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Simultaneous Bright Field and Dark Field STEM-IN-SEM Imaging of Hard-Soft Composites and Crystalline Materials

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Scanning transmission electron microscopy in scanning electron microscopy (STEM-IN-SEM) [e.g. 1] is a convenient technique for polymer characterization. Utilizing the lower accelerating voltages, larger field of view and, exclusion of post-specimen projection lens in an SEM; STEM-IN-SEM has shown results comparable to transmission electron microscopy (TEM) observation of polymer morphology [2]. Various specimen-holder geometries and detector arrangements have been used for STEM-IN-SEM imaging [e.g. 3, 4]. The authors have developed a technique to facilitate simultaneous bright field (BF) and dark field (DF) STEM-IN-SEM imaging [5]. The geometrical configurations and design of specimen holder for this application can be found elsewhere [5]. While the primary focus of this technique is to characterize soft materials there are potential applications of STEM-IN-SEM imaging to harder materials including crystalline samples. Here potential applications are explored.

Figure 1 shows a pair of BF and DF images from an ultramicrotomed and unstained epoxy-based composite filled with nanosilica particles. The low magnification BF image (left) exhibits mass-thickness contrast. The DF image (right) seems more intuitive to interpret because the contrast mainly relates to local average atomic number (Z). In the high magnification DF image, the darkest and brightest regions are the epoxy matrix and the nanosilica particles, respectively. BF and DF STEM-IN-SEM can be used for measuring particle size and distribution of hard-soft composites from a relatively large field of view. In addition, the time efficiency of this technique allows for the screening of many samples as a precursor to electron energy loss spectroscopy (EELS) analysis of hard-soft composite interfaces via STEM.

Figure 2 shows a pair of BF and DF images from a focused ion beam (FIB) cut undoped alumina with nickel particles. The BF image (left) exhibits mass-thickness contrast. In the BF image, darker regions represent the nickel particles and the different grey-scale regions represent the undoped alumina. The DF image (right) shows mixed contrast with the undoped alumina regions exhibiting diffraction contrast while dark regions represent nickel. In this case, higher Z regions appear darker in DF image, which no longer represents Z contrast. Since these images were acquired at 30 kV in SEM, the electron penetration power is limited, especially in higher Z materials. Therefore, both BF and DF signals in the Ni particles are reduced simply due to lack of electron penetration. Obviously, the lower electron penetration power is a limitation for STEM-IN-SEM imaging of crystalline materials. However, the time efficiency of BF & DF STEM-IN-SEM can be used to readily evaluate the electron transparency of FIB-cut samples during final thinning before subsequent analysis by TEM or STEM.

Further characterization of hard materials by BF & DF STEM-IN-SEM is ongoing. In addition to imaging, an accessory for our BF & DF STEM-IN-SEM holder [5] is being developed to allow for energy-dispersive X-ray spectroscopy (EDX) and electron backscatter diffraction (EBSD) analysis of crystalline materials.

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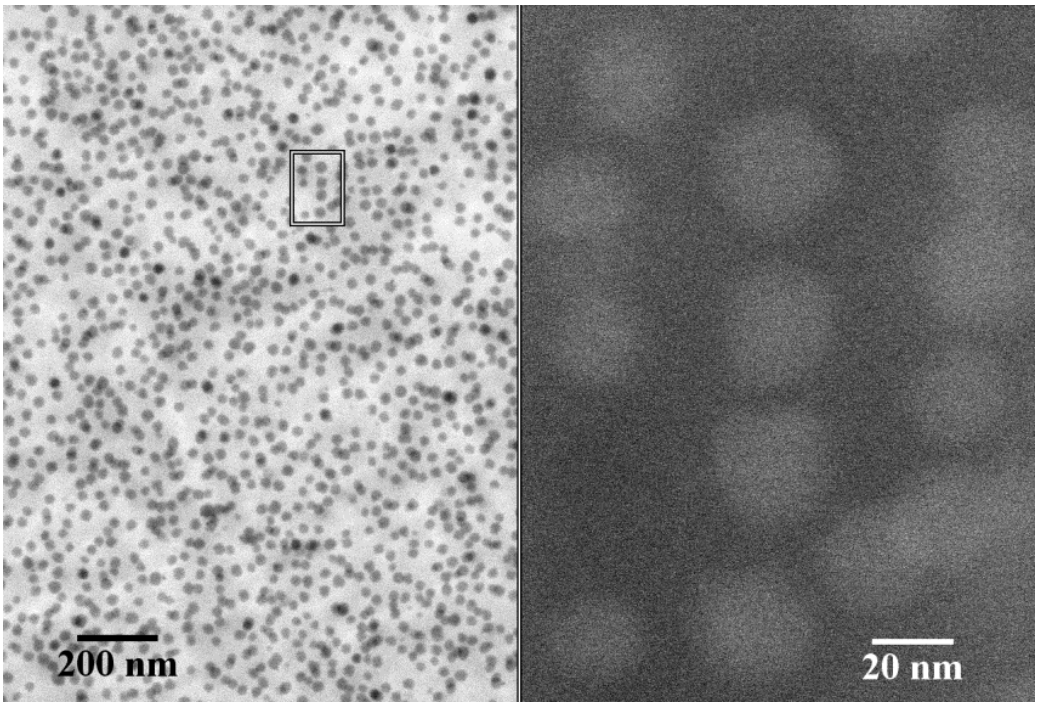

FIG. 1: BF (left) and DF (right) images simultaneously acquired from an ultramicrotomed and unstained epoxy-based composite filled with nanosilica particles by the STEM-in-SEM technique at 30 kV.

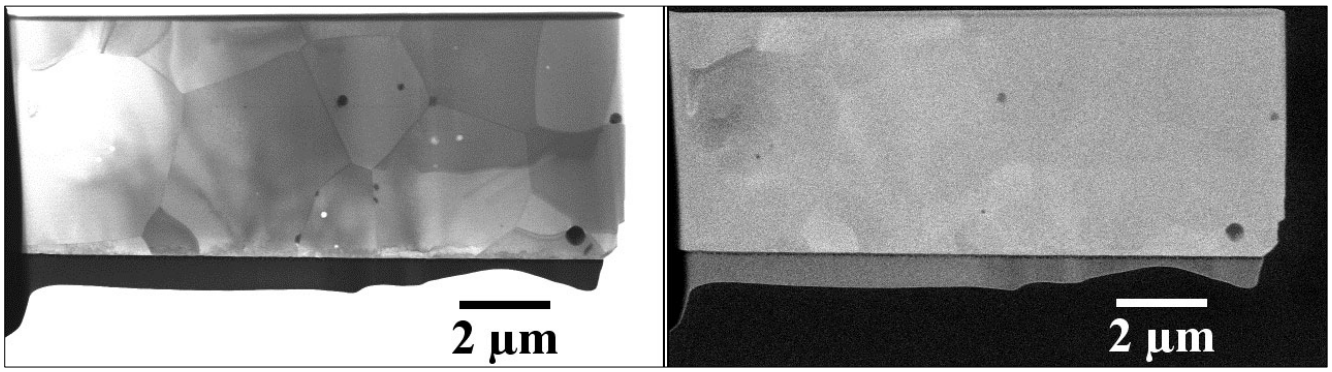

FIG. 2: BF (left) and DF (right) images simultaneously acquired from a FIB cut undoped alumina with nickel particles sample by the STEM-in-SEM technique at 30 kV.

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