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CRYSTAL STRUCTURES AND TOPOLOGY OF NEW Ba BORATES

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Seven new Ba borates, namely $Ba_2[B_5O_9]ClO_{0.5}H_2O$ (I), $LiBaB_9O_{15}$ (II), $Ba_2[B_5O_8(OH)_2](OH)$ (III), $Na_2Ba_2[B_{10}O_{17}(OH)_2]$ (IV), $LiBa_2[B_{10}O_{16}(OH)_3]$ (V), $Ba_5[B_{20}O_{33}(OH)_4]H_2O$ (VI) and $Ba[B_5O_8(OH)]H_2O$ (VII) were recently synthesized and studied by XRD. The topology of the borate frameworks and sheets in I-VII may be described by means of the fundamental building block (FBB) consisting of BO_3 triangles and BO_4 tetrahedra. FBB in I ($3BO_4$ tetrahedra and $2BO_3$ triangles) is the same as compared with that revealed in hilgardite. In contrast to I 3-membered polyhedral rings composed of $2BO_3$ triangles and $1BO_4$ tetrahedron can be considered as FBB within the framework of II. II comprises borate framework with the tunnels filled by Ba atoms in 12-fold coordination and by Li atoms in unusual 3-fold coordination. III is a sheet borate: its pentaborate $[B_5O_8(OH)_2]^{3-}$ sheets are characterized by the same ratio between BO_4 tetrahedra and BO_3 triangles as compared with the pentaborate framework in I. IV also contains pentaborate FBB formed by 3 tetrahedra and by 2 triangles. Two pseudohexagonal sheets in IV form the double layers, which are connected by Ba- and Na-polyhedra. V is characterized by new borate sheet, which is formed by 5 unequivalent BO_4 tetrahedra and by $5BO_3$ triangles. Two pentaborate groups are included in its FBB. One of them comprises 3 borate triangles and 2 borate tetrahedra, whereas the second one is formed by 3 borate tetrahedra and by 2 borate triangles. Similar FBB were recognized in double borate sheets of VI. However in contrast to V, in VI two adjacent layers are related by 2-fold axes to form a double borate sheets. The topology of the borate sheet of VII is practically the same as compared with that revealed in beringuccite, $Na_2[B_5O_8(OH)]H_2O$, where FBB is formed by $3BO_3$ triangles and by $2BO_4$ tetrahedra. The interlayers in Ba-beringuccite and Na-beringuccite contain the Ba atoms and water molecules.

Similarly with most of natural borates all studied structures contain three-membered borate rings formed either by 2 tetrahedra and 1 triangle or by 2 triangles and 1 tetrahedron.

Keywords: CRYSTAL STRUCTURE, XRD, BORATES

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3D-XRD: 3-DIMENSIONAL MAPS AND DYNAMICS OF GRAINS

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3D X-Ray Diffraction (3DXRD) microscopy is an emerging tool for fast and non-destructive characterization of the individual grains and sub-grains inside bulk materials. The method is based on diffraction with hard x-rays (E > 50 keV), enabling 3D studies of millimeter - centimeter thick specimens. Ray tracing with several detectors is applied. The position, volume, orientation, elastic and plastic strain can be derived for hundreds of grains simultaneously. Likewise for coarse-grained materials grain boundary maps can be generated. With the present set-up at the 3DXRD microscope at ESRF, the spatial resolution is 5-20 mu, while diffracting units of size 200 nm can be observed.

A set of novel reconstruction methods will be presented. Based partly on the concept of Radon transforms they generate grain maps as well as the intrinsic orientation distribution within each grain or sub-grain. Furthermore, simultaneous phase and diffraction information is obtainable by on-line coupling of 3DXRD and absorption contrast tomography.

3DXRD microscopy for the first time enables dynamic studies of the individual grains in polycrystals. (Generally surface probes are not representative with respect to kinetics due to stress relaxation, pinning, a-typical growth or diffusion etc.) Results will be presented for 1) nucleation and growth kinetics during recrystallization of metals, 2) phase transformation kinetics in steels and ceramics, and 3) rotation and brake-up of grains during tensile deformation of metals.

Keywords: MICRODIFFRACTION; 3DXRD; TEXTURE

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HARD X-RAY MICRODIFFRACTION ON SOFT MATERIALS

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I will discuss recent developments in microdiffraction experiments on soft materials at the ESRF. Instrumentation and experimental applications have been strongly influenced by applications in protein crystallography. Two main experimental lines can be distinguished: (i) single crystal diffractometry, (ii) scanning diffractometry including scanning small-angle scattering. Progress in microbeam protein crystallography has been linked to the development of a dedicated goniometer allowing rapidly positioning small samples in a microbeam. This concept has been extended to small molecule experiments. Scanning diffractometry has been used for mapping extended samples, such as fibers, using the same detection system as for single crystal diffractometry. Examples for both types of experiments will be discussed. A projection into the future suggests a convergence of both approaches into a single endstation

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SUBMICRON RESOLUTION 3D X-RAY STRUCTURAL MICROSCOPY

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The availability of high-brilliance synchrotron x-ray sources and new, high-precision x-ray focusing optics have made possible revolutionary advances in microbeam x-ray capabilities for the investigation of materials microstructure on mesoscopic length scales of tenths-to-hundreds of microns. Exploiting these capabilities, we have developed a new and powerful technique for submicron resolution 3D x-ray structural microscopy. Differential-aperture (knife-edge) profiling of Laue diffraction patterns from white x-ray microbeams makes it possible to extract full diffraction patterns from submicron voxels (volume elements) in bulk materials. For the first time, nondestructive measurements of the local crystallographic orientation, structural phase, morphology, and full stress/strain tensor can be performed in single-crystals, polycrystals, composites, multi-layers, and deformed materials.

The technique for differential-aperture x-ray microscopy (DAXM) with submicron point-to-point spatial resolution in three-dimensions will be presented and the following applications of the method will be discussed: (1) Micron resolution measurements of the local structure, orientation, and grain size in hot-rolled aluminum, (2) Micron resolution measurements of the local orientation and elastic strain tensor as a function of depth in cylindrically bent silicon, (3) Micron resolution measurements of the plastic strain and geometrically necessary dislocation distributions in a nano-indented copper single-crystal.

These newly developed measurement capabilities provide a direct - and previously missing - link between the actual microstructure and evolution in materials on mesoscopic length scales and the results of theory, simulation, and modeling of materials processes.

Keywords: MICROBEAM MICROSCOPY THREE-DIMENSIONALTHREE-DIM