

its Q-resolution is about 0.3 % at back scattering detectors. By utilizing the broadband width of neutron wavelength, it can observe a wide Q range of $0.01 < Q < 100 \text{ \AA}^{-1}$ in one experiment. These features provide diffraction pattern with reasonable resolution for PDF analysis as well as Rietveld refinement. Also, with the low Q data, larger scale structure and/or fluctuation will be observed. The expected neutron flux at the sample position of NOVA is 5×10^8 neutron / sec at 1 MW operation of the proton accelerator. Typical measurement time is expected to be several minutes for full Q-range measurement. By introducing state-of-art data acquisition technique, each neutron detection event at detectors will be recorded separately and it is possible to relate values of outer fields of the sample with the time of the neutron scattered. In some cases such as structural changes in reversible phenomena, msec order measurements of the full Q-range can be realized with the technique. In-situ measurements of hydrogen absorbing/desorbing process are planning to understand local structural changes in hydrogen storage materials. Despite it is still open question how we can analyze the full Q-range data consistently, it is promising that NOVA will provide new opportunities for non-crystalline structure of materials. This research is supported by NEDO (New Energy and Industrial Technology Department Organization) under "Advanced Fundamental Research Project on Hydrogen Storage Materials"

Keywords: neutron diffraction techniques, noncrystalline materials, hydrogen storage

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Negative thermal expansion and local crystal structure

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The structures of a significant group of materials which contract on heating consist of flexibly-linked polyhedra. This group is typified by ZrW_2O_8 , with a maximum negative thermal expansion (NTE) of -9.1 MK^{-1} and isotropic NTE over $\sim 1000 \text{ K}$. The WO_4 and ZrO_6 polyhedra are joined together at their corners by oxygen atoms and this flexibility, enhanced by the presence of a non-bonded oxygen atom on each WO_4 tetrahedron, gives rise to the NTE in ZrW_2O_8 [1,2]. Bonding via a molecular bridge---even one as simple as CN---can give rise to more extreme behaviour. For example, $\text{Ag}_3[\text{Co}(\text{CN})_6]$, whose CoC_6 octahedra are joined via a pentanuclear --CN--Ag--NC-- linkage, displays colossal NTE and positive thermal expansion of more than $\pm 100 \text{ MK}^{-1}$ along orthogonal directions throughout its $\sim 500 \text{ K}$ stability range [3]. This presentation will describe how analysis of the local structure of ZrW_2O_8 and $\text{Ag}_3[\text{Co}(\text{CN})_6]$ from reverse Monte Carlo modelling of total scattering data has been used to understand the local structural changes associated with unusual thermal expansion behaviour. Furthermore we will show how pressure-induced amorphisation might be explained in ZrW_2O_8 [4] and how weak argentophilic interactions govern the thermal expansion in $\text{Ag}_3[\text{Co}(\text{CN})_6]$ [5].

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High-resolution structure of disordered materials by high-energy X-ray diffraction

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Atoms in countless materials, both nature and man made, are not arranged in a perfectly periodic pattern over long-range distances as they are in crystals. The x-ray diffraction (XRD) patterns of such materials show only a few, Bragg-like peaks, if any, and a very pronounced diffuse component rendering traditional crystallographic techniques very difficult to apply. An alternative and very successful approach is that of the atomic pair distribution function (PDF) analysis (1,2). In the talk we will show results from recent high-energy ($\sim 100 \text{ keV}$) XRD and atomic PDF studies on materials of various degrees of structural disorder including water, ultra small ($\sim 1 \text{ nm}$) metallic clusters grown inside polymeric hosts, semiconductor QDs, fast Li-ion conducting glasses and nanosized ferroelectric ceramics. The experimental and data reduction procedures needed to obtain good quality PDF data with x-rays will be discussed as well.

1. Y. Waseda in *The structure of non-crystalline materials: liquids and amorphous solids*, (Mcgraw-Hill, 1980)

2. T. Egami and S.J.L. Billinge in *Underneath the Bragg peaks: structural analysis of complex materials* (Pergamon Press, Amsterdam, 2003).

