evropský sociální fond v ČR
EVROPSKÁ UNIE
MINISTERSTVO ŠKOLSTVÍ, MLÁDEŽE A TĚLOVÝCHOVY
OP Vzdělávání pro konkurenceschopnost
INVESTICE DO ROZVOJE VZDĚLÁVÁNÍ

Název: Technická specifikace zařízení vhodných pro analýzu magnetických částice

Školitel: Vojtěch Adam

Datum: 5.11.2013

Reg.č.projektu: CZ.1.07/2.4.00/31.0023

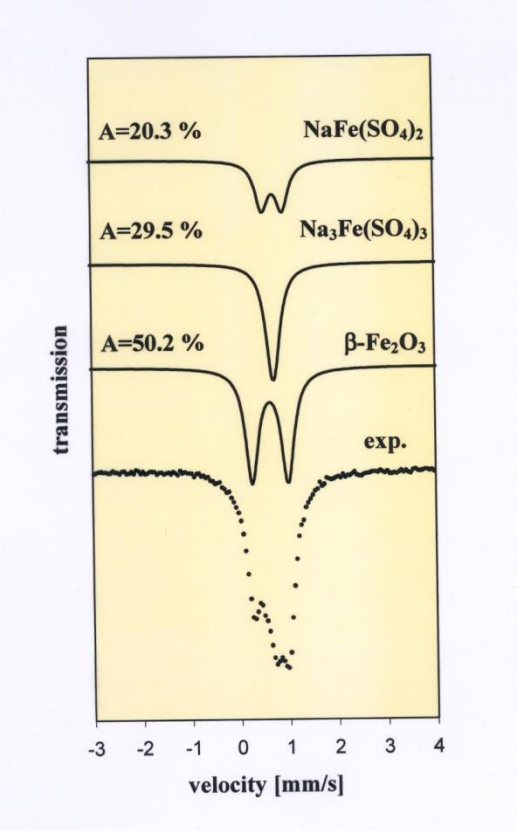
Název projektu: Partnerská síť centra excelentního bionanotechnologického výzkumu

NanoBioMetalNet

1) β-Fe_2O_3 powder prepared by thermally induced solid-state reaction of NaCl and $Fe_2(SO_4)_3$ in air at 400°C

Reaction mechanism

R. Zboril et al: *Chem. Mater.* **14**, 969-982 (2002).



XRD identification of reaction products:

$NaFe(SO_4)_2$ - monoclinic

$Na_3Fe(SO_4)_3$ - hexagonal

β-Fe_2O_3 -cubic

Na_2SO_4 - orthorhombic

ratio of Fe^{3+} ions: 5/3/2 ⇒ molar ratio:
β-Fe_2O_3 / $Na_3Fe(SO_4)_3$ / $NaFe(SO_4)_2$ =5/6/4

$$30NaCl + 10Fe_2(SO_4)_3 + 15/2O_2 \rightarrow$$

$$6Na_3Fe(SO_4)_3 + 4NaFe(SO_4)_2 + 5\beta\text{-}Fe_2O_3 + 4Na_2SO_4 + 15Cl_2$$

Theoretical weight loss:
14.2%

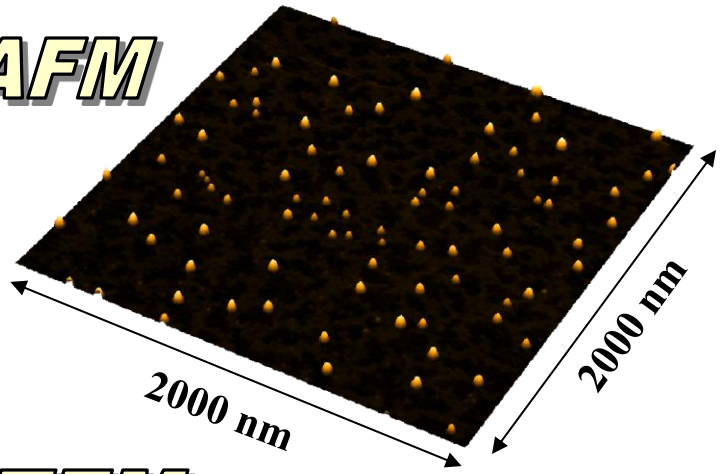
Experimental weight loss from isothermal TG:
14.3% (for initial mixture of $NaCl/Fe_2(SO_4)_3$ =3/1)

