A novel method of growing bulk single crystals

Organic single crystals of substituted benzene derivatives with high optical nonlinearties and low melting temperatures are very promising materials for future optoelectronic and nonlinear optical applications. However, it is difficult to grow large size organic single crystals. Recently, Sankaranarayanan of Alagappa University, Karaikudi and Ramasamy of Anna University, Chennai[1], have successfully grown a large size single crystal of benzil having hexagonal facets (Figure 1) by a novel seeding method using a microtube and based on Czochralski pulling technique. The conventional Czochralski technique involves three steps, viz. melting the source material, seeding the melt and pulling the crystal. In this recent study, an attempt is made to seed the melt with a stainless steel microtube of 6 μ ID. Due to capillary rise, a fine column of melt is crystallized inside the microtube which is used as a primary seed. In their experimental set-up (Figure 2), the material is melted in a static glass crucible, which is kept inside an independently controlled two-zone Khanthal wire-wound furnace and the microtube is dipped in the melt. The

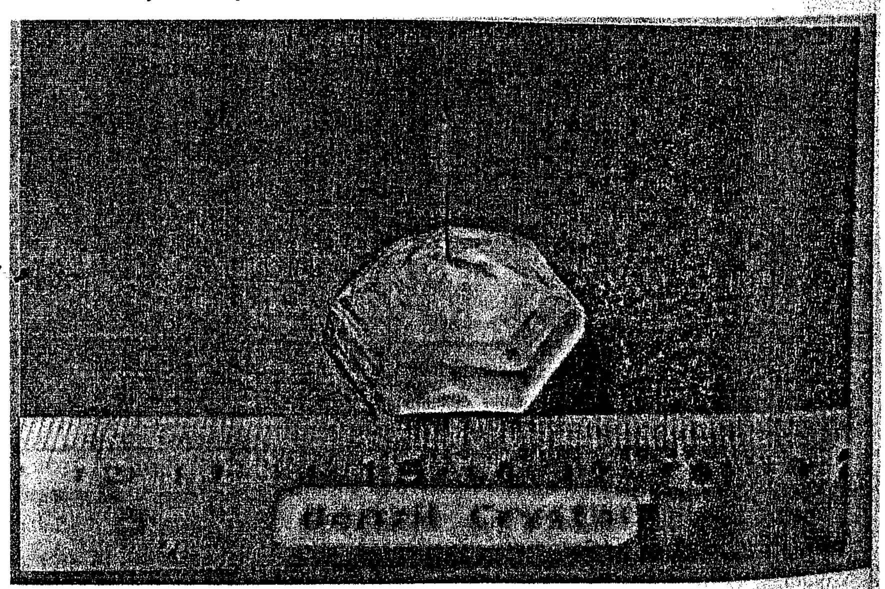

Figure 1. Benzil single crystal 'C' with the microtube as a seed. Reprinted from *Journal of Crystal Growth*, vol. 193, K. Sankaranarayanan and P. Ramasamy, 'Microtube-Czochralski technique (μT-Cz): a novel way of seeding the melt to grow bulk single crystal', pp. 252–256, © 1998, with permission from Elsevier Science.

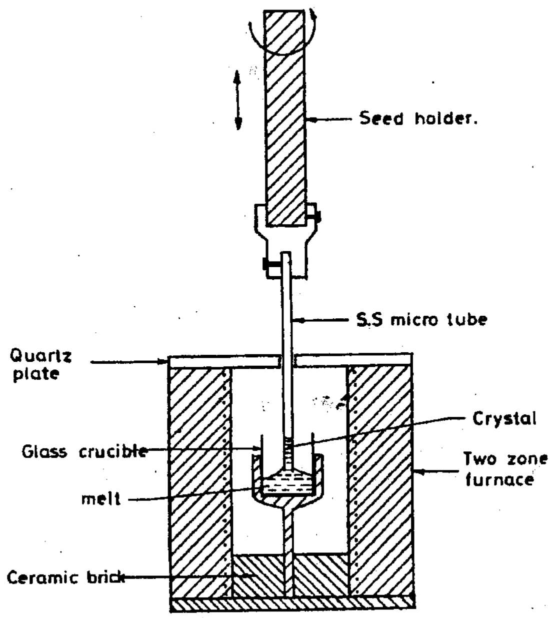

Figure 2. A schematic diagram of the experimental setup. Reprinted from *Journal of Crystal Growth*, vol. 193, K. Sankaranarayanan and P. Ramasamy, 'Microtube-Czochralski technique (μT-Cz): a novel way of seeding the melt to grow bulk single crystal', pp. 252–256, © 1998, with permission from Elsevier Science.

nucleation inside the tube is controlled by the following experimental parameters: radius and rotation rate of the microtube, melt temperature at which the microtube seeding is made (t_{ms}), pulling rate, axial temperature gradient and the length of the microtube underneath the melt surface (l_{ums}). The authors have reported three growth runs corresponding to l_{ums} = 0.5, 1 and 1.5 mm. Once the growth run is completed, the system temperature has to be reduced at the rate of 1°C/h. Any rapid cooling results in a crack in the crystal grown. For l_{ums} = 1.5 mm and with modified axial gradient a benzil single crystal with high quality transparency, having near planar crystal–melt interface is obtained. The crystal pulling rate is maintained at 1.2 mm/h at t_{ms} = 90°C. The crystal rotation rate is 6 rpm. There also exists a critical rotation rate relative to the radius of the microtube. Only below this rate, the change in crystal rotation is influential in deciding the morphology of the resultant crystal. They have also analysed the effect of l_{ums} on the nucleation. If the melt level and the crystal orientation inside the tube along two sides are the same then the value of l_{ums} is not vital. If the crystal orientation along two sides of the tube are different then the value of l_{ums} plays a crucial role. The value of l_{ums} should be sufficient enough to allow any one of the orientations at the end of the microtube as the deciding crystal orientation. This technique is viable to grow a bulk single crystal from the melt without a pre-grown-seed and the reproducibility for getting single crystals is about 80%.

1. Sankaranarayanan, K. and Ramasamy, P., *J. Crystal Growth*, 1998, **193**, 252.

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