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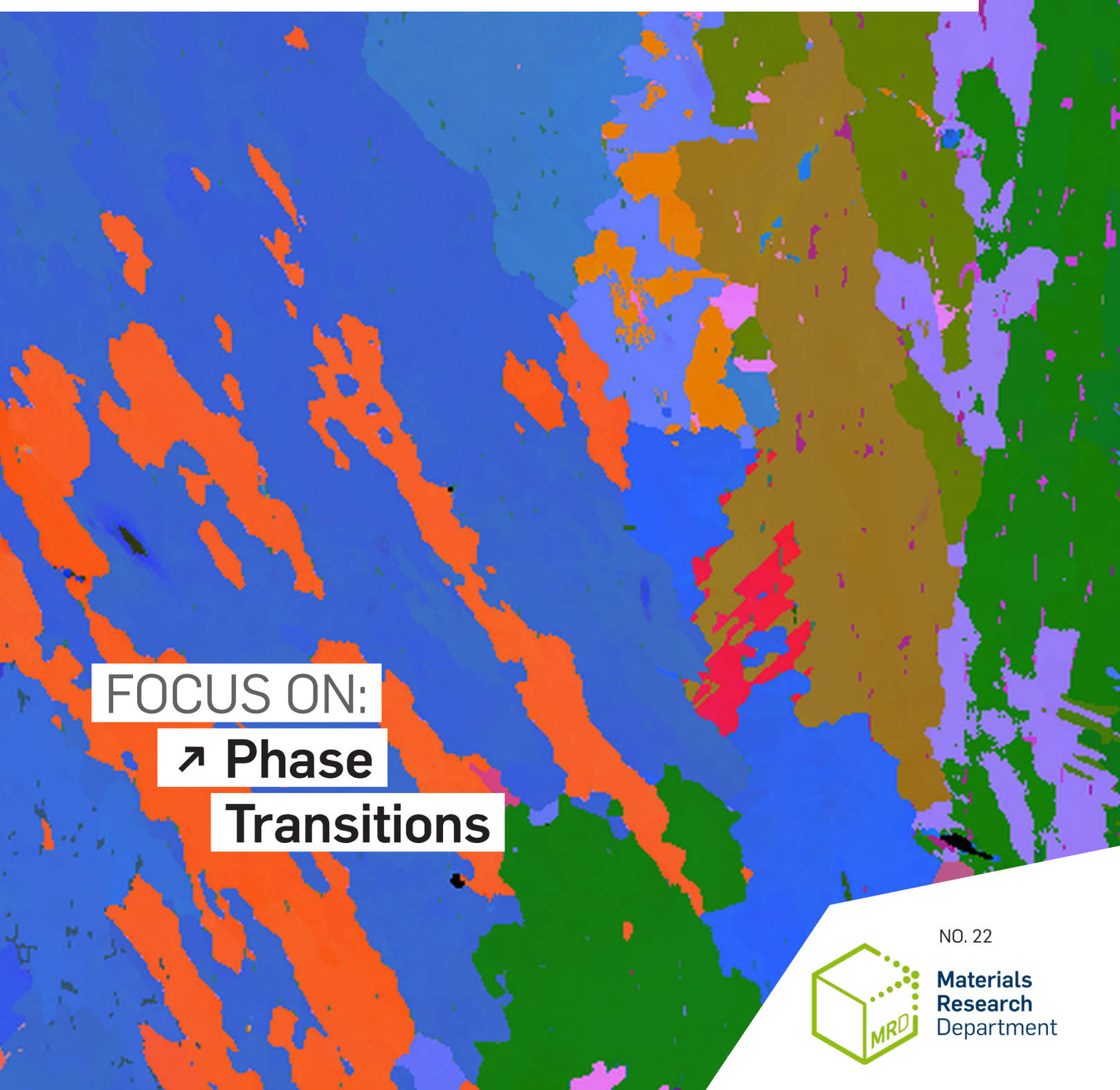

FOCUS ON:

↗ Phase Transitions

NO. 22

Materials Research Department

MRD

MATERIALS RESEARCH
DEPARTMENT

FOCUS ON:
PHASE TRANSITIONS

Controlling phase transformations enables the design of materials with specific properties, whether for energy storage, electronic materials, or structural alloys. This Newsletter highlights recent work and collaborations of our members in this area.

The issue is also available on the MRD website. We welcome topic suggestions for the coming MRD newsletters.

Enjoy reading,

Ralf Drautz and Tong Li
MRD Speakers
Denisa Voicu
MRD Science Manager

CONTENT

On the cover: Euler orientation map obtained through EBSD analysis of an austenitic stainless steel additively manufactured using DED-LB/M (authors: F. Großwendt, J. Lentz, S. Weber).

RELIABLE DETECTION OF PHASE **TRANSFORMATIONS** DURING HOT **ISOSTATIC PRESSING**

Introducing a new method for in-process analysis

Modern high-performance materials, for example for tools, energy systems, or aerospace applications, increasingly require complex thermomechanical processing routes. One important step in this context is Hot Isostatic Pressing (HIP), a process in which powder materials are consolidated and heat treated simultaneously at high temperatures and high gas pressure. This technique enables the production of nearly pore-free components as well as complex material compositions that are often difficult or impossible to achieve with conventional manufacturing methods.

This work presents a new experimental method that enables the reliable detection of phase transformations in alloys during HIP processes. The results were published in 2025 in the journal “High Pressure Research”.

Microstructure formation under heat treatment conditions
The mechanical properties of metallic materials depend on the microstructure formed during heat treatment. A key microstructural evolution is the diffusionless transformation of austenite into martensite, which largely determines properties such as hardness, strength, and toughness in steels.

However, under high pressure and rapid cooling rates, conditions typical for HIP processing, these transformations can behave differently compared to conventional heat treatments. Until now, robust measurement methods capable of detecting these transformations directly during the HIP process have been lacking.

In-situ measurement during processing
To address this challenge, an experimental method based on in-situ four-wire electrical resistance measurements was developed to detect phase transformations during HIP treatment.

A key element of this approach is a specially designed meander-shaped sample geometry. This geometry creates a long electrical conductor path within a very small area, allowing even small changes in electrical resistance to be detected with high sensitivity. At the same time, the design provides a robust and reproducible measurement setup under high-pressure conditions. In contrast to earlier wire-based sample designs, the new geometry offers significantly improved mechanical stability. Under HIP-WB conditions, it is not susceptible to displacement or pronounced geometric deformation, ensuring reliable measurements throughout the process.

Another advantage of this design is that the sample geometry can be applied to a wide range of metallic materials, making the method broadly applicable in materials research. Changes in electrical resistance provide information about the temperatures at which phase transformations occur.

Using this method, it was shown that the process pressure has a significant influence on the martensite start temperature (Ms) of the studied high speed steel. Increasing pressure stabilizes the austenite phase and shifts the onset of martensitic transformation to lower temperatures.

Relevance for research and industry
The developed measurement strategy enables, for the first time, a precise and reproducible analysis of phase transformations under realistic HIP processing conditions. This allows heat treatment processes to be designed and optimized more accurately.

In the long term, this approach opens new opportunities for:
- more energy-efficient manufacturing processes
- optimized properties of high-alloy materials
- improved post-processing of additively manufactured metal components.

Overall, this work demonstrates how innovative measurement techniques can help to better understand and control complex materials processes, an important step toward the development of the next generation of high-performance materials.

