Magnetic Properties of Magnetite Nano-Particles Manufactured by Reverse Micelles

J. í Hjøllum [1,2], L.T. Kuhn [1], M. B. Madsen [2], E. E. Carpenter [3], E. Johnson [2], K. Bechgaard [1]

[1] Risø national Laboratory, DK-4000 Roskilde, Denmark, [2] The Niels Bohr Intitute, Copenhagen University, [13] Virginia Commonwealth University, US.

Abstract

We present a study of magnetite manufactured by the reverse micellar method. In particular we study the stoichiometry of the manufactured magnetite by Mössbauer spectroscopy and the size distribution of the particles by TEM imaging.

Mössbauer spectra

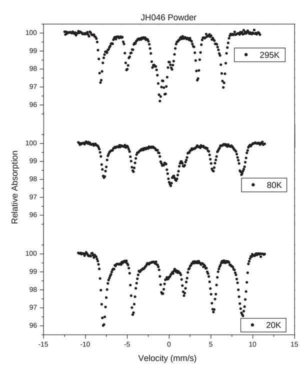

Figure 1: *Mössbauer spectra as a function of temperature of the sample 1 Powder.*

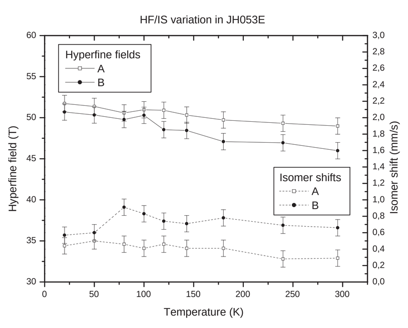

Figure 2: *Hyperfine field and isomer shift of the A and B magnetite components as function of temperature in sample 2E. Notice the shift in hyperfine field and the isomer shift tendencies in the area of 100-118 K. The hyperfine field error bars are 1 T and the isomer shift error bars are 0.1 mm/s. These error values are estimated.*

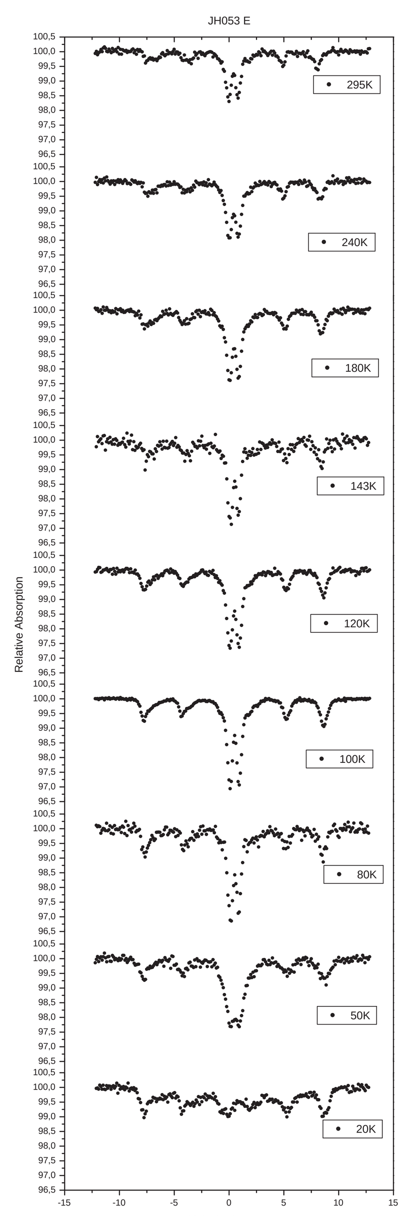

Figure 3: *Mössbauer spectra as a function of temperature of the sample 2E.*

Size distributions

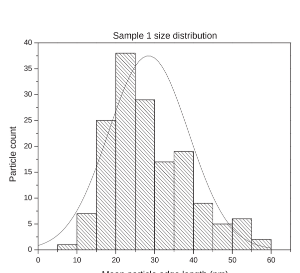

Figure 4: *Particle size distribution of sample 1 as determined at 27.5k magnification.*

