

## Transmission EBSD – Bridging the Gap between SEM and TEM

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With evermore powerful SEMs available on the market, the boundary between what could traditionally be accomplished using SEMs and TEMs is being continuously eroded. We have seen over the last several years the development of 3D reconstruction using FEG-SEMS with resolution approaching conventional TEM [1]. STEM imaging on SEM systems can achieve a resolution better than 7 Å in all three modes (BF, DF and HAADF). This resolution capability, in conjunction with the ease of operation and versatility of the SEM, has made this a more accessible and useful system for materials research. The primary drawback of this approach has been the lack of crystallographic information to accompany the imaging data. In addition to already existing STEM imaging in various modes, we now have the ability to obtain fully automated transmission-EBSD maps on TEM samples in an SEM. This new platform makes it possible to conduct, with relative ease, in-depth experiments combining EDS mapping and crystallographic analysis with conventional imaging techniques [2-3]. One point that will have to be closely investigated is the volumes from which the information originates. However, this concern applies to combined EDS and EBSD in the SEM mode as well. The flexibility to load up to six samples with a standard FEI STEM holder in a Nova 200 Nano-lab, shown in Fig. 1a, is a very useful, productive, and efficient aspect of the technique.

The new generation of integrated EDS-EBSD systems can acquire both chemical composition and crystallographic information as full 2D maps or (point measurements) simultaneously, and allows immediate identification of the crystal phase and orientation (Fig. 1b). (We note that NanoMEGAS introduced the ASTAR system a few years ago for automated acquisition of diffraction information in TEM. It is still in its early stages, and is not yet widely available in modern TEMs). When chemical and crystallographic analysis is combined with dual beam FIB capability, a system is available that allows the user to prepare a site-specific TEM sample and completely characterize it in the SEM.

In this paper we will present examples of data obtained from different lift-out samples. We will examine the orientation relationship of an Al<sub>2</sub>O<sub>3</sub> scale formed at high temperature on a Ni-base bond coat substrate, and chemical composition and crystal structure of a multi-component oxide slag. As the main example, a lift-out sample from a Ni-base super-alloy will be shown that was extracted from several grain boundaries that experienced significant strain accumulation, as had been determined by full field strain mapping [4]. TEM analysis and conventional EBSD analysis carried out on the same sample will also be presented, to demonstrate how multiple characterization techniques can be used for optimum data interpretation (see Fig. 2).

These recent developments have the potential to usher in a new generation of SEMs, equipped with fast position-sensitive detectors that can be inserted below the sample and will enable in the future extracting different signals (t-EBSD, BF, DF, and HAADF) and obtaining the desired image, based on software integration of the signal, pixel by pixel, in different configurations. Another possible outcome may be

microscopes with 40kV e-beams or higher on SEMs to obtain better resolution and the ability to work with thicker samples.

#### References:

- [1] A Avishai *et al.*, *Microscopy and Microanalysis*, **17**, Supplement S2, June (2011), p 666.  
 [2] R R Keller and R H Geiss, *Journal of Microscopy*, **245** (2012), p. 245.  
 [3] P W Trimby, *Ultramicroscopy*, **120** (2012) p. 16.  
 [4] J L W Carter, *et al.*, in *Superalloys 2012*, TMS, ed. E S Huron, (John Wiley & Sons, Inc., Hoboken, New Jersey) (2012), p. 43.  
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