

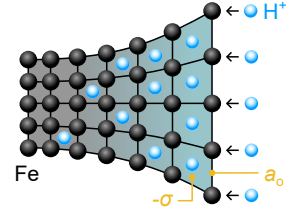
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TIME- AND DEPTH-RESOLVED CHARACTERIZATION OF HYDROGEN DIFFUSION INTO DUPLEX STEEL

Lattice Swelling and Stress Evolution

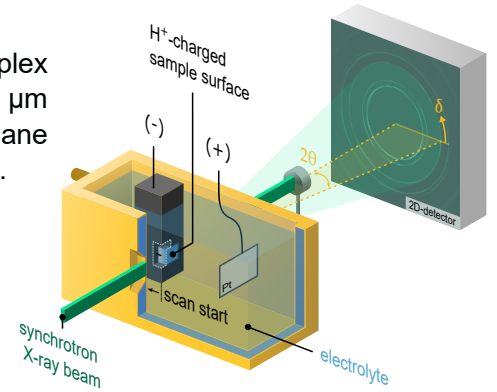
Amidst the increasing interest in hydrogen, it faces the challenge of balancing its potential as one of the most promising green energy sources and its detrimental impact on mechanical properties when interacting with steel components. This contribution addresses the significant issues stemming from hydrogen embrittlement in steels, offering support in this area with an innovative **SYNCHROTRON-BASED CHARACTERIZATION METHOD**.



Experimental

The analysis is based on in-situ electrolytic hydrogen (H) charging of a duplex steel sample, which underwent repeated line scanning until a depth of 500 μm for multiple hours. Debye-Scherrer rings are then used to calculate in-plane stresses and unstrained lattice parameters using the software package pyFAI.

- Duplex steel
- In-situ electrolytic hydrogen charging
- HE XRD cross-sectional analysis of in-plane stresses & unstrained lattice parameters

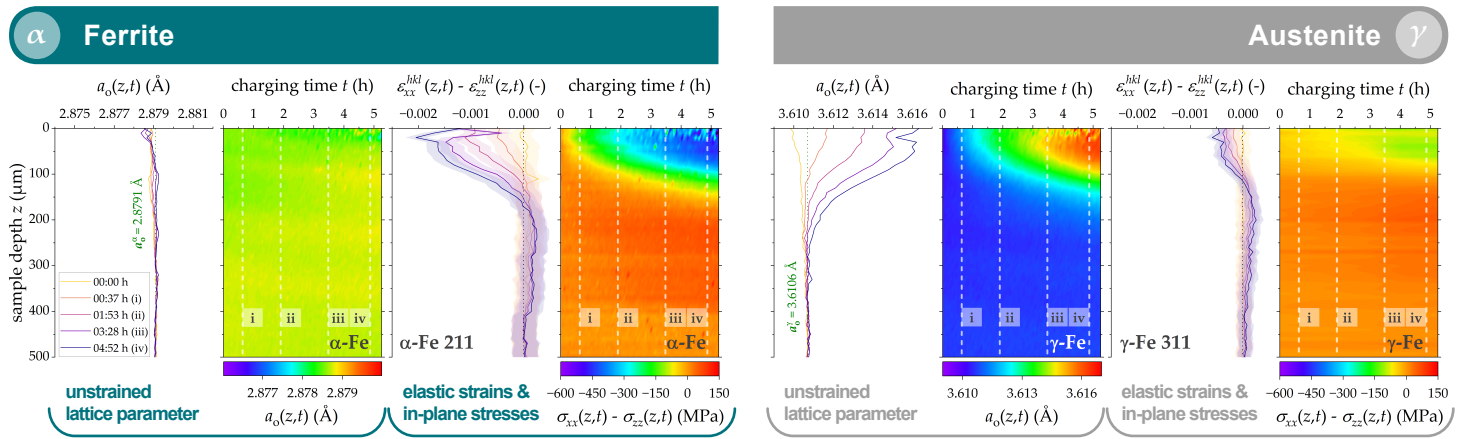


$$\frac{\partial d^{hkl}(\delta, z)}{\partial \sin^2 \delta} = \frac{1}{2} S_2^{hkl} \cdot (\sigma_{xx}(z) - \sigma_{zz}(z)) \cdot d_0^{hkl}(z)$$

↪ strain-free direction δ_0

Results

The developed method enables the decoupling of isotropic and anisotropic stresses, revealing that solely the austenite phase absorbs hydrogen in the investigated material, as inferred from unstrained lattice parameters. Consequently, these lattice expansions induce surface-near compressive stresses of up to -600 MPa in the ferrite and -150 MPa in the austenite phases.



