consisting of predominantly α-Ti ($P6_3/mmc$) [1]. The Ti-6Al-4V rod was machined using both conventional and high-pressure jet–assisted methods. The depth profile of residual stress was measured using x-ray diffraction. It was found that the compress residual stress is higher and the deeper under which the compress residual stress exists, for sample cut by high-pressure jet–assisted than for sample cut by conventional method [2].

Using transmission electron microscopy the cross-section of the surface layer was found to consist of a thin outer layer with nano-sized crystals (~10 nm) and the substrate of large grains with very high density of dislocations. Electron diffraction reveals that the nano-sized outer layer is highly textured. Furthermore, the study shows that the nano-sized layer has twice the thickness for the high-pressure jet–assisted cut sample (~1,000 nm) than for the conventionally cut sample (~500 nm). This shows that high-pressure jet–assisted cutting resulted in a thick and highly modified outer layer and provides an explanation for the large and deep compressed residual stress after high-pressure jet–assisted cutting of Ti-6Al-4V.

[1] PDF File No.44-1294. [2] Vosough M., Liu P., Svenningsson I., *Mat. Sci. Forum*, 2005, **490-491**, 545-551.

Keywords: titanium alloy, residual stress, metal cutting

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New Tools to Integrate Data Analysis and Data Collection at SSRL: Web-Ice

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Recent developments in automation at the Stanford Synchrotron Radiation Laboratory Macromolecular Crystallography beamlines have been driven by the needs of the Structural Genomics projects and feedback from the user community. Current capabilities at all beamlines include automated sample mounting and centering; crystal screening; fluorescence scan measurements and analysis; and wavelength changes at side stations with automatic table motion to track the beam. These features are implemented on the beamline control software program Blu-Ice.

The most recent software developments at SSRL aim at integrating data analysis and beamline hardware to the point where only minimal input by the user will be required to carry out a complete experiment. In order to facilitate remote access to the experiment, the software is accessible remotely through a web-browser interface known as Web-Ice. Web-Ice currently provides tools to view diffraction images as they are being collected; analyze the diffraction pattern and display statistics (such as number of spots, shape, diffraction strength, etc.) and autoindex and calculate a strategy to maximize data completeness based on two images selected by the user.

The next Web-Ice release will include a crystal screening interface to analyze and score images from multiple samples. Ultimately, the software will fully integrate data analysis and beamline control software for automated data collection.

Keywords: data collection, data analysis, automation

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Study of Micro Structural Defect Parameters in Nickel Dispersed Silica Nano Composites by Warren-Averbach Method and Modified Rietveld Technique

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Detailed Fourier line shape analysis has been performed on the X-ray diffraction profiles of Nickel dispersed silica nano composites, SiO_2-Ni (Wt % Ni-7.5 : Sample I, Ni-10 : Sample II, Ni-15 : Sample III, Ni-20 : Sample IV and Ni-25 : Sample V) by employing Warren-Averbach and modified Rietveld techniques.

The nickel dispersed silica nano composites were prepared through sol-gel route from a homogeneous solution of tetraethyl ortho silicate (TEOS), C_2H_5OH, required amount of $NiCl_2.6H_2O$, $C_6H_{12}O_6$ and water. The mixture was left at room temperature for gelling. The gel samples thus prepared were washed, dried and used for X-ray analysis.

The micro structural parameters like domain size, micro strain within the domains, deformation stacking fault densities (Intrinsic $\alpha^{/}$, Extrinsic $\alpha^{//}$ and Twin fault β) and dislocation density ρ were evaluated by Fourier line shape analysis taking silicon as standard for instrumental broadening correction. It has been observed from these two analyses that the $\alpha^{/}$ and $\alpha^{//}$ faults are totally absent whereas the twin β has significant presence. It has also been found that the β initially increases up to Sample III and then decreases. This is an observation on twin fault variation with Ni content in this SiO_2 - Ni nano composite system.

