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3D insights into nickel converter matte phases: Direct observations via TEM and FIB SEM tomography

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ABSTRACT

A prior application of mineralogy to the analysis of nickel converter matte was based on two dimensional in-plane projections of three dimensional phase structures. Recent developments in electron microscopy have established suitable techniques to base further analysis on actual three dimensional projections. Focused ion beam scanning electron microscopy tomography in arrangement with transmission electron microscopy was considered suitable in acquiring three dimensional projections of nickel converter matte phase structures at the mesoscale with subsequent reconstruction for 3D visualization and analysis.

Transmission electron microscopic section analysis was particularly useful in signifying that phase structures were geometrically arranged within an underlying nickel-sulfide microtexture. Tomography reconstruction and rendering of a rectangular particle volume allowed for color and grayscale based 3D visualization of the nickel-sulfide microtexture, copper-sulfide and NiCu-alloy phase structures. Color based 3D visualization was specifically effective in assigning a cubic morphology to smaller alloy phase structures. Grayscale based 3D visualization of alloy phase structures illustrated compositional zones correlating to the presence of bright Pt-dominant cores and darker Ni-dominant rims. High-fidelity reconstruction of developed Pt-dominant lobes was produced illustrating insightful morphological detail. It would be important to consider the three dimensional insights gained to the downstream metallurgy of nickel converter matte.

1. Introduction

Fast cooled nickel converter matte particles within the South African pyrometallurgical industry are fine-grained, multiphase and a complex intermediate product with respect to downstream comminution and hydrometallurgical unit operations. The application of mineralogy to the analysis of pyrometallurgical products is particularly useful for the control of furnace process conditions (Hagni et al., 1987; Andrews and den Hoed, 2011). A particular relevant case study describe the analysis of nickel converter matte as a function of iron endpoint and yielded significant opportunity for optimal monitoring and control of nickel converter blow conditions (Thyse et al., 2011a). The study was conducted applying scanning and transmission electron microscopy (SEM and TEM) integrated with energy-dispersive X-ray spectroscopy (EDX) to two dimensional (2D) planes of polished matte particle areas. Mineralogical analysis from particle areas revealed that the microstructure is dominated by a nickel-sulfide microtexture representing solid phase, embedding euhedral copper-sulfide and NiCu-alloy phase structures. Further analysis revealed that alloy phase structures would act as collectors of PGEs and exhibited a variety of morphological forms and associated compositions. Their most noticeable occurrence was as well developed and thus relatively larger cored dentritic-like phase structures (Thyse et al., 2011b). The analysis was based on 2D in-plane projections of three dimensional (3D) phase structures and it would follow to base further analysis on actual 3D projections.

Tomography includes specialist techniques in 3D image acquisition and reconstruction wherein the nature of the specimen in conjunction with the analytical requirements would commonly determine the suitability of such a technique. Electron tomography with typical resolutions below 1 nm^3 and a volume of view estimated to 100 s nm^3 are commonly applied in material science to study the morphology and composition of materials at the nanoscale (Midgley and Dunin-Borkowski, 2009). X-ray computed tomography can have resolutions below 100 nm^3 and has been readily integrated into the study of crack distribution and mineral dissemination of sphalerite particles (Ghorbani et al., 2011) and heap leaching (Dhawan et al., 2012). Recent advances in dual

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(electron and additional ion)-beam scanning electron microscopy have established a suitable technique for tomography at the meso-scale, 20 nm to 20 μm (Midgley and Dunin-Borkowski, 2009). Focused ion beam scanning electron microscopy (FIB SEM) tomography in versatile combination with TEM was therefore deemed suitable in acquiring 3D projections of nickel converter matte particle areas with subsequent reconstruction for 3D visualization and analysis.

2. Methods

The TEM specimen preparation was completed using a FEI HELIOS Nanolab 650 FIB SEM. The microscope is fitted with a Tomahawk Ga ion beam column for milling, a carbon precursor gas injection system for deposition of carbon and an Omniprobe autoprobe 200 for manipulation of TEM lamella.

