# Mössbauer Spectroscopy
# &
# Physics of Materials Laboratory

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Mössbauer Spectroscopy
Data Acquisition
Instrumentation

Mössbauer Spectroscopy
Methodology & Software

Mössbauer Spectroscopy
&
Physics of Materials Laboratory

Materials Samples
Synthesis
Solid State & Wet
Chemistry

Structural, Electronic & Magnetic
Properties of Materials
Characterization & Study
Focus: Magnetic Materials

Mössbauer Spectroscopy $^{57}Fe$ & $^{119}Sn$
Sample temperature 10-650 K (transmission geometry)

77-300 K

77-300 K

powder, films, single crystal & frozen liquid samples

## Mössbauer Spectroscopy $^{57}Fe$ & $^{119}Sn$
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10-300 K

300-650 K

powder, films & single crystal samples

# Mössbauer Spectroscopy $^{57}$Fe & $^{119}$Sn

## Special conditions/samples: applied magnetic field/backscatter Conversion Electron (CE) & X-rays

77-300 K, 0-10 kOe

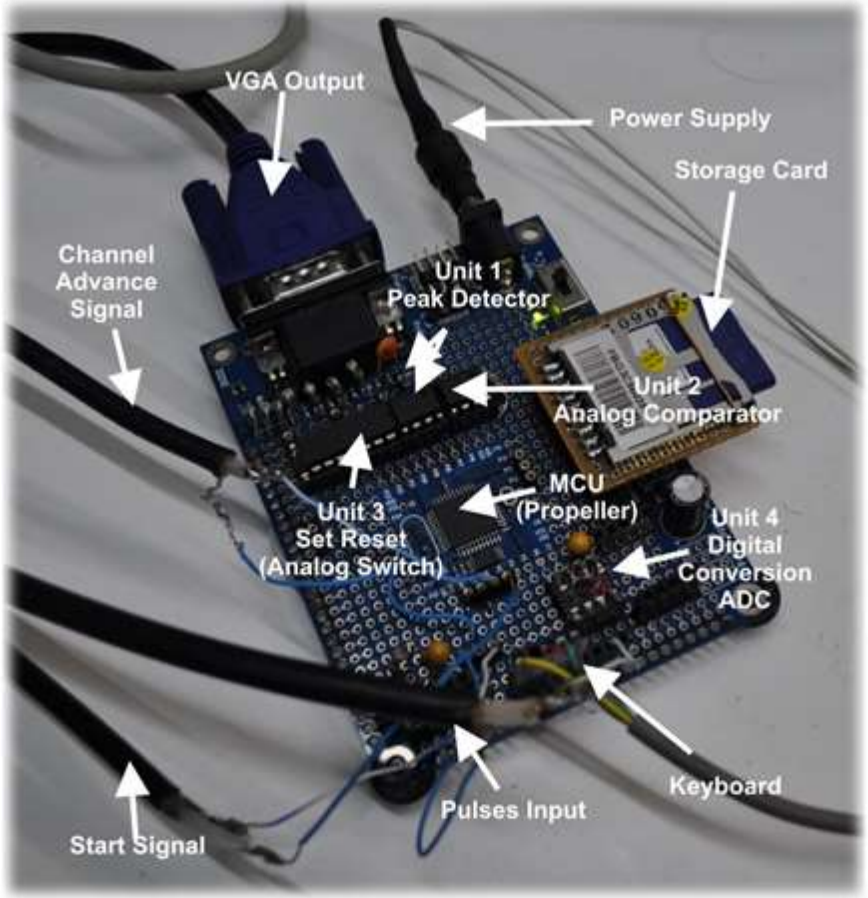
powder, films & single crystal samples

CE, X-rays backscatter Mössbauer spectrometer (300K)

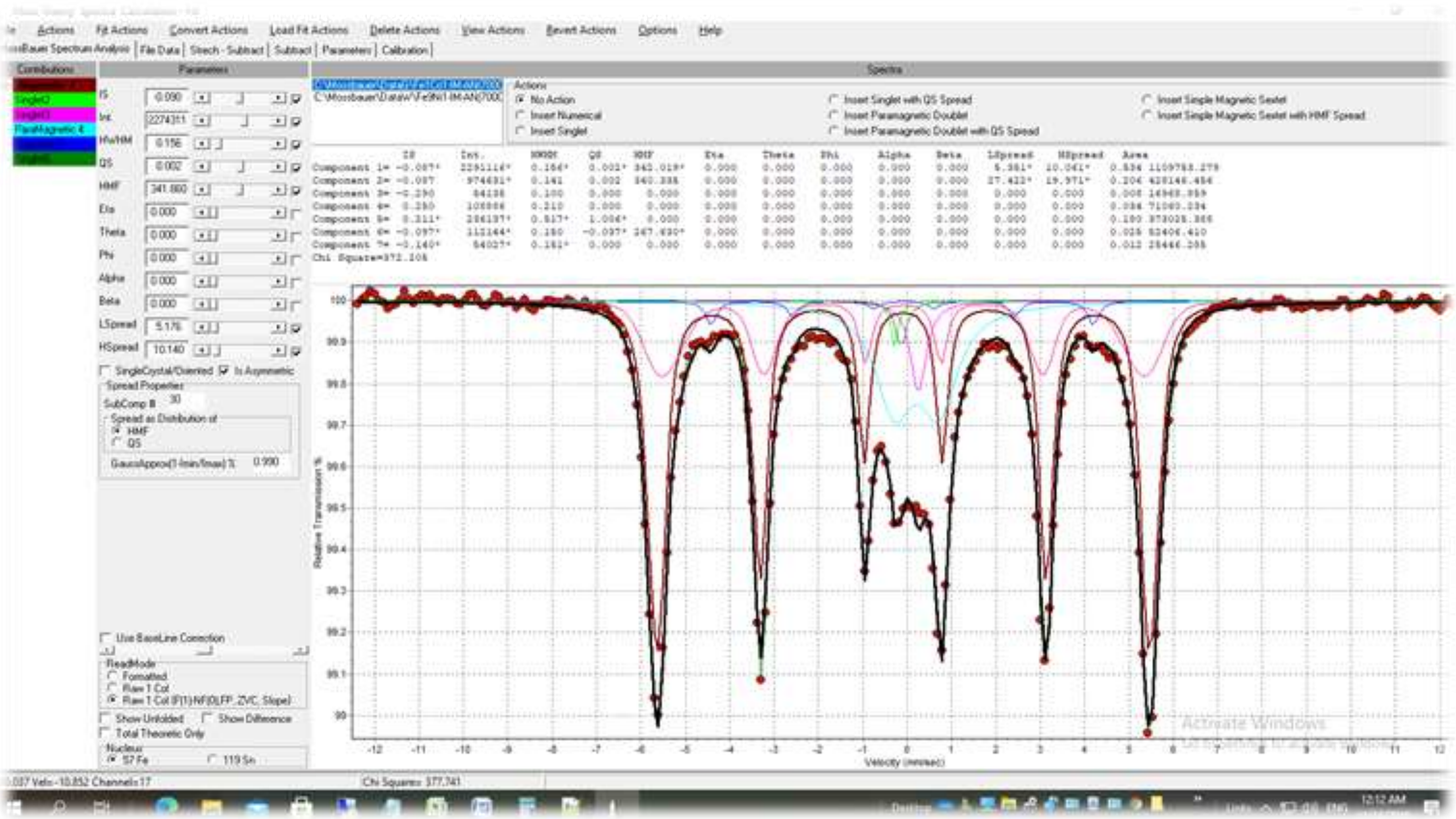
surface properties of thin and thick films

# Mössbauer Spectroscopy

## Continuous development of data acquisition instrumentation, experimental methodology and software




Mössbauer spectroscopy prototype digital data acquisition card (MossCard)




Mössbauer 1st on-the-fly spectra fitting software (IMSG)
$^{57}Fe$, & $^{119}Sn$ full Hamiltonian, real multi-component
HMF-QS distributions for powder & single crystal samples

Vibrating Sample Magnetometer (77-1300 K, 0-20 kOe)
• powder, films and single crystal samples
• vector VSM (combined x & y axes magnetic moments)
• magnetoresistivity measurements

Powder XRD (300 K)
• Quantitative analysis – Rietveld Refinement

## Tube Furnaces

max Temp. 1700 °C

max Temp. 1000 °C with gas flow

Samples in quartz ampules evacuation ($10^{-3}$-$10^{-6}$ Torr ) and sealing (acetylene flame)

Sample annealing (up to 1000 °C)
under continuous vacuum ($10^{-3}$-$10^{-6}$ Torr )

Arc melting (metals and alloys)

# Structural, Electronic & Magnetic Properties of Materials-Characterization & Study

## Biomimetic $[Fe_4S_4]_x[Sn_nS_{2n+2}]_y$ n=1, 2, 4 Amorphous Chalcogels (Mercouri Kanatzidis Group)

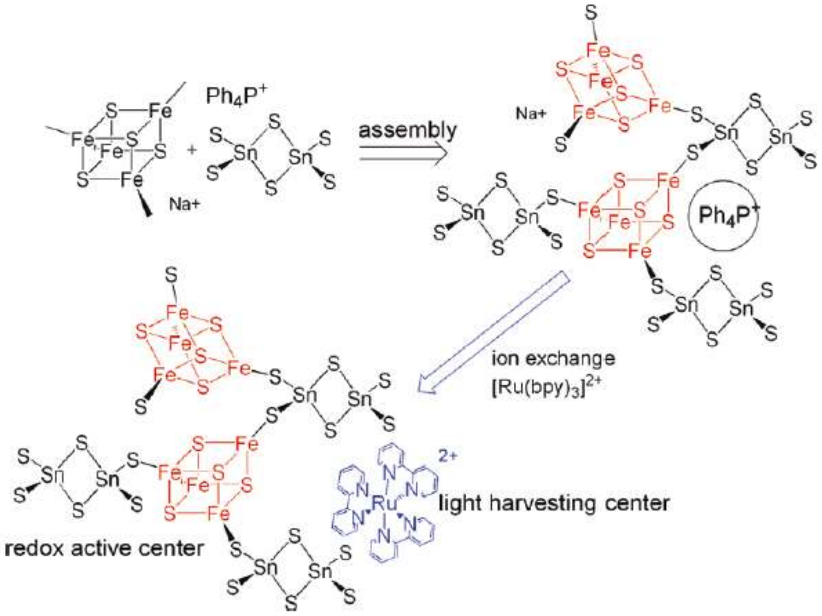
ITS-cg1 ($[SnS_4]$)

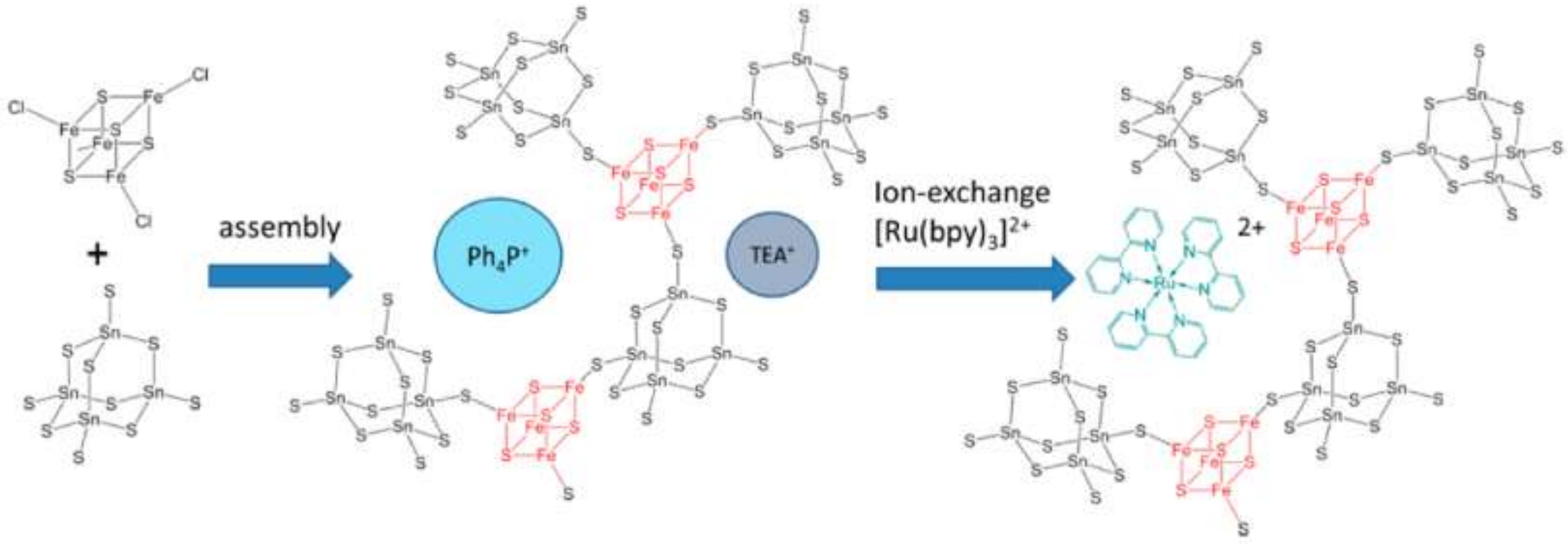
ITS-cg2 ($[Sn_2S_6]$)

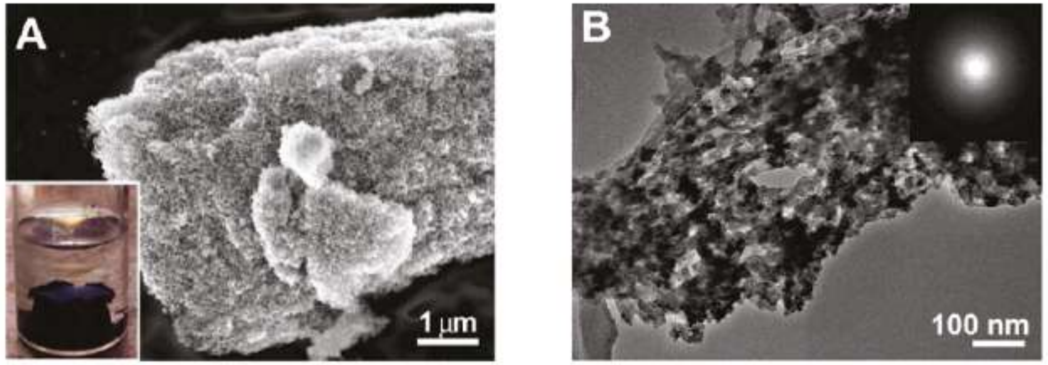
ITS-cg3 ($[Sn_4S_{10}]$)

