

AN OPTICAL HIGH PRESSURE CELL FOR LIQUID CRYSTALS

R. SHASHIDHAR, S. RAMASESHAN* AND S. CHANDRASEKHAR

Raman Research Institute, Bangalore 560 006

ABSTRACT

A new type of optical high pressure cell for visual observation of phase transitions in liquid crystals under pressure is described. This cell has been useful in identifying the pressure induced mesophases of *p*-methoxy and *p*-ethoxy benzoic acids.

INTRODUCTION

It was predicted theoretically¹ that certain compounds which do not form liquid crystals at atmospheric pressure can show mesomorphism on application of pressure. Using a differential thermal analysis (DTA) high pressure cell designed by us², we have been able to establish experimentally that *p*-methoxy and *p*-ethoxy benzoic acids, which are normally non-mesomorphic, show liquid crystalline behaviour when subjected to pressure^{1,3}. Though DTA could unambiguously detect the phase transitions, it was not possible to identify the pressure induced phases. This requires an optical cell wherein the sample may be subjected to pressure and at the same time examined through a polarizing microscope. We have designed and constructed such a cell and taken photographs of the textures exhibited by the pressure induced phases.

DESCRIPTION OF THE CELL

A schematic diagram of the cell is given in Fig. 1. It consists of a stainless steel stationary plate (SP) with a central hole and a coaxial step. A fused silica anvil (A_1) is press fitted into this step. A guard ring (R) also made of stainless steel is shrunk fitted to this anvil by heating both of them to nearly 400° C. This ring has a slightly larger inner diameter towards its top portion so that the other anvil (A_2), also made of fused silica, can just rotate inside the ring. This anvil is fixed in the top stainless steel plate (TP). The two anvils can be pressed against each other by screwing the top plate. A major difficulty that we encountered was to contain the liquid crystal under pressure. After trying out various types of binding rings it was found best to use a teflon spacer (thickness $\sim 100\mu$) of the type shown in the figure. A special feature of this spacer is that it is machined on both sides to have cone angles which perfectly match the corresponding angles of the anvils. Only then was it possible to contain the sample without leaks.

* Permanent address : Materials Science Division, National Aeronautical Laboratory, Bangalore 560 017.

The cell was enclosed in a small electrical oven provided with windows. The temperature of the sample was probed by a chromel-alumel thermocouple. The entire unit could be placed on the optical stage of a Leitz polarizing microscope equipped with a camera so that observations could be made in transmitted light. As the thickness of the cell was rather large ($\sim 1''$) a low power objective had to be used and consequently this was the limiting factor for the magnification.

