

PS16.05.03 PROTEIN CRYSTAL PERFECTION AND THE CRYSTAL GROWTH PROCESS. E.H. Snell, J.R. Helliwell, A. Cassetta, T.J. Boggon, Chemistry Department, University of Manchester, Manchester, M13 9PL. UK, N.E. Chayen Biophysics Section, The Blackett Laboratory, Imperial College, London SW7 2BZ. UK, E. Weckert, K. Holzer, K. Schroer, Institut für Kristallographie, University of Karlsruhe (TH), Kaiserstrasse 12, Postfach, 6980 D-76128, Karlsruhe. Germany, V. Stojanoff, D.P. Siddons, NSLS, Brookhaven National Laboratory, Upton, NY 11973. USA

A program of work is underway involving the improved characterisation of protein crystals, grown in microgravity and on earth, and the monitoring of the process of crystal growth. Characterisation techniques include detailed mosaicity and topography measurements, which has involved use of SRS, ESRF and NSLS synchrotron X-ray facilities. Monitoring of the crystal growth process is being done via CCD video and interferometry (the latter to follow refractive index changes). Assessment of microgravity versus earth based methods is made possible by use of ESA's APCF flown on the NASA Space Shuttle. Improved quality of lysozyme crystals grown in microgravity (by dialysis) has been demonstrated. Apocrystacyanin C1 (grown by vapour diffusion) shows an improvement but not as marked. Comparison of methods of crystal growth, including CCD video monitoring of motion of crystals suggests that dialysis methods are superior to vapour diffusion for microgravity crystal growth. A depletion zone around each growing apocrystacyanin C1 crystal is seen in the video data. The X-ray diffraction characterisation can be summarised via plots of mosaicity versus peak reflection height which allow sensitive diagnosis of improved perfection. Correlation of topographic mosaic block size can be made with the rocking curves of the microgravity crystals. The combination of diagnostic techniques and perfection analyses is giving better insights into the optimum conditions for protein crystallization for the realisation of the best signal- to-noise ratio in X-ray reflection intensity measurements.

Crystal Growth From Solution And Melt

PS16.06.01 NEW CRITERION OF QUANTITATIVE ESTIMATE OF THE CORRELATION OF SURFACE AND VOLUME PROCESSES DURING CRYSTAL GROWTH FROM SOLUTION. A.M. Askhabov, Inst. of Geol., RAS, Syktyvkar, Russia

Criterion is based on real concentration conditions, taking place at the time of crystal growth on boundary layer. The criterion is introduced as a relation of surface σ_s and volume σ supersaturation, i.e. $\eta = \sigma_s / \sigma$, (1). If $\sigma_s = \sigma$ then $\eta = 1$, and crystal growth is fully controlled by the surface processes. If $\sigma_s = 0$ then $\eta = 0$, and growth regime is fully diffusion. Both extreme cases do not take place in real life and always $0 < \eta < 1$. Quantitative character of the criteria η makes it comfortable to use it for comparison of growth, regimes of different crystals, growing in different conditions, but in the same supersaturation of solutions. In fact η is a dimensionless criterion of similarity, characterising the relative role of surface and volume processes. The unknown supersaturation on the growing surface σ_s , necessary for calculation of η , for layer crystal growth is given by the following expression $\sigma_s = \sigma^* [(1 + 4\sigma/\sigma^*)^{1/2} - 1]/2$ (2). Substituting (2) into (1) we get that $\eta = \sigma^* [(1 + 4\sigma/\sigma^*)^{1/2} - 1]/2\sigma$, or transforming $\eta = (1 + \eta\sigma/\sigma^*)^{-1}$ (3). In the expressions (2) and (3) constant for given conditions of growth parameter $\sigma^* = K_d \rho / \beta C_0$, where K_d - coefficient of mass-transfer, ρ - crystal density, β - average kinetic coefficient, C_0 - equilibrium concentration.

Parameter σ^* directly defines out of experimental dependencies of growth rates on supersaturation in the volume of solution. From the same dependencies $V(\sigma)$ we find K_d , through which hydrodynamic conditions influence on growth regime. This approach was used for establishment of growth regime of crystals, growing from water solutions. Calculation kinetic data for some crystals, growing in different conditions, the role of surface and volume processes is comparable in all the range of reasonable supersaturation.

PS16.06.02 SEGREGATION OF Ce IMPURITIES IN KMgF₃ SINGLE CRYSTALS. O. Bouzanov*, M. D' Elena, C. Sanipoli, A. Scacco, Dipartimento di Fisica, Università La Sapienza, P. le A. Moro 2, 00185 Roma, Italy

Effects of impurity segregation in the growth of single crystals from the melt have been quantitatively determined in several ionic systems. In general, evaluation of segregation coefficients is experimentally achieved by measuring with appropriate techniques the distribution along the growth direction of the dopant species and making use of, the normal freeze equation. No satisfactory information has been obtained up to now about segregation of impurities in the growth of single crystals of perovskite-like compounds, which turned out recently to be attractive as active media for solid state laser or radiation dosimetry applications. In this work, results concerning the segregation of Ce impurities in the growth of KMgF₃ crystals are reported. All samples were obtained from the melt with the Czochralski method by using Pt crucibles in an inert atmosphere. A very detailed study has been carried out on the distribution of the dopant, added as CeF₃ to the melt formed by stoichiometric mixtures of KF and MgF₂, as a function of the growth rate and of the initial concentration in the liquid phase. The impurity concentration has been determined from the measurements of the optical absorption spectra in the ultra-violet region. The data show that the dopant concentration increases with the grown volume in all crystals, indicating for the effective segregation coefficient a value lower than 1. A quantitative determination of such a value is under way.

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PS16.06.03 CRYSTAL GROWTH AND CHEMISTRY OF RARE EARTH ALUMINUM BORATES. D.Y. Choi, S.T. Jung, S.J. Chung, Dept. of Inorganic Materials Engineering, Seoul National Univ., Seoul 151-742, Korea

The crystal structures of rare earth aluminum borates with the general chemical formula of $\text{ReAl}_3(\text{BO}_3)_4$ (Re=Y, Gd and Nd) are well known as the isostructure of huntite. Several single crystals of these borates and their solid solutions were grown by top seeded solution growth (TSSG) technique. The space group of grown crystals could be confirmed as R32 for $\text{YAl}_3(\text{BO}_3)_4$ (YAB) and C_2/c for $\text{NdAl}_3(\text{BO}_3)_4$ (NAB), irrespective of their growth conditions. But the space group of $\text{GdAl}_3(\text{BO}_3)_4$ (GAB) grown above 1030°C could be determined as C_2 whereas the space group of this crystal grown at about 1000°C is R32. The phase diagrams of YAB-NAB and GAB-NAB were determined by x-ray powder diffraction and differential thermal analysis. The structural relations between these borates and the huntite could be explained with their unit cell transformations. The growth forms of YAB and GAB with the space group of R32 are composed of six rhombic and six prismatic planes. The morphological changes of NAB crystal were dependent on the flux compositions. X-ray topography of the grown crystals were carried out and the optical and laser properties of the prepared specimen were examined.