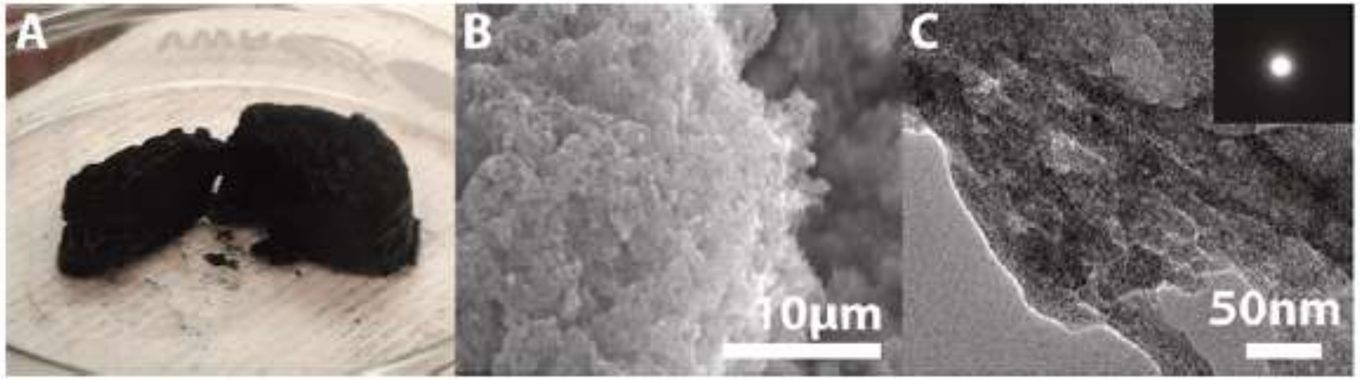
$$[Fe_4S_4Cl_4]^{2-} + [Sn_2S_6]^{4-} \rightarrow \{[Fe_4S_4]_x[Sn_2S_6]_y\}^{m-} + yCl^-$$

$$\{[Fe_4S_4Cl_4]\}^{2-} + [Sn_nS_{2n+2}]^{4-} \rightarrow \{[Fe_4S_4]_x[Sn_nS_{2n+2}]_y\}^{z-} + yCl^-$$







dx.doi.org/10.1021/ja111275t

dx.doi.org/10.1021/ja311310k

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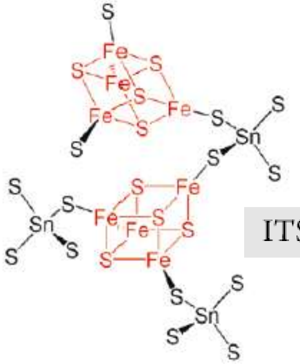
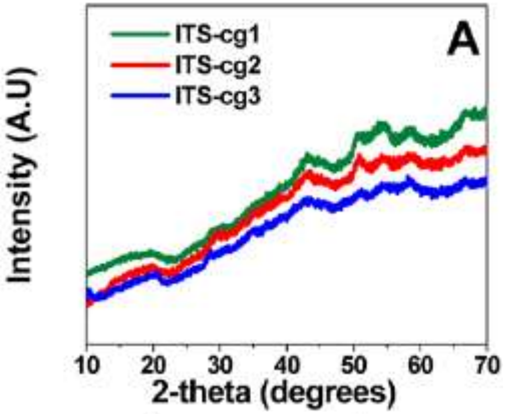
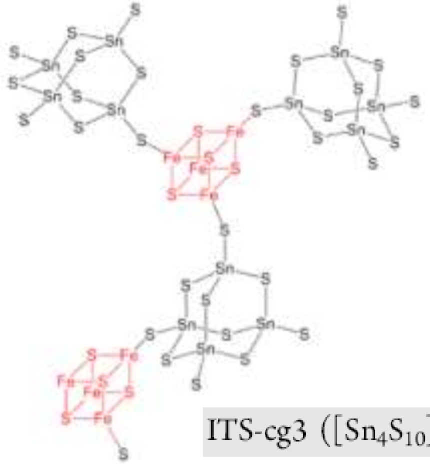
The $[Sn_nS_{2n+2}]$ building blocks' size determines the magnetic interactions between the $[Fe_4S_4]$ cubanes

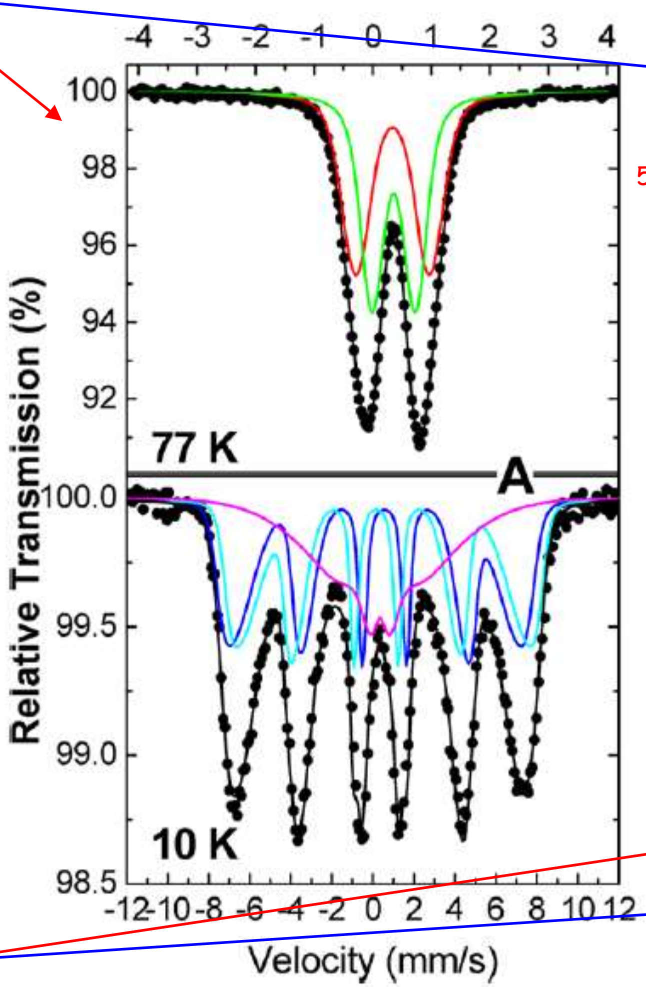



ITS-cg1 ($[SnS_4]$)

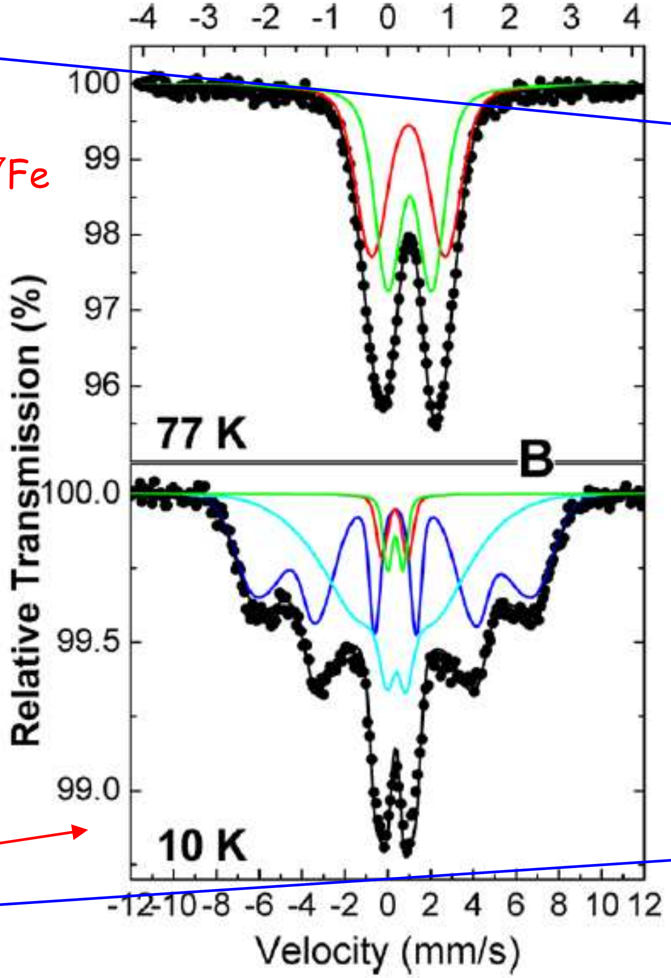


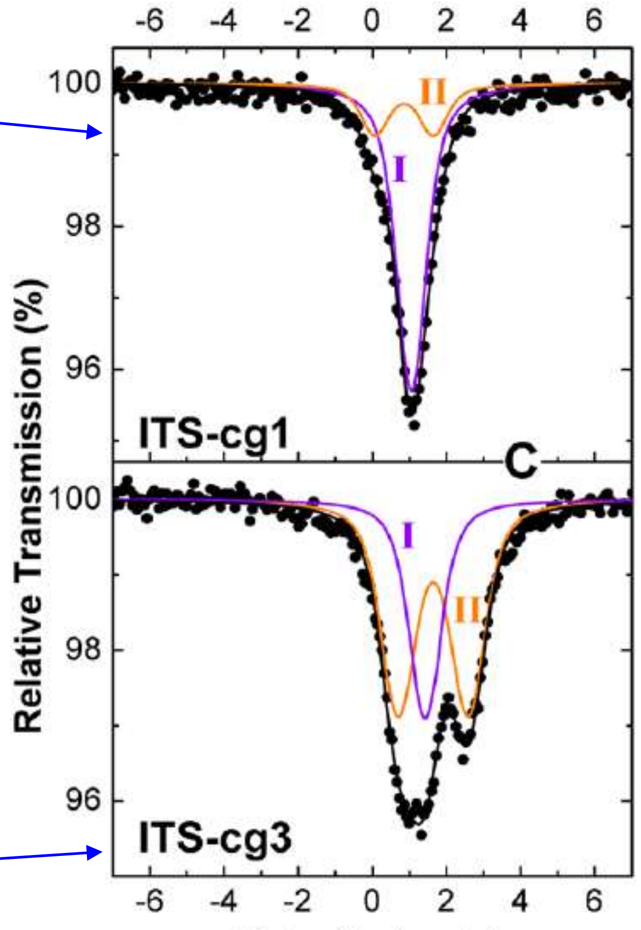




ITS-cg3 ($[Sn_4S_{10}]$)




$^{57}$Fe




$^{119}$Sn

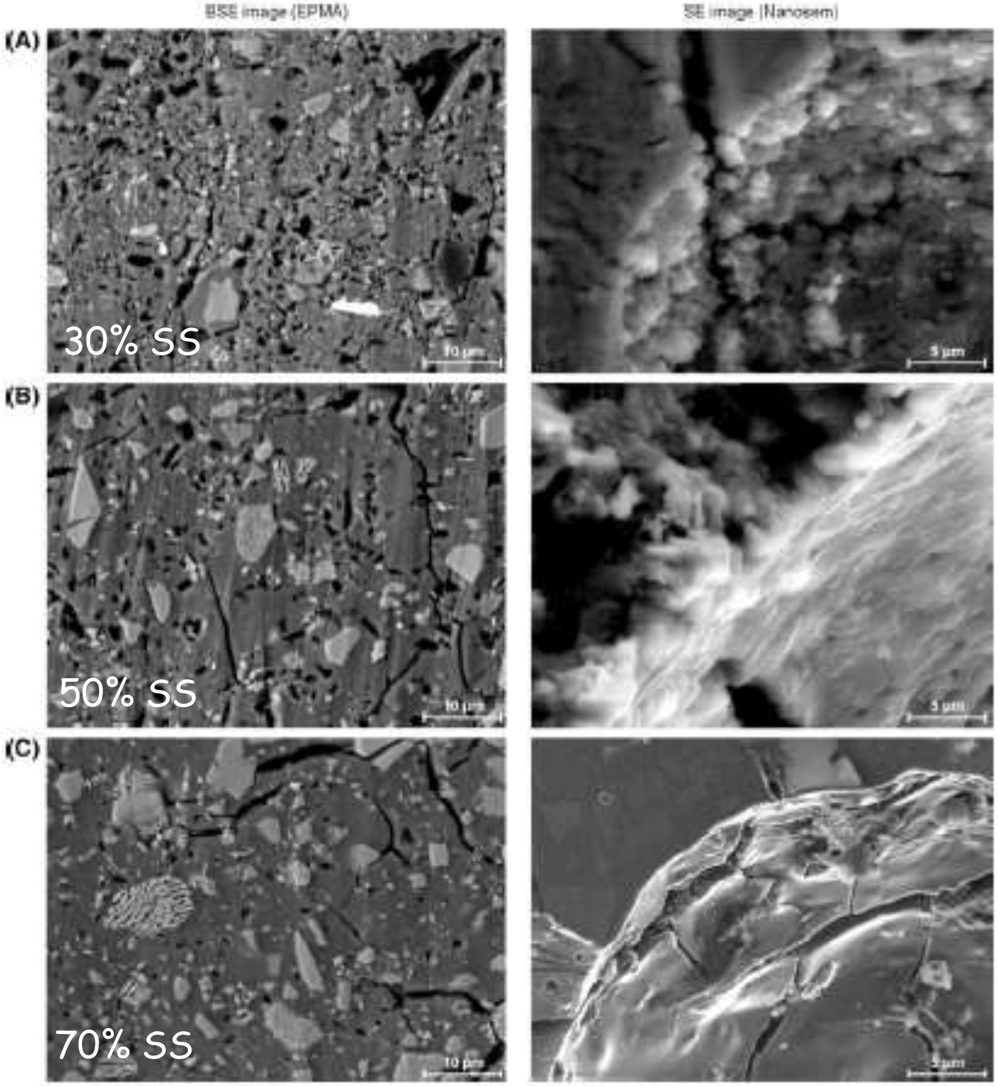
ITS-cg1 ($[SnS_4]$)

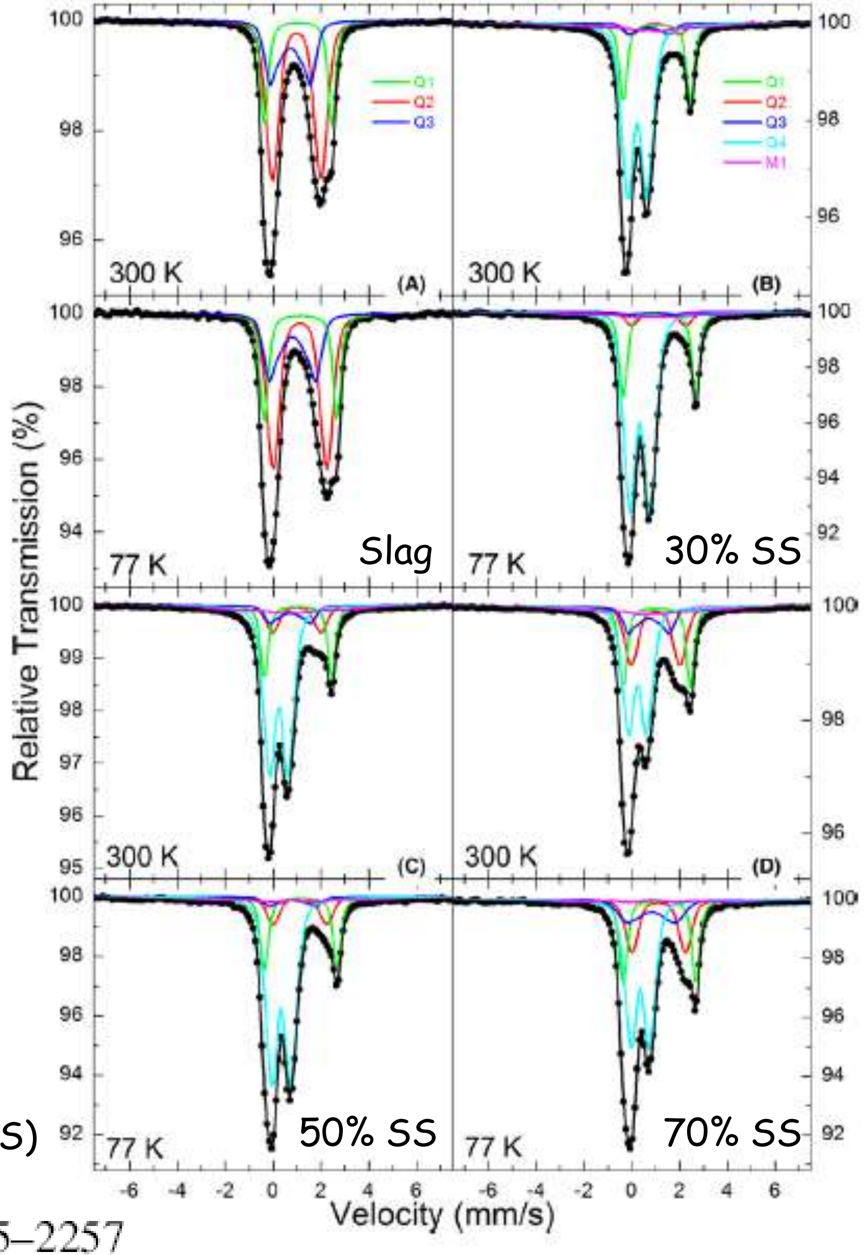
ITS-cg3 ($[Sn_4S_{10}]$)

**77 K**

# Structural, Electronic & Magnetic Properties of Materials-Characterization & Study

## Inorganic Polymers From $CaO$-$FeO_x$-$SiO_2$ Slags alternatives to ordinary Portland cement (Yiannis Pontikes Group)




Fayalite ($Fe_2SiO_4$) Slag + NaOH (SH) + $(Na_2O)_x \cdot (SiO_2)_y$ (SS)
alkali solution activation