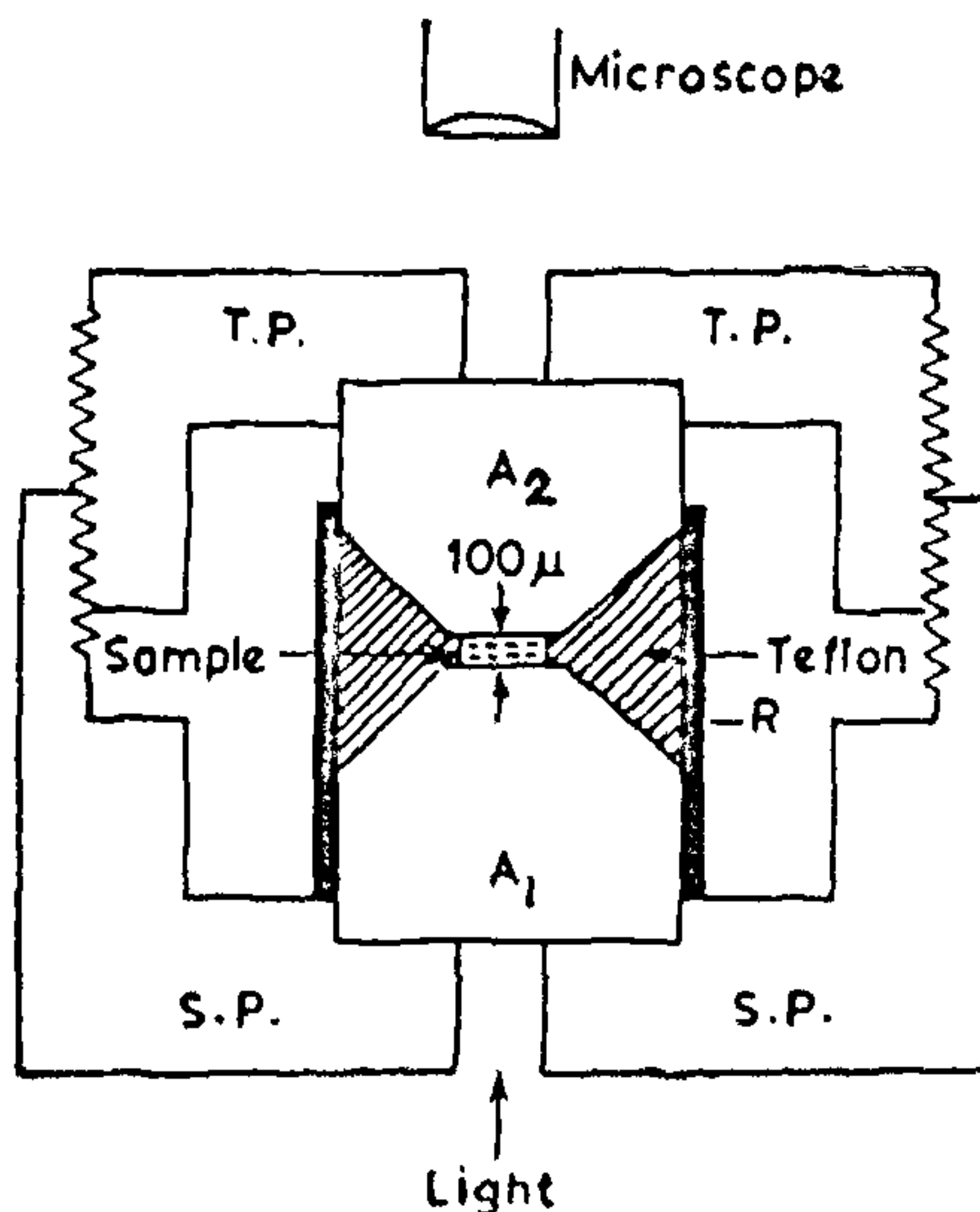


FIG. 1. Schematic diagram of the optical high pressure cell.

IDENTIFICATION OF PRESSURE INDUCED PHASES USING THE CELL

The pressure developed was very roughly estimated in terms of the number of screw turns of the top plate by measuring (by visual observation) the solid-nematic and nematic-isotropic transition temperatures for *p*-azoxyanisole for which dT/dP values are known^{4,5}. Experiments were then undertaken on *p*-methoxy and *p*-ethoxy benzoic acids

to identify the pressure induced phases. The sample which is in the solid phase at room temperature is packed within the spacer. The anvils are gradually pressed together by rotating the top plate. Initially the teflon is compressed but on further turning the sample itself experiences the pressure. Keeping the pressure constant, the temperature is then increased so that the phase transitions are studied at that pressure.

At low pressures *p*-ethoxy benzoic acid (which at atmospheric pressure has just the solid-isotropic liquid or melting transition at $\sim 194^\circ\text{C}$) shows a single intermediate phase which exhibits the *schlieren* texture typical of a nematic liquid crystal (Fig. 2). At higher pressures it was found that on

