

samples with varying B contents. This BO_3 group connects the octahedral chains perpendicular to the *c*-axis (Fig. 1). The indices of distortion indicate that the incorporation of boron leads to more regular polyhedra and a rotation of the AlO_6 octahedra. Nevertheless, distance least squares refinements (DLS) were necessary to determine the local BO_3 configuration. The DLS calculations yielded split positions for the oxygen atoms directly linked to B leading to a local distortion of the octahedral geometry verified in the crystal-structure refinements. This crystallographic model including the split positions for the oxygen atoms of the AlO_6 octahedron provides an explanation for the anisotropic behavior of the lattice parameters upon B-incorporation.

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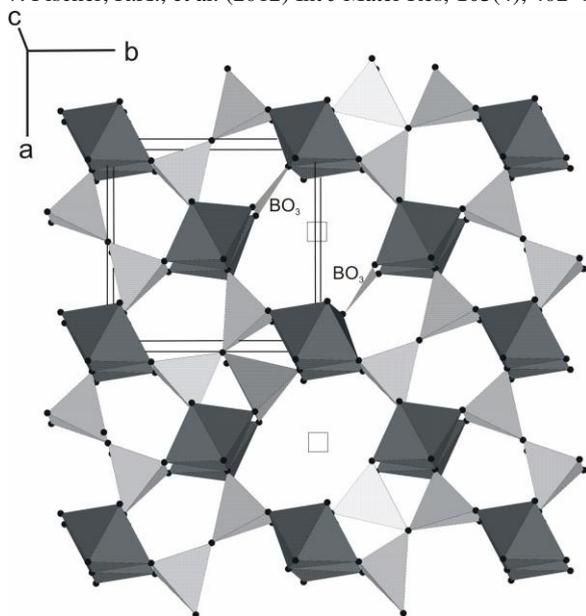


Fig. 1: crystal structure of B-mullite. Oxygen vacancies are indicated by squares. Only one out of many possible distributions [7] of oxygen vacancies, T_3O groups and BO_3 groups is shown.

MS20-T05

Structural Characterisation of shocked AlN-powders

K. Keller^{1,2}, T. Schlothauer^{1,2}, M. Schwarz^{2,3}, E. Brendler⁴, E. Kroke^{2,3}, G. Heide^{1,2}

¹TU Mining Academy, Institute for Mineralogy, Freiberg, Germany

²TU Mining Academy, Freiberg High-Pressure Research Centre, Freiberg, Germany

³TU Mining Academy, Institute for Inorganic Chemistry, Freiberg, Germany

⁴TU Mining Academy, Institute for Analytical Chemistry, Freiberg, Germany

Aluminium nitride, which has a wurtzite structure (w-AlN), undergoes a phase transition to the rocksalt structure (rs-AlN) at

pressures of about 14-23 GPa in static experiments [1-3]. Shock wave experiments have been performed with subsequently sample recovery to investigate the high-pressure phase [4].

The powders were characterised with X-ray diffraction and Rietveld full-pattern fitting. However, no pure rs-AlN phase was produced, but a phase mixture of w-AlN, rs-AlN and different Al-O-N phase. The oxygen-bearing phases, espγ-AlON with spinel structure and the 27R polytypoid of Al(O)N, gives a rough estimation of the shock temperature. We observe a sluggish phase transition caused by the kinetic barrier and a temperature-activated re-conversion.

The Rietveld-refinement of the lattice constant of rs-AlN gives a value of $a = 4,044\text{\AA}$, which is in good agreement with the HRTEM results. The crystallite size of the starting w-AlN and the synthesised rs-AlN was determined with XRD and TEM. We come to the conclusion, that the phase transition has a crystallite size dependence.

A vibration band in the IR-spectrum at about 490 cm^{-1} is assigned to the rs-AlN. Furthermore, solid state ²⁷Al-NMR studies were conducted, showing a chemical shift of 2 ppm for the sixfold Al-N coordination.

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MS20-T06

Structural and electrochemical studies of the Li-In alloys

I. Chumak¹, M. Knapp¹, H. Ehrenberg^{1,2}

¹Karlsruhe Institute of Technology, Institute for Applied Materials - Energy Storage Systems, Karlsruhe, Germany

²Karlsruhe Institute of Technology, Institute of Inorganic Chemistry, Karlsruhe, Germany

Two new binary Li-In phases are detected during galvanostatic Li-extraction from the alloy $\text{Li}_{13}\text{In}_3$ ($\text{Li}_{4,33}\text{In}$). The crystal structures of new compounds were determined by single-crystal X-ray diffraction. The compositions obtained after structure refinements were: $\text{Li}_{9,95}\text{In}_{5,05}$ (trigonal crystal system, Pearson symbol hP15, space group P-3m1 (No. 164), $a = 4.7480(7)\text{\AA}$, $c = 14.283(3)\text{\AA}$) and $\text{Li}_{8,37}\text{In}_{3,63}$ (hexagonal, hP12, P6/mmm (No. 191), $a = 4.6975(7)\text{\AA}$, $c = 11.526(2)\text{\AA}$). The structure of $\text{Li}_{9,95}\text{In}_{5,05}$ can be described as derivative from the CdI_2 typ. $\text{Li}_{8,37}\text{In}_{3,63}$ can be derived from the Li_2Pt structure.