*J Am Ceram Soc.* 2018;101:2245–2257

# Structural, Electronic & Magnetic Properties of Materials-Characterization & Study

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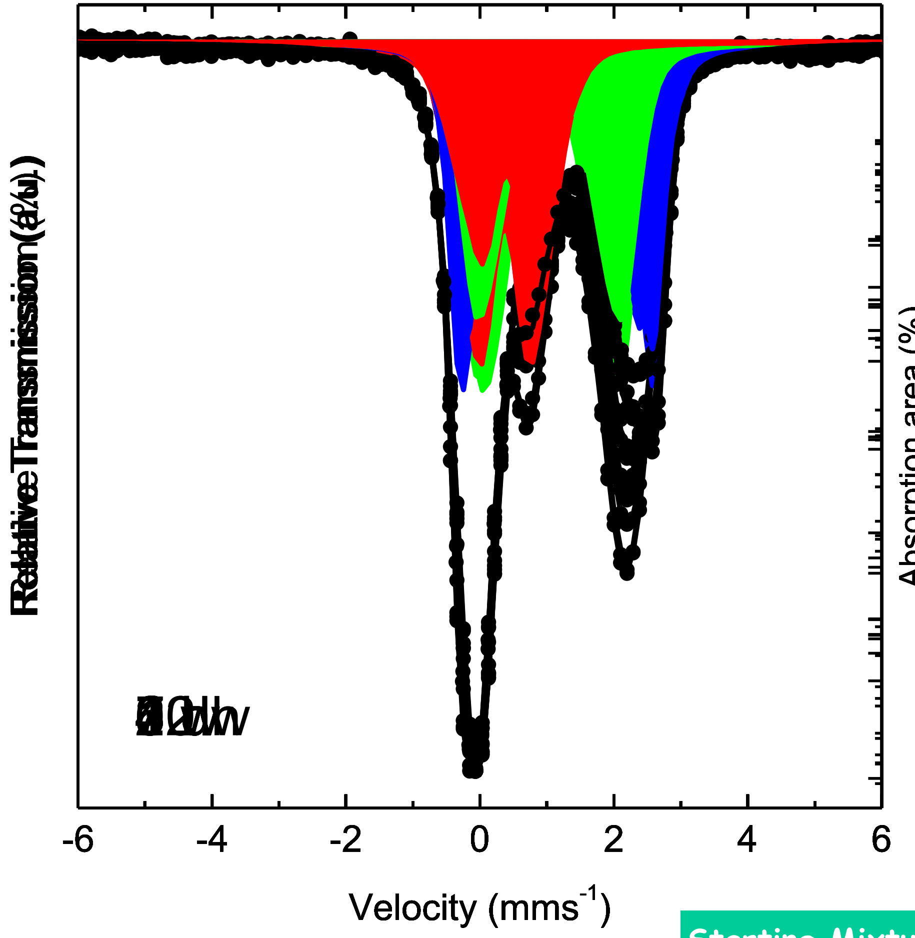
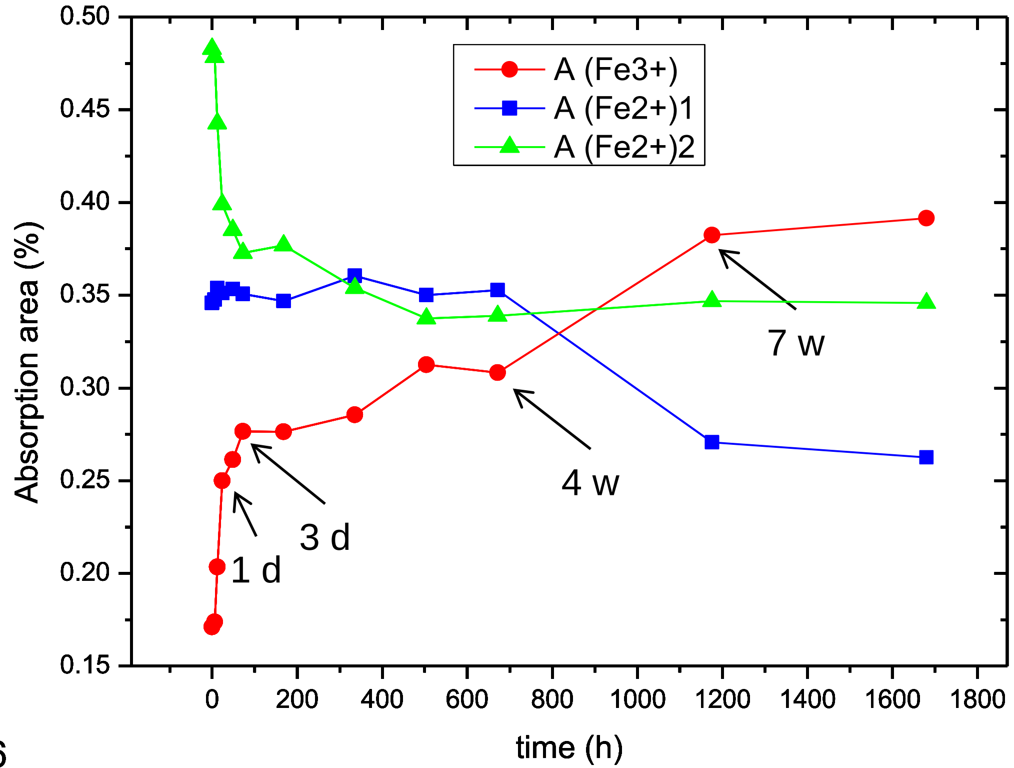
Evolution of the 77K $^{57}Fe$ Mössbauer spectra at different reaction stages




| Starting Mixture Components wt% | | | | |
|---|---|---|---|---|
| FeO | $SiO_2$ | CaO | $Al_2O_3$ | MgO |
| 47 | 34 | 12 | 5 | 2 |

- Activating solutions with molar ratios $SiO_2/Na_2O$=1.6 and $H_2O/Na_2O$=20.

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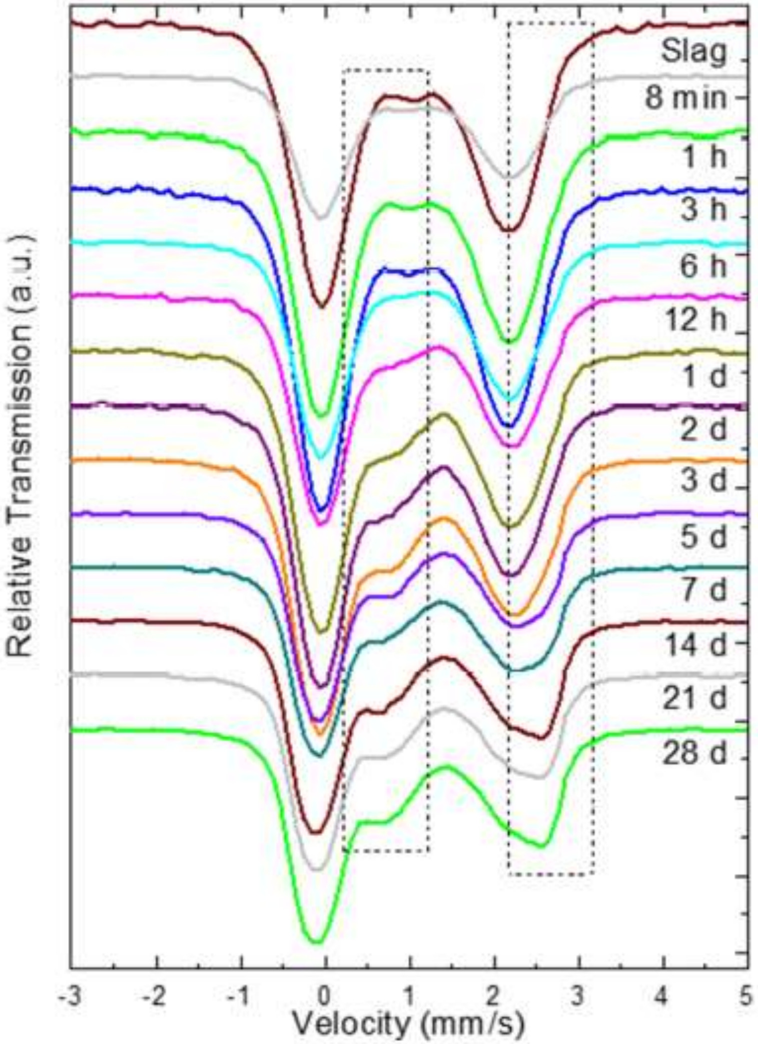


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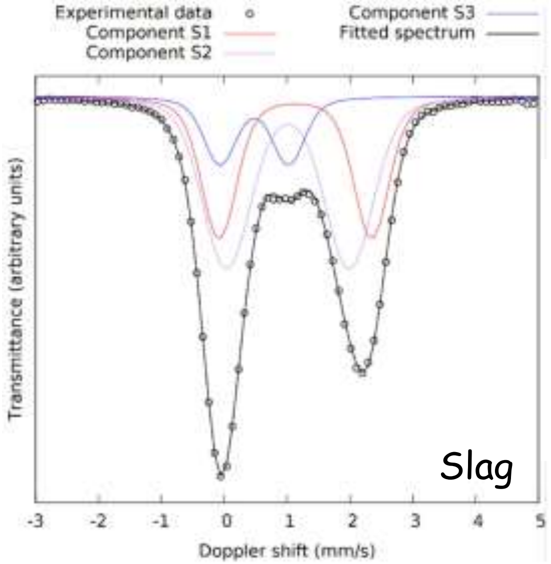


