Materials Characterization Tutorial

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What can be Obtained From the TEM Images of Defects?
Sosuke Kondo

Transmission electron microscopy has been used successfully to study the structure and distribution of the extended microstructural defects that often modify the macroscopic material properties. Among irradiation induced defects, perhaps the most frequently observed subject to date is voids or dislocation. For example, swelling can be estimated directly from observation of the void distribution. Radiation hardening is often associated with the number density of dislocation loops. In the beginning of this lecture, you would be specifically to learn about the traditional observation techniques of voids and dislocation loops. Here we will spend time sharing specific ideas, such as weak-beam-dark field imaging, needed to make dislocation images clearer with the so-called conventional 200 keV TEM. Of course, thin foil processing method using focused ion beam (FIB), which is a prerequisite for successful observation, is also covered in the section. Obtaining the physical parameters of the material, such as migration energy of point defects, from the obtained defect image is another reason for increasing the utility value of TEM. The migration energy of interstitials and vacancies would be essential parameters for predicting the microstructural development in the neutron irradiated materials. Recalling the diffusion equation, it is readily apparent that these material-specific values, along with sink concentration and irradiation temperature, determine the point-defect flux in the material. In other words, by examining the temperature dependence of each nucleation and defect growth, we can theoretically know the defect mobility in combination with simple rate equations. The migration energy of point defects can also be estimated from the analysis of dislocation loop free zone near the sink, where the concentration of a point-defect is always lower than the critical concentration for forming loops, based on the same theory as described above. The middle part of this lecture will focus on how to image the grain boundary denuded zone (DZ) with the conventional TEM and how to estimate the migration energy of interstitial atoms from the irradiation temperature dependence of DZ width. Some recent results on the microstructural evolution of neutron or ion irradiated silicon carbide, many of which are surprisingly similar to FCC metals, will be presented at the end of this lecture.

STEM and AEM Applications in Fusion Materials
Chad M. Parish

Scanning transmission electron microscopy (STEM) uses a fine electron probe to explore a thin foil of material. Opposite to TEM, the STEM depends on rastering this probe and obtaining a digital representation of the material's response. STEM is capable of replicating any of the contrast modes of conventional TEM, and therefore makes a powerful complement to TEM methods. However, unlike TEM, STEM is can obtain

additional image contrast modes, such as annular dark field, that provide vital scientific insight not available to TEM. Even more importantly, STEM allows the acquisition of chemical and elemental information via electron energy loss (EELS) and X-ray energy dispersive spectroscopy (XEDS). These techniques, collectively, can be referred to as Analytical Electron Microscopy (AEM).

In this tutorial, we will discuss: (1) the differences between TEM and STEM; (2) the use of STEM to obtain microstructural images of radiation-induced defects, such as dislocation loops; (3) the use, advantages, and disadvantages, of AEM methods—EELS and XEDS—in the characterization of radiation effects and plasma effects on materials, and (4) a brief introduction to data analytics and machine learning to explore the large datasets produced by modern AEMs.

The goal of this tutorial will be to (1) familiarize the students with the capabilities of modern STEM / AEM experiments, (2) enumerate how fusion materials problems can be solved using STEM / AEM methods, and (3) preview the rich future of data-science based support for STEM and AEM experiments.

Synchrotron-based Diffraction and Scattering and Opportunities at Advanced Light Source Facilities

David Sprouster

In recent years, synchrotron radiation facilities have become widely used as sources for x-ray measurements. Synchrotron radiation is emitted by electrons (or positrons) traveling at near speed-of-light velocities in a circular storage ring as they move through magnetic fields. These powerful x-ray sources, which are thousands to millions of times more intense than laboratory x-ray sources, have become indispensable tools for a wide range of structural investigations in numerous fields of science and technology. Diffraction and scattering-based techniques play a leading role at modern synchrotrons with most modern facilities accommodating numerous scattering and diffraction beamlines. The ultra-high fluxes available at modern synchrotron beamlines have opened up opportunities to perform rapid, non-destructive, high-throughput and in-situ experiments that would take exceedingly long times with conventional x-ray sources.

At a basic level, x-rays incident on any atomic surface (be it solid, liquid or gas), primarily interact with electrons and undergo elastic or Thompson scattering. When atoms are arranged in a periodic pattern, as in crystalline materials, the scattered x-rays constructively interfere leading to intense diffraction peaks in certain directions. This diffraction condition is known as Bragg diffraction and the diffraction angle "θ" measured during an x-ray diffraction (XRD) experiment can be used to determine the interplanar spacing "d" of the diffracting material through the Bragg formula: $n\lambda=2d\times\sin\theta$ (where n is an integer and λ is the wavelength of the incident x-rays). Analysis of XRD patterns allows the quantitative determination of lattice parameters, phase fraction(s) and microstructure.

At very small 2θ angles (<3 degrees), scattering very near the un-deviated transmitted x-ray beam contains size and shape information corresponding to structures ranging from tens to thousands of Å's. This diffuse scattering signal is more commonly known as small angle x-ray scattering (SAXS). Variations in the measured SAXS intensity with 2θ or scattering vector Q ($Q = 4\pi\times\sin\theta$), directly result from electron density fluctuations between the scattering objects and the background or host matrix. Analysis of SAXS patterns allows quantitative determination of size, size distribution and volume fraction of particles within a host matrix.

In this tutorial, the basics of synchrotron based XRD and SAXS will be described. The current and near-term capabilities available to the fusion community at advanced Department Of Energy facilities, including the National Synchrotron Light Source-II and the Advanced Photon Source, will be presented. Opportunities in leveraging synchrotron-based techniques to address fundamental and applied materials science challenges at various length scales to support modeling efforts will be highlighted and recent examples of neutron irradiated fusion materials systems will be presented.

Atom Probe Tomography for Fusion Materials
Philip D Edmondson

Atom probe tomography (APT) is a technique capable of providing high spatially and chemically resolved three-dimensional atom-by-atom rendering of a material specimen. The development of laser pulsing enables the analysis of significantly less conductive specimens, giving the technique a broader appeal. The combination of laser assisted APT and the development of robust specimen preparation techniques based on focused ion beam (FIB) have revolutionized the technique, not only enabling the analysis of poorly conducting specimens, but also site-specific sample fabrication and minimizing sample volume thereby reducing the radiological exposure of highly irradiated neutron exposed specimens. The unique traits of APT are the three-dimensionality of the analysis of the capacity to resolve the chemical identities of atoms, even at extremely dilute concentrations or of very light species that are not easily distinguishable via electron microscopy-based techniques. (APT chemical sensitivity is mass invariant) APT also has the added benefit of being able to identify and precisely locate individual atoms in non-crystalline materials.

In this tutorial, the fundamentals of APT will be introduced including sample preparation techniques, APT instrumentation, methods of reconstruction of the data, and basic analysis techniques. Finally, some examples of the use of APT to fusion specific materials issues will be presented.

The goal of this tutorial is: 1) provide familiarization of the technique and instrumentation; 2) give and understanding of how to reconstruct the APT data and provide best practices for doing so; 3) introduce some data analysis techniques and show how this may be applied to fusion materials applications.