Transmission electron microscopy and characterization of $NiFe_2O_4$ nanoparticles dispersed in SiO_2 matrix

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Keywords: Nickel ferrite, TEM, X-ray diffraction and SQUID magnetometry

Transmission electron microscopy is a versatile technology by which nowadays it is possible to analyze the shape and size of very small particles in nanometer range. Nickel ferrite is one of the soft ferrites ordered in the inverse spinel structure. Its physical properties get dramatically changed when prepared at nanoscale. This is because of large surface to volume ratio and disordered surface spins due to which magnetic surface anisotropy becomes dominant in determining magnetic properties [1].

$NiFe_2O_4$ nanoparticles dispersed in SiO_2 matrix (25 wt%) were synthesized by sol-gel method with tetraethyl orthosilicate (TEOS) as a precursor for SiO_2 [2]. Chemical phase identification was done by X-ray diffraction analysis. Average particle size was evaluated by Debye-Scherrer's formula and compared with size-distribution statistics taken from an image analysis of transmission electron micrographs (TEM). For TEM microscopy, the nanopowder was mixed with hexane and dropped on a carbon grid. The hexane will be evaporated and nanoparticles will stay on the carbon grid. Micrographs were taken at various scales to study the distribution of particle size. The particle shape is nearly spherical and no facets are observed. The average particle size from a log normal distribution fit comes out to be 25nm in good agreement with X-ray diffraction evaluation (23nm from Debye-Scherrer's formula). The formation of ferrite nanoparticles embedded in SiO_2 matrix was also studied by Fourier transform infrared spectroscopy (FTIR) in the far and mid infrared range. Nickel ferrite and SiO_2 matrix show characteristic bands in the far and mid infrared range. Magnetic characterization was done by using superconducting quantum interference device (SQUID-magnetometry). The nickel ferrite nanoparticles show a soft magnetic behavior with saturation magnetization Ms = 23.9 emu/g and coercivity Hc = 161 Oe at temperature T = 4.2K, after post annealing at 700°C to get nearly single phase spinel structure (with small amount of hematite 7%).

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3. This work is granted by Austrian national fund network projects (NFN) S10407-N16 and S10405-N16. K. Nadeem thanks to Higher Education Commission (HEC) of Pakistan for providing PhD scholarship.

DOI: 10.3217/978-3-85125-062-6-424 ,

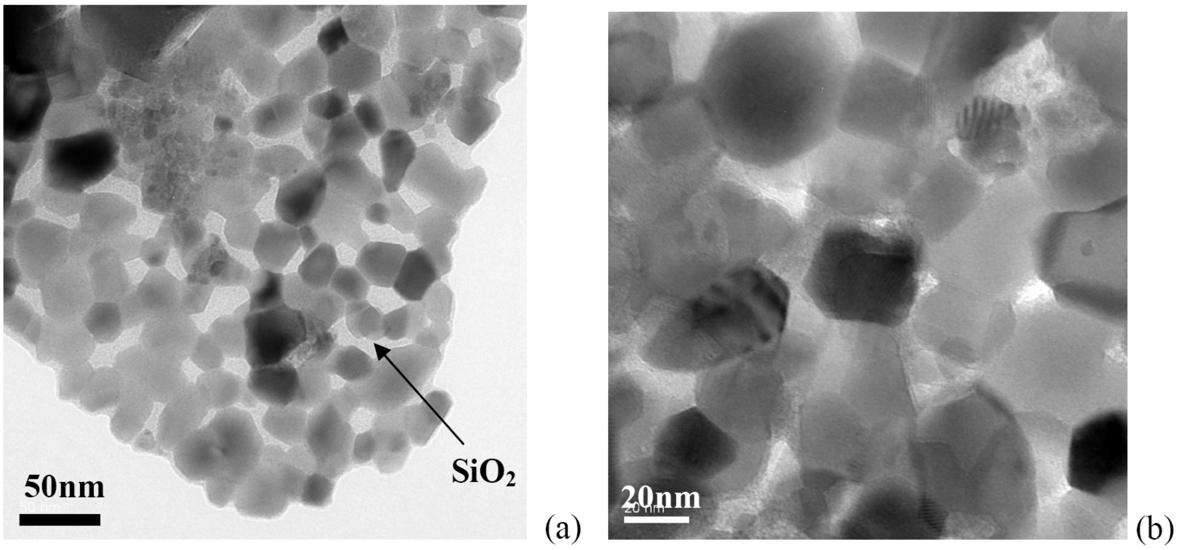

Figure 1. (a) TEM graph at 50nm scale and (b) TEM graph at 20nm scale of particulate

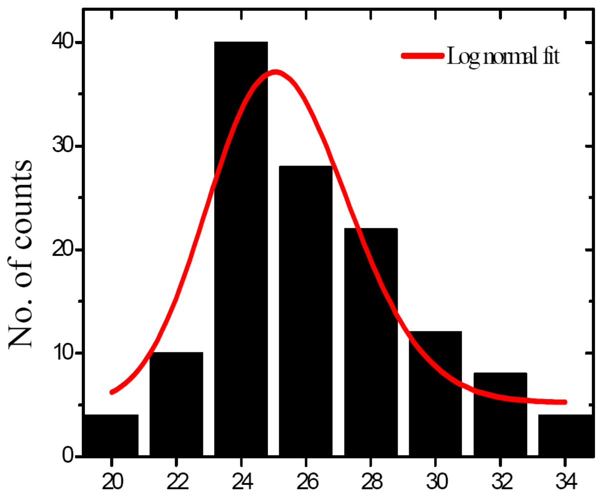

Figure 2. Particle size distribution fitted with log-normal distribution function.

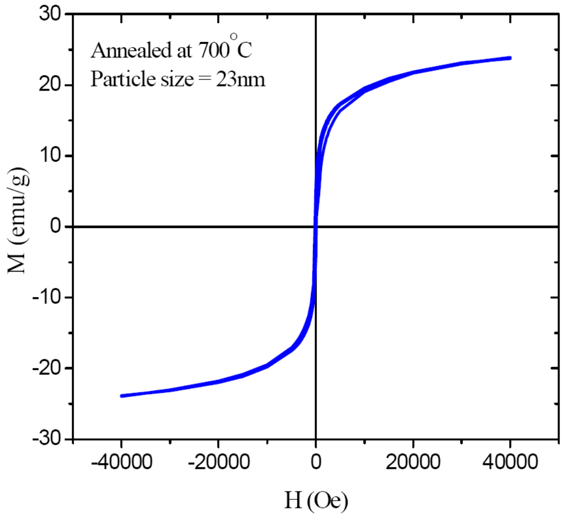

Figure 3. Hysteresis loop taken by using SQUID at T = 4.2K.

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