P.25.01.1

Acta Cryst. (2005). A**61**, C485

Characterization of Heteropoly Acids by X-ray Powder Diffraction at SPring-8

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It is important to know in short time whether a material synthesized is the material actually desired. The single crystal X-ray diffraction method is one of the typical and popular methods for this purpose, especially in the field of the structure analysis on the heteropoly acids. In this study, another method was examined, where the structure analysis was done by the powder diffraction method using the synchrotron radiation. Synchrotron radiation has a very small divergence and a high brilliance. Therefore, the method would make it possible to measure a peak separation, which would be difficult using the conventional laboratory system. In addition, it also makes the measurement time much shorter. Powder diffraction profiles using a large Debye-Sherrer camera with an imaging plate at the synchrotron public industry beamline BL19B2/SPring-8. The wavelength of the incident X-ray used was 0.1237 nm. The powder of a Keggin-type [$K_8SiW_{11}O_{39}$] complex was sealed in a soda glass capillary. The structure parameter of heteropoly acids was determined using the following two methods direct method (EXPO2004)[1] and Rietveld method (RIETAN2000)[2]. (Supported by Hyogo prefecture CREATE, JST.)

[1] Altomare A., et al., *J Appl. Cryst.* , 2004, **37**, 1025-1028. [2] Izumi F., Ikeda T., *Mater. Sci. Forum*, 2000, **198**, 321-324.

Keywords: heteropoly acids, powder diffraction in industry, synchrotron radiation application

P.25.01.2

Acta Cryst. (2005). A**61**, C485

Convergent Beam Method in X-ray Diffractometry to Determine Single Crystal Cuts' Orientation

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The scheme of orthogonal incidence of primary convergent x-ray beam is applied. Two linear PSDs symmetrically placed on both sides of primary beam register the diffraction picture in Bragg plane with primary beam and detectors' wires in it. We take account of interference maximums from the planes being at the diffraction angle or at a close angle, perpendicular to the surface. Example: single crystal Si, surface – (111) ± 2°, primary beam convergence angle – 8°, beam axis is orthogonal to the single crystal surface, the diffraction plane – {220}, angle between planes (111) and (220) – 35,27°, radiation Cr K_α, λ= 2,286, diffraction angle for plane (220) - 36,43°. The task: determine angles of rotation and tilting to bring plane (111) into the plane of the single crystal surface with minimum manipulations. Solution: Any angular deviations of the diffraction cone axis in the directions perpendicular to the Bragg plane lead to closing in of the interference maximums, and in the directions within the Bragg plane – to their shift along the detectors wires to the left or to the right.

Processing software allows real time control of the Bragg's plane tilting and turning, producing within a few seconds the necessary orientation of the single crystal.

Keywords: convergent beam, X-ray, orientation

P.25.01.3

Acta Cryst. (2005). A**61**, C485

New Model-free Method of Aberrations Correction for X-ray Powder Diffractometry

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The influence of instrumental aberrations is different for different experimental XRD set-ups. Proper correction for aberrations is required for accurate analysis, comparison, and storage of patterns in databases. Most of the current methods for aberration correction are based on simulation, which requires the introduction a model of peaks in the pattern. The introduction and refinement of peaks model is not always possible based on the available information.

We will describe the new method capable to correct instrumental aberrations while considering the diffraction pattern as a single "unknown" continuum. No input information about the sample microstructure or peak positions is required, and generally, there are no variable parameters to be refined.

An algorithm named "cleaning procedure" [1] incorporating the mathematical model of instrumental aberrations [2], converts an experimental pattern to one corrected for instrumental aberrations. The peaks in the corrected pattern appear to be symmetrical and positioned in the ideal (Bragg) positions regardless of the instrumental setup. The corrected pattern may be processed later by any analytical package or stored in the database.

The advantage of this approach is that the aberration correction stage is split from the analytical stage and may be performed with minimum sample information. The application of the method to patterns of different structures including bio-polymers will be presented for reflection, transmission, focusing and parallel beam geometries.

[1] *patent pending*. [2] Kogan V.A., Kupriyanov M.F., *J.Appl.Cryst*, 1992, **25**,16-25.

Keywords: instrumental computing, instrumentation and software, X-ray diffraction theory

P.25.02.1

Acta Cryst. (2005). A**61**, C485

Residual Stress Measurements for Rocks by TOF Neutron Diffraction Methods

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When using the rock, to know the pressure which rock had received in underground is important. It is thought that the rock is maintaining the stress received in underground as residual strain. It is possible to presume the pressure under the earth in order to measure the residual stress for the rocks.

In this report, we have made an experimental study to measure the residual strain of granite rocks by neutron diffraction using Sirius diffractometer at KENS, KEK. The d-spacing from powder sample was used to calculate the residual strain instead of the unstressed d-spacing. The maximum residual stress for quartz in granite used for this experiment is 34.4Mpa.

We also made the uniaxial compression experiments using the compression-testing device for Sirius diffractometer to get the relationship between strain by neutron diffraction and applied stress. The residual stress for quartz evaluated from the result of compression experiments is 5.5 MPa. The difference in two result of residual stress will be discussed. The result using marble rocks will be also reported.

Keywords: residual stress measurements, neutron diffraction, granite rock

P.25.02.2

Acta Cryst. (2005). A**61**, C485-C486

Microstructural Evolution of Titanium Alloy (Ti-6Al-4V) after Metal Cutting Assisted by High Pressured Jet Cooling

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Titanium alloys are used in aerospace industry owing to their high strength to weight ratio but difficult to machine. High-pressure jet–assisted machining of titanium alloys is beneficial. It increases productivity and improves the properties of the work-piece.

The titanium alloy used in the present study was Ti-6Al-4V

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

P.25.03.1

Acta Cryst. (2005). A**61**, C486

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

P.25.07.1

Acta Cryst. (2005). A**61**, C486

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

P.25.07.2

Acta Cryst. (2005). A**61**, C486

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

P.25.07.3

Acta Cryst. (2005). A**61**, C486-C487

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