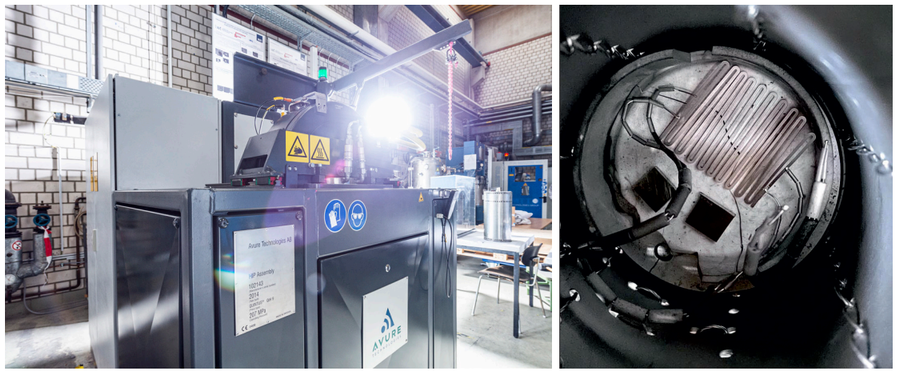

Figure 1: Left: HIP facility at the Chair of Materials Technology (LWT); right: newly designed sample geometry installed inside the HIP furnace chamber.

QR code provides access to the author’s original publication.

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ALTERNATIVE **HIGH TEMPERATURE** MATERIALS

A balancing act: Designing refractory alloys for superior high-temperature performance

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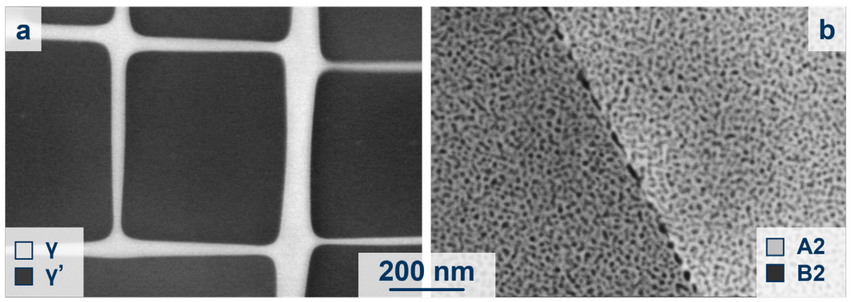
In the quest for sustainable aviation, developing advanced metallic alloys capable of withstanding extreme temperatures is paramount. Traditional Ni-based superalloys reach their physical limits, which are defined by their solvus and solidus temperatures. The former limits the stability of the strengthening phase, while the latter serves as an intrinsic reference temperature, dictating the contribution of diffusion-controlled processes. The outstanding mechanical, high-temperature properties of Ni-based superalloys arise from their peculiar microstructure consisting of a ductile disordered (γ) matrix, which is strengthened by a high volume fraction of ordered (γ') cuboidal precipitates, see Figure 1a. Additionally, the microstructure is further optimized for high-temperature applications by lattice misfit control and eliminating grain boundaries, i.e., by manufacturing single-crystalline components in a favorable crystallographic orientation.

Several strategies have emerged to circumvent the physical limitations of Ni-base superalloys. As these alloys have an excellent mechanical stability at high temperatures and an unparalleled resistance against harsh environmental conditions, one proposed strategy is to strengthen these alloys with a phase that is not formed by precipitation, but is introduced directly during synthesis, such as extrinsic oxide particles, remaining insoluble at elevated temperatures [1, 2]. This approach has recently attracted tremendous research interests. Still, the materials remain intrinsically restricted by their melting ranges.

An alternative strategy is to use a base element other than Ni, with a higher solidus temperature. Examples include refractory elements or their combinations [3]. These metallic elements from groups four to six in the periodic table possess melting temperatures beyond 2000 °C. The mobility of atoms at a certain absolute temperature is thus expected to be lower than in Ni and other late-transition-metal-based alloys with solidus temperatures below 1500 °C. This, in turn, limits diffusion-controlled creep deformation and improves microstructural stability. However, substantial barriers must be overcome in alloy design when relying on refractory metals. In the past, two of these barriers appeared insurmountable: (i) catastrophic oxidation at relatively low temperatures and (ii) a lack of ductility at room temperature. However, the advent of the high-entropy alloy concept has been a game changer, and it is now possible to design a concentrated alloy that is oxidation resistant and ductile.

Indeed, an outstanding oxidation resistance was obtained in concentrated, near equimolar Ta-Mo-Ti-Cr-Al alloys even at temperatures beyond the current capability limits of Ni-based superalloys, owing to the formation of complex



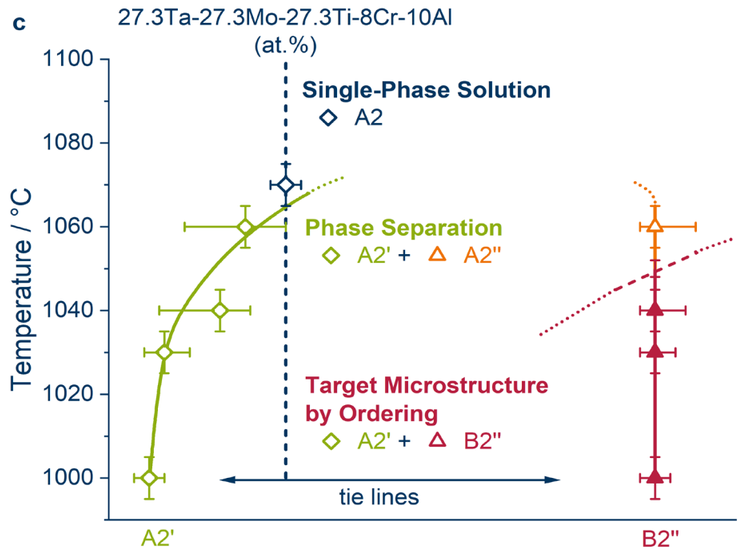


Figure 1: Microstructures of high temperature materials: (a) scanning transmission electron micrograph of a single-crystalline Ni-based superalloys CMSX-4 from Ref. [17], (b) scanning electron micrograph of a two-phase A2+B2 refractory-element-based Ta-Mo-Ti-Cr-Al alloy from Ref. [12]. (c) Semi-quantitative illustration of phase equilibria in the alloy from (b) collected from data in Refs. [12, 16].

rutile-type $(Cr,Ti,Ta)O_2$ scales [4]. However, detailed investigations revealed the existence of long-range order in these alloys [5], compromising the alloy's ability to plastically deform [6]. While disordered alloys composed of elements from groups four (Ti, Zr, Hf) and five (V, Nb, Ta) [7, 8] were confirmed to be ductile, their microstructural stability [9, 10] and creep resistance turned out to be not competitive [11]. An alternative approach is thus required.

In this context, we mimic the microstructure of Ni-based superalloys, with a disordered matrix reinforced by ordered precipitates, but change the base composition from Ni-rich to a concentrated mixture of refractory elements, thereby also modifying the lattice from face-centered cubic to body-centered cubic. The considered concentrated refractory alloys exhibit a disordered matrix (Strukturbericht designation A2) that is reinforced by finely dispersed, ordered precipitates (Strukturbericht designation B2), which provide a competitive creep resistance even with polycrystalline microstructure [12]. The formation of the B2 phase is typically achieved by alloying Al [13], which concentration needs to be finely tuned to establish oxidation resistance [14]. While the binary Ni–Al phase diagram, which includes the precipitation of the γ' phase (Ni_3Al) in the γ matrix (Ni-rich solid solution), serves as a simple reference system for Ni-base superalloys, there is no such simple base system with an A2+B2 two-phase region for designing refractory-element-based alloys. Extrapolation of thermodynamic data into unexplored composition regions is therefore required. Based on rigorous application of such thermodynamic calculations, model alloys were successfully developed [15] that exhibit the desired microstructures, e.g., 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al. The microstructure of this alloy does not only follow the desired A2+B2 template, see Figure 1b, but is also formed by a precipitation process, according to the semi-quantitative phase diagram in Figure 1c, with crystallographically coherent and chemically sharp interfaces exactly like in Ni-base superalloys [16]. Current research activities concern the identification of deformation mechanisms at low and high temperatures, the dislocation-precipitate interactions in such alloys as well as the tuning of the lattice misfit, oxidation response and microstructural design.

