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Nano-scale 3-D characterization of materials using FIB-SEM

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Nano-scale characterization in three dimensions can provide essential information regarding structural, optical or chemical behavior of materials. Dual-column Focused Ion Beam–Scanning Electron Microscopes (FIB-SEMs) have been successfully used for microstructural characterization of materials and devices through sequential milling and imaging ('slice-and-view' technique). Combined with image reconstruction, the approach demonstrates ability to study key microstructural features in 3-D [1-2].

In this paper, we describe the challenges and achievements of the 'slice-and-view' approach applied to complex material systems and taken to nano-scale. We will demonstrate results of technique application to various materials such as metal-polymer, nano-scale gamma-prime phase in superalloys, nanotubes and nanorods. One of the examples is metal nano-particles imbedded into polymer matrix on a glass substrate. The typical dimensions of the particles are 50-75nm. The slice-and-view sequences have been collected to study spatial distribution of the Au particles in 3-D. Typical slices were about 25 nm thick. The study was carried out using an FEI Nova 200 Nano Lab FIB-SEM. Amira 3.1 software package has been used for 3-D reconstruction.

Figure 1 shows 3-D reconstruction of the 3.4 x 3.4 x 0.75 μm data set with the sample being cross-sectioned normal to Z direction in the image. The data obtained from such 3-D characterization is critical for correct nano-particles density evaluation. Typically, particle concentration is estimated from SE images of the FIB cut surface (Figure 2). However, we need to take into account that SE images are 2-D representations of 3-D objects, since metal particles exhibit contrast even if located 15-20 nm below the polymer surface. The image analysis algorithm does take this into account, but only the 3-D data from FIB-SEM 'slice-and-view' sequence provides complete information on spatial distribution and particles concentration.

References

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[2] R.E.A. Williams, M. D. Uchic, D. M. Dimiduk, H.L. Fraser, Microsc. Microanal. 11 (Suppl. 2), 2005

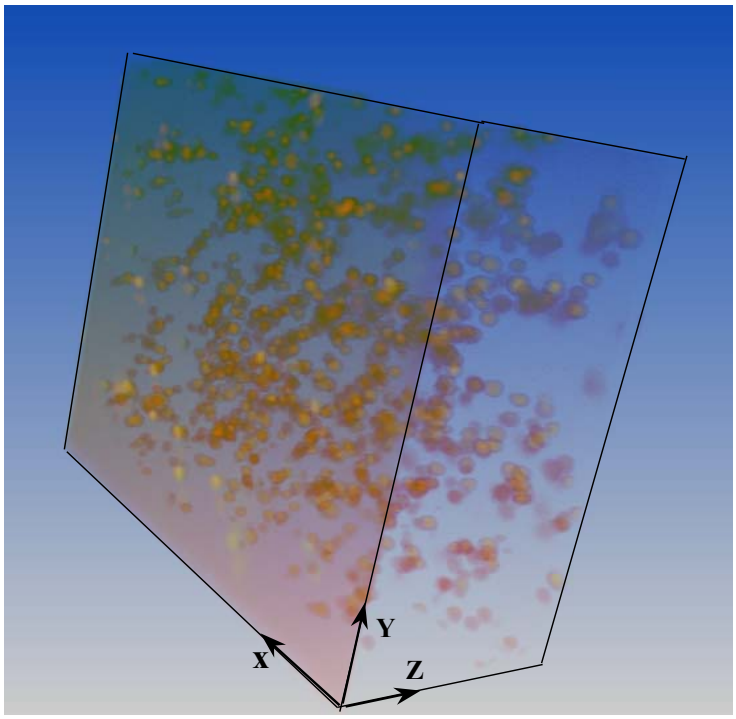

FIG. 1. 3-D representation of data set from FIB serial sectioning normal to Z direction in the picture. Lateral dimensions of the cut: X=Y=3.4μm.

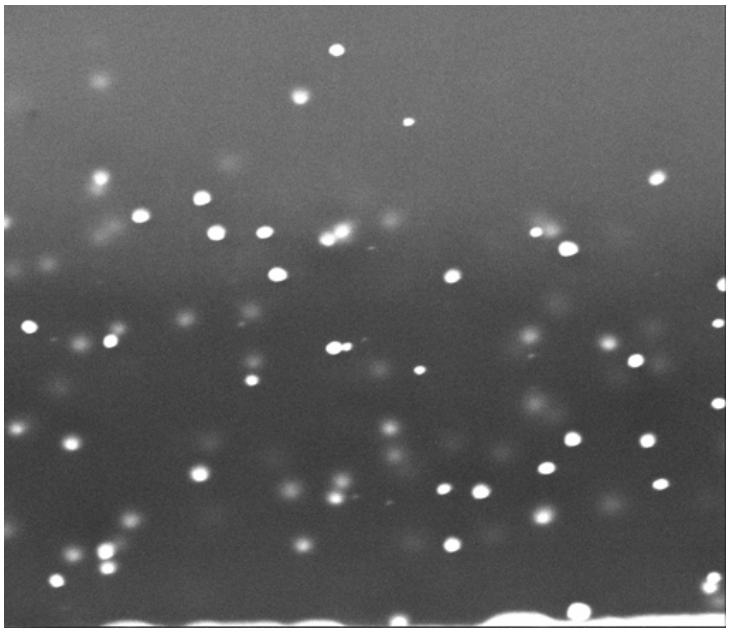

FIG. 2. SE image of the cross-section made by FIB showing ~75nm Au particles in PVA. Same magnification as in the 3-D rendering (Figure 1).

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