Conclusion • First in-situ observation of H-absorption • H primarily stored in γ • Surface-near compressive stresses

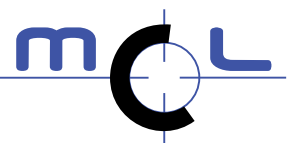


Dipl.-Ing.
Thomas Pogrietz

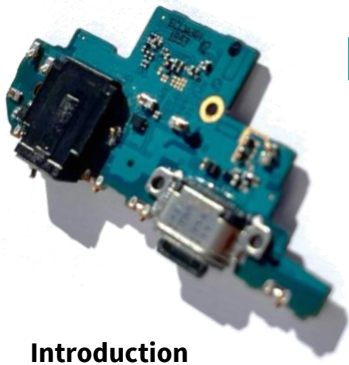
Department of Materials Science
Chair of Materials Physics
thomas.pogrietz@unileoben.ac.at



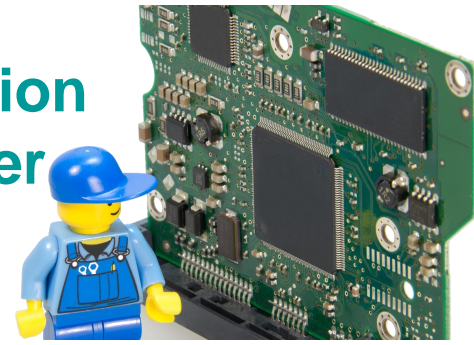
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Micromechanical evaluation of microelectronic solder bumps

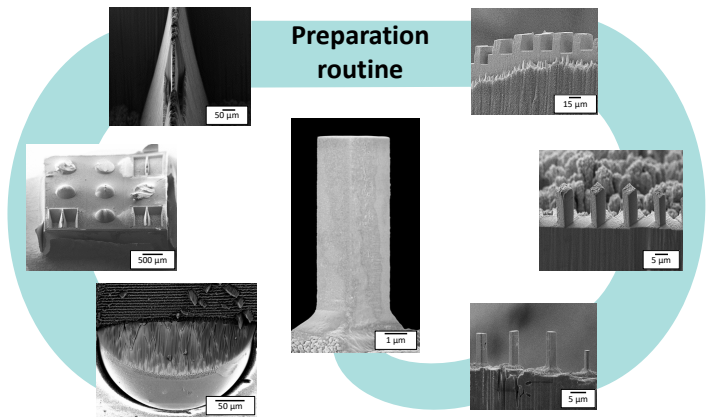
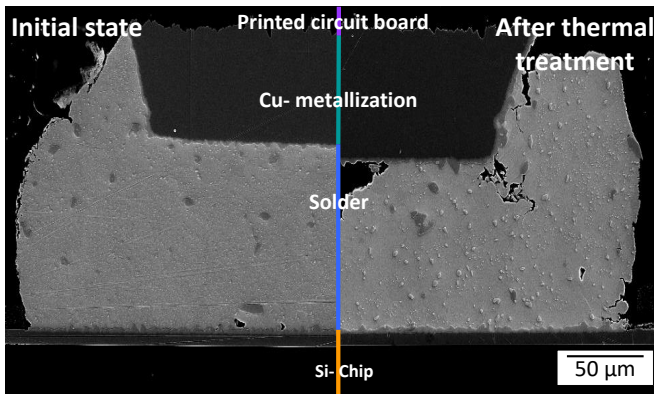


Introduction

Modern electronic packages have to withstand various mechanical and thermal stresses due to their range of applications. Here SAC305, a promising candidate as lead-free soft solder material, is micromechanically investigated by advanced nanoindentation experiments and in-situ microcompression experiments.

Microcompression experiments

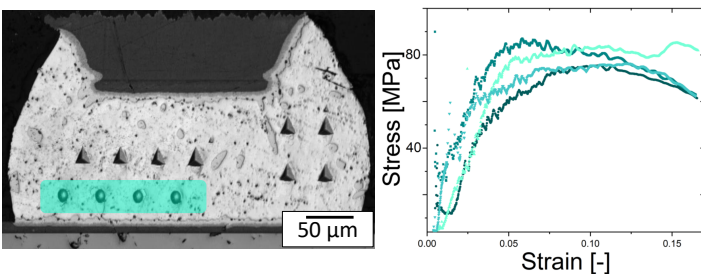
Microcompression experiments enable us to determine the mechanical properties of single crystallin beta-tin, which allows the deformation behavior of the matrix to be better understood. Therefore, rectangular micropillars with an estimated cross-section of $5 \mu\text{m} \times 5 \mu\text{m}$ were fabricated using a focused ion beam microscope



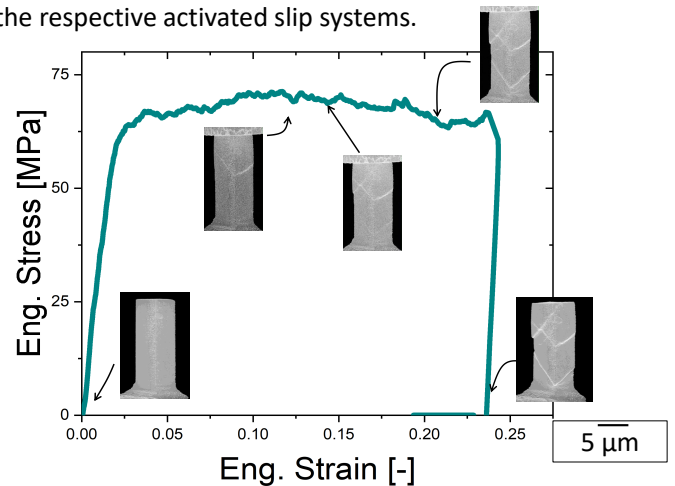
Nanoindentation Experiments

To introduce mechanical stresses mimicking the application conditions, the samples undergo two different heat treatments that lead to a change in the microstructure. Subsequent advanced nanoindentation experiments, spherical flow curves, strain-rate jump tests and creep experiments offer the opportunity to analyze the time dependent material behaviour over a wide range of strain rate.

