The Lecture Contains:

EXPERIMENTAL DETAILS

The apparatus used for experiments of the present study are described in the following sections. In experimental procedure followed for growth of KDP crystals is also discussed.

CONVECTION IN A RECTANGULAR CAVITY

Experiments are reported for studying fluid convection in a rectangular cavity. The temperature difference maintained across the horizontal surfaces of the cavity leads to unstable density gradients in the fluid medium. In turn, fluid motion in the form of closed loops is initiated in the cavity. The configuration studied, called Rayleigh-Benard convection is a problem of fundamental as well as practical importance. The flow patterns associated with Rayleigh-Benard convection show a sequence of transitions, leading from steady laminar to unsteady turbulent flow. The strength of convection is quantified by the Rayleigh number, a dimensionless parameter. The Rayleigh number is a measure of the ratio of energy released in the buoyancy field to that lost by viscous dissipation. The values realized in the present experiments are quite large in comparison to the critical Rayleigh number of 1707 reported for a horizontal differentially heated infinite fluid layer at the onset of convection.

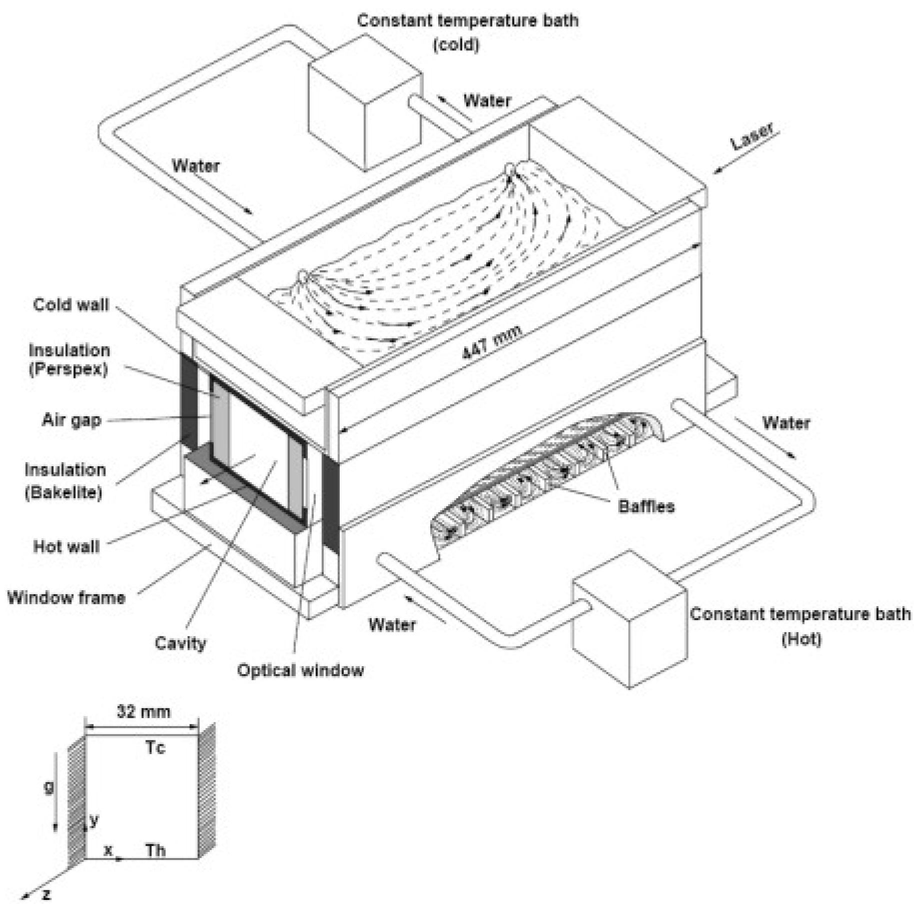

Figure 5.12: Schematic Drawing of the rectangular cavity experimental set up.

