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Editorial

Metals Challenged by Neutron and Synchrotron Radiation

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1. Introduction and Scope

In the past one and a half decades, neutron and synchrotron radiation techniques have come to the forefront as an excellent set of tools for the wider investigation of material structures and properties [1,2], becoming available to a large user community. This holds especially true for metals, which are a fascinating class of materials with both structural and functional applications. With respect to these application classes, metals are used to engineer bridges and automotive engines as well as to exploit magnetic and electric properties in computer storage, optics, and electronics. Both neutron sources and synchrotrons are large user facilities of quantum-beam installations [3] with the implementation of a common accelerator or nuclear reactor-based source, often serving over 50 beamlines simultaneously and even more end stations. Up to a few thousand experiments are undertaken yearly, utilizing specialized beam conditions, sample environments, and detection systems. Their variations range across spectroscopy, diffraction, small-angle scattering, and inelastic scattering for sample sizes ranging from nanometers to meters. Examples of such installations can be found in the Topical Collection *Facilities of Metals'* sister journal *Quantum Beam Science* [3].

The scope of the present Special Issue in *Metals* comprises articles on research case studies on individually selected systems. Fields of interest range from engineering, through materials design, to fundamental materials science, including non-exclusively, strain scanning, texture analysis, phase transformation, precipitation, microstructure reconstruction, crystal defects, atomic structures (both crystalline and amorphous), order and disorder, kinetics, time-resolved microstructure evolution, local structure correlations, phonons, deformation and transformation mechanisms, response to extreme conditions, local and integrated studies, both within the bulk and at interfaces. Regarding the breadth of the discipline, this contribution is not exhaustive by far, but stimulates important and evolving studies throughout the metals community.

2. Contributions

Twenty articles have been published in the present Special Issue of *Metals*, encompassing the fields of sintering techniques, titanium aluminides and titanium alloys, metallic glass and disorder in crystals, and thin layers and interfaces. This grouping is not hard-bound, and thematic links can be established beyond them, which shall be emphasized in the following presentation.

2.1. Sintering Techniques and Microstructure Evolution

In situ investigations for following morphology and phase constitution in sintering and annealing processes have been enabled by the high flux of modern neutron and X-ray beams. While wide-angle diffraction allows for the recording of the time-resolved crystallographic structural

evolution, small-angle scattering determines precipitations on the nanometer scale, complemented by tomographic imaging into micrometer-sized features.

Such in situ phase evolutions are presented by Chen et al. upon vacuum sintering of Ni-Ti powder compacts [4]. The authors present neutron diffraction patterns of both Ti-Ni and TiH_2-Ni mixtures taken every 34 s upon a heating ramp to 1373 K, unraveling the appearance and evolution of intermediate and final product phases. Hydrogen and its release not only influence the residual pore size of the product, but also highly affects the intermetallic diffusion and reaction during the sintering process.

Sub-micrometer-resolution synchrotron X-ray computed tomography has been used by both Xiao et al. [5] and Ma et al. [6] for in situ investigations of microwave sintering metals. Xiao et al. [5] work on titanium powder, which was sintered at 1173 K and data recorded in 900 s time steps. The reconstructed data allows for the extraction of phenomena such as particle densification together with neck formation and growth. The objects of Ma et al. [6] were packed stainless-steel fiber felts sintered at 1273 K–1473 K. Similarly, the neck forming between two crossing sintering fibers were evaluated. The data is interpreted analytically with atom migration rates due to atom and vacancy diffusion processes and the surface energy of the neck. Because of the complicated heating process in microwave sintering driven by electro-magnetic wave interaction, anomalous sintering rates result from the observations.

In situ investigations on microstructure and phase evaluations from the bulk of material are most important to develop modern high-strength, transformation-induced plasticity steels. Here, Nishijima et al. [7] present small-angle neutron scattering simultaneously taken with dilatometry in order to determine the precipitation and evolution of ferrite in austenite, after quenching from 1173 K to 573 K and subsequent holding. Furthermore, the quantitative phase evolution is monitored in wide-momentum-transfer neutron diffraction, revealing not only phase fractions but also strain in the lattice parameters.