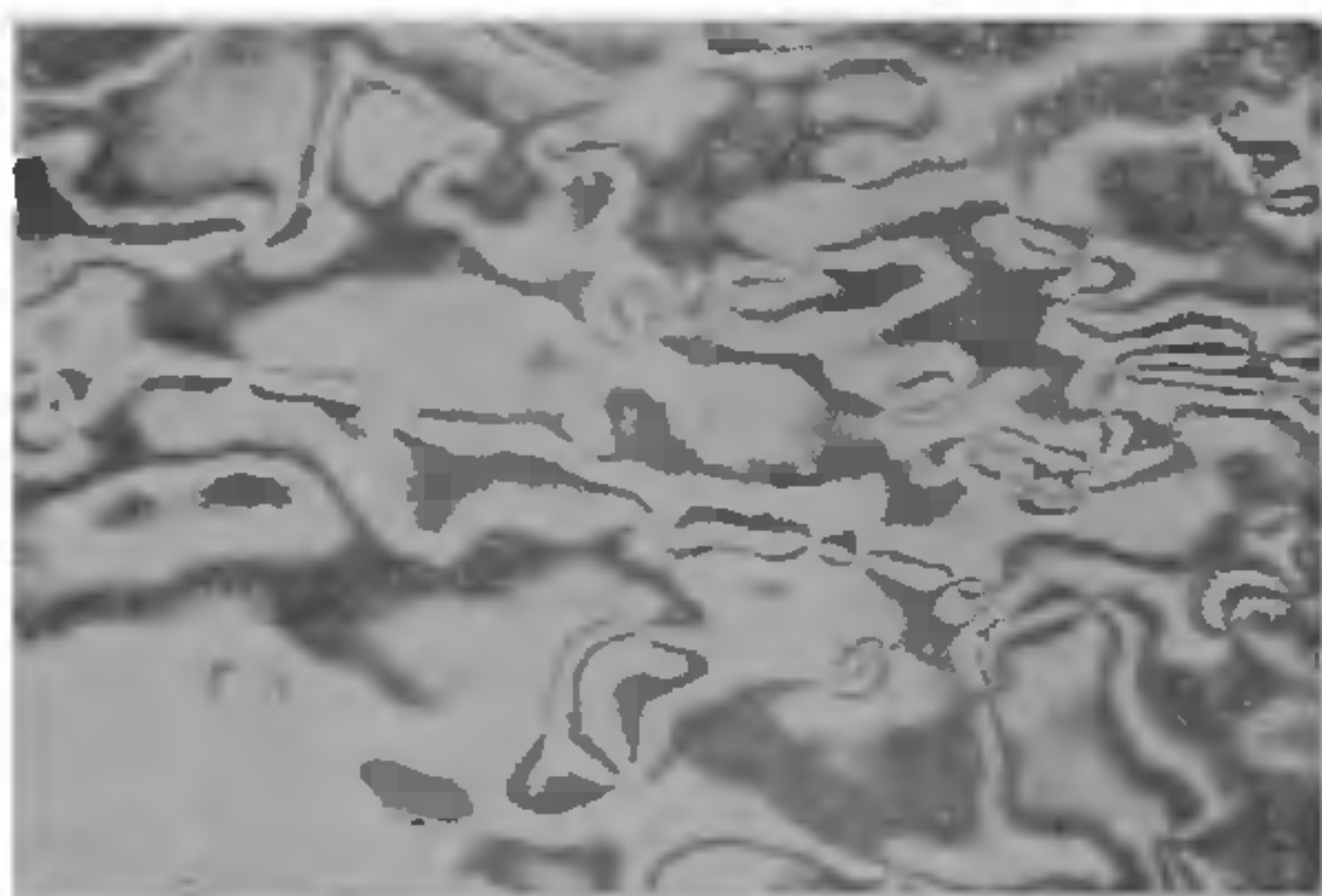


FIG. 2. Schlieren texture exhibited by *p*-ethoxy benzoic acid (crossed polars, $\times 90$).

cooling the isotropic liquid there appears the nematic phase (with a schlieren texture) which at a lower temperature transforms to another phase with 'focal conics' and 'batonnets' characteristic of a smectic liquid crystal (Fig. 3). On further

