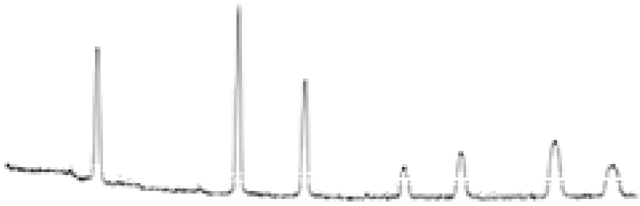
MLZ Conference 2022: Neutrons for Mobility



Report of Contributions

https://indico.frm2.tum.de/e/MLZ-Conference2022

Contribution ID: **1** Type: **Invited talk**

Neutrons for Mobility: A View into the Future (Keynote 1)

Tuesday, May 31, 2022 3:20 PM (45 minutes)

Presenter: Prof. SCHREYER, Andreas (ESS)

Session Classification: Tuesday afternoon

Track Classification: Main

Contribution ID: **2** Type: **Invited talk**

Future challenges in materials characterisation & analytics moving towards further circularity, higher functionalization and decarbonisation in Aviation (Keynote 2)

Tuesday, May 31, 2022 4:05 PM (45 minutes)

Presenter: Dr WEIMER, Christian (Airbus Munich)

Session Classification: Tuesday afternoon

Track Classification: Main

Contribution ID: **4** Type: **Invited talk**

Explaining Deviatoric Residual Stresses and Load Transfer in Aluminum Alloys and Composites with Complex Microstructure

Thursday, June 2, 2022 11:00 AM (35 minutes)

Explaining Deviatoric Residual Stresses and Load Transfer in Aluminum Alloys and

Composites with Complex Microstructure

S. EVSEVLEEV, I. SEVOSTIANOV, T. MISHUROVA, M. HOFMANN, R. KOOS, G. REQUENA, G. GARCES, G. BRUNO

The residual stresses and load transfer in multiphase metal alloys and their composites (with both random planar-oriented short fibers and particles) will be shown, as studied by neutron diffraction, by X-ray computed tomography, and by a model based on the reformulation of classic Maxwell's homogenization method.

Contrary to common understanding and state-of-the-art models, we experimentally observe that randomly oriented phases possess non-hydrostatic residual stress. Moreover, we disclose that the unreinforced matrix alloy stays under hydrostatic compression even under external uniaxial compression.

The recently developed modeling approach allows calculating the residual stress in all phases of the composites. It rationalizes the presence of deviatoric stresses accounting for the interaction of random oriented phases with fibers having preferential orientation. It also allows the explanation of the unconventional in-situ behavior of the unreinforced alloy and the prediction of the micromechanical behavior of other similar alloys.

Presenter: Prof. BRUNO, Giovanni (BAM Berlin)

Session Classification: Thursday Morning

Track Classification: Main

Contribution ID: **7** Type: **Invited talk**

Superalloys for mobility applications –In-situ characterization at high temperatures for optimized properties(Keynote 3)

Thursday, June 2, 2022 9:00 AM (45 minutes)

Superalloys for mobility applications –In-situ characterization at high temperatures for optimized properties

S. Neumeier

Friedrich-Alexander-Universität Erlangen-Nürnberg, Department of Materials Science & Engineering, Institute I: General Materials Properties, Erlangen, Germany
*steffen.neumeier@fau.de

Superalloys are key materials of our modern society. They are not only used in harsh environments of power plants for energy conversion but also in aerospace or marine applications, as they combine excellent mechanical properties at high homologous temperatures with very good oxidation and corrosion resistance. To further improve the efficiency of engines, advanced superalloys with improved properties are needed that can operate at significantly higher temperatures, for example at temperatures higher than the maximum application temperature of the most widely used polycrystalline wrought Ni-base superalloy IN718.
In this work, examples of new Ni- and Co-based superalloys are presented whose development and characterization was supported by neutron and high energy X-ray diffraction and scattering methods. It will be shown how neutron diffraction on simple, coarse grained experimental Co-Al-W-X alloys helped to determine the temperature-dependent lattice misfit between the main constituent phases, how in-situ high energy X-ray diffraction measurements revealed the deformation behaviour and formation of unwanted intermetallics phases during high temperature deformation and how small angle neutron scattering results could be used to adjust the alloys'heat treatments to optimize their mechanical properties.

Presenter: Dr NEUMEIER, Steffen (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU))

Session Classification: Thursday Morning

Track Classification: Main

Contribution ID: **10** Type: **Invited talk**

Inelastic and quasielastic neutron scattering on microporous polymer membranes for green separation processes

Wednesday, June 1, 2022 9:00 AM (35 minutes)

Inelastic and quasielastic neutron scattering on microporous polymer membranes for green separation processes

Andreas SCHÖNHALS1, Paulina SZYMONIAK1, Reiner ZORN2, Martin BÖHNING1

1Bundesanstalt für Materialforschung und -prüfung, Unter den Eichen 87, 12489 Berlin, Germany
2Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS-1) and Institute for Biological Information Processing (IBI-8), 52425 Jülich, Germany

Polymers with intrinsic microporosity are promising candidates for the active separation layer in gas separation membranes. These polymers are characterized by a high permeability and reasonable permselectivity. The latter point is somehow surprising because for microporous systems a more Knudson-like diffusion is expected then a size dependent temperature activated sieving process. It was argued in the framework of a random gate model that molecular fluctuations on a time scale from ps to ns are responsible for the permselectivity [1].
Her a series of microporous polynorbornenes with bulky Si side groups and a rigid backbone are considered which have different microporosity characterized by BET surface area values.
First inelastic time-of-flight neutron scattering measurements were carried out to investigate the low frequency density of state (VDOS). The measured data show the characteristic low frequency excess contribution to the VDOS above the Debye sound wave level, generally known as the Boson peak in glass-forming materials. It was found the maximum position of the Boson peak correlates with the BET surface area value [2].
For two selected comparable polynorbornenes elastic scans as well as QENS measurements by a combination of neutron time-of-flight and backscattering are carried out [3]. A low temperature relaxation process was found for both polymers. This process was assigned to the methyl group rotation. It was analysed in terms of a jump diffusion in a three-fold potential. The analysis of the dependence of the elastic incoherent structure factor on the scattering vector yields the number of methyl groups which might be immobilized.

[1] R. Inoue, T. Kanaya, T. Masuda, K. Nishida, O. Yamamuro Macromolecules 45, 6008 (2012)
[2] R. Zorn, P. Szymoniak, M. A. Kolmangadi, A. Wolf, D. Alentiev, M. Bermeshev, M. Böhning, A. Schönhals Physical Chemistry Chemical Physics 22, 18381 (2020)
[3] A. Schönhals, P. Szymoniak, M. A, Kolmangadi, M. Böhning, M., Zamponi, B. Frick, M. Appel, G. Günther, M. Russina, D. Alentiev, M. Bermeshev, R. Zorn, R. Journal of Membrane Science 642 119972 (2022)

E-mail for corresponding author: andreas.schoenhals@bam.de

Presenter: Prof. SCHÖNHALS, Andreas (BAM Berlin)

Session Classification: Wednesday morning

Track Classification: Main

Contribution ID: **13** Type: **Invited talk**

Design of Solid State Battery Materials and Prototypes

Wednesday, June 1, 2022 1:45 PM (45 minutes)

Design of Solid State Battery Materials and Prototypes
Jennifer L.M. Rupp
Technical University of Munich & TUM International Energy, Germany
jrupp@mit.edu

Next generation of energy storage devices may largely benefit from fast and solid Li+ ceramic electrolyte conductors to allow for safe and efficient batteries and fast data calculation. For those applications, the ability of Li-oxides to be processed as thin film structures and with high control over Lithiation and phases at low temperature is of essence to control conductivity. Through this presentation we review the field from a new angle, not only focused on the classics such as Li-ionic transport and electrochemical stability window for Li-solid state battery electrolytes, but focusing on opportunities and challenges routes in thermal and ceramic processing of the components and their assemblies with electrodes. Oxide vs. Sulfide based solid state battery materials and designs will be reflected on.Also, we will carefully review and give perspectives on the role of solid state battery ceramic strategies for the electrolyte on the electrode interfaces and towards charge transfer and vs. current densities. In other words, it will be a little ceramicist (own) love story on the good and the evil we can design by smart ceramic manufacture at the interfaces originating by the very first choices made in the electrolyte ceramic structure and material design. In the second part of the talk we will discuss new opportunities on low temperature processing of solid state electrolyte ceramics that do not technically require "classic sintering"and avoid prior particle calcination; instead demonstrating opportunities to use unique liquid based direct densification routes and vacuum techniques to design solid electrolytes and grafting interfaces to new hybrid and solid state battery prototypes targeted at processing below 700C for all parts. Collectively, the insights on solid state energy storage provide evidence for the functionalities that those Li-solid state material designs can have for cost and mass manufacturable solid state and hybrid battery prototypes.