Spherical flow curves



By carrying out the experiments in a scanning electron microscope, it is possible to combine the mechanical data with the recorded video to subsequently identify the formed slip steps. By testing different orientations, it is possible to calculate critical resolved shear stress (CRSS) for the respective activated slip systems.



Micro- & Nanomechanics
 Dipl.-Ing.
 Nadine Buchebner
 Department Materials Science
 Chair of Materials Physics
 nadine.buchebner@unileoben.ac.at

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Micromechanics of nano-3D-prints

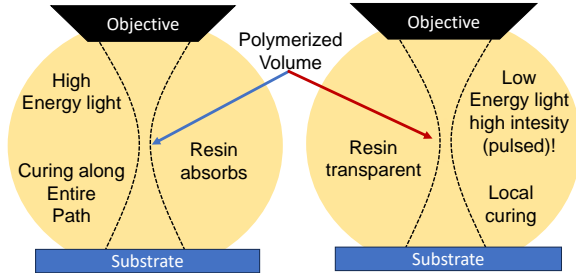
Miniaturized experiments on two-photon lithography derived specimens

What is the motivation?

- Two-photon lithography enables micron sized 3D prints with several nanometers precision.
- Mechanical properties are important for structure design and stability.
- Mechanical properties are accessible through mechanical experiments.
- Small structures require miniaturized mechanical experiments within a scanning electron microscope

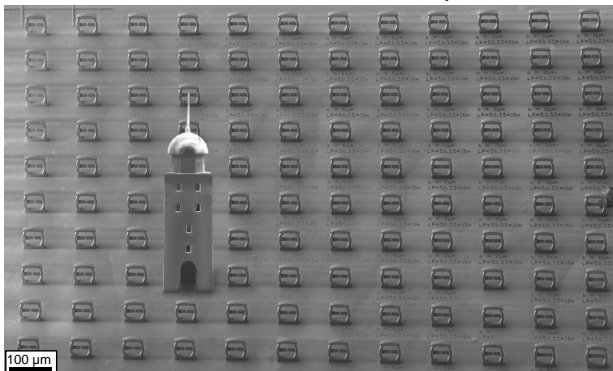
How are the specimens printed?

Standard (resin) printing Two-photon Lithography



What are the possibilities?

- High shaping freedom
 - Ready to test specimens
 - High reproducibility
 - Print-job automatization
- High throughput experiments



What is printed how are they tested?

	Tension specimens	Compression specimen Push- to-pull	Cantilever bending
Module			
Specimens printed			
Tested specimen			
Results	<ul style="list-style-type: none"> • Adhesion • Force, Energy • Strength • Yield, UTS 	<p>Evaluation</p> <ul style="list-style-type: none"> • Toughness • Essential Work of fracture 	<ul style="list-style-type: none"> • Toughness • K_{IQ} • J_{IQ} • J vs. da

Issues solved and fine tuning

- Overhanging feature → Taper (see above)
- Low stability → Higher power
- Low adhesion → Surface treatment



Micro- & Nanomechanics
 Dipl.-Ing.
 Alexander Jelinek
 Department Materials Science
 Chair Materials Physics
 alexander.jelinek@unileoben.ac.at

Acknowledgement



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Cracking the TiAl Code

An Interplay of Molecular Dynamics Simulations and Experimental Observation

Advanced TiAl alloys show superior performance in aerospace and automotive applications than conventional materials but lack ductility at room temperature. The fracture properties of a TNM⁺ alloy were studied using micro- and nanoscale experiments and molecular dynamics simulations, aiming to enhance the fracture properties of the alloy for broader applications.

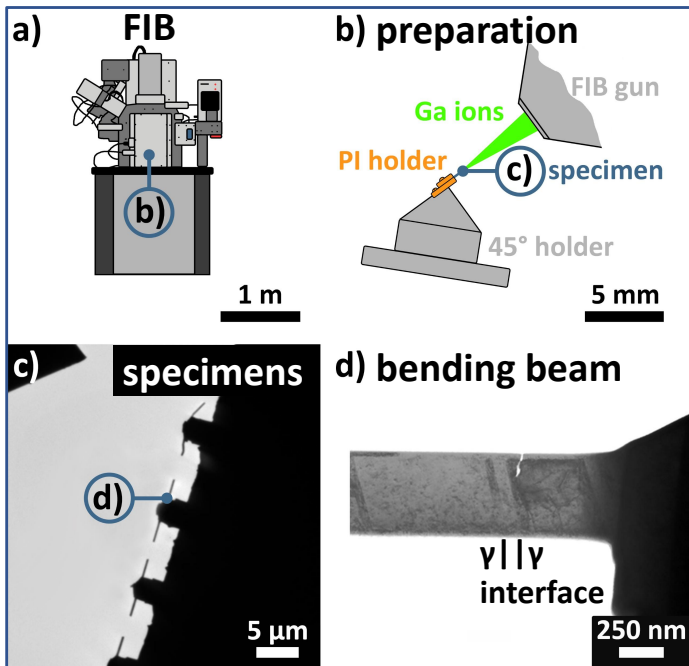


Figure 1: In a), a schematic of the utilized FIB device is presented. b) provides an outline of the specimen preparation on a 45° tilt-holder, housed within a Cu PI holder. c) and d) showcase both an overview and a detailed STEM image of the nanometer-sized bending beams, respectively.