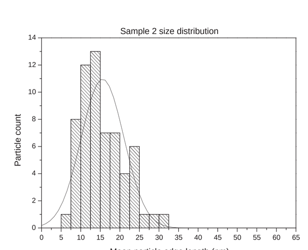

Figure 5: *Particle size distribution of sample 2 as determined at 250k, 400k and 600k magnifications.*

Sample #	Magn.	N	$\bar{d}$ nm	q nm	σ nm
Sample 1	27.5k	158	28.4	25.9	10.3
Sample 2	250-600k	61	15.6	13.9	5.5

Figure 6: *Size distribution data measured from TEM images of sample 1. $\bar{d}$ is mean particle size, q is the median and σ is the standard deviation. The sample 2 data is obtained at magnifications 250k, 400k and 600k*

Lattice distance

Magn.	N	$\bar{d}$ nm	q_d nm	σ_d nm
250k,400k,600k	6	0.24	0.24	0.005

Figure 7: *Measurement of the lattice distance of particles of sample 2D. $\bar{d}$ is the mean value, σ_d is the standard deviation and q_d is the median.*

TEM Images of sample 1

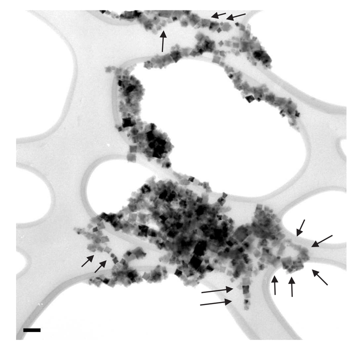

Figure 8: *The magnification is 27.5k. The bar is 100 nm.*

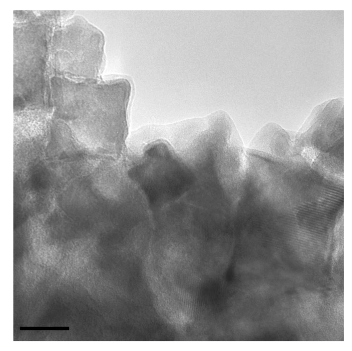

Figure 9: *The magnification is 390k. The bar is 20 nm.*

TEM Images of sample 2

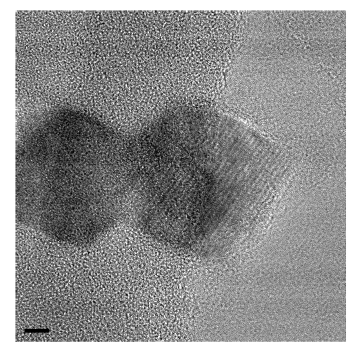

Figure 10: *The magnification is 600k. The bar is 5 nm.*

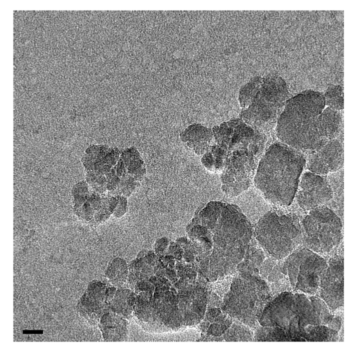

Figure 11: *The magnification is 250k. The bar is 10 nm.*

Particle shapes

The particle shape of magnetite depends on the pH in which it is synthesized[1], and many of the particles appear quadratic on the TEM images.

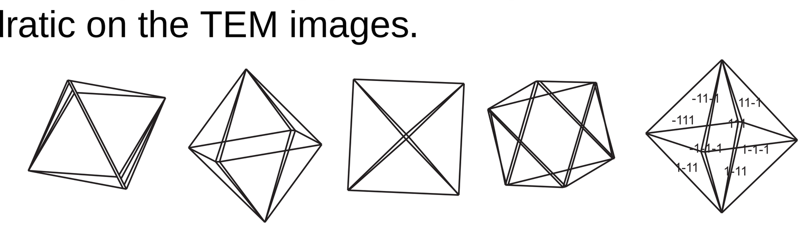

Figure 12: *An octahedral crystal, here magnetite, in various orientations. Constructed using an applet on webmineral.com [2]*

