

**s7.m4.p1** Vapor transport of organic thin films in microgravity and unit gravity. M. Ittu Zugrav<sup>1</sup>, W.E. Carswell<sup>2</sup>, G.B. Haulenbeek<sup>2</sup> and F.C. Wessling<sup>3</sup>, <sup>1</sup>Center for Microgravity and Materials Research, <sup>2</sup>Alliance for Microgravity Materials Science and Applications, <sup>3</sup>Department of Mechanical and Aerospace Engineering, University of Alabama in Huntsville, Huntsville, AL 35899, USA.

Keywords: NLO material, thin film, vapor transport.

This work is specifically focused on explaining previous results obtained for the crystal growth of an organic material in a reduced gravity environment. On STS (Space Transportation System)-59, in April 1994, aboard the USA Space Shuttle Endeavour, two experiments were conducted with N,N-dimethyl-p-(2,2-dicyanovinyl) aniline (DCVA), a promising nonlinear optical (NLO) material. The space experiments were set to reproduce laboratory experiments that yielded small, bulk crystals of DCVA. The results of the flight experiment, however, were surprising. Rather than producing a bulk single crystal, the result was the production of two high quality, single crystalline thin films. Repeated attempts on the ground to reproduce these results were fruitless.

A second set of flight experiments was conducted on STS-69 in September 1995. This time 8 DCVA experiments were flown, with each of seven experiments containing a slight change from the first reference experiment. The reference experiment was programmed with growth conditions identical to those of the STS-59 mission. Changes were made to the temperature profiles, the seed and the mounting technique, and the nature of the substrate. Once again the results were surprising. In all eight cases oriented and ordered thin films were grown again, albeit with varying quality.

The space growth parameters and samples were analyzed and the results were used to guide a systematic terrestrial investigation through the optimization and control of the growth cell orientation to the gravity vector, substrate temperature, deposition rate, and background pressure. We have extended our work to include vapor pressure measurements for the evaluation of the experimental transport situation, nitrogen background pressure variation during the heat-up and steady-state growth, thermal profile variation during the heat-up period, heat-up rates variation, supersaturation modification during the steady-state growth, and the substrate material variation.

We have established two thresholds behind which we must stay in order to grow DCVA thin films on the ground. The first is a threshold for background nitrogen pressure below which bulk crystals grow rather than thin films. The second is related to the limiting conditions in terms of temperature. The successful development of the ground thin film technology is a direct result of information gleaned from experiments conducted in microgravity.

**s7.m4.p2** Automated crystallization of polar biomolecules in the metastable regime using microfluidic silicon devices and a variable temperature incubator. R.E. Cachau<sup>1</sup>, J.R. Casas-Finet<sup>2</sup> and A. Sanjoh<sup>3</sup>. <sup>1</sup>Advanced Biomedical Computing Center and <sup>2</sup>AIDS Vaccine Program, SAIC Frederick, NCI-FCRDC, Frederick, MD 21702-1201, USA (cachau@ncifcrf.gov); <sup>3</sup>Protein Wave Co. Ltd. 1-16-5 Nishi-tomigaoka, Nara, Nara 631-0006, JAPAN.

Keywords: instrumentation, techniques for crystallisation.

Protein crystal growth studies are a demanding task, which frequently involve the use of variable chemical composition settings used to factor the best combination of conditions for crystallization. Large amounts of material are frequently involved in these settings. An additional disadvantage of these approaches is that, for the most part, the successful crystallizations will take place in the supersaturated regime with precipitation competing with nucleation. Ideally, we would hope for a setting where the nucleation will take place in the meta-stable region (similar to a seeding experiment), thus minimizing precipitation, and where the crystallization conditions are electronically controlled instead of chemically controlled. We explore one such setting by combining an incubator with highly accurate control over temperature ramps to reach the metastable regime while inducing nucleation using microfluidic silicon devices. Although for the moment the silicon devices offer a discrete number of combinations, the results obtained suggest the scan of the temperature-voltage regime as a simpler more economical and reliable way to explore nucleation conditions and obtain crystal growth around the metastable regime.