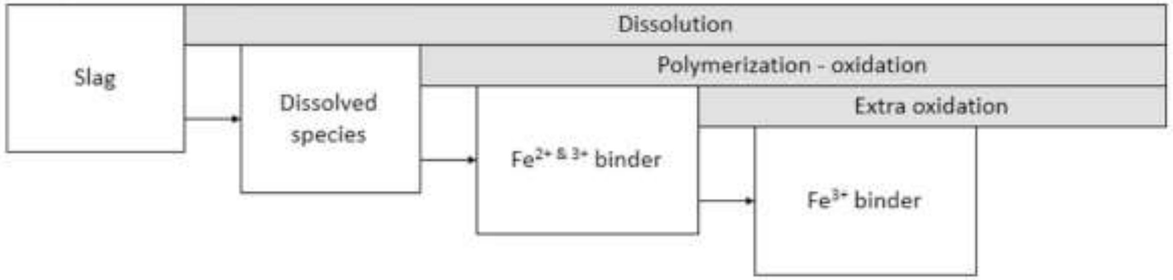


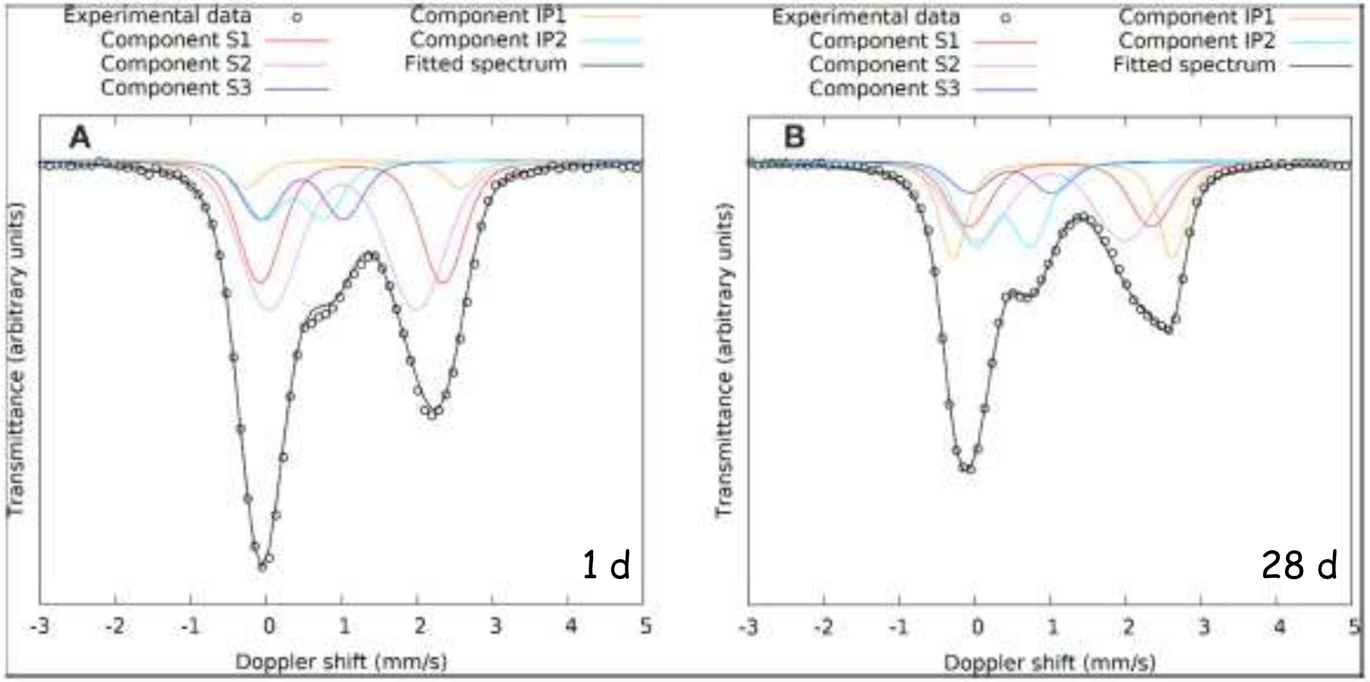


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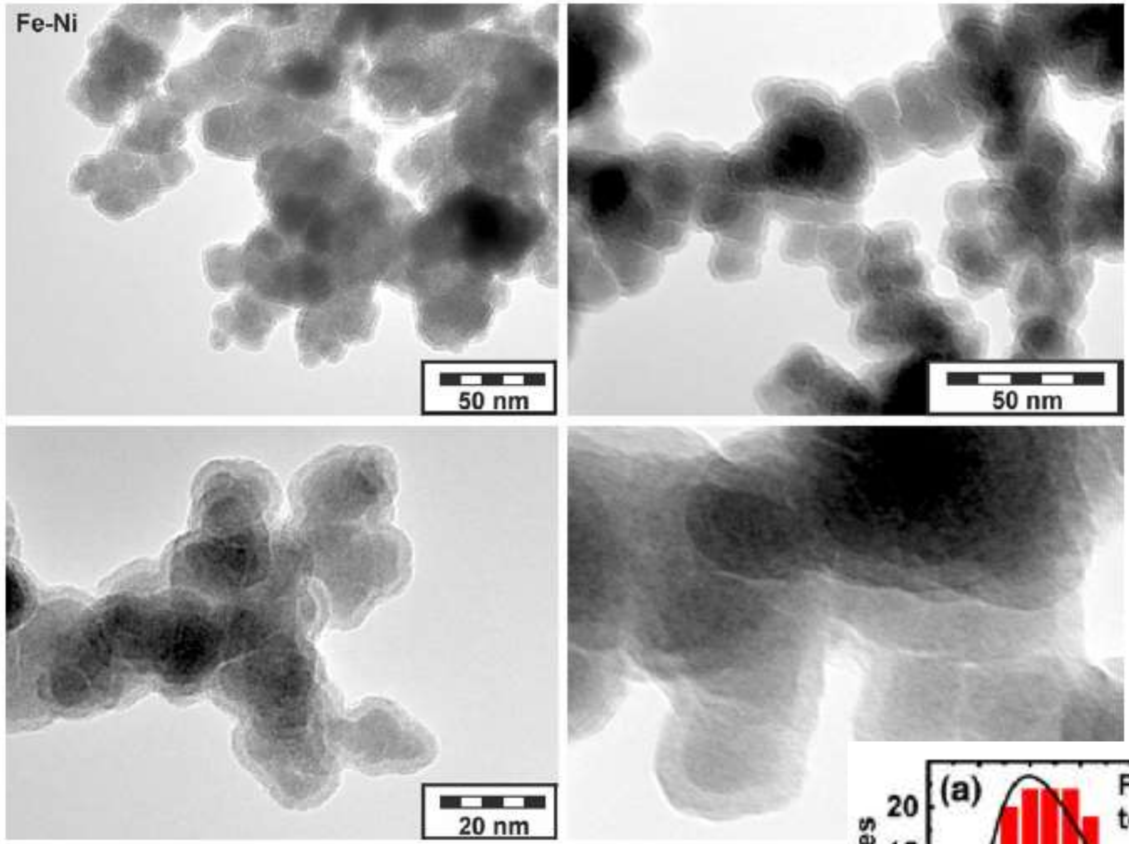
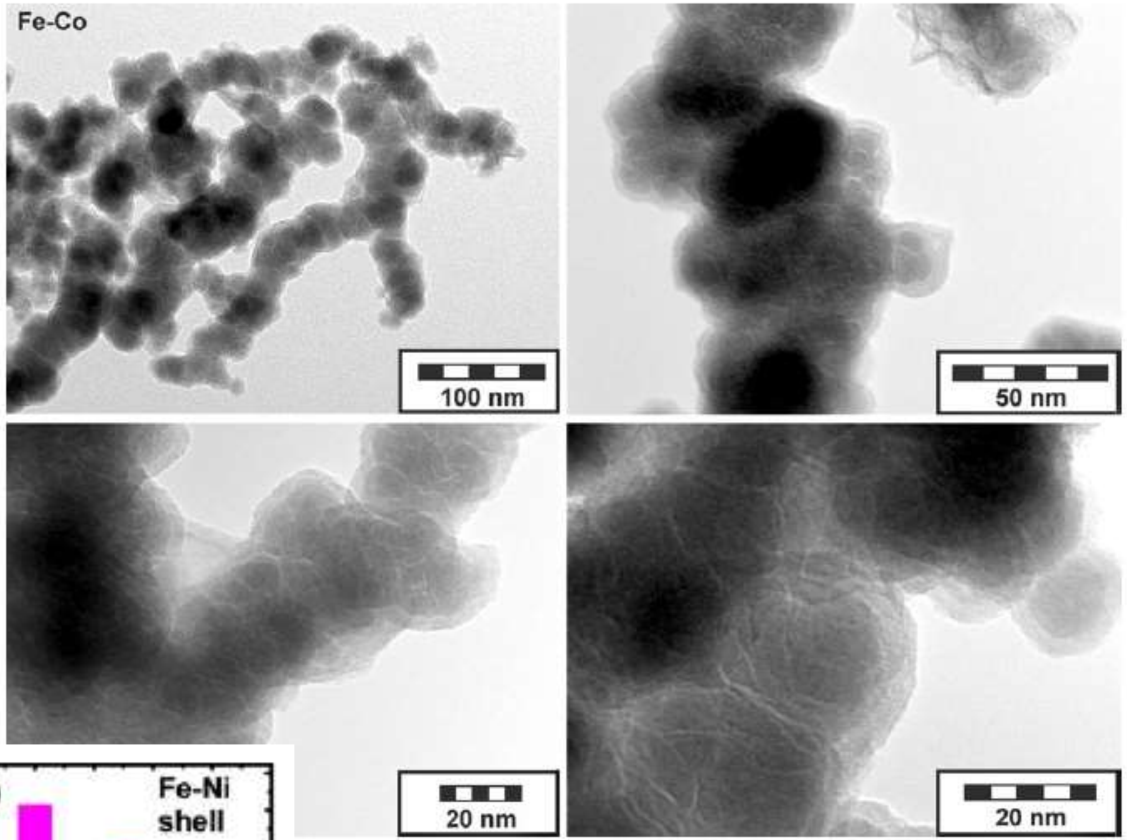
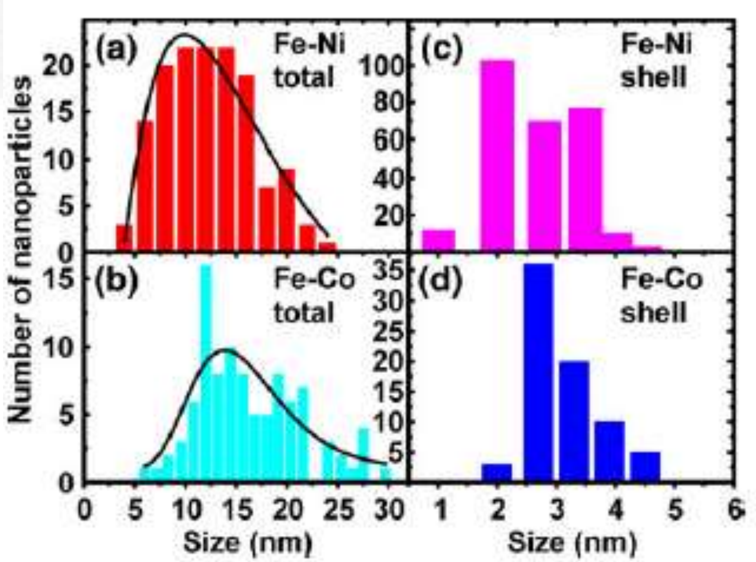
# Structural, Electronic & Magnetic Properties of Materials-Synthesis, Characterization & Study

## Fe-Ni/Fe-Co core-oxide shell Nanoparticles




$NaBH_4$ method @ RT (A. Bourlinos)

J Nanopart Res (2012) 14:1130
DOI 10.1007/s11051-012-1130-z

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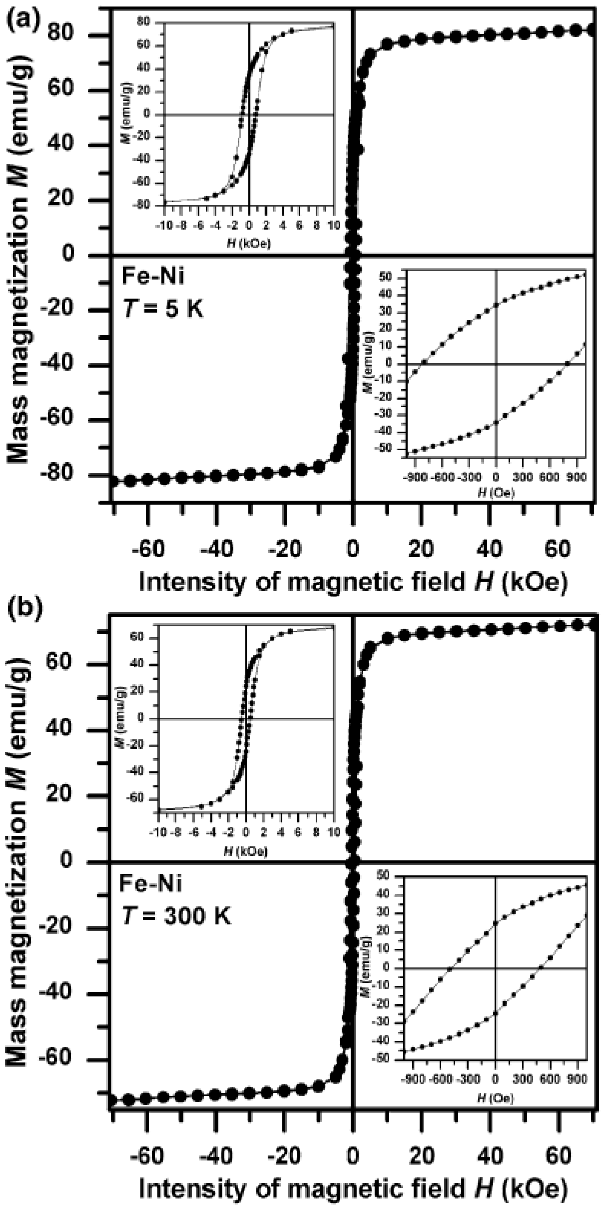


