FA5-MS06-O1

Pressure Induced Structural Changes in Nano-sized Materials Studied with On-Line Brillouin Spectroscopy and XRD. V.B. Prakapenka[a], I. Kantor[a], A. Kantor[b], S. Sinogeikin[c], M.L. Rivers[a], S.R. Sutton[a]. [a]*CARS, University of Chicago, USA.* [b]*Bayerisches Geoinstitut, Germany.* [c]*HP-CAT, Carnegie Institution of Washington, USA.*
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The combination of Brillouin spectroscopy (BS) with x-ray diffraction (XRD) was shown to be very effective in the high pressure studies of single crystals in the diamond anvil cell [1]. However, in a number of cases, high pressure induced, single crystal re-crystallization due to phase transition and stress significantly limited the high pressure-temperature range suitable for reliable BS and XRD measurements. In this work, we present a new approach in high pressure research employing the combination of BS with synchrotron XRD for characterization of nano-sized materials that could be readily synthesized in-situ at high pressure [2]. The main reasons for using nano-structural materials are to (1) reduce the anisotropy of samples probed with BS/XRD and (2) avoid significant effects on the Brillouin spectra due to the high sensitivity of acoustic velocities to crystallographic direction. The ability to perform simultaneous measurements of velocities and bulk modulus Ks (by BS), and the volume/density (by XRD) independent of any pressure standard in the same pressure-temperature environment provides essential information to resolve discrepancies between experimental data and theoretical calculations. The advantages and excellent performance of this technique will be illustrated by the characterization of elastic and structural properties of nano-sized ZnO at the pressure induced phase transition from hexagonal (wurtzite, B4) to cubic (rock salt, B1) phase.

[1] Sinogeikin S., Bas J., Prakapenka V.B. et al, *Rev. Sci. Instrum*, **2006**, 77, 103905. [2] Prakapenka V.B., Shen G.Y., Rivers M.L. et al, *J. Syn. Rad.,* **2005**, 12, 560.

Keywords: nano-crystallography; brillouin spectroscopy; high pressure

FA5-MS06-O2

High-Pressure Studies of Transition Metal Compounds Located Near the Insulator-Metal Borderline. Karl Syassen. *Max-Planck-Institut für Festkörperforschung, D-70569 Stuttgart, Germany.*
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Experimental high pressure studies of crystalline phases take advantage of numerous recent developments in diamond-anvil-cell techniques. Major advances have occurred in microscopic analytical methods that utilize synchrotron x-ray radiation (diffraction and inelastic scattering), optical spectroscopies, and synchrotron infrared spectroscopy. Concerning correlated electron systems, the subjects of interest range from pressure-induced structural changes to illuminating the interplay between more subtle changes in atomic arrangements, electron delocalization, magnetism, and superconductivity. Some recent results will be highlighted in this presentation. The main focus will be on structural and electronic properties of perovskite-related transition metal oxides located near the insulator-metal borderline, specifically titanates and vanadates. New findings for cuprate superconductors and Fe-based pnictides will be addressed briefly.

Work performed in collaboration with X. Wang, I. Loa, S. Karmakar, I. Efthimiopoulos (MPI/FKF Stuttgart), M. Hanfland, M. Merlini (ESRF Grenoble), and Y.-L. Mathis (ANKA Karlsruhe).

Keywords: transition metal oxides; high-pressure; X-ray diffraction

FA5-MS06-O3

Pressure-Induced Crystallographic Transitions Related to Electronic/Magnetic Phenomena in Iron(II,III) Compounds*. Gregory Kh. Rozenberg[a], Moshe P. Pasternak[a], Weiming M. Xu[a], Alexander Kurnosov[b], Leonid S. Dubrovinsky[b], Sakura Pascarelli[c], Manuel Munoz[c], Marco Vaccari[c], Michael Hanfland[c]. [a]*School of Physics and Astronomy, Tel-Aviv University, Ramat-Aviv, 6997, Tel Aviv, Israel.* [b]*Bayerisches Geoinstitut, University Bayreuth, D-95440 Bayreuth, Germany.* [c]*European Synchrotron Radiation Facility, BP 220, 38043 Grenoble Cedex, France.*
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The main issue of this paper is structural aspects of electronic/magnetic transitions in iron (II, III) compounds in a regime of very high static density. The experimental tools used were (a) synchrotron X-ray diffraction (XRD) as a structural probe (b) Mössbauer spectroscopy with its hyperfine interactions as magnetic probes, (c) electrical resistivity as a tool to identify gapped or gapless states, and (d) *K*-edge X-ray absorption spectroscopy as an atomic-scale structural and valence state probe. We concentrated on the following phenomena and corresponding structural transformations:
(i) PI metal–metal *intervalence charge transfer* in the layered antiferromagnetic $Cu^{+1}Fe^{3+}O_2$ delafossite as a result of the increase in overlap of atomic orbitals. This process results in part of the Fe^{3+} converting into Fe^{2+} concurrent with $Cu^{1+} \rightarrow Cu^{2+}$ partial transition with the creation of the new Cu^{2+} - Fe^{2+} magnetic sublattice. XRD studies have shown a sequence of *R-3m* → *C2/c* → *P-3m* structural phase transitions at the 18 – 30 GPa range leading to the breakage of the dumb-bell like O-Cu-O bonds and formation of a CuO_4 tetrahedral. This results in the stabilization of the antiferromagnetic order and further culminates into the discussed valence transformation. The transitions are completely reversible in pressure and with no noticeable hysteresis. This pressure-induced sequence of structural phase transitions, the first triggered by the structure instability of *R-3m* beyond 18 GPa and the second by the electronic overlap of the Cu and Fe *d*-bands adds

