

## TEM Studies of Fe<sub>1-x</sub>Ni<sub>x</sub> Nanowires by Magnetic-Field-Induced Synthesis

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The nanoscale iron-based structures in form of nanowires (NWs) have been considered as one of the most promising multifunctional materials in applications in many fields, including: data storage devices, biomedicine, catalysis, energy storage and conversion devices [1, 2]. They have received a great attention because they are inexpensive, environmentally friendly and exhibit unique physical and chemical properties which can be precisely controlled by changing their aspect ratios [3]. It is also important that the iron-based materials tend to be self-oxidized in the presence of oxygen. This leads to the formation of complex core-shell nanostructures [4]. Hence, the purpose of this work is to investigate determine the morphological and structural properties of iron-based nanowires with use of TEM.

Three iron-iron oxide core-shell nanowires (Fe-Fe<sub>x</sub>O<sub>y</sub> NWs) and iron-nickel NWs with Fe<sub>0.75</sub>Ni<sub>0.25</sub> and Fe<sub>0.5</sub>Ni<sub>0.5</sub> compositions were selected for investigation. These samples were fabricated by applying a magnetic-field-induced synthesis. Details of synthesing these nanowires were previously published [4, 5]. Powder samples were suspended in absolute anhydrous ethanol, and sonicated for 2 min. A small drop of suspension was pipetted onto a holey carbon grid, and dried in air with a cover glass. Specimens were then examined in a JEOL 2100F (S)TEM equipped with equipped with GIF/EELS (Tridium 863).

Low magnification TEM images show that the Fe-Ni and Fe-Fe<sub>x</sub>O<sub>y</sub> NWs are composed of nanoparticles (NPs) which are linked and aligned in long and almost linear wires with the diameter of 30 – 90 nm regardless of particle size (Figs. 1, 2 and 3). ED pattern (inset in Fig. 1a) and HRTEM image (Fig. 1b) reveal Fe NWs are crystalline. Moreover, those Fe NPs are separated by 3~5 nm amorphous layer (inset in Fig. 1b) and confirmed as Fe oxide by EELS analysis (Fig. 1c), and thus the nanowires may consider as Fe-Fe<sub>x</sub>O<sub>y</sub> core-shell structure. The morphology of bimetallic Ni-Fe NWs (Fe<sub>0.75</sub>Ni<sub>0.25</sub> and Fe<sub>0.5</sub>Ni<sub>0.5</sub>) reveal similar structure, i.e., linearly aligned NWs assembly that consisted of NPs (Figs. 2 and 3) and also covered by 3~5 nm-thick amorphous layer which most likely due to oxidation of Ni-Fe (Fig. 2c and 3c) when exposed in air. In addition, the diameter of NWs decreases from 30~90 nm (Fe-Fe<sub>x</sub>O<sub>y</sub> core-shell NWs) to 30~80 nm (Fe<sub>0.75</sub>Ni<sub>0.25</sub>) and 30~60 nm (Fe<sub>0.5</sub>Ni<sub>0.5</sub>). This also implies that not only the diameter will decrease but also the crystallinity will degrade when increasing the Ni stoichiometry in bimetallic Ni-Fe NWs. To understand the difference of morphology/microstructure of Fe-Ni and Fe-Fe oxide nanowires during their growth, the intrinsic structure and property of Fe and Ni should also be considered. Electron diffraction revealed that the Fe<sub>0.75</sub>Ni<sub>0.25</sub> and Fe<sub>0.5</sub>Ni<sub>0.5</sub> NWs are exist as amorphous-like (or very short range ordered materials) while the Fe-Fe<sub>x</sub>O<sub>y</sub> core-shell NWs are typical nanocrystallites. Although all samples revealed the specific core-shell-like structures, more research is needed especially the formation of amorphous coating layer [6].

