

STRIKING NANO-ONION NANOPARTICLES WITH CORE (γ -Fe) / DOUBLE SHELL (α -Fe/ Fe-oxide): SYNTHESIS, MICROSTRUCTURE AND MAGNETISM

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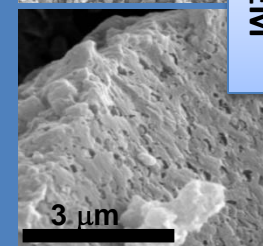
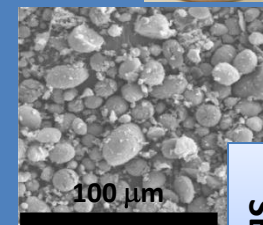
STATE OF THE ART

- Biomedical applications and magnetic separation
- Stabilization and protection of Fe-NPs with Fe-oxide layer
- Magnetic properties of Fe core/shell NPs at the nanoscale
- γ -Fe nanomagnetism:
 - In bulk form, only established above 1183 K
 - Stabilized on thin films, CNTs and Fe-NPs
 - Contradictory T_N results

a (Å)	Magnetic ordering
> 3.6	FM
< 3.6	AFM

CHEMICAL PREPARATION

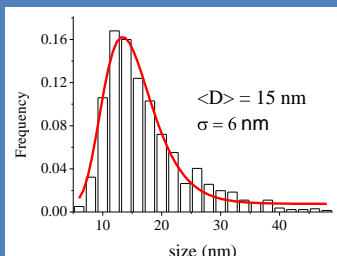
- Simple, inexpensive, massive (grames) technique
- Diameter of the porous
- Amorphous carbon filled with Fe nitrate solution
- Pyrolysis at the interconnections of porous carbon at 1173 K (α -Fe to γ -Fe bulk transition)
- 17 wt.% Fe
- Fe-NPs catalyze the phase conversion from amorphous carbon to graphite (2700 K \rightarrow 1100 K)



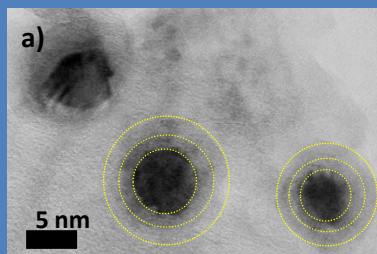
SEM

A.B.Fuertes et al. *Chem.Mater.* **18**, (2006)
M.Sevilla et al. *Carbon* **44**, 468 (2006)

MICROSTRUCTURE AND CRYSTAL STRUCTURE

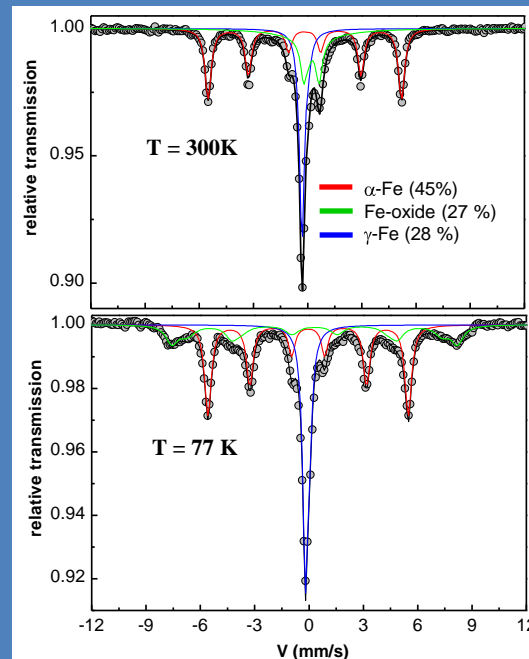
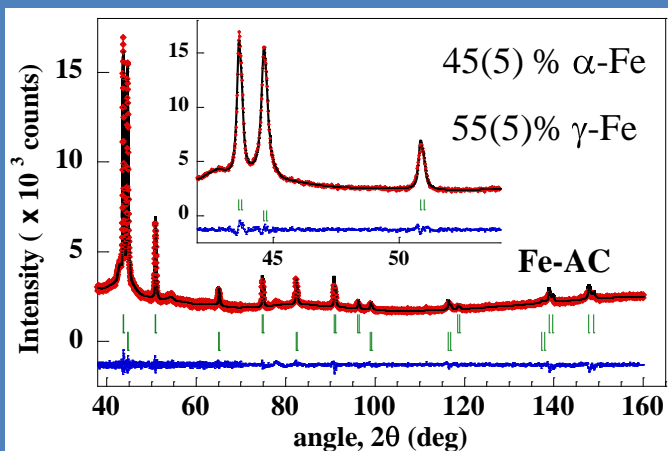


