Fe_3C NANOPOWDER PREPARED BY LASER-INDUCED PYROLYSIS

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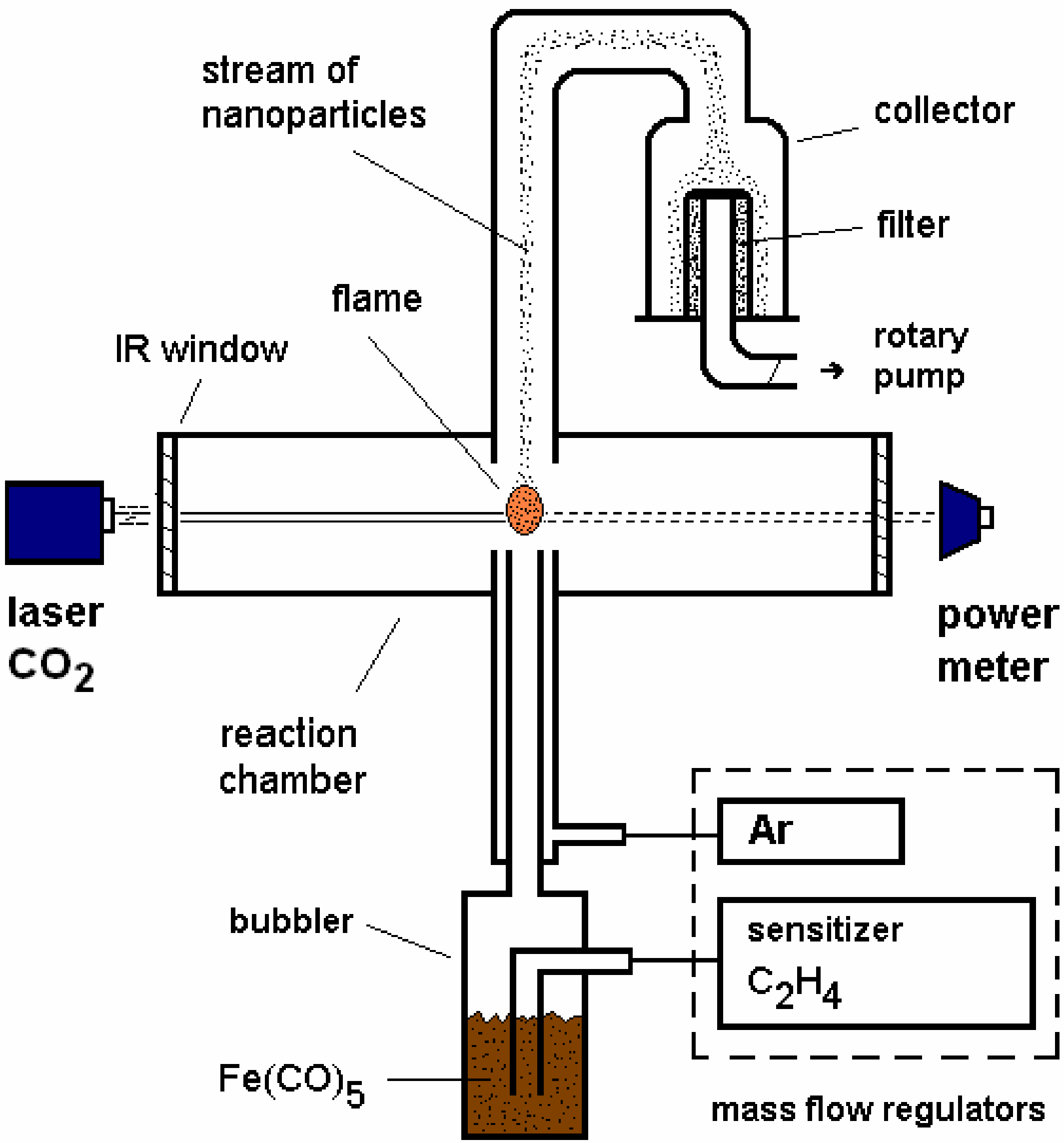


LASER-INDUCED PYROLYSIS OF GASES

J.S. Haggery, W.R. Canon
in Steinfeld (Ed.): Laser-Induced
Chemical Processes,
Plenum Press, New York, 1981

$$C_2H_4 + h\nu \rightarrow C_2H_4^*$$

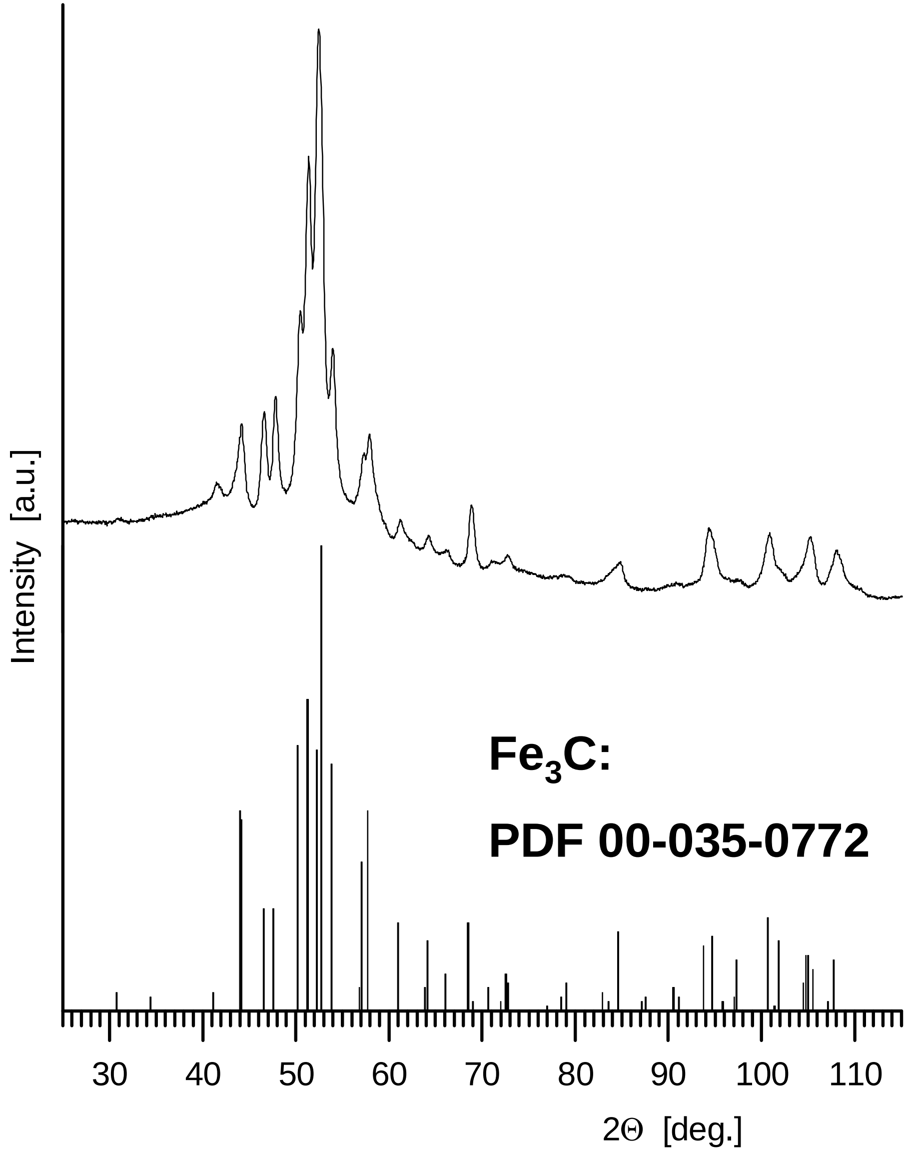
$$Fe(CO)_5\,(l) \rightarrow Fe\,(s) + 5\,CO\,(g)$$

