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IT-4-P-2553 Nanostructure Imaging with Topographic and Compositional Contrasts in a Cold-Field Emission Scanning Electron Microscope at Low Accelerating Voltage and with Energy-Filtration

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For developing new technologies, it is important to characterize the microstructure of materials with high spatial resolution at the nanoscale. To achieve high resolution, field emission scanning electron microscopes (FE-SEM) were developed. These microscopes allow working at low accelerating voltage, below 5 kV, to take advantage of the reduction of the interaction volume with accelerating voltage (from 1 µm in Al at 10 kV to 10 nm at 1 kV). Furthermore, their higher gun brightness compared to conventional thermo-electronic emitters, allow a probe size at the nanoscale. However, technical problems arise when SEM operates at low kV, i.e., the source brightness decreases and the chromatic aberration increases, all SEM parameters being equal. Using deceleration mode minimizes these problems and further improvement is achieved by using a cold-field emitter, which has a smaller energy spread and providing the highest brightness and the smallest source size of a FE-SEM. At low accelerating voltage, the emission volume of backscatter (BSE) and secondary (SEII, emitted by BSEs) electrons signals approach that of SEI (emitted by the primary electrons) signals. However it is not enough to reach the highest resolution. A magnetic field above the sample improves the spatial resolution by collecting mostly high-resolution signals. In addition, the energy-filtration of the electron signals allows selecting the type of contrast detected: topographic, compositional, or crystallographic.

Examples of high spatial resolution imaging are shown in Figure 1. Topographic imaging at a very low accelerating voltage of 50 V is possible with the deceleration mode with still an excellent spatial resolution of 2.8 nm as calculated with SMART-J (Figure 1A). The energy-filtration allows the observation of small compositional contrast as shown in Figure 1B where Al3Li precipitates (δ’) were observed in an AA2099 Al-Li-Cu alloy. A resolution of 2.2 nm was obtained for a combination of SE and BSE signals with a mix of topographic and compositional contrasts (Figure 2A). Simultaneously, an energy-filtered BSE signal was acquired with a resolution of 2.7 nm and a compositional contrast was observed (Figure 2B). Furthermore, Monte Carlo simulations were used to understand and to optimize the SEM parameters of these different imaging modes.

The HITACHI SU-8230 CFE-SEM provides low accelerating voltage, deceleration mode and energy-filtration of the electron signals and thus allows the characterization of the microstructure of materials with high spatial resolution at the nanoscale with various types of contrasts. The development of these new technologies permits to extend the imaging capabilities of the SEM towards new nanoscale applications.

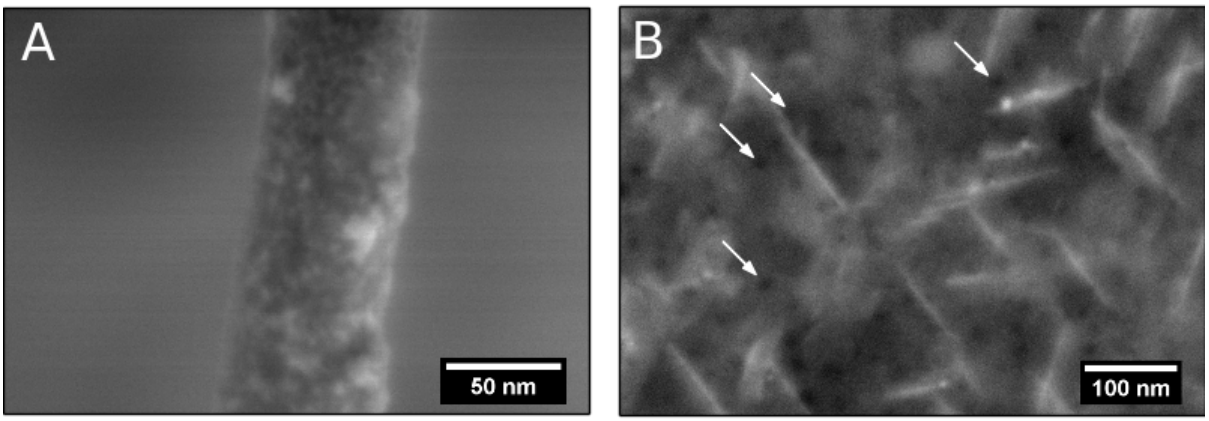

Fig. 1: High resolution micrograph obtained with a CFE-SEM. (A) SE micrograph of a CNT decorated with Pt nanoparticles was acquired at 50 V in deceleration mode with the top detector. (B) Energy-filtered BSE micrograph of an AA2099 Al-Li alloy acquired at low energy with the upper detector.

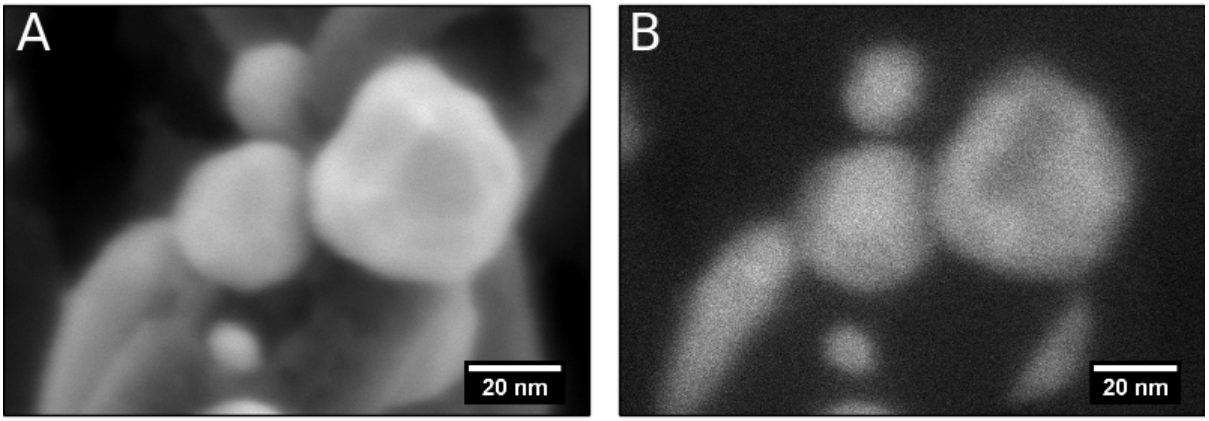

Fig. 2: High resolution micrograph obtained with a CFE-SEM. CNTs decorated with Pt nanoparticles micrographs were acquired at 1 kV with: (A) combination of secondary and backscattered electron (SE+BSE) signals by the upper detector; (B) energy-filtered BSE signal by the top detector.