The apparatus used to study the buoyancy-driven convection in a horizontal fluid layer is shown schematically in Figure 5.12. The cavity is 447 mm long with a square cross-section of edge 32 mm. The test cell consists of three sections namely the top plate, the fluid layer enclosed in a cavity and the bottom plate. The top and bottom walls of the cavity were made of 3 mm thick aluminum plates. The flatness of these plates was manufacturer-specified to be within $\pm 0.1 mm$ and was further improved during the fabrication of the apparatus. The central portion of the experimental apparatus is the test section containing the fluid medium. The side walls of the cavity were made of a 10 mm Plexiglas sheet. In turn the Plexiglas sheet was tightly wrapped with thick bakelite padding in order to insulate the test section with respect to the atmosphere. The height of the test section was 32 mm and was measured to be uniform to within $\pm 0.1 mm$. A window was provided in the direction of propagation of the laser beam. It was held parallel to the longest dimension of the cavity for recording the projected convective field in the form of two-dimensional images. The apparatus was enclosed in a larger chamber made of thermocole to minimize the influence of external temperature variations. The room temperature during the experiments was a constant to better than $\pm 0.5°C$ over a 10-12 hour period. Experiments were initiated by flowing water over the hot and cold surfaces respectively. After the start of the experiments, the surfaces reached a steady state within a few minutes. The thermal fields in air evolved over a longer period of time and stabilized over 5-6 hours. In water, dynamically steady patterns were realized in 1-2 hours. The thermally active surfaces were maintained at uniform temperatures by circulating a large volume of water over them from constant temperature baths. Temperature control of the baths was rated as $\pm 0.01°C$ at the cavity location, direct measurements with a multi-channel temperature recorder showed a spatial variation of less than $\pm 0.1°C$. For the upper plate, a tank-like construction enabled extended contact between the flowing water and the aluminum surface. Special arrangements were required to maintain good contact between water and the lower surface of the plate. Aluminum baffles introduced a tortuous path to flow, thus increasing the effective interfacial contact area.

For air, the temperature differences applied across the cavity walls were in the range of 5-50 K. These correspond to Rayleigh numbers of 14×10^3 *to* 14×10^4. In experiments with water, the temperature differences applied were in the range of 3-13 K. These correspond to Rayleigh numbers of 25×10^5 *to* 13.5×10^6 respectively. The Rayleigh numbers realized in the experiments can be seen to be quite large in comparison to the critical value of 1707, indicating the presence of strong fluid motion in the cavity.

Previous Next

CONVECTION AROUND A GROWING CRYSTAL

The apparatus in which experiments have been conducted on convection around a crystal in a top-hanging arrangement is discussed. The apparatus in which the crystal is held over a platform is described in the section titled Crystal Growth Chamber For Tomographic Projections.

Experiments that compare images of convection around the crystal recorded by interferometry, schlieren, and shadowgraph have been conducted in a growth chamber made of glass (17 cm diameter and 22 cm height) as shown in Figure 5.13. Glass used for the experimental chamber has the advantage of being a smooth surface on which unwanted nucleation cannot take place. In addition, glass is chemically non-reactive towards the KDP solution. The crystal growth chamber is in turn, placed in a Plexiglas tank. Temperature of the solution inside the growth chamber is maintained at prescribed levels by thermostatically controlled water circulating in the outer tank. The temperature of water is stabilized in the outer tank to within $\pm 0.1°C$ with the help of a programmable temperature controller (*Eurotherm*). A K-type thermocouple wire fixed to the outer surface of the growth chamber provides the feedback to the controller. With this arrangement, ramp rates could be applied to cool the solution over long periods of time. For example, a linear change in temperature of the solution from $36\ to\ 25°C$ over a period of 60 hours was closely realized in the present set of experiments. Sufficient insulation is provided on the outer surface of the entire assembly to reduce thermal interaction between the growth cell and the ambient.

For visualization of the concentration field by the schlieren technique, circular optical windows (*Stranda*, BK-7, 60 mm diameter, 5 mm thickness, $\lambda/4$ flatness) have been fixed on the walls of the growth chamber at opposite ends. The growth process is initiated by introducing into the growth cell a spontaneously crystallized seed crystal fixed at one end of a thin glass rod. The glass rod (diameter 3 mm) is small enough not to disturb the convection plume rising above the crystal. Rotation to the growing crystal is imparted with the help of a stepper motor (12V, 0.6A, *RS components)*.

