15-Crystal Growth

$BaO-B_2O_3$ flux, whereas Mo ions were detected 1 - 3 at% in the crystals grown from a modified K_2O-MoO_3 flux. The orientation of the seed crystal affected crystal growth and morphology. In the case of seeding in the <10$\bar{1}$> direction, twins with the twin law of 3-fold rotation axis were usually formed. Untwinned single crystals could be grown by seeding perpendicular to the <10$\bar{1}$> direction. The crystal faces {010}, {111}, {11$\bar{1}$}, {001}, {021}, {102} were well-developed in the untwinned crystals. The changes of morphology according to the growth conditions were investigated. Optical and electron microscopic observation of the striation and crystal defects were carried out.

PS-15.02.06 ATTEMPTS TO GROW DELTA-CRYSTALS FROM BINARY SYSTEMS BY ZONE-MELTING. By W. Uebach, H. Bradaczek and G. Hildebrandt, Institut für Kristallographie, Freie Universität Berlin, Germany.

Delta-crystals, i.e. bulk crystals with an intrinsic continuous change in lattice spacing of at least 0.1% per centimeter, would open a new field in X-ray and neutron optics. A crystal grown from two substances which form the same lattice and have perfect miscibility will in general have lateral as well as axial changes in lattice parameters when grown by directional solidification of the melt. Almost all crystal growers try to avoid this well-known effect, we, however, tried to enhance it by subsequent zone melting and zone crystallization with the system lead chloride and bromide. For this purpose we constructed a programme controlled furnace made of fifty independent heater elements allowing for a wide range of temperature regimes. Temperatures around 500 °C could be stabilized within less than 1°C. A fine grain carbon crucible, about twelve centimeters high and protected by a silica tube was put in vertical position. A two centimeter melting zone was run from bottom to top without any external movement. All possibilities for preheating and annealing were given.
The main difficulty was that the crucibles used to crack when the crystal tip at the bottom was heated for a second time. It was observed in these cases that in the first zone run the salts had obviously reacted with the crucible walls and thus did not give way for the thermal expansion of the crystal tip in the second run. Furthermore the compositional change from one crystal end to the other was not by far as high as expected from model calculations. These calculations, however, were made under the assumption that in every stage of the growth the melt zone was completely homogenious. A control of this assumption seems impossible.
Until now our best results stem from a directional solidification experiment with the system potassium and rubidium chloride showing a lattice parameter difference of 0.4% over a distance of four centimeters. The quality of this single crystal was encouraging, the 'delta', however, could have been as high as 4% - theoretically.
New attempts will be made with the well-known and widely investigated system indium and gallium antimonide.

PS-15.02.07 CLUSTER FORMATION IN THE $LiNbO_3$ MELT BEFORE SOLIDIFICATION by P. Andonov*, CNRS-LMMM, Bellevue, Meudon Cedex, France; S. Kimura and T. Sawada, NIRIM, Tsukuba-shi, Ibaraki, 305 Japan

Structural analysis of the $LiNbO_3$ melt has been carried out by means of the Small Angle X-ray Scattering (SAXS) using the synchrotron radiation in the range of momentum transfer 0.020 $Å^{-1} \leq k \leq 1.400\ Å^{-1}$. Formation and evolution of the scattering particles were studied from 1673 K to 1513 K including a large undercooling domain. Their radius of gyration, size, shape and distance have been determined. Microclusters are present in all the T-domain and their number is sufficient to give an interference effect. By lowering the temperature, their size increases and macroclusters appear from 1550 K onwards. Previously, it was verified, in the same T-domain, that octahedrally coordinated niobium atoms exist in the melt as a fundamental local structure. So cluster models have been built using octahedral NbO_6 molecules. Single molecules could represent the smallest particles at high temperature. Dimers, chains of at most three or four NbO_6 octahedra and blocks of 2x2x1 NbO_6 octahedra could appear below 1670 K and subsist up to the solidification with also blocks having 3x2x1 NbO_6 octahedra and regular or deformed small aggregates constituted by two layers of four corner-shared niobium oxide molecules bonded by a lithium atom. Macroclustering due to a regrouping of microclusters explain the rapid increase of the viscosity. The numbers and the volumes of different particles were obtained from viscosity data and SAXS measurements. This cluster analysis using both determinations, is a very effective tool to study the nucleation and the clustering in the domain of the pre crystallization.

PS-15.02.08 CRYSTAL GROWTH OF $KTiOPO_4$ AND X-RAY TOPOGRAPHIC OBSERVATION. By J. H. Kim and S. J. Chung, Department of Inorganic Materials Engineering, Seoul National University, Seoul, 151-742, Korea.

Single crystals of potassium titanyl phosphate($KTiOPO_4$:KTP) were grown by top-seeding and slow cooling technique using $3K_2WO_4 \cdot P_2O_4$ as flux. Seeding was in the <010>, <001> and <011> direction, respectively, and the seed crystals were placed on the surface of the solutions for better observation and control of the growth process. Cooling rates were slower than 0.05℃/h at the beginning of growth and gradually increased up to 0.2℃/h. Inclusion-free crystals up to $30x40x40mm^3$ in size could be obtained by 3 weeks growing time. To obtain large crystals from a given crucible size, seeding in the <010> direction seems to be most suitable.

Grown crystals seeded in the <010> direction exhibit a slightly different morphology from that reported by L.K. Cheng, J.D. Bierlein and A.A. Ballman, J. Crystal Growth, 110, 1991, 697. The cleavage face of (200) is less developed and in some cases the (110) face appeared. The variation in crystal form arises from seed locations and orientations.

The structural quality of the slice cut from $KTiOPO_4$ crystal was studied by X-ray topography and electron microscopy. Dislocations, growth sector boundaries, inclusions, and growth striations were observed.