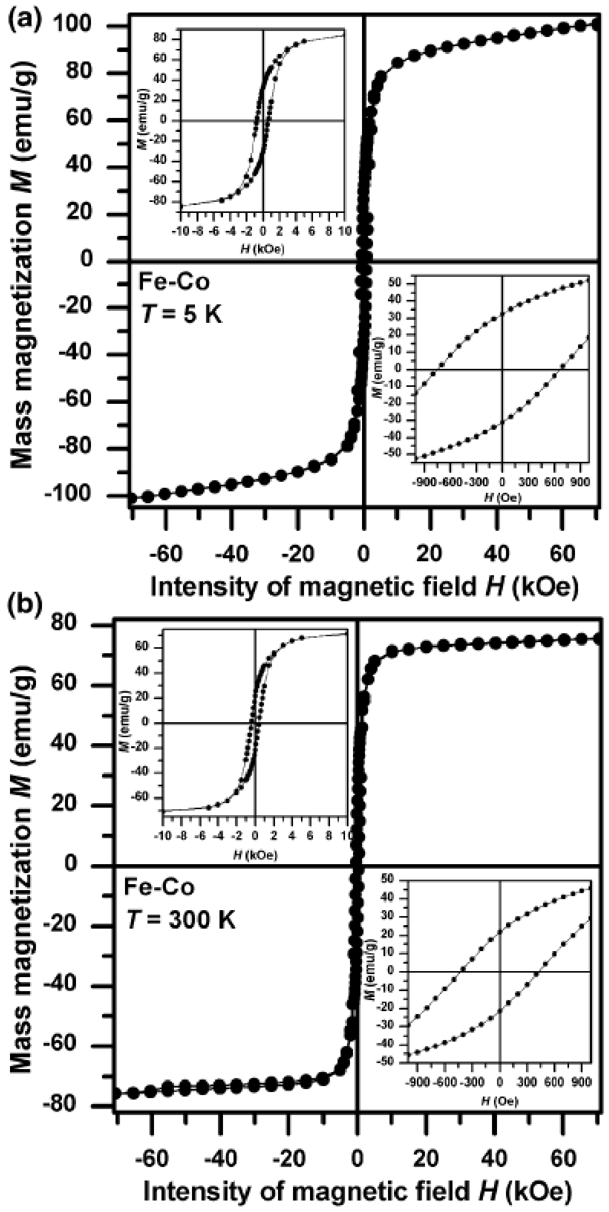


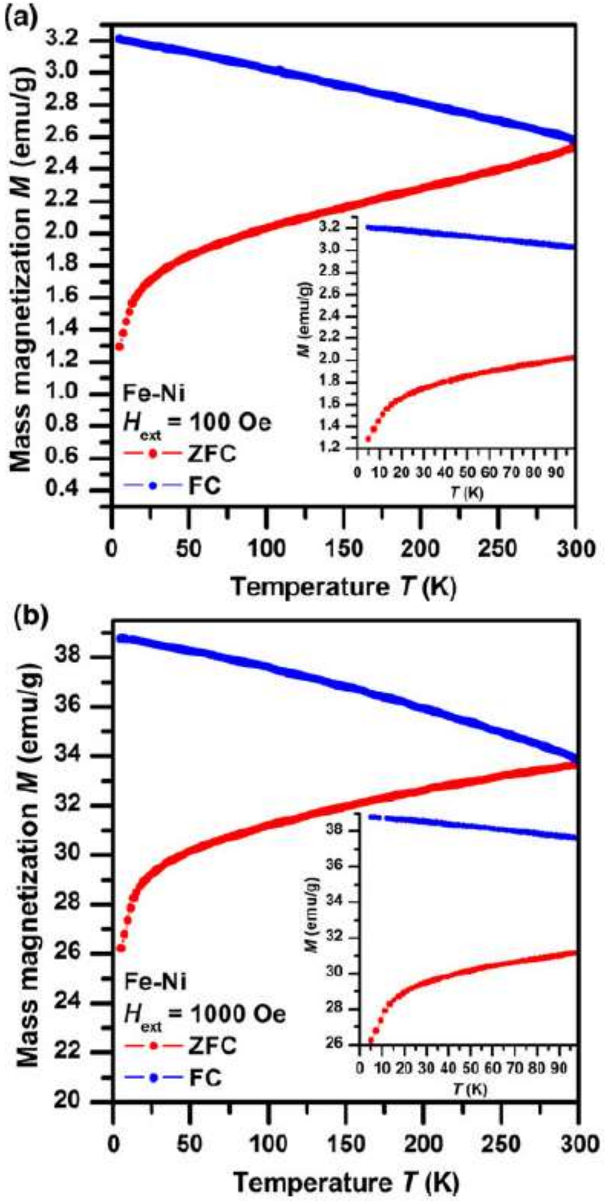


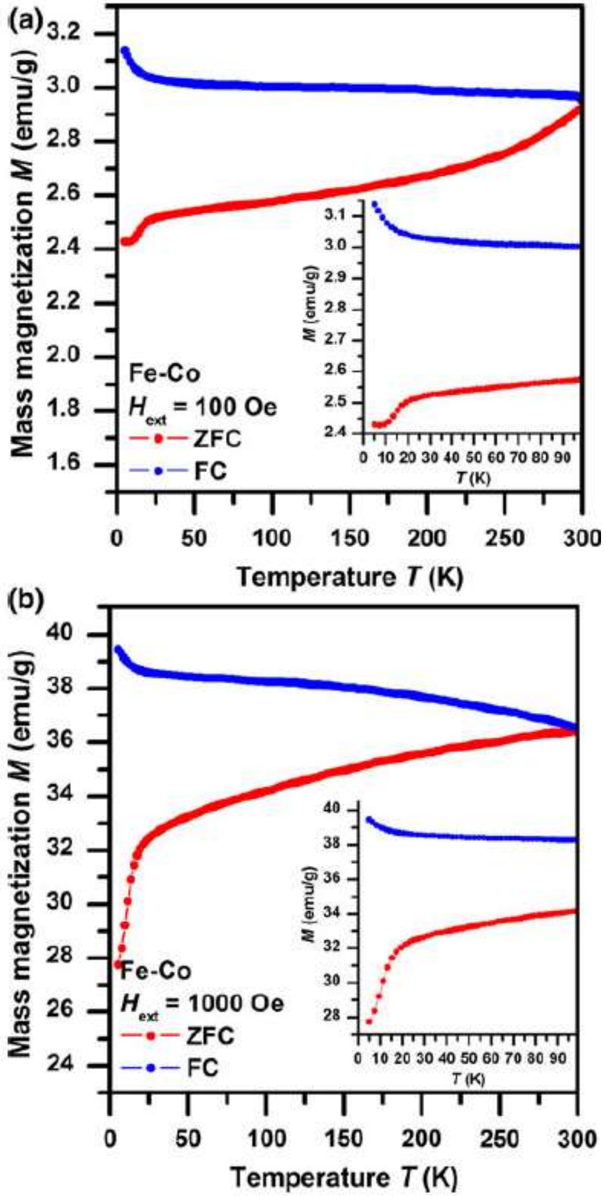

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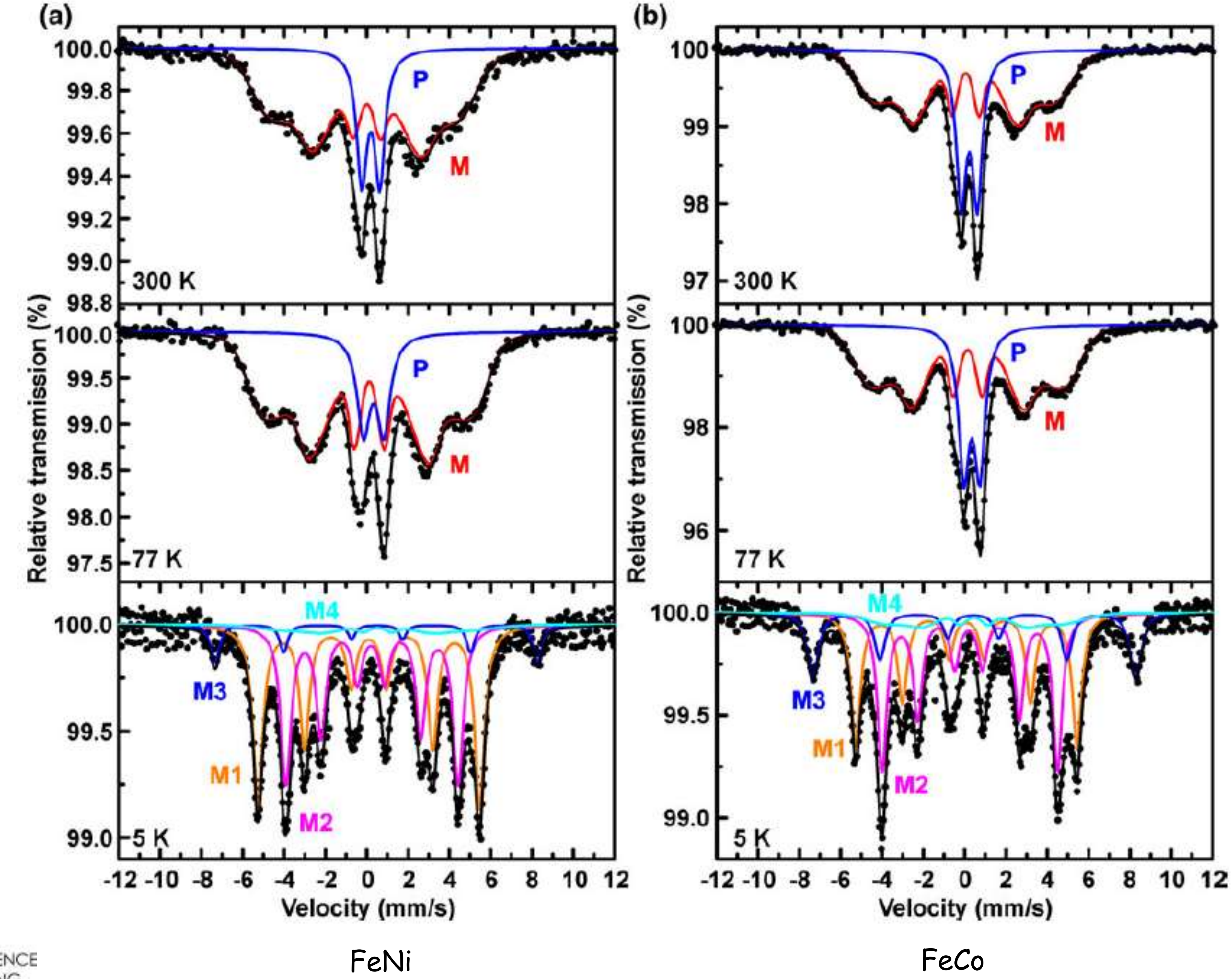
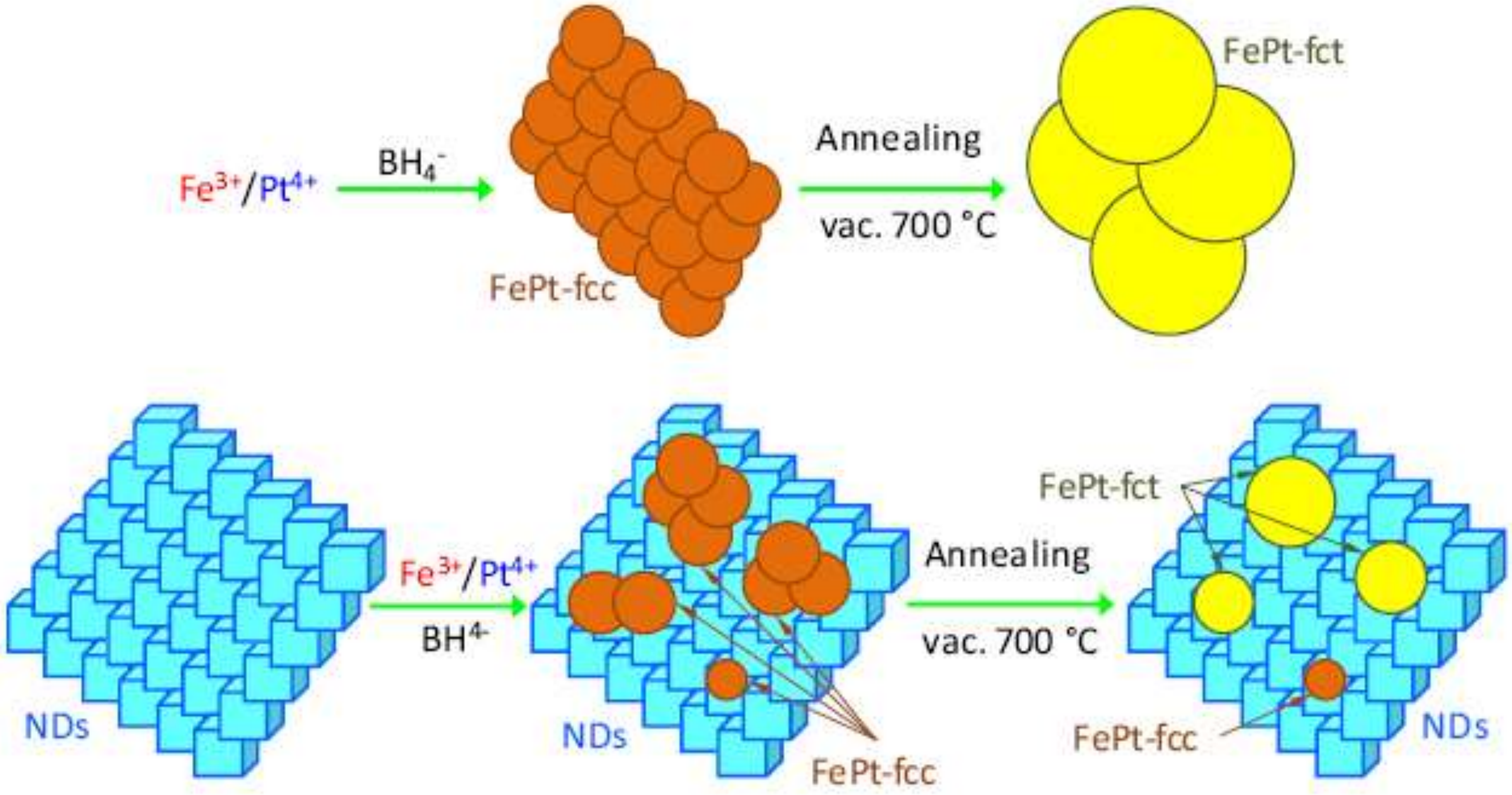



Fe-rich & Fe-poor regions in the NPs core

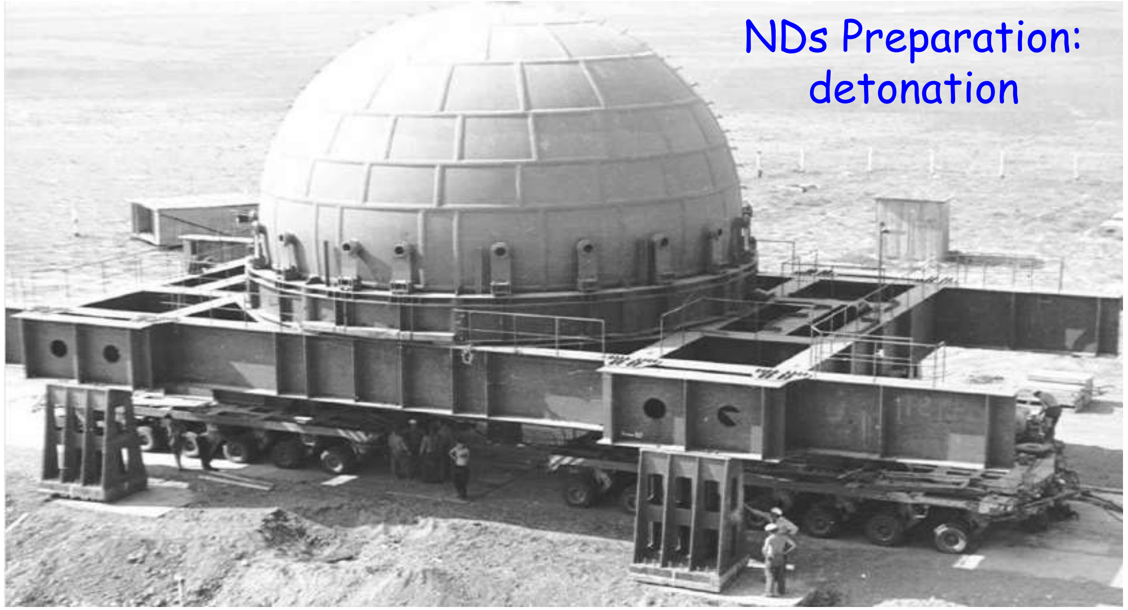
## Magnetic FePt/Nanodiamond (NDs) Hybrid Nanostructures




J Nanopart Res (2016)18:115
DOI 10.1007/s11051-016-3424-z

# Why NDs:

- exceptional structural and mechanical properties
- motor lubricants, plating, coating and polymeric composite agents
- highly biocompatible, vast field for research and applications in biomedicine
- B-doped forms have shown potential for electronic device applications



NDs Preparation: detonation

# Structural, Electronic & Magnetic Properties of Materials-Synthesis, Characterization & Study

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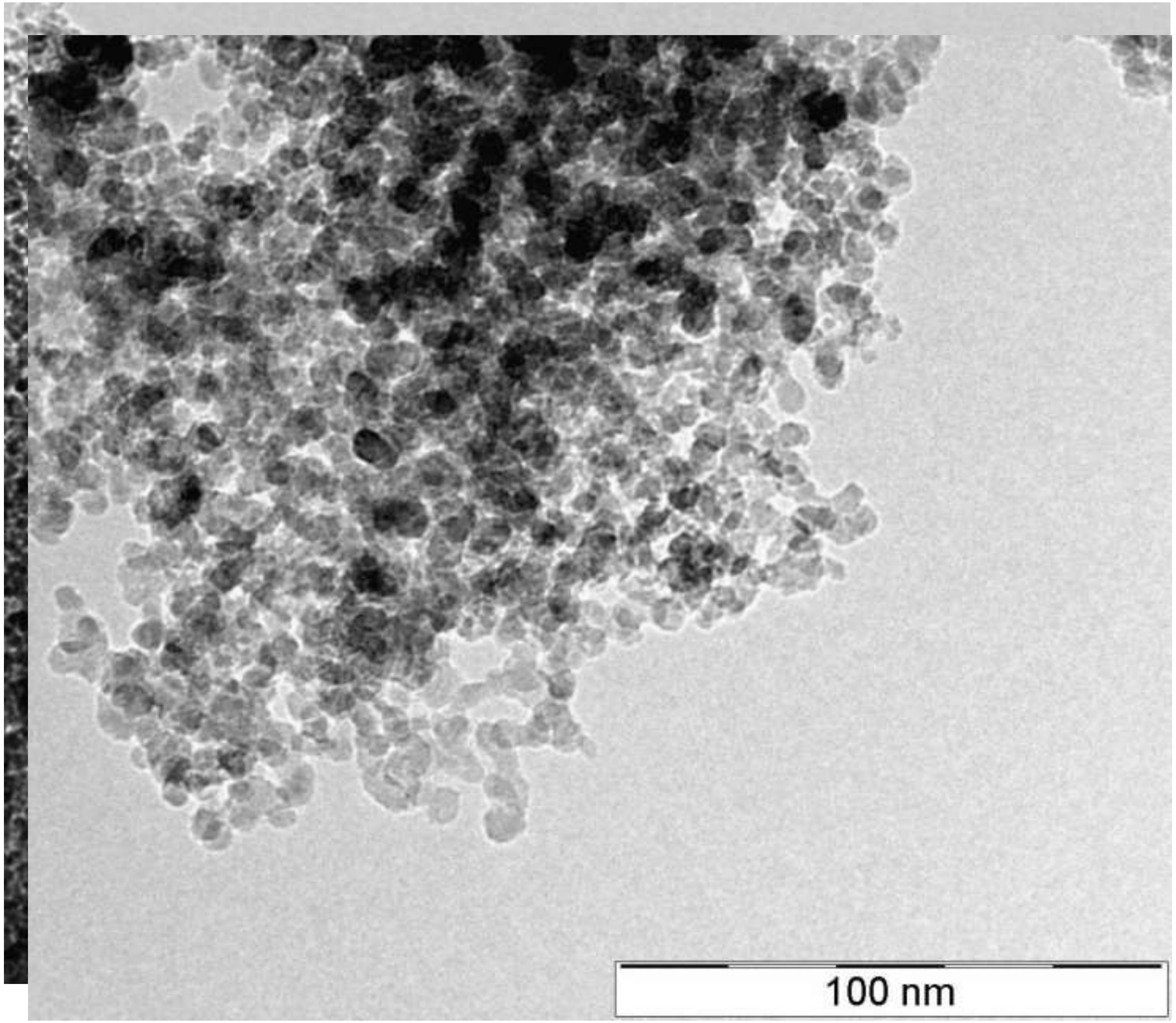


NDs

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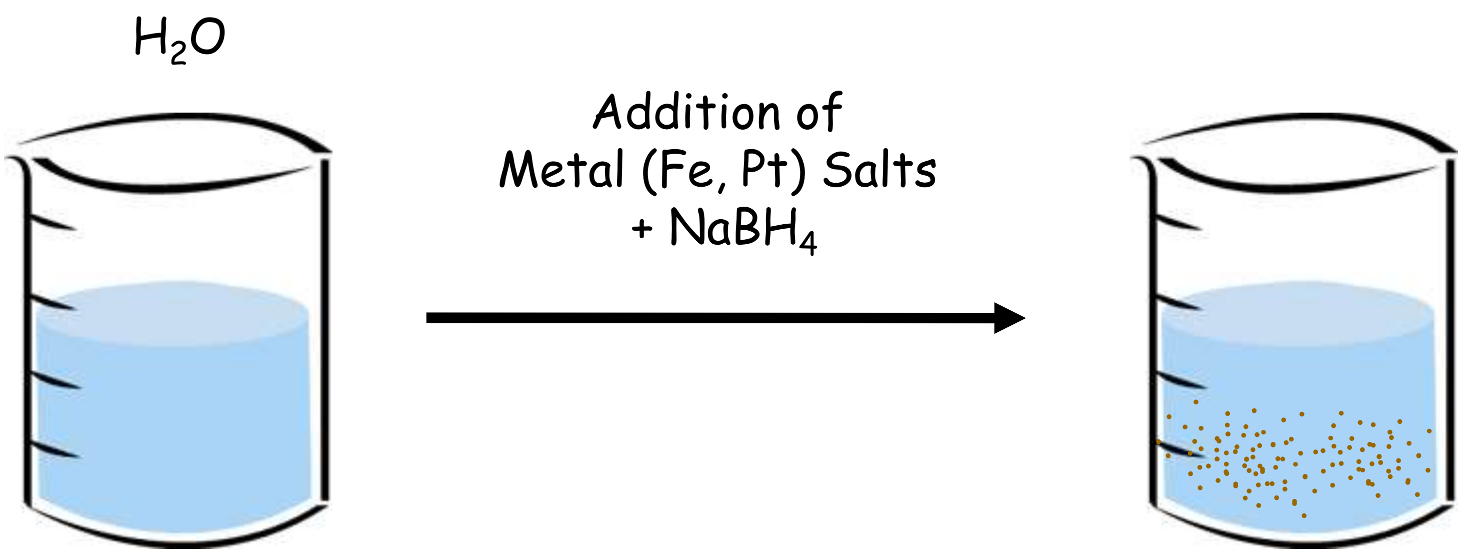
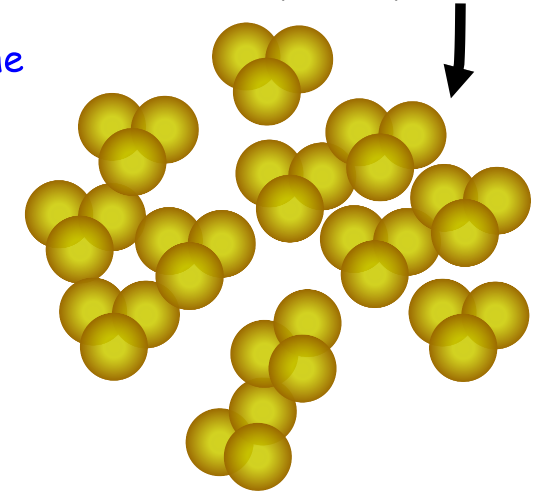



FePt MNPs
co-precipitation



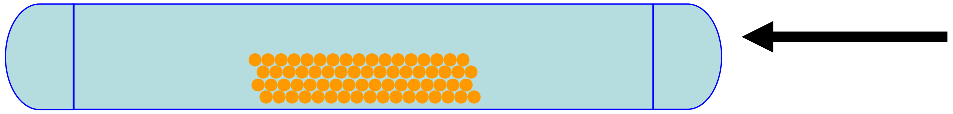
FePt soft (fcc) MNPs

free FePt MNPs

FePt MNPs:

- as prepared: received in the disorder fcc structure
- annealing at high temperatures (>600 °C) to transform to the high anisotropy fct structure
- limitation: annealing leads to sintering effects destroying the microstructural uniformity

