Title

3D Microstructure Analysis and Tomography in the Micro, Nano and Atomic scale

Module Coordinator

Prof. Frank Mücklich

Teaching Staff



Prof. Peter Felfer, Department of Materials Science and Engineering, Institute I, Friedrich-Alexander Universität Erlangen-Nürnberg (FAU), Erlangen, Germany

Prof. Frank Mücklich, Department Materials Science and Engineering, Institute for Functional Materials, Saarland University, Saarbrücken, Germany



Prof. Guillermo Requena, Deutsches Zentrum für Luft und Raumfahrt, Institute of Materials Research, Cologne, Germany



Dr. Di Wang, Karlsruher Institut für Technologie (KIT) Institut für Nanotechnologie, Eggenstein-Leopoldshafen, Germany



Prof. Ehrenfried Zschech, Fraunhofer Institute for Ceramic Technologies and Systems, Dresden, Germany

Talks

- Serial Sectioning Techniques from HR FIB-Tomography to large volumes with adequate resolution –
 Frank Mücklich
- Lab-based X-ray Tomography Ehrenfried Zschech
- Synchrotron Tomography Guillermo Requena
- TEM Tomography Di Wang
- Atom Probe Tomography Peter Felfer

Abstract

Three dimensional microstructure characterization at different scales plays the key role for understanding the quantitative relationship between processing, microstructure and properties. Starting from statistical analysis of simple shaped microstructures and their stereological 3D estimation, the necessity of tomographic techniques for complex microstructures will be explained. The tutorial will give an overview of tomographic imaging techniques in different length scales, namely serial sectioning techniques based on advanced mechanical as well as FIB-SEM techniques, X-ray tomography including Lab based as well as synchrotron techniques, TEM tomography, and finally the Atom Probe Tomography. State of the art, recent application examples and new trends for each method will be briefly discussed.

Serial Sectioning Techniques

For many engineering materials there is a tradeoff between adequate resolution and statistically relevant volume in order to understand the relevant microstructural features. The most relevant microstructural level can be covered by sub-µm **mechanical serial sectioning** in terms of large microstructural volume and a variety of metallographic contrast mechanisms. This enables to investigate essential geometrical features such as spatial connectivity e.g. in complex multiphase steel and to find quantitative correlations of this microstructures to properties. Subsequently, the FIB-tomography links the metallographic and the nano scale. By including well established EDS, EBSD and SEM, a lot of crucial challenges in material science can be coped by the **FIB-SEM Serial Sectioning** with a resolution down to a few nm.

X-Ray Tomography

Laboratory-based X-Ray Tomography, a nondestructive technique for 3D analysis of materials, is a highly ranked request from advanced materials development, and it is an industrial demand in several branches including microelectronics, energy storage and lightweight construction. Due to the particular properties of X-rays, i. e. high penetration of matter and good material contrast in absorption, micro and nano X-ray tomography are versatile tools for nondestructive 3D bulk analysis of materials and for the investigation of complex 3D structures. Examples for high-resolution in-situ X-ray imaging studies will be shown, including studies of kinetic processes in materials: Physical failure analysis in 3D-stacked microchips, kinetic reactions for energy storage and conversion processes, crack initiation and propagation in microchips and composites. Novel high-flux X-ray sources and novel X-ray optics will be discussed. These developments provide potential advantages for both micro XCT and nano XCT: reduction of measurement time and/or improvement of resolution.

Synchrotron Tomography can provide spatial resolution down to sub-µm range, high time resolution critical for in situ experiments and multimodal experimental data by simultaneous combination with other synchrotron techniques, e.g. diffraction. All these characteristics will be introduced in the frame of basic theoretical concepts and practical examples for which this technique has been decisive to understand the thermo-mechanical behaviour of structural materials such as Al-, Ti-alloys and composites.

TEM Tomography

TEM and STEM Tomography, benefiting from high spatial resolution in transmission electron microscope, provide 3D structural information down to sub-nm. They have been widely used to characterize e.g. mesoporous and microporous materials and supported metallic nanoparticles used as catalysts. In addition,

spectroscopic imaging, such as elemental mapping by EDX and EELS or EFTEM imaging can be combined with tomographic techniques to provide 3D elemental distributions. The tutorial will introduce the basics of TEM/STEM imaging, experimental tomography techniques, alignment procedure, reconstruction and quantification of material features. Plenty of applications in nanomaterials will be presented.

Atom Probe Tomography

In recent years, atom probe tomography has brought us much closer to the dream of knowing the location and identity of each atom in metallic, semiconducting and ceramic materials. This is based on the ability to field evaporate single atoms from sharp tips using either voltage (metals) or laser (semi and nonconductors) pulses. Detection of the field evaporated ions on a 2D detector, together with the arrival sequence enables 3D reconstructions. In this tutorial, we will cover the fundamentals and limitations of the technique as well as current topics to engage materials scientists in the plentiful possibilities of the technique.

Format

Lectures including discussion

Prerequisites

Background in Materials Research

Intended Stage

PhD Students, Post-Docs or Experienced Researchers

Duration

5 hours including breaks