Such combined techniques open a pathway for new alloy development in a larger sense.

2.2. Titanium Aluminides and Titanium Alloys under Extreme Conditions

Already two of the examples presented in Section 2.1 emphasize the importance of titanium and its alloys [4,5]. Much focus on alloys designed for extreme operating conditions is on the titanium aluminide intermetallics, complemented by a nice feature of a Ti-Zr solid solution.

Neutron and synchrotron in situ characterization methods have contributed tremendously to the development of modern γ-based titanium aluminides, as reviewed by Erdely et al. [8]. The Ti-Al phase diagram containing *fcc*-based γ- and *hcp*-based α_2-phase at operating temperature is not only complex by nature; moreover, alloying is undertaken to provide a ductile, *bcc*-based β-phase at processing temperatures above 1600 K. All phases can be ordered and fully or partially disordered, adding to the complexity, while the prevailing structure strongly influences the mechanical and thermal properties. The very particular neutron scattering behavior of titanium, possessing a negative scattering amplitude—in contrast to most other elements—is uniquely exploited to determine the atomic order in such phases. Superstructure peaks, uncloaking the order, are extremely strong while the main reflections, seen with X-rays, literally disappear, forming a so-called null matrix. Therefore, synchrotron X-ray diffraction can determine the overall crystal structure, and neutron scattering can determine the atomic order therein very sensitively. Similar to the above on steels, Erdely et al. [8] utilize small- and wide-range-scattering to determine in situ fingerprints of microstructure and phase evolution in reciprocal space. State-of-the-art, real-time, in situ studies during plastic deformation at 1573 K, where the morphology of intensity distribution along the two-dimensional Debye-Scherrer rings is streaked in time, reveal crystal deformation, dynamic recovery, and dynamic recrystallization processes. The paper not only reviews many of the pioneering methods cited therein, but also gives comprehensive reference to the thermo-mechanical properties and the development of such intermetallics for industrial application.

A greater emphasis on plasticity is presented by Stark et al. [9], in a particular case study using the abovementioned method to investigate γ-based titanium aluminum alloyed with niobium. In Ti-42Al-8.5Nb (at. %), the ductile β- and potentially ordered β_o-phase is abundant above ~1400 K, evolving even further at higher temperatures. In their experiment, two-dimensional in situ synchrotron high-energy X-ray diffraction is undertaken upon plastic deformation to 40% in 100 s, and is continuously recorded with 4 s time frames. The azimuthal intensity distribution leads to a detailed texture analysis and an illustration of their evolution, revealing intensity fluctuations that indicate dynamic recrystallization. It is revealed that phase composition, recrystallization, and the amount of deformation influence the texture significantly. The study demonstrates the potential of in situ diffraction techniques in a so-called Materials Oscilloscope [10] for the optimization of hot-forming multi-phase alloys.

More often, titanium aluminides are processed under extreme conditions, including high pressure, which gave myself and others, Liss et al. [11], the motivation to investigate a γ-based titanium-aluminide under high pressure and temperature. We used a 15 MN press to impose on a volume containing a 1.5 mm^3 sample inside a heating element while synchrotron X-ray diffractograms were recorded. Pressurizing at room temperature reveals the equation of states, introduction of crystallographic disorder, and continuous phase transformation of γ in favor of α/α_2. Regarding the high ordering energy of γ, this transformation is favored despite increasing the volume per atom, which is counterintuitive under pressure. Heating at 10 GPa to melting reveals shifting of the phase transformation temperatures. Moreover, the β-phase is found at high pressure and temperature in 3- and 2-phase fields. Such a ductile β-phase opens processing windows under high pressure, while it would not be abundant at operating conditions under ambient pressure, reducing problems of creep under constant load.