References for further reads
Photo-enhanced ionic conductivity across grain boundaries in polycrystalline ceramics
T. Defferriere, D. Klotz, J.C. Gonzalez-Rosillo, J.L.M. Rupp, H.L. Tuller
Nature Materials, 1-7 (2022)

Processing thin but robust electrolytes for solid-state batteries
M. Balaish, *J.C. Gonzalez-Rosillo*, K.J. Kim, Y. Zhu, Z.D. Hood, J.L.M. Rupp
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Photo-enhanced ionic conductivity across grain boundaries in polycrystalline ceramics
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Nature Materials, in press (2022)

Solid-State Li–Metal Batteries: Challenges and Horizons of Oxide and Sulfide Solid Electrolytes and Their Interfaces
K.J. Kim, *M. Balaish*, M. Wadaguchi, L. Kong, J.L.M Rupp
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Lithium-film ceramics for solid-state lithionic devices
Y. Zhu, J.C. Gonzalez-Rosillo, M. Balaish, Z.D. Hood, K.J. Kim, J.L.M. Rupp
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K.J. Kim, J.J. Hinricher, J.L.M. Rupp
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All ceramic cathode composite design and manufacturing towards low interfacial resistance for garnet-based solid-state lithium batteries
K.J. Kim and J.L.M. Rupp
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A low ride on processing temperature for fast lithium conduction in garnet solid-state battery films
R. Pfenninger, M. Struzik, I. Garbayo, E. Stilp, J.L.M. Rupp
Nature Energy, 4, 475–4832019 (2019)
Spray Pyrolysis Processing of The Electron - Ion Insulator for Metal Anode Adhesion in Solid State Batteries
S. Chakravarthy, W.S. Chang, H. Chu, J. Hinricher, Z.D. Hood, Y. Huang, S.Y. Kim, J. Li, A. Maurano, K. Pei, J.L.M. Rupp, Z. Wang, Y. Zhu MIT-Samsung IP case : 23978J (2022)

Dual-Phase Composite Li-Conducting Thin Film and Method of Making the Same
Y. Zhu, Z. Hood, J. Hinricher, W.S. Chang, H.C. Lee, L. Miara, J.L.M. Rupp IP: US 63/180,150 (2021)

Electron-ion insulator for metal anode adhesion in solid state batteries.
Z. Wang, J. Rupp, Y. Chen, A. Maurano, S. Chakvararthy, K. Pei, J. Li IP: US 17/144,687 (2021)

Amorphous Nitrogen-Rich Solid State Electrolyte
J.L.M. Rupp, W.S. Chang, Z. Hood, L. Miara IP: US 63/051024 (2020)

Bilayer component for Li metal battery and their use therein.
Z.D. Hood, W.S. Chang, L. Miara, J.L.M. Rupp. IP: US 62/994,466 (2020)

Lithium Solid Electrolyte and Method of Manufacture Thereof
Y. Zhu, W.S. Chang, L. Miara, J.L.M. Rupp IP: US 62/863,059 (2019

Presenter: Prof. RUPP, Jennifer (Technical University of Munich)

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **27** Type: **Talk**

Enhancing battery performance by clever tuning of anode materials

Wednesday, June 1, 2022 2:30 PM (20 minutes)

Li-ion batteries are not only omnipresent in portable devices, they are also being increasingly used to power electric vehicles which demands them to possess even higher energy densities. Energy storage capabilities, rate performances and cycling stabilities of Li-ion batteries are strongly dependent on the electrode materials. Practical energy densities accessible with cathode materials are already close to their theoretical limits with the currently available electrolytes. For advanced LIBs technologies, the improvement or replacement of anode materials becomes more paramount than the cathode materials. The widely used graphite anode is stable, but offers low energy density, and suffers from side reactions, which are severe at higher charging rates and low temperatures, leading to eventual capacity fading. Lithium titanium oxide anodes offer much longer life cycles, but have even lower energy densities. Li metal anodes offer the highest possible energy density, but are prone to dendrite formation and thus not preferred for safety reasons. Si anodes offer second highest possible energy densities, but suffer from large volume changes leading to fast capacity fading. This contribution will discuss advances in anode material engineering in above mentioned anodes. It will be demonstrated how battery performances can be enhanced by either altering the anode morphology, or preparing composite anode mixtures, or by applying coatings to anode surfaces. Deeper insights into aging mechanism can be described very well with neutron based analytical methods.

Authors: Dr PAUL, Neelima (Technical University of Munich, Heinz Maier-Leibnitz Zentrum (MLZ)); GILLES, Ralph

Presenter: Dr PAUL, Neelima (Technical University of Munich, Heinz Maier-Leibnitz Zentrum (MLZ))

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **28** Type: **Talk**

The contribution of neutron techniques for electro mobility and optimization of air- and land-based gas turbines

Wednesday, June 1, 2022 11:10 AM (20 minutes)

Batteries play an important role in the field of electric mobility because they can replace fossil fuels in many areas. Neutrons enable the detailed characterization in situ and operando of electrochemical processes on different length scales. Examples are presented how neutron diffraction, neutron imaging and neutron depth profiling improve the understanding of batteries and individual components.
Air- and land-based gas turbines are essential for airplanes and energy production. A major goal is to reach higher operating temperatures to reduce both fuel consumption and pollution. Neutrons as a probe enable the characterization of high-temperature alloys under real working conditions such as stress/compression or high temperature. Such conditions are realized with demanding sample environments. Neutron diffraction and small-angle neutron scattering monitor the phase transformations and the precipitation kinetics. The deeper understanding of the microstructure results in an optimization of the alloy development.

Reference:
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Author: Dr GILLES, Ralph (TU München, MLZ)

Presenter: Dr GILLES, Ralph (TU München, MLZ)

Session Classification: Wednesday morning

Track Classification: Main

Contribution ID: **29** Type: **Talk**

Texture evolution of a graphene nanoparticles reinforced copper matrix laminated composites

Wednesday, June 1, 2022 4:05 PM (20 minutes)

The recrystallization texture that develops during annealing process of deformed polycrystalline metal is vital and largely responsible for the anisotropy mechanical properties of the materials. The origin of this kind of texture is always great source of scientific interests. Especially, for cube texture which is considered as a gift in fcc metals used as conductive materials like Cu, Al, etc., for many electrical and electronic devices, an extensive researches have already been conducted to investigate the formation mechanism of the cube texture. However, although lots of mechanisms have already been proposed, contradictions always appear by the results of different researchers. Thus, the formation mechanism of the cube texture in fcc metals is still in debate and worth thoroughly investigation.
In our work, graphene nanoparticles (GNP) reinforced copper (Cu) matrix laminated composites were fabricated through three steps consisting of electrophoretic deposition (EPD), hot-pressing sintering and hot-rolling process. The results from EBSD, neutron diffraction as well as synchrotron radiation all shows that when the raw copper foil thickness is 30μm, the Cu-GNP composites always obtain nearly pure cube texture, while the pure copper obtains coarse grains with random orientation. This may provide us a new technique to obtain highly textured material from polycrystalline materials and reveal the mechanism of the recrystallization and orientation monopolization mechanism of the laminated materials.

