2007

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Recommended Citation

Davidhazy, Andrew, "Applications of photography in distillation research" (2007). Accessed from
http://scholarworks.rit.edu/article/427

APPLICATIONS OF PHOTOGRAPHY IN DISTILLATION RESEARCH

by Andrew Davidhazy

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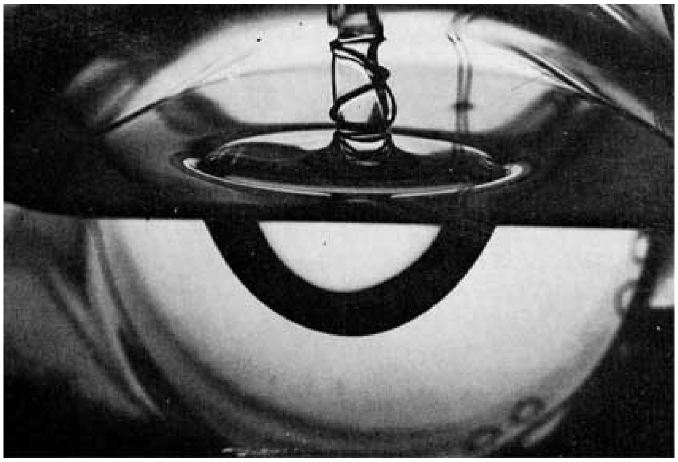
Figure 1. A 30 cc. water boule floating on water. There is a slight temperature difference between the two liquids. A narrow (25 micron) vapor gap or shroud separates the two liquids and is responsible for the black band outlining the shape of the boule below the surface.

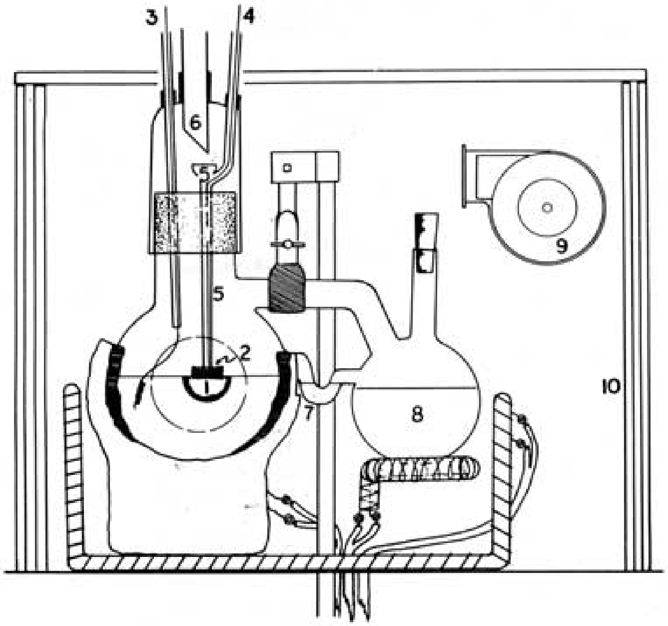

Figure 2. Boule growing and schlieren photography apparatus. Boule, 1,. is anchored to wire mesh stabilizer tube, 2, attached to applicator tube 5 which includes electrode 4. Second electrode 3 goes to support liquid. Vapor supply flask 8 provides a large amount of vapor, which is condensed by aerial condenser 6. There is a trapped overflow weir at 7; 9 is the air circulator and the glass walls of the thermal box are indicated at 10.

The Distillation Research Laboratory at RIT comprises a group interested in the behavior of liquids during evaporation and condensation. The main area of research is the study of floating liquid "boules"'. When a liquid, water for instance, is evaporated and the condensate returned to the superheated liquid, the drops of condensate will sometimes float. These drops are called boules (Figures 1 and 2). With time and a steady supply of condensate, boules grow up to a critical size when the vapor shroud that separates them from the bulk liquid suddenly breaks down and "sinks" the boule into the hot liquid below.

Typical questions which call for answers by means of optical and photographic techniques are: how small is the vapor gap that separates the boule from the support liquid? where and how fast does this gap break down? what does the merging of the two liquids look like? what are the characteristics of the liquid the boule floats on? All of the effects that call for photographic solutions are too fast, too small or otherwise visually imperceptible.