Related to this topic is the making of a titanium-zirconium null-matrix material, reported by Okuchi et al. [12], which is being developed as a neutron-transparent material for gaskets and high-pressure cells in neutron scattering experiments under extreme conditions, as outlined above. In a null-matrix material, coherent neutron scattering is suppressed by alloying elements of positive and negative scattering amplitude, Zr and Ti, respectively, in a solid solution. The composition is chosen such that the average coherent neutron scattering length, which determines the strength of the Bragg peaks, is equal to zero. The forging method presented here results in finer lamellar microstructures, improving the mechanical properties of the material while still exposing excellent neutron-optical properties.

Interestingly, the scattering process of the titanium-zirconium null-matrix material produced by Okuchi et al. [12] relates to the investigations summarized by Erdely et al. [8], because titanium-aluminides equally form a null-matrix when fully disordered. In their study, the authors use the latter to determine the aforementioned crystallographic order parameter upon disorder, rounding up this section.

2.3. Metallic Glass and Disordered Crystals

Crystallographic disorder, as described in Section 2.2., can be detected by diffuse neutron or X-ray diffraction, which is presented in an article by Goossens [13]. As ordered intermetallics undergo disorder, for example, atoms randomly swap sites and their species on a given lattice site are determined by their random probability as given by their concentration, i.e., they become a solid solution, and their coherent scattering differences contribute to incoherence and scatter into an all solid angle. If there is remaining order, i.e., short-range order existing just over a few unit cells, this diffuse scattering peaks at particular locations in a reciprocal lattice. There exist numerous kinds of disorder, such as anti-site swaps, vacancies, interstitials, spin-orientation, magnetism, two-body correlations, and thermal vibrations—phonons. Such local order and defects occur through a variety of complex metals, including intermetallics, alloys, and quasicrystals, and can strongly influence mechanical as well as functional properties of the material. Detailed modeling of the diffuse scattering can be a challenge,

and is the object of Goossen's contribution [13], realized here by an inverse Monte-Carlo method. Crystal structures and defects are simulated minutely in a computer program and the corresponding diffraction pattern is calculated and iterated by randomization of the defects step by step, until a realistic, averaged diffraction pattern is obtained. The author shows in three-dimensional reciprocal space the response to different defect contributions and crystal symmetries in various configurations.

A further degree of disorder is achieved when atoms do not sit on crystal lattice sites at all, which is the case in amorphous materials, and in the present case of metallic glass. Within the context of X-ray, neutron, or even electron beam diffraction, the concept of Bragg diffraction, based on crystal planes, breaks down. As Bragg diffraction is a discrete Fourier series of an infinite periodic structure, the discrete points Q_i forming the reciprocal lattice, and coefficients expressed by structure factors S_i, amorphous disordered systems have to be treated in a continuous Fourier integral with a continuous scattering function $S(Q)$. The back-transformation from an amorphous diffraction pattern leads to the pair distribution function or radial distribution function, as briefly introduced by Egami et al. [14]. Similarly, electrons scatter from atoms and form diffraction patterns, which can be obtained by measuring the extended X-ray absorption fine structure, known as EXAFS. In this process, electrons are emitted from an element-specific atom by tuning the X-ray energy across the absorption edge, scattering at surrounding atoms, and forming an interference pattern. Similarly, Fourier back-transformation leads to local atom distribution functions.

