MS37 O1

X-ray Microdiffraction on Individual Self-Assembled Nanostructures

C.Mocuta[1], B.Krause[1], R.Mundboth[1,2], T.H.Metzger[1], J.Stangl[2], G.Bauer[2], I.Vartanyants[3], C.Deneke[4], O.G.Schmidt[4] [1]European Synchrotron Radiation Facility (ESRF), Grenoble-France. [2] *Johannes Kepler University, Linz-Austria.* [3] *Hasylab at Desy, Hamburg-Germany.*[4]*Max-Planck-Instiutut fürFestköperforschung, Stuttgart-Germany.*

X-ray diffraction is a powerful non-destructive tool for the analysis of strain fields and chemical composition of nanostructures. In standard diffraction, ensembles of objects are characterized yielding averaged, statistical properties. It is of high interest to measure the structural properties of individual sub-micron sized objects in order to understand the change in physical properties, when the nanoscale is approached. To this end a new microdiffraction setup was developed and is now available on ID-01 beamline at ESRF.

In first experiments we investigated one-dimensional (wires, tubes) and zero-dimensional (dots) objects by diffraction techniques of individual objects in a focused x-ray beam. We will describe the way to find individual self-assembled objects on a macroscopic flat substrate and show microdiffraction results on single rolled-up semiconductor nanotubes [Krause *et al.*, Phys. Rev. Lett. 96 165502 (2006)], following the same tube from the part attached to the substrate to its freestanding part. It is demonstrated that the lattice parameter distribution and strain can be measured and modeled using elastic theory.

In a second example we will show a similar approach for micron-sized SiGe pyramidal islands on Si(001) grown by Liquid Phase Epitaxy. From the experimental data on particular individual objects and using mathematical modeling (Finite Element Methods), the variation of structural parameters such as strain, composition and shape was measured from island to island. Complementary microscopy investigation was performed on the very same objects measured in diffraction.

This approach shows some limitation of standard "ensemble average" diffraction methods, and opens up the possibility of combining x-ray microdiffraction technique with other micro-probe experiments on the same individual objects.

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Mössbauer Spectroscopy of Monodisperse Iron Oxide Nanoparticles.

I.S. Lyubutin. *Shubnikov Institute of Crystallography, RAS, Moscow 119333, RUSSIA.*
E-mail: lyubutin@ns.crys.ras.ru

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The ^{57}Fe-Mossbauer spectroscopy is extremely well suited for studies of nanostructured iron oxides. The technique is not restricted to studies of well-crystalline materials, but is applicable irrespective of crystal size and crystallinity. It can give information not only on static properties – such as phase composition, crystal structure, magnetic properties, valence states – but also on dynamic properties, such as electron hopping, superparamagnenic relaxation, diffusion, vibrations, etc. The Mossbauer means of characterizing the various steps of the preparation process, the as-prepared nanostructured materials, and their evolution during various treatments, can be successfully applied in nanotechnology. Mossbauer spectroscopy can give most valuable information about superparamagnetic behavior of iron oxide nanoparticles. Determination of magnetic moment, frequency, and thermal fluctuations permits one to evaluate particle volume by means of the formula $t = t_0\exp(KV/kT)$, where t and t_0 are times of magnetic moment relaxation, V is particle volume, and K is magnetic anisotropy. Temperature variations of Mossbauer spectra can be described by distribution of static magnetic hyperfine fields, and the Mossbauer blocking temperature T_b can be evaluated. The blocking temperature is an effective measure of the superparamagnetic energy barrier, which is given by the product of KV. Nanoparticles have a large fraction of the atoms at the surface. When the particle size is lower than 10 nm in diameter, the structure and properties of the surface is a challenge, and Mossbauer spectroscopy can give selective information about the inner part and surface properties of the nanoparticle. The Mossbauer blocking temperature has been observed to be very sensitive to surface characteristics of the particles. It is well known that, in small magnetic particles, surface and strain contributions to K dominate, producing magnetic anisotropy densities two orders of magnitude higher than the magnetocrystalline anisotropy of the corresponding bulk material. Therefore, Mossbauer spectroscopy can often give useful information about surface effects. Several examples of successful applications of the ^{57}Fe Mossbauer spectroscopy to investigation of magnetic, structural and oxidation states of iron ions in monodisperse iron oxide nanoparticles will be given in this report. One of the important problems in the iron-oxide particles preparation process is to identify and select the wüstite FeO, hematite α-Fe_2O_3, magnetite Fe_3O_4 and maghemite γ-Fe_2O_3 phases. Whereas wüstite has the cubic structure and hematite has the corundum-type crystal structure, both magnetite and magnetite have the spinel-type structure and thus can not be distinguish by X-ray technique. It will be shown in the report that the hyperfine parameters of Mossbauer spectra can be very helpful to resolve this important problem of nanotechnology.

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Nano-structured thin films characterized by high-temperature X-ray diffraction

J. Keckes *Erich Schmid Institute, Austrian Academy of Sciences and Department of Materialphysik, University of Leoben, Leoben, Austria*
E-mail: keckes@unileoben.ac.at

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Nano-structured thin films with characteristic length scales on the order of few to tens of nanometers provide an immense potential for the design of microeletronic devices, sensors based on mutli-functional surfaces and new tools with unprecedented wear resistance. In comparison with bulks, the nanocrystalline films exhibit a variety of new qualities like extraordinary strength and very high flow stress. For the practical application of those films, it is very important to characterize their behaviour at high temperature in order to assess structural changes