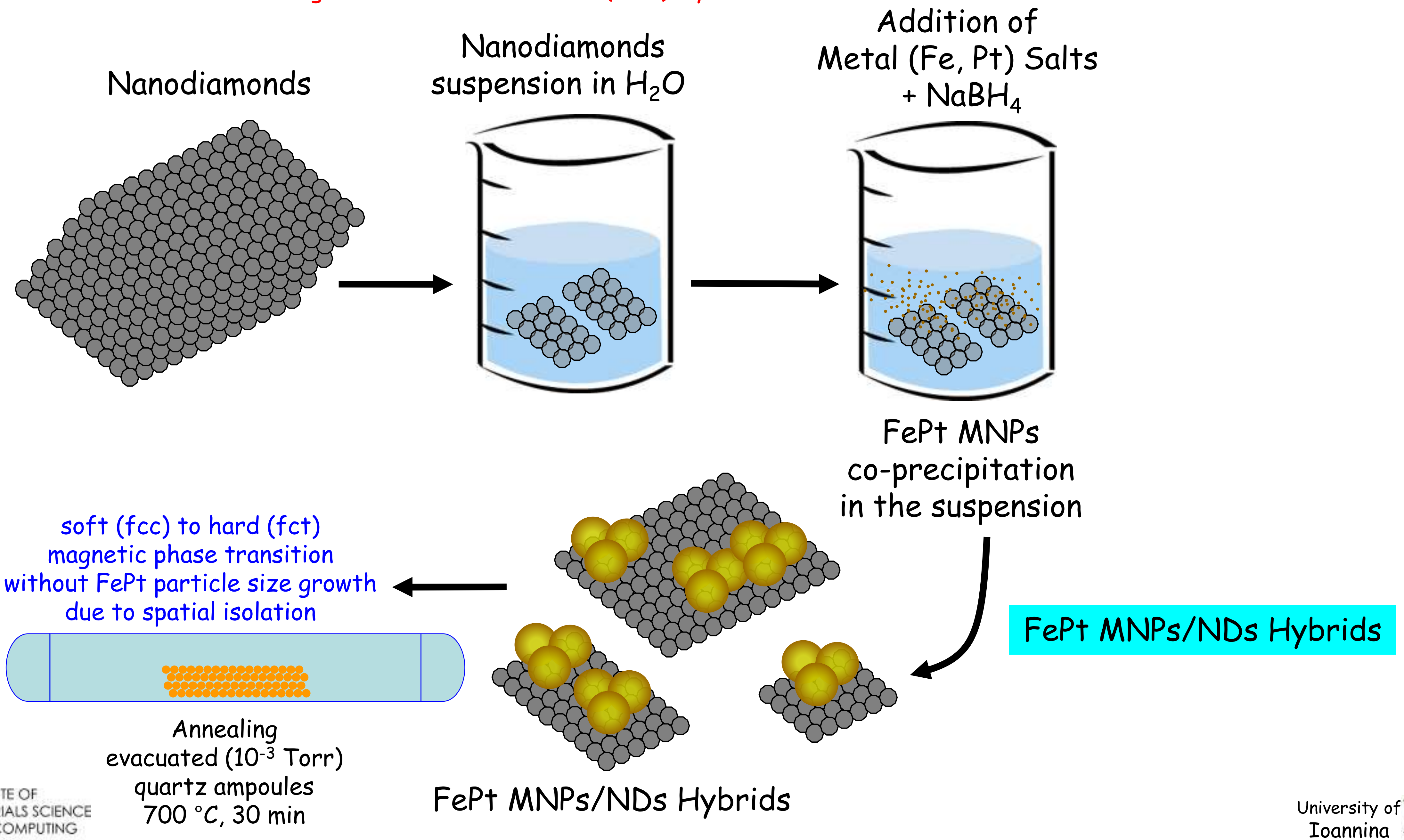
soft (fcc) to hard (fct)
magnetic phase transition



Annealing
evacuated ($10^{-3}$ Torr)
quartz ampoules
700 °C, 30 min

# Structural, Electronic & Magnetic Properties of Materials-Synthesis, Characterization & Study

## Magnetic FePt/Nanodiamond (NDs) Hybrid Nanostructures



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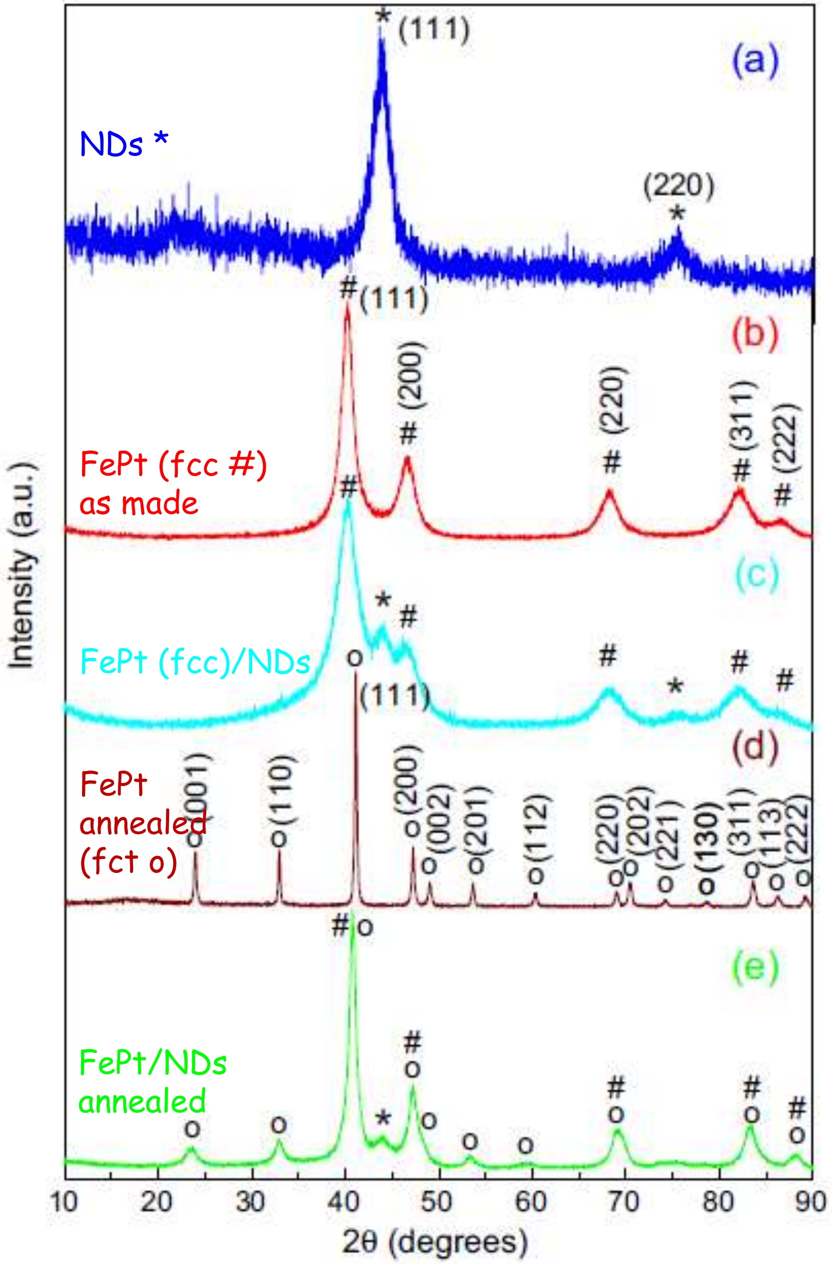
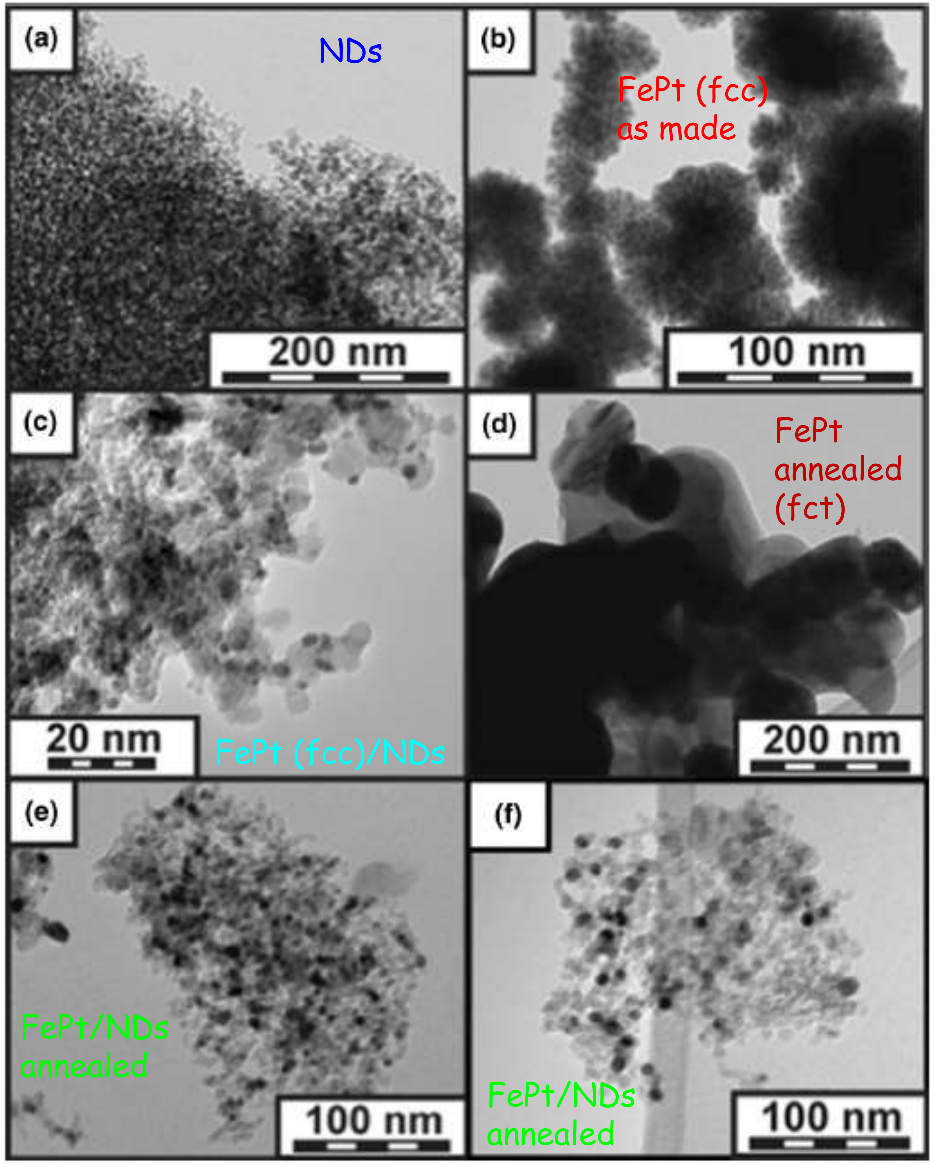
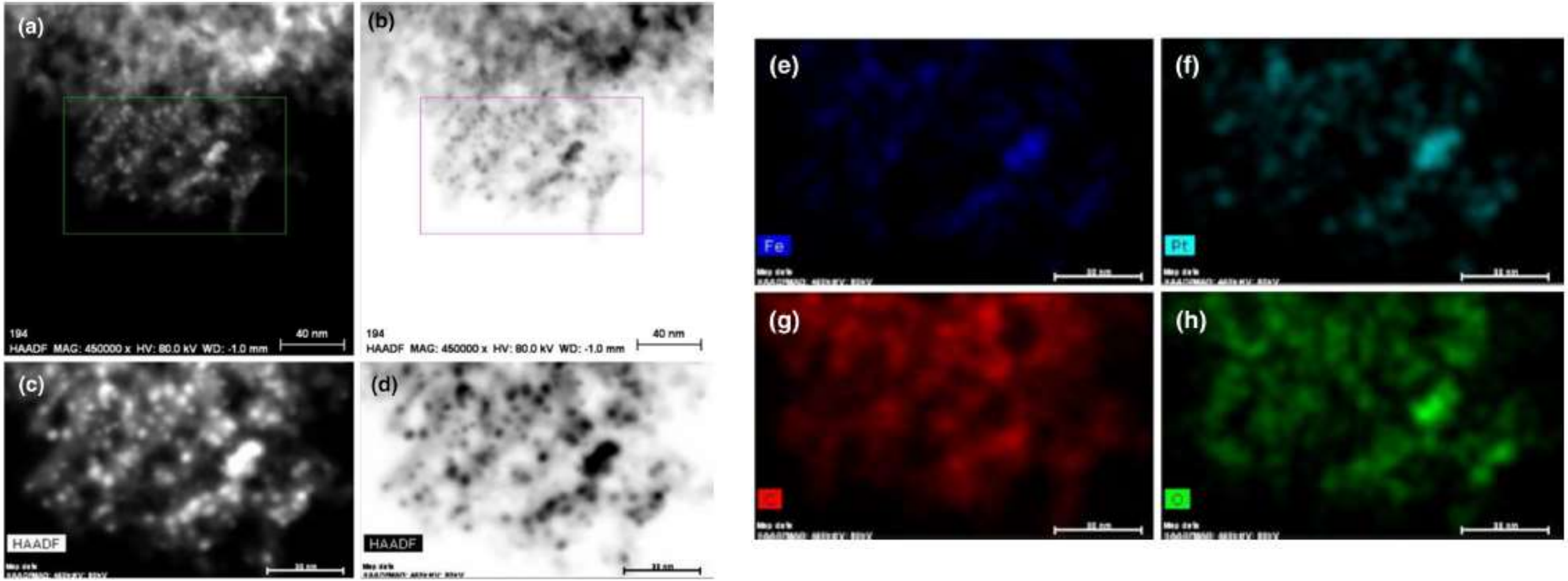


Magnetic FePt/Nanodiamond (NDs) Hybrid Nanostructures

# TEM: chemical mapping



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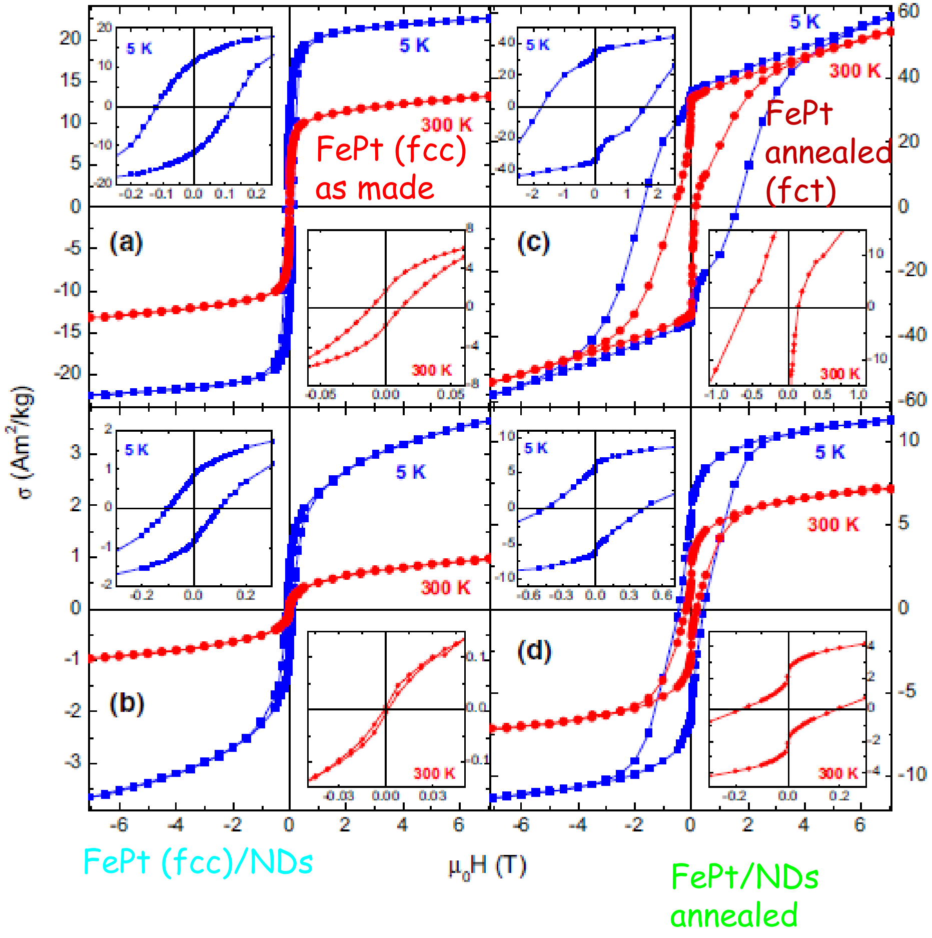