Catalytic decomposition of C_2H_4 on a hot Fe-particle surface

C diffuses into particle interior

Fe_3C (cementite) is formed

Fe_3C NANOPOWDER - XRD



Fe_3C clearly detected:

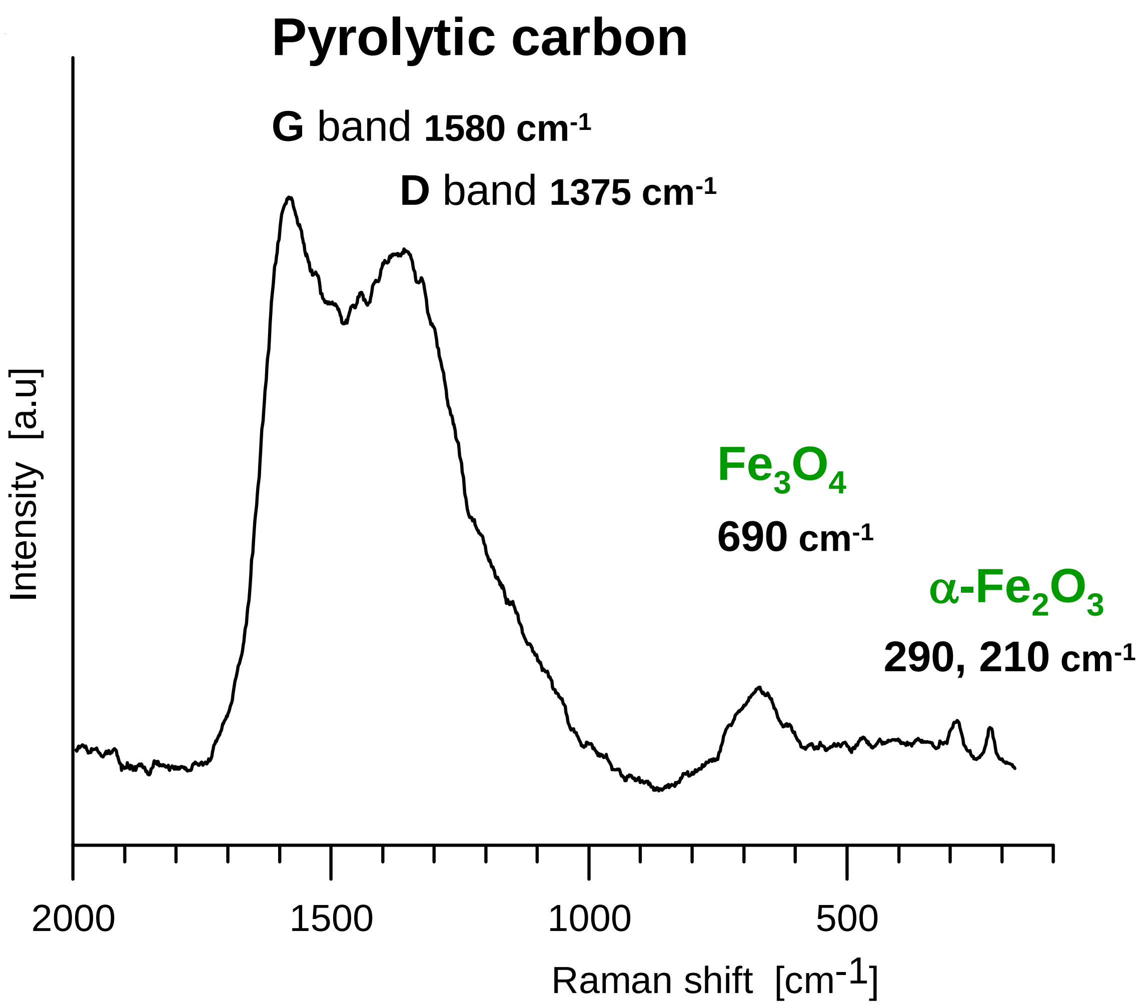
the mean coherence length
<u><L> = 20 nm</u>

The presence of Fe_XO_Y not excluded

due to small particle size !

α-Fe not proved !