THE VAPOR GAP

In most experimental measurements the one factor that usually determines the eventual technique to be used is the extent to which the method of measurement interferes with the experiment. In the case of the boule all physical means of measuring the separation between the boule and the support liquid were impractical, as these would destroy the gap. Instead, a simple optical principle was applied. It indirectly gave an indication of the thickness of the vapor gap. A grain of wheat light bulb was attached to the end of a glass tube and submerged near the floating boule. An observer, looking through the flat windows of the boule flask, could see a reflection on the side of the boule of the bright monofilament.

A camera (Canon FT-QL) equipped with a 100mm focal length Ektar enlarging lens at the end of a long extension tube was used to take macro photographs of the reflection (Figure 3a). The angles of incidence and reflection, as well as lens extension, were carefully annotated.

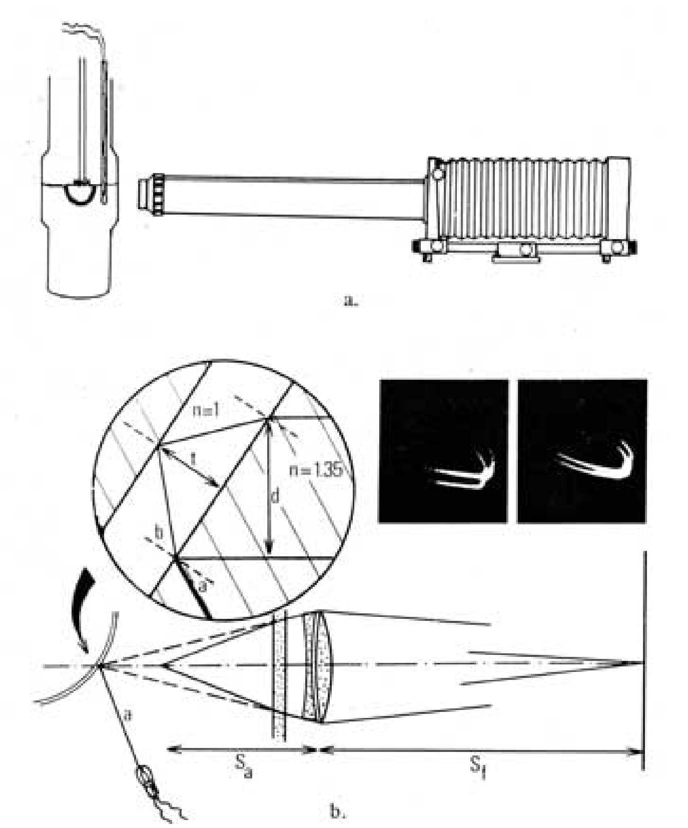

Figure 3. Camera arrangement, optical path diagram and double reflection photos of filament used in vapor shroud thickness measurement.

As expected, two images of the filament could be seen reflected from the boule (Figure 3b), one from the bulk liquid vapor interface and one from the vapor-boule interface. The real separation of the two interfaces depends on the apparent separation of the two reflections, the angle of observation and the magnification or reduction of the subject at the film plane.

To determine magnification all that was considered was the distance from the lens to the film plane divided by the virtual subject to lens distance. This takes into account index of refraction and curvature effects past the observation window. This virtual distance, Sa, was derived from the following equation:

$$1/Sa + 1/Sf = 1/f$$

where Sf is lens to film distance and f is the focal length of the objective. With a camera extension of 63 cm and a lens focal length of 10 cm, the virtual subject to lens distance was calculated to be 11.9 cm. Therefore, the magnification of the system was 5.3x. Since the measured separation of the filament images on the film was 0.25 mm, the thickness of the vapor shroud uncorrected for the angle of reflection was dm = 0.25/5.3 = 0.047 mm.

However, the ratio of actual thickness, 0, to photographed thickness, dm, is determined by

$$0 = dm/(2 \tan B \cos A)$$

where A is the angle of incidence and B the angle of refraction at the first surface as pictured in Figure 3b.

