Characterization of Nonconductive Polymer Materials Using FESEM

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One of the most challenging SEM characterization areas at our Electron Microscopy Lab [1] is currently found for characterizing nonconductive nano materials, especially for increasing demands from nano polymer research. This article gives an overview of using our Zeiss Ultra 55 and Supra 55VP SEMs for characterization of un-coated nonconductive materials.

For conventional SEM imaging of nonconductive materials, specimens need to be coated for preventing the accumulation of electrostatic charge. However for porous nano-composite polymer [2], sputter coating would potentially have the impact on SEM imaging of the nanoenhanced particles. Fig. 1 shows a variable pressure (VP) SEM micrograph of the nano particle enhanced polymer from Zeiss Supra SEM. The SEM chamber gas pressure was carefully controlled to 8 Pa for achieving beam charge stabilization, and also for stabilizing the porous polymer by reducing shirking under electron irradiation. VPSEM resolution is strongly limited by the complicated beam-specimen interactions, and only the VPSE detector is available for SE imaging. To get higher resolution low voltage SE imaging is applied for the relative condensed polymer-particle characterization [3]. In low voltage mode by controlling the electron landing energy, a dynamic charge balance can be built for the incoming and outgoing electrons on the sample surface. At 1 kV, SE1 raises rapidly from a small interactive volume with a narrow angular distribution. Together with using collecting-efficient through-the-lens (In-lens for Zeiss) detector, the resolution at low voltage mode could reach at the nanometer level [4]. Fig. 2 shows a SEM micrograph of the polymer particles from Zeiss Ultra In-lens detector at 3 kV with a working distance (WD) of 3 mm, where the artifacts were caused from slightly over charging. As decreasing down to 1 kV and WD to 1 mm, the dynamic charge balance was nearly reached as shown in Fig. 3, which is the enlargement of the white rectangular region in Fig. 2. The horizontal streaks on the particles in Fig. 3 were also caused by tiny over charging from 1 kV incident beam, which could be fully removed by further decreasing to 0.5 kV as shown in Fig. 4. All of these micrographs were recorded by using a 10 μm in diameter aperture to limit beam divergence, which gives benefits to reduce both the beam spherical and chromatic aberrations, and further increasing depth of field at such a short WD. By using this optimized setting with the detailed surface information, a series of nanoindentation tests were carried out for understanding particle nano-mechanics [3].

Compositional characterization can also be performed at this low voltage mode by using the Zeiss EsB detector [5] through filtering potential control for collecting only low-loss BSE signals. The initial test result is shown in Fig. 5 with metallic coating, and the low-los BSE indicates coating segregation to the triple junction with the filtering grid of 330 eV.

References

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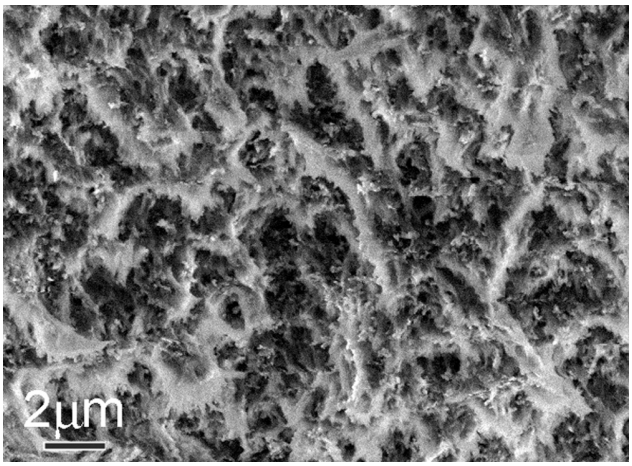

FIG. 1. VPSEM micrograph of a nano composite polymer under the 8 Pa gas pressure at 5kV.

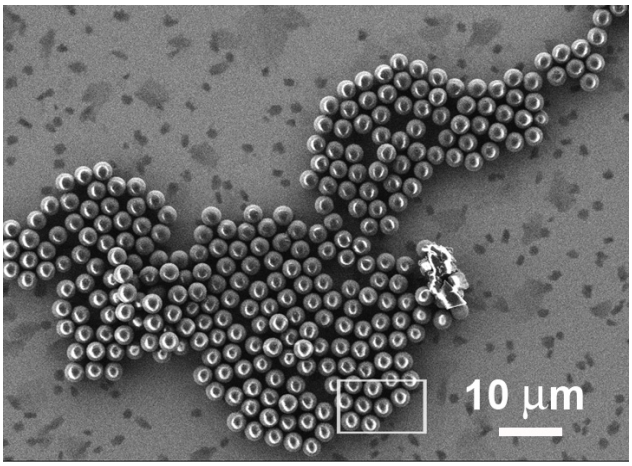

FIG. 2. LVSEM micrograph of the polymer particles at 3 kV and WD of 3 mm.

