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CONCLUSIONS

The characterization of convection patterns around a crystal growing from its aqueous solution is the subject of the present discussion. The study has been conducted using three laser measurement techniques that rely on the variation of refractive index with density, and hence the solutal concentration. These techniques have been validated in a thermal buoyancy experiment in a rectangular cavity. Subsequently, they have been used to study convection patterns around the growing crystal for varying process parameters. While images contain line-of-sight averaged information in a vertical plane, the possibility of reconstructing concentration contours on horizontal planes has been studied *via* tomography. Experiments reported in the study show that convection does influence crystal growth rate as well as its quality. The laser measurement techniques succeed in capturing the solutal concentration field (or its derivatives) around the crystal. Hence they can be used for online process control. Specific conclusions arrived at are given in the following paragraphs.

Comparison of optical techniques

Three refractive index-based optical techniques, namely interferometry, schlieren and shadowgraph have been applied to image the convective flow field in a differentially heated rectangular cavity. Air and water have been considered as working fluids. Experiments have been conducted over a wide range of temperature differences. The following conclusions have been arrived at in the present work.

1. In low temperature gradient experiments, all the three techniques correlate well with one another. Interferograms are limited by few fringes in air, and too many fringes in water. The shadowgraph image does not show sufficient contrast for analysis. In this respect, the schlieren technique is most amenable to data reduction.
2. In high gradient experiments, both schlieren and shadowgraph yield clear images. The interferograms are however corrupted by refraction errors. Schlieren and shadowgraph track the temporal response of the fluid medium in the form of the light intensity variation.
3. In high Rayleigh number experiments with water, the flow field is turbulent. Shadowgraph images are seen to be meaningful, as against interferograms and schlieren. They reveal a considerable amount of physical information, including boundary-layers, plumes and time scales.

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Optical techniques applied to crystal growth

A direct comparison of interferometry, schlieren and shadowgraph in the context of growth of optical crystals from an aqueous solution has been conducted. The three techniques reveal similar trends in terms of the spatial distribution of concentration gradients in the vicinity of the growing crystal. The initial dissolution and unsteadiness, a stable and symmetric growth regime and the final stratification of the solution are brought out. The free convection crystal growth experiment is thus limited in the long run by the localization of high concentration gradients around the growing crystal. The stratification here is stable and suppresses fluid motion. This limits the size of the crystal that can be grown. Based on the ease of instrumentation, image clarity and simplicity of data analysis, the laser schlieren technique emerges as better suited for process control, as compared to interferometry and shadowgraph.

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Influence of process parameters on crystal growth

The dependence of the convection patterns, concentration gradients and the crystal growth rate on ramp rate, rotation and crystal size is examined. Laser schlieren technique is employed as the measurement tool for determining the concentration distribution in the solution. Major conclusions to emerge from the study are:

1. The stable growth regime of the crystal at 0 rpm comprises symmetric buoyancy-generated convection plumes. When rotation is imparted to the crystal, the stable growth regime reveals a helical flow path.
2. Buoyancy-driven convection intensifies with increasing size of the crystal as well as the ramp rate. It is ultimately limited by salt depletion and solute stratification in the growth chamber that bring the fluid particles to rest. Crystal rotation diminishes the concentration gradients, equalizes them over all faces, and improves the symmetry of the concentration distribution in the solution. This leads to a crystal of better quality. The rpm of the crystal required to improve crystal quality depends on the cooling rate of the solution.
3. Over the range of parameters studied, the large-scale convection pattern is buoyancy-driven, even when the crystal is rotated.
4. Growth rates of the crystal correlate with the magnitude of the concentration gradients over its individual faces. The gradients in turn respond to the convective field around the crystal.
5. Experiments with crystals of various sizes immersed in an aqueous solution show that the convection transients are short-lived. Thus, the growth process is quasi-steady and can be controlled by adjusting the ramp rate and crystal rotation.

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Three dimensional reconstruction

Concentration distribution around a growing KDP on selected horizontal planes is reported. Partial images recorded at various view angles have been analyzed using the convolution back projection algorithm. A suitable extrapolation scheme has been employed to generate information about the entire measurement volume using the partial projection data. The performance of the extrapolation scheme has been assessed by reconstructing the numerically simulated three-dimensional temperature field in an axisymmetric differentially heated fluid layer. Steady crystal growth experiments have been conducted in two regimes that respectively are dominated by diffusion and buoyancy. The following conclusions emerge from the present study:

- The extrapolation technique used in the present work was seen to perform adequately with simulated data. In particular, no new artifacts were seen even when the original data set was as small as 30%.
- The concentration field in the vicinity of a crystal growing in a diffusion-dominated regime showed symmetry along the centerline of the beaker indicating a uniform deposition of solute on the crystal surfaces over a long period of time. The field in the bulk of the solution was entirely axisymmetric in the initial stages of the growth process.
- The transition from diffusion-dominated growth to the onset of convection induced temporary unsteadiness in the flow field that was accompanied by a breakdown of the symmetry of the concentration field.
- With an increase in the crystal size, the convection current was seen to increase in strength and thus determine the overall transport of solute from the bulk of the solution to the crystal surfaces. The geometry of the grown crystal fixed the orientation of the rising plume. Nearly symmetric distribution of concentration was realized in the stable growth regime in regions away from the crystal.

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NOMENCLATURE

C	Solute concentration, kmol of salt/kg of solution
ΔC_s	Concentration difference between two successive fringes
ΔC	Concentration difference between the saturated and supersaturated solution
D	Distance of the screen from the optical window (also diameter of the growth chamber), m
E_1	Absolute maximum temperature difference
E_2	RMS error
f	Focal length of the de-collimating mirror, m
g	Acceleration due to gravity, m^2/s
H	Height of the fluid layer (rectangular cavity), m (also, image size as seen through the optical window)
I	Intensity distribution (digitized)
L	Distance traversed by the laser beam through the test cell (also, crystal thickness in the vertical direction), m
N	Molar concentration of the solution, moles per 100 gm of KDP solution (also, total number of grid points on the reconstructed plane for tomography reconstruction)
Nu	Local Nusselt number defined as wall heat flux×cavity height/fluid conductivity
n	Refractive index of the fluid
n_a	Refractive index of the ambient
Pr	Prandtl number of the fluid, v/α
R	Spatial frequency in the CBP algorithm
Ra	Rayleigh number ($thermal = g\beta\Delta TH^3/v\alpha$)
Re	Rotational Reynolds number, $\omega L^3/v$
r	radial coordinate
s	Perpendicular distance of the data ray from the center of the object
t	Time, hr
T	Temperature, K
T_0	Reference temperature, K
ΔT_s	Temperature difference between successive fringes, K

$W(R)$	Filter function
x, y, z	Cartesian coordinates, with z parallel to the direction of propagation of light

Greek symbols

Subscripts

a, o	Reference value
c	Cold
h	Hot
$orig$	Original (temperature)
$recon$	Reconstructed (temperature)