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An Automatic Dilatometer for Radiation Polymerizations

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Synopsis

An automatic dilatometer particularly suitable for radiation polymerizations is described. The volume contraction may be followed outside the irradiation room by two methods with great accuracy.

INTRODUCTION

The determination of polymerization rate using the technique of dilatometry presents a number of difficulties when the reactions are initiated by ^{60}Co gamma radiation. In such cases, the irradiation has to be interrupted each time the position of the meniscus in the capillary of the dilatometer is measured, thus perturbing the polymerization reaction.

Systems that allow one to follow automatically the progression of a polymerization reaction have been described[1-3]; they usually involve recording the displacement of a mercury column.

Over the past few years, we have used successfully a simple automatic dilatometer containing no mercury, which is particularly useful for following polymerizations carried out in irradiation rooms. The direct optical determination of the level of the polymerization solution is obtained with a very satisfactory degree of accuracy.

DESCRIPTION OF DILATOMETER

Figure 1 is a schematic representation of the apparatus, which consists of the following systems:

Optical System. The light source is a galvanometer bulb with a very thin linear filament (1); the light beam is focused very accurately by the lenses (2) onto the center of the dilatometer tube (4) and passes through lenses (3) to be refocused onto a Ge photodiode (5). The optical system is mounted inside two watertight metal cylinders (6), the electric wires passing through lateral tubes. The two cylinders, very firmly fixed together, are attached to the moving block (7) and are shielded from the radiation by lead plates. The dilatometer is mounted on an adjustable support which can be moved to center the capillary.

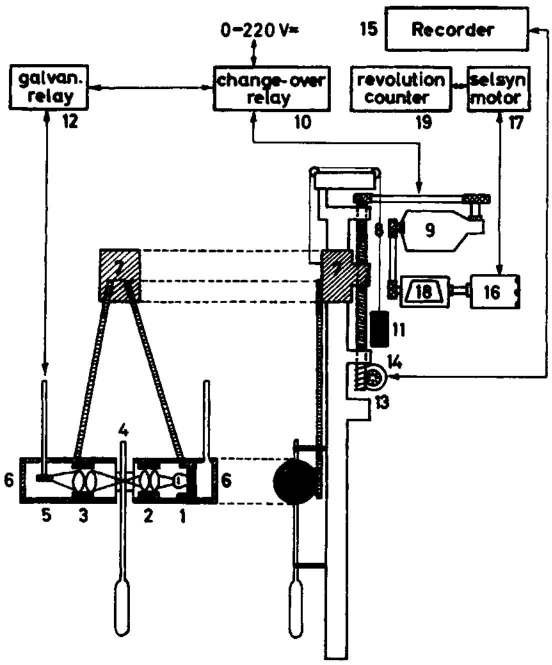

Fig 1. Schematic diagram of dilatometer: (1) linear filament galvanometer bulb (4V, 0.5 A); (2, 3) biconcave lenses (focal length 21 mm, aperture 20.5 mm); (4) capillary of the dilatometer; (5) Ge photodiode (Philips OAP 12); (6) light source cylinder (length, 70 mm; diameter, 37 mm) and photodiode cylinder (length 125 mm; diameter, 37 mm); (7) mobile block; (8) precision threaded rod (thread 0.5 mm); (9) drive motor (G.K. Keller NHS 12 R, 0–220 V); (10) change-over relay (MBLE R1A/120/KKK); (11) counterbalance weight, (12) galvanometric relay (AOIP RP 221); (13) worm screw; (14) helicoidal potentiometer; (15) recorder; (16, 17) selsyn motor; (18, 19) revolution counter.

Mechanical System. On rotating the precision screw (8) (thread 0.5 mm), the mobile block (7) holding the optical system is displaced. Maximal displacement is 110 mm and is limited by two microswitches. Rotation of the screw is effected by means of a direct current motor (9) through a gear reducer and with the help of pullies and a belt. The rotation can be reversed by means of a change-over switch (10). A counterbalance weight (11) reduces wear of the screw and avoids slack of the threaded parts.

Electrical System. The photodiode (5) is connected to a galvanometric relay (12) whose cells command the change-over relay (10) and thus the drive motor (9). The optical system, therefore, functions as a meniscus seeker. The use of a galvanometric relay is much simpler than that of an electronic one, as the former can be more easily adjusted.

Recording System. The displacement of the optical system can be recorded in two ways:

1. The worm screw (13) commands a helicoidal potentiometer (14) which is incorporated into a circuit with a recorder (15).

2. A primary selsyn motor (16) and a revolution counter (18) are coupled to the drive motor (9); the secondary selsyn motor (17) is located outside the irradiation room and is itself also coupled to a revolution counter (19). On completing a polymerization, one can check the proper functioning of the apparatus by comparing the readings on the two revolution counters.

ACCURACY

Taking into account the gear-reducing system and the thread of the driving screw, one turn on the revolution counter is equivalent to a displacement of 4×10^{-3} mm of the optical system. However, it is difficult to focus perfectly the light beam, so that in practice the error on the position of the meniscus has been shown to be of the order of 4×10^{-2} mm. The precision obtained is thus quite sufficient to follow even the most rapid polymerization rates.

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