Fe_3C NANOPOWDER - Raman spectrum



R.M. Cornell-U. Schwertman
The Iron Oxides
VCH Publishers (1999) p. 135

Fe_3O_4 **676**, 550 cm^{-1}

γ-Fe_2O_3 **740, 650, 252,** 505, 380, 350, 263, 193 cm^{-1}

α-Fe_2O_3 613, 500, **412,** 299, 247, 225 cm^{-1}

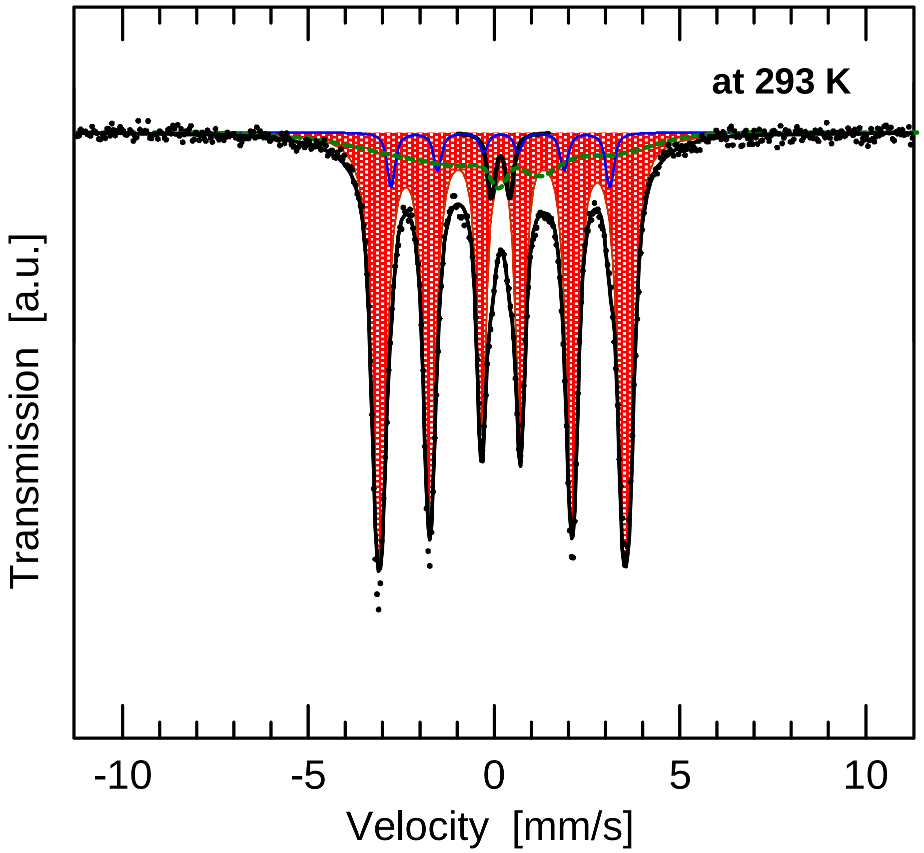
α-FeOOH 560, 470, **385,** 300, 250 cm^{-1}

Fe_3C NANOPOWDER - Mössbauer spectra

Fe_3C superposition 87 r.a.%

Fe_S B_{hf} = 21.2 T ε_Q = 0.02 mm/s δ = 0.21 mm/s 29 r.a.%

Fe_G B_{hf} = 20.3 T ε_Q = 0.02 mm/s δ = 0.21 mm/s 58 r.a.%



Sextet 3 r.a.%

B_{hf} = 18.3 T ε_Q = -0.01 mm/s δ = 0.18 mm/s

Distribution 8 r.a.%

B_{hf} = 15.9 T ε_Q = 0.36 mm/s δ = 0.08 mm/s

ΔB_{hf} = 14.2 T

Doublet 2 r.a.%

ε_Q = 0.25 mm/s δ = 0.18 mm/s

Fe_3C NANOPOWDER - Mössbauer spectra

Fe_3C superposition 84 r.a.%

Fe_S B_{hf} = 25.5 T ε_Q = 0.00 mm/s δ = 0.35 mm/s 30 r.a.%

Fe_G B_{hf} = 24.3 T ε_Q = 0.02 mm/s δ = 0.35 mm/s 54 r.a.%

surface/interface-Fe_3C or

Fe_XC_Y superposition 6 r.a.%

B_{hf} = 21.7 T ε_Q = 0.21 mm/s δ = 0.09 mm/s

B_{hf} = 18.6 T ε_Q = -0.03 mm/s δ = 0.43 mm/s

Fe_3O_4 superposition 11 r.a.%

B_{hf} = 53.7 T ε_Q = 0.08 mm/s δ = 0.37 mm/s

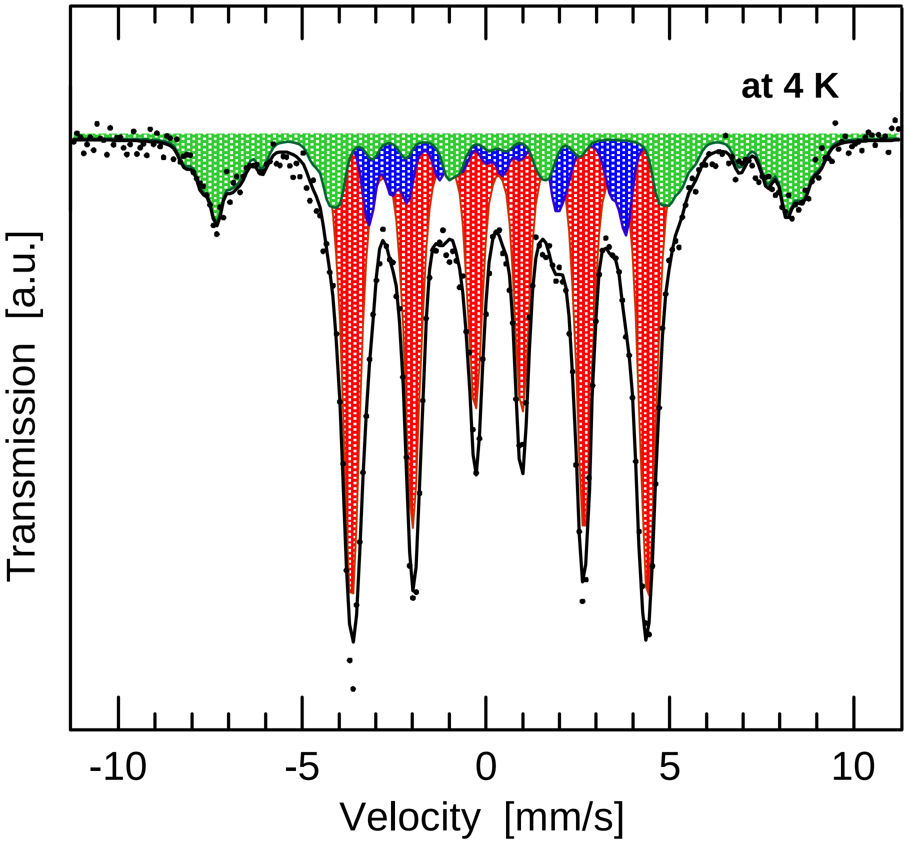
B_{hf} = 48.6 T ε_Q = 0.36 mm/s δ = 0.54 mm/s