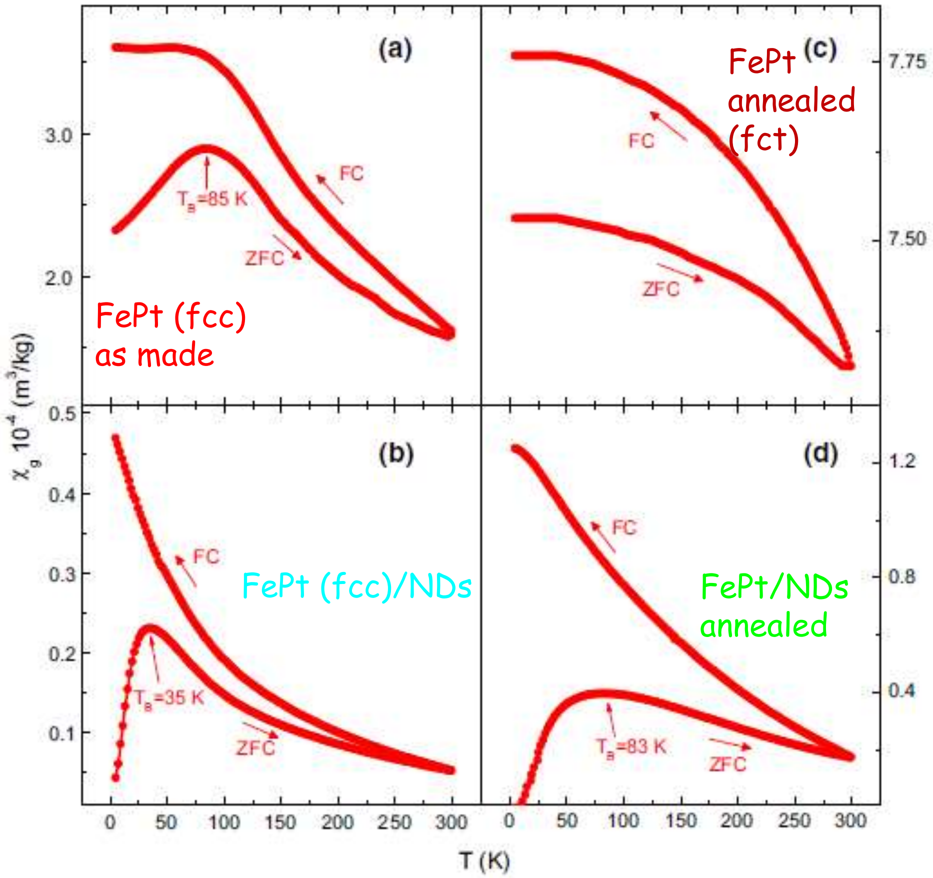

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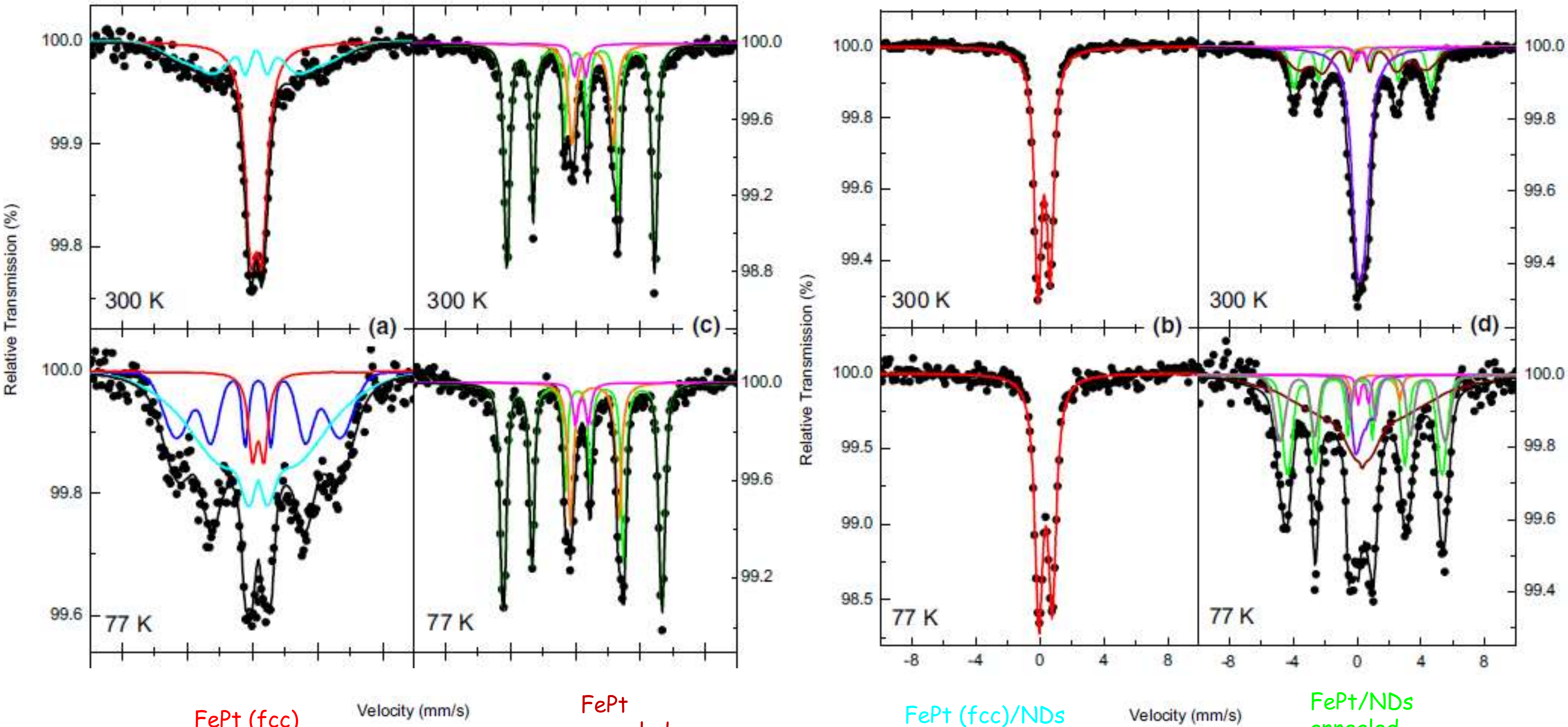


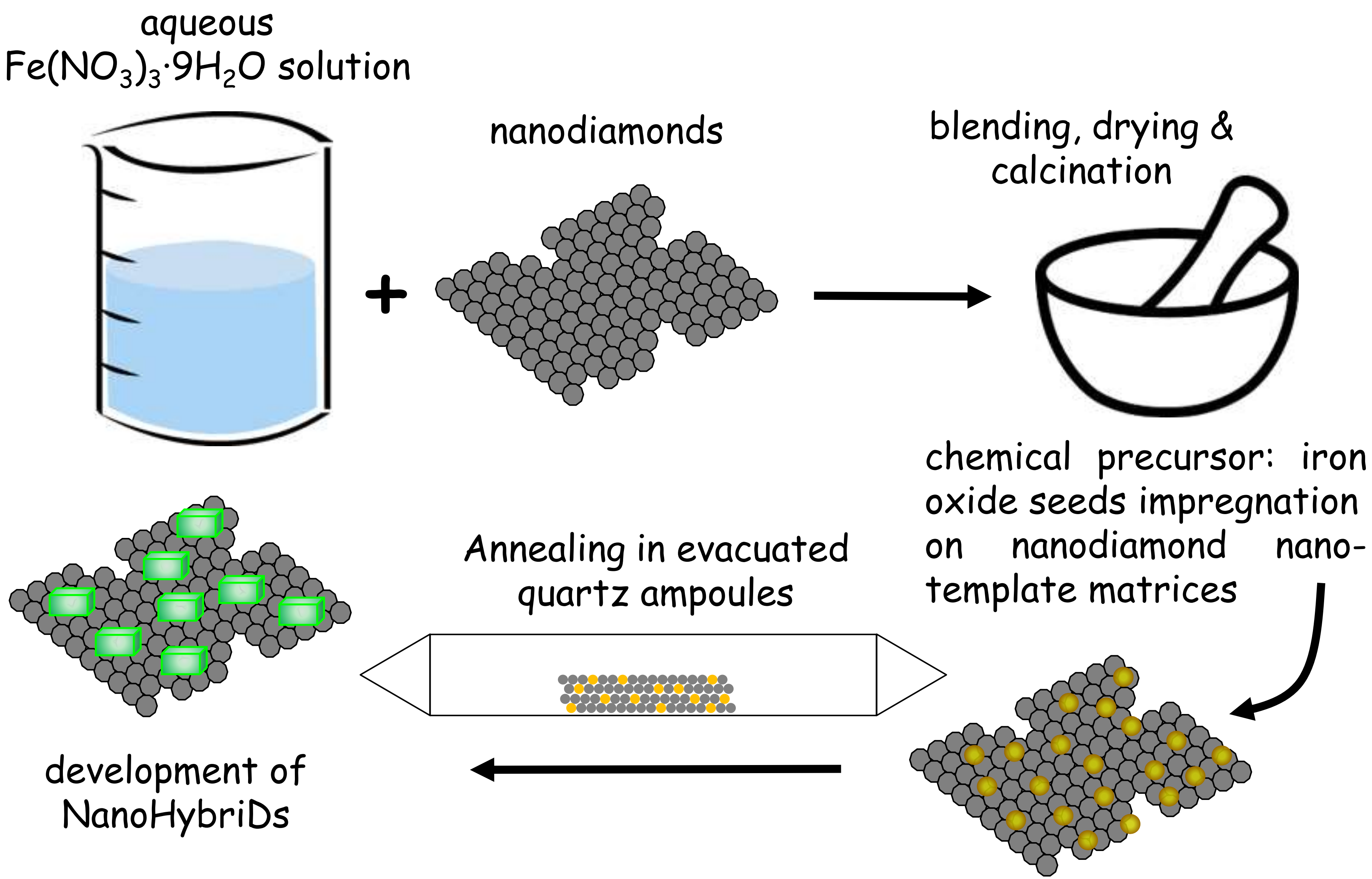

FePt (fcc) as made

FePt annealed (fct)

FePt (fcc)/NDs

FePt/NDs annealed

## Iron Oxide - Iron Carbide NPs/NDs NanoHybrids



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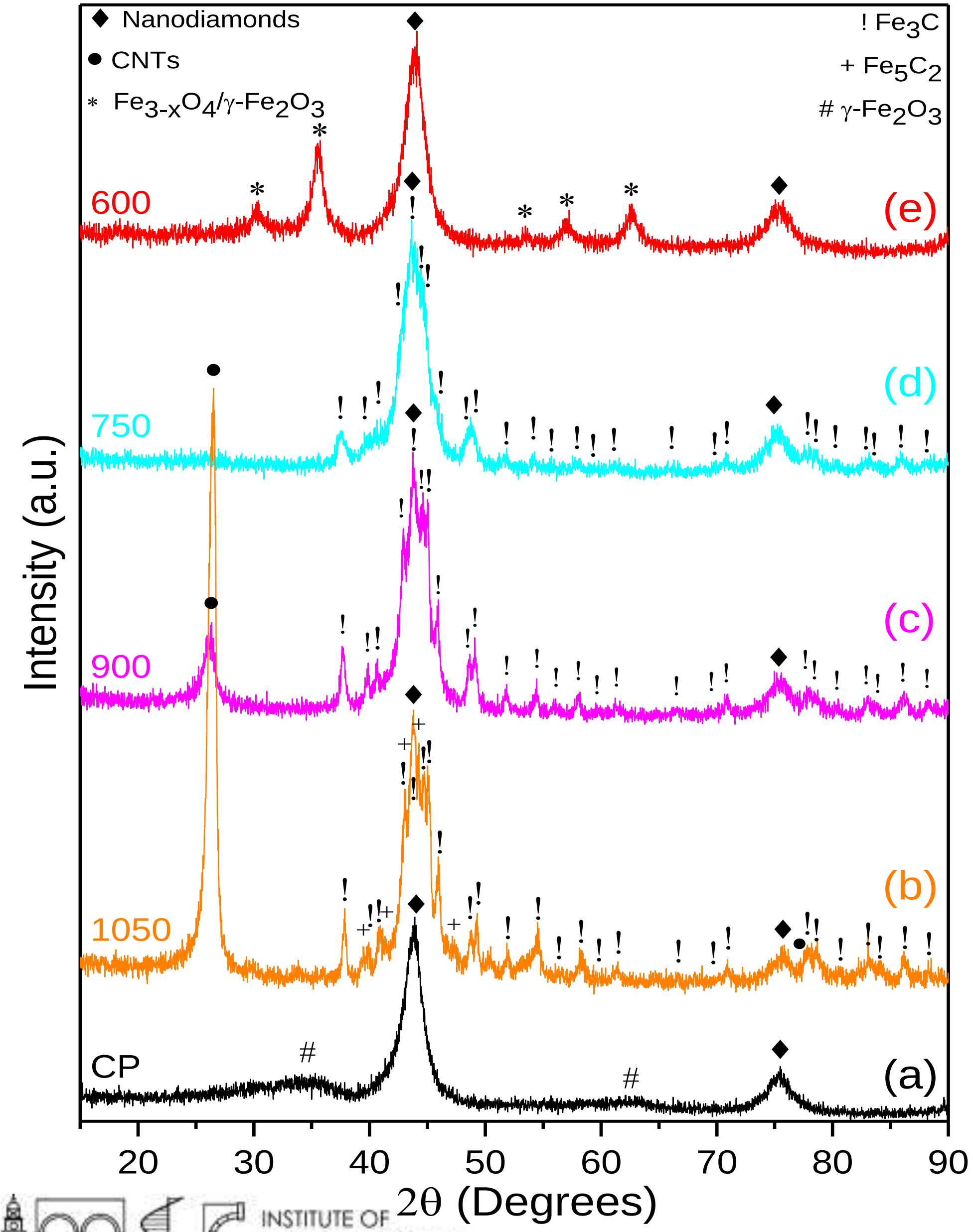
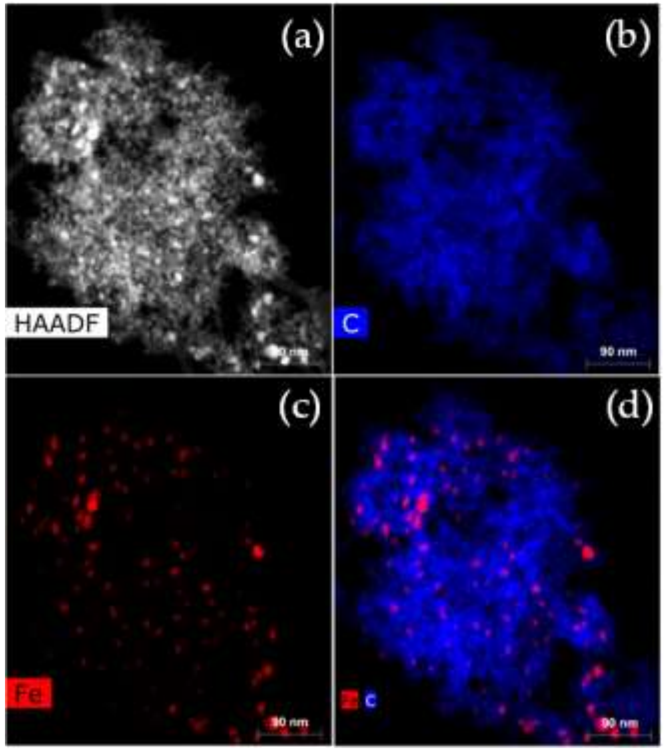
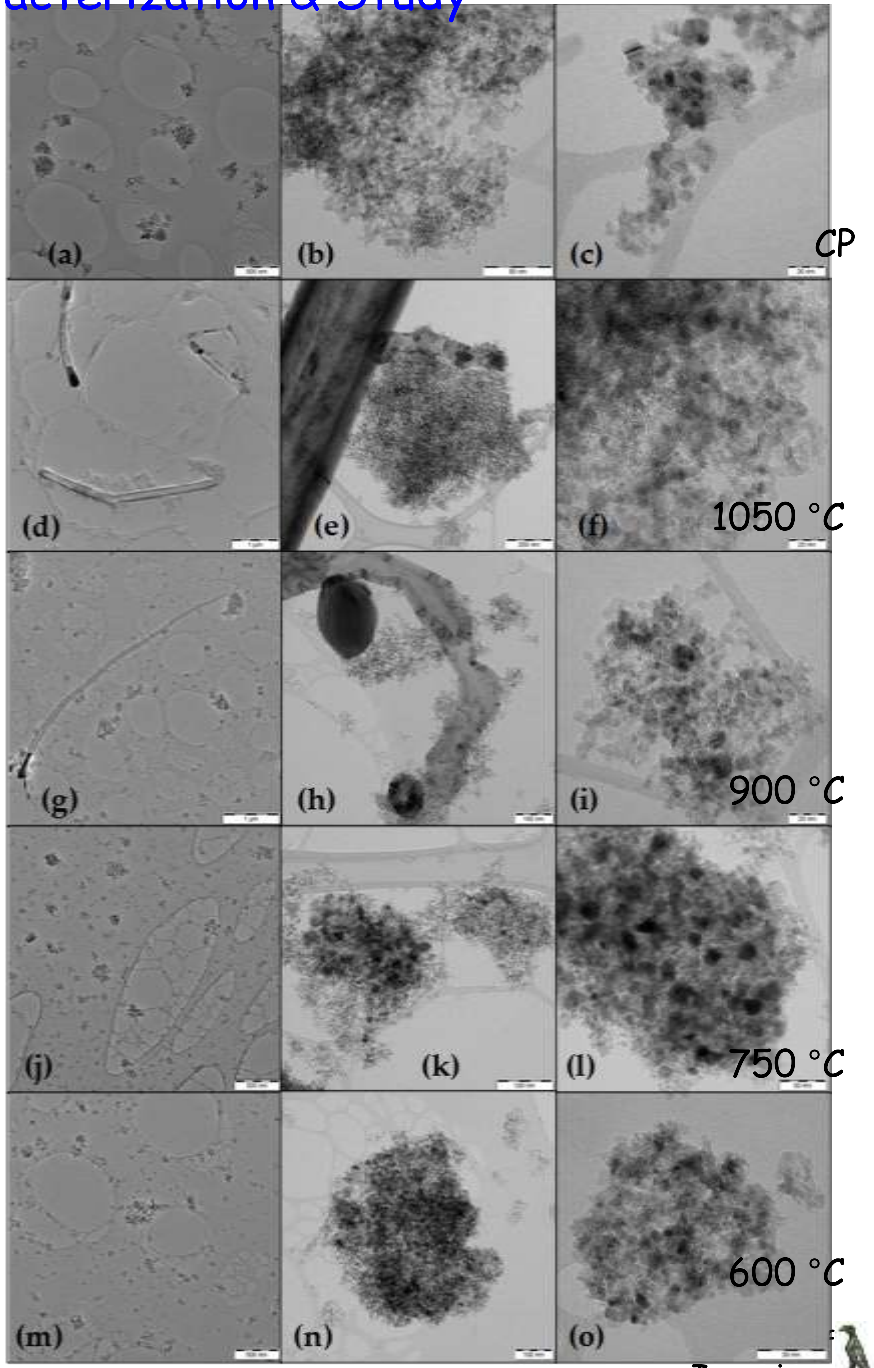



