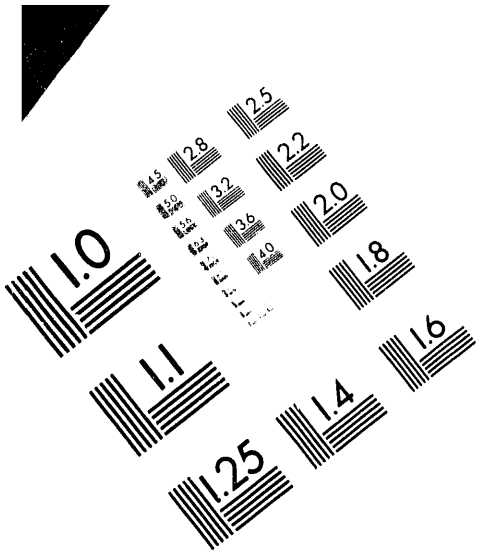
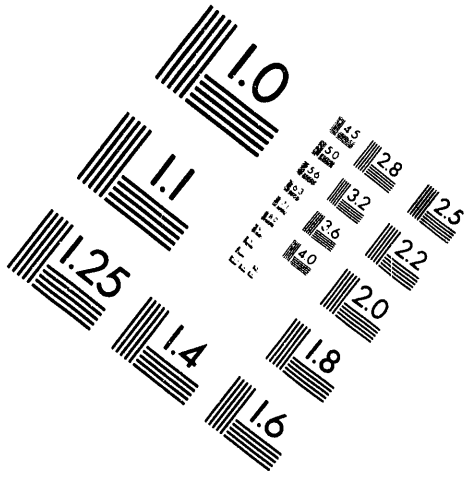
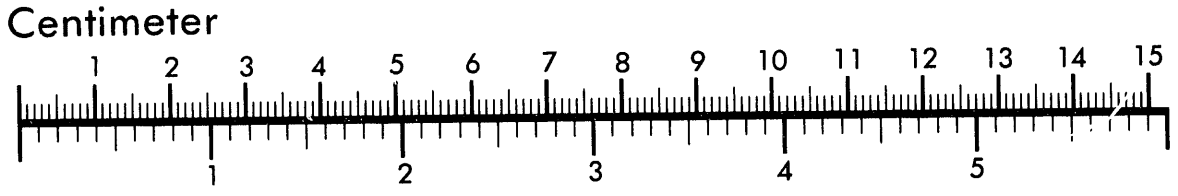
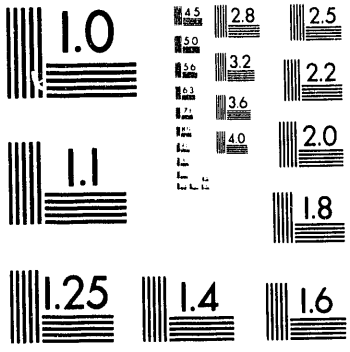
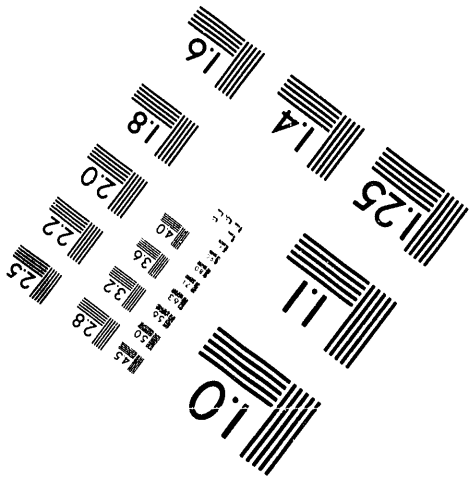


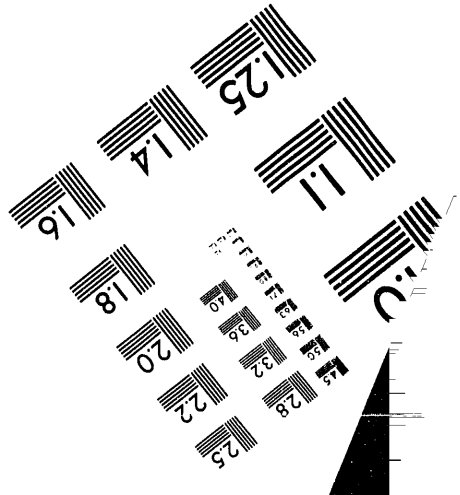
Association for Information and Image Management
1100 Wayne Avenue, Suite 1100
Silver Spring, Maryland 20910
301/587-8202











MANUFACTURED TO AIIM STANDARDS
BY APPLIED IMAGE, INC.