In essence, these new high temperature material candidates display an exciting development with largely unexplored design potential. While classical concepts of microstructure optimization, evolution and microstructure-property relationships might be transferred to another base crystal structure, fundamental theories of phase formation, deformation and precipitation strengthening must be re-evaluated and extended to capture common and unique features among the alloy classes.

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FROM **ICE** TO **MAGNETS**

Exploring phase transitions in modern magnetic materials

The discovery of novel magnetic materials continues to drive progress in condensed matter physics by revealing emergent phases and enabling new functionalities, particularly in the understanding and control of novel phases. Beyond their fundamental significance, such systems provide insight into longstanding problems while opening pathways toward technologically relevant applications. For example, unconventional superconductors, including cuprates [1] and iron-based compounds [2, 3], have broadened our understanding of superconductivity, as they frequently also exhibit complex magnetic phases and show potential for elevated transition temperatures.

But what exactly is the phase transition? A helpful way to approach this question is through a familiar example: water. Ice melting into liquid or water boiling into steam are everyday transitions between different states of matter. These changes may seem simple, but they reflect physical principles about how atoms and molecules organize themselves. In a solid, particles are arranged in an ordered structure; in a liquid, that order is partially lost; and in a gas, particles move freely with almost no correlation, as depicted in the inset boxes of the Figure 1. These different states- or phases- are separated by sharp boundaries where the material undergoes a sudden transformation.

Modern condensed matter physics extends this concept to far more complex systems. In magnetic materials, the focus shifts from atomic positions to magnetic moments, or “spins,” and how they organize collectively in a lattice. Phase transitions in these systems describe how magnetic order emerges, evolves, and eventually disappears under the influence of temperature, applied magnetic fields, lattice dimensionality, and competing interactions.

Our research group focuses on the physics of modern magnetic materials, where strong interactions between electrons lead to complex and often emergent behavior. These systems include unconventional superconductors, frustrated magnets, Mott insulators, and quantum spin liquids. We investigate how magnetic order, such as ferromagnetism and antiferromagnetism, develops, evolves, and ultimately breaks down, giving way to novel quantum states. Particular emphasis is placed on regimes where magnetism is suppressed, as in quantum spin liquids or superconducting phases, revealing the underlying quantum fluctuations that govern these transitions. Our approach combines the growth of high-quality single crystals with precise calorimetric, spectroscopic, and transport measurements, which are conducted under extreme conditions, including low temperatures and high magnetic fields, where new and exotic states of matter can emerge. For example, Figure 2 represents the single crystal of $GdCuAs_2$, which exhibits antiferromagnetic ordering at T_{N1} = 10.6 K, followed by an incommensurate-to-commensurate antiferromagnetic transition at T_{N1} = 6 K. These transitions have been observed in resistivity and magnetization measurements [4]. Furthermore, Figure 3 also represents the as-grown single crystal of the quasi-low-dimensional spin-chain compound Li_2CuO_2, along with its crystal structure, followed by confirmation of an antiferromagnetic transition at T_N = 9.3 K through magnetic susceptibility (χ) and heat capacity measurements [5].

In addition to leveraging state-of-the-art research facilities, our group is actively developing and extending experimental probes to accurately determine structural phase transitions, lattice distortions, and elastic properties of novel materials. This includes high-precision, non-destructive techniques such as resonance ultrasound spectroscopy and dilatometry, which are complementary methods for studying phase transitions in novel materials.

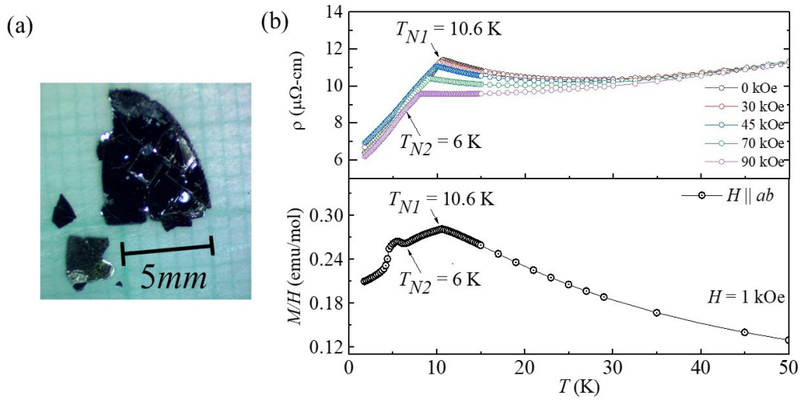

Figure 2: (a) Single-crystal image of $GdCuAs_2$. (b) Top panel: Temperature dependence of electrical resistivity under different magnetic fields, showing antiferromagnetic transitions at T_{N1} and T_{N2}. Bottom panel: Temperature dependence of magnetization for fields applied parallel to the ab plane also showing these phase transitions in $GdCuAs_2$ [4].

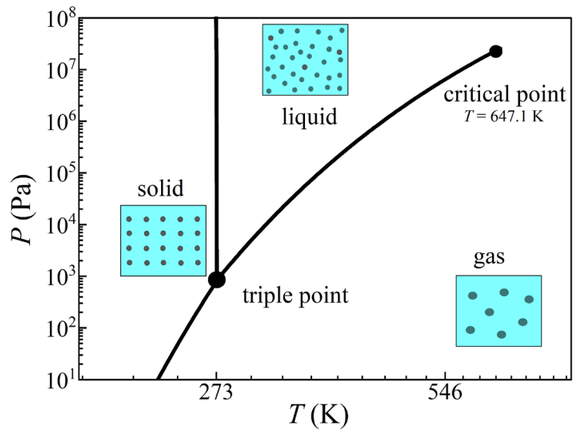

Figure 1: Water phase diagram showing solid, liquid, and gas regions as functions of temperature and pressure, including the triple point and critical point, with particle illustrations for each phase.

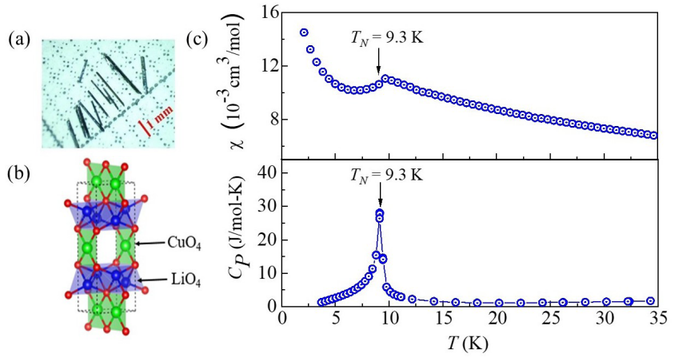

Figure 3: (a) Optical image of single crystals of Li_2CuO_2. (b) Crystal structure of Li_2CuO_2. (c) Top panel: Temperature dependence of magnetic susceptibility, χ with an antiferromagnetic transition at T_N = 9.3 K. Bottom panel: Heat capacity, *Cp*, versus temperature, confirming the magnetic ordering transition [5].