Author: Dr GAN, Weimin (Helmholtz-Zentrum Hereon)

Co-authors: Dr MAAWAD, Emad (GEMS, Helmholtz-Zentrum Hereon, Geesthacht, Geramny); HOFMANN, Michael; Dr HAILONG , Shi (Harbin Institute of Technology, Harbin, PR China); Prof. WANG, Xiaojun (Harbin Institute of Technology, Harbin, PR China); Dr ZHANG, Yudong (LEM3, Université de Lorraine, Metz, France)

Presenter: Dr GAN, Weimin (Helmholtz-Zentrum Hereon)

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **30** Type: **Poster**

Unique Materials’Characterization by In-Situ Neutron Diffraction and Small Angle Neutron Scattering and the new Quenching and Deformation Dilatometer

Wednesday, June 1, 2022 5:00 PM (20 minutes)

A Quenching and Deformation Dilatometer (TA instruments DIL 805A/D/T) operates at the MLZ for performing in-situ neutron diffraction (phase, texture, stress/strain) at STRESS-SPEC and small-angle neutron scattering (nanostructure) at SANS-1. Imaging applications are under preparation at ANTARES. With this setup, the evolution of the sample length during heating or quenching can be accurately monitored while scattering data are being acquired. Thanks to induction heating and gas cooling very high rates are accessible. Forces up to 20 and 8 kN can be applied in compression and tension, respectively. Besides, special sample holders for powders will soon extend the range of applications.
The combination of the neutron scattering and dilatometry measurements yields a unique view on the microstructural evolution under thermomechanical treatment. In this work, we will present some results of different materials, i.e. high entropy alloy (HEA), light weight TiAl alloy and Cu - Ce0.8Gd0.2O2-⊠ (CGO) composite. The combination of dilatometry and in-situ diffraction allows an accurate investigation of phase transformations (type and temperature) in AlCrFeNiTi HEA. TiAl alloy study will be focused on the bulk texture evolution induced by hot compression performed with the dilatometer. The aim is to investigate the mechanisms of hot compression and further to optimize the mechanical properties. Last example is the first results on Cu-CGO composites for high temperature green energy applications (solid oxide fuel cells, electrolyzers and catalytic membrane reactors). Here we studied the thermal expansion coefficient of the Cu-CGO cermets as a bulk at the same time as we obtain in-situ high temperature microstructural information on both Cu and CGO phases (diffraction measurements performed at the synchrotron Desy).

Authors: SOLIS, Cecilia (Helmholtz-Zentrum Hereon); LI, Xiaohu; HOFMANN, Michael; Dr BALAGUER, María (Instituto de Tecnología Química); Dr NAVARRETE, Laura (Instituto de Tecnología Química); Mr KEITA, Mohamed (HZ Hereon); Dr ZHANG, Yudong (CNRS); GAN, Weimin (Helmholtz-Zentrum Hereon)

Presenter: SOLIS, Cecilia (Helmholtz-Zentrum Hereon)

Session Classification: Posters

Track Classification: Main

Contribution ID: **31** Type: **Poster**

Neutron Spin Echo Spectroscopy with the J-NSE ”PHOENIX”

Wednesday, June 1, 2022 5:20 PM (20 minutes)

Neutron spin echo (NSE) spectroscopy provides a very high energy resolution, making slow diffusive processes accessible on molecular length- and time-scales. The J-NSE “PHOENIX” spectrometer at the MLZ has been modernized in the last years with superconducting main solenoids with optimized field shape which increases the resolution of the spectrometer by a factor of 2-3 [1]. Molecular motions of polymer chains or diffusion of hydrogen or hydrogen containing molecules can be studied with NSE, making it a valuable tool e.g. for diffusion studies in fuel cell membranes [2]. Besides a description of the new J-NSE “PHOENIX” we will present examples of typical applications in the area of soft matter studies with a focus on materials relevant to energy studies and show perspectives on future studies in this area by NSE.

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Authors: HOLDERER, Olaf (JCNS); FOMINA, Margarita

Presenter: HOLDERER, Olaf (JCNS)

Session Classification: Posters

Track Classification: Main

Contribution ID: **32** Type: **Talk**

Developments for 4D Neutron Depth Profiling at the N4DP Instrument

Thursday, June 2, 2022 11:35 AM (20 minutes)

Neutron Depth Profiling (NDP) is a non-destructive, isotope-specific, high-resolution nuclear analytical technique, which is often used to probe concentration profiles of lithium, boron, nitrogen, helium and several other light elements in different host materials. The N4DP instrument is located at the Prompt Gamma Activation Analysis (PGAA) facility of Heinz Maier-Leibnitz Zentrum (MLZ), which provides a cold neutron flux up to $5 \times 10^{10}\,\mathrm{s}^{-1}\mathrm{cm}^{-2}$. When a neutron is captured by a specific nuclide, charged particles with well-defined energies are emitted. The energy loss of the charged particles traveling through the host material is related to the depth of origin at a resolution level up to tens of nanometers.

We applied NDP to study the lithium-ion concentration gradient in energy storage systems, e.g. Li-ion batteries. Here, NDP reveals the evolution of immobilized lithium, which is one of the main causes of battery lifetime limitation. Furthermore, the status of the ongoing development towards 4D profiling is presented, where not only the concentration gradient, but also the lateral position of probes as well as its time evolution will be measured. For this, a highly segmented Si-based detector with 32×266 stripes, including integrated electronics, were tested. Using a camera-obscura geometry setup, we aim for lateral resolutions down to $100\mu\mathrm{m} \times 100\mu\mathrm{m}$ and highest time resolutions using a newly developed elliptical focussing neutron guide. This project is supported by the BMBF, Contract No. 05K16WO1, 05K19WO8.

Author: NEAGU, Robert

Co-authors: TRUNK, Markus; WERNER, Lukas (TUM); GERNHÄUSER, Roman (TU-München); GILLES, Ralph; REVAY, Zsolt (PGAA); MÄRKISCH, Bastian (Physik-Department, TUM)

Presenter: NEAGU, Robert

Session Classification: Thursday Morning

Track Classification: Main

Contribution ID: **33** Type: **Talk**

In-situ high temperature neutron scattering study on the hardening phase precipitation in the new VDM® Alloy 780

Thursday, June 2, 2022 9:45 AM (20 minutes)

In order to improve the microstructure and mechanical properties of newly developed Ni-base superalloy VDM® Alloy 780 it is necessary to understand the γ'hardening phase precipitation process. Here the precipitation process was studied in-situ by time-of-flight (TOF) neutron diffraction (ND) and small-angle neutron scattering (SANS) experiments at high temperature, which allowed us to characterize the obtained γ'precipitates, fraction and sizes (by SANS from the very early stages) and the misfit between matrix and precipitates (by ND). Besides, atom probe tomography (APT) and scanning electron microscope (SEM) provided further details on microstructural and chemical composition.
The precipitation of γ'phase at 720 ℃, i.e. its size and volume fraction as a function of time, was monitored in two differently solution-annealed samples. It appears that the obtained results depend on the heat treatment history of the sample. Two particle size distributions of γ'precipitates were detected by SANS after 2 h in the case of the sample with an extra step after solution-annealing. Variation in heating rates of SANS and TOF ND measurements results in different precipitates nucleation and growth kinetics. A final heat treatment at 620 ℃ does not lead to a similar precipitation or growth process.
The in-situ SANS measurements at 750 ℃ of the fully precipitation hardened sample with two particle size distributions of γ'precipitates at RT confirms the matrix-diffusion-controlled Ostwald ripening of the precipitates and shows smaller coarsening kinetics than other reported Ni-based superalloys.
The project is funded by the BMBF through the project "An innovative testing machine for heating, quenching, tension, compression and cracking studies of industrial relevant high-temperature alloys—HiMat"(project number 05K19WO7).

Authors: Dr SOLIS, Cecilia; Mr KIRCHMAYER, Andreas (Friedrich-Alexander-Universität Erlangen-Nürnberg); Dr DA SILVA, Ivan (ISIS); KUEMMEL, Frank; MUEHLBAUER, Sebastian; BERAN, Premysl (Nuclear Physics Institute CAS); Dr HAFEZ HAGHIGHAT, Massod (VDM Metals); NEUMEIER, Steffen (Friedrich-Alexander-Universität (FAU) Erlangen-Nürnberg); GILLES, Ralph

Presenter: Dr SOLIS, Cecilia

Session Classification: Thursday Morning

Track Classification: Main

Contribution ID: **34** Type: **Talk**

Hydrogen motion in Li4(BH4)(NH2)3 investigated by Quasielastic Neutron Scattering (QENS)

Wednesday, June 1, 2022 9:35 AM (20 minutes)