RESULTS

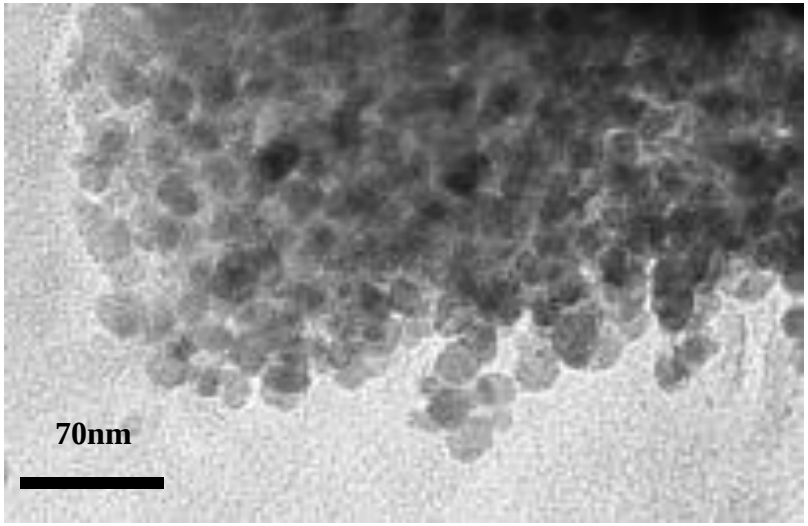
1) β-Fe_2O_3 from solid-state reaction of NaCl and $Fe_2(SO_4)_3$

RESULTS

AFM



TEM

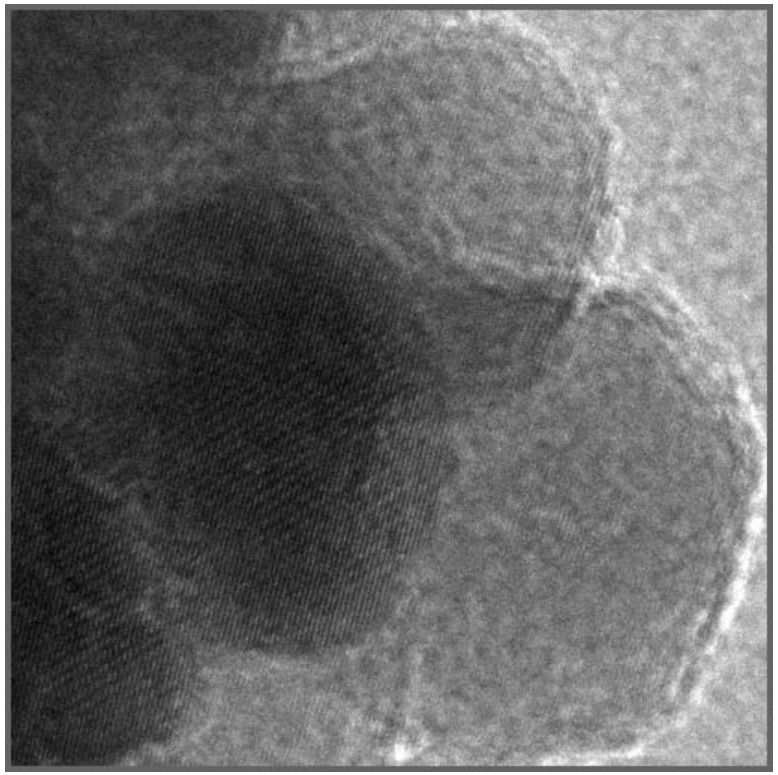


Microscopic observations

AFM vertical dimensions: 14-22 nm

TEM lateral dimensions:15-25 nm

HRTEM



2) Amorphous Fe_2O_3 nanopowder prepared by thermally induced oxidative decomposition of $(NH_4)_4[Fe(CN)_6]$

