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Reduction of iron oxides prepared by sol-gel method for the production of microscale and nanoscale metallic iron particles

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Abstract – Microscale and nanoscale metallic iron particles were prepared through the hydrogen reduction of the sol–gel precursor powder product. The precursor powders were obtained after the low temperature treatment of an aqueous diluted PVA and metal nitrate solution. The various hydrogen reduction temperature products were characterized by X-Ray Diffractometry, Scanning Electron Microscopy, Vibration Magnetometry and Mossbauer Spectroscopy to reveal the effects of hydrogen reduction on microstructure and magnetic properties. With the increase of the hydrogen treatment, the iron oxides particles reduce to iron particles in micro and nanoscales.

The hydrogen reduction of oxides has been proved to be an important method for the production of metallic nanoparticles when applied to Fe_2WO_6 [1] , MoO_3 [2] and $CuFe_2O_4$ [3]. So, in the present work, the hydrogen reduction was used to syntheze micro and nano particles of metallic iron from iron oxides prepared using a relatively simple, inexpensive and rapid sol–gel method.

Microscale and nanoscale metallic iron particles were prepared through the hydrogen reduction of the sol–gel precursor powder (figures 1 and 2). The precursor powder was obtained after the low temperature treatment at 250^{o}C of an aqueous diluted PVA and iron nitrate solution [4]. The various hydrogen reduction temperature products were characterized by X-Ray Diffractometry, Scanning Electron Microscopy, Vibration Magnetometry and Mossbauer Spectroscopy to reveal the effects of hydrogen reduction on microstructure and magnetic properties. The precursor powder was consisted of iron oxides, mainly hematite and magnetite as shown by Mossbauer spectroscopy (figure 3) and confirmed by X-Ray Diffraction. The reduction processes are followed by Mossbauer spectroscopy and X-ray diffraction. With the increase of the hydrogen treatment, the iron oxides particles reduce to α-iron particles (figure 3). X-ray diffraction patterns from the sample treated at 500^{o}C indicate only an α-iron phase. At the same temperature, the Mossbauer spectroscopy shows an almost free sample of oxides. The Scanning Electron Microscopy is used to estimate the particle size. In the sample treated with hydrogen at 500^{o}C, the obtained particle sizes are about 200nm and about 1000nm (figures 1 and 2). A very high enhancement of the magnetic moment was observed with the temperature increase of the hydrogen treatment.

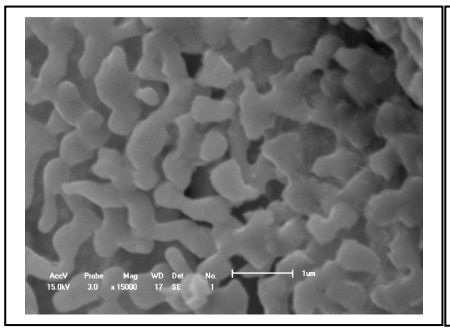

Figure 1: micro metallic iron particles

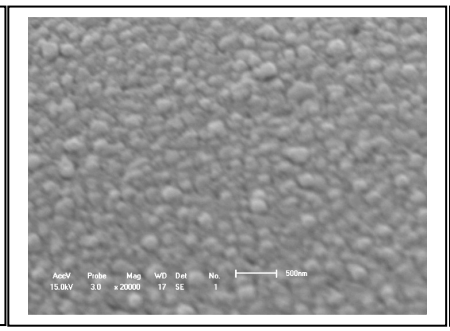

Figure 2: nano metallic iron particles

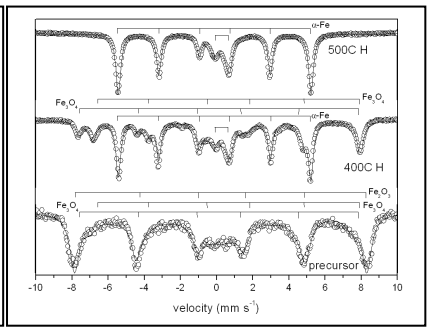

Figure 3: Mossbauer Spectroscopy of precursor, and the samples treated at 400 and 500 ^{o}C.

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