Emphasis is given not only on the structure, but also on the strain response to elastic and plastic deformation. Suzuki et al. [15] report on elastic deformation mechanisms in Zr-Cu-Al metallic glasses under as-cast and heat-treated conditions. Like in any object under uniaxial tensile loading, atom distances expand in the loading direction and normally contract transversely, reflecting in changes and anisotropy of the diffraction patterns. Peak shifts can be observed and evaluated both in reciprocal space mapping and after back transformation to direct space. A salient feature of this paper is that elastic response, i.e., the Young modulus, depends on the length of the scale observed. It differs for its nearest neighbors, for long-range atomic average, and for macroscopic distances. The latter can contain pseudo-elastic strain, which is localized plastic strain, where atoms in building blocks change their morphology in a reversible manner, leading to a larger overall strain. Diffraction processes detect only elastic strain, such that this pseudo-elastic effect is not seen directly. As the method can distinguish nearest-neighbor distances, second-nearest-neighbors etc., it is sensitive to separating strain response on different length scales. Suzuki et al. [15] apply these aspects to determine the stress behavior in various eutectic, hypoeutectic, and heat-treated glasses, and reveal strongly- and weakly-bonded regions and clusters. By the way of elastically perturbing the system, the authors have shown how to obtain structural information on various length scales, not otherwise possible to reveal from diffraction and macroscopic methods alone.

Likewise, Egami et al. [14] treat the atomic response of metallic glass to perturbations by a uniaxial mechanical load, breaking the symmetry of the system and extracting further information on the amorphous microstructure over length-scales. Non-affine atomic rearrangements below the yield stress have been found to be localized and reversible, leading to the abovementioned pseudo-elasticity. As an analysis method, the authors present the difference pair-distribution function, showing residuals between the loading and transverse direction, and allowing the extraction of the non-affine response of deformation in so-called localized shear-transformation zones. Increasing temperature leads to avalanche collapses of the latter, resulting in creep.

The structure of metallic glass is further discussed by Guo et al., particularly how synchrotron radiation [16] and combined simulations [17] are employed to determine icosahedral and icosahedral-like clusters. Both techniques, diffraction and absorption spectroscopy, EXAFS, are used to index Voronoi clusters in a tessellation. Because the determined popular clusters in Zr-30Pd (at. %) glass share configuration similarities with the Zr_2Pd quasi-crystalline phase, the latter precipitates upon annealing before transformation into a fully crystalline phase.

All contributions lead to the conclusion that a metallic glass is largely made up of various kinds of close-packed building fragments which link together in an amorphous network. There are localized hard centers, with softer regions in-between, which can reversibly deform in a non-affine way and lead to pseudo-elasticity. Diffraction and scattering methods primarily probe for atomic length-scales, but when coupled with response to an applied uniaxial stress, it can deliver representative structural information on larger, intermediate length-scales, which is not otherwise accessible.

2.4. In Situ and Time-Resolved Response to Mechanic Load and Shock

The concept of real-time and in situ studies has already been touched in the previous chapters, where examples of thermally driven reactions and transformations, as well as response to mechanical stress, were investigated. In particular, the brilliance of synchrotron radiation allows one to focus the investigation under highly advanced resolution, be it in time or spatial.

The deformation behavior of individual grains in polycrystalline Cu-Al-Mn super-elastic alloy is presented by Kwon et al. [18], who use a 15 × 15 μm^2 beam of white high-energy X-rays in the transmission of a 200 μm thick tensile specimen with a 400 μm grain size. When scanned over the grains and their boundaries, the recorded white-beam Laue diffraction patterns minutely reveal grain orientation, as well as gradients therein. In particular, streaking of the Laue spots occurs according to the gradients in the illuminated volume. On top of this, the Laue spots have been analyzed by an energy-dispersive detector, altogether resolving locally reciprocal space in three dimensions, namely orientation and depth. Data is taken in situ while the specimen is being loaded, giving local strain feedback to the applied stress. It is found that the resulting strain distribution not only varies from grain to grain, but, furthermore, orientation and lattice-spacing gradients evolve within one grain. Reversible phenomena during super-elastic deformation have been observed and are interpreted by a reversible martensitic transformation. In this way, super-elasticity shows parallels to the non-affine pseudo-elastic deformation described in Section 2.3 on metallic glasses.

