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Apparent mismatch between XAFS and XRD structure of crystalline and amorphous electrochromic WO_3

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WO_3 are used in a great variety of inorganic materials dealing with a wide range of applications. We have studied four different crystalline WO_3 materials (monoclinic (1), monohydrate (2), hexagonal (3) and pyrochlore (4)) as precusors to amorphous electrochromic species obtained by mechanosynthesis. The structures of the crystalline phases were determined by various diffraction techniques (single crystal, X-ray or Neutron powder diffraction). EXAFS was used to compare the local structure around W of these crystalline phases and those of the corresponding amorphous materials. The EXAFS W-O radial distribution functions were determined as sums of gaussians, in the frame of the errors and quality of fits estimation procedures recommended by the International XAFS Society (IXS) standard and criteria committee (2000). From this comparison, two main results are obtained :
The XAFS and diffraction rdf of (1) and (2) are in agreement, but the powder diffraction rdf of (3) and (4), which are metastable phases, are in total disagreement with the EXAFS results. In these two last cases, diffraction structures miss the existence of short and long W-O bonds, revealed by the EXAFS study. We explain this mismatch by disorder effects which lead to averaged crystal structures. On the contrary the EXAFS study is able to give a more precise picture of the local W structure, even if the long range order informations are poorer;
The W-O rdf of the corresponding amorphous materials converge to a unique local structure, independent of its crystalline precursor.

Keywords: inorganic materials, XAFS and XRD, quality of fit in EXAFS

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High pressure and high temperature EXAFS and diffraction study of AgI

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We have determined the precise P-T phase diagram of AgI by in-situ high-pressure high-temperature synchrotron experiments [1]. We will report on structural details and effective potentials in various high pressure phases. EXAFS and X-ray diffraction measurements were performed up to 6.0 GPa and 1100 K using a multi-anvil high-pressure device and synchrotron radiation from Spring-8, Hyogo. In the disordered rock-salt phase, Ag ions occupy both octahedral and tetrahedral sites and twenty percent of Ag ions occupy the tetrahedral site as a maximum value at 2GPa. The transition between the rock-salt type and disordered rock-salt type phases is a broad disorder type within the same structure. From the viewpoint of the local structure analyses, some sudden changes are recognized near the phase transition point. The Debye Waller factors in AgI phases were investigated by both the diffraction and EXAFS methods. Analysis of EXAFS Debye-Waller factor is useful because the force constant can be decided directly even at high pressure and high temperature [2]. Pressure influences greatly the effective pair potential and anharmonicity decreases with increasing pressure. Phonon dispersion relations in various phases have been derived from high pressure experiments.
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Keywords: high pressure, superionic conductor, Debye-Waller factor

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In situ XRD and XAFS studies of oxidation/reduction and water gas shift reactions of Cu doped ceria

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Metal doped and impregnated ceria has been shown to be an active catalyst for the water gas shift (WGS) reaction. The structural transformations under REDOX and WGS conditions have been shown to involve reversible movement of the Cu out and back into the cerium atomic positions in the ceria structure. The diffraction studies were carried out with high energy X-rays and the high Q data allowed for improved profile refinement[1, 2] and pair distribution function (PDF) studies [3]during structural transformations (typical sampling time 3 minutes). The XAFS studies were collected in Quick EXAFS mode[4] during transformations (typical sampling times 15 seconds). Intermediate Cu2O like structure was demonstrated from principal component analysis of the time resolved QXANES data and subsequent modeling of the QEXAFS data. A comparison of PDF and EXAFS results will be presented. The research carried out at BNL was financed through contracts DE-AC02-98CH10886, DE-FG02- 03ER15476 and DE-FG02-05ER15688 with the US Dept. of Energy (Division of Chemical Sciences).
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Zooming into the overall architecture of the giant muscle protein titin

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