In our case the refractive index was n = 1.35, the angle of incidence an estimated 33 degrees 30' and therefore the angle of refraction 48 degrees 15'. This leads to the following ratio:

$$0/dm = 1/\ 2\ (1.11)\ (0.834)$$

from which 0 = dm/ 1.85 and thus the real thickness of the shroud becomes 0.047/1.85 = 0.025mm (25u) at the specific point photographed.

VAPOR GAP COLLAPSE

As mentioned earlier, a boule will not grow indefinitely but upon reaching a critical size it suddenly merges with the support liquid. To determine where, and also maybe why, a boule bursts, the visualization of the event, too quick for the eye to follow, was required. The greatest problem in most high speed single or multiple exposure photography is the synchronization of the exposure to the event. Various methods were attempted. It was known that a small potential (5 - 10 v.) applied across the vapor shroud would cause immediate merging. The first still photographs of a burst were taken with a Kodak View Camera with the shutter acting as an electrocution switch. One lead from the synchronization post led to the boule while the other was connected to the + terminal of a 22 v. battery. The-terminal led to the support liquid. After the boule had reached a size of about 30 ml., an exposure was made at M synchronization under photoflood illumination. The time advance built into the shutter provided enough time lag for the vapor shroud to collapse from 50 to 70% before shutter operation. The resulting photographs proved to be too blurred to be acceptable. Better definition was accomplished by using a standard rotary switch and a Graflex Stroboflash II. The switch had its common terminal connected to the boule and to the ground input of the flash. The second position went through the battery to the boule while the third position completed the triggering circuit for the flash.

The camera was set up and the boule focused by incandescent lighting. At the appropriate time this was turned off and in almost total darkness the shutter was opened. By quickly rotating the switch (by hand or spring) the time delay between boule electrocution and flash could be varied to obtain photographs after the shroud had collapsed from 10 to 5 0% (Figure 4), The results were good but still better definition was desirable.

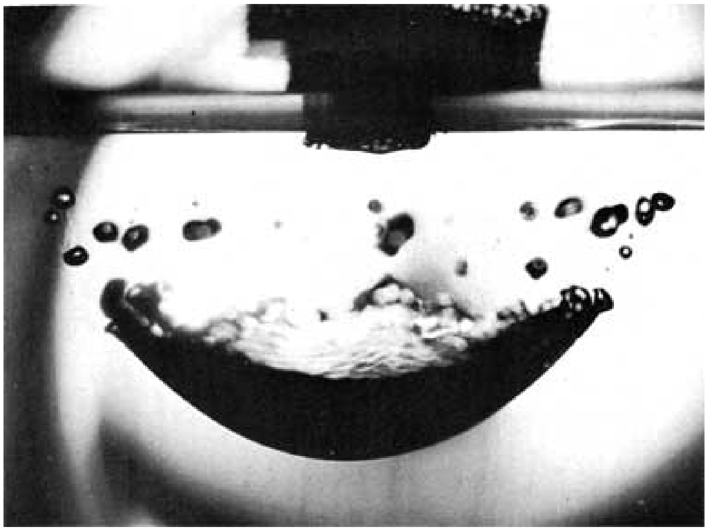

Figure 4. Boule burst after 70% collapse by Stroboflash lighting. Note that shroud was moving too fast for the duration of this flash. Detached bubbles remaining from shroud float towards surface.

The question remained whether electrocuting the boule was affecting the results obtained. To reduce this possibility a Tektronix 535 oscilloscope was used to monitor the change in the strength of a sine wave that could be picked up by connecting the boule to one input of the oscilloscope. This was the 60-cycle signal probably produced by the heater around the boule flask. The wave would gain amplitude whenever the electrode was submerged into the support liquid. It would also gradually gain amplitude as the boule grew. This characteristic was applied to set off a General Radio Strobotac the instant the electrode attached to the boule came in contact with the support liquid when the shroud collapsed.

