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Analysis of $MnFe_2O_4$ phase transition induced by the energy of electron beam in an iron-manganese oxide nanoparticle

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The iron-manganese oxide (FeMn-ox) is a material that has carried great interest in recent years because of the promising application in environmental remediation, hydrogen production, and catalytic degradation of organic pollutants [1-3]. Since the strong relationship of structure and properties was found in the crystalline materials, the analysis of in-situ changes of the structure has carried attention due to constitute a useful tool for developing formation mechanisms [4, 5]. In addition, it is known that the energy content in the electron beam of the transmission electron microscope (TEM) can promote repositioning of the atomic species and structural changes in metal oxide nanoparticles [6]. Therefore, the beam energy can be used to induce a phase transformation of FeMn-ox nanoparticles. In this work, iron-manganese oxide (FeMn-ox) nanoparticles were synthesized by the hydrothermal method. Four aqueous solutions were required: sodium dodecyl sulfate (0.15 M), sodium hydroxide (2.5 M), iron (III) chloride (0.3 M), and manganese chloride (0.3 M). The solutions were mixed, and a total volume of 25 mL was introduced into an autoclave for heating the entire system at 80 ^{0}C, under an isothermal process, for 1h. Afterward, the sample was cleaned in an ultrasonic bath of deionized water. The characterizations were focused to study the phase transition in a single FeMn-ox nanoparticle by transmission electron microscopy in a JEOL ARM200F. Thus, the phase transition was induced by a conventional acceleration voltage of 200 keV.

A summary of the results is given in Figures 1 and 2. Figure 1 shows a comparison between a FeMn-ox nanoparticle imaged by TEM after 0 s and 9 s. These results suggest that the energy of the electron beam promotes structural changes in the nanoparticle and lead to a nanostructure formation on the border of nanoparticles (covered with a square in Figure 1d). This is corroborated by the high resolution-TEM (HRTEM) in Figure 1c and Figure 1f and the selected area electron diffraction (SAED). On the other hand, Figure 2 shows the HRTEM characterization of phase transition. The nanoparticle border has a specific atomic arrangement when time equals 0 s Then, after 3 s, the atomic structure starts to suffer modifications. A dramatic change is observed once 6 s have elapsed, atom migration outside the nanoparticle interface has driven to the formation of nanostructure placed at the surface. When the time reaches 9 s, a complete change of phase in the observed region is achieved and no further atomic migration was observed. The transition of phase to MnFe2O4 can be clearly identified by the ED calculated by fast Fourier transform (FFT). The ED pattern at time equals to 9 s is in good agreement with that reported for the MnFe2O4 phase [7]. The results suggest that phase transition can promote morphological modification in FeMn-ox nanoparticles by the effect of the energy in the electron beam. These observations open the door toward post-synthesis morphological modifications of metal-oxide nanoparticles assisted by electron microscopy.

