MS37 O1

X-ray Microdiffraction on Individual Self-Assembled Nanostructures
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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.

MS37 O2

Mössbauer Spectroscopy of Monodisperse Iron Oxide Nanoparticles.
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Keywords: iron oxide nanoparticles, Mössbauer spectroscopy, surface effects

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.

MS37 O3

Nano-structured thin films characterized by high-temperature X-ray diffraction
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Keywords: thin film analysis, stress analysis, in-situ temperature diffraction

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

caused by thermal cycling like elastic and plastic deformation of film and substrate, recrystallisation, phase transformation, annealing of defects [1,2]. The purpose of this contribution is to present a new complex high-temperature X-ray diffraction (XRD) approach which can be used to *in-situ* characterize structural changes occurring in thin film structures during thermal cycling. The novelty of the approach resides in the characterization of a free standing thin film-substrate composite which can bend freely in the high-temperature chamber (DHS900, Anton Paar GmbH). This gives an opportunity to characterize the substrate curvature by measuring substrate symmetrical reflections at different sample positions [3]. In this way, the macroscopic stress imposed on the film can be correlated with other structural parameters like elastic strain, size of coherently diffracting domains, point defect density. The approach provides thus an opportunity to perform a complex thermo-mechanical and structural characterization of films. By comparing measured stress and strains, absolute magnitude of temperature dependent X-ray and mechanical elastic constants can be determined. In the case of multilayered coatings, a comparison of the macroscopic stresses imposed on the whole film composite with the elastic strain behaviour of individual sublayers can be used to study thermo-mechanical effects in complex thin films structures. The new approach was applied to a variety of thin film systems *e.g.* TiN, CrN/Cr, Al, Cu, CrN on Si(100) measured using laboratory and synchrotron sources (BESSY and Hasylab) in the temperature range -100 to 550°C [4]. In the case metal thin films with the thickness down to 50 nm, the approach was used to determine flow stresses which exceed 800 MPa in Al. Moreover, first high-temperature X-ray elastic constants of textured Cu, Al and TiN thin films were evaluated. In the case of hard CrN films with an average crystallite size of about 10 nm deposited on Si(100) and steel, the new approach allowed for the calculation of exact XECs and quantify intrinsic and extrinsic stresses. Also other examples of high-temperature XRD analysis of thin films will be provided.

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MS37 O4

Structural and Compositional Investigation of Yttrium-doped HfO_2 Nano-films

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Keywords: anomalous scattering, epitaxial structures, synchrotron radiation

Cubic phase yttrium-doped HfO_2 (YDH) ultra-thin films were grown epitaxially on GaAs (001) and Si (111) substrates by molecular beam epitaxy (MBE). The C-V and I-V measurements of a film of 7.7 nm thick yield an enhanced dielectric constant ~32 and thus an equivalent oxide thickness ~0.94 nm, close to the theoretical value of cubic phase HfO_2. Thorough structural and morphological investigations by x-ray scattering and transmission electron microscopy reveal the YDH thin films are epitaxially grown on the substrates. The interfaces between YDH and these substrates are atomistic sharp and free of reacted interfacial layer. We have also determined yttrium content of YDH films to be 19% by using anomalous x-ray diffraction (AXD) across Y *k*-edge and angle resolved X-ray photoelectron spectroscopy (AR-XPS). The agreement between the AXD and AR-XPS results manifests that the incorporated Y atoms indeed homogeneously substitute Hf atoms in the crystalline lattice and form a substitutional solid solution.

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MS37 O5

Generating High Brilliance X-ray Beams for X-ray Diffraction and Scattering Applications

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Keywords: single-crystal X-ray diffraction, X-ray optics, X-ray diffractometer instrumentation

Today a large fraction of the X-ray analytical systems used in two-dimensional diffraction and scattering applications are still equipped with non-optimized beam-generating schemes that combine high power sealed tubes or rotating anodes with large source-sizes with inefficient optical schemes. With the advent of single reflection graded multilayer optics and efficient, low power micro-focus sealed tubes, it has become advantageous and cost-effective to replace these high power systems with this more efficient and robust technology.

The key to this new technology are Xenocs' high performance, single reflection X-ray optics that couple optimally to small x-ray sources. The relative figure-of-merit for X-ray beams is the brilliance, which typically is expressed as photons/mrad²/mm²/s in the relevant part of the spectrum (i.e. Cu K-alpha), and which can never exceed the brilliance of the source (Liouville's Theorem). Xenocs' single reflection optics optimally conserve the brilliance of these sources, resulting in extremely bright X-ray beams.

The GeniX product line from Xenocs combines a micro-focus X-ray tube with high-efficiency Xenocs X-ray optics, and offers a high performance solution with clearly defined characteristics (beam size, divergence, spectral purity, flux…). Compared to high power sealed tubes, the Genix solution offers superior brilliance enabling faster data collection in a package with a small footprint, low power consumption, and low facility requirements. The low maintenance requirements of this solution also make it appealing as a replacement for traditional rotating anodes.

In addition to presenting the GeniX product platform we present data obtained with the GeniX to demonstrate its performance and its value as an efficient, cost-effective X-ray beam delivery solution for a variety of applications including single crystal and protein crystallography, high pressure diffraction, and SAXS.