Ratio of edge lengths

The relative deviation from the quadratic form is:

$$e = \frac{z_1 - z_2}{\frac{(z_1+z_2)}{2}} \qquad (1)$$

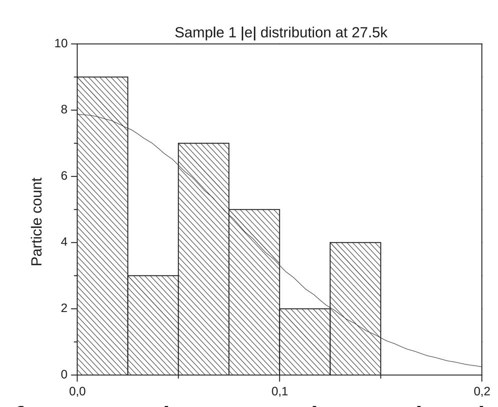

Figure 13: *Distribution of $|e|$ for sample 1 as determined at 27.5k magnification. The normal distribution in this graph has a mean of 0, and a σ which has been constructed from the added datasets $|e|$ and $-|e|$. From the data we conclude that the particles are quadratic.*

Ratio of diagonal lengths

The particles edge corners have right angles, if the diagonals have equal lengths. The relative deviation from equal diagonals (d_1, d_2) lengths:

$$f = \frac{d_1 - d_2}{\frac{(d_1+d_2)}{2}} \qquad (2)$$

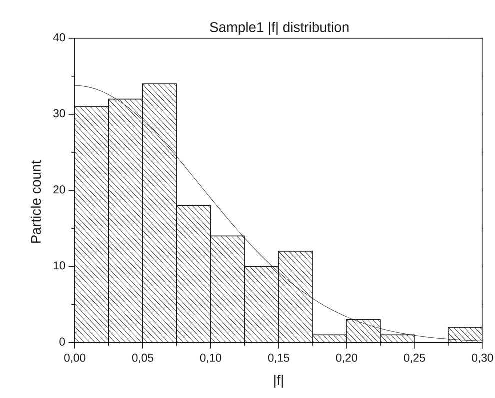

Figure 14: *Distribution of |f| for sample 1 as determined at 27.5 k magnification. The normal distribution with the same mean and standard deviation is displayed for comparison. The data indicate some asymmetry. However, we have arrived at the conclusion that the particles are octahedra and that the deviations from the quadratic form in the images, is due to randomness in particle orientation.*

Particle size control

The samples sample 1 and sample 2 are practically identical, except the parameter ω.
The ratio, a, between the ω's of the samples is

$$a = \frac{\omega_{Sample1}}{\omega_{Sample2}} = \frac{11.22}{5.95} = 1.9 \pm 0.2\,, \qquad (3)$$

the ratio, b, between the mean particle sizes of the samples is

$$b = \frac{\bar{d}_{Sample1}}{\bar{d}_{Sample2}} = \frac{28.4\,nm}{15.6\,nm} = 1.8 \pm 0.2\,, \qquad (4)$$

and the ratio, c, between the medians of the particle size of the samples is

$$c = \frac{q_{Sample1}}{q_{Sample2}} = \frac{25.9\,nm}{13.9\,nm} = 1.9 \pm 0.2\,, \qquad (5)$$

where we have estimated the uncertainty values.

Conclusion

- We have implemented the reverse micellar technique to manufacture materials of a predetermined composition and size.
- Synthesis using the AOT/iso-octane system has in all cases results in either maghemite or magnetite.
- We have shown that particle size control is possible, and that it is dependent on the water to surfactant ratio, ω.
- We found that sample 1 consists of octahedral particles, and sample 2 contains a wide variety of shapes. The particle shape can most likely be attributed to the pH value of the synthesis solution[1].
- We have manufactured one sample which is close to stoichiometry, which shows clear signs of the Verwey transition.
- We have found the magnetic hyperfine field of the iron oxides in the samples to be lower than that of bulk due to canting of the surface spins.

Contact

Do not hesitate to contact me if you have suggestions, comments etc.

My data:
Jari í Hjøllum
Building 227, AFM, Risø National Laboratory
Fredriksborgvej 399
4000 Roskilde

Email : jari.hjoellum@risoe.dk

References

[1] R. M. Cornell and U. Schwertmann. *The Iron Oxides, Structure, Properties, Occurrence and Uses.* VCH Verlagsgesellschaft, Weinheim (Federal Republic of Germany), VCH Publishers, New York, NY (USA), 1996.

[2] Webmineral.com. Mineralogy database (www.webmineral.com). The internet.

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