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UNRAVELING **PHASE TRANSITIONS** IN METAL **HYDRIDES**

Atomic-scale insights into hydrogen ordering

Provoking or suppressing phase transitions in metals is an established strategy to control the material performance. In many cases, however, these transformations do not manifest as obvious structural changes but instead proceed through subtle atomic-scale processes, such as the occupation of interstitial sites. A particularly important example is the formation of metal hydrides, where hydrogen incorporation induces above a certain threshold a phase transition within a metallic host. These transformations are central to challenges ranging from hydrogen embrittlement [1] to the development of solid-state hydrogen storage systems [2].

Hydride precipitation in metals exemplifies a diffusion-driven phase transition: hydrogen atoms, owing to their small size and high mobility, diffuse rapidly through the metallic lattice and occupy interstitial sites. However, their solubility in many metals is low, promoting the formation of hydride precipitates. In titanium, a model system for such studies, hydrogen uptake leads to the formation of several hydride phases, including γ-TiH and ε-TiH_2 (see Figure 1a). These phases are distinguished by ordering phenomena, specifically, how hydrogen atoms arrange within available interstitial sites. Understanding how hydrogen distributes within hydrides, particularly at interfaces between metal and hydride phases, is key to tune the structure-property relationship in material systems involving metals and hydrogen.

Despite its importance, probing a light element such as hydrogen in metals remains experimentally challenging. In the last years, advanced electron microscopy and spectroscopy techniques have therefore become powerful tools. In particular, Scanning Transmission Electron Microscopy (STEM) combined with Electron Energy Loss Spectroscopy (EELS) enables the investigation of hydrogen through its influence on plasmons (oscillation of free electrons) and phonons (quantized lattice vibrations). In our ongoing study (publication in preparation), we combine these advanced techniques to probe hydrogen ordering within hydride precipitates. Figure 1b shows a STEM image of a typical Ti-hydride precipitate within the Ti-matrix. The high-resolution STEM image in Figure 1c shows the metal/metal-hydride interface.

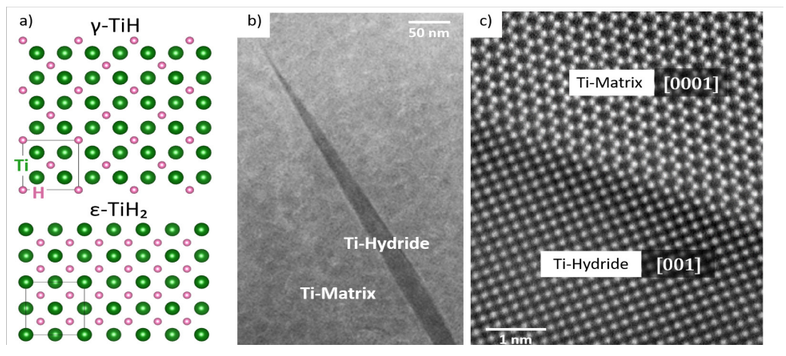

Figure 1: Microscopy and spectroscopy study of the Ti-H sample system. a) Crystal structure of the two face-centered tetragonal phases γ-TiH and ε-TiH_2 in [001] orientation. b) Overview STEM image of a hydride precipitate within the Ti-Matrix. c) High-resolution STEM image of the metal/metal-hydride interface.

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The precipitate is oriented in [001] with respect to the Ti-matrix (hexagonal) [0001], which is promoted by the small mismatch of their interplanar spacing in that orientation [3].

Electron energy loss spectroscopy (EELS), which measures the energy loss that electrons of an electron beam experience when traveling through a sample, is sensitive to changes in valence electron density. In metal–hydride systems, this allows hydrogen to be probed indirectly through its influence on the electronic structure of the host lattice. In metal hydrides, hydrogen donates electrons to the host lattice, leading to a measurable shift in the plasmon peak [4]. In titanium, the plasmon peak shifts from approximately 17.4 eV in pure Ti to higher energies with increasing hydrogen content, reaching values characteristic of specific hydride phases (19-22 eV). In our samples, we observed slight upward shifts near the hydride, indicating the presence of solute hydrogen in the titanium lattice, and consistent shifts within the hydride precipitates corresponding to the γ-TiH phase.

While plasmon analysis already gives an idea about the hydrogen content within the precipitate, it is only sensitive to electron oscillations and not the hydrogen atom itself. Beyond plasmons, the low-loss regime of EELS allows probing of phonon vibrations, as well. Advances in EELS setups have enabled energy resolutions in the range of a few meV, making it possible to probe vibrational modes in the vicinity of the zero-loss peak [5]. While we could not observe differences in the plasmon peak within the hydride phase, however, the spectra obtained in the low-loss regime for phonon vibrations shows variations. We observe distinct phonon features around 150 meV, corresponding to hydrogen–hydrogen vibrational modes (see Figure 2a). In combination with high spatial resolution, using the SuperSTEM 3 Nion microscope, even localized new techniques are feasible [6] which enables us to observe distinct variations of the peak features closer to the metal/metal-hydride interface compared to the center of the precipitate. To interpret these observations, density functional theory calculations provide crucial insight. They show that even subtle changes in hydrogen configuration, such as the presence of point defects or partial occupancy of interstitial sites, can significantly alter phonon spectra. These effects are particularly pronounced in phases like γ-TiH, where only a subset of available interstitial sites is occupied, making the system sensitive to local fluctuations. Complementary 4D-STEM measurements allow us to correlate these spectroscopic observations with structural information (see Figure 2b). By recording diffraction patterns at each scan position, we could detect subtle differences in peak intensities associated with the TiH phase, providing further evidence for local variations in hydrogen ordering.

Together, these complementary techniques provide a coherent picture: hydride phase formation is not a homogeneous (i.e. spontaneous) phase transition, but a spatially complex process in which hydrogen distribution varies across precipitates. While no single method can unambiguously resolve hydrogen ordering in these systems, the combination of electron spectroscopy and advanced microscopy offers a detailed and internally consistent view of how hydrogen is distributed within the metallic lattices and the precipitates. These insights provide a foundation for tailoring hydrogen storage materials, where controlling atomic-scale ordering and interface properties is essential for improving performance.

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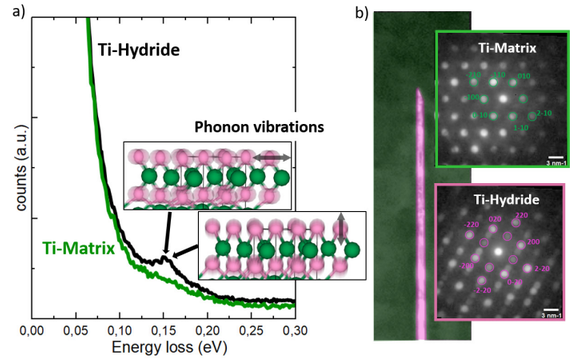

Figure 2: a) Comparison of two EELS spectra (Ti-Matrix vs. Ti-Hydride) for the low-loss regime. H-H phonon vibrations at 35 THz (in ab-plane) and at 39 THz (in c-direction).
b) 4DSTEM analysis showing exemplary diffraction pattern of the Ti-Matrix [0001] and the Ti-Hydride [001].

