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EELS and EDS Compositional Imaging of B_4C Nanostructures Synthesized by a Solid-Liquid-Solid Method

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Boron carbide (B_4C) is a material well known for its extreme hardness, chemical inertness, low density and excellent nuclear properties as a neutron absorber. This material has been extensively studied in its bulk form. However, nanostructures are a recent addition to the family of boron carbide structures. Recently, a lot of research has been carried out to synthesize one-dimensional nanostructures such as nanorods, nanowires and nanoribbons owing to their potential applications in high-temperature devices and doped semi conductors [1-3]. In this work, we report detailed electron microscopy characterization using scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM), energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectrometry (EELS) spectrum imaging of boron carbide nanostructures synthesized using a solid-liquid-solid method.

In a systemic growth study, the effect of temperature (1000, 1150 and 1250°C) and boron-to-carbon ratio (B:C = 0.25, 1, 4 and 8) were investigated. In addition, different amounts of silicon additive to produce silicon-doped nano structures were also studied. Various nanostructures including nanowires, nanorods, and nanoribbons with different aspect ratios were observed. Structural and chemical analyses were employed to help understand the growth mechanisms of these structures.

HRTEM imaging, selected-area electron diffraction (SAED) and nanoprobe diffraction analysis were performed using a Topcon 002B microscope operating at 200kV. EELS and EDS spectrum imaging were conducted using a Philips CM200-FEG TEM equipped with a Gatan Imaging Filter and operating in the scanning transmission electron microscopy (STEM) mode at 200kV. All spectra were acquired Emispec acquisition system and processed with ES Vision software. EELS data were also exported for further analysis with Gatan EL/P software. Data were acquired with a STEM probe of ~1.2 nm full-width at half-maximum (FWHM) and 1 nA probe current. All EELS and EDS spectrum images were automatically drift corrected and acquired for areas of 60 by 120 nm with 1 s dwell/pixel.

Figure 1 shows typical bright-field and HRTEM images of a B_4C nanowire doped with silicon and processed at 1150°C. Both Fourier Transforms of HRTEM images and nano-probe diffraction analysis show that the structure obtained is B_4C. Nanostructures of diameter larger than 100 nm show some stacking fault defects and multiple growth events. EELS and EDS spectrum images were also collected from nanostructures processed with these same conditions. Figure 2 shows typical EDS and EELS spectra from the center of a nano wire (the position is shown on the inset image). Figure 3 shows compositional maps of Ni, Co, Si, B and C extracted from the spectrum images. The maps show that the structure consists mainly of B and C with the ~ 5% Si content concentrated at the surfaces. Work is underway to understand the exact growth mechanisms and to optimize processing conditions.

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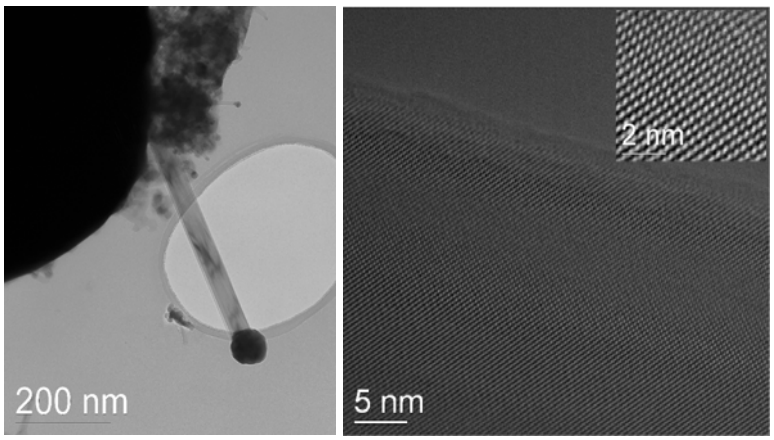
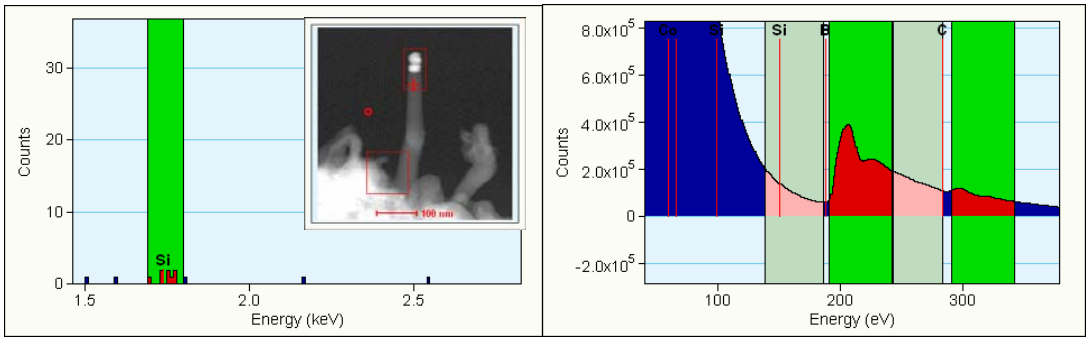


(a) (b)

Fig. 1. (a) Bright field image showing catalysis particles at the tip and (b) HRTEM image of B_4C nano wire



(a) (b)

Fig. 2. (a) EDS and (b) EELS spectrum images simultaneously collected from B_4C nano wire shown in inset.

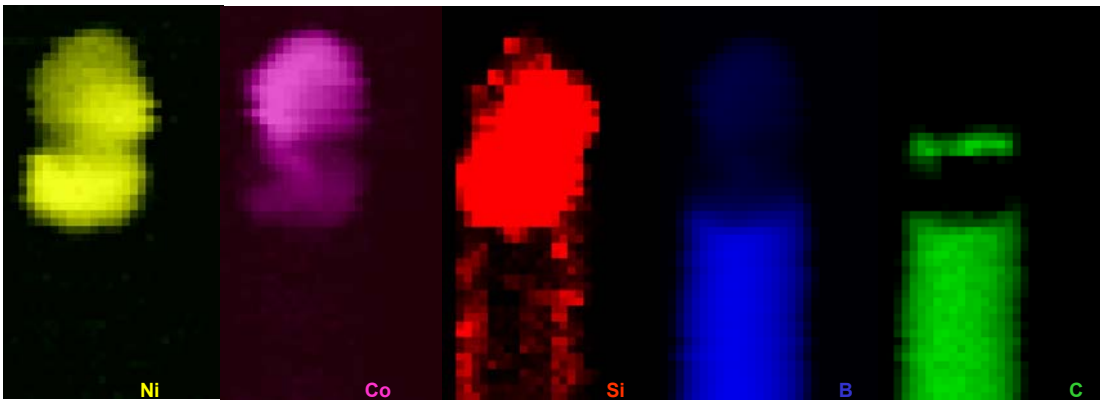


Fig. 3. Compositional maps of Si doped B_4C nano wire showing all the detected elements (Ni, Co, Si, B and C)

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