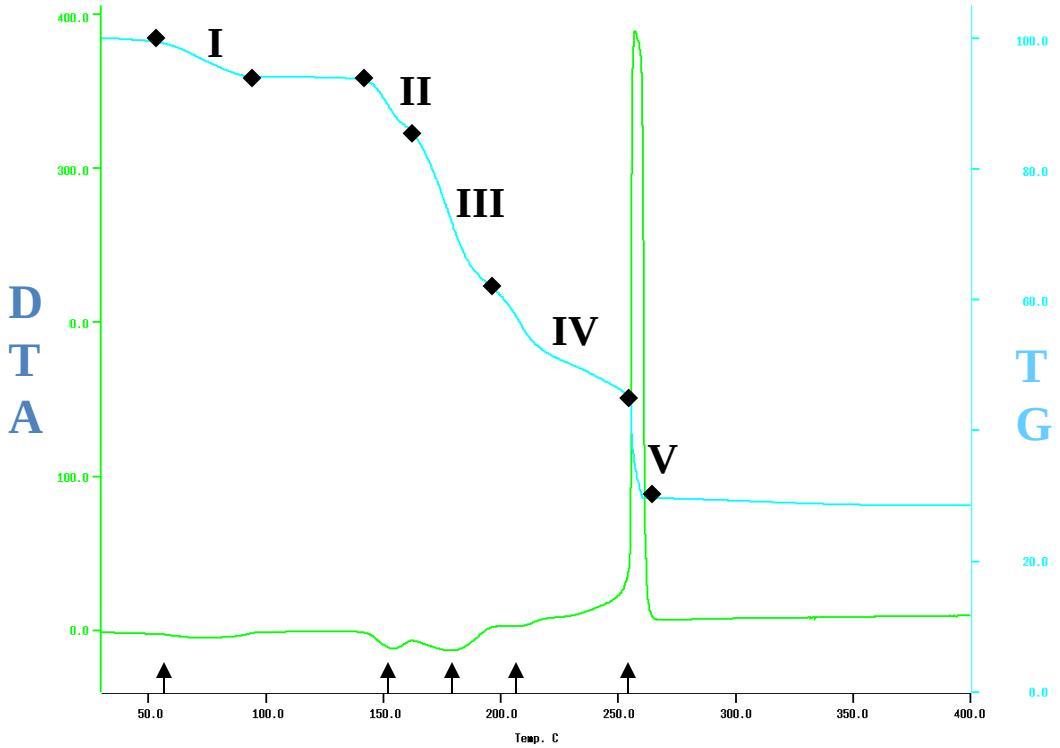
Formation mechanism - TG/DTA

Zboril, et al.: *Crystal Growth & Design* **4,** (2004) 1317.

Five steps

ΔT [°C]	Δm	intermediate
50-100	6.2%	$(NH_4)_3[Fe(CN)_6]$
140-160	9.2%	$(NH_4)_3[Fe(CN)_5]$
160-190	21.9%	$NH_4[Fe(CN)_4]$
190-240	19.3%	$Fe_4[Fe(CN)_6]_3$
240-255	15.1%	Fe_2O_3

$\Sigma\Delta m = 71{,}7\%$



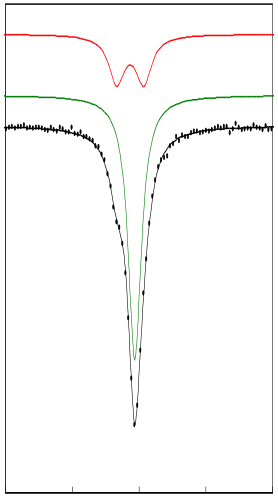
$\Sigma\Delta m_{teor.} = 71{,}9\%$

$$2(NH_4)_4[Fe(CN)_6] + 7/2O_2 \rightarrow Fe_2O_3 + 6\,(CN)_2 + 8NH_3 + 4H_2O$$

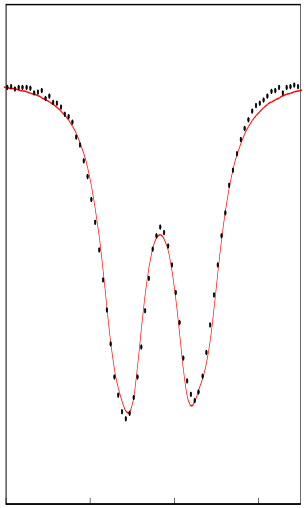
2) Amorphous Fe_2O_3 nanoparticles from $(NH_4)_4[Fe(CN)_6]$