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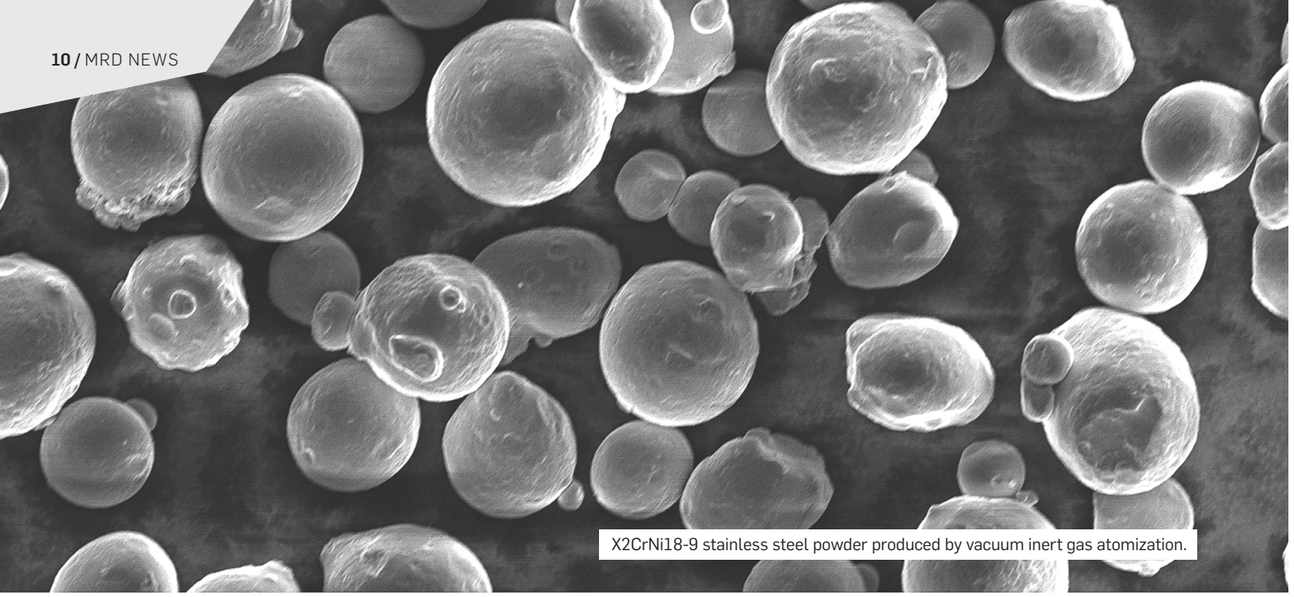

X2CrNi18-9 stainless steel powder produced by vacuum inert gas atomization.

CHEMICAL **HOMOGENEITY** AS A KEY FACTOR IN THE **MARTENSITIC** TRANSFORMATION OF METASTABLE AUSTENITIC STAINLESS **STEELS**

Insights and perspectives on phase stability and transformation behavior

Phase transitions are often associated with familiar changes of state, such as liquid turning into solid or gas. In structural alloys, however, equally important phase transitions occur entirely within the solid state. In austenitic stainless steels, the transformation of austenite to martensite is one such example, with far-reaching consequences for material behavior.

This is particularly evident in demanding environments such as future hydrogen infrastructure, where phase transformation becomes a critical factor in determining material reliability. Austenitic stainless steels such as X2CrNiMo18-14-3 (DIN EN 1.4435) are widely used in hydrogen related structural applications because of their high ductility and comparatively low hydrogen diffusivity. However, the situation changes markedly when the face-centered cubic (fcc) austenite transforms into body-centered cubic (bcc) martensite, for example during plastic deformation. Owing to its bcc crystal structure, martensite is considerably more susceptible to hydrogen-assisted damage and, because hydrogen diffuses much faster in this phase, it also promotes rapid hydrogen transport toward stress-concentrated regions. Understanding which factors promote or suppress this phase transition is therefore not only of scientific interest, but also of major technological importance.

This is especially important in metastable austenitic stainless steels such as X2CrNi18-9, which contain less Ni than X2CrNiMo18-14-3. In these alloys, austenite stability is not determined by the nominal chemical composition. Two materials with the same average chromium and nickel contents can behave very differently if those elements are not distributed equally throughout the microstructure [1]. Local nickel-depleted regions, for example, are less stable than the surrounding matrix and can act as preferred nucleation sites for deformation-induced martensite. Once transformation starts in such regions, it can spread further into neighboring areas, even where the austenite is intrinsically more stable. In this sense, phase transformation is not just a thermodynamic event; it is also a locally triggered and spatially evolving microstructural process [1, 2].

This is where processing becomes decisive. Conventional casting and hot forming tend to leave behind mesoscale segregations inherited from solidification. These segregations often appear as elongated bands enriched or depleted in alloying elements such as nickel and chromium. As a result, the microstructure becomes chemically heterogeneous, leading to local variations in phase stability, as schematically illustrated in Figure 1. Under mechanical loading, especially at low temperature, these inhomogeneities provide ideal starting points for martensitic transformation.

Powder-metallurgical processing routes offer a fundamentally different starting point. In atomized powder, solidification occurs so rapidly that the large-scale segregation typical of cast materials is largely suppressed. When such powder is consolidated by hot isostatic pressing, the resulting steel can exhibit much higher chemical homogeneity on the mesoscale (see Figure 1). This means that the local stability of austenite is more uniform across the material. Consequently, there are fewer chemically weak regions in which martensite can nucleate easily [2].

Mechanical testing under low-temperature tensile loading reveals how important this difference can be. Chemically heterogeneous, conventionally processed material shows a strong tendency for deformation-induced martensitic transformation and pronounced work hardening associated with transformation-induced plasticity [1, 2]. By contrast, hot-isostatically pressed material retains much more austenite under the same loading conditions and deform more uniformly [1]. The perspective is that the hot-isostatically pressed condition exhibits a higher resistance to hydrogen embrittlement, as schematically illustrated by the stress-strain curves in Figure 1.

For materials researchers, this offers an important perspective. We often discuss phase stability in terms of global composition or established empirical parameters, but local fluctuations can be just as decisive. A steel may appear sufficiently stable on average, while still containing small regions that trigger transformation much earlier than expected. Once these regions are activated, the resulting phase evolution can dominate macroscopic behavior.

The broader message is clear: chemical homogeneity should be regarded as an important parameter. It influences where phase transitions are initiated, how they propagate, and how strongly they affect material performance. This is particularly relevant for alloys intended for hydrogen-related applications, where suppressing martensite formation can significantly improve resistance to embrittlement.

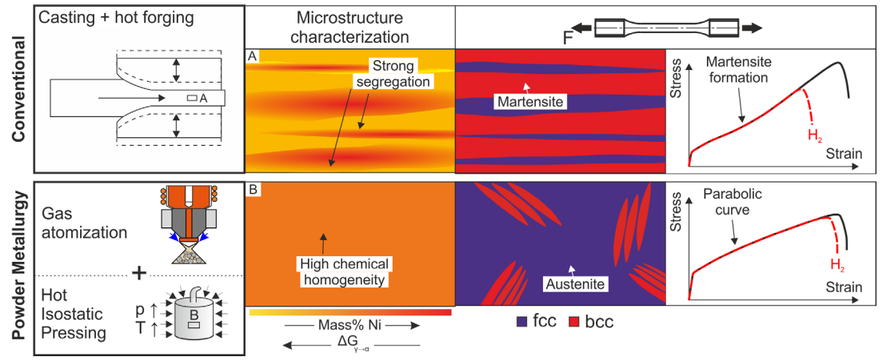

Figure 1: Schematic illustration of the influence of the processing route, conventional versus powder metallurgical, on chemical homogeneity and, consequently, on the transformation behavior during mechanical loading. The figure further highlights the perspective that higher chemical homogeneity reduces the tendency for martensite formation and thereby lowers susceptibility to hydrogen embrittlement.