Methodology and Results:

Notched bending beams were prepared by focused ion beam microscopy techniques (see Figure 1) and exhibit a size of 1500 x 300 x 100 nm. Consequently, their size is one-hundredth of the width of a hair. This sophisticated preparation allows the determination of different stages of the crack propagation, which are analysed by in-situ transmission electron microscopy and complementary molecular dynamic simulations. Furthermore, the current strain state was recorded by 4D scanning techniques at certain stages of the experiment.

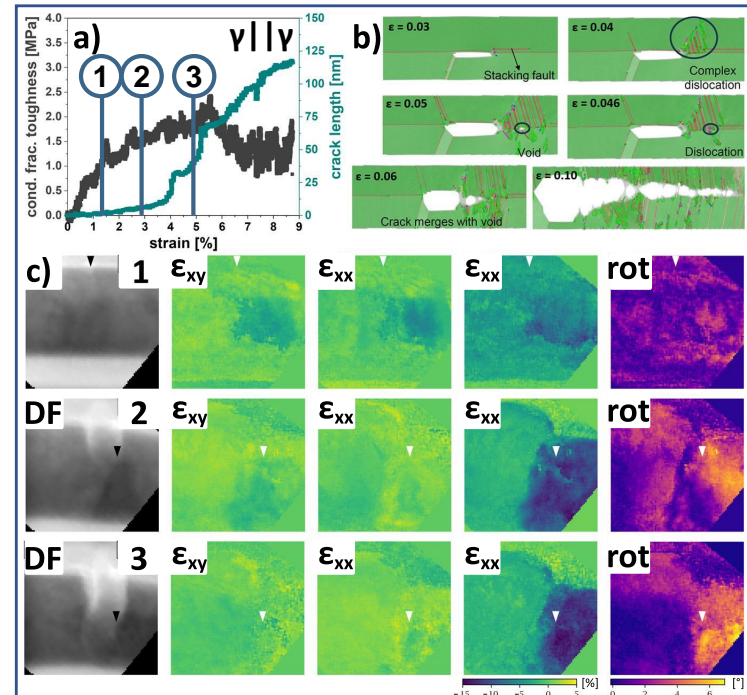


Figure 2: a) conditional fracture toughness and crack length against the calculated strain of a cantilever notched along a $\gamma||\gamma$ interface. b) molecular dynamic simulation of the crack propagation along a $\gamma||\gamma$ interface. c) measured strain maps and calculated rotation from an in-situ fracture experiment.

Conclusions and Outlook:

The combination of different in-situ methods provides new insights into the fracture behaviour of this semi-brittle material system. Based on the conditional fracture toughness, fracture strain, dislocation emission behaviour and simulated pre-crack tip void formation, new design criteria can be developed. The improved micro- and macroscopic mechanical properties of the next generation of TiAl alloys may therefore enable their use in a wider range of applications.



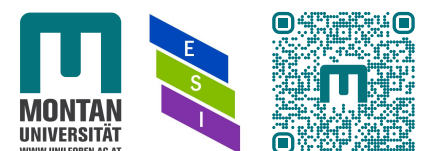
Dipl.-Ing. Dr. mont.
Michael Burtscher
Materials Science
Materials Physics
michael.burtscher@unileoben.ac.at

Acknowledgement:

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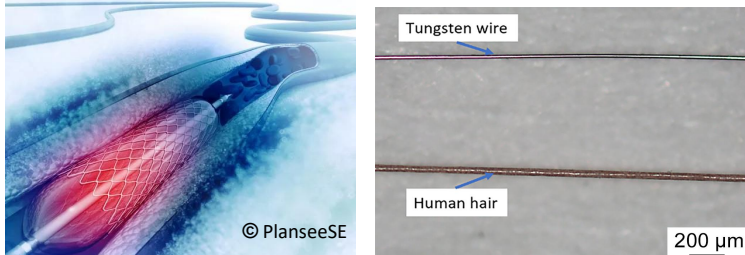
Where research meets the future!

Doped tungsten for applications in extreme environments

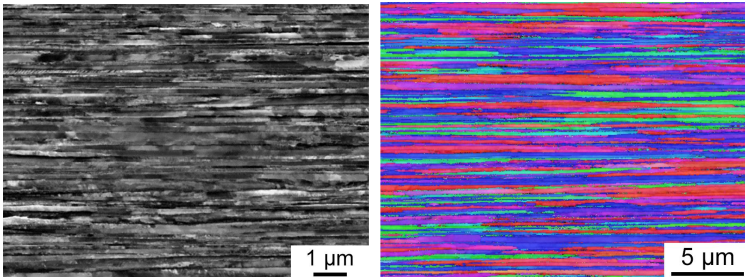
Employing materials in extreme environments requires investigation of novel concepts in materials design to achieve respectable service life in challenging conditions. Such applications necessitate a profound understanding of structure-property relationships, involving the investigation of damage and failure mechanisms on the microscale.

Tungsten fine wire

Tungsten fine wires are used in highly-specialized fields, such as surgical instruments and silicon crystal pulling. Their microstructure features highly elongated grains, resulting in anisotropic mechanical properties.