The Tektronix 535 has a "+ gate out" which produces a + 20 v. pulse whenever triggered by the input signal exceeding a preset level. As the amplitude of the signal grew with increasing boule size, the peak was kept below the triggering level by manually lowering its position. Upon shroud collapse the signal would suddenly exceed the triggering level and cause a pulse to be sent to the Strobotac that started flashing. To prevent multiple exposures at the rate of 60 per second, the oscilloscope's "+ gate out" was led to the Strobotac through a limiting circuit (Figure 5). This circuit consisted of a normally closed relay to allow the pulse to reach the flash with the first enhanced sweep of the oscilloscope. However, the first flash was picked up by a GE-X2 photocell that caused the normally closed relay to open. Upon opening, the relay also completed a secondary circuit that maintained it in open position. In this manner multiple exposures were prevented and although the start of a burst could not be photographed, the appearance following the first 10 - 20% of merging could be recorded after any desired interval (Figure 6).

Exposures were in all cases determined by using a 4x5 Polaroid back on the view camera and making test shots on Type 52 film. The speed of Type 52 is very close to that of Kodak Tri-X sheet and roll film that was used to make all final photographs. The values obtained with Type 52 were used directly with Tri-X,

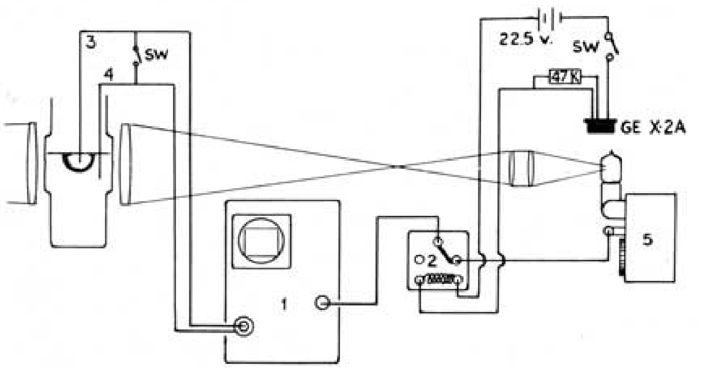

Figure 5. Basic wiring diagram for self-triggered boule photographs. Electrodes 3 and 4 connect the boule and liquid to the oscilloscope 1. A switch is used across them to start a boule growing. When boule bursts a +20-volt pulse goes from the scope through normally closed relay 2 to stroboscope 5. After flashing, transistor GE-X2A conducts, energizing relay. This disconnects the scope from the stroboscope and prevents double exposures.

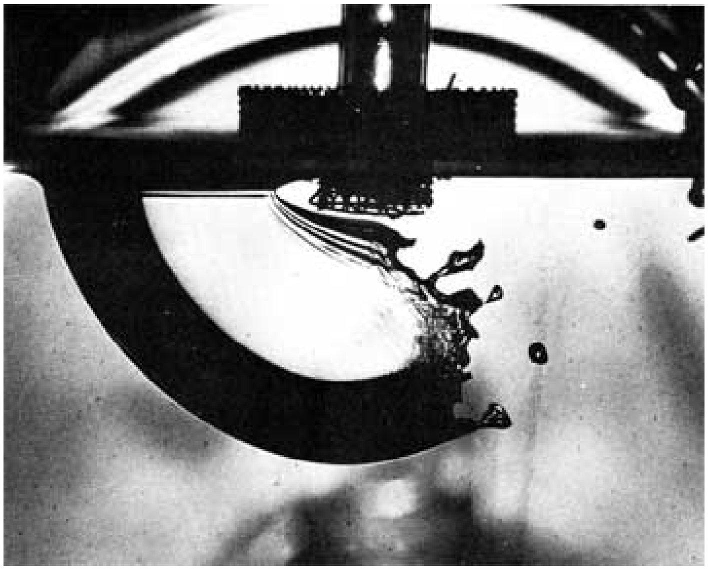

Figure 6. Self-triggered boule burst. Note area of collapse preceded by waves on the remaining shroud. Exposure time 1 microsecond.

Although we gained much valuable information just from still photographs, the mechanism of merging remained to be visualized. The main problem encountered was the fairly erratic lifetime of a boule. This made the use of a regular framing high-speed camera rather impractical, for the amount of wasted footage promised to be rather high. It was decided that the still photographs taken by electrocuting the boule were not much different from the ones taken by monitoring the sine waves with the oscilloscope. For a preliminary experiment we decided that a drum camera in streak mode would be used in conjunction with the Strobotac. A simple drum camera was constructed in this laboratory. It consisted of a wooden box 12 x 12 inches in which a flat circular drum covered with a strip of 35mm film was revolved at 600 to 800 rpm.