The electrochemical tests were performed in Swagelok-type cells assembled in an argon-filled dry box with electrolyte 1M LiPF_6 in a 1:1 mixture of dimethylcarbonate and ethylencarbonate and the

Li metal foil as the negative electrode. The electrode material was prepared by grinding the powders of 80 wt. % $\text{Li}_{4.33}\text{In}$ (acting as active material), 10 wt. % carbon black (electronically conductive additive) and 10 wt. % PVDF (binder) in an agate mortar. The structural changes in $\text{Li}_{13}\text{In}_3$ electrode during electrochemical lithium extraction-insertion were studied by *ex situ* XRD (STOE STADI/P powder diffractometer, Mo- $\text{K}\alpha_1$ radiation). In situ diffraction during galvanostatic charging was performed at beamline B2 (DESY, Hamburg, Germany).

MS20-T07

POLYMORPHISM OF MONOMERIC NICKEL ACETATE WITH 2-PYRIDINEETHANOL

M. Trdin¹, N. Lah¹, I. Leban¹

¹University of Ljubljana, Faculty of Chemistry and Chemical Technology, Ljubljana, Slovenia

The investigation of the coordination chemistry of simple pyridine alcohols in combination with different carboxylate ligands resulted in the preparation and characterization of three polymorphic forms of mononuclear Ni(II) acetate with 2-pyridineethanol, $\text{Ni}_2(\text{ac})_2(2\text{-pyEtOH})_2$: two of them concomitantly crystallize from the mixture of nickel acetate and 2-pyEtOH in acetonitrile (triclinic and monoclinic form), while a third polymorph (another monoclinic form) was obtained from the mixture of nickel acetate, 2-pyEtOH and potassium oxalate in methanol.

The coordination geometry and the overall molecular structures are similar in all three polymorphs. Ni(II) atom is located on an inversion center and is surrounded by two 2-pyEtOH ligands coordinated in a chelating manner and two acetates coordinated as monodentate ligands through one of the two carboxylate O atoms. Thus, almost perfect octahedral N_2O_4 geometry is achieved. Noncoordinated carboxylate oxygen enhances the stability of the complex through a strong intramolecular H-bond with the hydroxy group of the 2-pyEtOH ligand.

The three polymorphs differ primarily in their packing arrangement which is governed by weak intermolecular interactions. The three crystal structures will be presented and compared.

MS20-T08

High temperature elastic properties of a pseudo-single crystal of the nickel-base superalloy CMSX-4

K. Demtröder¹, H. Buck², P. Wollgramm², G. Eggeler², J. Schreuer¹

¹Ruhr-Universität Bochum, Kristallographie, Bochum, Germany

²Ruhr-Universität Bochum, Lehrstuhl für Werkstoffwissenschaften, Bochum, Germany

Today composite pseudo-single crystals of nickel-base superalloys are widely used in gas turbines and jet engines. The extreme operation conditions of high temperatures, corrosive atmosphere and high mechanical stresses may result in microstructural changes of the turbine blades. The characteristic microstructure consists of a coherent arrangement of γ' precipitates with an ordered Ni_3Al -structure embedded in a matrix of γ -phase [1,2]. This exsolution structure enables extraordinary high mechanical strength at high temperatures [3]. In order to improve the superalloys for high temperature applications a better understanding of the correlations between their mechanical properties and their microstructure as a function of temperature, mechanical stresses and composition is crucial.

To this end we studied the elastic and anelastic properties of pseudo-cubic as cast and heat treated nickel-base superalloy CMSX-4 using the innovative technique of resonant ultrasound spectroscopy (RUS) between room temperature and 1250°C. In addition, the thermal expansion was investigated with the aid of a commercial dilatometer. Microstructural characterization was performed using a combination of atomic force microscope (AFM), optical microscope, electron microprobe analysis and scanning electron (SEM) microscopy.

The elastic behavior of the pseudo-single crystals of CMSX-4 show a pronounced pseudo-cubic anisotropy at room temperature (Fig. 1). Between room temperature and 1000°C the elastic constants soften non-linearly by about 28% (Fig. 1). At higher temperatures strong ultrasound dissipation effects develop indicating an increasing number of point defects or increasing mobility of defects or grain boundaries. This coincides with the dissolution of the γ' -phase which also starts at around 1000°C.

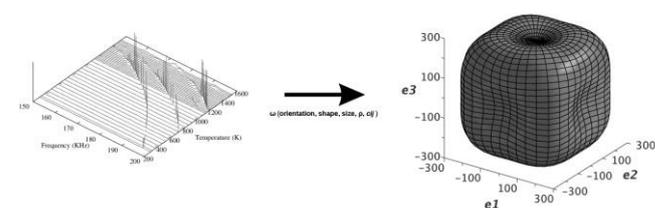


Fig.1: Section of experimental resonance spectra of CMSX-4 (left); Representation surfaces of the longitudinal elastic stiffness at room temperature (right).

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MS21 – Cutting Edge Techniques in Analysis and Preparation

MS21-T01

Current Status of the Liquid-Metal-Jet X-ray Source Technology

B. Hansson¹, O. Hemberg¹, T. Tuohimaa¹, M. Otendal¹, P. Takman¹

¹Excillum AB, Kista, Sweden

High-end x-ray diffraction and scattering techniques such as high-resolution XRD, protein crystallography, and SAXS rely heavily on the x-ray source brightness for resolution and exposure time. Traditional solid or rotating anode x-ray tubes are typically limited in brightness by when the e-beam power density melts the anode. The liquid-metal-jet technology has overcome this limitation by using an anode that is already in the molten state.

We have previously demonstrated prototype performance of a metal-jet anode x-ray source concept [1-3] with unprecedented brightness in the range of one order of magnitude above current