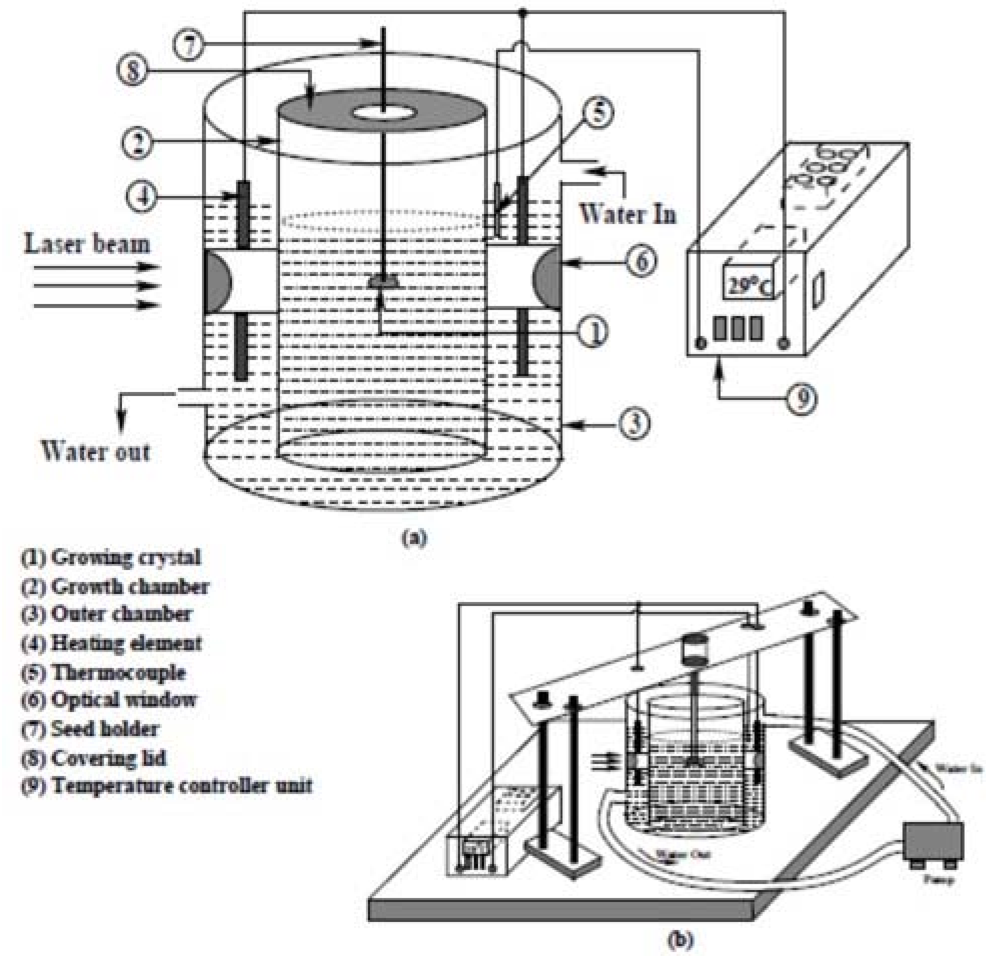

Figure 5.13: (a) Schematic diagram of the crystal growth chamber; and (b) Complete assembly.

Previous Next

For experiments employing interferometry, a double-walled compensation chamber is placed in the path of the reference beam. In order to ensure identical conditions in terms of the undisturbed optical path length, the volumetric capacities of the compensation chamber and growth chamber are kept equal. The inner cell of the compensation chamber contains the KDP solution that has a salt concentration corresponding to saturation conditions at the ambient temperature of $25°C$. In the growth process, the temperature range utilized is $25°C\ to\ 36°C$ and no deposition was seen in the experiments anywhere in the cell. The solution in the test section and the compensation chamber are at practically identical temperatures throughout the duration of the experiment. This is accomplished by splitting the outlet of the water-circulating pump into two parts, one leading to the growth chamber and the other to the compensation chamber. In the absence of a crystal, the optical path lengths in the test and the compensation chamber could be balanced during the cooling of the solution. Consequently, there was no fringe formation (in the infinite fringe setting mode) and fringe deformation (in the wedge fringe setting mode) from either thermal disturbances or those related to small differences in concentration. Fringe formation in the presence of the crystal occurred due to salt concentration differences in the solution with respect to the average value in the compensation chamber.

In the present set of experiments, the growth of the seed crystal is initiated by slow cooling of the supersaturated solution. Two values of ramp rates have been employed: $0.05\ °C/hour\ and\ 0.1\ °C/hour$. The rate at which the solution is cooled plays an important role in controlling the strength of convection currents, and hence the growth rates and crystal quality. Low rates keep the degree of supersaturation to a small value. The growth process is then diffusion-dominated and is inefficient in terms of the size of the crystal grown. On the other hand, very high values of the ramp rate give rise to vigorous convection currents resulting into deterioration of the crystal quality. At intermediate cooling rates, high crystal quality is to be expected.

