13.5–5 NEUTRON DIFFRACTION STUDY OF DOMAINS IN SMALL Fe_3O_4 PARTICLES. By H. Boysen, Institut für Kristallographie und Mineralogie der Universität München, E. Schmidbauer, Institut für Allgemeine und Angewandte Geophysik der Universität München, Theresienstr.41, 8000 München 2, E. Steichele, Fakultät für Physik der Technischen Universität München, E 21, 8046 Garching, FRG.

The magnetic structure of small, spherical ferro- or ferrimagnetic particles slightly above the true single domain size is characterized according to theory by the formation of a single domain wall in the center, which may cover an essential part of the particle. In a two domain sphere, presumably, two domains are formed which are separated by one 180° domain wall. The critical diameter D of a Fe_3O_4 sphere in the true single domain state appears to be $D_{cr} \approx 30$ nm, the domain wall width for bulk Fe_3O_4 is ≈ 150 nm. For a spherical Fe_3O_4 particle of D ≈ 200 nm one may expect a large fraction of the volume to be part of the domain wall, in which a spiral-type structure of the atomic magnetic moments should exist. The features of such magnetic structures should be visible in magnetic Bragg peaks by high resolution neutron diffraction.

The Fe_3O_4 spheres were prepared by reduction of commercially available $\alpha - Fe_2O_3$ grains in a H_2/H_2O gas atmosphere at ≈ 400° C. An analysis of the resulting Fe_3O_4 grains by SEM techniques showed rather spherical grains with a mean size of ≈ 230 nm. The Rietveld analysis of a diffraction pattern from a conventional two-axes-powder diffractometer gave structural results, which agreed quite well with those from a standard Fe_3O_4 sample: u = 0.3801(2), M(A) = $4.0(1)\mu_B$, M(B) = $3.8(1)\mu_B$. From the broadening of the reflexions a particle size between 300 Å and 1300 Å and indications of internal strains could be estimated. Moreover some reflexions showed additional weak wings and the peak-to-background ratio was lower by about 20% due to some kind of disorder in the small particle sample.

Line profiles and particle size broadening were studied in more detail with a high resolution Time-of-Flight diffractometer (E. Steichele and P. Arnold, Phys.Lett.(1973) 44A,165), which gives a resolution $\Delta d/d \approx 6 \times 10^{-4}$ for the low order (111) and 2×10^{-4} for the higher order (444) reflexions. The mainly magnetic (111) reflexion shows a clear broadening by a factor 7 compared to the standard Fe_3O_4 sample; the (444) peak, which is mainly nuclear, is broadened by a factor 1.3. However, these and all other peaks in between, magnetic and nuclear, lie on a common straight line in a $\Delta d - d^2$ - plot, from which we derive a coherently scattering block size of 950 ± 50 Å. From the line profiles we cannot conclude the existence of Bloch walls with long periodically ordered spin rotation. From the particle size result we find that the spherical grains are split up into crystalline domains of about half the sphere size and that there are no magnetically ordered domains smaller than that (≈ 950 Å). Because of the crystalline subdivision of the particles no conclusions can be drawn from these measurements about the maximum magnetic domain sizes. Our results appear to be in contradiction to the simple concept of one 180° wall in a single particle.

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