The camera was mounted on a tripod, focused, and its position noted. Then it was removed to the darkroom where the film was taped to the drum. After being repositioned on the tripod, the drum was set in motion. Directly behind the boule the Strobotac was turned on and set to 400fps. The boule was now electrocuted on M synchronization at 1/50 second at f/16. A perfect shutter would need to be set to 1/13 second to prevent double exposure but due to the small aperture being used the exposure time set on the shutter has to be much shorter. Actually now only about 75% of the film was being exposed but the start and the progression of the shroud collapse could be easily interpreted in about 20-25 frames (Figure 7). From these pictures the following quantitative and qualitative results were obtained: For isopropyl alcohol boules 4 to 5 cm in diameter, the collapsing front moves at between 50-70 cm/see. the opaque areas left behind then float upwards at about 8-16 cm/sec. and the lifetime of a burst is between 0.02 and 0.08 sec. The wavelengths of the "wave front" preceding the merge line increase with time to about 0.5 mm. The photographs suggest that breaks in the shroud start at either the rim or the bottom and at only one of these areas each time 2. The cause of the bursts has not been determined.

SCHLIEREN TECHNIQUES

The research into the characteristics of the life and demise of a boule constitute only one part of this laboratory's -investigations. The liquid the boule floats on is the second area of interest.

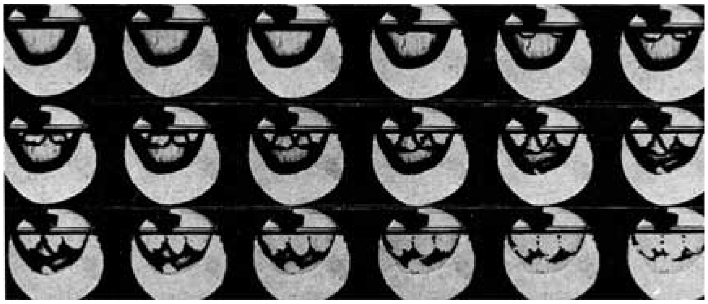

Figure 7. Induced burst sequence with drum camera and stroboscope. Burst initiated at frame 2. Note burst starts at two points at the top. This is probably due to the unnatural method of inducing the burst.

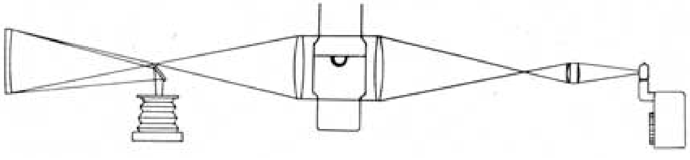

Figure 8. Basic schlieren system illustrating the use of a mirror as an objective for the view camera with the deflecting secondary mirror acting as a knife-edge.

Some questions that are raised are:

1. What does the liquid a boule floats on look like?
2. What effect do convection patterns within the liquid have on boule behavior?
3. Are there differences in convection patterns between various liquids?
4. How do convection patterns below the surface affect the surface of liquids?
5. Which areas are elevated? depressed? hotter? or cooler?

The study of convection in liquids has often been accomplished by the use of various schlieren techniques. These are optical methods for emphasizing the effect of refractive index differences within gases or liquids on a beam of light.

The schlieren system used in this laboratory consists of a 1000 watt projector lamp condensed by a small lens, two 60" focal length 4" diameter astronomical objectives and a 35mm camera fitted with a telephoto lens or a 4x5" camera with a 40" focal length mirror as an objective (Figure 8). The liquid under test is held in a container (with plane parallel windows) between the two objective lenses. The condensed light source is directed at these lenses from a distance of about 60". A parallel beam of light leaves the first field lens, traverses the liquid sample, and after passing through the second lens is brought to a focus about 60" away. At this point the image of the light source is positioned on the diaphragm blades (equivalent to a knife edge) of the telephoto lens that is focused on the test liquid. The camera is adjusted so that 70 to 50% of the image on the knife-edge is cut off by it, allowing the rest to pass on to the camera's ground glass. If there are no refractive index differences in the liquid a uniform grey image is seen on the ground glass. When the liquid is heated, the edges between hot and cold areas (corresponding to lower and higher refractive index areas) bend light rays away from their former path. This causes some of the light, which previously passed by the knife-edge, to be intercepted and some which was stopped before to pass by. In this manner a qualitative analysis of convection within liquids can be performed.