Stirring the solution reduces the natural convection-induced temperature oscillations by homogenizing the bulk solution. Hence the importance of optimum rates of rotation in crystal growth processes has gained recognition. The two most widely used stirring mechanisms are the rotation of the seed and/or the rotation of the crucible. In the present work, experiments have been conducted by continuously rotating the growing crystal at 15 rpm.

Previous Next

CRYSTAL GROWTH CHAMBER FOR TOMOGRAPHIC PROJECTIONS

Tomographic projection data are recorded by viewing the convection pattern at various angles over the range of $0 \ to \ 180°$.If the pattern is strictly steady, projections can be recorded by turning the crystal through small incremental angles. This approach has the drawback of creating unsteadiness in the flow field. Results obtained by turning the crystal have been discussed in the context of validating the tomographic algorithm with measured data. An appropriate technique is to keep the growth chamber fixed and turn the axis of the source-detector system. This route could not be followed in the present study owing to the physical size of the laser, CCD camera and the accompanying optical components. Instead, the growth chamber along with the crystal was turned through definite angles to image the concentration field. The curvature of the beaker led to ray-bending problems and prevented scanning of its entire width. The portion of the solution visible through optical windows alone has been used for measurement. Unlike the top-hanging arrangement of previous sections (where the supporting rod disturbed the convective plumes), experiments have been conducted here using a crystal mounted above a platform.

The crystal growth chamber used in the present experiment is shown schematically in Figure 5.14.