B_{hf} = 48.2 T ε_Q = -0.04 mm/s δ = 0.47 mm/s

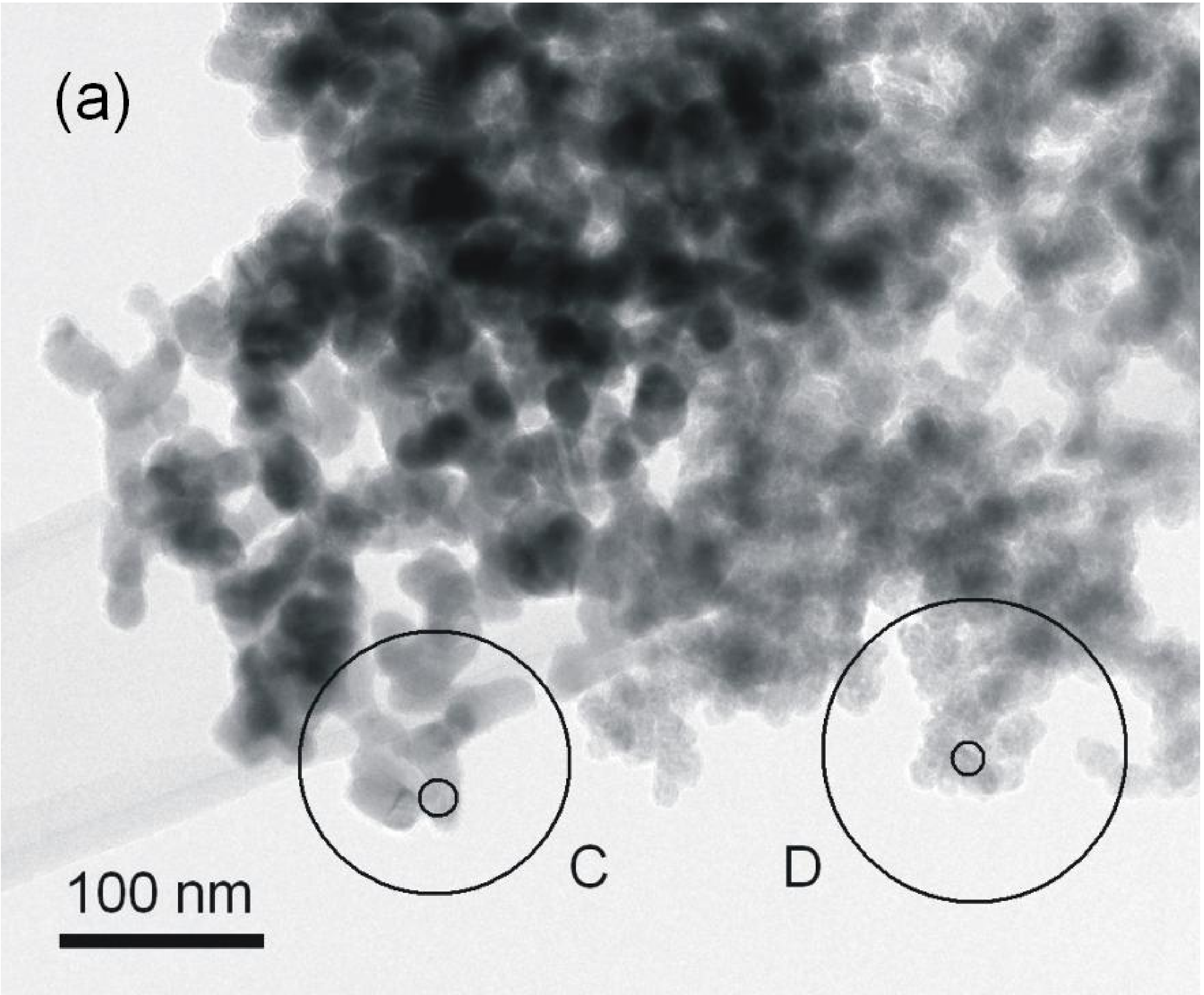
B_{hf} = 47.8 T ε_Q = -0.13 mm/s δ = 0.09 mm/s