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ATOMIC-SCALE CONTROL OF COMPETING **ELECTRONIC PHASES** IN 2D MATERIALS

Phase engineering of molybdenum disulfide

Fabricating cutting-edge semiconductor devices and materials requires manipulation at an atomic level, calling for completely new, disruptive manufacturing processes. This is particularly true when it comes to transferring this new type of electronics to industrial production.

Transition-metal dichalcogenides provide in this context significant advantages over conventional semiconductors. Their high carrier mobility, thickness-dependent band gap and strong anisotropic material properties make them ideal for miniaturization. Meanwhile, the polymorphism presents new opportunities for engineering emergent properties and devices. The concept of phase engineering in two-dimensional (2D) materials has emerged as a powerful way to overcome the limitations of conventional semiconductor scaling and enable new device functionalities that go beyond the CMOS paradigm. Significant advantages in terms of low-resistance contacts, reconfigurable device architecture and monolithic function integration in electromechanical devices can be demonstrated.

Molybdenum disulfide for example is the most researched 2D material after graphene and has three stable or metastable phases: the semiconducting trigonal prismatic 2H phase; the metallic octahedral 1T phase; and the less researched 3R phase.

The synthesis of single- and few-layered 2D materials on a wafer-scale is an essential first step in investigating layer-dependent property changes and providing pathways for large-scale applications. Examples of 2D fabrication methods include hydrothermal self-assembly and chemical vapor deposition. Recently, however, (plasma-assisted) atomic layer deposition has also come into the focus of research, as it allows for the highest level of layer thickness control due to surface limitation, enables direct processing on flexible substrates thanks to low process temperatures, and allows for highly conformal deposition on 3D substrates.

These films are polycrystalline, and Raman spectroscopy reveals the semiconducting 2H-phase across the entire 200 mm wafer [1] (Figure 1). Careful adjustment of the process parameters enables the crystallinity and homogeneity to be adjusted and controlled according to the desired application. The deposited 2D films are then comprehensive characterized using advanced materials characterization techniques that include X-ray diffraction, scanning electron microscopy (SEM), atomic force microscopy (AFM), Raman spectroscopy, transition electron microscopy (TEM), Rutherford backscattering spectroscopy (RBS), ellipsometry and IV characteristics (Figure 1).

In general, various phase manipulation strategies can be employed to alter the phase across the entire wafer. To date, these have been studied primarily on monocrystalline layers. Thereby an effective method of inducing phase transformations in MoS_2 involves alkali ion intercalation, which means inserting alkali atoms or ions between the layers of layered materials.

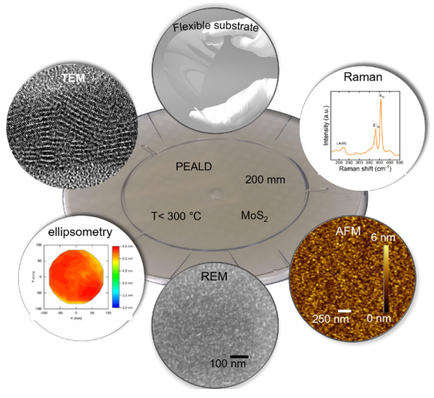


Figure 1: Initial 2D material film on a 200 mm wafer and comprehensive film characterisation.

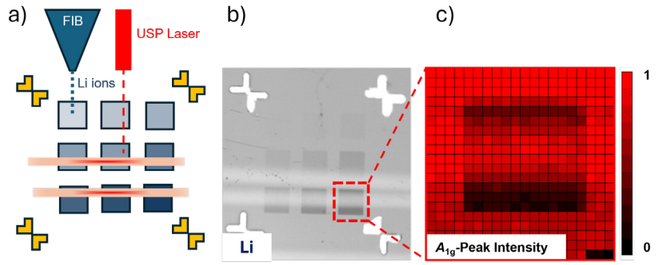


Figure 2: (a) Schematic representation and (b) optical micrograph of a 150 µm × 150 µm writing area, in which 20 µm × 20 µm squares were written using a lithium-ion beam. The fluence increases from 10^{13} cm^{-2} to 10^{16} cm^{-2} from top left to bottom right. Subsequently, two lines were written using a USP laser beam. A very strong Raman signal from the 2H phase is observed there, whereas it disappears completely in the area written by FIB alone (black areas in subfigure c).

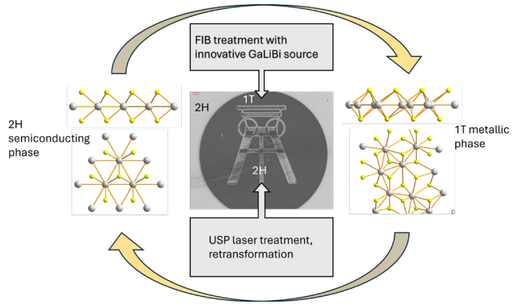
Various intercalation strategies have been developed, including chemical intercalation from the gas or liquid phase, and electrochemical intercalation using solid or liquid electrolytes. However, manipulating their correlated electronic states spatially resolved is rather unexplored especially on atomic layer deposited 2D materials.

In cooperation with RAITH GmbH, we focus on new phase engineering concepts offering the highest levels of lateral and fluence control. This is achieved using a focused ion beam (FIB) equipped with an innovative multi-species ion source consisting of a eutectic alloy containing lithium (Li), gallium (Ga) and bismuth (Bi) [2]. Compared to solution-based chemical alkali metal intercalation, this direct writing method exhibits significantly lower risks of excess Li contaminating clean room facilities. By selecting appropriate process parameters, it is possible to remain well below the ablation threshold of MoS_2, preserve the layered structure of the 2D material, and eliminate signatures of the 2H phase from the Raman signal [5]. Laser processing using ultrashort-pulse lasers can also offer a promising, liquid-free alternative to conventional methods of nanofabrication. It is a modern, sustainable process for surface modification and patterning. Even for ultrathin MoS_2 films deposited by PEALD, laser radiation could be used to ablate, anneal, or recrystallize the 2D material, or to generate laser induced periodic surface structures [3, 4]. Here the USP laser is used as a powerful method for controlling the conversion of metastable metallic phase into the semiconducting 2H. The E^1_{2g} and A_{1g} peaks of the initial MoS_2 film, which vanished after FIB laser treatment, were successfully recovered after USP laser irradiation, indicating retransformation into the semiconducting 2H phase (Figure 2).

The capabilities of this innovative phase-control technology in two-dimensional materials can finally be successfully demonstrated by using a USP laser to inscribe a typical Ruhr region headframe motif within a circle that has previously been treated with an FIB Li ion beam (Figure 3).

As a mask-free and wafer-compatible technique, FIB and USP laser processing connects laboratory-scale phase engineering with scalable 2D electronics. This establishes phase control as a design parameter for next-generation electronic systems.

Figure 3: Schematic illustration of the technology for the spatial transformation of a MoS_2 film produced by atomic layer deposition (ALD). A Li beam from a focused ion beam (FIB) with a liquid GaLiBi source is used to transform the originally semiconducting 2H phase of the MoS_2 film (the light grey outer region in the optical microscope image) into the metallic phase (the dark grey circle in the central optical microscope image). Subsequently, a headframe is written into the FIB-treated area using a USP laser beam, triggering a retransformation to the semiconducting 2H phase. This phase then appears light grey again in the optical microscope image.



