demonstrate that similar results can be obtained by other cold-working techniques, quenched cylinders of linear polyethylene, 0.25 in. in diameter by 0.5 in. long, were placed in a steel cavity 0.25 in. in internal diameter and 1.5 in. long. A steel rod 0.23 in. in diameter was then pushed into the cold blank in order to extrude a cold-formed sleeve of polyethylene back up around the rod. It was found easy to form tubes of 0.01 in. wall thickness and over 6:1 length/diameter ratio by this method.

References

1. Gruenwald, G., *Modern Plastics*, **38,** 137 (1960).
2. Rothschild, P. H. and B. Maxwell, Princeton University Plastics Laboratory Technical Report No. 59C, October 1960.
3. Matsuoka, S., and B. Maxwell, *J. Polymer Sci.*, **32,** 131 (1958).

Bryce Maxwell

Princeton University Plastics Laboratory
Princeton, New Jersey

Paul H. Rothschild

General Engineering Laboratories
General Electric Company
Schenectady, New York

Received February 23, 1961

An Improved Microscope Hot Stage

The recent discovery of a large number of crystalline polymers with well-defined melting points[1] has resulted in the increased use of melting point determinations of polymeric materials, mainly for identification, but also for structural elucidation.[2] As many crystals of polymers exhibit birefringence, the measurements are generally performed on a heated microscope stage, under crossed Nicol prisms or Polaroids, the melting point being taken as the temperature at which the birefringence entirely disappears. A number of suitable microscope hot stages have been described in the literature,[3] and several are commercially available,[4] the Kofler stage being most commonly used. This model consists of an electrically heated block on which the sample is placed, being protected from air currents by means of a glass baffle and cover plate. Temperatures are measured with a thermometer, inserted through a well in the block, which is so marked that readings coincide with melting points of standards measured with the equipment.

Satisfactory results are obtained with this stage when a precision of about 1°C. is required. For better measurements, however, certain modifications are necessary because sample and block temperatures differ considerably and cannot always be precisely related. For this purpose a second electrically heated block was constructed to be placed over the original one (Fig. 1). It was provided with a small indentation (I) in the center for accommodating the sample, a lip (L) for seating, and a cover glass (G) in a small circular indentation at the top, held in place by a circular clip (C), which sealed the center hole (H) to air currents. In other respects it was similar to the parent block and in operation could be simply lifted for the introduction

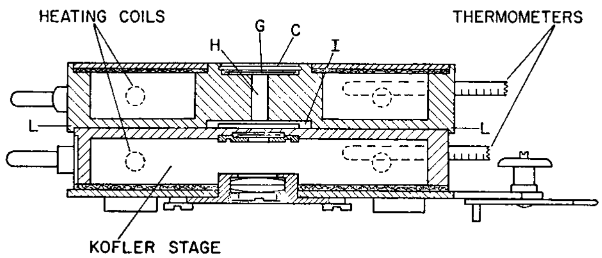

Fig. 1. Schematic diagram of modified stage.

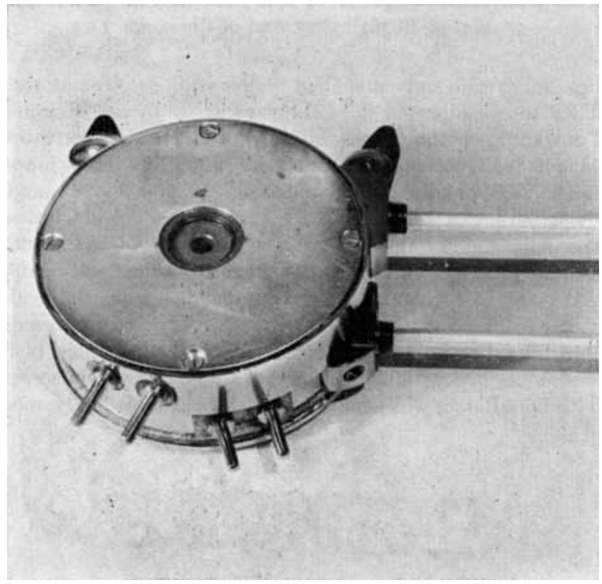

Fig. 2. Photograph of modified stage.

or removal of the sample. The joining faces of both blocks were polished and chrome-plated to reduce temperature gradients between them. A photograph of the modified stage is shown in Figure 2.

TABLE I
The Apparent Melting Point of Phenacetin as a Function of Sample Distance from Heat Source[a]

Stage	Observed m.p., °C.	Distance, mm.	Remarks
Kofler	139.4	2.8	Sample on two microscope slides (each 1.4 mm. thick) and covered by 0.14 mm. thick cover glass[b]
Kofler	137.2	0.14	Sample between two cover glasses (0.14 mm)[b]
Modified	135.5	0	Sample between two cover glasses (0.14 mm.)

[a] U.S.P. grade phenacetin, recrystallized three times from a 50/50 (vol.) mixture of $CHCl_3$ and *n*-hexane.

[b] The usual baffle and top glass cover was also used.

Two matched thermometers with 0.1°C. divisions covering a 20-degree range and equipped with reading lenses were

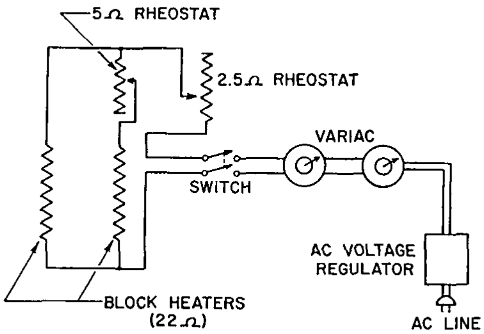

Fig. 3. Electrical system of the stage.

used for precise and convenient temperature determinations. They were calibrated in the apparatus by means of iron–Constantan thermocouples by use of a Leeds and Northrup Model 8662 potentiometer and ice–water reference junctions. The precision, using phenacetin, was ±0.02°C., and the accuracy is estimated as ±0.1°C. Table I illustrates the magnitude of the temperature difference of block and sample obtained with the use of the conventional and modified stage as a function of sample distance from the heat source. The electrical system (Fig. 3) consisted of an a.c. controller, two Variacs connected in series, and a control box containing two variable resistors, of which one was in series with both blocks while the other was in series with only one. The function of the first resistor was to provide fine control of the heating rate, while the second permitted matching of the temperature of both blocks. Heating rates were less than 0.2°C./min.

We thank Mr. Q. A. Trementozzi for a helpful discussion.

References

1. See, for example, Gaylord and Mark, *Linear and Stereoregular Addition Polymers*, Interscience, New York–London, 1959, particularly Chaps. 4 and 5.

2. Campbell, T. W., and A. C. Haven, Jr., *J. Appl. Polymer Sci.*, **1**, 73 (1959); M. Dole and B. Wunderlich, *Makromol. Chem.*, **34**, 29 (1959).

3. Zscheile, F. P., and J. W. White, Jr., *Ind. Eng. Chem., Anal. Ed.*, **12**, 436 (1940); F. W. Mathews, *Anal. Chem.*, **20**, 1112 (1948).

4. McCrone, W. C., Jr., *Fusion Methods in Chemical Microscopy*, Interscience, New York, 1957, pp. 20–25.

E. Kohn
H. Medina, Jr.
J. V. Cavender

Plastics Division
Monsanto Chemical Company
Texas City, Texas

Received January 30, 1961