FIB/DualBeam Techniques for Quantitative Analyses

Lucille A. Giannuzzi

FEI Company, 5350 NE Dawson Creek Drive, Hillsboro, OR 97124

The use of Focused ion beam (FIB) and combined FIB plus scanning electron microscope (SEM) dual platform instruments have continued to increase in popularity over the past decade in both the physical and biological sciences. FIB based techniques enable site specific regions to be prepared for direct quantitative analysis and/or extracted for quantitative analyses using other analytical instruments in material systems that were once too difficult or unfeasible to observe by other means. In addition, since FIB techniques in general offer higher throughput than conventional methods, more samples may be quantified than previously possible. In this paper, quantitative techniques facilitated by either direct FIB imaging or FIB-based specimen preparation methods are discussed.

One of the most popular uses of focused ion beams is for transmission electron microscopy (TEM) specimen preparation. The ubiquitous FIB lift-out techniques allow for site-specific extraction of interfaces, inclusions, failure sites, etc., from a wide range of materials [1]. Therefore, quantitative x-ray energy dispersive spectrometry (XEDS) techniques can be directly applied to e.g., interdiffusion studies [2], or grain boundary segregation effects on grain boundary diffusion [3].

A particular advantage to FIB-based imaging is the channeling contrast generated by ion induced secondary electron imaging which is unsurpassed compared to conventional SEM imaging. Thus, FIB imagining can provide direct capabilities for quantification of microstructural features. This ability to use FIB channeling contrast imaging on a mechanically polished Cu-Ni diffusion couple revealed regions where diffusion induced recrystallization (DIR) occurred at the diffusion couple interface [2]. Site specific TEM specimen preparation of DIR and non-DIR regions allowed for direct quantitative analysis of the Cu-Ni diffusion couple showing an enhancement of lattice interdiffusion due to grain boundary contributions [2]. FIB channeling across grain boundaries was also used to prepare TEM specimens for the quantification of the effects of Bi segregation on the Ni diffusion through Cu twist boundaries [3]. In addition, dopant profiles of p/n junctions within Si-based transistor gates can be quantified using electron holography methods with the utilization of back-side FIB milling plus low energy polishing steps [4].

The basic FIB lift-out techniques have also been employed to prepare samples for quantification in surface science instruments such as x-ray photoelectron spectroscopy [5], Auger electron spectroscopy [6], secondary ion mass spectroscopy (SIMS) [6], as well as 3D atom probe [7,8]. The same techniques used to prepare atom probe specimens can be used to make pillar-shaped TEM specimens for on-axis TEM tomography [9,10].

Quantitative crystallographic orientations can be obtained by acquiring electron backscattered diffraction (EBSD) patterns in the SEM from 30 keV and 5 keV FIB prepared surfaces [11]. The combined FIB lift-out plus EBSD techniques have been used to quantify crystallographic texture and phase distribution in materials [12]. With new FIB instruments able to produce low energy ions, specimens can be prepared with minimal ion implantation damage enabling sub-angstrom information from HRTEM specimens [13]. These low energy polishing techniques may also be extended to improve EBSD pattern quality and quantification for difficult materials [14].

Finally, many of the 2D quantitative analyses described above can be extended to 3D. Direct FIB channeling contrast imaging methods or combined FIB/SEM techniques have been used for quantitative analysis of microstructure and morphology, crystallography, texture, or elemental distribution using sequential FIB slicing with either direct FIB [15] or SEM imaging [16], or SEM combined with EBSD [17], XEDS [18], or FIB/SIMS analysis [19].

References

[1] L.A. Giannuzzi et al., in Introduction to Focused Ion Beams, eds. L.A. Giannuzzi and F.A. Stevie, Springer 2005
[2] S.M. Schwarz et al., *Acta Materialia*, 51 (2003) 2765.
[3] B.W. Kempshall et al., in Mass and Charge Transport in Inorganic Materials - II, Vol.37 Advances in Science and Technology (2003) 239.
[4] M.R. McCartney et al., *Micros. Microanal*., 9(Suppl 2) (2003) 776.
[5] A.C. Ferryman et al., *Surf. Interface Anal.*, 33, (2002) 907.
[6] F.A. Stevie et al., *Surf. Interface Anal*., 31 (2001) 345.
[7] R.L. Martens,et al., *Microsc. Microanal.* 6 (Suppl. 2) (2000) 522.
[8] M.K. Miller et al., *Ultramicroscopy*, 102 (2005) 287.
[9] S.M. Schwartz, et al., *Microsc. Microanal.* 10(Suppl. 2) (2004) 142.
[10] P.G. Kotula et al., *Microsc. Microanal*. 13(Suppl 2) (2007) this issue.
[11] T.L. Matteson et al., *J. Electronic Materials*, 31(1), (2002) 33.
[12] J.R. Michael et al., *Microsc. Microanal.* 12(Supp 2), (2006) 926 CD.
[13] Lucille A. Giannuzzi et al., *Micros. Microanal*., 11(Suppl 2) (2005) 828.
[14] J.R. Michael et al., *Microsc. Microanal*. 13(Suppl 2) (2007) this issue.
[15] B.J. Inkson et al, *Scripta Mater*., 45 (2001) 753.
[16] L. Holzer et al., *J. Microscopy*, 216, (2004) 84.
[17] J.J.L. Mulders & A.P. Day, *Materials Science Forum*, 495-497 (2005) 237.
[18] Paul G. Kotula et al., *Microsc. Microanal*. 12, (2006) 36.
[19] D.N. Dunn. & R. Hull, *Appl. Phys. Lett.* **75**, (1999) 3414.