analysis is used for it purpose. Therefore the elemental electrolyte composition are determined from phase concentrations. There are some analytical problems of XRD analysis. A mineralogical content a sample of cooling electrolyte may include until ten phases. Phase content depends on CR, elemental content and cooling speed. High speed of cooling induces that some Ca-contained phases are half-amorphous and some Li-contained phases have a heavy overlapping of all strong lines. Several different quantitative XRD or combined XRD and XRF [1] methods based on using of calibration standard samples have been developed for bath control at Russia aluminum smelters. Standard samples with precision values of phase concentrations are problem too because their getting by mix of the synthesis phases impossible. This presentation focuses on implementation this methods and decision of mentioned problems including phase quantification of industrial electrolytes as standard samples.

1. S.Kirik, I.Yakimov. Advances in X-ray Analysis, Vol. 44, p.85-90.

Keywords: quantitative phase analysis, aluminum bath ratio, cryolite ratio

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Diffraction experiments with high-energy X-rays during PVT growth of SiC

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An inductively heated PVT growth furnace was built for the growth of SiC crystals by the modified Lely method. Crystals of 2 inch diameter can be grown.The furnace is designed for in situ diffraction experiments and absorption contrast imaging with high-energy x-rays (50keV and higher) during crystal growth. With this furnace placed in the beam of a laboratory high-energy x-ray tungsten anode, the growth process can be monitored by x-ray diffraction, e.g. with the white beam Laue diffraction technique in focussing or non-focussing mode. Absorption contrast images of the crystal boule give information about the growth velocity. Diffraction and absorption contrast images are recorded in intervals of 15 minutes exposure time by a CCD-detector and off-line readable image plates. Up to now, the diffraction images allow the 'real-time' visualization of the thermo-mechanical response of the crystal to variations of growth parameters like argon pressure and temperature changes. Polytype changes like the 6H to 15R transition can be observed in situ in the diffraction patterns. The observation of the characteristic tungsten emission lines in the diffraction image allows to observe the macroscopic bending of the lattice planes over the crystal wafer and to determine its radius of curvature. In the initial stages of growth the shape of the developing growth front seems to be susceptible to parameter changes like e.g. repeated growth interruptions. During growth interruptions the mechanical stress is relieved. Thus with the aid of diffraction on growing SiC crystals we are able to monitor in situ transient temperature phenomena and their response to changes in the external parameter values which have been chosen for crystal growth.

Keywords: silicon carbide, crystal growth and perfection, high-energy x-ray diffraction

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Dislocation assisted intermetallic layer formation at the interface of Sn-Pb solder and Cu

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When near eutectic Sn-Pb Solder containing solid resin as flux is applied on Cu substrate ,an intermetallic layer comprising mainly of Cu_3Sn & Cu_6Sn_5 is formed at the interface ,the thickness of which determines the quality of soldering (dry or perfect).This work reports that dislocation on the Sn component favours the formation of intermetallic layer. The average dislocation density was calculated from the xrd profile of 101 line of beta-tin component of a perfectly & dry soldered Cu strip using the 4th moment($M_4(q)$) method proposed by groma & Borbely,2001.The thickness of the intermetallic layer was measured using scanning electron microscopy.

Reference:
1.A.Borbely & I. Groma, Appl.Phys. Lett.,2001,**79**,1772

Keywords: dislocations, material chemistry, metal alloys

P25.02.05

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Characterization of strain in cubic thin film with <hkl> fiber texture in anisotropic stress state

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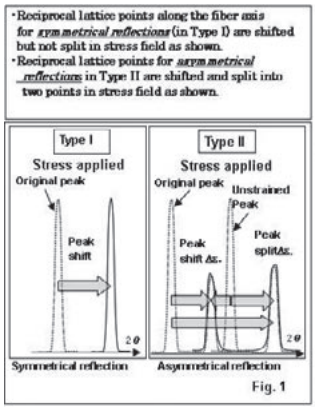
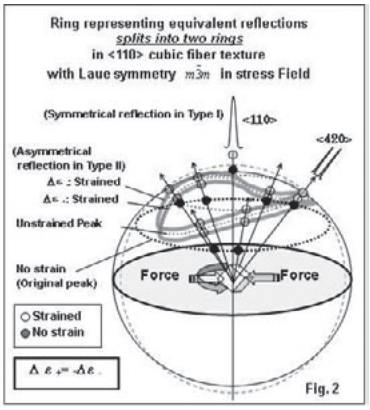
A technique for obtaining residual stress in poly-crystalline materials was proposed at AsCA' 07 (P05-019). The technique provides the residual stress based on Reuss model through an examination of overlapping reciprocal lattice points which meet Bragg condition with the Laue symmetries. This paper shows the residual stress in TiN with <110> fiber texture or other fiber textures in cubic having the Laue symmetry m-3m and analyzes the strain from the peak splits and peak broadening in the XRD profiles with the technique. Thus the equivalent reflections of the crystallites form a ring around the fiber axis in reciprocal lattice space(RLS). In the case of TiN with <110> axis, the reflections are divided into two groups by the orientation of their crystallites. The crystallites are deformed differently due to the elastic constants determined by their orientations. Type II in Fig. 1 shows the peaks of the two groups shift in opposite directions away from the unstrained peak. Thus the ring is divided into two rings as shown in Fig.2. With the above consideration of RLS, the described technique suggests its application to characterizing the strain of general poly-crystalline materials.





