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Phase States Cycling Sequence in Complex Oxides Received by Decomposition of the Melt-Solution of the Simple Oxides (Nitrates) in NH_4NO_3

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$Y_3Al_5O_{12}$, $Y_3Ga_5O_{12}$ (garnets), $YAlO_3$, $LaAlO_3$ (perovskites) $LuBO_3$, $GdBO_3$ ($Lu_xGd_{(1-x)}$)BO_3, YBO_3 (borates) and $Eu_2(MoO_4)_3$ (molybdates) have been received by the dissolution of simple oxides (or nitrates) in the melt of NH_4NO_3 and by the following decomposition of the solvent at the increasing temperature. Amorphous like precursor states have been received as a result of such procedure. X-ray analysis has shown that the consequent annealing of the precursors at elevated temperatures gives a row of structure states which are characterized by the cyclic sequence of phases known for solid state synthesis.

For example, α-phase is known for macro-sized samples of $Eu_2(MoO_4)_3$ at low temperature. It undergoes phase transition into β-phase at ~800 C. In the case of annealing of the precursor of $Eu_2(MoO_4)_3$ the phase sequence is as follows: β-phase (not α-phase) arises at~500 C before other things, then it transforms into α-phase at ~600 C and finally, at T>800 C α-phase undergoes a phase transition into β-phase again. Simultaneously the dimensions of the synthesized crystallites are changed from nano- into micro- and then into macro-scale.

The explanation of the phenomenon is based on the assumption that the temperature of phase transitions depends on the total energy only and that this energy depends on the crystallite sizes.

Keywords: phase diagrams, X-ray, oxygen compounds

P.11.16.4

Three-dimensional Birefringence Imaging with a Microscope Tilting Stage

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We show that by combining the MetriPol™ imaging system (see www.metripol.com) with a microscope tilting stage it is possible to obtain very precise birefringence information on crystals. The MetriPol™ microscope uses a combination of a rotating polarizer and a circular analyzer to separate out three types of images, one representing the light transmission through the specimen, one showing the orientation of the optical indicatrix at any point in the image, and one giving quantitative information on $|\sin\delta|$, where δ is the phase difference introduced by the birefringent sample.

We demonstrate that it is possible to simulate the data closely using our optical equations for uniaxial and biaxial crystals in any general alignment, starting from the known birefringence values of the sample. In samples with a small retardance, where $|\sin\delta|$ is known to lie within the first period of the sine function, it is often possible to obtain values for the birefringence without any prior knowledge of the optical properties. We are currently investigating the use of multiple wavelength techniques to determine the absolute values of δ, as in the work of Geday, Kaminsky, Lewis & Glazer, (2000), in order to extend this technique to samples with a higher retardance. In addition, we hope to show that by constructing a suitable database of known optical properties of different materials, it should be possible to identify unknown crystalline grains in a microscope rock section.

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Keywords: birefringence, microscopy, minerals

P.11.16.5

Dynamic Method for Measurement of Piezo-Optic Coefficients

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The influence of mechanical fields on the propagation of light through a crystal is described by the piezo-optical effect (inducing quantity (i.q.): mechanical stress) or the elasto-optical effect (i.q.: mechanical strain). Dynamic strain fields are usually realised by ultrasonic waves ("acousto-optics") and the theory of the interaction of light and ultrasonic waves with anisotropic materials (e.g. Raman-Nath theory [1]) yields a number of experimental approaches to the elasto-optical constants, e.g. Acousto-optic diffraction or Brillouin scattering. The realisation of homogenous low frequency dynamic stress in crystals rises experimental difficulties. In terms of quality of data to our best knowledge the static method of Pockels [1] could not be surpassed by any dynamic method given in literature. In this work we present a dynamic device for the generation of a homogeneous low frequency (1 – 10 Hz) mechanical stress field, based on a piezo-ceramic translator that generates a periodic uniaxial pressure. The crystal sample (parallelepiped with parallelity of faces of ±1 μm) is arranged in a sandwich-like system "translator-quartz-crystal-quartz", where the quartz slabs act as force sensors that control the stress homogeneity. For the measurement of the piezo-optic coefficients via optical retardation this device is either integrated into a Jamin interferometer or into the arrangement of rotating analyser [2].

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Keywords: optical properties of crystals, acousto-optics, stress

P.11.16.6

X-ray Powder Diffraction Characterization of Nanoparticles

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Nanometer size particles show chemi-physical properties which largely differ from those of the parent bulk material. Because of the complexity and novelty of these materials, a fundamental simple task, such as determining and controlling the NP size distribution needs a complex experimental work, which often requires the combination of different characterization techniques. Among the most widely used, X-ray powder diffraction has shown a great potential to meet the increasing demands of microstructural material characterization. Indeed, powder diffraction data analysis methods – exported from micrometer-sized polycrystalline materials characterization research field – have to be specifically tuned for particle size falling much below 100 nm. In fact, even for crystalline NPs, due to the small size, Bragg peaks may be so much broadened to be hardly separated and many approximations, commonly accepted for micrometer size domains, fail. In addition, surface-related strain fields and size effects cannot easily be separated and affect both peak position and width. In the most complex cases, also non-crystallographic structures may occur. In these extreme cases, the classical crystallographic formalism becomes quite useless, being the Debye scattering function (that is, the direct evaluation of the NP structure factor from the interatomic distances) the only possible choice.

We will present examples on several nanoparticles, namely Au, II-VI compounds and CeO_2. Based on the specific material and data analysis demand, we will make use of a shape-convolution method to calculate the diffraction pattern of the NPs powder or alternatively of a computing approach based on the Debye scattering function.

Keywords: nanoparticles, X-ray powder diffraction techniques, quantitative X-ray analysis

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