Ultra-high time resolution is reviewed by Ichiyanagi et al. [19], investigating the dynamic structural response to shock waves in a laser-pump X-ray-probe setup. High-power lasers are used to induce pressure waves into the 10 GPa range within nanoseconds, synchronized with a given time shift to 100 ps probing X-ray pulses. Repeating the experiment with different time delays results in scanning the temporal evolution of the structural changes. From an X-ray optical point of view, 'pink' beams covering a wide bandwidth are used to match enough reciprocal-space acceptance for given crystal orientations. Similar to the abovementioned Laue method, streaking of the reflection spots determines lattice gradients in a CdS single crystal at each snapshot in time. In contrast to quasi-static compression, the structural phase transition at 3.25 GPa was not observed on nanosecond dynamic loading-unloading, revealing an over-compressed state with an incubation time greater than 10 ns. Furthermore, temporal response is demonstrated for polycrystalline and amorphous materials. Different behavior between single-crystalline and polycrystalline specimens allow for the elucidation of nucleation and growth mechanisms of the pressure-induced phase transformations.

Back to more conventional time-scales, Lee & Huang et al. [20] and Lee & Wang et al. [21] employ neutron scattering on cyclic loading for fatigue testing a crack tip in stainless steel under steady and overloaded fatigue conditions. They also study the twinning-detwinning behavior in magnesium alloy under reversed loading. The crack tip analysis was performed by scanning a 1 mm^2 neutron beam along the crack-propagation direction, revealing internal lattice strains which concentrate at the tip. A comparison of the overloaded with the steady fatigued specimen shows large compressive stresses behind the crack tip, suggesting a mechanism for crack-growth retardation after overloading. The magnesium study considers load partitioning between the different crystallographic grain orientations and texture evolution in Mg-8.5Al (mass %) alloy in four scenarios, compression followed by reverse tension and vice versa, respectively starting with extrusion texture and a reoriented texture. In both cases, extension twinning is activated in the first deformation step, basically flipping the basal pole from transverse to axial in the first case, and axial to transverse in the second case. There

are asymmetries and non-linearities in unloading and reverse deformations, which are discussed in detail with the stress states of the different plane families.

These contributions round up important insights into the potential of in situ deformation studies, comprising lattice-orientation resolved strain and texture. The knowledge, measurements, and modeling of these inherently relate to the mechanical and physical properties of a material.

2.5. Thin Films and Layers

Besides the bulk studies presented here, metals and materials in the form of thin films play a highly important role in modern technology, in the form of functional materials, such as magnetic nano-layers in data storage, catalysts, optical components, and electronic devices, and even beyond hard condensed matter physics in biological systems, such as peptide layers or cell membranes.

Neutron and X-ray reflectometry and derived grazing-incident diffraction are important methods where evanescent waves are used to probe depths into the films from atomic distances to micrometers. When these depths are scanned by varying the incident angle to the surface, or more quantitatively by varying the scattering vector, accurate depth profiles are obtained. Demkowicz and Majewski [22] review the basic scattering theory and concepts behind this method, starting from the very basic concepts of diffraction, the definition of scattering vector, evanescent wave functions, and interaction potential expressed by atomic scattering lengths. Both neutron and X-ray scattering theories are very similar, differing mostly on the different scattering amplitudes, resulting in distinct interaction potentials, which are also expressed in refractive index. First of all, the thickness of the layers leads to thickness fringes, which is an important measure, particularly for in situ studies. For example, in magnetic metallic multilayers, fringes and intensities minutely depend on the spin orientations and help to understand, e.g., exchange couplings through a non-magnetic layer under applied conditions. In addition, the sharpness and the inter-diffusion of atomic layers can be revealed from the overall reflection curves.

