HIGHLY CRYSTALLIZED MN-ZN FERRITE NANOPARTICLES AS STARTING MATERIAL FOR PREPARING HIGH-DENSITY SOFT MAGNET CERAMICS WITH VERY FINE-GRAINED MICROSTRUCTURE

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Nanoscale magnets hold great promise in a wide range of electronic technologies. They are needed for improving the performance of microwave filters, power supplies, etc. Although nanoscale magnets have been synthesized for many years, the magnetic particles have generally been produced with a broad size distribution and poor crystallinity, whereas most applications require particles with a narrow size distribution (monodisperse particles) and a very low structural disorder in order to have magnetic characteristics, such as saturation magnetization, as close as possible to those of bulk samples.

We have succeeded in preparing nanoscale manganese zinc ferrite (MZFO) monodisperse particles with good crystallinity by using forced hydrolysis in a polyol. This is a novel preparative route to powdery oxide materials developed in our laboratory, as an alternative sol-gel *chimie douce* method. It consists in the hydrolysis of metallic salts dissolved and heated in a polyol. Polyols are crystal growth media of particular interest for the synthesis of fine particles with a narrow size distribution and high crystallinity [1-3]. Such features, in the case of ferrimagnetic oxide nanocrystals, are related to improved magnetic characteristics [4;5]. The particles prepared are polycrystalline since the average crystallite sizes determined by XRD analysis (from 14 to 18 nm as the Zn content increases) are significantly smaller than the average particle diameters determined by TEM analysis (from 78 to 29 nm as the Zn content increases) (Fig. 1).

The microstructural properties of these particles are characterized by high resolution transmission electron micrographs (HRTEM) which confirm their polycrystalline character and show that they are highly crystallized (Fig. 1). There is no evidence of defects such as dislocation and stacking faults. The nanocrystals are almost all oriented in the same direction. The fast Fourier transformation (FFT) diagrams show a Laüe-like pattern demonstrating that each polycrystalline particle is textured and can potentially be used as starting material for preparing non-porous soft magnet ceramics with very fine-grained microstructure.

The particles are superparamagnetic at room temperature with a blocking temperature ($T_B \approx$ 250 K) which depends on the crystallite size. The saturation magnetization at room temperature is slightly lower than that of bulk MZFO material. The lower value of saturation magnetization of nanocrystals compared to the bulk samples is usually attributed to surface effects that are due to finite-size scaling of nanocrystallites, which in turn leads to a non-collinearity of magnetic moments on their surface [6]. In the present case, the high crystallinity and the mosaic-like texture of the MZFO particles are not consistent with a high spin disorder at the grain boundaries. Only broken exchange bonds at the external layer of the polycrystalline particles (the surface spin having nearest neighbours on one side, none on the other side) may result in a decrease in the saturation magnetization. This high crystallinity is responsible for the enhanced saturation magnetization of the MZFO nanocrystals prepared here, as compared to that of similar materials prepared by other routes (hydrothermal [7], co-precipitation [8], reverse micelle [9;10], thermal decomposition [11], …).

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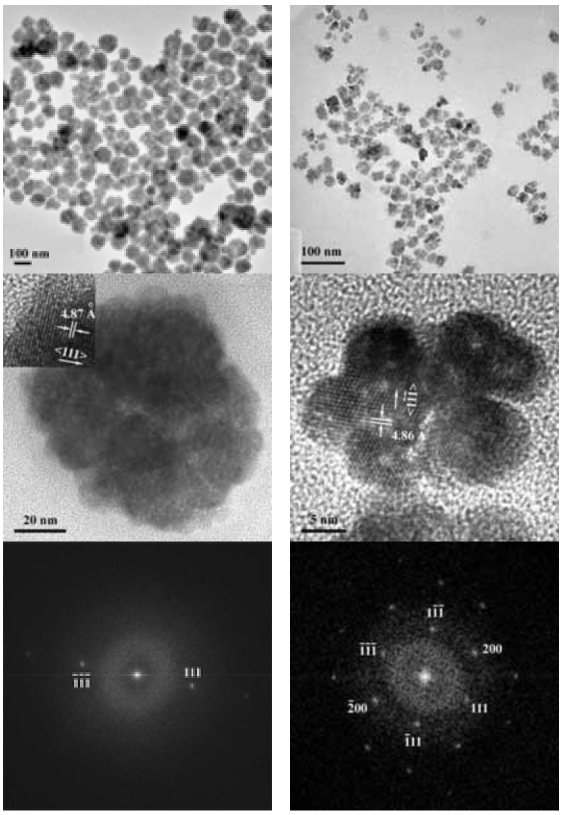
Figures:



(a) (b)

Fig. 1. TEM and HRTEM images and FFT patterns of representative polyol-prepared MZFO particles: a) (Mn:Zn) = 20:80 and b) (Mn:Zn) = 80:20.