786
doi:10.1017/S1431927611004806

Microsc. Microanal. 17 (Suppl 2), 2011
© Microscopy Society of America 2011

Applications of Atomic-Resolution EELS Mapping at Low kV

B. Schaffer,*,** F. Azough,*** D. Abou-Ras,****, S.S. Schmidt,**** M. Schaffer,*,***** M. Sarahan,*,**, Q. Ramasse,*,***** and R. Caballero***

* SuperSTEM, STFC Daresbury Laboratories, Keckwick Lane, Warrington, WA4 4AD, UK
** Kelvin Nanocharacterisation Centre, SUPA School of Physics and Astronomy, University of Glasgow, Glasgow, G12 8QQ, Scotland, UK
*** Materials Science Centre, School of Materials, University of Manchester, Manchester M1 7HS, UK
**** Helmholtz-Zentrum Berlin, Hahn-Meitner-Platz 1, 14109 Berlin, Germany
***** Department of Engineering, George Holt Building, Ashton Street, Liverpool, L69 3BX, UK

In recent years, aberration corrected scanning transmission electron microscopy (STEM) has become a widely used technique in materials science, which routinely provides atomic-resolution Z-contrast imaging. The possibility to combine image detectors with analytical detectors for simultaneous mapping of these signals at similar sub-atomic spatial sampling gives STEM a distinct advantage over other atomic-resolution imaging techniques. 2D chemical maps of individual atomic columns have been demonstrated using electron energy-loss spectroscopy (EELS) [1-5] and, more recently, energy-dispersive X-ray spectroscopy (EDXS) [6].
As high probe currents and large EELS detection collection angles have become available in new instruments, the limiting parameter in applications is often the sample itself. Outstanding sample preparation is paramount. Even with perfectly prepared samples, beam damage and specimen drift induced by electron beam interactions often dictate a narrow set of suitable experimental parameters for 2D mapping, limiting the attainable signal-to-noise (SNR) ratio. Clearer maps can often be obtained by applying post-processing techniques such as principal component analysis (PCA), multiple linear least-square (MLLS) fitting of references, or by other multivariate statistical analysis techniques [7-9]. Such techniques are explored and developed in house.
Fig.1 shows an example of 2D EELS SI data of a $Ba_{6-3x}Nd_{8+2x}Ti_{18}O_{54}$ ceramic. The 2D EELS SI data set was acquired with the aberration corrected NION UltraSTEM at SuperSTEM/Daresbury equipped with a Gatan ENFINA electron spectrometer and operated at 100 kV.
Recently, the material class of sp^2-bonded monoatomic layers has gained strong attention. Many of these samples can not withstand electron irradiation sufficiently long to facilitate reliable EELS mapping at higher acceleration voltages. Reducing the voltage to 60 kV or below has proven a successful strategy to produce atomically resolved 2D EELS maps with the UltraSTEM in our lab.
However, if beam damage prevents even 1D line-scan acquisitions of sufficient SNR, distributed-dose acquisitions may also be applicable. The Smart Acquisition [10] routine scans the beam rapidly through a set of pre-defined positions during acquisition of a single spectrum. While some spatial information is sacrificed in the averaged spectrum, the increased exposure time or reduced beam damage often reveals otherwise inaccessible information.
A typical application of the Smart Acquisition approach is the averaged line-scan as shown in Fig.2. Here, elemental profiles of a $Cu(In,Ga)Se_2$ thin film across grain boundaries and defects can be gained with sufficient SNR. Notably, the atomic resolution is retained in the profile direction.

In this work, we present the application of atomic resolution EELS analysis on different materials systems, highlighting obstacles and solutions of the technique.

https://doi.org/10.1017/S1431927611004806 Published online by Cambridge University Press

References

[1] M. Bosman et al., *PRL* 99 (2007) 086102.
[2] K. Kimoto et al., *Nature* 450 (2007) 702.
[3] D.A. Muller et al., *Science* 319 (2008) 1073.
[4] Varela et al., *PhysRev B* 79 (2009) 085117.
[5] G.A. Botton et al., *Ultramicroscopy* (2010) 926.
[6] M. Watanabe et al., *Jeol News* 45/1 (2010) 8.
[7] N. Bonnet et al., *Ultramicroscopy* (1999) 97.
[8] M. Sarahan et al., *Ultramicroscopy* (2011) 251.
[9] F. de la Pena et al., *Ultramicroscopy* (2011) 169.
[10] K. Sader et al., *Ultramicroscopy* (2010) 998.
[11] The authors would like thank EPSRC for funding under grant EP/D040205/1.