Keywords: synchrotron X-ray scattering, atomic pair distribution functions, disordered materials

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Atomic pair distribution function analysis on nanomaterials

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Atomic pair distribution function (PDF) analysis is very powerful tool for the structural analysis of a nanomaterial [1], because the crystal structure cannot be well reproduced by a unit cell with periodic boundary condition due to the limited size. Recently, there are active researches about the structure determination of nanomaterials [2-7]. Here, we will discuss three finite size effects on PDF. The fast one is the particle form factor effect. The second one is the small angle scattering effect. The last one is the particle-particle correlation effect. So far, we have studied the former two cases [6]. Here, in addition, we would like to discuss the last effect for a

simple case, e.g., spherical particle-particle correlation. The particle-particle correlation effect without the atomic correlation gives us a background in PDF. One of important points in PDF analysis is to include background in a diffraction pattern. Interestingly, the background in PDF gives us information of particle-particle correlation when there is no atomic correlation between the particles. These three effects will be discussed by using PDF data sets of TiO₂ nanoparticle [6], Ge₂Sb₂Te₅ nanoparticle embedded in amorphous matrix [8], and C60 powder samples.

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High pressure studies of planetary ices

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In the context of the outer solar system, the ices (water-ice, ammonia, methane and mixtures of these) are minerals. Their high-pressure behaviour is thus crucial to models of the evolution of planets such as Uranus and Neptune and planetary satellites like Titan, Ganymede and Triton in the same way as the properties of ferromagnesian silicates are basic to models of the Earth. Over the past ten years, access to high-quality x-ray and neutron diffraction data has provided a wealth of new information on high-pressure structures and phase relations for many of the ices. This has greatly enhanced our understanding of these systems and placed models of planets on a firmer basis. In my talk, I will describe some of the results that I and co-workers have obtained in the methane, methane-water, ammonia and ammonia-water systems and describe the consequences for planetary models.

Keywords: high-pressure diffraction, high-pressure neutron diffraction, planetary interiors

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Nucleation and growth of ice XI -Study suggests the existence of ferroelectric ice in the Universe-

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From neutron powder-diffraction experiments, we have studied the growth process of ferroelectric ice XI with deuteron-ordered arrangements. We measured time-resolved neutron diffraction of compressed KOD or NaOD doped D₂O-ice. We observed the growth of ice XI at 58 - 74 K. The mass fraction *f* (the ratio of mass of ice XI to that of the doped ice) linearly increased with time for about 5 days. The de-transformed ice Ih, obtained after warming above the transition temperature of 76 K, retransformed to ice XI at 60 -66 K. The observed increase of *f* with time is in good agreement with the nucleation process of the ordering and the constant growth of the ordered domain. The results suggest that large quantities of ice on cold icy bodies (such as Pluto and Charon) in our solar system are able to transform to ice XI, which may be detectable by space telescope and planetary exploration.

Keywords: neutron powder diffraction, time-resolved structural studies, ice structures

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High-pressure phase transitions of deep Earth materials

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Recent developments in synchrotron XRD measurements combined with laser-heated diamond-anvil cell (LHDAC) techniques have enabled us to search for a novel phase transition at extremely high pressure and temperature. A phase transition from MgSiO₃ perovskite to post-perovskite was discovered through a drastic change in XRD patterns above 120 GPa and 2500 K, corresponding to the condition in the lowermost mantle (Murakami *et al.*, 2004; Oganov and Ono, 2004). A pressure-induced phase transformation from ABO₃-type perovskite to any denser structures was not known at that time. This new MgSiO₃ polymorph called post-perovskite has an orthorhombic symmetry (space group: *Cmcm*) with a sheet-stacking structure. The Mg site in post-perovskite is smaller than that in perovskite, which results in a volume reduction by 1.0-1.5% from perovskite structure. The electrical conductivity of post-perovskite is higher by three orders of magnitude than that of perovskite at similar pressure range (Ohta *et al.*, 2008). This is likely due to a shorter Fe-Fe distance in post-perovskite structure, while conduction mechanism is yet to be further examined. Phase transition boundary between perovskite and post-perovskite has been determined in a wide temperature range up to 4400 K at 170 GPa (Tateno *et al.*, 2008). Phase relations of Fe alloys have been also studied at core pressures (>135 GPa), although the generation of high temperature is more difficult at higher pressures. A new high-pressure B2 phase of FeS was recently discovered above 180 GPa (Sata *et al.*, 2008). The Fe-Ni alloys have a wide pressure-temperature stability field of fcc phase at the core pressure range, depending on the Ni content (Kuwayama *et al.*, 2008).

Keywords: high-pressure phase transformation, perovskite oxides, iron compounds