Experimental Report

15/09/2014

Proposal: **5-31-2259** **Council:** 10/2012

Title: Nuclear and magnetic study of Cobalt, Manganese and Copper Ferrite nanoparticles obtained by a new electrochemical method

This proposal is a new proposal

Researh Area: Materials

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Samples: MnFe2O4
NiFe2O4
CoFe2O4

Instrument	Req. Days	All. Days	From	To
D1B	2	2	02/07/2013	04/07/2013
D2B	1	1	15/07/2013	16/07/2013

Abstract:

In the last decade, nanoparticles have attracted great interests in the scientific community due to their optical, electrical and magnetic properties.
Cobalt ferrite has focused attention for its high crystalline anisotropy, high coercivity and moderate saturation magnetization, but most of its magnetic properties strongly depend on the size and shape of the nanoparticles, which are closely related to the preparation method. A new methodology based in the electrochemical synthesis has been developed in order to controlling particle size in the range 20-40 nm.
We propouse a neutron diffraction study of particles obtained by this new method in order to localize the cobalt and iron atom in the octahedral and tetrahedral sites; relate the nanoparticle size with the magnetic behaviour and study the role of cobalt through the subtitution of this atom by nickel and manganese.

We have studied spinel ferrites (MFe_2O_4; M= Fe, Co, Ni, Mn) prepared by a new methodology based in the electrochemical synthesis. This new methodology allows controlling particle size in a range that is difficult to achieve by other synthesic techniques (between 20-40 nm).
A bean time of 2 days in D1B and 1 day in D2B diffractometers were allocated in order to study the magnetic structure, order temperature and cation occupancy in the two possible sites, tetrahedral and octahedral, in the spinel.

In D1B, cryofurnace with ROC option and λ = 2.52 Å was used and data were taken between 10K and 535K. Magnetite and nickel and manganese ferrite were measured. The second day in D1B, the furnace was installed without ROC option and magnetite and cobalt, manganese and nickel ferrites were measured from room temperature up to 800K. Neutron diffraction patterns of all these samples were taken above the magnetic transition

In D2B, measures at low temperature, 100K, and high temperature, around 800 K, and λ = 1.594 Å were carried out in order to obtain the atomic and magnetic structure with high resolution.

Figure 1 shows the thermodiffractograms obtained in D1B of one sample. The thermal evolution of (111) peak allowed us to obtain the magnetic order temperature and the Rietveld analysis the evolution of both cation sites magnetic moment, Figure 2.
Figures 3 and 4 show the diffractograms at 100 and 800 K obtained in D2B of cobalt ferrite, which allowed us to draw the crystal and magnetic structure of the sample, Figure 5.

The joint analysis of both experiments allows us to observe a possible exchange of cations between tetrahedral and octahedral sites when sample is heating the first time, but due to the resolution, wavelength and angle range of D1B it is not possible to corroborate this exchange.

Also, the neutron diffraction data of manganese ferrite together with other techniques showed the obtained nanoparticles are magnetite with manganese oxide around them instead of real manganese ferrite.

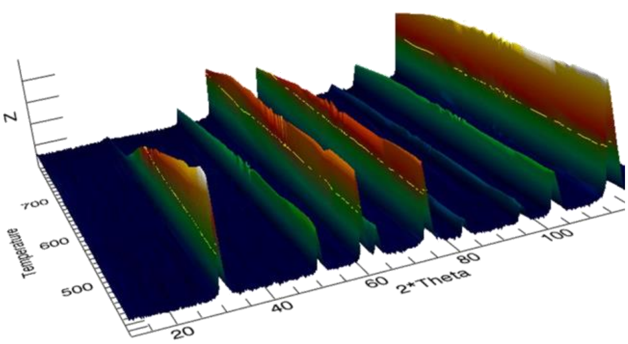

Fig 1. Thermodiffractograms of Cobalt ferrite.

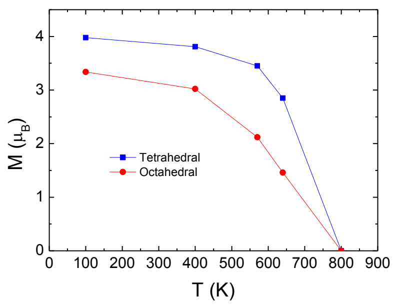

Fig 2. Magnetic moment evolution of both cation sites.

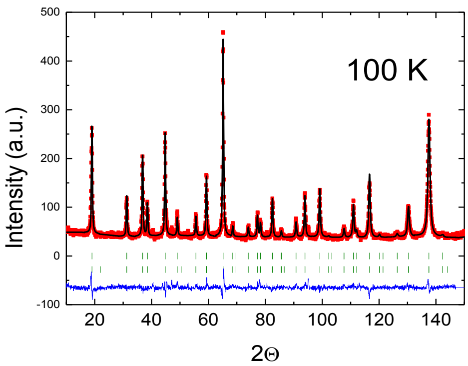

Figure 3. D2B diffractogram at 100K

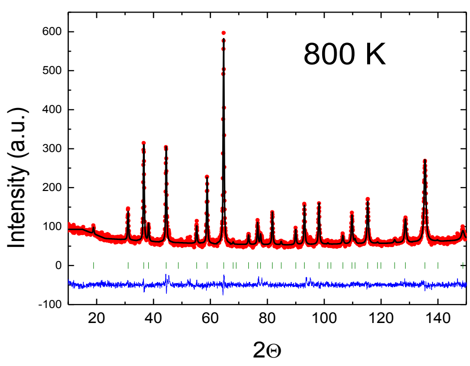

Figure 4. D2B diffractogram at 800K

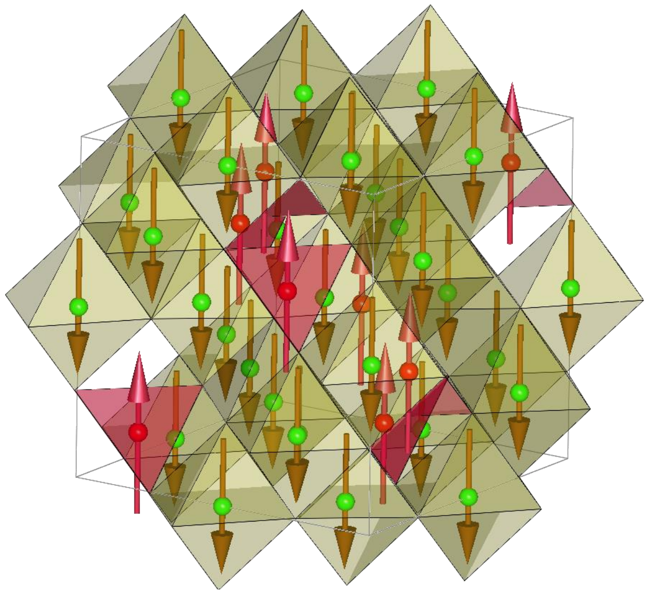

Figure 5. Crystal and magnetic structure of cobalt ferrite.