Keywords: nano crystals, defect analysis, diffraction

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Synchrotron XRD Study of ZrO_2-CeO_2 Nanopowders Synthesised by Gel-combustion

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Zirconia-ceria solid solutions are being widely investigated due to their excellent mechanical and catalytic properties. For example, these materials are extensively used as promoters in three-way catalysts.

In this work, the crystal structure of nanocrystalline ZrO_2-CeO_2 solid solutions, synthesised by a pH-controlled nitrate-glycine gel-combustion process, has been studied by using a high-intensity synchrotron X-ray diffractometer (D12A-XRD1 beamline of the LNLS, Brazilian Light Facility). Several weak Bragg peaks of the tetragonal phase, which correspond to forbidden reflections in the case of a perfect cubic fluorite structure, were detected. By determining the integrated intensity of the strongest of these reflections, (112), as a function of the CeO_2 content, the tetragonal-cubic phase compositional boundary was established to be at (85±5) mol% CeO_2. For a CeO_2 content up to (68±2) mol%, we identified a tetragonal phase with $c/a > 1$, whereas, in the range between 68 and 85 mol% CeO_2, the existence of a tetragonal phase with $c/a = 1$ and oxygen anions displaced from their ideal positions in the cubic phase (keeping the tetragonal symmetry) was verified. Finally, solid solutions with CeO_2 contents higher than 85 mol% exhibit the cubic fluorite-type phase.

Keywords: synchrotron powder diffraction, zirconia-ceria, nanocrystalline materials

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Characterization of Individual Zincoxide Nano-belts by using X-ray Nano-Diffraction Technique

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Nano-structures, such as wires, rods, belts and tubes, whose lateral dimensions fall in the range of 1 to 100 nm, have received growing interests due to their outstanding proprieties and their potential applications in electronic and biological fields. The development of these new structures into future nano-devices crucially depends on the development of new characterization techniques and theoretical models for a fundamental understanding of the relationship between the structure and properties [1].

X-ray diffraction technique has been successfully applied for characterization of bulk or powder nano-structured materials, where useful information, such as crystallite size distribution, crystallite

shapes and lattice defects were evaluated from the X-ray pattern. In those cases the determined quantities are characteristic to a large volume of sample.

In the present case the X-ray diffraction technique was employed for characterization of individual nano-belts. The measurements of X-ray diffraction lines from a single nano-belt were achieved by using the unique nano-diffraction technique described in [2]. The results were compared with those obtained from SEM/TEM.

[1] Zhao M. H., Wang Z.L., Mao S. X., *Nano Letters*, 2004, **4**, 587. [2] Xiao Y., Cai Z., Wang Z. L., Lai B., Chu Y. S., *J. Synchr. Rad.*, 2005, **12(2)**, 124.
Keywords: nano-belts, X-ray nano-diffraction, nano-structure

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Structural Characterization of Hybrid Carbon Nanomaterials
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Reflection High Energy Electron Diffraction (RHEED) has been used to investigate the structural features of a new class of nanostructured carbon materials, coupling nanosized diamond with single-walled carbon nanotubes. This innovative material is being produced in our laboratories in a modified CVD reactor by means of reactions between carbon nanopowders and atomic H.

We investigated samples grown at increasing deposition time, combining the structural RHEED data with the information achieved by complementary analysis techniques (Field-Emission Scanning Electron Microscopy (FE-SEM), transmission electron microscopy (TEM), Raman spectroscopy) and by a suitable theoretical approach using *ab initio* modelling [1].

We have been able to determine the growth sequence of the carbon nanophases and the architecture of the observed hybrid nanostructures. Their inner structures are found to be single-walled Carbon nanotubes (SWNT) or bundles of them, and the outermost deposit consist of faceted diamond nanocrystallites.

The experimental conclusions confirm the theoretical prediction [1] about the role of atomic hydrogen in creating localized sp3 hybridized defects on the outer wall of carbon nanotubes, able to promote the formation of suitable sites for nanodiamond nucleation.

