SAND2000-0407C

Small area analysis using micro-diffraction techniques

RECEIVED
FEB 24 2000
OSTI

R.P. Goehner, R.G. Tissot, and J.R. Michael

Sandia National Laboratories, Albuquerque, New Mexico 87185-1405

An overall trend toward smaller electronic packages and devices makes it increasingly important and difficult to obtain meaningful diffraction information from small areas. X-ray micro-diffraction, electron back-scattered diffraction (EBSD) and Kossel are micro-diffraction techniques used for crystallographic analysis including texture, phase identification and strain measurements. X-ray micro-diffraction primarily is used for phase analysis and residual strain measurements of areas between 10μm to 100μm. For areas this small glass capillary optics are used for producing a usable collimated x-ray beam. These optic are designed to reflect x-rays below the critical angle therefore allowing for larger solid acceptance angle at the x-ray source resulting in brighter smaller x-ray beams. Less than 10μm beams have been produced with a tapered bored single capillary optic using conventional x-ray sources.[1] A Xenon filled HiStar area detector used for x-ray micro-diffraction captures a large area of the diffraction cone which allows for rapid analysis (typically 10 to 20 minutes) and also visually shows texture and particle size influences from the sample. Fig. 1 is a 20 minute area detector image showing a textured Al (222) fine-grained thin film with much coarser grained random oriented LaB_6 used as an internal standard. Also visible is a Laue spot from the Si single crystal substrate. The determination of residual strain using micro-diffraction techniques is very important to the semiconductor industry. Residual stresses have caused voiding of the interconnect metal which then destroys electrical continuity. Being able to determine the residual stress helps industry to predict failures from the aging effects of interconnects due to this stress voiding. Stress measurements would be impossible using a conventional x-ray diffractometer; however, utilizing a 30um glass capillary these small areas are readily assessable for analysis. Kossel produces a wide angle diffraction pattern from fluorescent x-rays generated in the sample by an e-beam in a SEM. This technique can yield very precise lattice parameters for determining strain. Fig. 2 shows a Kossel pattern from a Ni specimen.

Phase analysis on small areas is also possible using an energy dispersive spectrometer (EBSD) and x-ray micro-diffraction techniques. EBSD has the advantage of allowing the user to observe the area of interest using the excellent imaging capabilities of the SEM. An EDS detector has been used for simultaneous element identification which enhances phase identification of unknowns.[2]

The x-ray area detector also allows for rapid microstructure information including crystallite orientation and size by directly observing the diffraction rings. Coarse crystallites would tend to show a more spotty rings (Fig.1) than fine crystallites. Fig. 3 shows highly textured orthorhombic (left) and rhombohedral (right) fine-grained PZT thin film. Slight omega rotation distinguishes Laue spots by showing trails perpendicular to the Debyė arcs. These Laue spots are from the single crystal substrate.

These techniques allow for small area analysis that in the past would have been difficult if not impossible to obtain. The future development in x-ray optics and the use of synchrotron sources will allow for the potential of nondestructive submicron x-ray diffraction analysis.

1. York, B., Fabrication and Development of Tapered Capillary X-ray Optics for Microbeam Analysis, 1995 MAS Meeting, Breckeridge, CO.
2. Industrial Applications of X-Ray Diffraction edited by Chung, F. and Smith, D. (2000). *Industrial Applications of X-ray Diffraction,* Marcel Dekker, 2000,p. 896-890.

3. This work was supported by the United States Department of Energy under contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy

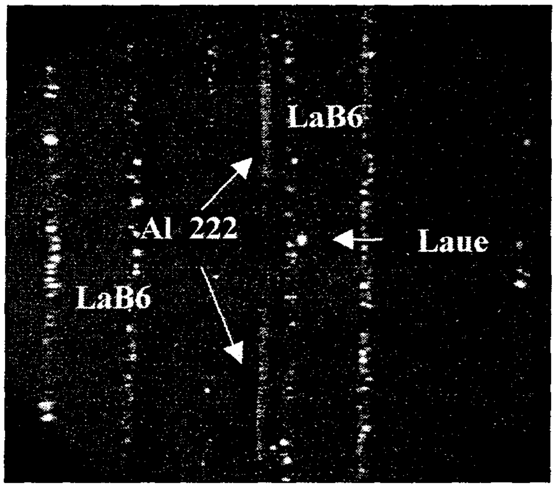

Fig. 1 Area Detector image of textured Al (1 1 1)

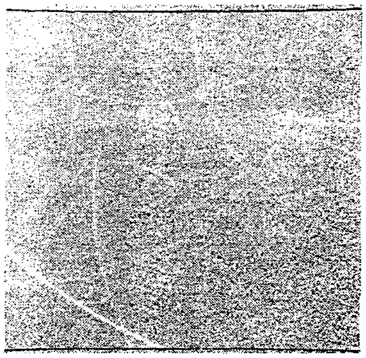

Fig. 2 Ni Kossel pattern

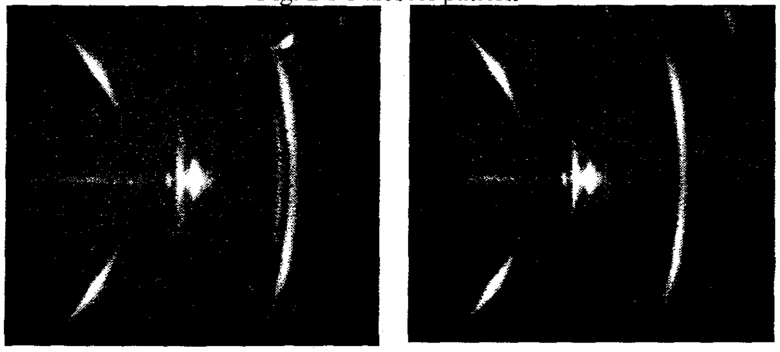

Fig. 3 Area detector image of textured PZT thin film left orthorhombic and right rhombohedral phase

DISCLAIMER