STRUCTURAL METHODS FOR STUDYING NANOCRYSTALLINE MATERIALS

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Nanocrystalline materials can be produced by various methods such as rapidly quenching, mechanical alloying, sputterig etc. The magnetic properties of such materials depend crucially on their actual magnetic micro structure, whose detailed knowledge hence is required to understand and to interprete their magnetic behaviour. The most essential parameter in this context is the mean size D of the nanocrystalline grains. In the case of soft magnetic nanocrystalline materials the coercivity scales with D^6. For hard-magnetic nanocrystalline materials the exchange length l_{ex} which is proportional to $(A/K)^{1/2}$, where A is the exchange energy and K the anisotropy constant, becomes comparable to the grain size thus causing an enhancement of remanence (i.e. $M_r/M_s > 0{,}5$).
Various methods exist for determination of the grain size: X-ray diffraction line profile analysis and neutron small-angle scattering (SANS) allow to determine the mean size of the grains, whereas tranmsission electron microscopy (TEM) allows to directly oberve local grain size distributions. From the angular broadening $\Delta\beta$ of X-ray diffraction peaks one can infer the mean size δ of the crystallites ("grains") from the relation

$$\delta = K\lambda / \Delta\beta\cos\theta \ ,$$

where the constant K~0.9-1 depends the crystallite shape, λ is the X-ray wavelength and θ the Bragg angle. Here the main problem is that also local stresses cause line broadening which is not easy to take into account during data evaluation and hence may be the origin of systematic errors.
Neutron small-angle scattering (SANS) is a less indirect method for the determination of the mean grain size of such samples. It allows to cover a broad range of momentum transfers and hence size intervals in real space just by varying the distance between sample and (2-dim.) detector array and by choosing an appropriate neutron wavelenth. Usually the individual measurements taken at different sample-detector distances and/or neutron wave lengths are normalized by comparison with a reference scatterer (H_2O, 1 mm) with known macroscopic scattering cross-section ($\Sigma \sim 1$ cm^{-1}). Magnetic materials have to be aligned by applying an external magnetic field in order to allow for a separation of nuclear and magnetic scattering contributions. This turns out to be a problem for hard-magnetic materials which require rather high fields for saturation. For small-angle scattering by non-interacting "particles" with given scattering length density difference $\Delta\rho$ with respect to the surrounding uniform medium (i.e., an amorphous soft-magnetic matrix) each of the two cross-section contributions can be written as

$$\frac{d\Sigma}{d\Omega}(Q) \propto (\Delta\rho)^2 \int_0^\infty K_n(r)\, r^6 P(Qr)\, dr \, .$$

There $K_n(r)$ is the number of particles with size r, and $P(Qr)$ is the normalized form factor of a particle of size r (radius). Evidently the size distribution can only be determined if assumptions about the particle shape are made, or vice versa, the shape can only be inferred for known size distribution. Needless to say, that for interacting particles with possible correlations between their distance and their shape such a simple relation no longer holds and data evaluation becomes appreciably more complex.