annealing @ 750 °C

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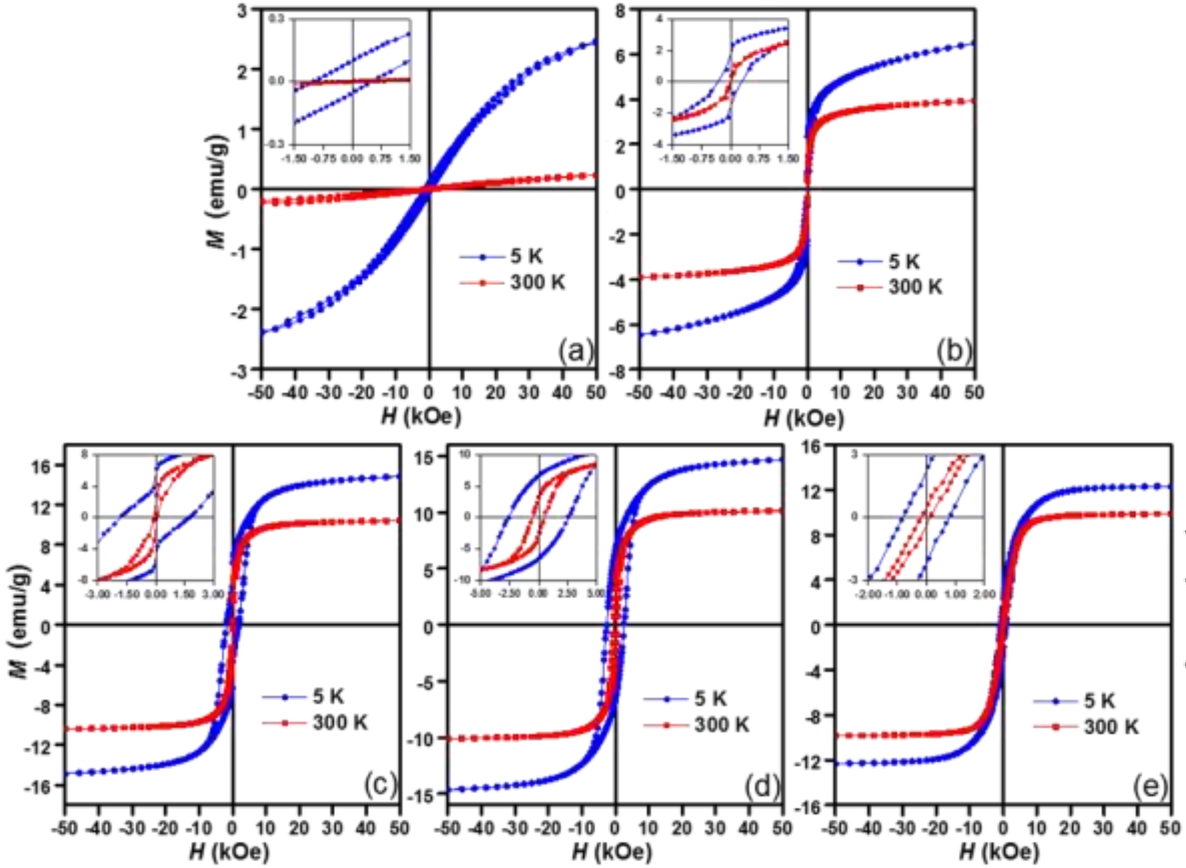


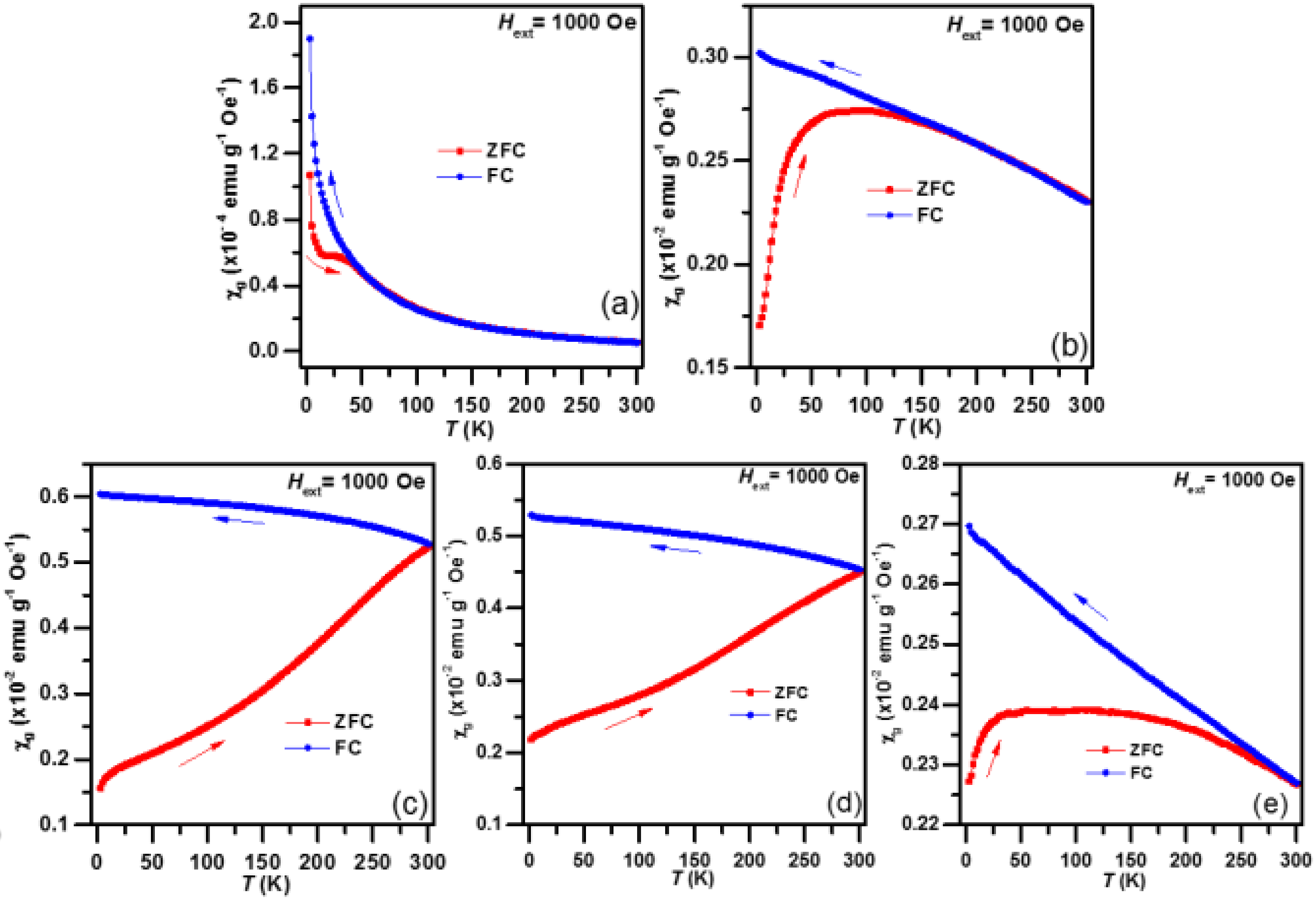


CP (a), NHD-600 (b), NHD-750 (c), NHD-900 (d) and NHD-1050 (e)

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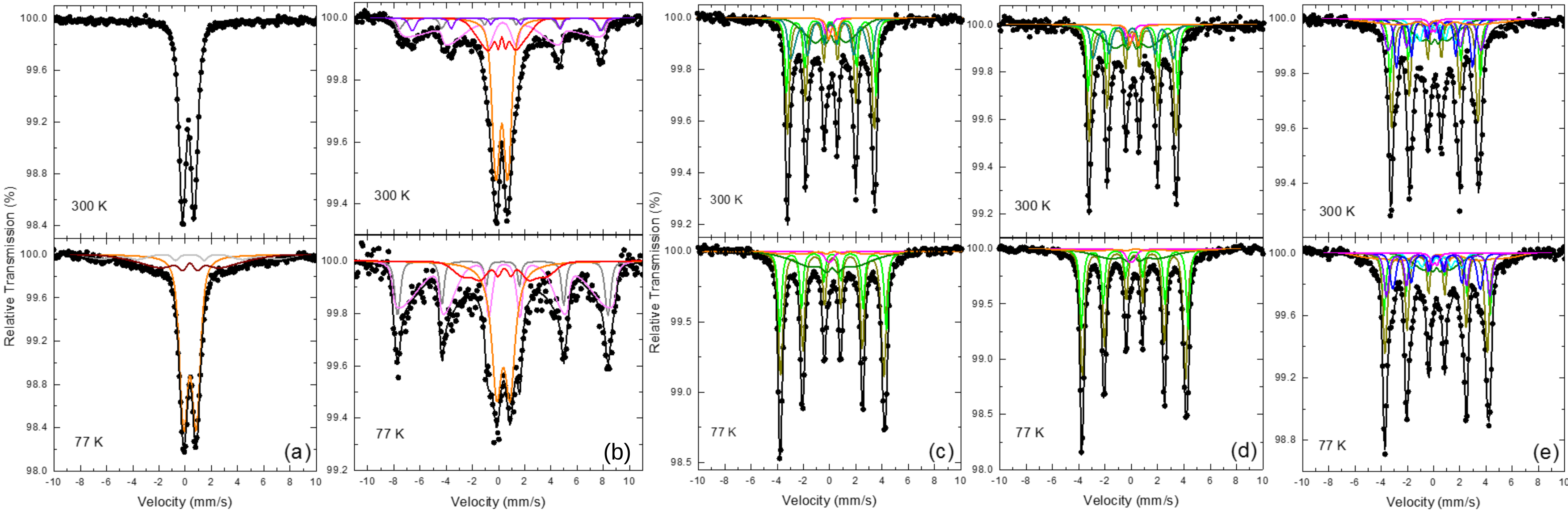



CP (a), NHD-600 (b), NHD-750 (c), NHD-900 (d) and NHD-1050 (e)

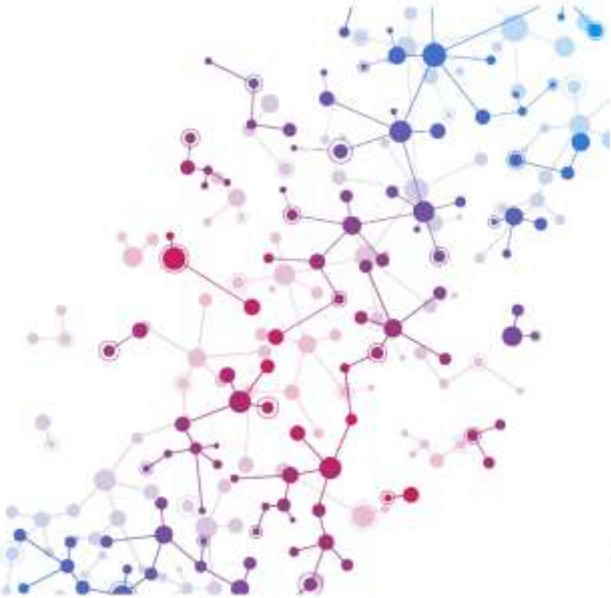
# Mössbauer Spectroscopy & Physics of Materials Laboratory

## Participation in National Research Infrastructure Networks: Innovation-el (https://innovation-el.net/)

innovation-el
Nanotechnology, Advanced Materials & Micro/ Nanoelectronics

# Welcome to Innovation-el

Your Gateway to the Greek Infrastructure Network for Nanotechnology, Advanced Materials and Micro/Nanoelectronics



PARTNERS

DEMOKRITOS

FORTH Institute of Electronic Structure and Laser

THEORETICAL AND PHYSICAL CHEMISTRY INSTITUTE (TPCI)
NATIONAL HELLENIC RESEARCH FOUNDATION (NHRF)

FORTH INSTITUTE OF CHEMICAL ENGINEERING SCIENCES

ElMicLab

University of Ioannina

EIM

## INNOVATION-EL

is an **open-access** large-scale distributed **research infrastructure** of cutting-edge facilities that covers all fronts from materials synthesis, characterization and functionalization to micro-nanofabrication, device/system design, development and testing. The network is complemented by multiscale computer simulations and theory, and is supported by more than **200 skilled scientists** of long-standing expertise and interdisciplinary experience.

## OUR GOAL

is to provide academic, industrial and governmental sectors with **tools and solutions** to achieve **scientific excellence** and develop **high added-value products.** Innovation-el aims at becoming the **innovation ecosystem** par excellence of Southeastern Europe, where the knowledge triangle will boost knowledge-intensive products and services.

# Mössbauer Spectroscopy & Physics of Materials Laboratory

Members
Assoc. Prof. A. Bourlinos
A. Polymeros (PhD)
P. Ziogas (PhD)

Collaborators (Europe)
Y. Pontikes (KU Leuven-Belgium)
R. Zboril (RCPTM Olomouc-Czech Rep.)
J.M.D. Coey-M. Vekatesan (TCD-Ireland)

Collaborators (USA)
M. Kanatzidis (Northwestern)
J. Aitken (Duquense)
G. Hadjipanayis (Delaware)

Collaborators (Ioannina)
Y. Deligiannakis
M. Karakassidis
D. Gournis
S. Hadjikakou
I. Panagiotopoulos

Collaborators (Greece)
P. Trikalitis (UoC)
A. Lappas (IESL Crete)
A. Godelitsas (UoA)
G. Dimitrakopoulos (AUTh)
G. Papavassiliou (Demokritos)