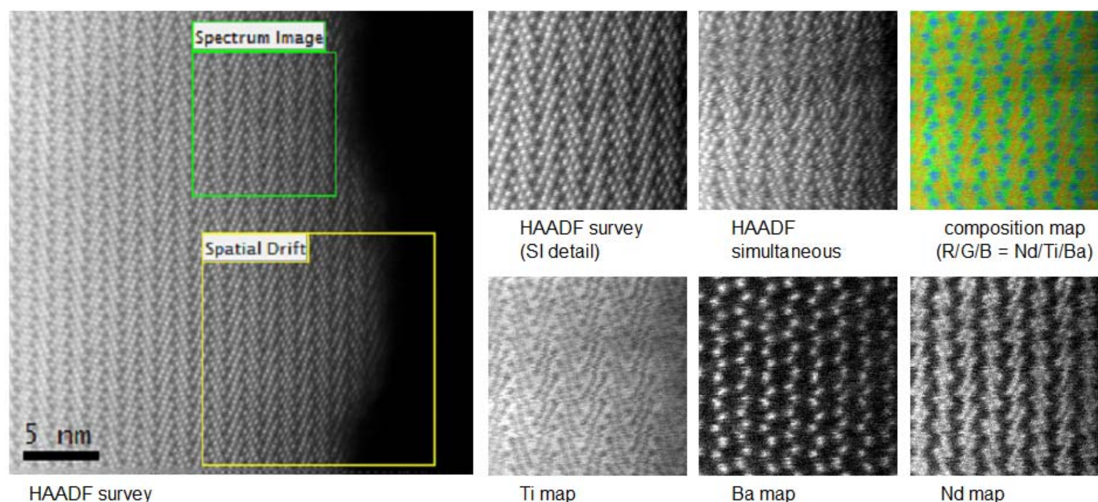

FIG. 1. Example of atomically resolved 2D EELS mapping of a $Ba_{6-3x}Nd_{8+2x}Ti_{18}O_{54}$ ceramic.

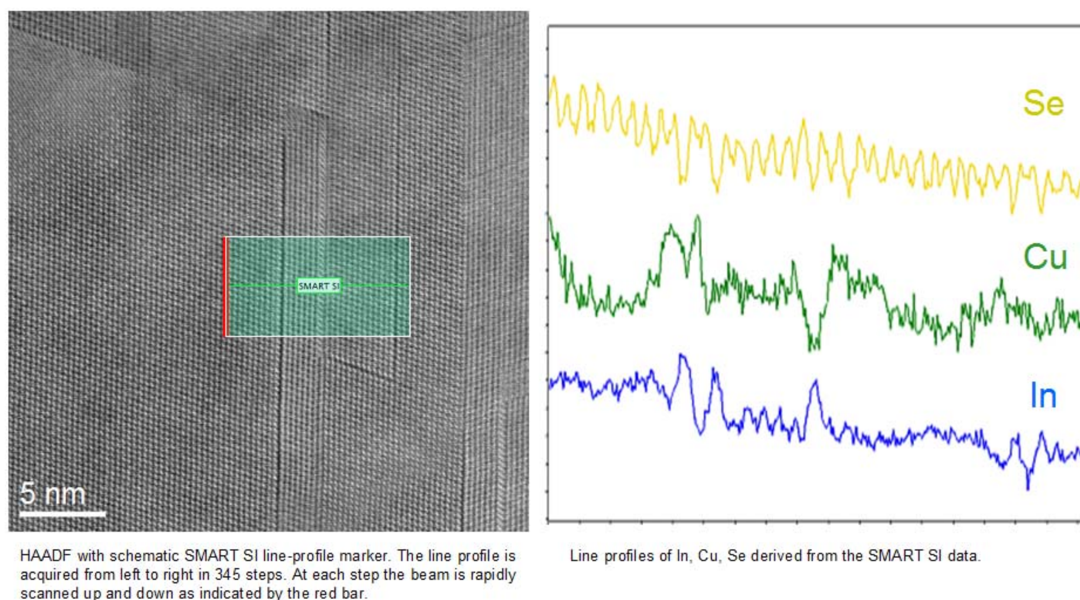

FIG. 2. Example of a Smart Acquisition line scan of a $Cu(In,Ga)Se_2$ thin film in a solar cell. Atomic resolution in scan profile is given, while the profile is averaged in perpendicular direction.

https://doi.org/10.1017/S1431927611004806 Published online by Cambridge University Press