can take several directions including comparison of the actual geometry to either theoretical models or experimental structures from the PDB or small molecular databases. In this contribution we will present integration of search and validation tools from the Cambridge Crystallographic Data Centre, in particular Mogul, into an automated structure solution and refinement workflow. Apart from a post-refinement assessment of structure quality, we explore the use of Mogul to compare programs applied to generate ligand parameter dictionaries for crystallographic refinement. Finally, trends in the quality of ligand geometry as function of structure determination parameters will be discussed based on kinase structures from the PDB as well as those solved in-house. Systematic errors in ligand structures will be highlighted along with potential pitfalls of this validation approach.

Keywords: structure validation, statistical analysis CSD PDB, kinases

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Nucleation of heat storage materials - search algorithms for similarities of crystal surfaces

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The demand for e・ciant use of energy leads to an increasing interest in energy storage. For the storage of thermal energy latent heat storage materials are most convenient. These materials use a phase transition solid - liquid in order to store heat. Because of large melting enthalpies and low melting points salt hydrates are of particular relevance for the storage of heat at moderate temperatures. Latent heat storage materials based on salt hydrates show a strong undercooling of the melt. This inhibits the technical development of latent heat storage as an economic useful product. The controled release of the stored energy is possible by triggering the crystallisation using nucleators. Heterogenous nucleators are found empirically or by comparing geometric crystal properties like lattice parameters. The disadvantage of these methods is, that the nucleating agents known for di・erant materials so far are unreliable. Heterogeneous nucleation is expected if the melt gets in contact with epitactic and che-mical similar surfaces. Search algorithms for similar crystal surfaces were developed in order to improve the developement of nucleators for latent heat storage materials and to gain a de-eper insight to the mechanism of nucleation. This approach provides an extension of existing search routines in crystal structure databases and enables more specific search results. The program ATBEL performs a morphology prediction based on the BFDH-method. For all predicted faces ATBEL creates entries in two di・erant databases. These database are used by the program EPITAX for the search of lattice match and chemical similarity of crystal surfaces. New algorithms for automatic handling of crystal structure data are introduced.

Keywords: nucleation, heat storage, morphology

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Hydration of activated belite cements studied by synchrotron X-ray powder diffraction

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In spite of its universal use in contemporary construction, ordinary Portland cement is one of the most environmentally contentious materials. On average, for every tone of cement produced, 0.97 tons of CO_2 are released into the atmosphere. So, cement industry contributes around 6% of all CO_2 anthropogenic emissions. Hence, cement chemists are making great efforts to find ways to reduce the environmental impact of the cement-production process. Belite cements may reduce 10% de CO_2 emissions but belite reactivity with water is slow and thus these cements develop low mechanical strengths at early stages. The reactivity of these materials may be increased by two complementary ways: i) stabilize high temperature belite polymorphs and ii) produce calcium sulfoaluminate (CSA) belite cements. CSA clinkers contain $Ca_4Al_6O_{12}SO_4$, which reacts rapidly with water forming ettringite, AFt or $Ca_6Al_2(SO_4)_3(OH)_{12.26}H_2O$, and enhancing development of early age mechanical strengths. CSA clinker manufacture may reduce CO_2 emissions up to 35%. Here, we report an in-situ synchrotron powder diffraction hydration study of these cements. Both alkaline oxides activated belite and CSA cements have been analysed. The patterns were collected in transmission in BM08 beamline of ESRF using the translating image-plate detector. This methodology minimise powder averaging errors which are critical for obtaining accurate analyses. All patterns have been treated by the Rietveld method in order to extract the quantitative phase contents. The water/cement weight ratio was kept fixed to 0.5 and the gypsum role has been investigated by adding different amounts. The starting crystalline phase assemblage and the evolution of the hydrate phases will be reported and related to the calorimetric studies.

Keywords: cement hydration, synchrotron powder diffraction, quantitative Rietveld cement phase analysis

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Bath quantitative XRD control at Russia aluminum smelters

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The electrolysis cell used in the production of aluminum is a dynamic system. The concentration of main components in the electrolyte must be rigidly controlled in order to maintain optimum conditions during the production process. Key parameters for control include cryolite ratio (CR) or bath ratio (NaF/AlF_3) and additions of calcium, magnesium or lithium fluorides The bath analysis must be done during about 2 to 3 minutes because there are often several hundred or even thousands of cells to measure. The XRD quantitative phase

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

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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.