Formation mechanism - RT Mössbauer spectroscopy

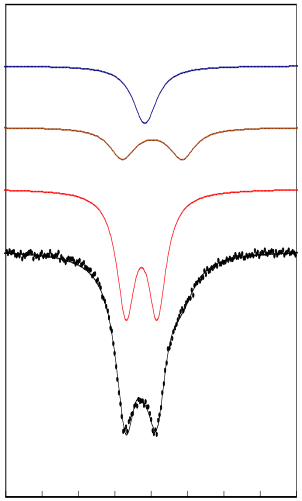
AmF, 40 °C



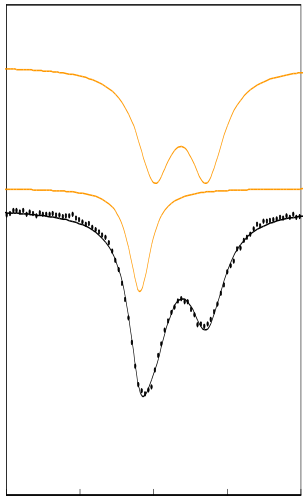
AmF, 100 °C



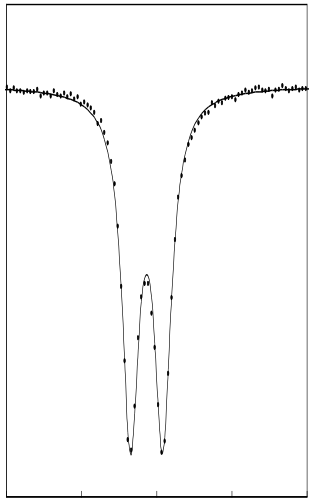
AmF, 160 °C



AmF, 200 °C



AmF, 250 °C



$(NH_4)_4[Fe(CN)_6]$	$(NH_4)_3[Fe(CN)_6]$	$(NH_4)_3[Fe(CN)_5]$	$Fe_4[Fe(CN)_6]_3$	Fe_2O_3
low-spin Fe^{2+}	low-spin Fe^{3+}	low-spin Fe^{2+}	low-spin Fe^{2+}	high-spin Fe^{3+}
δ =-0.02 mm/s	δ = -0.13 mm/s	δ = 0.02 mm/s	δ = -0.04 mms	δ = 0.33 mm/s
	ΔE_Q=0.43 mm/s	ΔE_Q=0.82 mm/s		ΔE_Q=0.80 mms
		$NH_4[Fe(CN)_4]$	high-spin Fe^{3+}	
		low-spin Fe^{3+}	δ = 0.40 mm/s	
		δ = -0.12 mm/s	ΔE_Q=0.69 mm/s	

valence–change mechanism

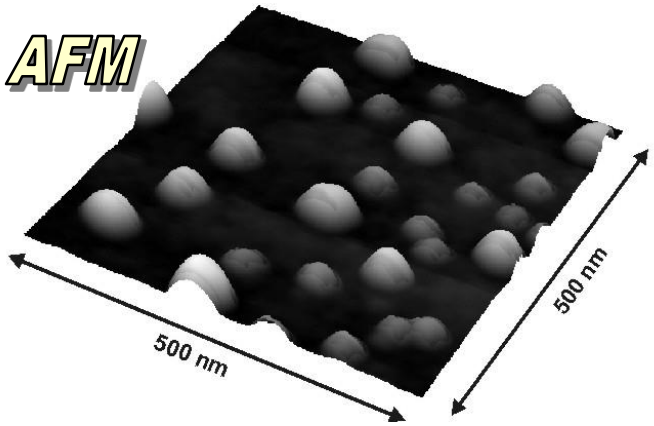
$NH_4^+ \rightarrow NH_3 \Leftrightarrow Fe^{2+} \rightarrow Fe^{3+}$

$2CN^- \rightarrow (CN)_2 \Leftrightarrow Fe^{3+} \rightarrow Fe^{2+}$

$(NH_4)_4[Fe(CN)_6] \rightarrow (NH_4)_3[Fe(CN)_6] \rightarrow (NH_4)_3[Fe(CN)_5] \rightarrow NH_4[Fe(CN)_4] \rightarrow Fe_4[Fe(CN)_6]_3 \rightarrow Fe_2O_3$

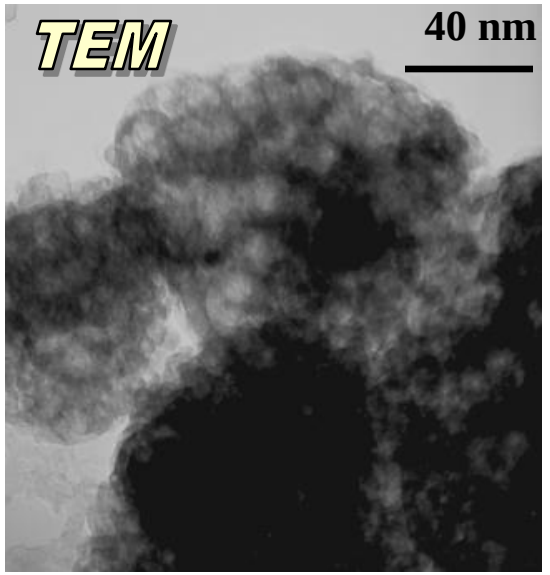
2) Amorphous Fe_2O_3 nanoparticles from $(NH_4)_4[Fe(CN)_6]$