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HYDROGEN IN COMPOSITIONALLY **COMPLEX ALLOYS**

Stability and hydride formation under high pressure and high temperature

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Compositionally complex alloys, often referred to as high-entropy alloys, are promising candidates for advanced and sustainable technologies. Compositionally complex alloys contain multiple alloying elements in chemically complex local environments, which can strongly influence their structure, stability, and functional properties. Although first studied mainly for structural applications, these materials are now being explored for functional and energy-related uses, including heterogeneous catalysis and hydrogen storage.

In our recent studies, we focus on single-phase, well-characterized compositionally complex alloys as model systems for hydrogen-related applications. Recently, we investigated the Cantor alloy, a single-phase *fcc*–structured CoCrFeNiMn alloy, under high-pressure, high-temperature conditions to examine its stability and behaviour in extreme environments as well as under a hydrogen atmosphere. The Cantor alloy is among the most widely studied compositionally complex alloys. Using hydrogen as a reactive gas at elevated pressure and temperature, we explored the alloy's resistance to hydrogen uptake and identified the conditions under which this resistance breaks down, leading to the formation of a compositionally complex hydride (we also call it high-entropy hydride).

To access these regimes, we employed diamond anvil cells and large-volume presses combined with modern characterization techniques. These methods allow us to reach extreme conditions and to explore the stability fields of high-entropy hydrides. The use of hydrogen as a pressure-transmitting medium and reactive gas enables the study of reactions between solids and hydrogen under extreme conditions, at pressures above 50 GPa at room temperature (Figure 1). In addition, external resistive heating helps to accelerate kinetics and to probe the limits of hydrogen uptake at elevated temperatures, which are relevant for industrial applications and beyond. Our results show that the Cantor alloy represents a remarkable example within the family of compositionally complex alloys. At room temperature, it does not absorb hydrogen even at pressures above 40 GPa. This unusual resistance to hydrogen uptake points to potential applications as a hydrogen-resistant material [1], with an exceptional stability comparable to that of conventional Cu–Be alloys. At the same time, our results show that this resistance is not unlimited. Although the Cantor alloy remains resilient to hydrogen even at 15 GPa and 100 °C (Figure 2), at higher pressures it forms a *fcc*–structured (CoCrFeNiMn)H high-entropy hydride [2].

This observation is important for several reasons. Most importantly, it represents the first *in situ* observation of a high-entropy hydride derived from the Cantor alloy. It therefore highlights the dual role of this material. On the one hand, it is exceptionally resistant to hydrogen uptake across a wide range of conditions. On the one hand, it is exceptionally resistant to hydrogen uptake across a wide range of conditions. On the other hand, it can transform into a distinct hydride phase

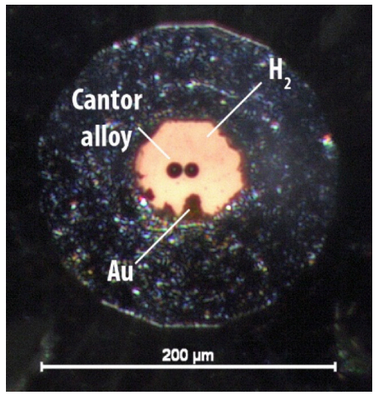

Figure 1: Microphotograph showing two small balls of Cantor alloy loaded in a diamond anvil cell with rhenium gasket and hydrogen as pressure transmitting medium and reactive gas. Au powder has been used as an internal pressure calibrant.

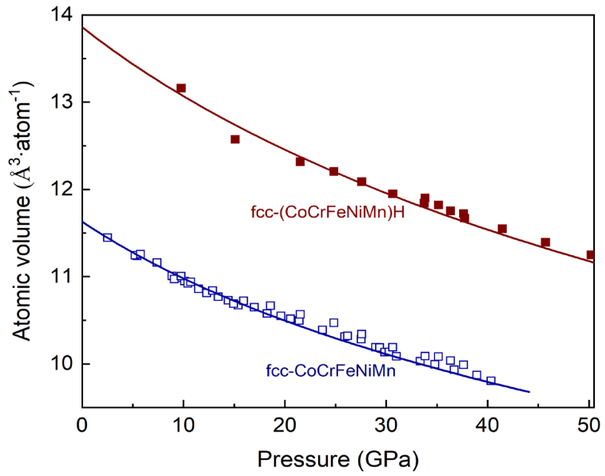

Figure 2: Atomic volumes for Cantor alloy upon compression at room temperature (its compressibility curve below) and after heating showing the formation of *fcc*-structured (CoCrFeNiMn)H high-entropy hydride with much larger atomic volume die to hydrogen uptake (above).

when the thermodynamic driving force becomes sufficiently strong. In this sense, the Cantor alloy provides an especially useful model system for understanding how hydrogen interacts with chemically complex metallic hosts.

Our study also opens the door to the broader field of high-entropy hydrides, which remains comparatively underexplored. Experimental insight in their stability, hydrogen content, and atomic-scale structure remains limited, particularly for hydrides derived from multicomponent transition-metal alloys. In this context, the Cantor alloy offers an important starting point for identifying the conditions under which hydrogen incorporation becomes favourable and for understanding how compositionally complex alloys respond to hydrogen under extreme conditions.

Our experimental findings were supported by density-functional theory (DFT) calculations, which helped explain the relative stabilities of the phases involved. Among other results, we showed that the *fcc*-structured hydride is more stable than its *hcp* counterparts and provided a reasonable estimate of the resulting hydrogen concentrations. In addition, neutron experiments combined with DFT calculations provided important atomic-level insight, revealing that hydrogen occupies the octahedral sites in the resulting *fcc* structure (Figure 3). This structural information is essential for understanding the stability of the hydride phases and the mechanisms of hydrogen incorporation in chemically complex alloys.

Our decompression results further show that hydrogen leaves the bulk of the material upon quenching to lower pressure and under ambient conditions. This finding suggests that hydrogen uptake is reversible under the investigated conditions and provides further insight into the stability limits of the hydride phase. Overall, our work contributes to a better understanding of how compositionally complex alloys interact with hydrogen and helps clarify the conditions under which high-entropy hydrides can form.

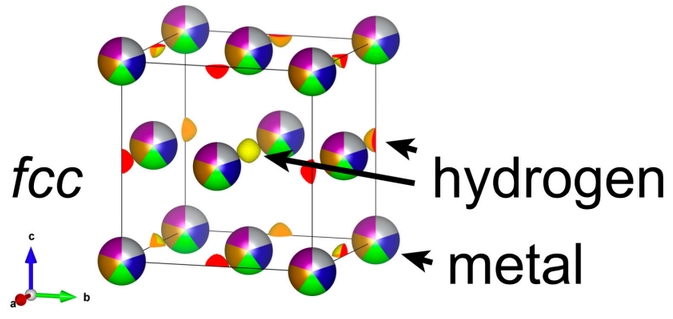

Figure 3: Structural model of the *fcc*–(CoCrFeNiMn)Dx high-entropy hydride obtained from in-situ neutron diffraction study and confirmed by DFT modelling.

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Figure 1: Manufacturing environment used within the research unit FOR 5620.

