic control, these alloys are considered promising candidates for actuators, sensors, and advanced cooling technologies [1–3]. Among them, multicomponent Ni–Co–Mn–In-based alloys are of particular interest because their transformation temperatures, phase stability, and magnetic properties can be tailored by chemical modification [4]. However, the production of these materials in powder form suitable for powder metallurgy and additive manufacturing remains challenging, especially due to difficulties in controlling phase composition, chemical homogeneity, and microstructural evolution during processing [5].

The aim of this work was to investigate the phase and microstructural evolution of mechanically alloyed multicomponent Ni–Co–Mn–In–Fe–Cr Heusler alloy powders and SPS-consolidated materials. Mechanical alloying was carried out using different milling times and ball-to-powder ratios (BPR) in order to determine their influence on powder morphology, structural refinement, phase formation, and chemical homogeneity.

The crystallization process from the partially amorphous state and structural transformations occurring during heating were analyzed using in situ X-ray diffraction (XRD). Microstructural and chemical characterization was performed by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy-dispersive X-ray spectroscopy (EDS). The characterization of obtained material was focused on the morphology and size of powder particles, local chemical distribution, phase composition, and the development of fine-grained microstructures after consolidation. The mechanically alloyed powders were further consolidated by spark plasma sintering (SPS) at different temperatures to evaluate the effect of thermal processing on phase constitution and microstructural stability.

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The results showed that high-energy mechanical alloying led to the formation of highly refined multicomponent powders with a partially amorphous structure. In situ XRD revealed progressive crystallization and formation of the Heusler-type $L2_1$ phase during heating. After SPS consolidation, dense materials with a fine-grained two-phase microstructure were obtained, composed mainly of the Heusler phase with γ -phase particles. Electron microscopy observations confirmed that the applied powder metallurgy route strongly affected particle morphology, phase distribution, and local chemical homogeneity. Although the martensitic transformation was limited in the as-sintered state, post-sintering heat treatment resulted in a detectable functional response, indicating that further optimization of thermal treatment and processing parameters may improve the shape memory behavior.

This study highlights the importance of combined microstructural, chemical, and phase analysis in understanding the processing–structure–property relationships in multicomponent Heusler alloys. The obtained results demonstrate that mechanical alloying combined with SPS consolidation is a promising route for producing refined Ni–Co–Mn–In–Fe–Cr Heusler-based materials for future applications in additive manufacturing and functional composite systems.

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Microstructural insights into the mechanisms of hydrogen embrittlement in materials for hydrogen-fuelled gas turbine systems

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Keywords: Ni-based superalloys, hydrogen embrittlement, mechanical properties,
microstructural evolution

1. Introduction

Hydrogen is expected to become an important carbon-free fuel for future gas turbine systems. However, replacing conventional fuels such as natural gas with hydrogen changes not only the combustion conditions but also the degradation mechanisms experienced by structural materials. It is related to an absorption of hydrogen and as a consequence reduction in the ductility of a metal [1]. This is especially an issue in terms of duplex or superaustenitic steels [2]. In this context, the Ni-based alloys seems to be very promising, especially in terms of hydrogen cracking. This points to a fundamental issue which is the decarbonization of Poland and the international economy. The two main problems are connected, first with the hydrogen corrosion resistance and second with a much higher exposition to water vapor at a very high temperature. In addition to oxidation and high-temperature corrosion, hydrogen-assisted degradation, including hydrogen embrittlement, becomes a critical concern for components exposed to hydrogen during storage, transport, fuel delivery and operation. Therefore, understanding the interaction between hydrogen and structural materials is essential for the safe and reliable deployment of hydrogen-fuelled gas turbine systems.

Gas turbine systems comprise components operating under a wide range of temperatures and mechanical loads and therefore employ different classes of structural alloys [3]. Duplex stainless and super austenitic steels are attractive for hydrogen storage and fuel delivery systems owing to their high strength and corrosion resistance, whereas nickel-based superalloys are indispensable for high-temperature components because of their exceptional mechanical stability and oxidation resistance. Despite their widespread use, these alloy systems exhibit markedly different susceptibility to hydrogen-assisted degradation. The microstructural origins of these differences, including phase distribution, hydrogen trapping behaviour, precipitate characteristics and deformation mechanisms, remain insufficiently understood.