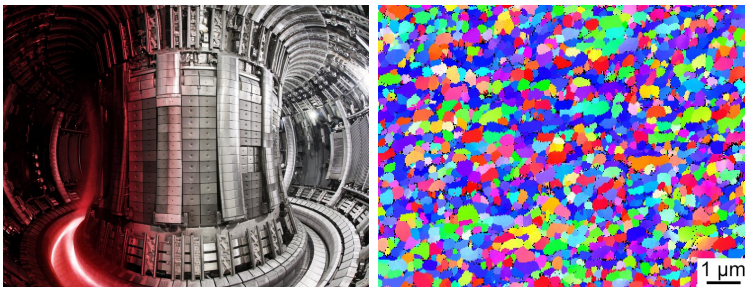
Left: stent, right: comparison of tungsten wire with human hair.



Left: Scanning electron microscope (SEM) micrograph of the elongated microstructure, right: electron backscatter diffraction (EBSD) micrograph of the microstructure.

Tungsten for fusion applications

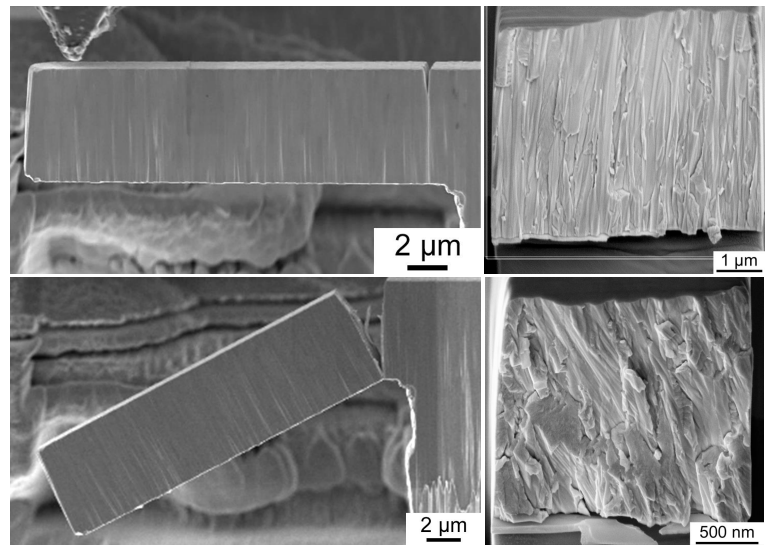
Bulk tungsten is used as plasma-facing material in fusion reactors (e.g. Tokamaks), where it is subjected to heat fluxes of up to 20 MW/m², particle bombardment, and temperature cycling.



Left: Fusion reactor, right: EBSD micrograph of the ultrafine-grained bulk tungsten microstructure.

In-situ micromechanical testing

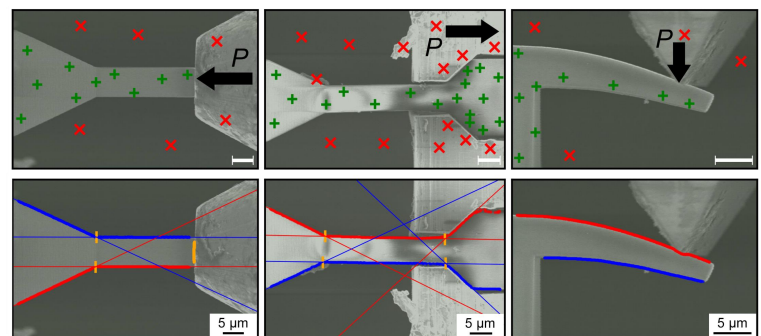
In-situ micromechanical testing proves to be a powerful tool for microscale characterization of mechanical performance, deformation mechanisms and fracture.



Microcantilever testing (left column) and SEM micrographs of fracture surfaces (right column).

Advanced computer vision

Recent developments in object detection and image segmentation enabled by artificial intelligence allow to continuously track specimen deformation. Data evaluation routines incorporating this approach can correct experimental inaccuracies and provide image-based quantification.



Three different experimental geometries tracked using image segmentation.

Micro- and Nanomechanics
Dipl. Ing.
Julius Keckes
Department Materials Science
Chair of Materials Physics
julius.keckes@unileoben.ac.at

Micro- and Nanomechanics
Dipl. Ing.
Hannah Lichtenegger
Department Materials Science
Chair of Materials Physics
hannah.lichtenegger@unileoben.ac.at

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On the Road to Characterize Small Scale Failure

Automatic visual detection of fracture characteristics from *in situ* fracture experiments below the micron scale

The ongoing trend of device miniaturization and decreasing internal length scales requires testing of material properties. This includes fracture behavior, which must be examined on increasingly smaller length scales, reaching down to the micron and sub-micron regime. Experiments are conducted to gain insights into the material- and fracture behavior at this scale.

In Figure 1a), the setup of such an experiment is shown. A wedge applies a force on a pre-notched cantilever with crack of length a . On the z-axis, an electron microscope is mounted. Figure 1b) shows a single frame, with the wedge visible in the upper right corner. The marked green area represents the region of interest. The frames are recorded as a force is applied to the cantilever (Figure 1c). Manual measurements in these frames are a tedious task and rely heavily on human expertise. Therefore, we developed a system that extracts essential fracture characteristics from all images automatically (Figure 1c). The important fracture characteristics are the crack length a , the crack tip opening displacement (CTOD, δ), and -angle (CTOA, α) (Figure 1d).

