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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

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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

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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

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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

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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

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