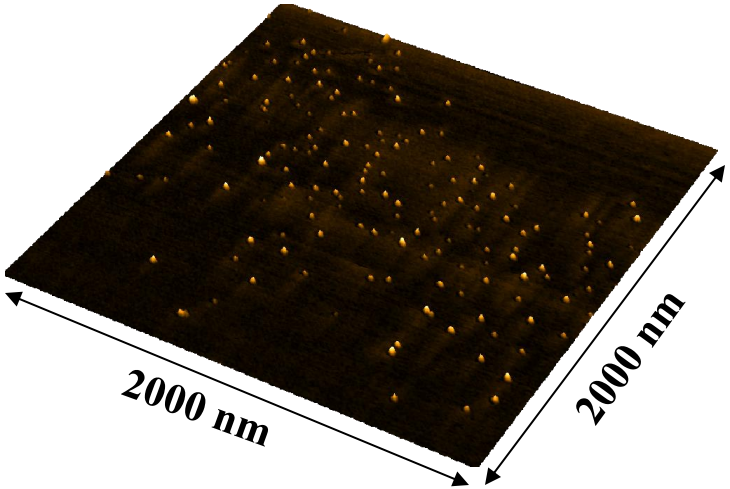
RESULTS



Globular spongelike agglomerates:

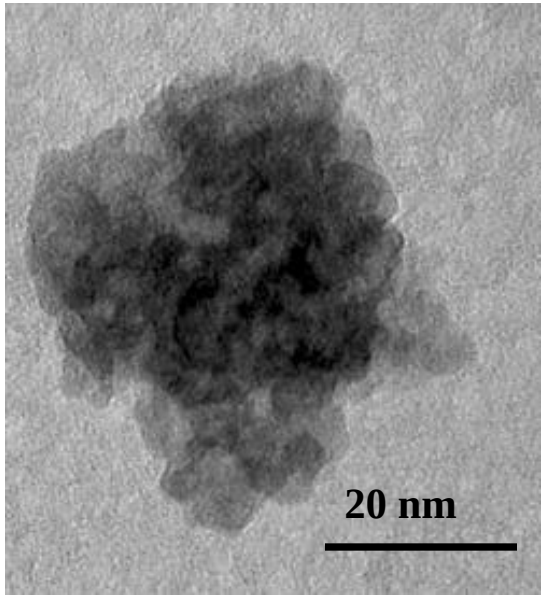
50-150 nm

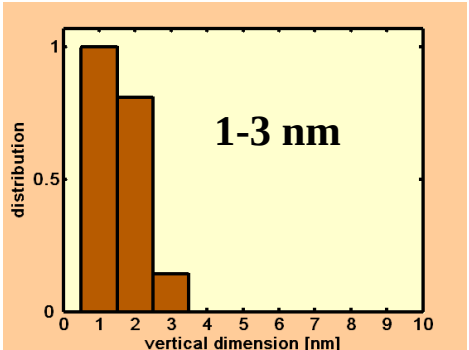


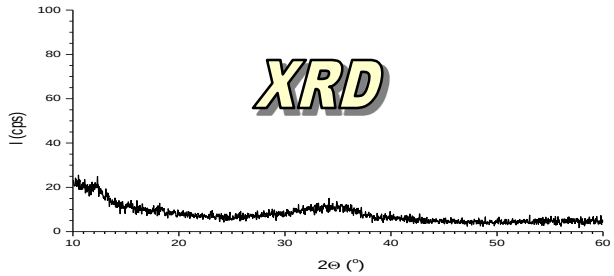


surface area from BET:

415 m^2/g







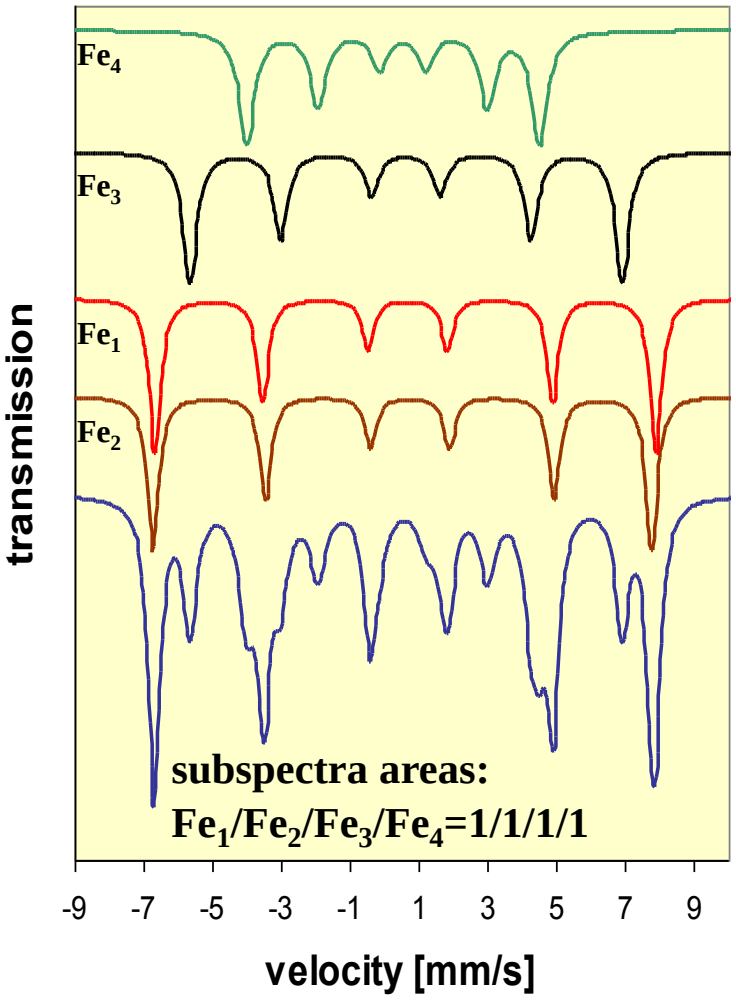
3) ε-Fe_2O_3 from $Fe_2(SO_4)_3$

Zboril et al., *J. Nucl. Radioanal. Chem.* **255** (2003) 413.

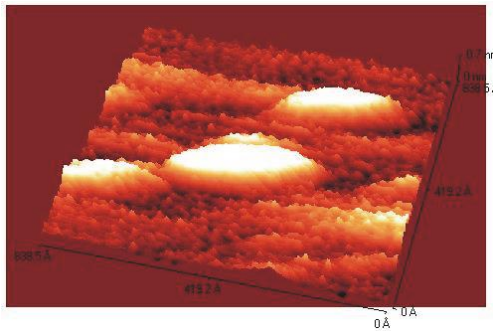
$$Fe_2(SO_4)_3 \xrightarrow{530\ ^\circ C,\ \text{air-limited}} (\beta+\varepsilon)\ Fe_2O_3 + 3SO_3$$

+ postprocessing magnetic separation

300 K Mössbauer spectrum

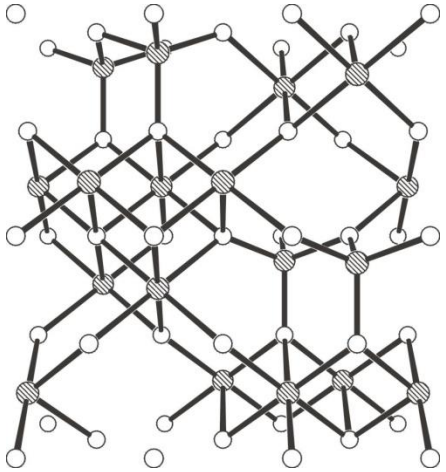


AFM



40-60 nm, elongated particles

Structure



Orthorhombic *Pna*2_1 space group

ferrimagnetic at RT, T_C~480 K

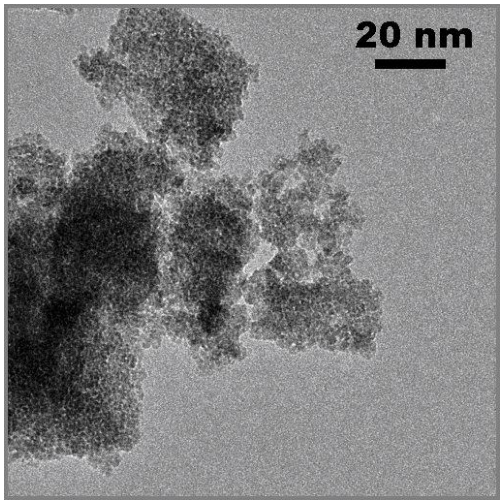
Nanocomposite of ε-Fe_2O_3 in a silica matrix obtained by combining reverse-micelle and sol-gel methods: giant Hc value of 2.0 T at RT

Jin et al. *Advanced Mater.* **16** (2004) 48.

4) Thermal syntheses of Fe_3O_4 and α-Fe nanoparticles embedded in nanocrystalline MgO matrix

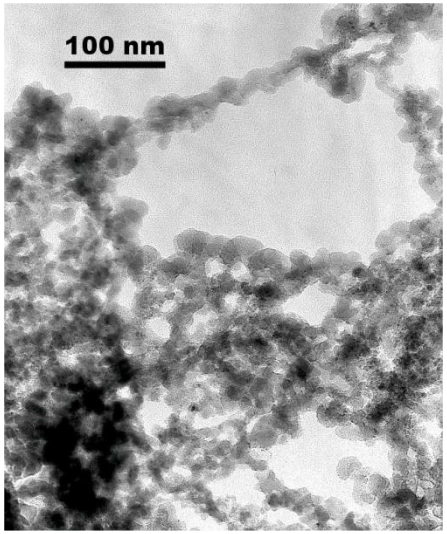
O. Schneeweiss, R. Zboril, N. Pizurova, M. Mashlan, E. Petrovsky, J. Tucek: *Nanotechnology* **17**, 607-616 (2006).