A very nice application of the capabilities of neutron and X-ray reflectometry is on hydrogen absorption in metallic thin films and heterostructures, presented by Callori et al. [23]. Safe hydrogen storage not only plays a paramount role in chemical energy storage, but hydrogen can also alter physical properties, such as magnetism and electric conductivity in a functional device. In contrast to characterization by X-rays, neutron scattering is the ultimate probe for atomic structures containing hydrogen. This is because of the large neutron scattering lengths of hydrogen. Moreover, the isotope ^{1}H possesses a negative coherent scattering length of -3.7406 fm while its heavier counterpart deuterium, ^{2}H or D, diffracts with 6.671 fm. Mixing the two in pre-determined concentrations allows for contrast variation, including the null-matrix effect as discussed in Section 2.2 and other contrast matching. It shall be noted that hydrogen also possesses a nuclear spin, which in principle can be polarized for complementary analysis, but is not part of the present study. In contrast, X-ray scattering is poorly sensitive to hydrogen atoms, as they only possess one scattering electron which most often is not even localized. Callori et al. [23] demonstrate that the refractive index varies in the Pd-H system, related to the scattering-length-density changes due to mixing negative H with positive Pd scatterers, reducing the critical angle for total reflection in a reflectivity curve, from which the H concentration is obtained. In metallic crystal lattices, hydrogen diffuses on interstitial sites, which can be barely filled, to be fully occupied in a stoichiometric metal hydride, straining the original metal lattice. The authors present data for several Fe-Nb multilayers before and after hydrogen loading, showing shifts of the thickness fringes due to swelling of the layer thicknesses. On top of this, polarized neutron reflectivity is sensitive to the magnetism in the system carried by the Fe layers, showing that its ferromagnetism is not affected by hydrogen loading the Fe/Nb multilayers. All three effects, namely refractive index, thickness fringes, and magnetic polarization, are obtained simultaneously. The results are further discussed with X-ray diffraction results and various other methods to quantify phase transformations, anisotropic expansion, and magnetism. Such investigations not only play an important role in the microstructure engineering of hydrogen-storage devices, but also for the functional properties of sensors.

Hydrogenated diamond-like carbon film with titanium doping is a lubricant in spacecraft applications, and is investigated by Kidena et al. [24] with respect to resistance to hyperthermal oxygen exposure, as found in the lower orbits around Earth. Thin films of 400 nm have been investigated by a multitude of quantum-beam methods, including Rutherford back-scattering and elastic recoil detection analysis of He, X-ray photo-electron spectroscopy, and synchrotron X-ray absorption fine structure (NEXAFS) measurements. The results of these methods lead to the determination of film thickness, layer profile, and hydrogen content. It has been found that the material is excellently stable under low-orbit conditions, as bombardment with oxygen forms a stable, 5 nm thick titanium oxide layer, having no influence on lubrication but protecting the degradation of hydrogen.

The examples given are not only important pieces of research, but also demonstrate the unique capabilities of neutron and synchrotron radiation for the investigation of thin films, coupled to complementary methods.

3. Conclusions and Outlook

A variety of interwoven topics have been compiled in the present Special Issue of *Metals*. The materials range from bulk metals to thin films, under extreme conditions in space, temperature, pressure, and shock, with applications as structural materials, sensors, as well as in data storage, energy storage, and neutron optics. The methods of investigation and niche applications of neutron and synchrotron radiation are similarly widespread, covering their diversity which includes spectroscopic methods, diffraction, polarization analysis, reflectometry, contrast matching, and coherent and incoherent scattering.

There is a large overlap of questions and scientific concepts shared by the various research communities, such as response to stress in glass and crystals, non-affine super-plasticity, order and disorder, phase transformations and chemical reactions, localization, thermal equilibrium, and materials under extreme conditions. The reader will benefit from cross-disciplinary work in this widespread field designated by the word *Metals*. Moreover, it is demonstrated that one probe alone, i.e., synchrotron radiation or neutron scattering, is not sufficient for an in-depth understanding of the relevant problems. Furthermore, cross-disciplinary application of different quantum beams can benefit from synergies beyond them. Therefore, as a next step addressing various classes of materials beyond the metals alone, a new sister journal to *Metals* has been created, entitled *Quantum Beam Science* [3], to open broader opportunities for future challenges by neutron and synchrotron radiation in *Metals*.

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