PHASE TRANSITIONS IN ADDITIVELY MANUFACTURED AUSTENITIC STEELS

Determining local austenite stability

Within the DFG Research Unit FOR 5620 at Ruhr University Bochum, the Chair of Materials Technology (LWT) addresses the materials-related fundamentals of wire-based DED-LB/M for large, load-optimized freeform components. The consortium seeks to establish a robust process chain that combines robot-guided additive deposition with sequential subtractive finishing in a single manufacturing environment. Therefore, a manufacturing environment including two robotic arms mounted to a rail and combined with a tilt-and-turn table is applied (Figure 1). From the perspective of materials technology, the central question in the subproject 5 is how the thermal history of the process influences local phase stability and, with it, the structural integrity and reliability of the final component.

This question is highly relevant for future applications in which large additively manufactured steel components must combine geometric freedom with reproducible performance, for example in corrosive environments or hydrogen-related systems. The investigations focus on corrosion-resistant metastable austenitic steels, especially type 304L (1.4301, X2CrNi18-9), which are technologically relevant and highly suitable model systems for studying microstructure formation during additive manufacturing. However, the additively manufactured state differs fundamentally from conventional hot formed material. Rapid solidification, repeated reheating, and geometry-dependent heat accumulation generate microstructures that are spatially graded, chemically heterogeneous, and crystallographically complex (Figure 2).

At this point, phase transitions become central to the materials-related understanding of the process. In DED-LB/M structures, solidification-induced microsegregation produces local variations in alloy chemistry on the micrometer scale. These variations directly influence the local stability of austenite. In destabilized regions, deformation can trigger martensitic transformation, producing harder but less ductile constituents. Such local phase changes are not merely of academic interest: they may alter crack initiation behavior, reduce corrosion resistance, and increase susceptibility to hydrogen-embrittlement. The performance of a large additively manufactured component may therefore depend on transformations that occur only in small, locally distinct regions.

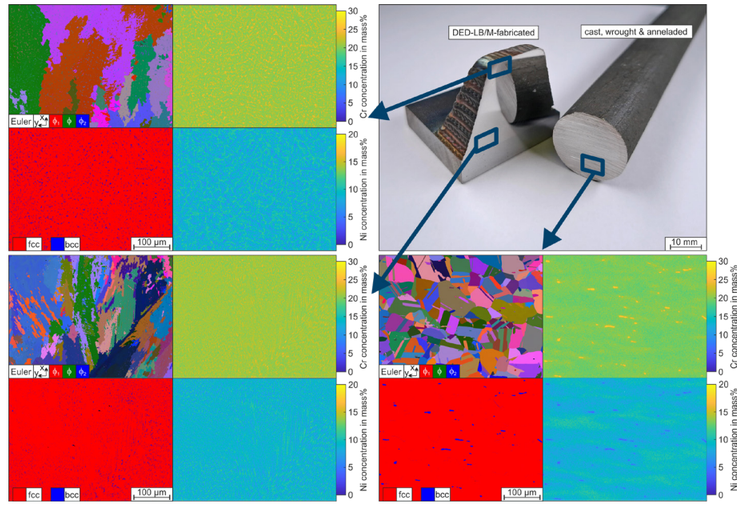

Figure 2: Microstructures emerging from DED-LB/M-processing and conventional processing of 304L.

A particular challenge in wire-based DED-LB/M is that these mechanisms are strongly coupled to part geometry. Heat dissipation near the building platform or the substrate material is fundamentally different from that in regions with progressive thermal accumulation, for example near the top of a tapered structure. To study this systematically, model geometries such as pyramid-shaped samples are employed, intentionally promoting increasing heat build-up with increasing build-up height.

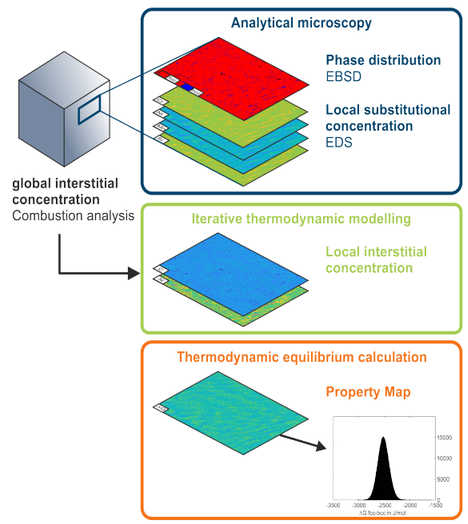

Figure 3: Approach for determining local segregation-dependent austenite stability.

Initial results reveal clear differences in grain structure, microsegregation, and ferrite content along the build direction. The results also show that conventional solution annealing, which is intended to reduce segregation and eliminate ferrite, may not be effective locally due to geometry-related variations in solidification conditions.

Compared with hot formed and annealed reference material, DED-LB/M-processed 304L exhibits coarser and more elongated grains on the one hand, while on the other hand the segregation pattern is finer while it is also varied with respect to the geometry-dependent solidification conditions. These distinctions help explain why phase stability, transformation behavior, and damage tolerance may differ even when nominal alloy chemistry remains unchanged. In the course of the project, advanced materials characterization including analytical microscopy coupled with thermodynamic calculation of property maps will be linked more closely with process control concepts in DED-LB/M developed by the project partners to move from observing heterogeneous microstructures toward actively engineering them (Figure 3). In parallel, the materials portfolio is being extended beyond classical CrNi steels to alternative systems such as Ni-free CrMnCN steels which in contrast exhibit intrinsically stable austenite at room temperature. For the MRD community, phase transitions in additively manufactured steels are therefore not merely a metallurgical detail, but a decisive factor in process reliability, component integrity, and the future industrial use of large-scale metal additive manufacturing.

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A **DIFFERENT** KIND OF **TRANSITION**

Materials Research Department becomes a Science Hub at the Ruhr-Universität Bochum

Earlier this year, the Materials Research Department was confirmed for a new five-year funding phase running from 2026 to 2030. Alongside four other science and humanities hubs, the MRD has been designated a Science Hub of the Ruhr-Universität Bochum, a structure introduced by the Rectorate to foster interdisciplinary, cutting-edge research across the university.

As part of the key research area “Matter, Materials and Energy”, the MRD contributes to investigating the fundamental properties of complex materials with a focus on sustainable technologies for the future.

This funding renewal follows an evaluation of the 2021–2024 period, which assessed members’ research, publications, collaborations, patents, transfer activities and internal MRD initiatives. The evaluation concluded with overwhelmingly positive feedback, reaffirming the MRD’s role in establishing materials science as a core focus area at Ruhr-Universität Bochum.

Looking ahead, the new funding phase sets out a series of targets for research excellence, support for early career researchers, internationalization, equal opportunities and knowledge transfer. To reach these goals, we rely on the continued success, innovative ideas and active involvement of the entire MRD community.

As we transition into this next chapter, we also take the opportunity to look back at important activities from previous years that reflect the commitment and collaborative work of our members.

The continuous growth of the MRD has strengthened the network for collaborative and interdisciplinary research. Today, MRD counts 88 members from the RUB, MPI SusMat, DLR and Forschungszentrum Jülich. Shown here are the members admitted at the assembly in November 2025.

MRD members contributed to the excellence strategy application of the RUB and TU Dortumnd „Unprecedented functionalities: Driving the AI revolution for mastering complexity of materials interfaces”.

The Early Career Researchers Network brings together more than 100 members from chairs affiliated with the MRD. The four ECR representatives organise the annual ECR Day in different formats (pictures from 2023, 2024, 2025).

At the MRD Innovation Day start-ups, industry representatives and university researchers come together to show how academic research can be translated into innovation (2024).

Established in 2024, the Science Slam for Materials Researchers challenges early career materials scientists to communicate their research in innovative and engaging ways.

Visit our LinkedIn page for updates on our activities.

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