B_{hf} = 47.0 T ε_Q = -0.18 mm/s δ = 1.10 mm/s

B_{hf} = 40.4 T ε_Q = -0.58 mm/s δ = 0.99 mm/s



Fe_3C NANOPOWDER - HRTEM



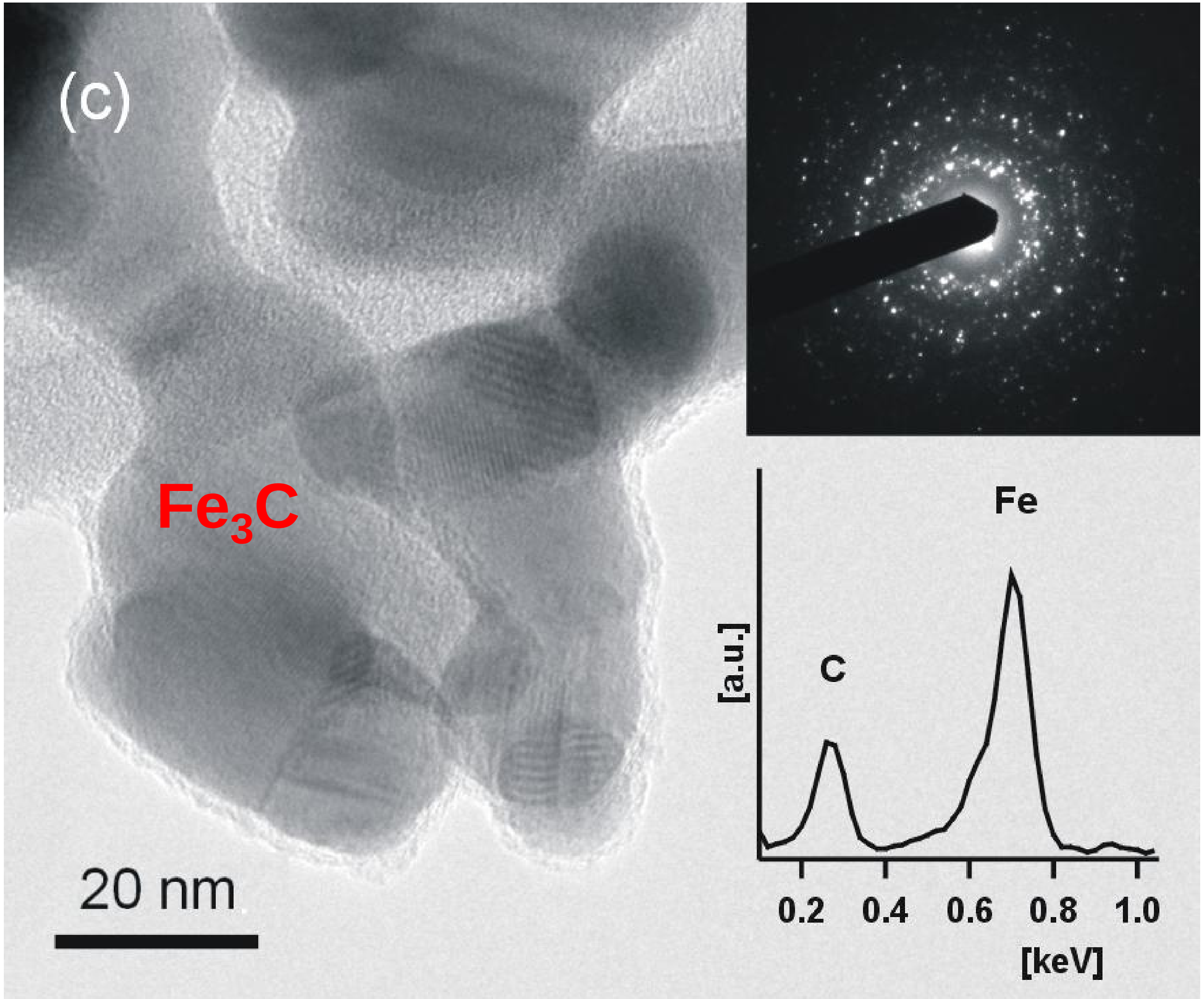
Two different types of nanoparticles observed:

