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An ELNES study of SiO_2 nanowires grown from a patterned reagent

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Progress has been achieved recently in the synthesis and device application of SiO_2 nanowires, however isolating and manipulating freestanding nanowires is still challenging, and it is of great interest to grow them in a controlled fashion, so that no post-growth manipulation is needed for further measurement or device use [1]. We have developed a novel process to synthesize SiO_2 nanowires from a patterned reagent into arrays with tunable size and pitch, using electron-beam lithographic patterning of Fe-SiO_2 composite films [2]. First, stable iron nanoparticles embedded in SiO_2 matrix are grown onto Si_3N_4 membranes in an ultrahigh vacuum system [3]. Such composite samples are then patterned and etched, using hydrogen silsesquioxane (HSQ) negative-tone resist, giving circular HSQ (exposed) nanodots, 50 nm in diameter and a 1μm pitch, covering the Fe-SiO_2 composite film. Taking advantage of the catalytic properties of the Fe particles, nanowires are grown from the patterned nanodots by annealing in a furnace reactor (in a N_2/H_2 atmosphere) as shown in Fig. 1. The number and diameters of the nanowires on each dot depend on those of the iron nanoparticles, which is controllable through the fabrication and patterning process [2].

Figure 2 shows a transmission electron microscope (TEM) image of a bundle of nanowires projecting from the substrate. The nanowires have a smooth surface and a closed end, in which a single iron particle is entrapped, indicating a gas-liquid-solid growth mode. A limited number of nanowires (Fig. 2, inset) were selected for analysis by electron energy-loss near-edge structure (ELNES) and nano-diffraction patterns (DP). The results are presented in Figs. 3 and 4; Auger spectra and images were also recorded for chemical analysis of the nanowires.

Electron energy-loss and Auger analysis showed that the nanowires contain silicon and oxygen; Spectra analysis carried out on individual nanowires was not affected by the presence of the substrate, as shown by the absence of the N-K edge in Fig. 3. The Si-L_{23} edges of SiO_2 and Si differ in their fine structure and threshold energy [4], allowing us to exclude the presence of elemental Si in the nanowires. We have performed multiple-scattering calculations of the Si-L_{23} edge (shown in Fig. 3) and O-K edge (not shown here) of SiO_2. These simulations give us confidence that the nanowires consist of SiO_2.

Pure iron is useful as high-activity catalyst but is susceptible to oxidation, which we sought to minimize by embedding the iron nanoparticles in a SiO_2 matrix. Absence of a postpeak at about 40 eV above the Fe-L_{23} confirms that oxidation of iron is low [5]. However, our measured white-line ratio: $L_3/L_2 = 2.9 \pm 0.2$, extracted by fitting the Fe-L_{23} edges (shown in Fig. 4), indicates some chemical reaction [5], perhaps the formation of iron silicides at the surface, as expected by the vapor-liquid-solid model. Judged from the intensity profile of the DP (showing as a photographic negative in the inset of Fig. 4), bcc-Fe is the main phase; there is evidence of iron silicides but not iron oxides. No SiO_2 peaks were found, indicating the amorphous nature of the nanowires.

The exposure product of HSQ is Si_xO[6], which evidently provides the reagent source for the growth, since no nanowires appeared in areas covered with unexposed HSQ or the SiO_2 of the

matrix itself. The embedded iron nanoparticles act as a catalyst, after diffusing to the surface. We are developing a model for the growth process of the nanowrires, on the basis of our microstructural and chemical analysis [2].

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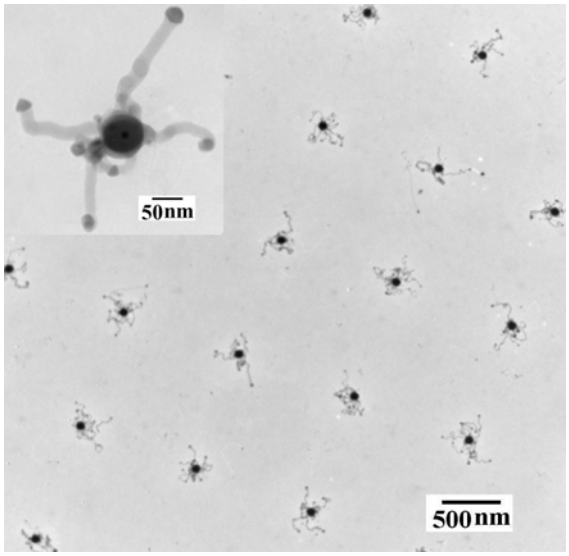

Fig.1: SiO_2 nanowires on the Fe-nanodot array.

