A New Approach for Contrast Segmentation of Microstructural Features for 3D-Imaging and Material Characterisation

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With the widespread application of microscopy in natural sciences, it is evident that two-dimensional information has limitations concerning connectivity of features etc. Conversely, three dimensional (3D) observations of objects often provide direct links to their functionality. As a result, the development of microscopic techniques has generally progressed into "3D microscopy". 3D microscopy information has also become more informative for materials science, and many analytical microscopy techniques have introduced 3D methods over the past twenty years.

A relatively new, in-situ ultramicrotome – containing scanning electron microscope (GATAN 3View placed in an FEI Quanta 250 FEG SEM) is ideal for 3D microscopy, giving tools for analytical probing at the microscale and nanoscale in a single instrument. The stage design does not require major image alignment, and tilting is not necessary as for conventional SEM imaging, but unlike focused ion beam approaches. Concerning the specimen, a sharp-edged diamond knife is utilized for generation of slice thicknesses in the range 10 to 100 nm. As a consequence of the previous sequential slicing, fresh, relatively smooth and non-contaminated surfaces of the specimen are available routinely for low voltage, high resolution imaging without charging in the pressure-controlled SEM chamber.

The sequential slicing and imaging steps yield a stack of images that are sampled equidistantly through the volume of interest and, finally, that are available for 3D reconstruction. Within the LATEST2 project, this novel and innovative approach has successfully generated 3D images to give better understanding of the materials of interest. In studies of metallic alloys and their performance, i.e. corrosion behaviour, it is important to quantify the distribution of intermetallic particles and their dimensions, grain size and grain orientation, and the locations of particles within the alloy microstructure, as well as the composition of the material of interest and connectivity of features and relation to the corrosion front.

Series of images are usually obtained using a backscattered electron (BSE) detector. The detector collects deflected primary electrons due to the strong electrical field of the specimen's atomic nuclei. Therefore, BSE imaging is highly dependent on the atomic numbers of the materials of interest. Figure 1 shows the relationship between the BSE coefficient and the average gray scale of intermetallics and the aluminium matrix in AA2024 T3 at 2.5 kV. In order to confirm the relationship with the gray scale level, composition quantification was obtained by energy dispersive X-ray analysis (EDX) in the green circled regions displayed in Figure 1. The BSE coefficient is in good agreement with the gray scale; the blue and red arrows indicate the Al_2MgCu (S) and Cu_2Al (Θ) phases respectively. It is also evident that such information is able to contribute to a database for various materials. In summary, the established database then provides information for elemental 3D imaging without further EDX analysis.

A further advantage of SEM imaging is the presence of electron channeling effects, which result from change of electron penetration depth with different crystal orientations in the same material. This penetration depth difference reflects the BSE signal intensity as a function of angle relative to the Bragg angle. These signals are generated from a few nanometres below the surface. This intensity difference is able to locate grain boundaries and, thus, enable measurement of the grain size distribution. However, the relatively low electron penetration depth renders the signal sensitive to the roughness and deformation of the block face (the block being the specimen that is sliced sequentially with the diamond knife). In fact, forces applied on the block face as a result of mechanical slicing with the diamond knife introduce some roughness and deformation on and immediately below the block face respectively. This roughness and deformation may change the surface crystal orientation locally, with the result that the image shows differences of regular gray level, with such differences being distinct in individual grains. Figure 2 shows the difference of regularity of gray level on individual grains of an aluminum alloy specimen, and reveals the 3D reconstruction, with segmentation imaging based on this difference.

The understanding of the relationship of the regular gray scale difference with roughness and deformation, and the reduction of associated damage on the block face are crucial for future advancement of this analytical approach. Current work is aimed at minimizing or avoiding damage to the block face, which will allow the next important advance of understanding through strain or deformation measurements and 2D and 3D EBSD.

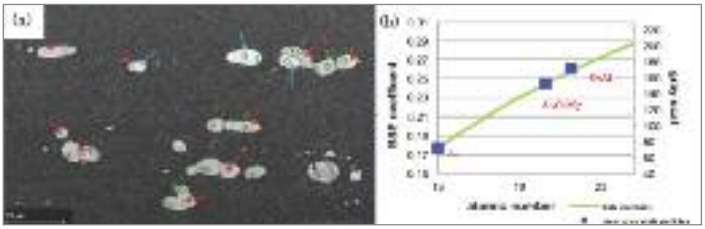

Fig. 1 (a) BSE image of AA2024 T3 at 2.5 kV. (b) Variation of the BSE coefficient with average gray scale of the intermetallics revealed in the image.

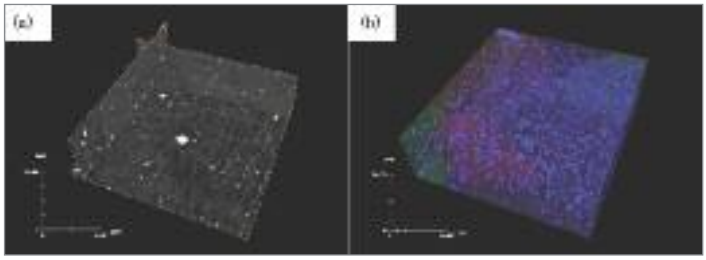

Fig. 2 3D reconstructed image of AA 2024 T3 aluminium alloy. The true intermetallic particle shapes and distributions provide accurate volume fractions and locations for informing thermomechanical processing for material performance: (a) orthoslice imaging of 3D volume; (b) segmented imaging of intermetallics and grains (the intermetallics are coloured blue and the aluminium rich matrix grains are coloured green, red, yellow and light blue).