1 of 1

To be published in *Proceedings of the 6th Analytical Electron Microscopy (AEM) Workshop*, Los Angeles, California, July 11–16, 1993

ATOMIC RESOLUTION ELECTRON ENERGY LOSS SPECTROSCOPY IN THE SCANNING TRANSMISSION ELECTRON MICROSCOPE

N. D. Browning, M. F. Chisholm, and S. J. Pennycook
Solid State Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-6030

SOLID STATE DIVISION
OAK RIDGE NATIONAL LABORATORY
Managed by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
under
Contract No. DE-AC05-84OR21400
with the
U.S. DEPARTMENT OF ENERGY
Oak Ridge, Tennessee

February 1993

ATOMIC RESOLUTION ELECTRON ENERGY LOSS SPECTROSCOPY IN THE SCANNING TRANSMISSION ELECTRON MICROSCOPE

N. D. Browning, M. F. Chisholm and S. J. Pennycook

Solid State Division, Oak Ridge National Laboratory, P. O. Box 2008, Oak Ridge, TN 37831. USA

Electron energy loss spectroscopy (EELS) in the scanning transmission electron microscope (STEM) is an invaluable tool for the microanalysis of materials, providing information on both compositional and electronic structure fluctuations on the sub-nanometre scale. To utilise fully the high resolution potential of the energy loss signal, it is essential to have a reference high-resolution image showing the atomic structure in the region of study. The recently developed high-resolution Z-contrast imaging technique[1] for the STEM, provides an intuitive reference image of the atomic structure that, as both imaging and microanalysis can be performed simultaneously, can be conveniently used to position the electron probe over individual atomic columns. The spatial resolution of both the image and the energy loss spectrum can be identical, and in principle limited only by the probe size of the microscope[2,3]. Therefore, for the 2.2Å probe size of the VG HB501 UX dedicated STEM, there exists the ability to be able to resolve individual atomic columns or planes.

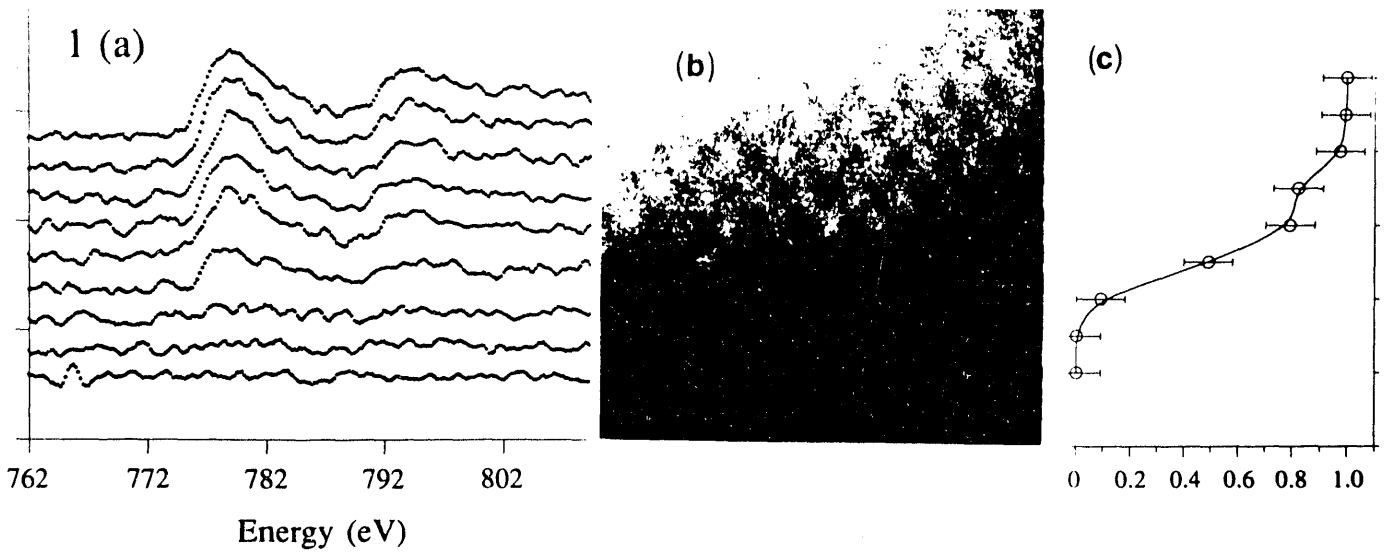
Experimentally, obtaining an energy loss spectrum from a single atomic column or plane requires that the range of the inelastic event be less than the interatomic spacing. For 100 kV electrons, using a collection aperture of 30 mrad the spatial resolution is effectively limited by the probe for energy losses above 300 eV.[2] Detecting sufficient signal in acquisition times of the order of a few seconds is necessary to reduce the effects of specimen drift and beam damage. As the Z-contrast operating condition uses a factor of 10 less beam current than is ordinarily used for microanalysis in STEM, a CCD based detection system with its uniform area, low noise and high gain is required.[4] For crystalline materials in major zone-axis orientations beam broadening in thicker specimens is reduced by beam channelling effects, though the effects of the probe profile must be considered (there exists a secondary maximum in the profile at ~3Å radius which has ~15% of the intensity of the main probe).[2]