Fast and efficient hydrogen storage is one of the key components for the use of hydrogen in a sustainable energy economy. Reactive Hydride composites have been considered for some time as potential solid state storage systems, among others also the amide based mixture $Mg(NH_2)_2$ + 2 LiH. The kinetic performance of the hydrogen exchange reaction in this system is significantly enhanced by the addition of $LiBH_4$ [Gizer et al. Inter. J. Hydrogen Energy 44, 11920-11929 (2019)] and the subsequent formation of the amide-borohydride compound $Li_4(BH_4)(NH_2)_3$. Here, we present a study of the structure and of the anion motion of in $Li_4(BH_4)(NH_2)_3$ investigated with synchrotron radiation powder X-ray diffraction (SR-PXD) and quasielastic neutron scattering (QENS) at temperatures close to operating condition. SR-PXD confirms the recrystallization of $Li_4(BH_4)(NH_2)_3$ into the α-phase during cooling from the melt. The QENS measurements prove a long-range diffusive motion of hydrogen containing species at 514 K with the diffusion coefficient $D \sim 10^{-6}\,\frac{\mathrm{cm}^2}{\mathrm{s}}$. At temperatures below 514 K, localized rotational motions were observed which have been attributed to (BH_4^- tetrahedra units undergoing rotations mainly around C_3 axes. The activation energy for this thermally activated process is found to be E_a = 15.5±0.9 and 17.4±0.9 $\frac{\mathrm{kJ}}{\mathrm{mol}}$ for the two instrumental resolutions utilized in the QENS measurements, respectively, corresponding to observation times of 55 and 14 ps. The results will be discussed in the context of the improved hydrogen exchange reaction that is observed in $Mg(NH_2)_2$ + 2 LiH with $LiBH_4$ additions.

Authors: PISTIDDA, Claudia (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); GIZER, Gökan (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); ASLAN, Neslihan (GEMS at Heinz Maier-Leibnitz Zentrum (MLZ), Helmholtz-Zentrum Hereon); DORNHEIM, Martin (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); MÜLLER, Martin (Helmholtz-Zentrum Hereon); BUSCH, Sebastian (GEMS at Heinz Maier-Leibnitz Zentrum (MLZ), Helmholtz-Zentrum Hereon); Dr LOHSTROH, Wiebke (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München)

Presenter: Dr LOHSTROH, Wiebke (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München)

Session Classification: Wednesday morning

Track Classification: Main

Contribution ID: **35** Type: **Talk**

Assessment of experimental residual stresses measured into an additively manufactured 316L austenitic stainless steel with finite element simulations and effect of heat treatment on stress relief

Thursday, June 2, 2022 10:05 AM (20 minutes)

Laser powder bed fusion (LPBF) is a method for selectively or partly melting a bed of powdered material using a laser, where each subsequent layer of molten or partly melted material is bonded to the previous layer [1, 2]. The rapid cooling rates inherent to this process result in large stress gradients, which can have a significant effect on the performance during service of metallic components, reducing fatigue lifetime or provoking sudden localized failures. Thermal treatments applied after the build but before the components are removed from the build plate usually release these residual stresses (RS). As a result, estimating the RS and their distribution has become critical for maximizing the viability of manufacturing parameters. 316L austenitic stainless steel is a well-known and characterized steel in Additive Manufacturing (AM) that is industrially relevant. Because this material is stable against microstructural changes in temperature and segregation, it is a good choice for this study.

Two arch-shape samples with a hollow region have been manufactured to measure the RS in as-built and heat treated (HT) conditions. This HT was developed for stress relief on this steel based on microstructural and experimental analysis i.e., tensile and distortion tests. The microstructure of both samples was characterized by means of different techniques (i.e., LOM, FEG-SEM, EBSD, XRD, and hardness maps) and the RS were measured with non-destructive depth-resolved syn-chrotron X-ray diffraction experiments. RS measurements are compared to computer-based simu-lations. The novelty of this research is the combined evaluation of these three fields i.e., microstructure, experimental RS and calculated RS, all in all evaluated in an end use geometry.

This work is being developed in the frame of the EU Horizon 2020 EASI-Stress project under grant agreement No 953219. The aim of the project is to standardized the use of non-destructive synchrotron x-ray and neutron diffraction based residual stress characterization and understand the formation and progression of RS to strengthen industrial access to these non-destructive large-scale facilities. As a result of this work, it is expected to produce more trust experimental data to develop more robust mathematical models.

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Author: Mr SANCHEZ PONCELA, Manuel (ArcelorMittal)

Co-authors: Dr CANELO, David (Hereon); Mr MARTINEZ, Juan Manuel (ArcelorMittal); Dr CABEZA, Sandra (ILL); Dr PIRLING, Thilo (ILL); Dr STARON, Peter; Dr ABREU, Guilherme (Hereon); Dr MAAWAD, Emad (Hereon); Dr SCHELL, Norbert (Hereon)

Presenter: Mr SANCHEZ PONCELA, Manuel (ArcelorMittal)

Session Classification: Thursday Morning

Track Classification: Main

Contribution ID: **36** Type: **Poster**

Investigation of Na+ diffusion in NASICON solid electrolyte materials by Quasi-Elastic Neutron Scattering

Wednesday, June 1, 2022 5:40 PM (20 minutes)

Over the last decades, sodium superionic conductor (NASICON) materials have been a widely studied class of solid electrolytes for Na-ion based all-solid-state batteries due to their high conductivity and facile synthesis conditions. The aim of this work is to clarify the reason for extremely high conductivity exhibited by some compositions, specifically by $Na_{1+x}Zr_2Si_xP_{3-x}O_{12}$ ($0 \leq x \leq 3$). The focus of this study is to explain the role of the monoclinic to rhombohedral phase transition for the material with x=2.4, which occurs at around 170 ℃, on the Na^+-ion occupancy in the crystal structure. Additionally, we investigate the role of Al/Y and Sc substitution on the sodium occupancy and the overall temperature dependence of the ionic conductivity in the temperature range of 297-640 K.

In general, neutron scattering techniques are highly suitable for measuring the properties of crystalline materials. Here, QENS (quasi-elastic neutron scattering) is applied to measure the spatial and temporal dynamic properties of diffusion of sodium ions in the crystal lattice. This technique is feasible, because sodium is the only diffusive element in the material and the only one scattering incoherently. The measurements were performed at the BASIS spectrometer at the Spallation Neutron Source, Oak Ridge National Laboratory in Tennessee, USA. For the evaluation of the QENS data, the DAVE software (NIST Center for Neutron Research) is used.

The Na^+-ion diffusion mechanism can be described by the right choice of the diffusion model, in this case the Chudley-Elliot jump diffusion model has been used. Important parameters, such as diffusion coefficients, activation energies, jump distances between the occupation sites and residence times are extracted from the measured and modelled QENS data.

Authors: PIVARNÍKOVÁ, Ivana; SEIDLMAYER, Stefan; Dr FINSTERBUSCH, Martin (IEK-1 FZJ); Mr DÜCK, Gerald (IEK-1 FZJ); Dr JALARVO, Niina; MÜLLER-BUSCHBAUM, Peter (TU München, Physik-Department, LS Funktionelle Materialien); GILLES, Ralph

Presenter: PIVARNÍKOVÁ, Ivana

Session Classification: Posters

Track Classification: Main

Contribution ID: **37** Type: **Poster**

Complementary Neutron-Based and X-Ray Measurements on the Lithiation Mechanism of LiAl Electrodes

Wednesday, June 1, 2022 6:00 PM (20 minutes)

Metal alloys, such as LiAl, are gaining more interest as anode materials for lithium ion batteries because they exhibit a high theoretical capacity while being inexpensive. Aluminium has an almost three times higher specific capacity with 993 mAh/g in relation to the commonly used graphite anode. [1] During Lithiation, aluminium begins to form a solid solution with lithium, the so called alpha-LiAl phase. Once the solubility limit is reached upon lithiation, the alpha-LiAl undergoes a phase transition to the beta-LiAl phase. [2]
Al electrodes were electrochemically lithiated to different state of charges in coin cells with Li metal as counter electrode. X-ray diffraction measurements were conducted on the disassembled anodes with the goal to understand the lithiation mechanism and to determine the fractions of alpha- and beta-LiAl phases. The Rietveld refinements yield that the samples charged to SoC25 show a higher amount of alpha-LiAl. Simultaneously, the amount of beta-LiAl in the samples increases with higher SoC as expected.
Additional neutron depth profile measurements were performed in NPI CAS Rez at the CANAM infrastructure to determine the Li distribution throughout the lithiated samples. First results have confirmed that the lithiation of the aluminium starts at the surface where a higher Li concentration was identified. The higher charged sample also shows a stronger lithiation in the bulk of the Al anode. This shows that the whole anode is lithiated with a concentration gradient from surface to bulk material. In the lower charged sample, no Li was detected near the backside of the anode, indicating that pristine aluminium is there still present. Additional operando diffraction measurements are planned to further investigate the early stages of the lithiation and the nucleation of the beta-LiAl.
This work was performed as collaboration between TUM (Heinz Maier-Leibnitz Zentrum, FRM II) and RWTH Aachen (ISEA) in the frame of the BMBF project ExcellBattMat cluster.