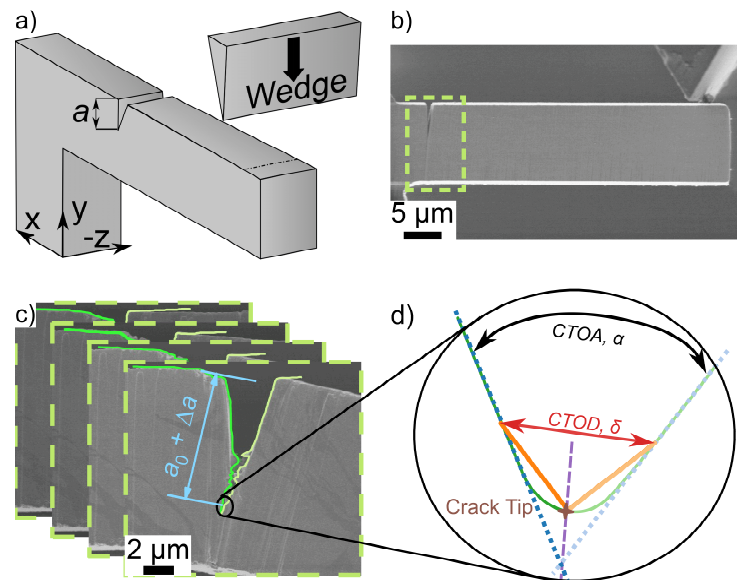


Figure 1: Illustration of in situ performed micro-mechanical experiments a) Schematic view of a test sample. b) Specimen before testing marked with the region of interest. c) Evaluated frames with the found contours by the algorithm. d) Schematic illustration of the essential fracture characteristics.

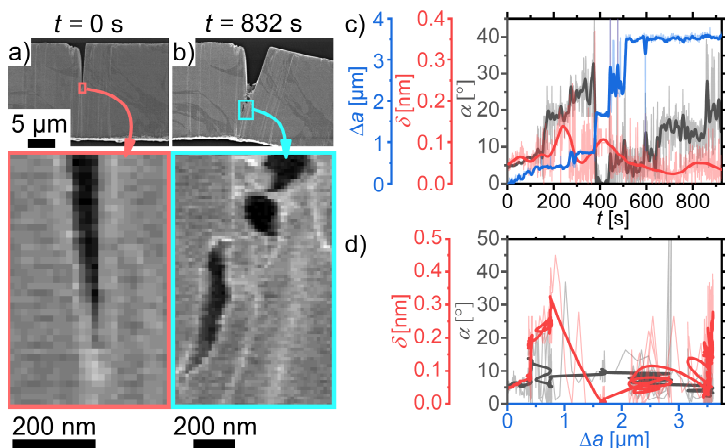


Figure 2: Analysis of the recorded in situ frames. a) Initial frame with the fabricated notch. b) Evolved crack for a semi-brittle fracture. c) Algorithmically determined fracture characteristics. d) Crack growth resistance curve obtained from the determined fracture characteristics.

Figure 2a) illustrates the fracture process of a semi-brittle material during loading. At frame $t = 0$ s, the sample with the initial crack is shown. A detailed view of the crack tip (highlighted by the red rectangle) is provided below. After $t = 832$ s (Figure 2b), the crack has evolved due to the applied force. The detailed view (highlighted by the cyan rectangle) reveals tearing and pore formation. This makes crack extraction challenging, leading to increased scatter of the determined characteristics (Figure 2c). Analysis of the fracture characteristics enables to construct crack-growth resistance curves (Figure 2d), which give further insight into the fracture process. From the data, critical fracture parameters can be evaluated. This allows researchers to compare different materials with respect to their damage tolerance, assess and enhance their applicability in specific scenarios.



Dipl.-Ing.
Klemens Schmuck
Material Science / Material Physics
klemens.schmuck@unileoben.ac.at



Dipl.-Ing. Dr. mont.
Martin Antenreiter
Information Technology
martin.antenreiter@unileoben.ac.at

Acknowledgement



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Where research meets the future!

PBF-LB of Soft Magnetic Fe-Based Metallic Glasses

Felix Römer¹, Parthiban Ramasamy², Florian Spieckermann¹, Martin Stockinger³, Jürgen Eckert^{1,2}

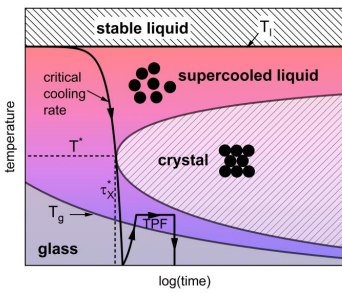
¹Department of Materials Science, Montanuniversität, Jahnstraße 12, 8700 Leoben, Austria

²Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Jahnstraße 12, 8700 Leoben, Austria

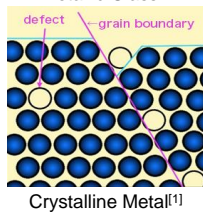
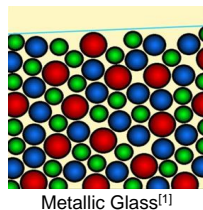
³Department Product Engineering, Montanuniversität Leoben, Franz-Josef-Straße 18, 8700, Leoben, Austria

Reducing the energy losses is a key requirement in the development of soft magnetic materials. Interest has focused on nanocrystalline soft magnetic alloys, whose magnetic properties benefit from their chemical and structural variations at the nanoscale. Laser beam powder bed fusion (PBF-LB) provides high heating and cooling rates, making it suitable for producing metallic glass parts with dimensions much bigger than their critical casting diameter. Understanding the behavior of metallic glasses under these high rates and complex treatments is the key to printing dense, crack-free parts with suitable magnetic properties. In the current study we show the effects of double scanning and random line strategies on the microstructure, crack formation and thermal properties of $Fe_{72.6}Si_{11.3}B_{10.9}Cr_{2.2}C_{3.0}$ (at%).