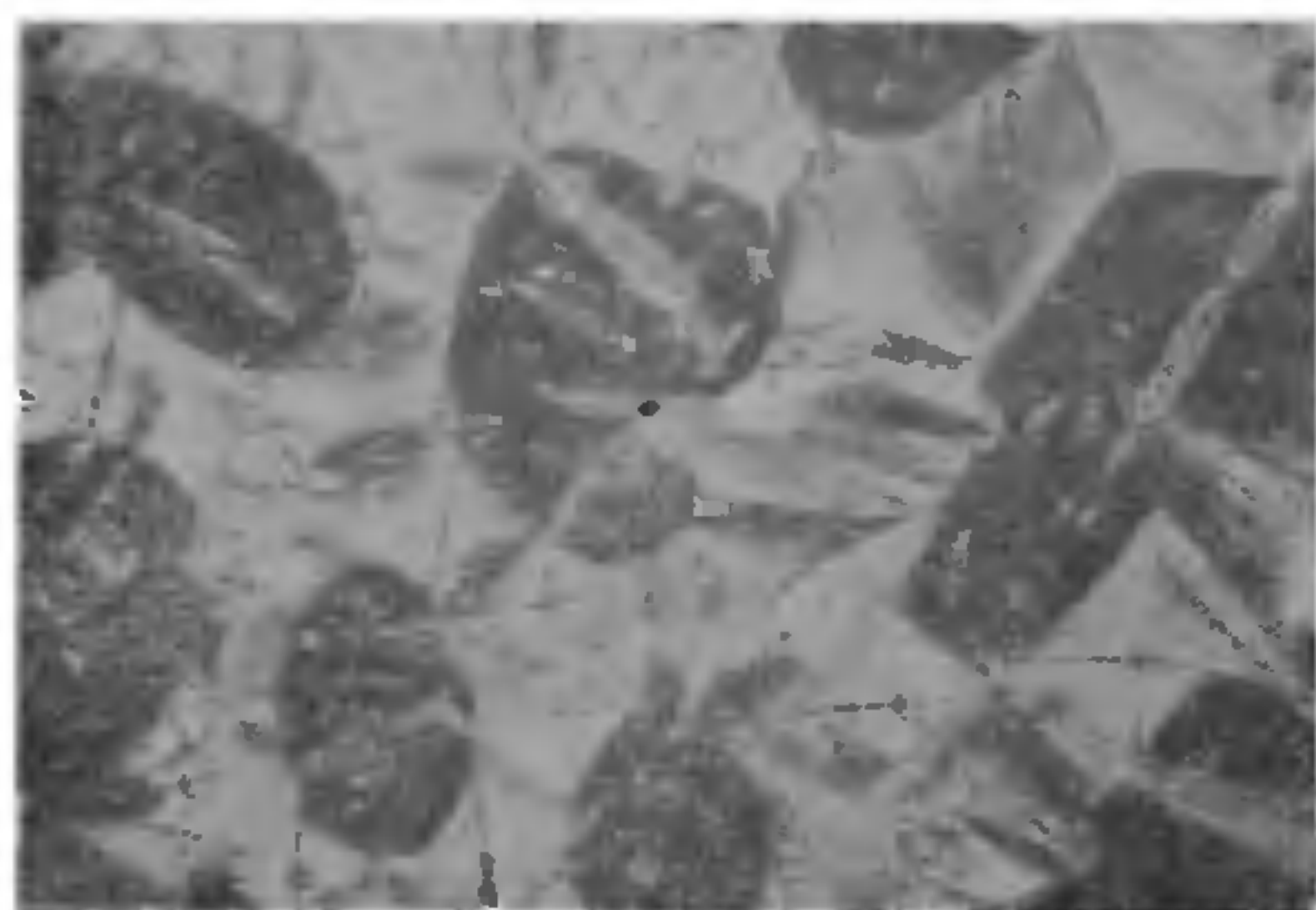


FIG. 3. Focal conics and batonnets shown by *p*-ethoxy benzoic acid (crossed polars, $\times 90$).

cooling the sample goes over to the solid phase and becomes opaque. It may be mentioned that it was

not possible to observe such well defined textures during every experiment. Special care had to be taken to clean the anvil surfaces and to have a uniformly thick sample ($\sim 100\mu$), in order to get good textures.