<u>Spot C</u>

crystalline particles

<u>Spot D</u>

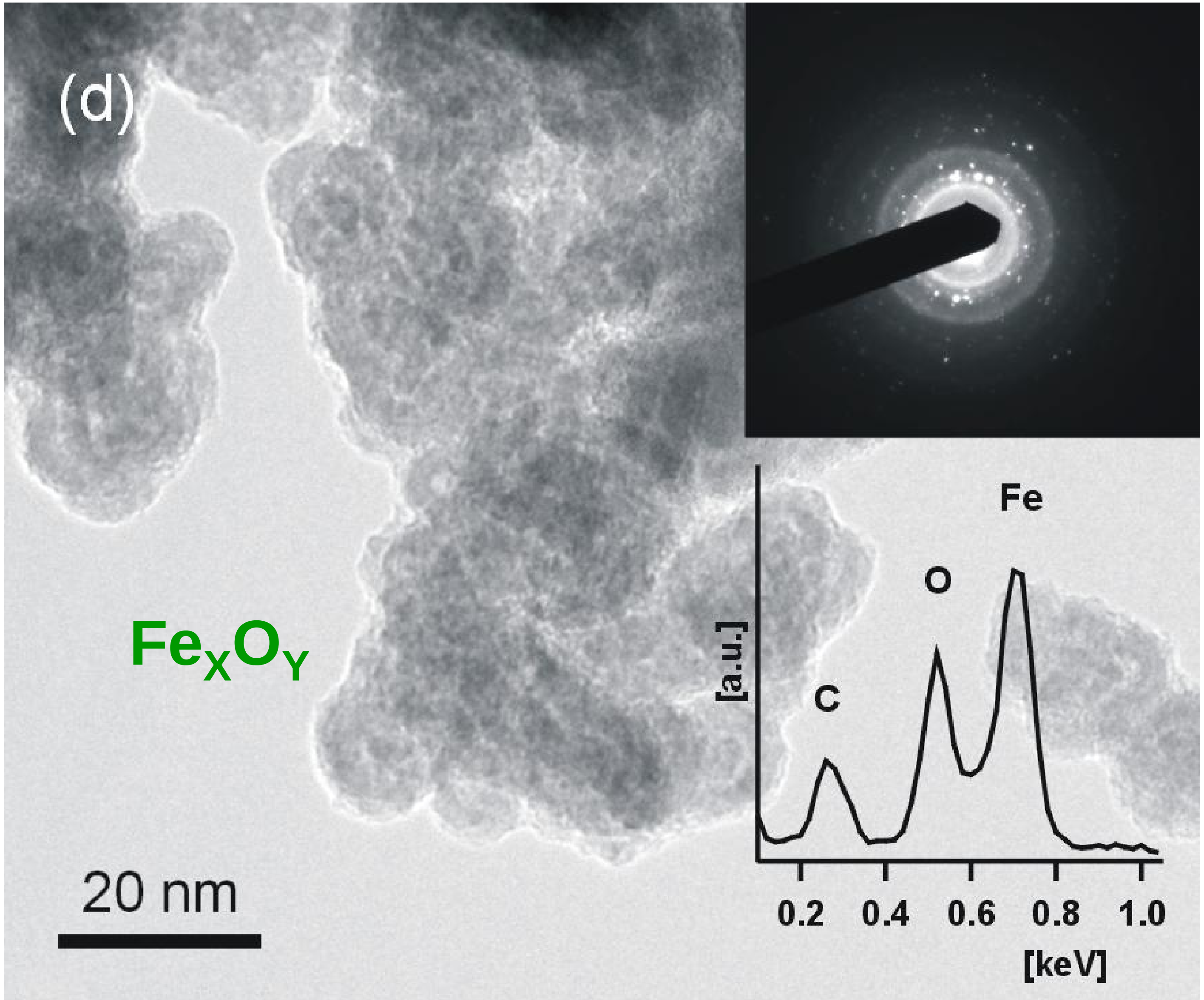
less crystalline



Spot C

crystalline particles

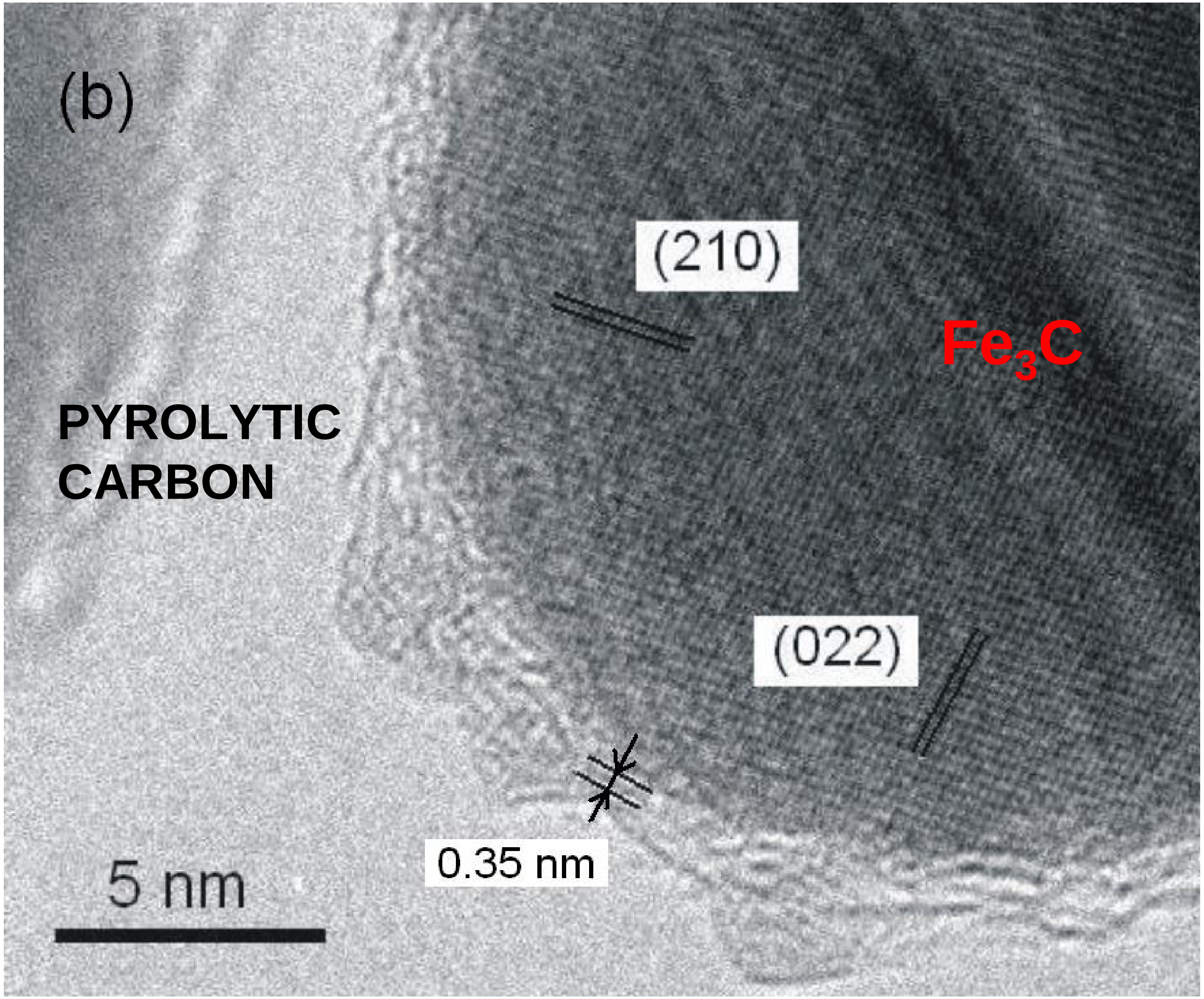
Fe-C



Spot D

less crystalline

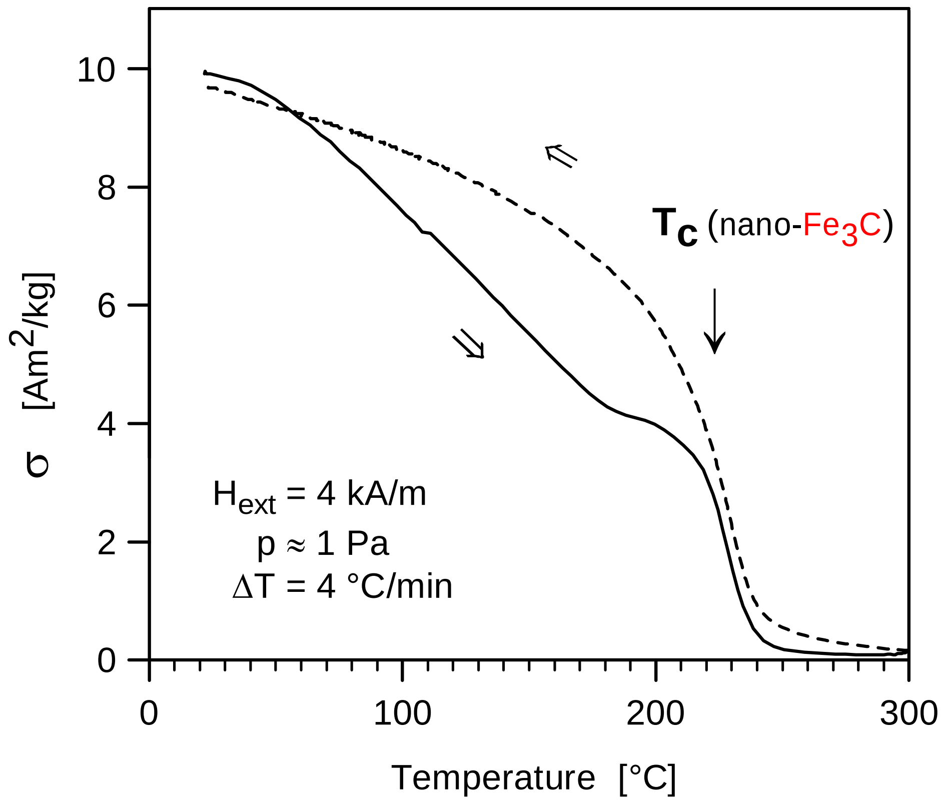
Fe-O



B. David et al.
Surf. Interface Anal.
38 (2006) 482

Fe_3C NANOPOWDER - Magnetic measurements: VSM