TEM

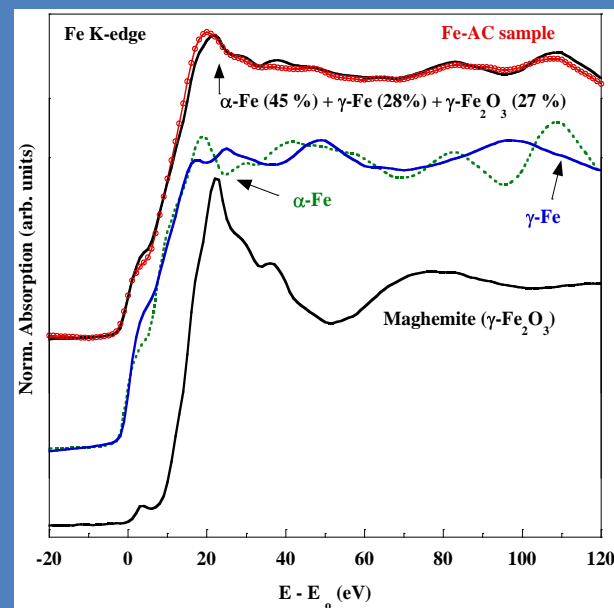


- Fe-NPs randomly dispersed
- Fe-NPs deposited at the interconnections of the porous carbon
- Thin Fe-oxide shell among the NPs under coherence length of XRD to be detected
- Unknown AC crystalline structure
- $a_{\text{BCC}} = 2.867 (1) \text{ \AA}$
- $a_{\text{FCC}} = 3.587 (1) \text{ \AA} < 3.6 \text{ \AA} !$

XRD @300 K



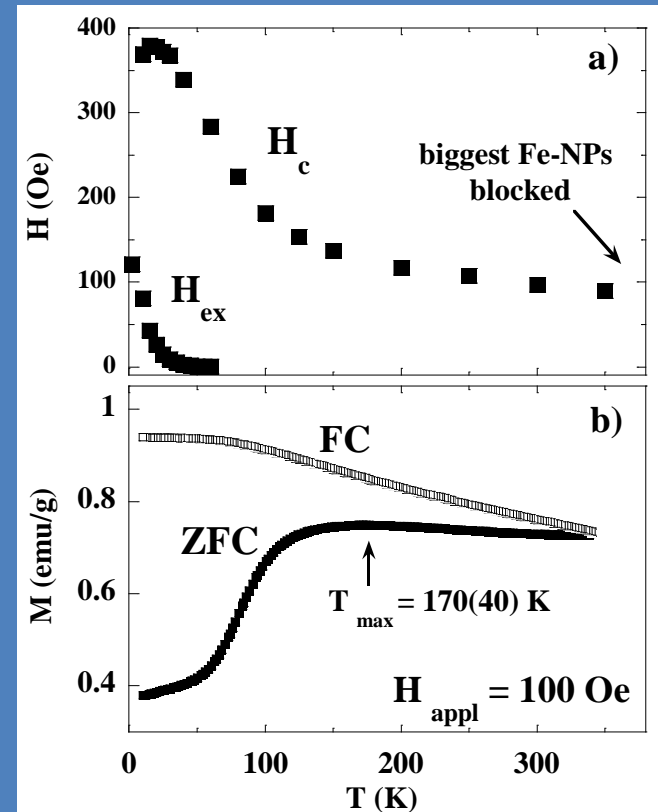
MÖSSBAUER SPECTROSCOPY



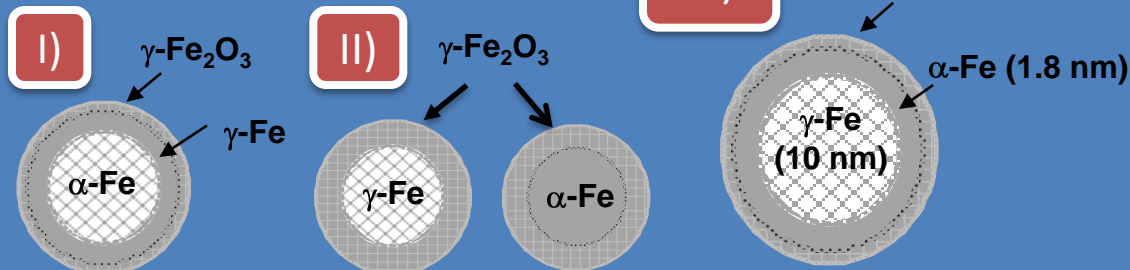
XANES

MAGNETISM

- Broad distribution of Fe-NPs sizes $\rightarrow T_B \approx 170(40)$ K
- Not complete SPM behaviour at 300 K
 - biggest NPs remain blocked
 - coercivity at room temperature
 - Irreversible behaviour between ZFC-FC curves
- Interface and surface progressively freeze into a low temperature spin-glass-like creating:
 - Exchange bias effect below 50 K
 - “Plateau-like” in low temperature ZFC-FC curves
 - $M(H)$ cycles not completely saturated at low temperature
- EB effect created by magnetic coupling between :
 - FIM Fe-oxide / FM α -Fe / possible AFM γ -Fe



NANO-ONION MORPHOLOGY



M. P. Fernández-García et al. *Phys. Rev. B* 81, 094418 (2010)

- 3 possible microstructures
- III) is the most probable due to magnetic properties (α -Fe cores not SPM at 300 K)
- Medium relative dimensions of nano-onion NPs estimated within this structural and magnetic combined study.