Metallic Glasses vs. Metals



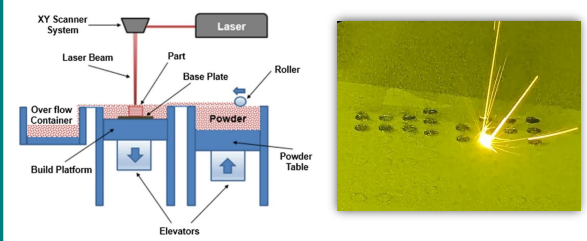
A typical Time-Temperature-Transformation (TTT) curves for the formation of metallic glasses (MGs)^[2].



- + High strength
- + High hardness
- + Excellent tribological properties
- + Excellent corrosion resistance
- + Excellent biocompatibility
- + Very good soft magnetic properties

- Near-zero tensile ductility
- Poor fatigue resistance

Additive Manufacturing PBF-LB



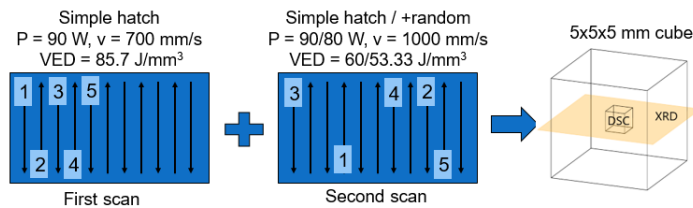
Powder bed fusion PBF-LB process configuration ^[4]

- Layer by layer process
- High cooling rate
- High geometrical freedom
- Complex heat treatment

Sample Geometry and Parameter

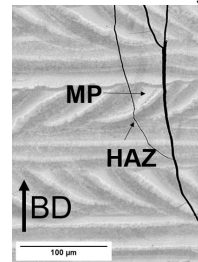
Aconity Midi+ ^[5]

- 1070nm Laser
- 80 μ m Laser spot size
- 50 μ m Layer thickness
- 30 μ m Hatch distance
- Each layer 90° rotation
- Argon atmosphere <100 ppm O₂

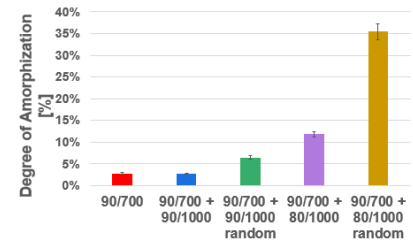


The first scan using higher energy density is used for layer densification. Following this, lower energy density scanning, or a combination of lower energy density and a random line strategy, can increase the content of amorphous phase in the material.

Microstructure, Hardness, and Crystallization



SEM BSE image of the samples microstructure



Degree of amorphization by comparing crystallization enthalpies of the DSC signals.

- Multi-scanning strategies could not counteract cracking.
- Cracks predominantly formed parallel and perpendicular to the build direction (BD).
- Melt pool MP (1621±58 HV_{0.5}) and heat affected zone HAZ (1203±45 HV_{0.5}) showed strong variations in hardness.
- Double scanning with a lower energy density resulted in minor vitrification.

By employing double scanning with a sufficiently reduced energy density and the random line strategy, a degree of amorphization of ~35% could be restored.



Dipl.-Ing.
Felix Römer
Department of Materials Science
Chair of Materials Physics
felix.roemer@unileoben.ac.at

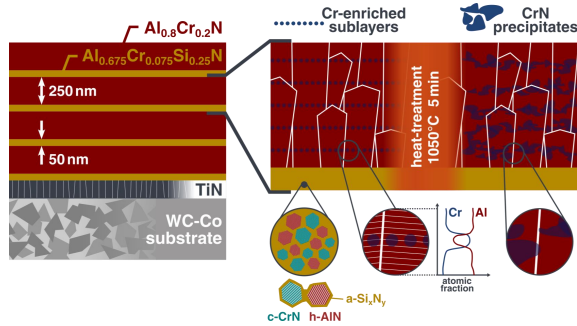
- Research focus:
 - Additive Manufacturing
 - Metallic Glasses
 - Crystallization



ENHANCING TOUGHNESS IN HARD CERAMIC THIN FILMS

Transition metal nitride thin films are widely used in demanding metalworking, microelectronic, automotive or aviation applications due to their exceptional hardness and wear resistance. However, these films tend to lack toughness and are prone to brittle failure. In this study, we are exploring a novel microstructural approach to increase the fracture toughness of AlCrSiN thin films while maintaining hardness, temperature and oxidation stability.

