15-Crystal Growth

PS-15.02.15 HOW CAN WE SOLVE PROBLEMS OF CRYSTAL GROWTH OF ORGANIC NLO MATERIALS ?-SELECTION OF METHODS, TEMPERATURE AND SOLVENTS FOR ORGANIC NLO CRYSTAL GROWTH. By Chaoguo Wang, Beijing Institute of Technology, China

High quality organic crystals are needed for X- ray diffraction structure analyses and for use as nonlinear optical devices. Current routine and nonroutine methods of growing organic crystals are discussed and the influence of temperature and organic solvents on the character of organic NLO crystal was examined experimentally. Growing method and conditions of MONA, MONDA, MMONS, NMDA, POM according to their physical and chemical properties are made and those are more suitable for organic NLO materials. In actual practice a growth method can not give suitable for difference organic crystals. Alternatives of growth method are according to the crystal chemistry and properties of the particular compound. Several examples from our recent research work serve to illustrate this point. The organic NLO materials have usually hyperpolar molecule, but most typical organic solvents are a dipole moment less than about 3 Debye. Therefore, The ideal solubility and similar dipole solvent is difficulty selected to growing large three-dimensional crystals can be expected to grow in the solution. The nonpolar solvents tend to form lowly dimensional crystal, and a polar solvents favors formation of bulk crystals. Many organic compounds usually melt below 200 ℃ and char at high temperature. Proposal of growing method is selected by thermal example DSC and optical request. As crystal growth practitioners we know that the art of crystal growth is much more highly developed than the science much work need to be done to improve existing methods for organic crystal preparation. The effort needs to be interdisciplinary involving organic chemists, crystallographers and materials scientists. As scientists, we believe that crystal growth mechanisms are rational and ultimately under- standable. Innovative, interdisciplinary approaches are needed to make advances in this field, and the future utility of organic solid as technologically important materials depends on such advances(Author, G. Zhang et al., J. of Crystal Growth 1990, 100, 411-415).

PS-15.02.16 **THE STUDY ON A NEW ACOUSTO-OPTIC MATERIAL---LEAD BROMIDE CRYSTAL.** By Chen Fusheng, Institute of Crystal Materials, Shandong University, Jinan 250100, China

Lead bromide crystal has become a very important Acousto-Optic (AO) material because of its excellent AO performance. A new Acoustic-Optic Tunable Filter (AOTF) made from the crystal has been used to fabricate Infrared Spectrometers which has been found wide applications.

Using directional crucible decreasing method, larger size up to φ20 * 60mm, colorless, transparnent lead bromide single crystal was grown, the growth rate is 1-2cm/day. Crystal growth experiments indicated that the orientation of seed crystal, G/V value (G: temperature gradient, V: decreasing rate) and the material purity were the major factors influencing the crystal quality. The appropriate G/V value ranged from 10 to 20, under this condition the solid -liquid interface appeared as a slightly convex or flat faces. Using [001] oriented seed crystal, it's easier to grow nearly perfect crystal, or the cracks will be serious in the grown crystal. The raw material purity should be higher than 99.999% to assure less scattering grains and better uniformity in the crystal.

The transmission range of the grown lead bromide crystal extends from 0.4 to 30 μm. X-ray powder diffraction pattern and Laue photography indicated the lead bromide belongs to mmm point group. The crystal defects and their causes were also studied.

PS-15.02.17 INVESTIGATIONS OF CONTROLLED REVERSION OF SOLID-LIQUID INTERFACE DURING GROWTH OF YAG:Nd CRYSTALS. By Xu Tianhua, Peng Weiqing, Zheng Qimeng, Huang Changmin and Xu Guangyu, Southwest Institute of Technical Physics, PO Box 238, Chengdu 610041, P.R. China.

There are four clearly differentiated states of liquid flows that can be found in various combinations of diameters and rotation rates of the crystals, namely, I. simple natural convection; II. coexistence of natural and forced convections with an asymmetric overcooling region caused by the "window effect"; III. coexistence of the same liquid flows but without that region and IV. simple forced convection. In general, states I and III last long, while state II is short; state IV occurs when the diameter of the crystal is large in comparison with the inner diameter of the crucible and when the difference in temperature is relatively small between the crystal and the crucible. In all mentioned transitions, namely, I → II, II → III and III → IV, abrupt changes in the diameter of the crystal and remelting of the growth interface to a certain extent were observed. Component supercooling and generation of dislocations occur frequently.

It is obviously undesirable to grow the crystals in states II and III. In order to grow in state III, it is necessary for the crystals to go through states I and II and two rapid transition processes which cause component supercooling and generate dislocations. In this connection, we have developed a method called "Contolled Reversion of Solid-LiquidInterface" (CRSLI), which allows the growth to go into state III immediately after a dislocation-free top has grown during state I, and thus to obtain crystals free from dislocations. The key to realize the "safe" transition from I to III is: 1. choosing a suitable position; 2. setting-up of a suitable thermal field; 3. keeping the crystal growth in a stable liquid flow rate; 4; not having the residue of the region of unstable growth appearing inevitably during the interface reversion.

When crystals are grown following this procedure, the growth process as a whole is not continuous in space and time, and thus has broken the normal procedure.

PS-15.02.18 COMPARISON OF THE GROWTH OF YAG:Nd CRYSTALS IN RESISTANCE AND RF HEATING SYSTEMS. By Xu Tianhua, Peng Weiqing, Zheng Qimeng, Huang Changmin and Xu Guangyu, Southwest Institute of Technical Physics, PO Box 238, Chengdu 610041, P.R. China.

We have grown YAG:Nd crystals in a graphite heating molybdenum crucible and in an RF heating iridium crucible, separately, and found that there is an appreciable distinction between them in the growth conditions (atmosphere, thermal field and liquid flow behaviours), the growth parameter control (stability of the diameter, permissible growth rate and doping level), as well as in the results on the growth of the crystal (external shape, surface morphology and absorption spectrum).

In the first case, when filled with argon (nitrogen cannot be used), the furnace was in a reductive atmosphere, the surface of the crystal was severely contaminated due to an interaction between graphite, molybdenum and the constituents of the crystal, and the crystal had an absorption peak near 3700 Å. In the second case, the furnace was in a neutral atmosphere (Ar and/or N_2), and the crystal had a wide absorption peak near 2600 Å. The above mentioned peaks can be eliminated by a long-term atmospheric annealing at a temperature ranging from 1200 to 1300°C. With regard to the setting-up of the thermal field, the first case usually shows a larger (or more suitable) regulating range of the longitudinal and radial temperature gradients, as well as a higher doping level and growth rate than in the second case. The doping levels (the atomic ratios in the starting materials) were larger than 5 % and ≈ 4 %, repectively, their pulling rates were 2.0 to ≈ 2.5 mm/hr and 1.0 to ≈ 1.3 mm/hr, respectively. Even in such conditions, the normal crystal/charge ratio for the first case (resistance

