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X-RAY MICROIMAGING OF ELEMENTAL COMPOSITION AND MICROSTRUCTURE FOR MATERIALS SCIENCE

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1. Abstract

X rays have many advantages over electrons and other charged particles for the microcharacterization of materials. X rays are more efficient in photoejecting inner shell electrons which results in characteristic x-ray fluorescence. X rays also produce less Bremsstrahlung which yields far higher signal-to-background than obtained with electrons. Minimum detectable limits (MDL) for X ray excited fluorescence can be a few parts per billion; 10^{-3} to 10^{-5} less than for electron excitation. The third generation synchrotron radiation sources such as the Advanced Photon Source will for the first time provide x-ray sources as brilliant as the most advanced electron probes. It will therefore soon be possible to develop a submicron x-ray probe with unprecedented low levels of detection in diffraction, EXAFS, Auger, Photoelectron and fluorescence spectroscopies for structural and chemical characterization. Some applications to materials science are shown.

2. Advantages of an X-ray Probe

We are at the beginning of a revolution in our ability to microimage elemental composition and structure with x rays. This revolution is the result of vastly more brilliant x-ray sources, new developments in x-ray optics and rapidly improving image processing (Fig. 1). The superiority of x rays for imaging internal structure and elemental composition has long been recognized (Fig. 2-5) yet efforts to construct x-ray microprobes have been largely dormant for 30 years due to the overwhelming brightness of electron sources (Fig. 6,7). With the construction of third generation synchrotron radiation sources we will, for the first time, have x-ray sources as brilliant as the most advanced electron sources. With simultaneous advances in x-ray optics it will be possible to deliver the same flux of x-rays to a 1 μm^2 spot as with electrons (Fig 9-11). The fluorescent signal from each x ray on a sample is typically 10-100 times greater than from electrons or ion excitation (Fig. 11). Even more dramatic is the signal-to-noise which is typically four to five orders of magnitude greater for x rays than for electrons (Fig. 12). An x-ray microprobe will, therefore, deposit much less power into the sample for the same minimum detectable limit (Fig. 13,14). Conservative estimates for the detection limits with a 1 μm^2 x-ray microprobe having 10^{-4} 8 keV

photons/sec far exceed that possible with alternative probes (Fig. 15). An x-ray microprobe will also yield better spatial resolution for thick samples (Fig. 16). The ultimate performance for fluorescence detection will be achieved using crystal spectrometers (Fig. 17). An x-ray microprobe offers several other advantages compared to charge particle microprobes. The most important of these is the ability to make measurements in the presence of air, water or other gases, and the ability to probe deep into a sample (Fig. 18).

3. Applications of an X-ray Microprobe to Materials Science

An x-ray microprobe on a third generation storage ring will have many important applications to materials science. The low MDL of an x-ray microprobe will be useful in mapping out trace element distributions in inhomogeneous samples. Microprobes using pinholes and solid state detectors are presently capable of detecting 100 ppb of metals in plastic with 10-60 μm diameter probe size (Fig. 19). The extended range of detectability of a third generation x-ray microprobe will be useful in mapping out the elemental distribution in microcircuits, particularly near junctions contacts and at interfaces (Fig. 20).

Information about elemental distributions near grain boundaries will help elucidate the role of microalloying in altering grain boundary brittle failure (Fig. 21). Similarly at 1 μm resolution we will be able to study diffusion along interfaces and grain boundaries with remarkable sensitivity (Fig. 22). Another interesting problem is the effect of microalloying on radiation induced swelling (Fig. 23).

An x-ray microprobe can be a valuable tool for nondestructive studies of microstructure in composite materials. Some possible applications are the radiographic or tomographic study of nuclear fuel particles and fiber reinforced composites (Fig. 24-26). Tomographic studies using x-ray fluorescence will be particularly sensitive to trace element distributions (27-28).

An x-ray microprobe will be a valuable tool for studying the crystallographic structure at grain boundaries, interfaces, and at composite boundaries. Some early experiments have already demonstrated the usefulness of x rays for studying structure at and near boundaries (Fig. 29-31). Particularly intriguing is the ability to study not only the structure but also the local strain near cracks, precipitates, flaws and other features of importance to materials properties.

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