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This work therefore aims to compare the microstructural response of duplex stainless steel, superaustenitic steel and a modified René N5 nickel-based superalloy to hydrogen exposure in order to identify the mechanisms governing hydrogen-assisted degradation in these two alloy systems. The results provide new insight into the role of microstructure in controlling hydrogen tolerance and may contribute to the selection and development of materials for future hydrogen-fuelled energy systems.

2. Experimental procedure

Duplex stainless steel, superaustenitic Al-6XN steel and a modified René N5 nickel-based superalloy were selected as model materials for this study. To evaluate their susceptibility to hydrogen-assisted degradation, several hydrogen charging experiments were performed. Electrochemical hydrogen charging was conducted in 0.1 M H_2SO_4 at a current density of approximately 10 mA/mm² with a pre-charging time of 15 min. To assess the influence of hydrogen on the mechanical response, electrochemical charging was combined with in situ tensile testing under identical charging conditions. In addition, immersion hydrogen charging was carried out by exposing the specimens to the same electrolyte at room temperature for four days. Immediately after immersion, the specimens were characterized by high-energy synchrotron X-ray diffraction. Spatially resolved measurements were performed from the specimen edge towards the interior with beam positions at 0.2, 0.4, 0.7 and 1.2 mm to investigate hydrogen-induced lattice changes and their penetration depth. Samples subjected to in situ hydrogen-assisted tensile testing were also examined by synchrotron diffraction using a finer spatial resolution, with measurements acquired from the specimen edge to a depth of 100 μm in 10 μm increments, to investigate the microstructural evolution associated with hydrogen-assisted deformation. Finally, detailed microstructural characterization was performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

3. Results and discussion

The initial microstructures consisted of austenite (FCC) in Al-6XN and both austenite and ferrite (BCC) in the duplex stainless steel (DSS) as well as a γ (FCC) matrix in the modified René N5 superalloy, as expected for these alloy systems. Following hydrogen charging, pronounced microstructural changes were observed in DSS, whereas the René N5 alloy remained largely unaffected (Fig.1). Comparison of synchrotron diffraction patterns before and after electrochemical hydrogen charging revealed significant changes in the relative peak intensities for DSS. Rietveld refinement showed that the BCC phase fraction increased to 75.5% at the expense of the FCC phase, representing an increase of approximately 40% compared with the initial condition and more than 20% compared with the tensile-tested specimen without hydrogen charging. These observations are consistent with hydrogen-assisted martensitic transformation in DSS. Furthermore, diffraction peaks became noticeably broadened after hydrogen charging, indicating increased lattice strain and internal stresses that may facilitate the phase transformation.

In contrast, the diffraction patterns of the modified René N5 superalloy remained nearly unchanged after hydrogen charging, indicating considerably higher resistance to hydrogen-induced microstructural degradation. Mechanical testing further supported these observations, as hydrogen-charged DSS exhibited increased strength accompanied by a significant reduction in ductility compared with the uncharged condition, consistent with the formation of strain-induced martensite.

Spatially resolved synchrotron diffraction following immersion charging demonstrated that hydrogen-induced lattice changes extended to a depth of approximately 150 μm in DSS, whereas they were confined to only 10–20 μm in the René N5 superalloy. This pronounced difference in hydrogen penetration provides further evidence of the superior resistance of the nickel-based superalloy to hydrogen-assisted degradation.