Precursors

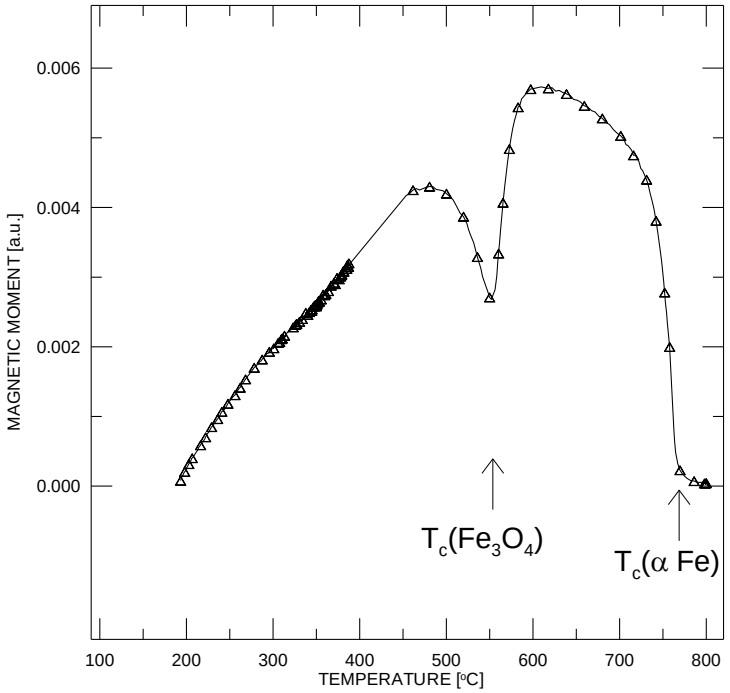


Amorphous iron(III) oxide prepared by solid-state route

Nanocrystalline Mg prepared by spark erosion technique



Thermomagnetic curve

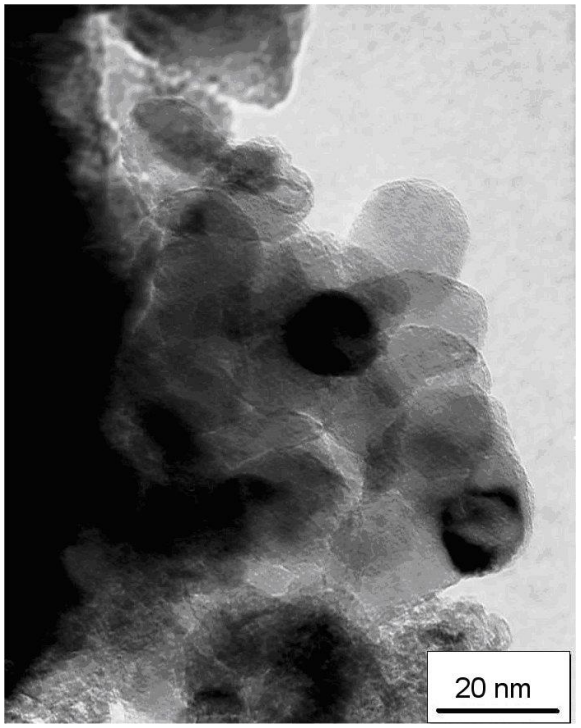


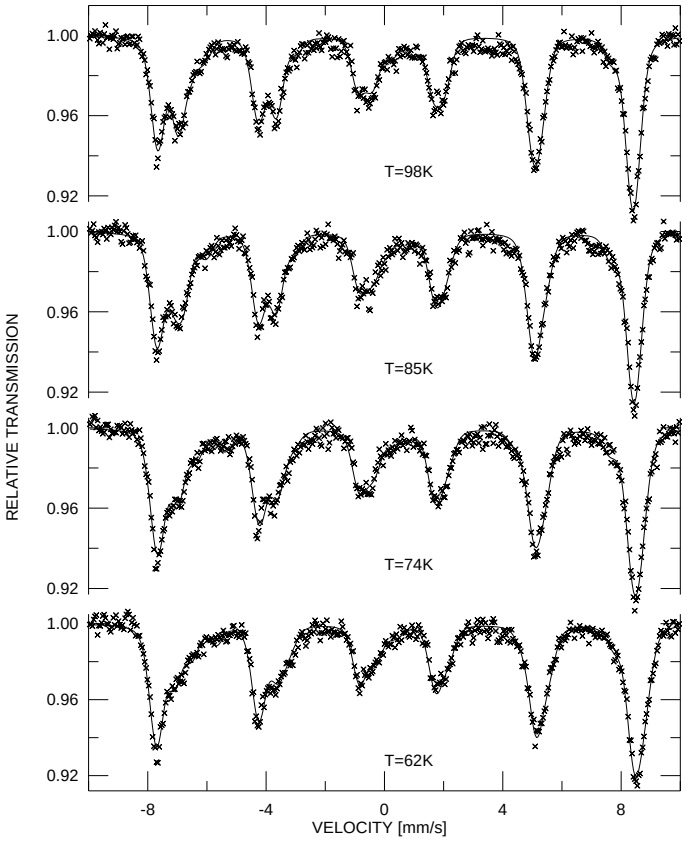
Mixture of precursors heated in hydrogen

