X-ray Optics For Polycrystalline Microdiffraction

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Introduction

The behavior of materials is inherently dependent on three-dimensional tensor distributions. Heterogeneous networks of second phases, un-oriented grains, voids, reinforcing particles and fibers act to redistribute macroscopic applied loads into complex, three-dimensional distributions of stresses and strains. X-ray diffraction with submicron resolution can detect the distribution of phase texture and strain in materials, but X-ray microdiffraction on polycrystalline materials is especially challenging and requires fundamentally new instrumentation and techniques; for a single grain, the probability of a Bragg reflection is vanishingly small with a monochromatic beam and an unknown crystal orientation. Whereas small single crystals can be bathed in large x-ray beams and studied with standard diffraction-rotation methods, polycrystalline microdiffraction patterns are complicated by sphere-of-confusion motions and by changing grain illumination as the sample is rotated (Fig. 1). The complexity introduced by sample rotation can be avoided by the use of broad-bandpass Laue methods.

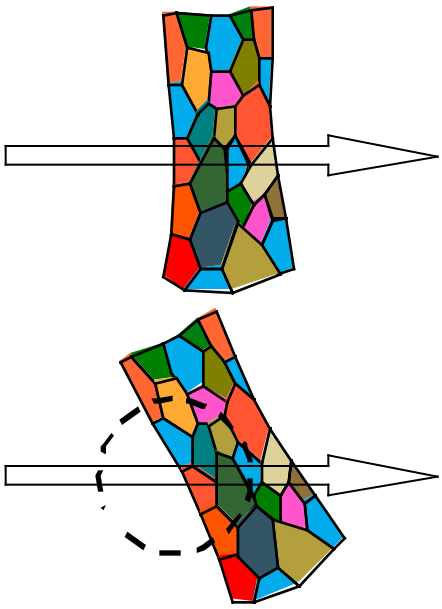

Fig. 1 The grains illuminated by a small x-ray microbeam change as the sample is rotated due both to sphere-of-confusion errors and due to the penetration of the beam into the sample.

Methods and Materials

Our approach is to use white-beam Laue methods to determine the phase, texture and strain of crystallites without rotating the sample. With white beam diffraction, the deviatoric strain tensor can be determined from the Laue pattern and the absolute strain tensor can be determined from the Laue pattern with the additional information obtained by accurately determining the energy of one Laue reflection. The essential elements for the white-beam x-ray microdiffraction station are shown in Fig. 2.

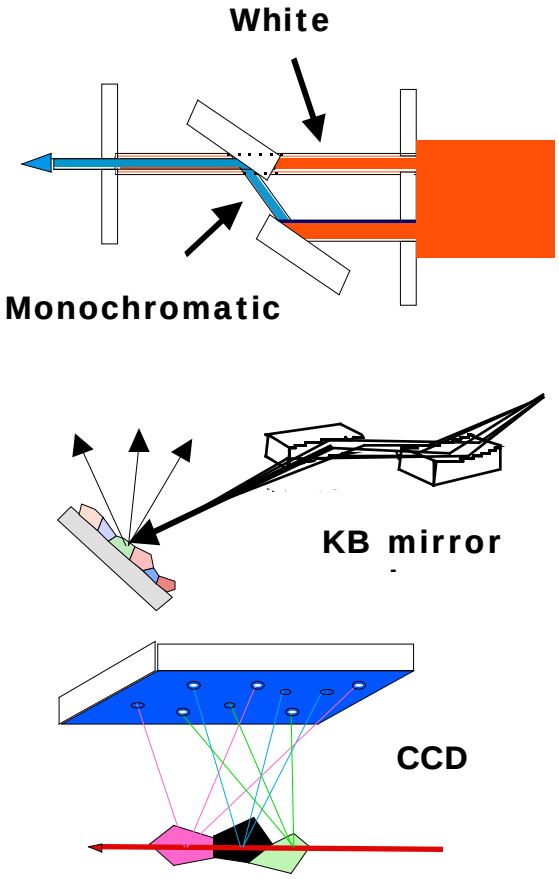

Fig. 2. The three essential elements for x-ray microdiffraction on a polycrystalline sample: (top) a microbeam monochromator for accurately measuring the x-ray energy; (b) nondispersive focusing optics and (c) a high accuracy x-ray area detector.

The specially designed micromonochromator allows the beam to cycle between white and monochromatic beam conditions with a fixed focal spot on the sample.[1] The Kirpatrick-Baez mirrors provide non-dispersive focusing and achieve outstanding focusing performance by using a special differential deposition technique.[2,3] The CCD camera collects the Laue image which is interpreted by a novel computer program capable of indexing overlapping Laue patterns from multiple grains.[4]The bandpass between and nominal x-ray energy of the incident beam is a compromise between the desire for many reflections, and the need

to improve the integrated reflectivity of reflections to accelerate the data collection.[5]

Results

The performance of the overall system has now reached a stage where fundamentally new information about the mesoscale dynamics of materials can be probed. The x-ray microbeam monochromator provides a stable absolute energy reference with an uncertainty of about 0.5 eV from 15-22 keV. The x-ray mirrors have achieved a focal spot size of less than 0.4x0.5 μm^2 (see Fig. 3), and the automated texture and strain mapping has shown a sensitivity to strain, under favorable conditions, of better than 2 parts in 10^5.

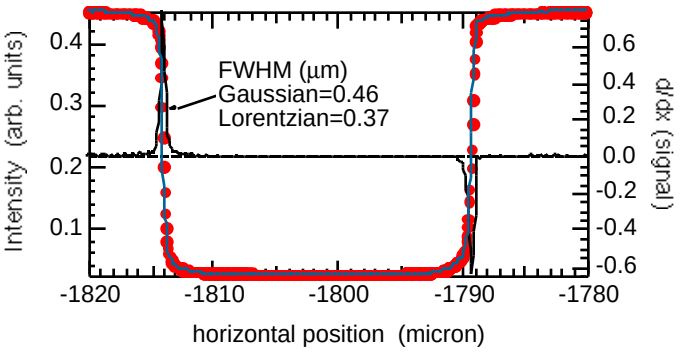

Fig. 3 Horizontal focus of the x-ray microbeam measured with a 22 μm-diameter wire passed through the beam. The derivative of the transmission gives an upper limit to the beam size. Note that the quality of the mirrors is such that there is little stray radiation outside the focus (low tails).

Some of the first materials studied were high temperature superconducting thin films where the penetrating x-ray beam allows the orientation of the substrate, buffer layer and superconducting grains to be quantitatively correlated on a grain-by-grain basis. This new information has yielded insights into the growth mechanisms of these films(see report by J.D. Budai et al.). Another early experiment studied the texture and strain in metallic interconnect wires in integrated circuits (see report by N. Tamura et al.). This work illustrates how the x-ray microdiffraction experiments can be used to map elastic and plastic deformation in adjacent grains with high spatial and angular resolution.[6] Other work includes measurements of deformed metals[7], weld samples (see report by J-.S. Chung et al.) and composite materials with strength near their theoretical limit.

Discussion

X-ray microdiffraction represents an important new application made possible by the ultra-high brilliance of the Advanced Photon Source. Although the power density of the beam is increased by more than four orders of magnitude by the focusing optics, the total power in the microdiffraction beam is small; the useable emittance from the beam is less than 0.1% of the raw undulator emittance for a 1 μm-diameter beam with less than 1 milliradian of divergence. As a result, the thermal rise on samples is small for most materials and the probe can be used for general-purpose studies of strain, texture and phase distributions in polycrystalline materials.[8]

Acknowledgments

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