### References:

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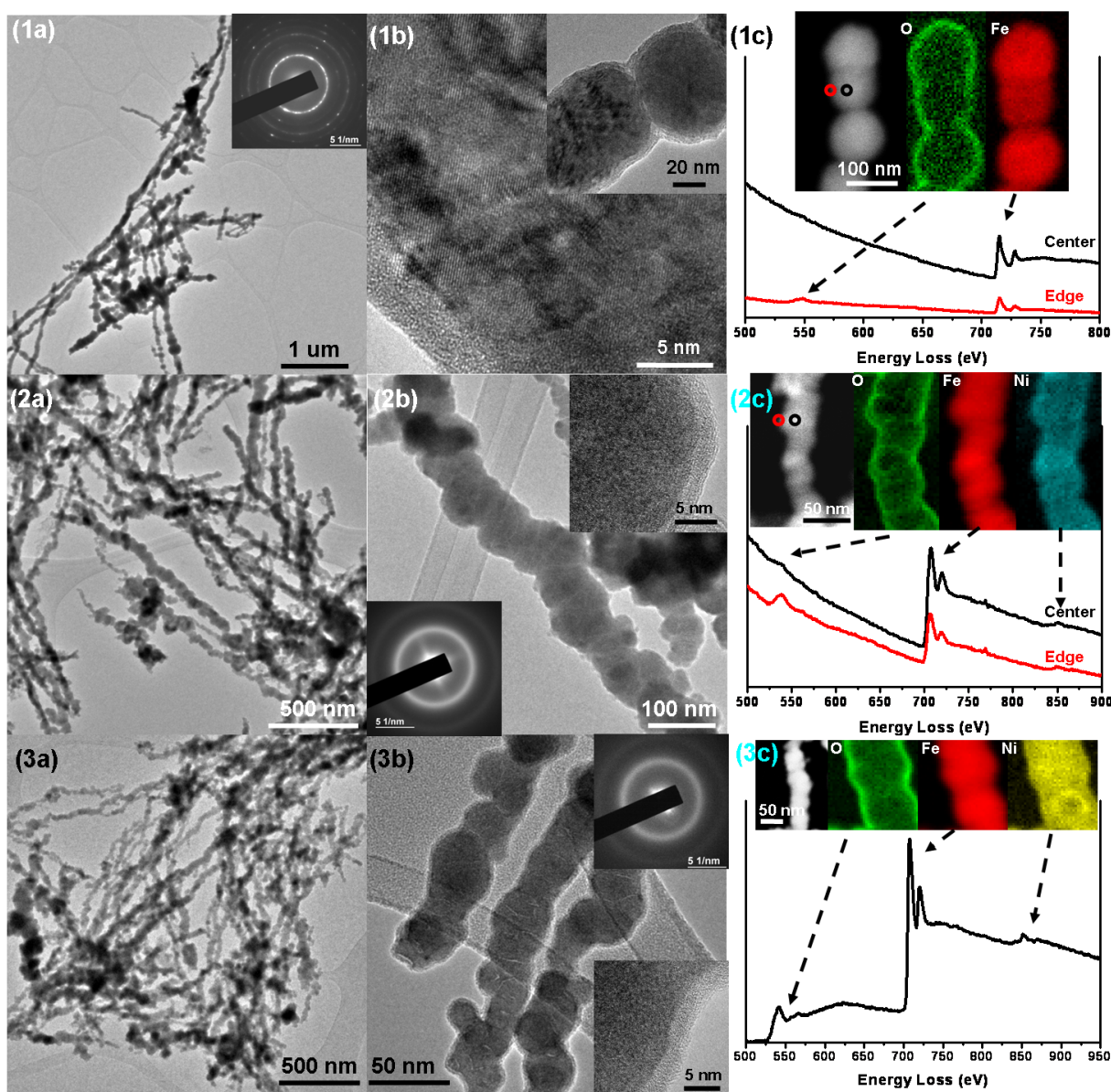
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**Figure 1.** TEM image (a) and HRTEM image (b) of Fe-Fe<sub>x</sub>O<sub>y</sub> NWs. Inset in (a) and (b) is the corresponding ED pattern and low mag. TEM image, respectively. (c) Position-dependent EELS spectra recorded from edge (red) and central (black) positions of NWs. HAADF images and elemental mapping of O and Fe, are shown in upper part of panel.

**Figure 2.** (a)-(b) TEM images of Fe<sub>0.75</sub>Ni<sub>0.25</sub> NWs. Inset in (b) are the corresponding ED pattern and HRTEM image, respectively. (c) EELS analysis (spectra and elemental mapping).

**Figure 3.** (a)-(b) TEM images of Fe<sub>0.5</sub>Ni<sub>0.5</sub> NWs. Inset in (b) are the corresponding ED pattern (upper) and HRTEM image (bottom), respectively. (c) EELS analysis (spectra and elemental mapping).