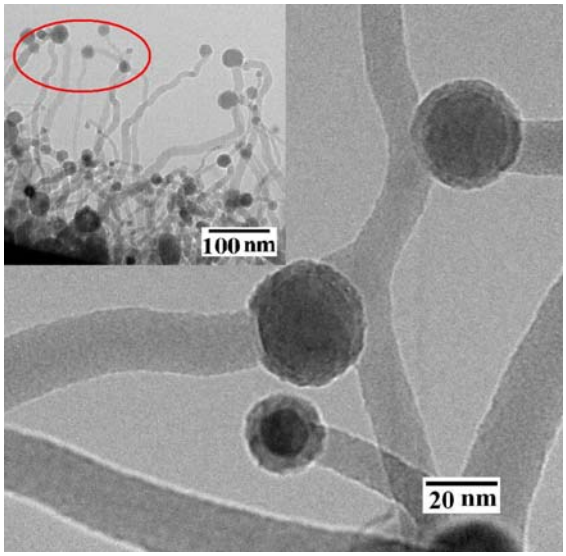

Fig. 2: SiO_2 nanowires and selected area for EELS analysis (inset).

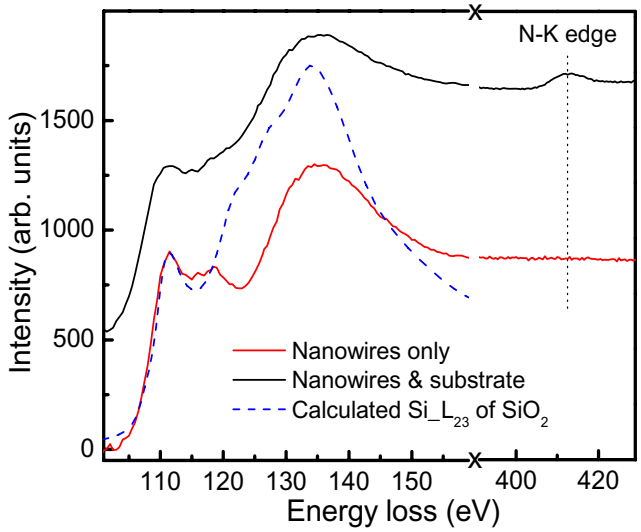

Fig.3: Experimental Si-L23 edges, compared with a Si-L_{23} edge calculated for SiO_2.

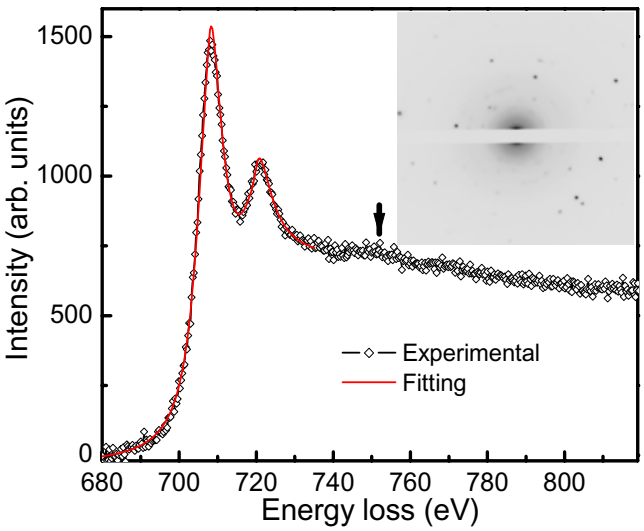

Fig. 4: Fe-L_{23} edges with a computer fit and DP (inset) of selected individual nanowires.

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