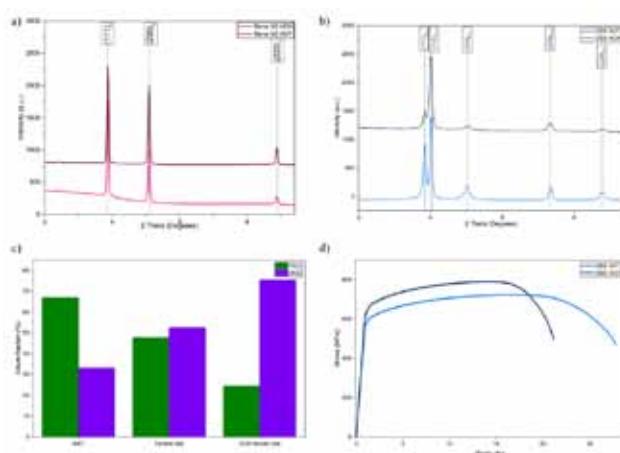


Figure 1. Comparison of synchrotron diffraction patterns in the initial state and after hydrogen charging for a) modified René N5 nickel-based superalloy and b) duplex stainless steel (DSS). c) Rietveld-based quantitative phase analysis and d) corresponding mechanical property comparison of DSS in the uncharged and hydrogen-charged conditions.

4. Conclusions

The results demonstrate fundamentally different microstructural responses of stainless steel and the modified René N5 superalloy to hydrogen exposure. While hydrogen promoted phase transformation, lattice distortion and deeper penetration into the duplex stainless steel, the nickel-based superalloy exhibited only minor hydrogen-induced structural changes. These findings highlight the critical role of alloy microstructure in controlling hydrogen-assisted degradation and provide valuable insight into the selection and design of structural materials for future hydrogen-fueled gas turbine systems.

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Effect of Specimen Thickness on Microstructural Stability and Oxidation Behavior of Modified CMSX-6 and René 5 Superalloys

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Keywords: superalloys, microstructure evolution, oxidation, heat treatment

1. Introduction

Nickel-based materials are recognized for their excellent mechanical strength and corrosion resistance at high temperatures, thus making them strong candidates for use in high-temperature applications such as gas turbines and aerospace engines [1–3]. The growing demand for higher performance of these industries has intensified the search for materials capable of maintaining structural integrity and withstanding harsh environments. Furthermore, the rapid development of advanced manufacturing technologies, particularly additive manufacturing (3D printing), has enabled the production of components with complex geometries and variable wall thicknesses. These design innovations place even greater emphasis on achieving superior mechanical performance and corrosion resistance to ensure the long-term reliability and durability of such components under demanding operating conditions. To elevate the high-temperature mechanical properties of nickel-based alloys, appropriate heat treatment processes are applied to promote formation of γ' phase [4,5]. However, formation of this γ' phase often compromises the integrity of oxide layers [6–8].

Turbine blades feature presence of complex geometries and non-uniform cross-sections, including regions of reduced wall thickness that are particularly susceptible to thermal effects. These geometric features can lead to variations in oxidation behavior and can alter the morphology and growth kinetics of oxide layers [9,10].

This study investigates the behavior of specific nickel-based superalloys with various thicknesses to evaluate the role of local geometry in microstructure stability and oxidation processes.

2. Experimental procedure

Two nickel-based superalloys, René 5 and CMSX-6, were selected for the investigations, both of which containing alloying elements additions such as Ta, Mo, Re and Cr, with concentrations ranging from 1% to 4%. From the bulk materials, specimens with thicknesses of 0.1, 0.25, 0.5, 1 and 2 mm were cut to investigate the effect of geometrical constraints on microstructure stability and oxidation behavior. All samples were subjected to a surface preparation ensuring

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reproducibility and surface uniformity. The surfaces were ground using SiC abrasive papers up to a grid size of 7000, followed by degreasing in acetone for 2 minutes. The samples were also cleaned in ethanol for additional 2 minutes to ensure the removal of any residual contaminants. Prepared specimens were then subjected to a three-stage heat treatment consisting of heating in 1270°C for 2 hours, in 1070°C for 4 hours and in 870°C for 20 hours. To evaluate the influence of sample thickness and alloying additions on microstructural stability and oxidation behavior, a combination of techniques was employed: phase identification and quantitative phase analysis were performed using high-energy synchrotron X-ray diffraction at the PETRA III facility (DESY, Hamburg, Germany). Cross-sectional microstructural characterization was carried out using scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and energy-dispersive X-ray spectroscopy (EDS). Surface morphology was investigated using scanning electron microscopy (SEM). More detailed microstructural characterization was performed with the use of transmission electron microscopy (TEM). The mass of each specimen was measured before and after the heat treatment, allowing the mass change associated with oxide scale formation to be determined.