This anticipated high spatial resolution of the energy loss signal was confirmed by the study of cobalt silicide-silicon interfaces. By using an oscilloscope linescan to center the beam accurately over successive planes across the interface, a series of 5 second exposure energy loss spectra were obtained of the cobalt $L_{2,3}$ edge (figure 1 (a)) from a cobalt silicide-silicon {100} interface prepared by ion implantation and annealing (figure 1(b)).[5]. The intensity of the L_3 edge at each atomic plane relative to the intensity of the bulk gives a measurement of the variation in cobalt composition in the region of the interface (figure 1(c)). Both image and energy loss scan indicate that the interface is not atomically abrupt, with the 2x1 reconstruction indicated in the image giving rise to a 50% intensity drop in the energy loss spectrum. In the same sample, however, a similar energy loss scan can be performed (figure 2(a)) from atomically abrupt facets in the {111} planes (figure 2(b)). The cobalt L_3 intensity profile of the interface confirms an atomically abrupt interface (figure 2(c)), with an almost 100% drop in edge intensity in moving one single plane into the silicon.

The resolution of the abrupt interface in the {111} facet agrees well with the theoretical estimate of the optimum spatial resolution. For a 3.1Å planar spacing, the effects of the probe tails should result in a ~7% change in intensity at the two planes either side of the interface. Although some effects are visible in figure 2(c), the signal-to-noise ratio ~10 makes it difficult to quantify. However, these results clearly demonstrate the possibility of obtaining atomic resolution chemical analysis by EELS. Since heavy atoms such as cobalt are easily observed by Z-contrast imaging, the technique is anticipated to have most value in the study of lighter elements which are masked by the heavier elements in the image. The combination of the two techniques can thus provide complimentary information on changes in composition at interfaces at the atomic scale.[6]

References

1. S. J. Pennycook and D. E. Jesson, Phys.Rev.Lett 64 (1990), 938
2. N. D. Browning and S. J. Pennycook, in press Microbeam Analysis
3. N. D. Browning and S. J. Pennycook, proceedings of the MRS Fall Meeting 1992
4. D. McMullan et al, Inst. Phys.Conf.Ser 98 (1990), 55
5. Sample supplied by S. Mantl of Institut für Schicht-und Ionentechnik, Forschungzentrum Jülich, Germany
6. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy systems Inc, and in part by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Program administered by the Oak Ridge Institute for Science and Education.



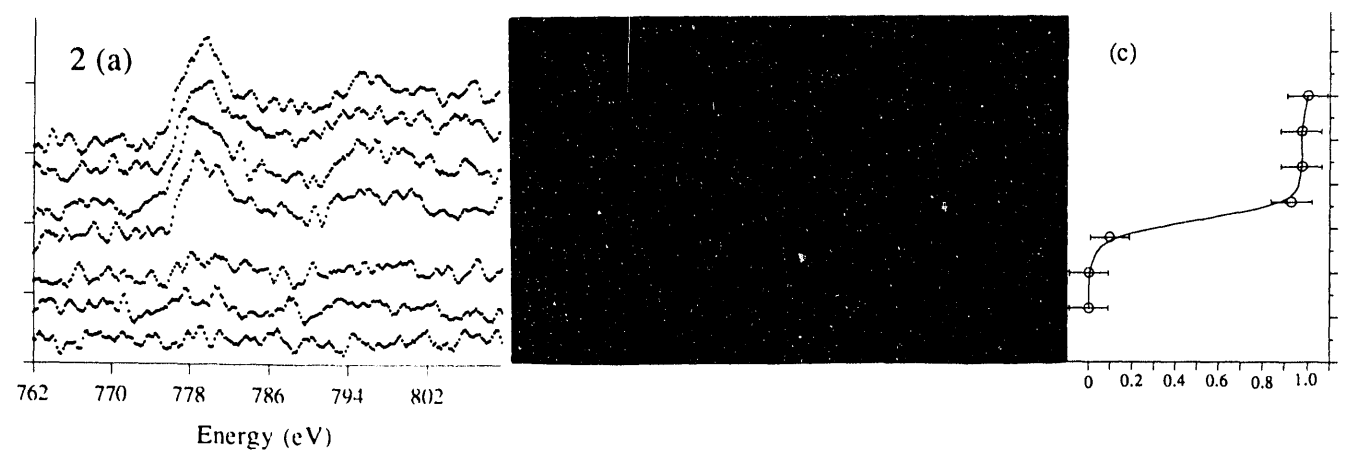


Figure 1 (a) A 5 second exposure Cobalt $L_{2,3}$ spectrum scan in single plane steps across the cobalt silicide-silicon {100} interface (b). The cobalt L_3 edge intensity as a fraction of bulk intensity at each atomic plane shows that this interface is not atomically abrupt (c).

Figure 2 (a) A 5 second exposure Cobalt $L_{2,3}$ spectrum scan in single plane steps across the cobalt silicide-silicon {111} facet interface (b). The cobalt L_3 edge intensity as a fraction of bulk intensity at each atomic plane shows the {111} facet interface to be atomically abrupt (c).

DATE
FILMED
9/22/93
END