4) Thermal syntheses of Fe_3O_4 and α-Fe nanoparticles embedded in nanocrystalline MgO matrix

300°C, hydrogen - magnetite nanoparticles

TEM



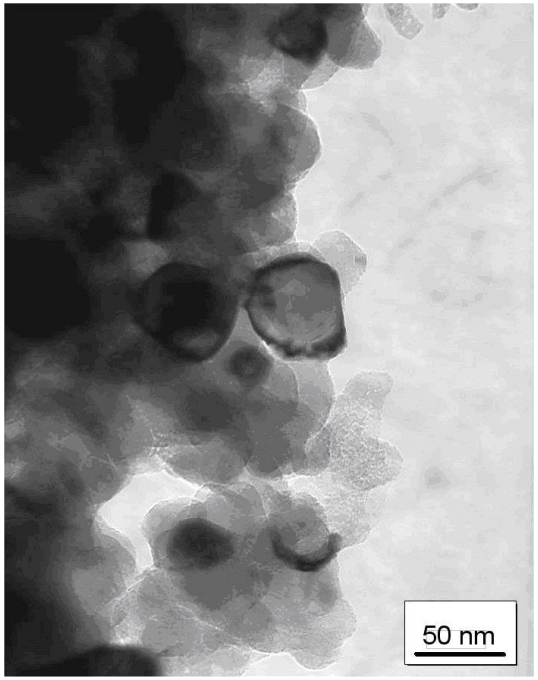


Mössbauer

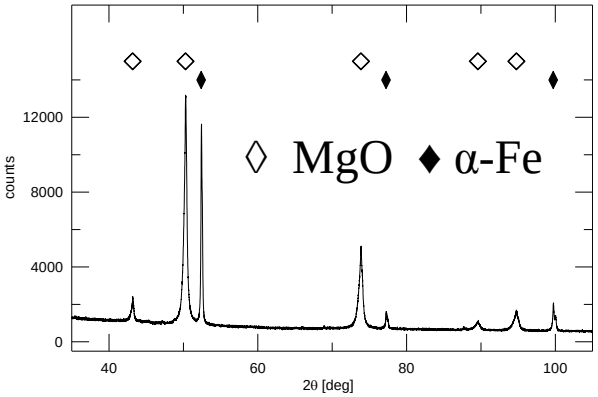
4) Thermal syntheses of Fe_3O_4 and α-Fe nanoparticles embedded in nanocrystalline MgO matrix

600°C, hydrogen - iron nanoparticles

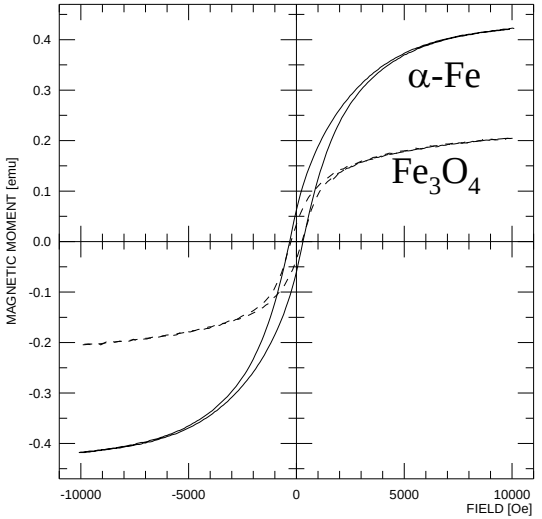
TEM



XRD



Magnetic measurements



Advantages of the used matrix:

- nanocrystalline character, low specific weight, high chemical and thermal stability, non-toxicity, biocompatibility ➡

Application potential of such magnetic nanocomposites:

biomagnetic separations, drug delivery, hyperthermia treatment

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Děkuji Vám za pozornost

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