3. Results

Immediately after heat treatment, it was evident that the sample surfaces differed markedly from one another, showing distinct types and extents of surface modification. In particular, the Rene N5 + 4Mo specimen with 0.1 mm wall thickness underwent severe degradation and disintegrated after the heat-treatment. This visual assessment was further confirmed during phase analysis performed using high-energy synchrotron X-ray diffraction.

Figure 1 presents representative synchrotron diffraction patterns illustrating the effect of specimen thickness on the phase composition of the investigated CMSX-6 and René 5 superalloys after heat treatment in air. Regardless of specimen thickness and alloy composition, the diffraction patterns are dominated by reflections corresponding to the γ matrix and the γ' strengthening precipitates. However, as the specimen thickness decreases, additional low-intensity diffraction peaks become apparent, particularly for specimens with thicknesses of 0.10 and 0.25 mm.

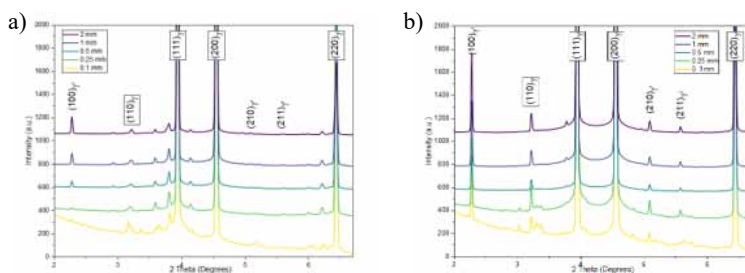


Figure 1. X-ray synchrotron diffraction pattern of the René 5 + 2Mo (a) and CMSX-6 + 4Re (b) superalloys.

The appearance of these additional reflections suggests enhanced degradation processes in the thinner specimens, most likely associated with oxidation during heat treatment in air [9]. Although oxide layers are expected to form on specimens of all thicknesses, they represent a proportionally larger fraction of the diffracting volume in thinner samples, which may account for the increased intensity of the additional reflections. Several of the observed peaks are located close to the expected diffraction positions of α - Al_2O_3 and Cr_2O_3 ; however, they cannot be unambiguously assigned to these phases. This may result from the formation of non-stoichiometric or mixed oxide phases, which are commonly observed during the early stages of high-temperature oxidation [10,11].

Alternatively, the additional reflections may indicate the formation of secondary phases induced by local compositional changes resulting from high-temperature exposure. Oxidation-driven depletion of alloying elements near the surface may alter the phase stability and promote the precipitation of new phases, leading to a more complex diffraction pattern in the thinner specimens [9]. Further phase identification is required to distinguish between these possible mechanisms.

4. Summary

Although definitive phase identification requires further analysis, the appearance of additional diffraction peaks in the thinner specimens suggests that reduced specimen thickness influences microstructural stability and oxidation behaviour during heat treatment in air. These findings indicate that specimen geometry may play an important role in the degradation mechanisms of Ni-based superalloys exposed to high temperatures. Further investigations using cross-sectional SEM/EDS and TEM will be performed to identify the newly formed phases and to clarify the relationship between specimen thickness, oxidation, and microstructural evolution.

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Surentall – database of physicochemical and thermodynamic properties of materials. Integration of Surdat and Entall databases

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Keywords: Surdat database, Entall database, Miedema model, HEA

The thermodynamic and physicochemical properties of metals and their alloys are among the fundamental data used in materials engineering as well as in many other fields of science and technology. The knowledge of these properties is essential for designing new materials and modelling technological processes. In the era of science digitalization, a key challenge is not only the collection and verification of such data, but also its integration and easy accessibility for researchers and engineers worldwide.