Author: PHAM, Thien An (Heinz Maier-Leibnitz Zentrum, Technical University of Munich (FRM II))

Co-authors: WELLS, Luke (RWTH Aachen University (RWTH) - Institute for Power Electronics and Electrical Drives); SEIDLMAYER, Stefan (Heinz Maier-Leibnitz Zentrum, Technical University of Munich (FRM II)); CANNAVO, Antonino (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); CECCIO, Giovanni (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); VACÍK , Jiří (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); MÜLLER-BUSCHBAUM, Peter (Heinz Maier-Leibnitz Zentrum, Technical University of Munich (FRM II)); FIGGEMEIER, Egbert (RWTH Aachen University (RWTH) - Institute for Power Electronics and Electrical Drives); GILLES, Ralph (Heinz Maier-Leibnitz Zentrum, Technical University of Munich (FRM II))

Presenter: PHAM, Thien An (Heinz Maier-Leibnitz Zentrum, Technical University of Munich (FRM II))

Session Classification: Posters

Track Classification: Main

Contribution ID: **38** Type: **Talk**

Flexible High-Energy Optics using Coated Hollow Capillaries

Wednesday, June 1, 2022 4:25 PM (20 minutes)

The guidance of high-energy wave and particle radiation is associated with numerous challenges. This severely limits the possible uses. However, flexible guidance and formation would open up numerous new fields of application.

In this talk, different selected coating technologies like Chemical Vapour Deposition (CVD) in order to coat even complex hollow glass capillaries[2] used as High- Energy Optics are introduced. These different coating methods are discussed intensively for the modification and functionalisation of different hollow glass capillaries with High-Z-metals[2] in order to use them as X-Ray Waveguides e.g. for analytical applications like High- Energy Optics for microanalysis. As High-Z-metals mainly d-block transition metals can be used. For this purpose, special organometallic, elementorganic or coordination compounds[3] mainly of d-block transition metals are decomposed chemically in these capillaries.

Thus, a specific tailored nanostructured surface for the respective applications in Functional Materials can be realized to guide even High- Energy radiation in these hollow capillaries like optical waveguides. To achieve this objective, the right choice precursor material is highly relevant.

These innovative surface coatings, which alter the properties according to the state-of-the-art of uncoated High- Energy Optics significantly, can enable these flexible modified capillaries to boost up their performance for different flexible applications.

In this talk, the latest developments in this exciting research area will be presented and discussed in detail.

Author: Prof. WOCHNOWSKI, Jörn Volkher

Presenter: Prof. WOCHNOWSKI, Jörn Volkher

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **39** Type: **Talk**

Interface growth and lithium concentration homogeneity in all solid state batteries with LLZO-LCO cathodes by neutron depth profiling

Wednesday, June 1, 2022 2:50 PM (20 minutes)

All-solid-state lithium batteries (ASSBLIBs) offer promising advantages in terms of energy and power density as well as safety over lithium-ion batteries (LIBs) with liquid electrolytes. However, they require solid electrolytes with high ionic conductivity comparable to established liquid-based systems. Urgent questions in the application of ASSLIBs are tied to the three key properties, (i) chemical stability during cell manufacturing and (ii) cycling, which can cause interface, and (iii) lithium gradient growth between the different solid phases [1, 2].
However, direct measurement and quantification of the Li distribution in LIBs and ASSLIBs is difficult or impossible with conventional methods, although it would be crucial for understanding the processes governing the key properties, stability, ionic conductivity and homogeneity of lithium distribution. Two of the most promising techniques for these investigations are neutron beam-based depth profiling (NDP) and ion beam-based particle induced x-ray (PIXE) and gamma ray emission (PIGE) spectroscopy. The non-destructive NDP method proved to be successful to investigate liquid systems [3, 4] at the FRM II neutron source in Garching. Other neutron sources have applied NDP for studying interfaces or for thin film cell investigations [5, 6]. Our partner group at IEK-1 produces oxide based ASSLIBs that show one of the best performing solid electrolytes and are thoroughly investigated using continuum modelling [7].
We study the distribution of lithium in our ceramic-based ASSLIB cells with LLZ (Li6.6La3Zr1.6Ta0.4O12) as the electrolyte and separator, and LiCoO2 as cathode active material by using both NDP and PIXE/PIGE measurements. The NDP data measured at the neutron source in NPI CAS Rez suggest that a thin interface is formed on the surface of LLZO.

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[4] Wetjen, et al., J. Electrochem. Soc., 2018, 165, A2340.
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[7] Finsterbusch, et al., ACS Appl. Mater. Inter., 2018, 10, 22329.

Author: Dr SEIDLMAYER, Stefan

Co-authors: CANNAVO, Antonino (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); CECCIO, Giovanni (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); SEOK, Ah-Ram (IEK-1 FZJ); FINSTERBUSCH, Martin (IEK-1 FZJ); VACÍK , Jiří (Nuclear Physics Institute of the CAS Nuclear Physics Institute CAS (UJF)); GILLES, Ralph

Presenter: Dr SEIDLMAYER, Stefan

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **40** Type: **Talk**

Targeted use of residual stress in electrical steel to increase energy efficiency

Friday, June 3, 2022 11:10 AM (20 minutes)

The magnetic flux guidance in an electric engine is usually achieved by introducing cutouts in the electrical steel (ES) sheets that make up its core. However, these cutouts create thin structures, reducing the mechanical strength of the ES sheets and limiting the achievable maximum rotational speed and therefore the energy efficiency of the engine.
Residual stress in ES sheets reduces the mobility of magnetic-domain walls due to the magneto-elastic effect. This can be utilized to create a novel type of magnetic flux barriers relying on the local decrease in magnetic permeability introduced by embossing of the ES sheet. Such barriers do not compromise the structural integrity of the sheets.
The influence of various embossing parameters on the residual stress state was calculated by Finite-Element simulations and probed using neutron grating interferometry (nGI). In nGI the dark field image (DFI) maps ultra-small-angle neutron scattering as resulting from the interaction of the magnetic moment of the neutron with the magnetic domain structure in the bulk of the sheet sample. In an applied magnetic field, a change in the domain size therefore results in a change of the DFI signal. Hence, enabling the visualization of the local distribution of magnetization.
Prototypes of magnetic flux barriers based on the magneto-elastic effect show similar flux guidance as traditional barriers while the mechanical strength is comparable to unworked ES.
In this presentation we will give a comprehensive overview of the effect of residual stress on the magnetic properties of electrical steel as well as show the applicability of nGI to probe such materials.

Authors: SCHAUERTE, Benedikt (Institut für elektrische Maschinen, RWTH Aachen); GILCH, Ines (Lehrstuhl für Umformtechnik und Gießereiwesen, Technische Universität München); SEBOLD, Simon (MLZ); SCHULZ, Michael; NEUWIRTH, Tobias

Presenter: SEBOLD, Simon (MLZ)

Session Classification: Friday Morning

Track Classification: Main

Contribution ID: **41** Type: **Poster**

Studying the temperature-dependent lattice misfit of γ′ strengthened superalloys with a varying Co/Ni-ratio using neutron and high-resolution X-ray diffraction

Wednesday, June 1, 2022 6:10 PM (20 minutes)

Superalloys strengthened by ordered γ′ precipitates are widely used in aircraft engines due to their excellent high-temperature properties and play, therefore, an essential role for mobility. The γ matrix phase and the coherently embedded γ′ precipitates have slightly different lattice parameters due to their different chemical compositions resulting in a constrained lattice misfit between both phases. In this study, the lattice misfit of the model alloy series Ni–Co–9Al–8W–8Cr (at.%) with a varying Co/Ni-ratio was investigated by neutron and X-ray diffraction. The room temperature lattice misfit of polycrystalline (PX) samples was investigated by means of neutron diffraction. Moreover, the lattice misfit of single crystalline (SX) samples as a function of the temperature was studied by employing high-resolution X-ray diffraction (HRXRD) up to 1000 ℃. The Co/Ni-ratio strongly affects the sign of the lattice misfit and its temperature-dependent behavior. The Ni-rich alloys show a negative lattice misfit at room temperature that becomes more negative at higher temperatures. In contrast, the Co-rich alloys exhibit a positive lattice misfit at room temperature that slightly tends towards zero upon heating. Since the sign and magnitude of the lattice misfit at high temperatures strongly affect the resulting coherency stresses within the alloys and thus mechanical properties, knowledge of the temperature-dependent lattice misfit is crucial for future superalloy development.