Keywords: fiber texture, strain, Laue symmetry

P25.07.06

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Local structure of the tetragonal phase in nanostructured zirconia-based solid solutions

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Zirconia-based ceramics are widely used because of their electrical and mechanical properties. Pure ZrO_2 exhibits 3 polymorphs of monoclinic (m), tetragonal (t) and cubic (c) symmetries. The m phase is stable at room temperature and transforms to the t one at 1170ºC during heating, while this phase transforms to the c one at 2370ºC. The c phase can be fully stabilized at room temperature by doping with other oxides (Y_2O_3, CaO, CeO_2, etc.). The t phase can not be fully stabilized, but it can be retained, in a metastable condition, in nanopowders and fine-grained ceramics. In previous works, we studied the crystal structure and the local order of ZrO_2-based nanomaterials with compositions close to the t/c phase boundary. EXAFS study showed that the t-to-c transition observed by XRD is only related to a symmetry change of the local order around Zr^{4+} cations. We also found that the expected crystallographic model (two oxygen subshells with 4+4 oxygen atoms) does not agree with EXAFS data, while a 5+2 model yields better agreement factors. It is worth to remark that, while the sevenfold coordination of Zr^{4+} cations has already been proposed for the c phase and attributed to the presence of oxygen vacancies that are preferentially located around Zr cations, only a few papers reported this coordination for the t phase. In this work, we present an EXAFS study of Zr-O bond in pure or lightly-doped ZrO_2 nanopowders that exhibit the retention of the t phase in metastable condition. The results on Zr-O bond were compared with those obtained by synchrotron radiation XRD analysis in order to establish a model for the local structure around Zr atoms that agrees with the crystal structure accepted for the t phase. The influence of the crystallite size was also investigated.

Keywords: ZrO_2, nanomaterials, EXAFS

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Phase separation inside the CdTe-CdSe type II quantum dots revealed by synchrotron PXRD and SAXS

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Using synchrotron powder X-ray diffraction and small-angle X-ray scattering (SAXS), we have studied the structures of the two CdSe/CdTe and CdTe/CdSe type II quantum dots (QDs), including the crystalline structure, particle shape and size, as well as phase separation of the two components. The X-ray results suggest that the spherical CdTe/CdSe QDs of a size of ~8 nm, synthesized in a two-step procedure with CdTe nanoparticles (4 nm) as nuclides, have a structure of a CdTe-rich core enclosed by a CdSe shell. On the other hand, the spherical CdSe/CdTe QDs of a size of ~9 nm, synthesized in a similar two-step procedure but with CdSe nanoparticles (~3 nm) as nuclides, show mainly one phase structure with a uniform distribution of CdSe and CdTe. The phase separation of the two components CdSe and CdTe inside the two QDs, a decisive factor in the photovoltaic applications, is furthermore examined using anomalous SAXS for a consistent conclusion. For comparison, single phase nanoparticles CdSe and CdTe are also examined. The correlation between the synthesis procedures and the structures of the QDs is discussed.

Keywords: CdTe-CdSe, quantum dots, PXRD and SAXS

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Teaching an old molecule new tricks: A novel tubular morphology of caffeine

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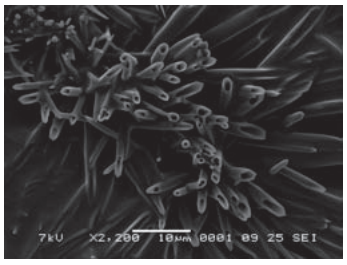
Crystals of caffeine with a novel tubular morphology have been prepared. Scanning electron microscopy (SEM) images of caffeine crystallised by rapid evaporation from solution show a network of interlinked hollow tubes with hexagonal cross-section and internal diameter of 0.5-5 μm. X-ray powder diffraction (XRPD) analysis demonstrates that these tubular structures are of the metastable, trigonal polymorph of caffeine, form I. The application of transmission electron microscopy (TEM) to the study of this species will be presented and the use of this technique as a tool to study pharmaceutical compounds in general will be discussed.



Keywords: nanotubes, pharmaceuticals, transmission electron microscopy