a new insight in the complexity of *Transition-Metal (TM) oxides* in the regime of high density of matter.
(ii) At P > 45 GPa a *high* to *low* spin crossover takes place of both iron species in $CuFeO_2$ high-pressure phase as a result of Hund's rule breakdown at very high density. Such a transition will be accompanied by a significant reduction of the TM ionic radii and therefore a volume decrease or even a structural transition. Finally, at P > 75 GPa a *Mott* transition occurs in $CuFeO_2$ resulting in a metallic phase.
(iii) Sluggish structural phase transitions in antiferromagnetic insulators FeI_2 and $FeCl_2$ attributed to the onset of a *Mott* transition (MT). These studies show a precursor intermediate pressure phase formation preceding the MT and allow establishing features of the structural transformation specifically attributed to the MT for different types of the electronic transitions (Mott-Hubbard and Charge-Transfer).
(iv)Volume dependence of the orbital term of the moment in FeI_2 and $FeCl_2$ results in its eventual collapse under pressure. For these compounds significant lattice distortion is observed attributed to the quenching of the orbital term, and this transformation further culminates into a Mott transition.

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Keywords: high pressure; crystallographic transition; magnetic/electronic transitions

FA5-MS06-O4

Total X-ray Scattering Studies of Nanocrystalline and Amorphous Organic and Inorganic Compounds. D. Beckers[a], M. Gateshki[a], J. te Nijenhuis[a]. [a]*PANalytical B.V., Lelyweg 1, 7602 EA Almelo, The Netherlands.*
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Recent interest in nanomaterials has increased the need to analyze structures on a local (nano) scale. However, the atomic structures of nanostructured and amorphous materials are not accessible by conventional methods used to study crystalline materials, because of the short ordering range in these materials. One of the most promising techniques to study nanostructures using X-ray diffraction is total scattering pair distribution function (PDF) analysis. The pair distribution function provides information of finding atoms separated at a certain distance. This function is not direction dependent; it only looks at the absolute value of the distance between the nearest neighbours, the next nearest neighbours and so on. The method can therefore also be used to analyze non-crystalline materials.
We have developed the application of PDF analysis on a standard laboratory system employing an X-ray tube with a silver anode as X-ray source. Previous measurements have shown that meaningful results have been achieved on various samples of different nature –crystalline, nanocrystalline, amorphous solid and liquid.
In this study we compare PDF calculations on different amorphous materials. We show the analysis of some inorganic samples like geopolymers. As an organic model substance we used lactose. We present results on the monitoring of crystallization processes of freeze- and spray-dried lactose as well as the analysis of lactose of different constitution (alpha-lactose monohydrate / beta-lactose mixtures).

Keywords: amorphous compounds; nanocrystallites; pair distribution function

FA5-MS06-O5

TEM Characterization of Axial CdTe/ZnTe Nano-Wires and Simulation of Growth by Finite Element Method. Holm Kirmse[a], Wolfgang Neumann[a], Paweł Dłużewski[b], Slawek Kret[c], Tomasz Wojtowicz[c], Detlef Klimm[d]. [a]*Humboldt-Universität zu Berlin, Institut für Physik, AG Kristallographie, Newtonstraße 15, D-12489 Berlin, Germany.* [b]*Institute of Fundamental Technological Research, Polish Akademy of Science, Swietokrzyska 21, 00-049 Warsaw, Poland.* [c]*Polish Academy of Science, Institute of Physics, Al. Lotników 32/46, 02-668 Warsaw, Poland.* [d]*Leibniz-Institut für Kristallzüchtung Berlin, Max-Born-Str. 2, 12489 Berlin, Germany.*
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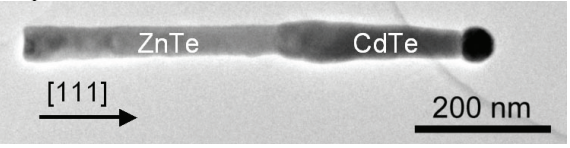
Nanowires (NWRs) are one-dimensional objects of only some 10 nm diameter and a length up to several micrometres. Semiconductor NWRs exhibit unique physical properties making the NWRs favourable for new applications. Electronic and even photonic functionality can be introduced by a p-n junction in axial heterostructure NWRs. For applications in the infrared range of light a materials combination of CdTe/ZnTe can be chosen.
Such NWRs were fabricated via a vapour-liquid-solid (VLS) growth process generated in a molecular beam epitaxy system. Liquid droplets of a gold-based catalyst promoted the NWR growth. In a first step, ZnTe nanowires were grown. After a growth interruption Cd and Te were provided for the further growth process resulting in NWRs with a CdTe/ZnTe interface.
For the TEM characterization the NWRs were harvested from the substrate and transferred to a holey carbon film. The TEM investigations were performed at a JEOL 2200FS operating at 200 kV.
The NWRs exhibit an unique shape comprising a concentric buckling of the CdTe part close to the interface. The buckling can be attributed to a combination of three effects: first, relaxation of lattice mismatch, second, lateral growth, and third, variation of the size/shape of the catalyst droplet.



TEM bright field image of a harvested CdTe/ZnTe NWR.

The CdTe/ZnTe interface is free of misfit dislocations. Hence, a pure elastic relaxation of the lattice mismatch has to be considered. CdTe is compressed and relaxes within a