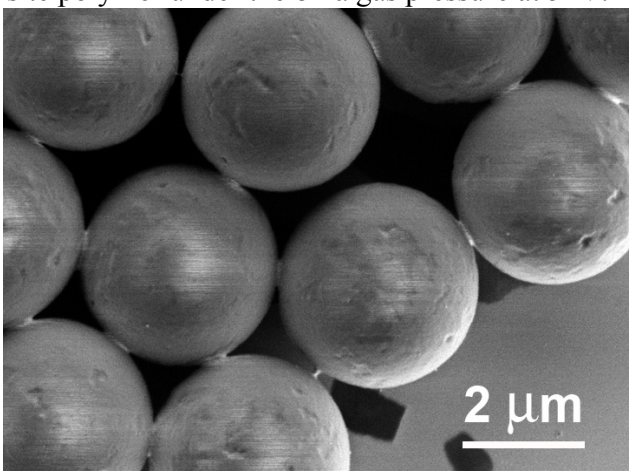

FIG. 3. LVSEM micrograph of the polymer particls at 1kV and WD of 1 mm.

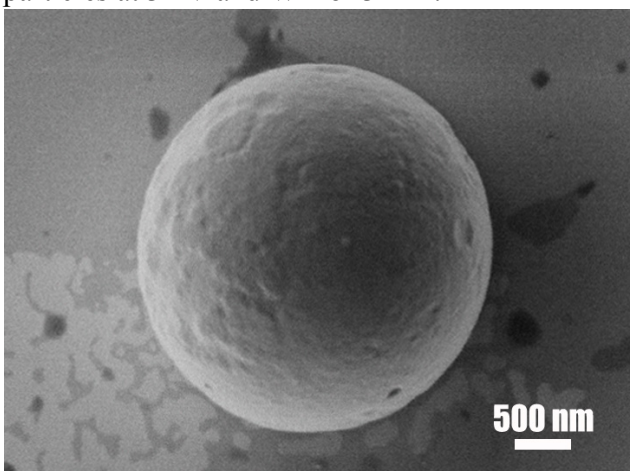

FIG. 4. LVSEM micrograph of the polymer particles at 0.5 kV and WD of 1 mm.

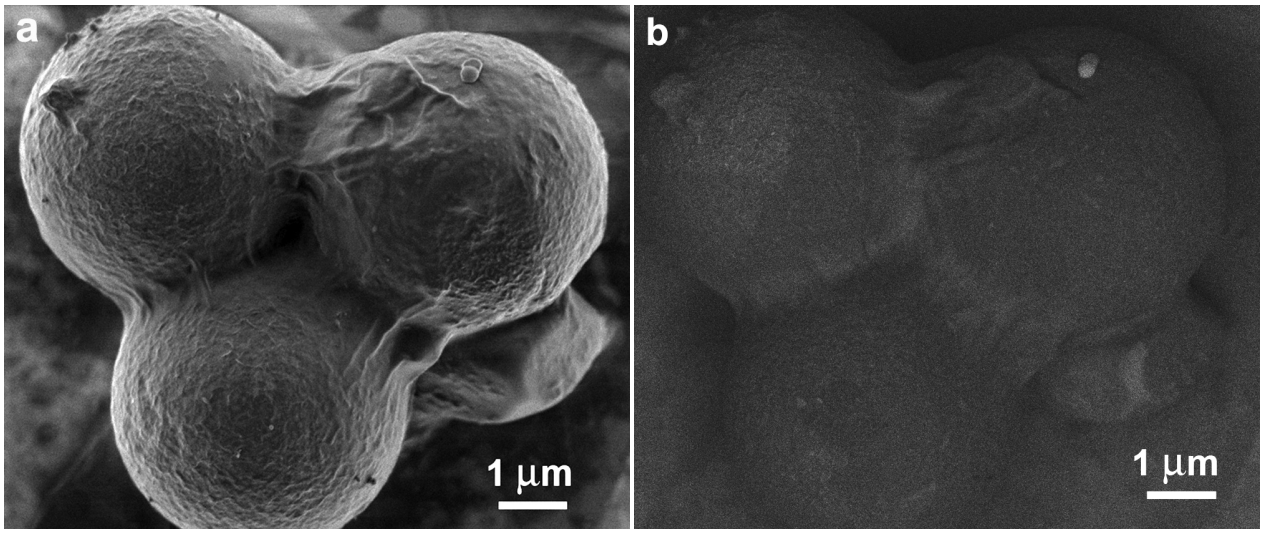

FIG. 5. LVSEM micrographs of the polymer particles with Au-Ni metallic coating. (a) In-lens SEM image at 0.5 kV and WD of 1 mm. (b) Low-loss BSE image of (a) at the filtering grid of 330 eV.