Author: BANDORF, Jakob (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU))

Co-authors: Dr ZENK, Christopher H. (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)); VOLZ, Nicklas (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)); Dr DOLOTKO, Oleksandr (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München); Dr NEUMEIER, Steffen (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU))

Presenter: BANDORF, Jakob (Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU))

Session Classification: Posters

Track Classification: Main

Contribution ID: **42** Type: **Talk**

Structure of Nafion membranes in humidity

Friday, June 3, 2022 10:50 AM (20 minutes)

Nafion membranes are frequently used as membranes in fuel cells and electrolyzers due to their chemical resistivity. In this study, we used Nafion membranes before and after the application. We investigated the structure of Nafion membranes in different humidity conditions using small angle neutron scattering (SANS) and prompt gamma activation analysis (PGAA). The SANS measurements reveal the swelling of the pores with water that base on the analysis of structural correlation peaks. Furthermore, the bare water content is also characterized in terms of the incoherent background. Using hydrogen polarization by spin pumping, a full contrast variation series is measured by SANS. Here, the finite pore size is clearly measured, and by the time dependence of the signals upon pumping, the thickness of the water/polymer interphases is given. PGAA reveals an independent cross check of the water uptake.

Authors: BABCOCK, Earl; Dr FRIELINGHAUS, Henrich (JCNS); APPAVOU, Marie-Sousai (Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH); SZEKELY, Noemi (Jülich Centre for Neutron Science JCNS); HOLDERER, Olaf; Prof. KOIZUMI, Satoshi (Ibaraki University); Dr NODA, Yohei (Ibaraki Univeristy)

Presenter: Dr FRIELINGHAUS, Henrich (JCNS)

Session Classification: Friday Morning

Track Classification: Main

Contribution ID: **43** Type: **Talk**

Analysis of Precipitation and Lattice Defects in Aluminum Alloys Using Positron Annihilation Spectroscopy

Friday, June 3, 2022 9:40 AM (20 minutes)

Light metal alloys are a vital part in industrial applications such as aviation, electric vehicle construction and space flight. Despite the advancements in composite materials, aluminum alloys are still the only choice for numerous technical components. Pure aluminum exhibits generally poor mechanical properties. By alloying aluminum with other metals such as copper, lithium or manganese, its mechanical properties can be vastly improved. For many of these light metal alloys, precipitation hardening causes a considerable gain in mechanical strength.

Positron annihilation techniques are usually applied to investigate lattice defects with highest sensitivity. In particular, Coincident Doppler Broadening Spectroscopy (CDBS) can be used to study the chemical surrounding of vacancy-like defects and precipitates in aluminum alloys. With CDBS, we analyzed various alloys after undergoing a wide variety of solution annealing and tempering steps in order to give a more detailed insight into precipitation behavior.

Authors: HUGENSCHMIDT, Christoph; CHRYSSOS, Leon

Presenter: CHRYSSOS, Leon

Session Classification: Friday Morning

Track Classification: Main

Contribution ID: **44** Type: **Talk**

Characterization of hydrogen storage materials

Wednesday, June 1, 2022 9:55 AM (20 minutes)

Hydrogen can be stored reversibly, safely and with high hydrogen densities in light metal hydrides. Because of their sensitivity to hydrogen, neutrons are the ideal probe for characterizing metal hydride materials and tank systems. Neutron imaging was used to study hydrogenation processes in metal hydride powders. In addition, neutron and X-ray scattering methods have been used to study phase transformations and changes in nanostructure in various hydrogen storage systems to gain deeper insight into the complex hydrogen sorption processes. A high-pressure cell for in situ neutron studies was developed to characterize processes at high pressures and temperatures. The results will enable the optimization of hydrogen storage systems in terms of capacity, kinetics and safety.

Author: Dr PRANZAS, P. Klaus (Helmholtz-Zentrum Hereon)

Co-authors: Dr BÖRRIES, Stefan (HZG); Dr KARIMI, Fahim (Helmholtz-Zentrum Hereon); Mrs ASLAN, Neslihan (Helmholtz-Zentrum Hereon); Mr METZ, Oliver (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); Dr PISTIDDA, Claudio (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); Dr DORNHEIM, Martin (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); Prof. KLASSEN, Thomas (Institute of Hydrogen Technology, Helmholtz-Zentrum Hereon); Prof. MÜLLER, Martin (Institute of Materials Physics, Helmholtz-Zentrum Hereon)

Presenter: Dr PRANZAS, P. Klaus (Helmholtz-Zentrum Hereon)

Session Classification: Wednesday morning

Track Classification: Main

Contribution ID: **45** Type: **Poster**

Influence of build direction on residual stresses and textures in lightweight alloys produced by additive manufacturing (AM)

Wednesday, June 1, 2022 6:10 PM (20 minutes)

The freedom in complexity of AM metal parts allows very stiff and extreme light designs with real parts made of AlSi11Mg0.5 alloy entering serial production in automotive industry amongst others. Nevertheless, the production process of selective laser melting (SLM) is prone to the formation of residual stresses (RS), which can be large enough to destroy the part already during manufacturing. Furthermore, the parts may have distortions and must undergo a subsequent heat treatment to release RS, which is not always possible and cost as well as energy intensive. Therefore, a pro-found understanding of the formed RS is mandatory for structural integrity assessment and successful industrial manufacturing suitable for air, space and vehicle industry. The SLM production process is closely linked to texture formation in building direction of AM parts, which results in anisotropic mechanical properties.
In the current presentation, we investigate the evolution of residual strains and texture of an additively manufactured AlSi11Mg0.5 alloy component as function of building directions. High energy synchrotron X-rays are used to assess the strains of the different phases in this alloy and corresponding pole figures are derived to correlate the texture with the resulting stress profiles. In addition the influence of heat treatment on the strain level is studied as a function of spatial position within the AM parts. A brief outlook how this results can be used to qualify and mitigate stress induced failure mechanism in real parts will also be given.

Authors: WALZ, Erik; LANDESBERGER, Martin (TUM); Dr HOFMANN, Michael (Technische Univesität München); PARMAR, Priyank; GAN, Weimin (Helmholtz-Zentrum Hereon)

Presenter: Dr HOFMANN, Michael (Technische Univesität München)

Session Classification: Posters

Track Classification: Main

Contribution ID: **46** Type: **Talk**

Investigation in the onset of plasticity in high strength metal sheets used in automobile industry

Friday, June 3, 2022 9:00 AM (20 minutes)

In view of efficiency and resource scarcity, lightweight construction is an important issue for automotive industry. Here, the use of high- and ultra-high-strength steels offer enormous weight reducing potential in car body construction. However, a manufacturing disadvantage of these materials is the strong elastic springback due to the high yield strength and the associated difficulties with regard to dimensional accuracy. Numerical prediction of springback in formed metal sheets is still a challenge nowadays. Especially in high strength steel sheet metals, the modelling of elastic behaviour is crucial in cases where the non-linearity and decrease of Young's modulus cannot be neglected during sheet forming.
In the current study a temperature-dependent evaluation method (TDEM) [1] is investigated and further improved. Matching the thermo-elastic effect with the microscopic material behaviour shows that the temperature minimum can be considered as the onset of yielding, i.e. the maximum yield stress at zero plastic strain (YS0). The assumption is that the temperature minimum is the equilibrium between elastic cooling and plastic heating induced by deformation. To verify this hypothesis, lattice strains for mild steels as well as for high strength steels are measured in-situ using synchrotron radiation, while monitoring the temperature changes in the respective samples. The suitability and validity of the thermo-elastic effect for material characterization and the determination of the onset of yielding has been proven by following and comparing the evolution of the measured lattice strains and the dislocation density during loading and unloading. The results clearly indicate that the thermo-elastic effect yields suitable values for the correct determination of the onset of yielding in sheet metals, which is an essential parameter for predicting springback in current material models.