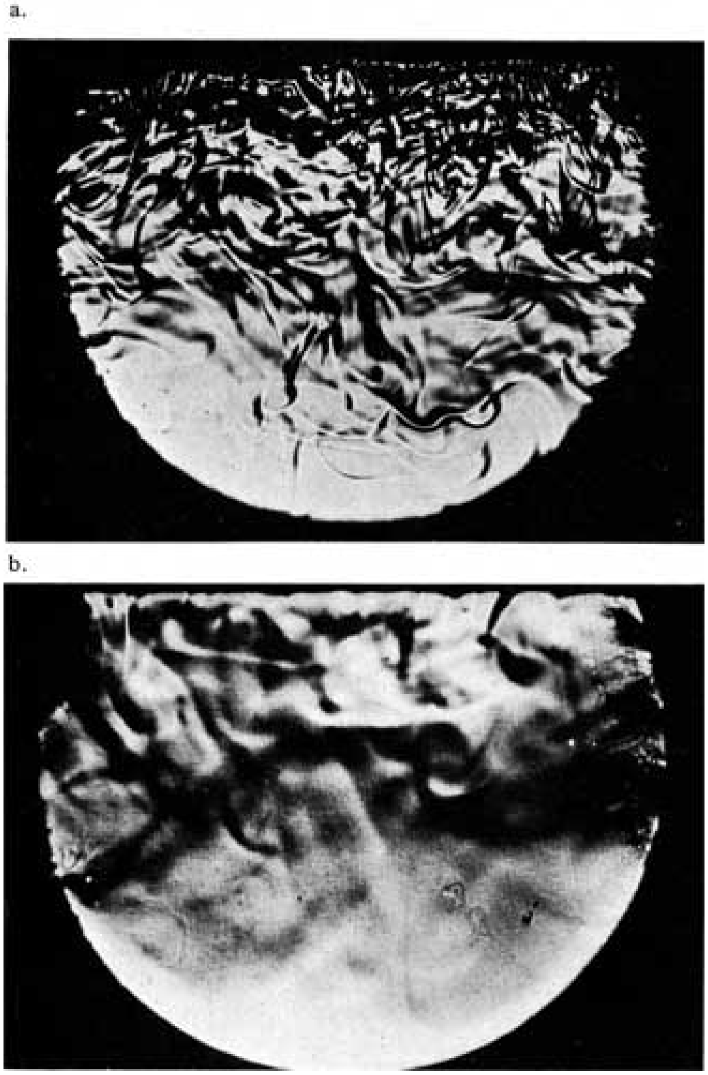

Figure 9. Schlieren photograph of 2-propanol, a, and water, b, at identical heat inputs.

The photographs obtained so far indicate that there are characteristic group differences but that one liquid, water, stands out clearly from the rest (Figure 9a,b).

A modification of the above system can be used to detect small variations in the relief of the liquid surface. Schlieren systems have traditionally been used as detectors of refractive index differences. However, the method is equally suitable to detect changes in the slope of a reflecting surface. This is accomplished by having either a light source which projects light perpendicular to a liquid surface or, as in the case illustrated here, the light is parallel to the surface but is deflected 900 to the horizontal (Figure 10). In this case a perfectly flat surface resembles the appearance of a liquid of uniform refractive index in the refractive schlieren system. Areas that tilt to left or right of horizontal equate themselves with higher or lower indices. A point that needs to be made is that schlieren systems are generally only sensitive to changes in refraction or angle of reflection that cause the light beam to move in a direction perpendicular to the knife edge.