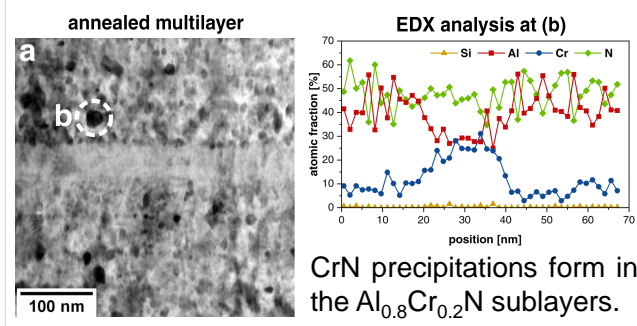
THIN FILM SYNTHESIS AND ARCHITECTURE



A multilayer thin film is deposited by cathodic arc evaporation.

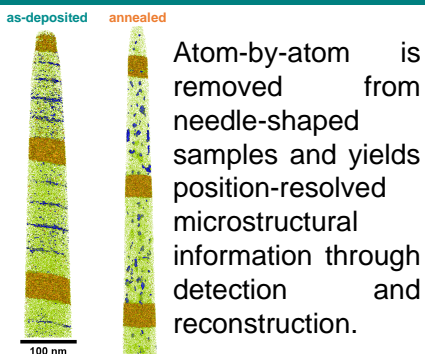
The heat-treatment under vacuum leads to sequential precipitation activity in the multilayer thin film.

TRANSMISSION ELECTRON MICROSCOPY



CrN precipitations form in the Al_{0.8}Cr_{0.2}N sublayers.

ATOM PROBE TOMOGRAPHY

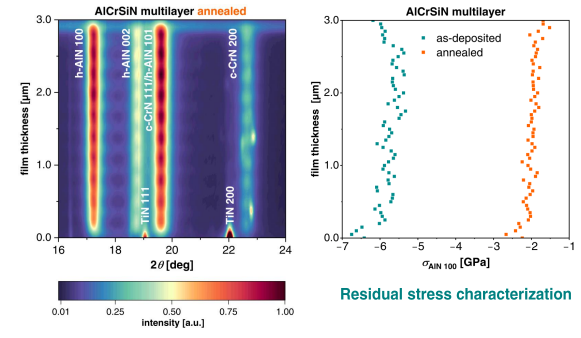
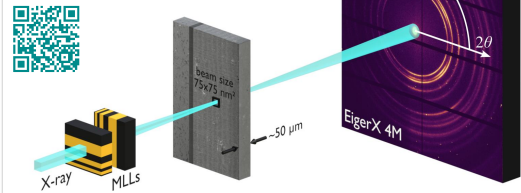


Atom-by-atom is removed from needle-shaped samples and yields position-resolved microstructural information through detection and reconstruction.

CROSS-SECTIONAL X-RAY NANODIFFRACTION AT THE ESRF

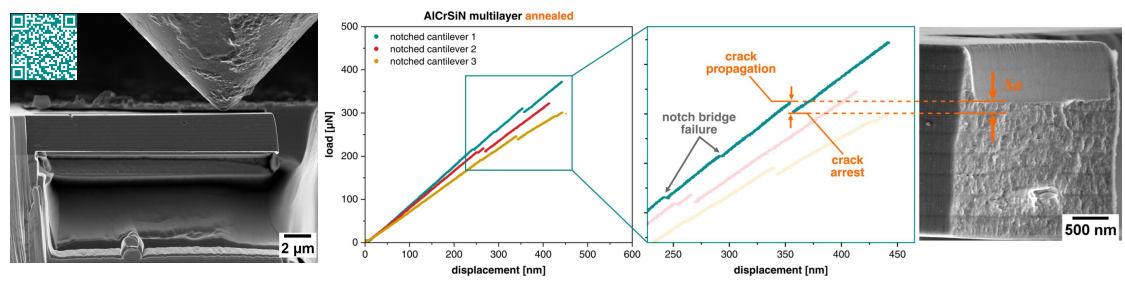
CSnanoXRD can reveal gradients of:

- crystalline phases
- residual stresses
- microstructure



Residual stress characterization

MICROMECHANICAL IN SITU BENDING TESTS



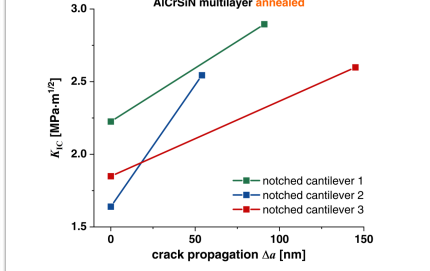
The distance between the notch tip (start of the crack propagation) and the next sublayer interface (crack arrest) was measured and is in good accordance with the analytical solution^[1].

stiffness changes after load drop $k_1 > k_2$ → $\int_0^a \frac{a}{t} Y \left(\frac{a}{t}\right)^2 da = \frac{(k_0 - 1)L}{18\pi(1 - \nu^2)r^2} a \rightarrow a + \Delta a$ analytical solution for the crack propagation

[1] M. Alfreider et al., Materials & Design, Volume 194, 2020.

CONCLUSION

Via nanoscale characterization methods the crack arrest mechanism was correlated to the multilayer microstructure that enhanced the fracture toughness K_{IC} of the thin film while maintaining its functional properties.



Dipl.-Ing.
KEVIN KUTLEŠA
 Department Materials Science
 Chair of Materials Physics
 kevin.kutlesa@unileoben.ac.at



RESEARCH TOPICS AND AIMS:

- synchrotron X-ray diffraction
- electron microscopy
- mechanical and electrical tests
- process-structure-property relationships