T_C(n-Fe_3C) = 227 °C
bulk: 210 °C



Transition

from ferromagnetic state

into paramagnetic state

is smeared out

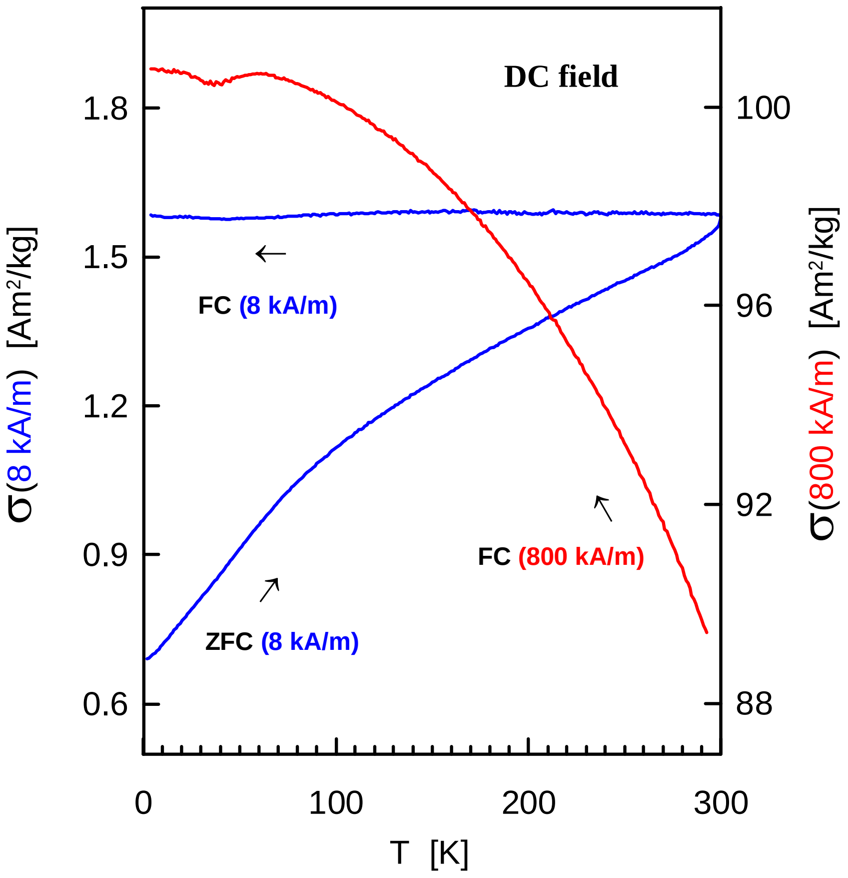
B. David et al.

JMMM 304 (2006) e787

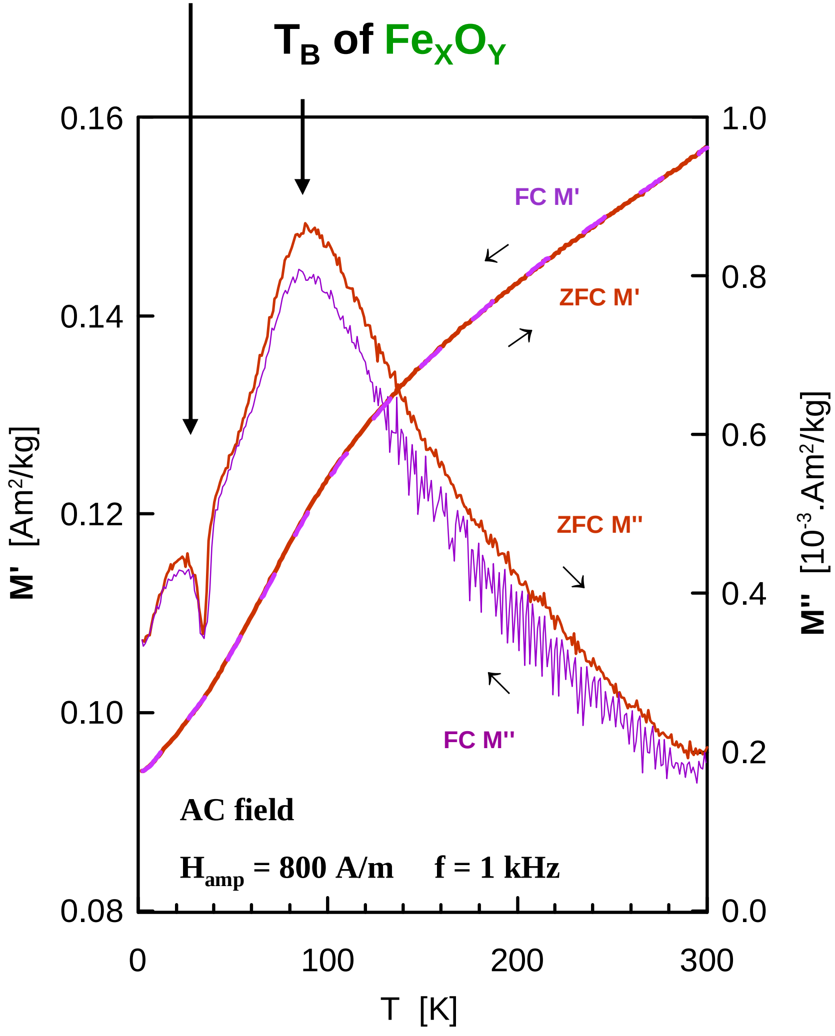
Magnetic measurements: PPMS

Interaction of ferromagnetic particles

=> **superferromagnetic behaviour**



ascribed to Fe_3O_4 (?!)