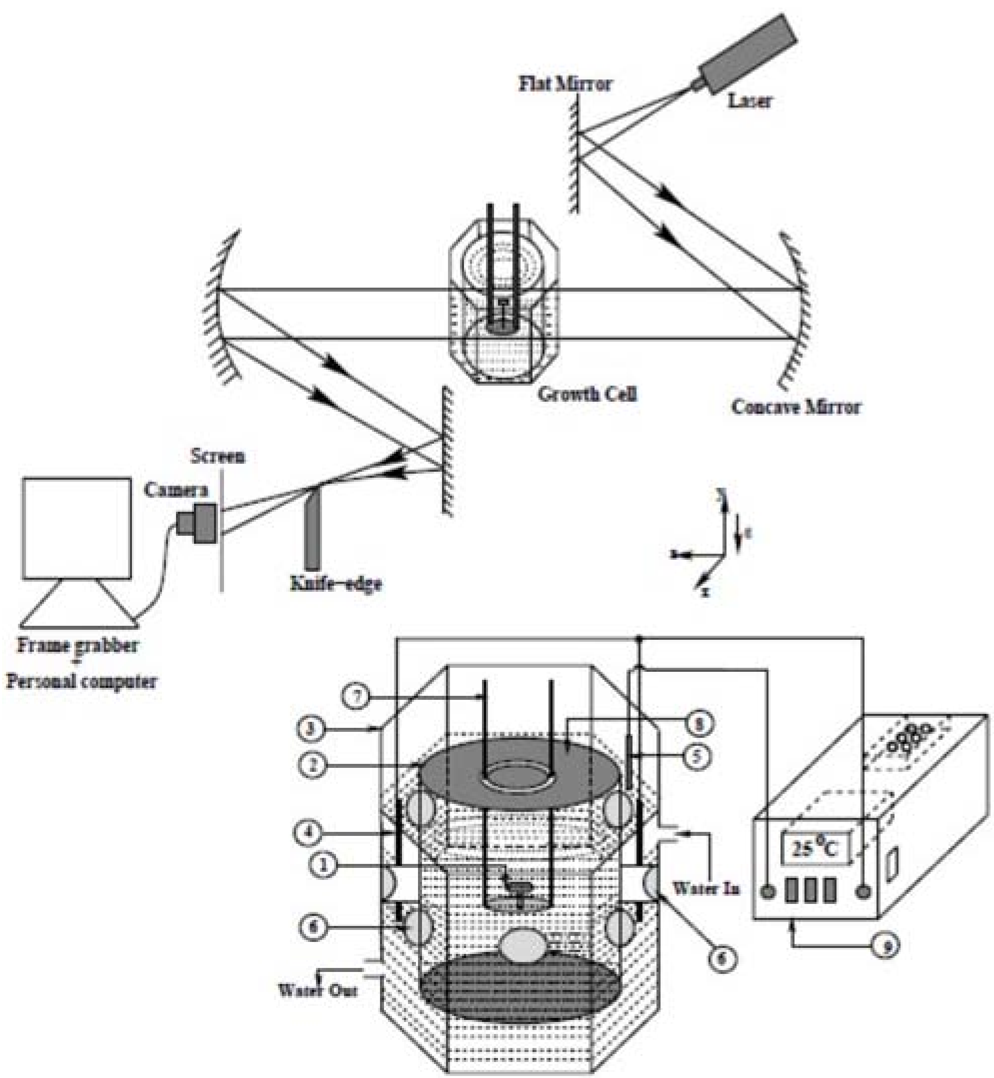

Figure 5.14: Schematic diagram of the four-view crystal growth chamber

placed in the path of the laser beam in a $Z-type$ schlieren set up. (1) Growing Crystal, (2) Growth Chamber, (3) Outer Chamber, (4) Heating element, (5) Thermocouple, (6) Optical window, (7) Seed holder (platform configration), (8) Covering Lid, (9) Temperature controller unit.

Previous Next

Module 5: Schlieren and Shadowgraph

Lecture 28: Crystal growth apparatus

Figure 5.14 comprises a glass chamber that holds the KDP solution, has a diameter of 16.5 cm with a height of 23 cm. For visualization of the concentration field by the schlieren technique, circular optical windows (BK-7, 40 mm diameter, 5 mm thickness, $\lambda/4$) are fixed on the glass beaker at opposite ends. A total of eight such windows permit passage of the laser beam for four view angles. Parallelism and straightness of the optical windows are crucial for generating meaningful images, and considerable precautions have been taken in this regard. The Plexiglas tank surrounding the growth chamber is octagonal in plan. It ensures large enough volume for the circulating water to keep the KDP solution at the required temperature level over a considerable period of time. Four heating elements placed diametrically opposite in the outer chamber maintain the temperature of circulating water, and hence the KDP solution. Electrical input to the heating elements is regulated by a programmable temperature controller (*Eurotherm*). As in the apparatus discussed in Convection Around A Growing Crystal (Figure 5.13) a K-type thermocouple wire fixed to the outer surface of the growth chamber provides the feedback to the controller. Uniformity of temperature within the solution is ascertained by recording temperatures at various locations using 26 gage K-type thermocouples. With this arrangement, it was possible to reduce the temperature of the solution from $36\ to\ 25°C$ linearly with time over a time frame of 60 hours.

The salt-solution has an initial temperature that is high enough to keep it from becoming fully saturated. A KDP seed crystal spontaneously crystallized in a second vessel is placed on a glass platform and introduced in the growth cell. When the bulk temperature is lowered, the solution becomes super-saturated with salt that in turn deposits on the crystal. The solution adjacent to the crystal is now close to being saturated with the dissolved salt. A crystal growing from its aqueous solution thus creates a three-dimensional solute distribution in its vicinity. The solutal concentration gradients and the accompanying gradients in density of the solution are responsible for the appearance and evolution of buoyancy-driven convection currents in the growth chamber.

Previous Next

EXPERIMENTAL PROCEDURE

The crystal growth experiments are performed in the following manner. A supersaturated solution of KDP powder is prepared in distilled, de-ionized water. The amount of KDP salt to be dissolved is determined from its solubility curve at an average temperature of $35°C$. The solution is stirred long enough to ensure complete dissolution of the solute. It is filtered using Whatman-100 filter paper to remove residual microscopic particles. The solution is maintained at relatively high temperature for about 4-5 hours using water circulation from the temperature control unit. Subsequently, the solution is cooled by applying a suitable ramp rate, $0.1\ °C/hour$ in the present study. When the solution reaches a value right enough for it to become saturated, a KDP seed is introduced into the solution using a thin glass rod. The orientation of the seed crystal is adjusted in such a way that the prismatic faces $(\{100\})$ are placed normal to the direction of the laser beam. Experiments are allowed to run for a considerably longer duration of time $(\approx 60\ hours)$ in order to allow the growth of large crystals. Precautions are taken to avoid external disturbances and vibrations that may introduce unsteadiness in the flow field. For the experiments discussed below, the crystal size increased from $0.3\ cm^3\ to\ 3\ cm^3$ in 60 hours, while preserving high crystal transparency.

Previous Next