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Authors: Prof. BATISTA, António C. (University of Coimbra); MAAWAD, Emad (Hereon); REBELO-KORNMEIER, Joana; Mr VITZTHUM, Simon (utg, TUM); Mr HARTMANN, Christoph (utg, TUM); Mr GRUBER, Maximilian (utg, TUM); Prof. VOLK, Wolfram (utg, TUM)

Presenter: Mr VITZTHUM, Simon (utg, TUM)

Session Classification: Friday Morning

Track Classification: Main

Contribution ID: **47** Type: **Poster**

Texture and microstructure evolution of TNM alloy during hot compression

Wednesday, June 1, 2022 6:10 PM (20 minutes)

Owing to low density (3.8-4.0g/cm3), high specific strength and stiffness, excellent creep resistance, and good corrosion resistance, the β-solidifying TNM alloys with properly aligned (α2+γ) lamellar structure have been considered as excellent candidates for modern turbine blades. Recently, it has been evidenced that when the γ lamellae are oriented to the load direction the mechanical properties of the alloys can be greatly increased, thus, lamella orientation control has become an interesting topic for property optimization.
In the present work, the microstructure and texture of a TNM alloy (Ti-43Al-4Nb-1Mo-0.1B (at%) hot compressed to different strains at various strain rates were characterized by synchrotron radiation diffraction at a macroscopic scale to obtain the bulk texture information and by SEM EBSD at mesoscopic scale to analyze with respect to the orientation relationships and microstructure stability. Results have shown that the microstructure at the initial state was composed of grains from three different phases, BCC β phase, hexagonal α phase and tetragonal γ phase. After the uniaxial compressive hot deformation at 1280℃ to different deformations at different strain rates, it seems that (1) the microstructure changed and the α to lamellar (α+γ) was inhibited with the increase of the strain rate. The microstructure became mainly composed of the alpha phase. (2) All three phases can be textured. For each phase, a different texture was obtained and the change of microstructure was revealed in details thanks to the complementary characterization methods. In conclusion, texturization of the TNM alloy seems to be possible thanks to the hot compression. It leads to different types of microstructures depending on the amount of deformation and the strain rates as well as the used temperature.

Keywords : TNM TiAl alloy, texture, microstructure, synchrotron radiation diffraction, SEM-EBSD

Authors: KEITA, Mohamed; Dr SOLIS, Cecilia; Dr ZHANG, Yudong (CNRS); Dr GAN, Weimin (Helmholtz-Zentrum Hereon); Prof. BOUZY, Emmanuel (LEM3 University of Lorraine)

Presenter: KEITA, Mohamed

Session Classification: Posters

Track Classification: Main

Contribution ID: **48** Type: **Talk**

Doping effect on the hydrogen production via microwave assisted water splitting in doped-ceria materials.

Wednesday, June 1, 2022 3:45 PM (20 minutes)

Nowadays, hydrogen is being used as an energetic vector for saving excess of renewable energy. The most used techniques to generate hydrogen are thermochemical looping's, electrolysers and hydrocarbons reforming. However, all these techniques have several drawbacks, namely the high temperatures needed, the use of sophisticated machinery and the long operation times required for the hydrogen production. Recently, the possibility to generate green hydrogen using electric energy, as microwave radiation, has been reported. This process uses metallic oxides as catalysts, e.g. CeO2, and it occurs in a reactor at temperatures lower than 250 ℃ in less than five minutes. The hydrogen production mechanism happens in two steps. First, the material is irradiated with a microwave electromagnetic field, producing the reduction of the material with the concomitant release of oxygen. This radiation is able to stabilize a higher amount of oxygen vacancies in the fluorite structure at lower temperatures than the conventional radiative processes, as it is the case of thermosolar (>1000 ℃). When the microwaves are turned off in the presence of water, the material splits the H2O molecule, therefore liberating a stream of molecular hydrogen and filling its oxygen vacancies. One example of this process is noted in the material Ce0.8Gd0.2O2. The release of oxygen is accompanied by an increase in the material electrical conductivity. Besides, it has been observed that a different conductivity behaviour can be inferred depending on the irradiated microwave power. If the microwave radiation power is lower than the activation energy (PTh), the material conductivity behaves like conventional heating process. On the other hand, for a microwave radiation power higher than the PTh, the material undergoes a sudden spike in the conductivity. This rise is mainly ascribed to an increase of the electronic conductivity. Ionic conductivity can be tuned by doping the ceria lattice with iso and aliovalent cations. For example. incorporating Zr into the structure, the ionic conductivity decreases, while Gd generates oxygen vacancies in the anionic sublattice, thus, increasing the ionic conductivity. In this work, we have synthesized and characterized a set of ceria doped materials, e.g. Ce1-xMxO (M = La, Y, Yb, Tb, Zr, and Gd) (x = 0.1 and 0.2). Their interaction with microwave radiation has been monitored to study their modulability regarding conductivity behaviour and hydrogen production capacity.

Authors: Mr DOMÍNGUEZ SALDAÑA, Aitor (Intituto de tecnología Química); Dr SANTOS, Joaquín (Instituto de Tecnología Química); Dr CATALÁ, José Manuel (Institute of Information and Communication); Dr SERRA, José Manuel (Instituto de Tecnología Química); Dr NAVARRETE, Laura (Instituto de tecnología química); Dr BALAGUER, María (Instituto de Tecnología Química); Dr PLAZA, Pedro José (Institute of Information and Communication)

Presenter: Mr DOMÍNGUEZ SALDAÑA, Aitor (Intituto de tecnología Química)

Session Classification: Wednesday afternoon

Track Classification: Main

Contribution ID: **49** Type: **Poster**

Energy Landscape for Li-Ion Diffusion in the Garnet Type Solid Electrolyte material $Li_{6.5}La_3Zr_{1.5}Nb_{0.5}O_{12}$ (LLZO-Nb)

Wednesday, June 1, 2022 6:10 PM (20 minutes)

The detailed investigation of innovative solid electrolytes featuring promising properties, such as a high ionic conductivity, that make it suitable for an application in next-generation batteries is one of the key strategies to expand the understanding of corresponding structure-property-relationships which than allows for further tailoring of the materials properties as demanded.
Here, we report on the investigation of the well-known and commercially available material $Li_{6.5}La_3Zr_{1.5}Nb_{0.5}O_{12}$ (LLZO-Nb) by powder X-ray and powder neutron diffraction as well as by temperature-dependent synchrotron powder diffraction experiments. Based on the experimental neutron data the Li-ion diffusion pathways are analyzed applying the maximum entropy method as well as the one-particle potential formalism. The obtained results allow for a visualization of the energy landscape for Li-ion motion within the garnet structure.

Author: Dr STRANGMÜLLER, Stefan (Technische Universität München - Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II))

Co-author: SENYSHYN, Anatoliy (Technische Universität München - Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II))

Presenter: Dr STRANGMÜLLER, Stefan (Technische Universität München - Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II))

Session Classification: Posters

Track Classification: Main

Contribution ID: **50** Type: **Talk**

Dynamic lithium distribution in 18650-type Li-ion batteries on multiple length scales

Wednesday, June 1, 2022 11:30 AM (20 minutes)

Lithium-ion batteries as energy sources in different kinds of portable electronics and electric vehicles play an important role in modern society. Permanent demand for improved energy/power densities and longevity results in the increasing engineered complexity of Li-ion batteries on different scale levels. Such complexity requires the application of non-destructive probes for characterization of batteries.
In-operando investigations of lithium-ion batteries (as closed electrochemical systems) are often not trivial as only a few methods exist to probe the conditions of the active materials locally in a non-destructive way. In such context diffraction-based methods are very popular in battery research (especially when combined with electrochemistry), either in *ex-situ* or *in-operando* modes. In the current work the dynamic lithium distribution in a high-power 18650-type lithium-ion battery has been investigated on multiple length scales using both neutron and synchrotron based X-ray diffraction. The lithium distribution during dis-/charging has been investigated using spatially-resolved neutron powder diffraction on the graphite anode. Higher-resolution *in-situ* investigation in the fully charged state on both anode and cathode has been performed applying synchrotron-based X-Ray diffraction computed tomography. Finally, the dynamic lithium concentration in a single cathode layer has been investigated using synchrotron radiation with μm-sized beam. Non-uniform lithium distributions were observed on all investigated length scales, which was attributed to the complex character of current distribution in the studied high-power Li-ion battery. Such knowledge is highly relevant for further improvement of current state-of-the-art lithium-ion batteries in terms of effective use and understanding of issues related to enhanced power fade, fatigue and stable operation.