SUMMARY

I. Synthesized **Fe_3C** nanopowder:

- according to <u>XRD and TEM</u>: particle size of **Fe_3C** is **Ø < 30 nm**
- <u>Mössbauer analysis</u>:

  Fe_3C contains cca 84 % of all Fe atoms

  Fe_3O_Y contains cca 11 % of all Fe atoms

II. It is difficult to obtain pure single phase **Fe_3C**:

- some synthesized small **α-Fe** nanoparticles have not reacted with **C_2H_4**

  ⇒ were not covered by carbon ⇒ oxidized and so **Fe_XO_Y** formed
- **<u>the synthesis parameters must be optimized</u>**

III. Magnetic properties of **Fe_3C** nanopowder:

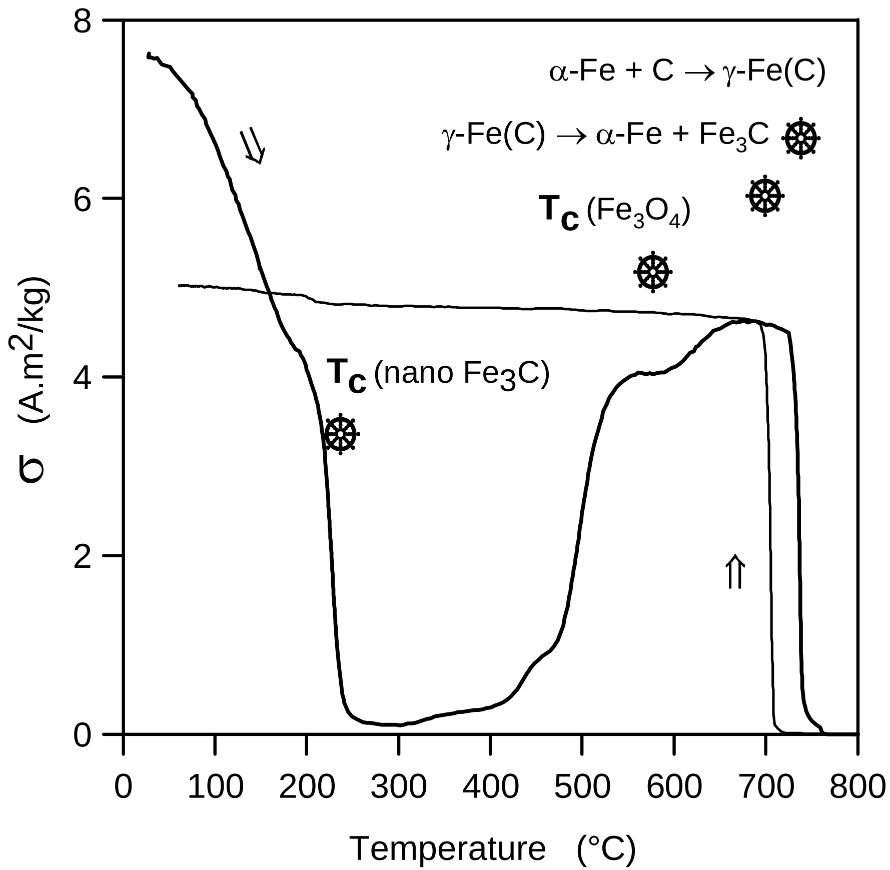
- values of **H_C(n-Fe_3C)** and **σ_S(n-Fe_3C)**

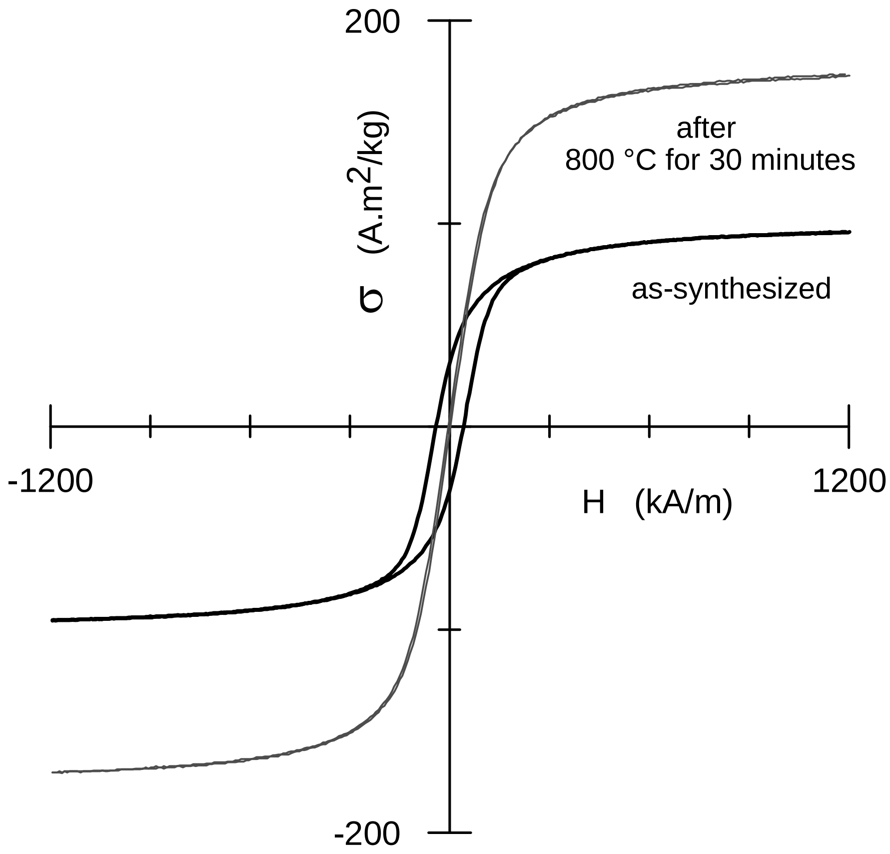
  <u>can be even higher if single phase non-agregated particles are obtained</u>

Fe_3C NANOPOWDER - Magnetic measurements

H_C(n-Fe_3C) = 42 kA/m bulk: ?

σ_S(n-Fe_3C) = 96 Am^2/kg bulk: 130 Am^2/kg





Fe_3C NANOPOWDER - TEM