The preparation of a TEM specimen using a FIB SEM is a challenging and lengthy process. For the preparation of the TEM lamella a site of interest (SOI) on the specimen was selected in electron backscattered imaging mode. Fig. 1a shows the proposed FIB section along the selected SOI. After identification of the SOI a protective layer of carbon was deposited over the area of interest before the commencement of 30 kV ion beam milling of trenches on either side of the SOI. This was done to expose and prepare a $5 \times 10 \times 2$ μm lamella for removal (see micrograph in Fig. 1b) from the SOI and attachment onto a copper finger on a special TEM grid. The removal of the lamella is achieved by attaching the Omniprobe needle to the lamella using C deposition. After the lamella is attached to the needle it is cut free using the ion beam and moved to a prepared position on the copper finger for attachment. After careful manipulation, the lamella is placed into slight contact with the copper finger and fixed into place by deposition of C. The lamella was then progressively thinned using the ion beam from both sides to a final thickness of approximately 30 nm as confirmed by edge on SEM imaging. Final polishing of the lamella was done using a 2 kV and 500 V ion beam energy to ensure removal of amorphous material from the surface of the lamella.

The TEM analysis of the lamella was done using a JEOL JEM 2100 LaB_6. Both TEM and scanning TEM (STEM) modes along with selected EDX was used to study phase and elemental distribution in the specimen.

A tomography reconstruction of several serially sectioned slices obtained from another SOI in the specimen was completed using the slice and view facility of the FIB SEM. After selection of an appropriate region, trenches were milled around a rectangular volume in the specimen. The rectangular volume was then sequentially sliced by the ion beam and the exposed cross sectional surface imaged in backscattered mode using the electron column. Each section or slice was 20 nm apart and resulted in 246 images or slices. The resolution of each slice is related to the features within and would be finer than the slice thickness. The overall resolution of the tomography reconstruction would therefore be a function of their combination. After acquisition of the images a normalization routine was used in the processing program ImageJ to remove any background contrast gradients on individual images and also to normalize contrast limits between images. Noise reduction was then done on the images. The image stack was then imported into the software package Amira, aligned and cropped. Following this, a slight smoothing of the image stack was done. Segmentation and 3D volume representation of the stack was then done using contrast thresholding. Fig. 2a and b shows the reconstructed rectangular volume and a single sectioned slice in backscattered electron mode at 17.5 and 8 μm size scales, respectively.

3. Results and discussion

TEM in arrangement with FIB SEM tomography were therefore applied to nickel converter matte particle areas for 3D visualization and analysis.

3.1. TEM analysis of lamella

The SOI as shown in Fig. 1a is characterized by a bright PGE structure as well as NiCu-alloy dentritic-like phase structures, seemingly embedded in the larger nickel-sulfide microtexture. Fig. 3 provides 2D in-plane imaging and analysis of the prepared lamella (section along SOI) using both the STEM and EDX attachments. Fig. 3a in particular, provides illustration of the distribution and morphology of phase structures present within the section to an approximate depth of 4 μm.

The section seems dominated by a morphologically indistinguishable and underlying nickel-sulfide microtexture. The previous PGE structure is observed to extend within the nickel-sulfide microtexture and EDX analysis revealed the presence of an Os-dominant core along with a lower concentration transition-zone and rim. The size of the Os-dominant phase structure in Fig. 3a can be estimated as just below 1 μm in the elongated *x* and *y*-direction. EDX analysis indicated the presence of a smaller Pt-dominant core located directly below the Os-dominant phase structure. Noticeable dentritic-like structures appears to extend from the

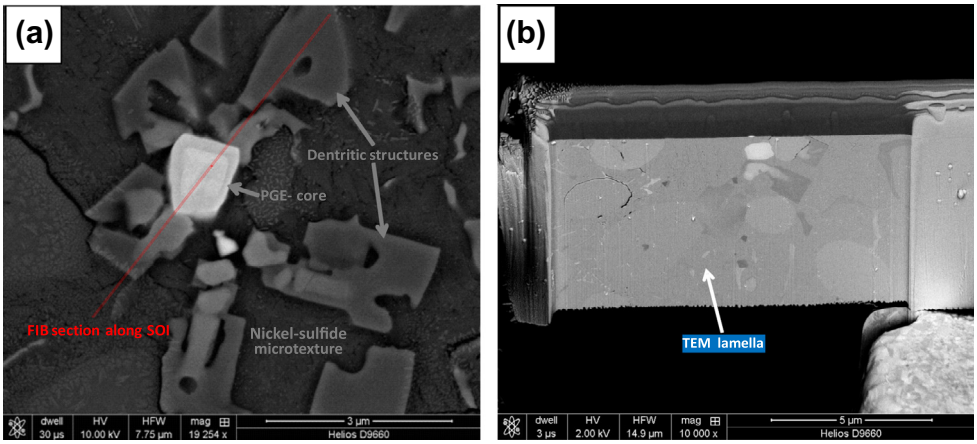

Fig. 1. Backscattered electron-induced micrographs showing (a) the proposed FIB section along selected site of interest at 3 μm size scale and (b) the manipulation of TEM lamella at 5 μm size scale.