

distance, to determine the average number of nanoparticles associated with each microsphere, and to define the correlations among the nanoparticles in the halos. Our observations, based on ultra-small-angle X-ray scattering, reveal the fundamental nature and structure of the haloing effect, and demonstrate that the nanoparticles are separated from the microsphere by a distance close to the Debye screening length of the solution.

Keywords: colloidal suspensions, photonic crystals, small-angle x-ray scattering

dodecahedral. These morphologies determine the structure of the colloidal crystals: in the case of octahedral morphology, the lattice structure is body centered cubic (BCC), while the lattice structure is face centered cubic (FCC) in the case of rhombic dodecahedral morphology. This relationship was not observed for spherical constituent particles. It is known that a repulsive force between particles is necessary for the formation of colloidal crystals. Magnetite has a magnetic force that results in strong attraction between the particles. It suggests that other particles that have no or weak magnetic force must be the precursor for magnetite colloidal crystal formation. These precursor particles could disperse in solution and subsequently form of the colloidal crystals, finally transformed into magnetite. A possible candidate is greigite, which was observed at the surface of the magnetite particles.

Keywords: colloids, magnetic particles, meteorite

MS.39.1

Acta Cryst. (2008). A64, C72

Total scattering: The key to the local and medium range structure of complex materials

Thomas Proffen

Los Alamos National Laboratory, Lujan Neutron Scattering Center, Mailstop H805, Los Alamos, NM, 87545, USA, E-mail : tproffen@lanl.gov

Structural characterization is mainly based on the measurement of Bragg intensities and yields the average structure of the crystalline material. However, this approach ignores any defects or local structural deviations that manifest themselves as diffuse scattering. It also fails in case of disordered materials, badly crystalline such as many nano-materials, or not crystalline at all, such as glasses. In some cases crystalline and amorphous phases coexist making the traditional crystallographic structure refinement difficult or incomplete. The total scattering pattern, however, contains structural information over all length scales and can be used to obtain a complete structural picture of complex materials. Suddenly one has access to a new parameter, the real-space range of the refinement and structures can be analyzed as function of length scale straight forwardly. Here we present different applications of this technique including data taken on the high resolution neutron powder diffractometer NPDF located at the Lujan Neutron Scattering Center at Los Alamos National Laboratory. This instrument is design for total scattering studies using the Pair Distribution Function (PDF) approach and length scales in excess of 200Å can be accessed.

Keywords: total scattering, disordered materials, neutron powder diffraction

MS.38.5

Acta Cryst. (2008). A64, C72

Self-assembled magnetite particles formed 4.6 billion years ago

Jun Nozawa, Katsuo Tsukamoto, Kenta Yamada

Tohoku University, Aramaki, Aoba, Sendai, Miyagi, 980-8578, Japan, E-mail : nozawa@ganko.tohoku.ac.jp

We report magnetite colloidal crystals formed in the space 4.6 billion years ago. In the meteorite parent body there were abundant waters, and some minerals were generated as a result of the aqueous alteration. Magnetite (Fe_3O_4) is one of typical minerals formed by the aqueous alteration. We found the colloidal crystals composed of magnetite with various morphologies, e.g. octahedral and rhombic

MS.39.2

Acta Cryst. (2008). A64, C72-73

New opportunity to explore noncrystalline materials by neutron total diffractometer (NOVA) at J-PARC

Toshiya Otomo

High Energy Accelerator Research Organization (KEK), J-PARC Project Office, 1-1 Oho, Tsukuba, Ibaraki, 305-0801, Japan, E-mail : toshiya.otomo@j-parc.jp

A neutron total diffractometer, named "NOVA", is constructing now at Japan Proton Accelerator Research Complex (J-PARC). NOVA will be used as very intense powder diffractometer since

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

MS.39.3

Acta Cryst. (2008). A64, C73

Negative thermal expansion and local crystal structure

David A Keen¹, Andrew L Goodwin², Matt G Tucker¹, Martin T Dove², John S O Evans³, Lars Peters³

¹Rutherford Appleton Laboratory, ISIS Facility, Harwell Science and Innovation Campus, Didcot, Oxfordshire, OX11 0QX, UK, ²Department of Earth Sciences, Cambridge University, Downing Street, Cambridge, CB2 3EQ, UK, ³Department of Chemistry, Durham University, University Science Laboratories, South Road, Durham, DH1 3LE, UK, E-mail : d.a.keen@rl.ac.uk

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].

[1] M G Tucker, A L Goodwin, M T Dove, D A Keen, S A Wells & J S O Evans, *Phys Rev Lett* **95** 255501 (2005)

[2] M G Tucker, D A Keen, J S O Evans & M T Dove, *J Phys CM* **19** 335215 (2007)

[3] A L Goodwin, M Calleja, M J Conterio, M T Dove, J S O Evans,

D A Keen, L Peters & M G Tucker, *Science* **319** 794-797 (2008)
 [4] D A Keen, A L Goodwin, M G Tucker, M T Dove, J S O Evans, W A Crichton & M Brunelli, *Phys Rev Lett* **98** 225501 (2007)
 [5] M J Conterio, A L Goodwin, M G Tucker, D A Keen, M T Dove, L Peters & J S O Evans *J Phys CM* (Submitted)

Keywords: thermal expansion, pair distribution function, cyanides

MS.39.4

Acta Cryst. (2008). A64, C73

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

Valeri Petkov

Central Michigan University, Physics, Dow 203, Mt. Pleasant, MI, 48858, USA, E-mail: petkov@phy.cmich.edu

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

MS.39.5

Acta Cryst. (2008). A64, C73-74

Atomic pair distribution function analysis on nanomaterials

Shinichi Shamoto, Katsuaki Kodama, Satoshi Iikubo, Tomitsugu Taguchi

Japan Atomic Energy Agency, Quantum Beam Science Directorate, #2-4, Shirakata, Tokai, Ibaraki, 319-1195, Japan, E-mail : shamoto.shinichi@jaea.go.jp

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