Research on the physical properties of lead-free alloys—substitutes for traditional solders based on Sn-Pb eutectic alloys began in 1998 at the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences (IMMS PAS). These studies led to the publication of the first version of the Surdat database in 2007 [1]. The database, provided as an installation package along with a monograph, was made freely available on the IMMS PAS website: <http://www.imim.pl>. It included experimental measurement results of surface tension and density for metals, binary alloys and selected multicomponent alloys, mainly based on Sn-Ag and Sn-Ag-Cu eutectics with additions of In, Bi, and Sb. Figure 1 shows the cover of the monograph and the installation disc containing the Surdat database installation program [1].



Figure 1. The Surdat database released as a monograph in 2007, together with the installation program [1].

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Work on the development of the physicochemical properties database continued and led to the creation of a new version, Surdat 2 [2]. This version also included the NIST (National Institute of Standards and Technology) database, which was available as a website or a PDF file. Figure 2 shows the cover of the monograph and the installation disc containing the Surdat 2 database program. The next version, Surdat 3 [3], shown in Figure 3, was released in 2019. All versions of the database were published as desktop applications that needed to be installed on a computer in order to be used. More than a decade has passed since the decision to create the Surdat database was made. During this time, web applications have developed significantly, which now makes the desktop format of the database a considerable limitation for its public use.



Figure 2. The Surdat 2 database – monograph with installation program [2].



Figure 3. The Surdat 2 database – monograph with installation program [3].

After the release of the second version of the Surdat database, work began at IMMS PAS on the development of a thermodynamic database called Entall [4], which was planned as a web-based application. Figure 4 shows the data presentation format in the Entall database [4].



Figure 4. Data presentation in the Entall database [4]. <https://entall.imim.pl/>.

The existing Surdat and Entall databases are valuable sources of information on the properties of metals and their alloys. However, these databases are not compatible with each other. Therefore, the aim of this work is to present a new solution that integrates the Entall and Surdat databases within a modern web application, enabling fast, intuitive and unified access to physicochemical and thermodynamic data of metals and their alloys.

The integration of the Entall and Surdat databases and their free release as the new Surentall application constitutes a significant step towards unlocking the potential of these databases. This new version of the web application provides users not only with access to the full range of data but also with the ability to visualize the data, export it in formats commonly used by users, and easily integrate it with other software. Thanks to the use of modern web technologies and optimization of the user interface, the application represents a breakthrough in access to information on selected thermodynamic and physicochemical properties, enabling scientists and engineers to conduct research and implement new technologies more efficiently. Additionally, the new form of the application not only meets the current needs of science and industry but also opens new perspectives for interdisciplinary research and modelling of technological processes. Figure 5 shows the data presentation format in the new Surentall database.

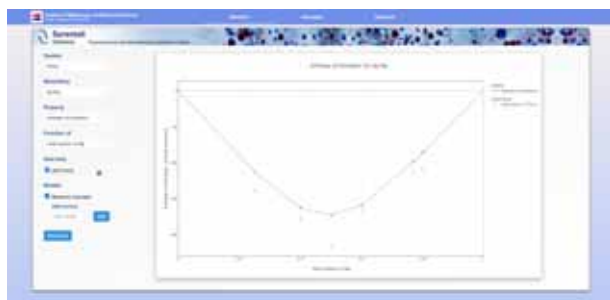


Figure 5. Prediction of the enthalpy of formation of intermetallic phases in the Ag-Mg system using the Miedema Model [4,5].