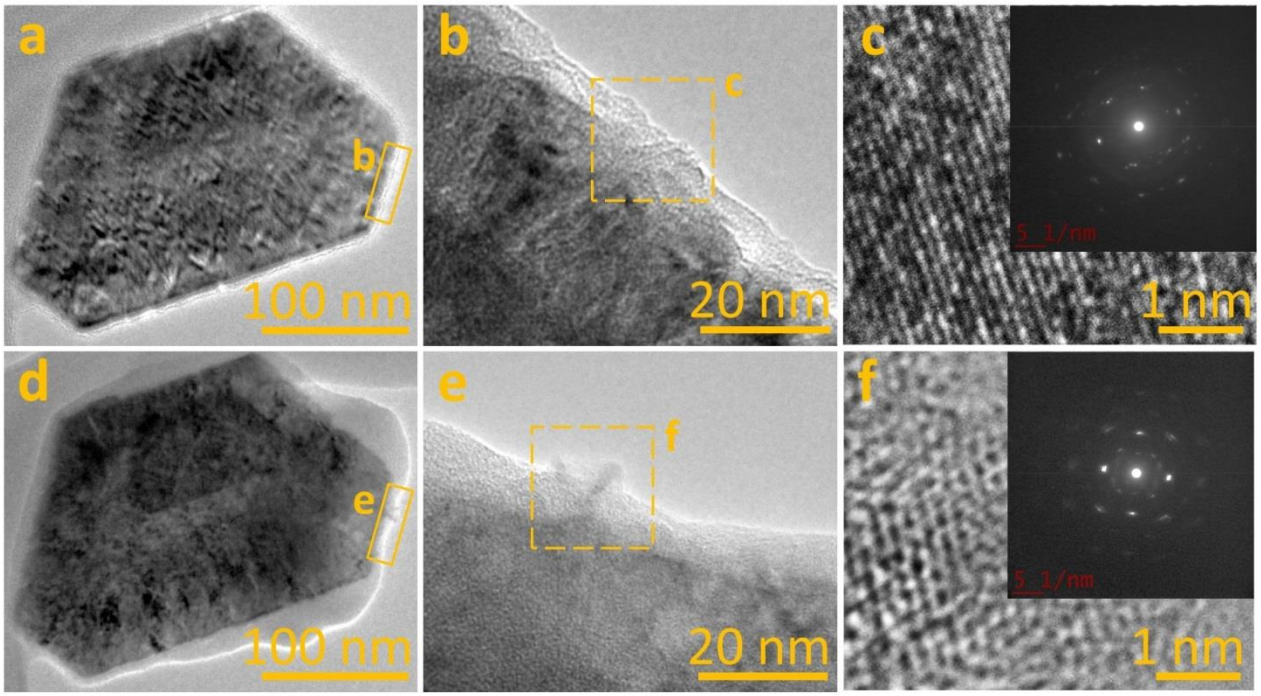

Figure 1. FeMn-ox nanoparticle imaged by TEM at (a) 0 s and (d) 9 s, (b), and (e) show a close-view at the edge of the nanoparticle. (c) and (f) show HRTEM images of the pointed section in (b) and (e), respectively (inserted shows the SAED).

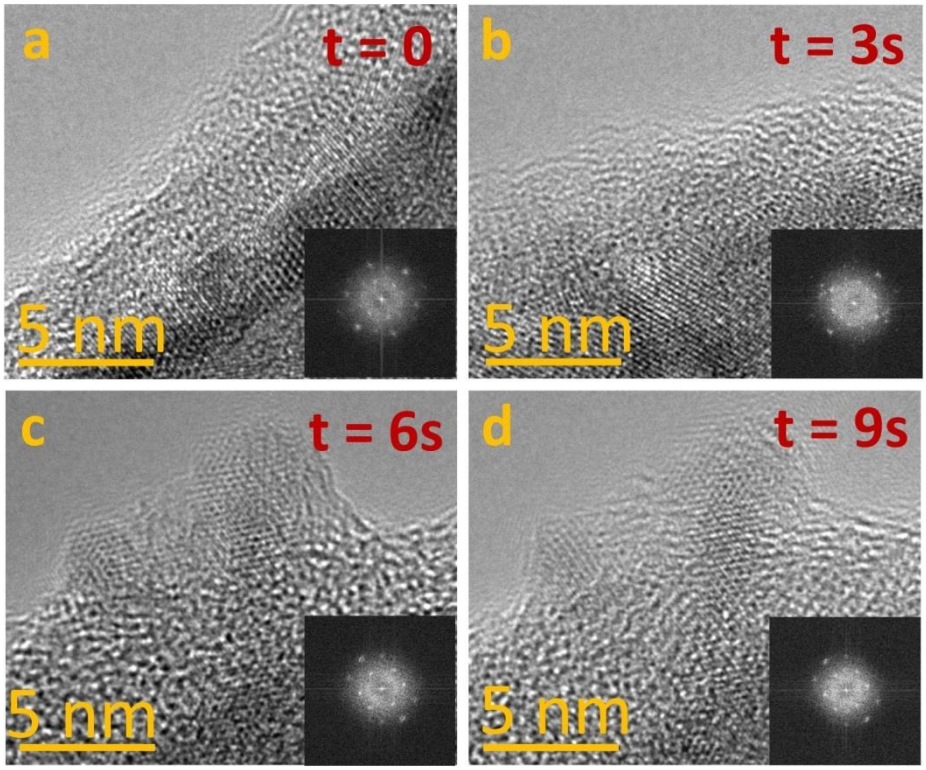

Figure 2. HRTEM image of the sample under electron beam for (a) 0 s, (b) 3 s, (c) 6 s and (d) 9 s. Inserts show the ED pattern calculated by FFT.

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