COMPETING SUPERPARAMAGNETISM AND EXCHANGE BIAS IN Fe NANOPARTICLES EMBEDDED IN AN AMORPHOUS CARBON MATRIX

M.P. Fernández García[a], *D.S. Schmool*[b], *A.S. Vieira*[b], *M. Sevilla*[c], *A.B. Fuertes*[c], *P. Gorria*[a], *J.A. Blanco*[a].

[a]*Dpto. de Física, Universidad de Oviedo, C/ Calvo Sotelo s/n,33007,Oviedo, SPAIN.*

[b]*IN-IFIMUP in Dpto. de Física, Universidade do Porto, Rua do Campo Alegre, 687, 4169-007, Porto, PORTUGAL.*

[c]*Instituto Nacional del Carbón, CSIC, Ap. 73, 33080, Oviedo, SPAIN.*

fernandezpaz.uo@uniovi.es

Research in magnetic nanoparticles (NPs) continues to have a high impact over the last decade not only for their fundamental scientific knowledge [1], but also for their unique properties and potencial technological applications such as ultrahigh-density recording and medicine. The synthesis of NPs is of key importance [2,3], because the properties of these nanocrystals are strongly dependent on their physical dimensions [4]. As a consequence we have used a commercial and low cost activated carbon (AC) (widely used before as adsorbents, as catalyst support and in Medicine because of their bio-compatible character) for embedding Fe NPs [5].

Another important issue for most applications relies on the magnetic order of NPs being stable with time. Sometimes, NPs loose this required stability (wanted for example in recording media) when the reduction of their sizes comes into conflict with the superparamagnetic (SPM) limit [6]. When this happens, magnetic exchange coupling [7] induced by the core and NP´s surface competes with SPM behaviour leading to magnetization stability. We have studied these and other interesting behaviours by combining structural characterization and physical properties of Fe NPs randomly dispersed in AC.

The sample in powder form, with average grain sizes of several microns, as SEM images reveals, contains around 17 wt.% of Fe. The average crystalline sizes for the Fe-AC NPs, obtained from TEM images (Figure 1), correspond to a broad distribution ranging from 5 to 50 nm with different crystal structures as the X-ray diffraction evidences.

The analysis of the room temperature Mössbauer spectrum of these NPs (Figure 2) was performed with three different contributions: a ferromagnetic α-Fe phase with B_{HF} = 33 T, a paramagnetic γ-Fe phase and an additional doublet, that could be associated with the NP´s shell or with an oxide phase probably located on the surface of the NPs. The relation between the percentage of α-Fe and both γ-Fe and the doublet contribution is $42/58 \approx 0.72(4)$. XRD pattern collected at room temperature confirms the existence of both BCC and FCC crystal structures giving a relative phase percentage of $\alpha - Fe/\gamma - Fe \approx 0.82(5)$ that agrees rather well with that of Mössbauer spectrum.

Magnetization vs. magnetic field curves, *M(H)*, are reversible above 200 K (Figure 3) as expected for a superparamagnetic-like behaviour. In addition $M_{ZFC}(T)$-$M_{FC}(T)$ curves do not overlap in the measuring range 10 - 340 K (Figure 3), suggesting that the whole system has not reached a SPM regime. Moreover, M_{ZFC}, exhibit a broad maximum that leads to a non unique well-defined blocking temperature owing to the broad distribution of NP´s size. Finally, a loop shift in *M(H)* curves indicating the existence of a magnetic exchange bias coupling (Figure 4), seems to suggest the presence of a core/shell morphology that makes Fe-AC NPs remain completely blocked below 60 K.

References:

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Figures:

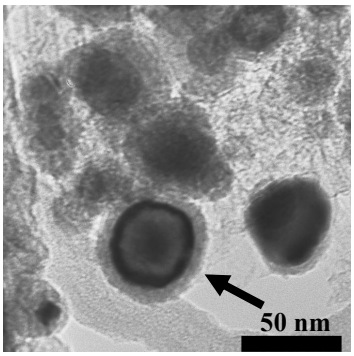

Figure 1. TEM image of Fe-AC NPs. The arrow points a core/shell Fe-AC NP.

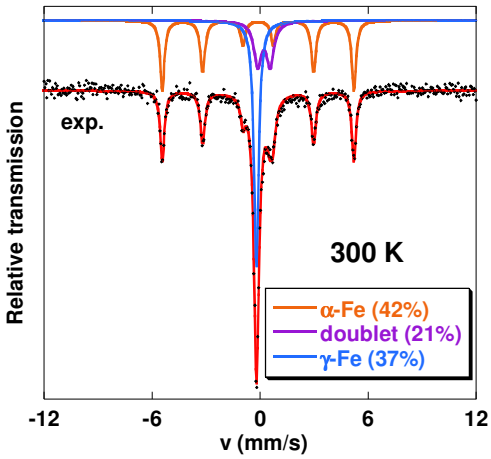

Figure 2. Room temperature Mössbauer spectrum of Fe-AC NPs together with the fit with three different contributions.

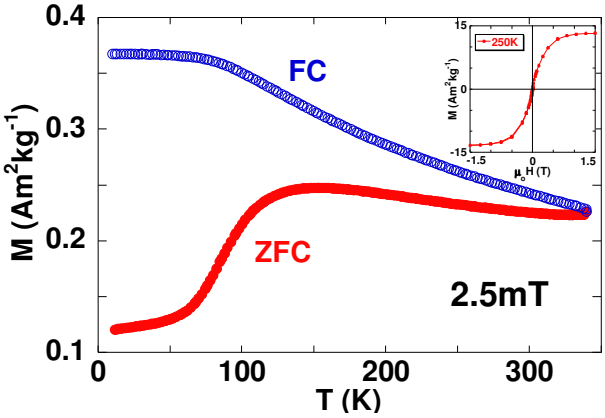

Figure 3. ZFC-FC magnetization variations under an applied field of 2.5 mT measured in the range 10-340K. The inset shows the SPM behaviour for *M(H)* curve at 250 K.

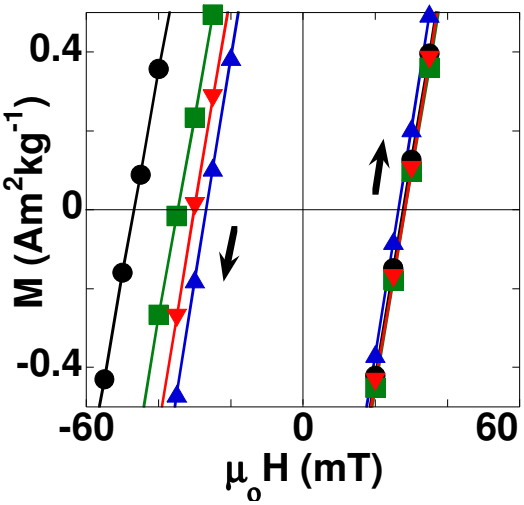

Figure 4. Details of the *M(H)* hysteresis loops around *M=0* under $\mu_o H_{cool}$=1.5T