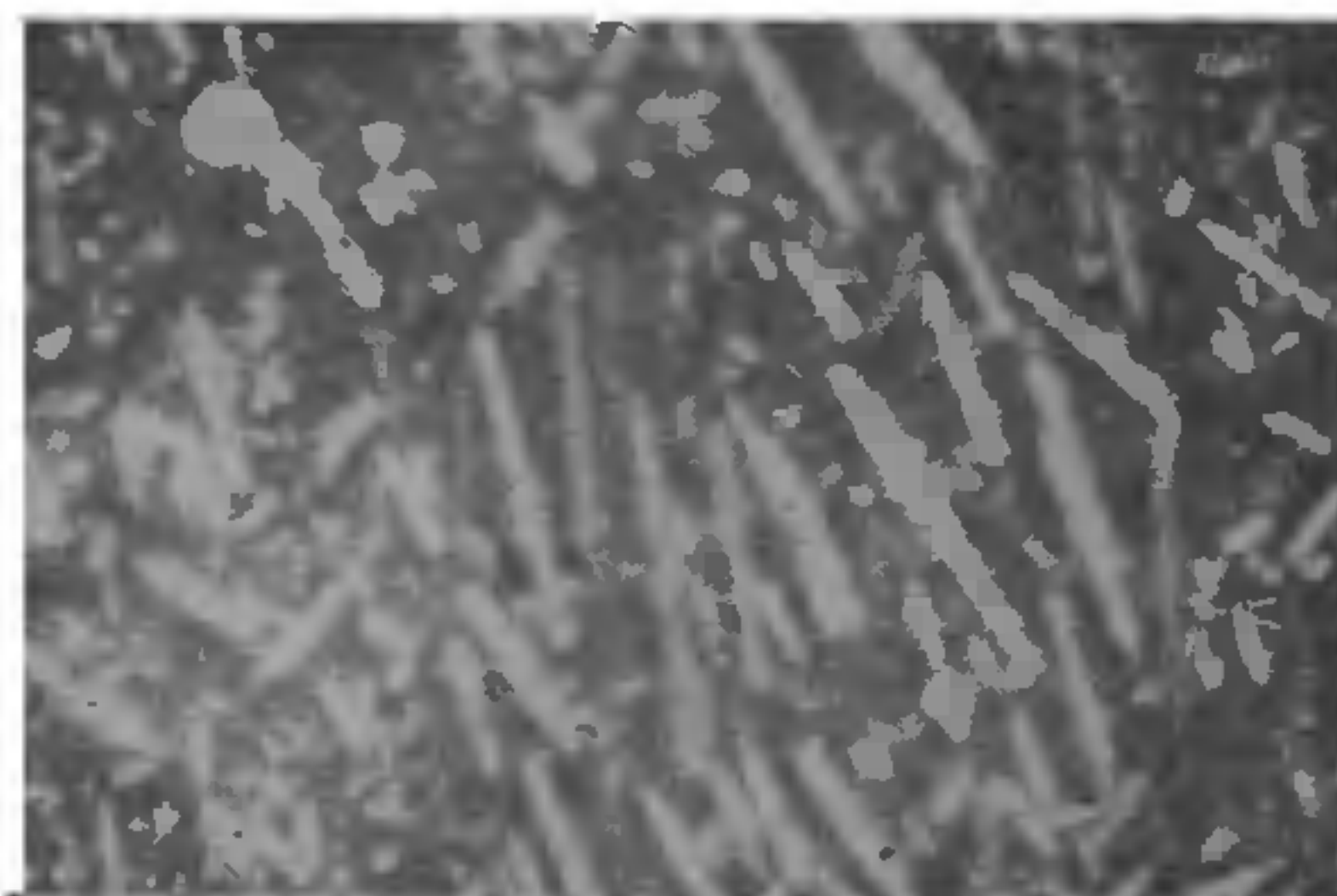


FIG. 4. Batonnets shown by *p*-methoxy benzoic acid (crossed polars, $\times 90$).

p-Methoxy benzoic acid also shows similar textures under pressure. Batonnets shown by the smectic phase of this compound are shown in Fig. 4.

In its present form this cell works for relatively low pressures (of the order of a few hundred bars) only. This is primarily because of the relatively large surface area of the anvil faces which are pressed together, though fused silica itself is capable of withstanding pressures up to 5 kbar. Furthermore, the calibration of pressure is not accurate. Experiments are in progress to modify the design of the cell so as to go to higher pressures and also to improve the accuracy of the pressure calibration.

ACKNOWLEDGEMENTS

We wish to acknowledge the help given by Mr. N. Narayana Iyer and Mr. Chatterjee in the construction of the cell.

1. Chandrasekhar, S., Ramaseshan, S., Reshamwala, A. S., Sadashiva, B. K., Shashidhar, R. and Surendranath, V., "Proceedings of the International Liquid Crystals Conference, Bangalore, December 3-8, 1973," *Pramāṇa Supplement*, 1, 117.
2. Reshamwala, A. S. and Shashidhar, R. (to be published).
3. Shashidhar, R. and Chandrasekhar, S., *J. de Physique*, 1975, 36, C1-49.
4. Hulett, G. A., *Z. Phys Chem.*, 1899, 28, 629.
5. Robberecht, J., *Bull. Soc. Chim. Belge*, 1936, 37, 597.