Author: PETZ, Dominik

Co-authors: SENYSHYN, Anatoliy; MÜLLER-BUSCHBAUM, Peter (TU München, Physik-Department, LS Funktionelle Materialien)

Presenter: PETZ, Dominik

Session Classification: Wednesday morning

Track Classification: Main

Contribution ID: **51** Type: **Talk**

Microstructure and texture evolution for high-temperature α phase in extruded β-containing TiAl alloy

Friday, June 3, 2022 9:20 AM (20 minutes)

Increasing demands on modern turbines require ($\alpha 2+\gamma$) lamellar-structured TiAl alloys with fine colony size and properly aligned lamellae. As the lamellar structure is formed by the $\alpha \rightarrow \alpha 2+\gamma$ phase transformation obeying Blackburn OR, the characteristics of lamellar structure depends directly on the high-temperature α phase. Thus, the lamellar structure optimization could be realized by the modification of high-temperature α phase through thermomechanical processing. In this work, the microstructure and texture evolution of high-temperature α phase in TNM alloy during hot extrusion at ($\alpha+\beta$) phase field was investigated by high energy X-ray diffraction (HEXRD) and SEM electron back scatter diffraction (SEM-EBSD). Results show that with a small extrusion ratio (E2.25), the microstructure exhibits uniform and equiaxed α grains with a weak $\langle 11\bar{2}0\rangle$//ED fiber texture. With the increase of extrusion ratio, the microstructure tends to exhibit bimodal structure (E7.11) consisting of deformed grains, fine primary DRXed grains with $\langle 10\bar{1}0\rangle$//ED, as well as coarse grown grains with $\langle 11\bar{2}0\rangle$//ED. The microstructure and texture evolution are resulted from a combination of extrusion parameter and the GB β phase. The increasing extrusion ratio, on one hand, increases the deformation degree and the extrusion rate, so that the considerable stored energy cannot be released in a short time. On the other hand, the large extrusion ratio elongates the GB β phase leading to more α/β interfaces which served as pining points inhibited the low-angle boundaries to evolve into high-angle boundaries. Both of them keep more deformed α grains orientated with $\langle 10\bar{1}0\rangle$//ED retained in the sample with high extrusion ratio. Accordingly, a preferred grain growth happened to the <11-20>-orientated grains due to the high interface energy.
Keywords: TiAl alloy, extrusion, texture, high energy X-ray diffraction (HEXRD), SEM-EBSD

Author: Dr ZHANG, Yudong (LEM3 UMR 7239 CNRS, Université de Lorraine)

Co-authors: Dr QIANG, Fengming (University of Lorraine); Prof. KOU, Hongchao (Northwestern Polytechnical University, China); Prof. LI, Jinshan (Northwestern Polytechnical University, China); Prof. BOUZY, Emmanuel (University of Lorraine, France)

Presenter: Dr ZHANG, Yudong (LEM3 UMR 7239 CNRS, Université de Lorraine)

Session Classification: Friday Morning

Track Classification: Main

Contribution ID: **52** Type: **Poster**

Structural response of electrochemical cycled $Li_xNi_{0.8}Co_{0.15}Al_{0.05}O_2$ (0<x<1) battery cathodes

Wednesday, June 1, 2022 6:10 PM (20 minutes)

In view of increasing greenhouse gas emissions, the demand for renewable energies is rising in parallel with the demand for energy storage materials. Thus, the development of lithium ion batteries with high nickel content paves the way towards better cycling stability, higher energy and power densities at lower costs. One of them is the commercially available state-of-the-art 18650-type battery of $Li_xNi_{0.8}Co_{0.15}Al_{0.05}O_2$ (NCA) and graphite chemistry, also used in the Tesla Model S of 1st generation [1,2]. The overall cell performance to large extent is limited by the diffusivity of Li-ions in the NCA structure, which becomes important in the context of the on-going discussion about occurrence of antisite disorder in nickel rich cathode materials, where the exchange of Ni^{2+} and Li^{+} leads to the blocking the diffusion pathways of Li-ions. Such processes typically result in the rapid degradation of the cell capacity as well as in structure instabilities of various kinds [3,4]. Especially for NCA materials, presence of antisite defects at different lithiation levels is often controversial and studied poor in literature. Therefore, in the present contribution the structural response of delithiated NCA electrode materials obtained from commercial 18650-type lithium-ion batteries was investigated by a systematic ex-situ powder diffraction study on the instrument SPODI at the FRM II reactor in Garching. The structural parameters were modeled by full-profile Rietveld refinement. During electrochemical cycling, the lithium occupancy was found to decrease linearly with higher charge states, which was confirmed by chemical ICP-OES measurements, that agree well with the on-going delithiation. The remaining transition metal occupancies showed consistent behavior during electrochemical cycling, demonstrating the absence of Li/Ni antisite defects in the structure of NCA materials.

Author: HÖLDERLE, Tobias

Co-authors: Prof. MÜLLER-BUSCHBAUM, Peter (TU München, Physik-Department, LS Funktionelle Materialien); Dr SENYSHYN, Anatoliy

Presenter: HÖLDERLE, Tobias

Session Classification: Posters

Track Classification: Main

Contribution ID: **53** Type: **Talk**

Welcome Address

Tuesday, May 31, 2022 3:00 PM (20 minutes)

Presenter: Prof. MÜLLER, Martin (Helmholtz-Zentrum Hereon)

Session Classification: Tuesday afternoon

Track Classification: Main

Contribution ID: **54** Type: **Invited talk**

Time to act!

Tuesday, May 31, 2022 8:30 PM (45 minutes)

Abstract:

The effects of climate change are becoming more and more tangible. Especially in recent years extreme events such as heat waves, heavy rains and flood events are becoming more frequent. Based on the results of the Intergovernmental Panel on Climate Change (IPCC), it is clear that in some regions of the world there would be fewer droughts, less heat waves, heavy rain events and floods if global warming could be limited to 1.5 ℃ instead of 2 ℃ by the end of this century. This poses new challenges for regions and many sectors. It is therefore high time to urgently drive forward climate protection and adaptation to the effects of climate change and to avoid any further warming, because every half degree count.

Presenter: Prof. JACOB, Daniela (Hereon)

Session Classification: Evening Talk

Contribution ID: **55** Type: **not specified**

Discussion / Concluding Remarks

Friday, June 3, 2022 11:30 AM (30 minutes)

Session Classification: Friday Morning

Contribution ID: **58** Type: **Talk**

Engagement with the Mobility Sector at ISIS Neutron and Muon Source

Friday, June 3, 2022 10:00 AM (20 minutes)

Traditionally used by academic researchers, neutron facilities also provide commercial advantages to those companies which are aware of the unique benefits of neutron scattering techniques for their research and innovation. The advanced techniques provided by neutron sources are powerful tools for multi-scale material and process characterisation down to atomic detail. As such, they are useful for a wide range of industry sectors for developing new advanced materials, new innovative products, and more efficient manufacturing processes. Within the mobility sector, ISIS Neutron and Muon Source has many collaboration points with industrial users, which clearly demonstrate the impact that neutron science has to business sectors ranging from optimisation of petrochemicals to integrity of rocket engine components, and from lightweighting of aircraft wings, to resilience of self-driving cars.
In order to improve its offer to industrial users, in 2011 ISIS established its flexible "Industrial Collaborative Research and Development"(ICRD) Route by which industrial users are quickly granted ISIS beamtime based on potential economic benefit to the UK, rather than based purely on scientific merit. To date, this access route has been used by more than 50 companies, and has resulted in an additional 50 days of beamtime per year allocated to industrial users. These have resulted in high quality case-studies which illustrate the impact of neutron scattering techniques to the mobility sector, while the economic data provided by the industrial users have formed the basis for assessment of ISIS contribution to the UK's economy. This presentation will summarise diverse examples of industrial measurements at ISIS within the mobility sector, and will demonstrate how these examples help to quantify the contribution of ISIS to economic growth in the UK.

Author: Dr APPLEBY,, Graham A. (ISIS Neutron and Muon Source, Rutherford Appleton Laboratory)

Co-author: FROST, C. D

Presenter: Dr APPLEBY,, Graham A. (ISIS Neutron and Muon Source, Rutherford Appleton Laboratory)

Session Classification: Friday Morning