

# **Study Structure-Property Relations in Materials Using Analytical and In-Situ Microscopy Characterization Techniques**

## **Agenda**

18 August 2019, Sunday

2:00 PM - 6:00 PM

2:00 PM **Registration**

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2:30 PM **Welcome Address**

Dr. Allen Zhang

*Vice President, ZEISS Research Microscopy Solutions*

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2:40 PM **In-Situ X-Ray Microscopy and LabDCT on Material Science Applications**

Mr. Chunjie Cao

*Senior Application Expert, ZEISS Research Microscopy Solutions*

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3:15 PM **Using Transmission Kikuchi Diffraction in a Scanning Electron Microscope to Quantify Geometrically Necessary Dislocation Density at the Nanoscale**

Prof. Hongbiao Dong

*Department of Engineering, University of Leicester*

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3:50 PM **Coffee Break**

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4:15 PM **FIB-SEM and Laser Ablation Applications in Material Science**

Dr. Qijun Ren

*Electron Microscopy Application Engineer, ZEISS Research Microscopy Solutions*

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4:50 PM **Internal Structures Evolvement Revealed by Laboratory X-ray Tomography**

Prof. Zhang Lei

*Shenyang National Laboratory of Materials Science, Institute of Metal Research, Chinese Academy of Sciences*

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5:25 PM **Machine Learning Segmentation of Metal Samples**

Dr. Roger Barnett

*Application Development Engineer: Steel & Other Metals, ZEISS Research Microscopy Solutions*

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6:00 PM **End of Workshop**

# **Study Structure-Property Relations in Materials Using Analytical and In-Situ Microscopy Characterization Techniques**

## **Speakers and Abstracts**

**Mr. Chunjie Cao**

*Senior Application Expert, ZEISS Research Microscopy Solutions*

**Title: In-Situ X-Ray Microscopy and LabDCT on Material Science Applications**

**Abstract:** The presentation will introduce the advantages of ZEISS X-ray microscopy and its applications in metal research. The first section of the presentation is focused mainly on the working principle and instrumentation in X-ray microscopy. The following sections will list the application cases of XRM on metal research, including alloy, AM, TBC, 4D in-situ experiment etc. The last sections will introduce diffraction contrast tomography technology on ZEISS x-ray microscopy. The application of DCT in metal crystal orientation analysis will also be introduced.

**Prof. Hongbiao Dong**

*Department of Engineering, University of Leicester*

**Title: Using Transmission Kikuchi Diffraction in a Scanning Electron Microscope to Quantify Geometrically Necessary Dislocation Density at the Nanoscale**

**Abstract:** Quantification of GND density on mesoscale has been achieved with recent advances in automated orientation imaging microscopy by electron backscatter diffraction (EBSD). In EBSD, the Kikuchi diffraction patterns arise as a consequence of diffraction within the subsurface interaction volume, which is fully contained within the bulk material. As a result, the spatial resolution for measuring GND density is restricted to approximately 100 nm. Therefore, it is challenging to quantify the geometrically necessary dislocation (GND) density at the nanoscale using conventional electron backscatter diffraction due to its limited spatial resolution.

To overcome this problem, in this study, the transmission Kikuchi diffraction (TKD) technique is used to measure lattice orientation and to calculate the corresponding nanoscale GND density. Using the TKD method, a variation of GND density from  $6.0 \times 10^{14}$  to  $1.0 \times 10^{16} \text{ m}^{-2}$  has been measured in a welded super duplex stainless steel sample. The distribution of dislocation density is shown to be in good agreement with transmission electron microscope (TEM) result. Compared with dislocation measurements obtained by TEM, the TKD-GND method is revealed to be a relatively accurate, fast and accessible method.

**Dr. Qijun Ren**

*Electron Microscopy Application Engineer, ZEISS Research Microscopy Solutions*

**Title: FIB-SEM and Laser Ablation Applications in Material Science**

**Abstract:** FIB-SEM is commonly used in material science for high resolution imaging, TEM sample preparation, and 3D reconstruction of samples, etc. However, the complexity of the workflow and difficulty to access regions of interests (ROI) deeply buried under sample surface hindered more effective applications of FIB-SEM in material characterizations. On the other hand, the analysis of materials such as elemental composition in high resolution is highly demanding, which is also beyond the scope of traditional EDS analysis.

To address the above issues, we introduced the new generation of ZEISS FIB-SEM system. With new designed workflows, it provides much higher efficiencies for traditional time consuming applications, such as TEM sample preparation and 3D tomography. The correlation of a Femto-second laser ablation and Ga FIB fine milling realizes the position and access to the ROIs deeply buried in bulk materials. These techniques pave the way for effective characterization of samples in nanometer scale and in 3D, and will find wide applications in Alloys, Metals, Semiconductors, and Additive manufacturing, etc. Moreover, correlation between FIB-SEM and a nano-SIMS (Secondary Ion Mass Spectroscopy) system allows for high resolution and 3D elemental analysis quantitatively, which helps for deep understanding of samples.

**Prof. Lei Zhang**

*Shenyang National Laboratory of Materials Science, Institute of Metal Research, Chinese Academy of Sciences*

**Title: Internal Structures Evolvement Revealed by Laboratory X-ray Tomography**

**Abstract:** Although X-ray generated from laboratory-based tubes cannot be compared with synchrotron radiation in brilliance and monochromaticity, they are still viable and accessible in-house for ex-situ or interrupted in situ X-ray tomography. This talk mainly demonstrates recent works using laboratory X-ray tomography coupled with the measurements of properties or performance testing under thermal, mechanical, and electrical conditions. Evolvments of correlated internal structures for some typical materials were uncovered. The damage features in a graded metallic 3D mesh and a metallic glass under mechanical loading were revealed and investigated. Micro-voids with thermal treatment and void healing phenomenon with electropulsing were clearly demonstrated and quantitatively analyzed. The substance transfer around an electrode of a Li-S battery and the protective performance of a Fe-based metallic glass coating on stainless steel were monitored through electrochemical processes. It was shown that in situ studies of the laboratory X-ray tomography were suitable for the investigation of structure change under controlled conditions and environments. An extension of the research for in situ laboratory X-ray tomography can be expected with supplementary novel techniques for internal strain, global 3D grain orientation, and a fast tomography strategy.

**Dr. Roger Barnett**

*Application Development Engineer: Steel & Other Metals, ZEISS Research Microscopy Solutions*

**Title: Machine Learning Segmentation of Metal Samples**

**Abstract:** Most routine assessment of steel and other metals requires microscopy to assess various microstructural parameters intrinsic to the metal or the effects of subsequent treatments. These may include grain size, porosity, coating layer thickness or a variety of other factors.

*Quantitative assessment of micrographs involves a segmentation step – dividing one region from another. Regions may be individual grains, pores, dendrites, layers or phases. Doing this by assessing colour/greyscale levels may be a challenging and time-consuming task.*

*We present a new approach for straightforward workflow-based segmentation of metal sample images using guided machine learning in ZEISS ZEN Intellesis. This facilitates faster routine analysis and even allows quantitative automatic assessment of challenging samples that would normally require manual labelling. The technique can be applied to light microscopy, electron microscopy, X-ray microscopy – any microscopic method*