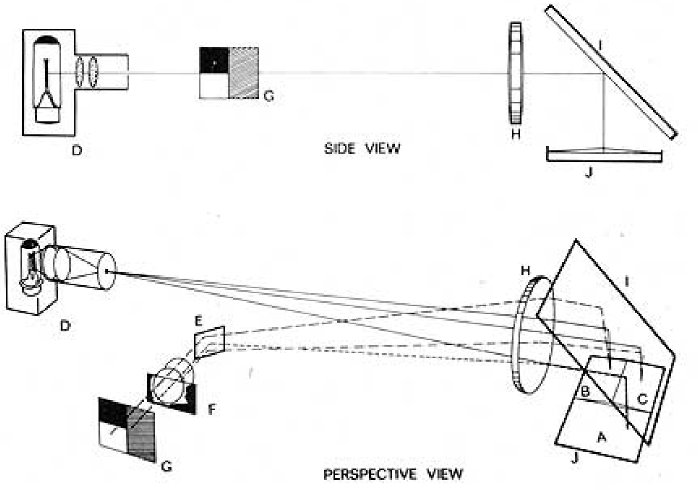

Figure 10. Basic reflection schlieren system. Light from source D is aimed at field lens H and deflected onto idealized surface J by large first surface mirror 1, placed at 45 to the surface but aimed slightly away from the source. This causes the light reflected from the surface to fall on secondary mirror E and to proceed into the camera lens and diaphragm F. Various slopes such as A, B or C on the surface produce visual effects such as illustrated on ground glass G.

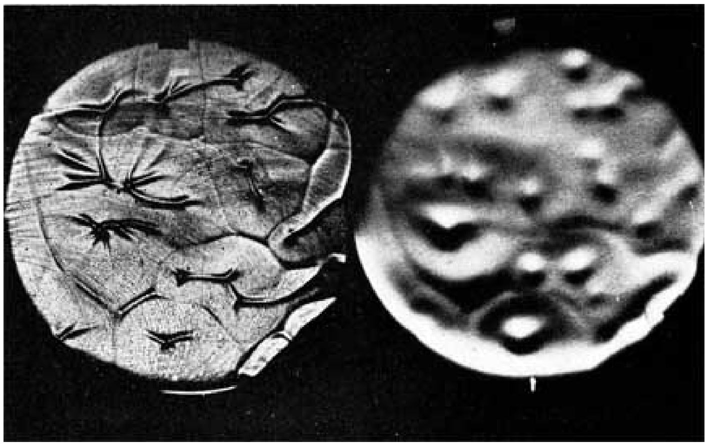

Figure 11. Simultaneous vertical schlieren views of the surface of a layer of paraffin wax, right, and of the liquid beneath, left. Areas of the surface that appear brighter on their upper side are small elevations caused by hot material rising to the surface.

The system has been developed to the stage that simultaneous vertical views of the surface relief and temperature gradients within a layer of molten paraffin wax can be visualized (Figure 11). A short film about this experiment is available for loan on request. Note that absolute heights cannot be measured. Flat areas, even though at different elevations, will all appear 50% grey, since the knife edge always intercepts half of the ray bundle reflected from these areas. However, since the density of the image at any point on the ground glass is a direct function of the slope of the surface, elevations can be approximately determined relative to a fixed reference point. Slopes that are more inclined than those which just produce 100% illumination or complete extinction cannot be measured. This "deficiency" can be corrected by making the light source larger or the field lens to knife edge distance shorter, thereby reducing the ability of the system to detect small changes in slope.

A simpler technique of detecting surface patterns has yielded excellent results. Clean aluminum powder is allowed to float on the surface of the liquid under investigation. The camera is placed at an angle of about 450 to the liquid surface and the lighting is arranged so that a near specular reflection is obtained from the surface. At this point the liquid surface still looks quite dark but the specks of aluminum reflect a great deal of the incident light. Time exposures are made at speeds of one-half or one second. The powder in motion on the surface produces a very easily interpreted record of the movements of the surface. Compare Figure 12a to 12b.

Figure 12, a & b. Instantaneous, top, and time exposures of a liquid surface with tracer powder added.

Photography plays a very important role in the research of the Distillation Laboratory at RIT. Phenomena which are undetectable to the eye are readily visualized by the application of various photographic and optical methods. Some of these methods are very basic indeed. However, the importance of photographic solutions to scientific problems reassert their value in view of the wealth of new knowledge gained through the medium.

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This article was published in:

Photographic Applications in Science, Technology and Medicine, pp. 27 – 31, November 1969