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Tailoring microstructure and martensitic transformation in TiHfZrCoCuNi alloys by varying Zr content

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Keywords: high-entropy shape memory alloys, martensitic transformation, SEM/EDS, X-ray diffraction

1. General

NiTi-based high-entropy and multi-principal element shape memory alloys have recently attracted increasing interest as promising high-temperature functional materials. Among them, TiZrHfNiCuCo alloys are considered a particularly interesting group due to their ability to undergo martensitic transformation between ordered B2 austenite and B19' martensite [1]. In these alloys, the B2 phase may be treated as an ordered complex solid solution, in which Ti, Zr and Hf preferentially occupy Ti-type sublattice sites, whereas Ni, Co and Cu occupy Ni-type sites [2]. Literature data show that transformation temperatures, phase stability and microstructural features are strongly affected by chemical composition, elemental partitioning and the relative content of Ti-type and Ni-type elements [2,3]. However, the individual influence of Ti-type elements, especially the effect of progressive Zr substitution in the Ti-Hf sublattice, still requires further clarification.

In this work, the influence of Zr content on the phase constitution, microstructure and martensitic transformation of $(\text{TiHf})_{50-x}\text{Zr}_x(\text{CoCuNi})_{50}$ alloys was investigated. Special attention was paid to the relation between Zr content, lattice parameter evolution, chemical contrast observed in backscattered electron images and transformation behaviour determined by differential scanning calorimetry.

2. Materials and methods

The investigated alloys had the nominal composition $(\text{TiHf})_{50-x}\text{Zr}_x(\text{CoCuNi})_{50}$, where $x = 0, 8, 16, 24$ and 32 at.%. The alloys were prepared by vacuum arc melting from pure elemental pieces under an argon protective atmosphere. To improve chemical homogeneity, the obtained ingots were subjected to homogenization annealing at 950°C for 100 h and then furnace cooled.

Phase constitution was analysed by X-ray diffraction. The lattice parameters of the cubic phases were determined using a modified quadratic extrapolation method. Microstructural observations were performed by scanning electron microscopy using backscattered electron imaging. Local chemical composition and elemental partitioning were analysed by energy-dispersive X ray spectroscopy. Martensitic transformation behaviour was studied by differential scanning calorimetry.

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3. Results and conclusions

X-ray diffraction showed that alloys containing 0–24 at.% Zr were mainly composed of a cubic B2 phase. In contrast, the alloy with the highest Zr content, 32 at.%, exhibited dominant martensite coexisting with the B2 austenite phase. The lattice parameter of the cubic phase increased linearly with increasing Zr content. This effect may be related to the larger atomic radius of Zr compared with Ti and its progressive incorporation into the Ti-type sublattice. At the same time, the difference between the lattice parameters of coexisting cubic phases decreased with increasing Zr content, suggesting that Zr promotes chemical and structural homogenization of the matrix.

DSC measurements revealed that alloys containing 0, 8 and 16 at.% Zr did not show clear martensitic transformation peaks in the investigated temperature range. Transformation effects were observed for the alloy containing 24 at.% Zr in the temperature range from approximately –50 to –120°C. A significant increase in transformation temperature was found for the alloy containing 32 at.% Zr, for which transformation effects occurred between approximately 50 and 130°C. These results indicate that Zr strongly affects phase stability and may shift the martensitic transformation to higher temperatures when its concentration in the Ti-type sublattice is sufficiently high.

SEM/BSE observations revealed chemically distinct microstructural features. In the Zr-free alloy, the matrix consisted of two phases visible in chemical contrast, which is consistent with the XRD results. This alloy also contained particles of a third phase enriched in Ti and containing Ni, Co and Hf. With increasing Zr content, the contrast between matrix regions became significantly weaker, indicating improved chemical homogeneity. The alloy containing 32 at.% Zr showed a plate-like morphology typical of martensitic microstructures, which agrees with the dominant martensitic phase identified by XRD. In all investigated alloys, Ti₂Ni like particles were observed.

The obtained results show that Zr substitution in the Ti-Hf sublattice has a pronounced influence on the phase constitution, lattice parameter evolution, microstructural homogeneity and martensitic transformation behaviour of (TiHf)_{50-x}Zr_x(CoCuNi)₅₀ alloys. Increasing Zr content promotes the formation of martensite and shifts the transformation temperature to higher values. Therefore, Zr content can be considered an effective compositional parameter for tailoring the phase stability and potential shape memory behaviour of TiHfZrCoCuNi multi-principal element alloys.

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