[1] Barnard A.S., Terranova M.L., Rossi M., *Chem.Mater.*2005, 17, 527.
Keywords: RHEED, nanophase systems, carbon nanotubes

P.25.07.5
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Strain, Size and Composition of Buried GaN Quantum Dots in AlN Using Grazing Incidence Anomalous Diffraction
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Structure determination of buried nano-structures represents a challenge due to (i) the nanometric scale of objects and (ii) the presence of strain fields, which produce a 3D (non-discrete) diffuse scattering.

We have developped the use of grazing-incidence multi-wavelength anomalous scattering, which allows to extract the scattering contribution of the (resonant) atoms only. By targetting the resonant edge of one atom of the nano-structures, it allows solving the sub-structures of the nano-objects without requiring any model or prior information.

We will show how this technique can be used to extract the sub-structure of GaN Quantum Dots (QD) in AlN, to obtain the size and strain of QD as a function of the number of layers of QD deposited.
Keywords: nanostructures, anomalous scattering, synchrotron

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Structure Indexing and Solution from Non-Ambient XRPD Data
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Despite excellent recent progress, crystal structure determination from powder data remains a challenge.[1] This is especially so for flexible and weakly diffracting organic (inc. pharmaceutical) compounds.[2] Of particular interest to the pharmaceutical industry is the full structural characterisation of crystalline forms under both ambient and non-ambient conditions. This arises from a desire to understand the behaviour of solids, and how the materials respond to a range of humidities and temperatures.

The necessary diffractometer hardware to conduct non-ambient experiments (*e.g*. an Anton Parr humidity stage or a TTK temperature stage) is well established,[3] but the application of such experiments to pharmaceutical compounds is less so.[4] Here we present the methodology required to obtain high resolution non-ambient data for crystalline forms that correspond to phase changes observed in other solid state analytical techniques (*e.g*. DSC or GVS). We have indexed and subsequently solved the structures of observed forms from this non-ambient data to give full structural information across the phase diagrams. In addition, the thermal expansion coefficient for the material can be determined by indexing over a range of temperatures. This value is key for the construction of a pressure *vs*. temperature thermodynamic phase diagram.

[1] a) see, for example, David W. I. F., Shankland K., McCusker L.B., Baerlocher Ch., *IUCr Monogr. Crystallogr.*, 2002, **13**, 337; b) Harris K.D.M., Cheung E.Y., *Chem. Soc. Rev.*, 2004, 33. [2] Shankland K., Markvardsen A.J., David W.I.F., *Zeit. Krist*., 2004, **219**, 857-865. [3]see, for example, Anton-Parr-Str, Graz, Austria: website www.anton-parr.com . [4] Brittain H.G., *Spectroscopy*, 2001, **16**, 14-16.
Keywords: powder diffraction under non-ambientconditions, pharmaceutical structure determination, hydrates

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Structure Analysis of Pharmaceutical Compounds from Powder Diffraction Data
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In recent years, the crystal form of pharmaceutical compounds has become important not only for identification of the quality but also for accession of the patent. However, powder diffraction data using standard in-house instruments has low resolution as compared to the synchrotron radiation data. Accordingly, we have been studying the usefulness of the synchrotron radiation and have identified the crystal form of pharmaceutical compounds. In this study we investigated polymorphs of carbamazepine, taurine and acetaminophen as an example of pharmaceutical compounds. Additionally, we made a study on the structure determination of carbamazepine and taurine using the synchrotron diffraction data.

All pharmaceutical compounds, except for carbamazepine form III, were purchased from Wako Pure Chemical Industries (Tokyo, Japan). Carbamazepine form III was prepared by heat treatment of form I at 443 K for 2 hrs. The concomitant samples of carbamazepine were prepared by mixing form I in form III with mortar and pestle moderately. Powder X-ray diffraction patterns were collected with BL24XU of SPring-8 using milled powder samples packed in a quartz glass capillary.

As the result of analysis for concomitant polymorphs of carbamazepine, the peaks of 0.5 % form I at 12.06° and 12.3° (2Theta) are detected. By Rietvelt refinement, Rwp of the carbamazepine (form III) is 6.00 % (Rp=4.02 %).
Keywords: pharmaceutical compounds, polymorphs, powder refinement