

NOTE

A Modified Balance-Type Stress Relaxation Apparatus for Chemical Relaxation Under Liquid Phase

Synopsis

A relatively simple and inexpensive automatic apparatus for chemical relaxation giving good accuracy has been developed for measurement of chemical relaxation in the gaseous phase in high polymers. The apparatus is based on a previously described balance-type stress relaxation apparatus intended for automatic operation.

The elastoviscous properties of high polymers pose a problem of considerable interest. In studies of the elastoviscous behavior of high polymers, decay of stress at constant extension is a very simple and fundamental rheological experiment. An apparatus which measures this stress relaxation is valuable for studying the mechanism of network breakdown and chemorheology in high polymers.¹⁻⁴

A simple device for this purpose, wherein an extended-ring test piece of high polymer was held under solution, was described by Nisizawa.⁵ Nisizawa's apparatus for measurement of stress relaxation in which the sample is held under solution so that chemical relaxation does not contribute to the various chemical relaxations has been somewhat modified to render it more versatile and convenient in use; the latest form is described below.

A constant-temperature vessel of this apparatus has been constructed in which a cap floats on mercury in a gutter on the top of the constant-temperature vessel. From this apparatus a complete stress relaxation curve of high accuracy under various gaseous phases can be constructed.

Construction and Procedure of Stress Relaxation Apparatus

Samples for examination in this apparatus take the form of a ring. The ring samples (7.0-cm. O.D., 6.0-cm. I.D.) was died out of cured sheet 0.05 to 0.10 cm. in thickness. It was stretched between two stainless steel pulleys A and B. The diameter of the pulleys is 1.0 cm. The upper pulley A is free to rotate on the bend rod C hanging from the beam D. A cap M floating on mercury in the gutter N on the top of the constant-temperature vessel is rigidly fastened to the bend rod C. The lower pulley B is also free to rotate on the reverse U-rod which is rigidly fastened to the vertically movable clamp F. Also, a cap O floating on mercury in the gutter P on top of the constant-temperature vessel is rigidly fastened to the reverse U-rod E. The elongation chosen can be varied from 0 to 250% by setting the clamp F in the proper position. Higher elongations can be obtained by use of a ring sample of smaller diameter. The reverse U-rod E extended downward from the top of the constant-temperature vessel which is filled with solution or gaseous phase can be manipulated from outside. Thus, samples can be supported over the pulleys and can come to the desired temperature; they are then quickly brought to the desired elongation by pushing the clamp F onto the collar G which is previously set in the desired position.

The fulcrum of the balance consists of stationary agate v-blocks in which a hardened steel edge is held to the beam. The adjusting screws H is adjustable so that the beam can be balanced with the chainomatic weight I at each end of the beam. The chainomatic weight I is automatically controlled (J,K). The finest division of the dial L which winds up the chainomatic weight is 0.1 g., but it can be read to within 0.01 g. The maxi-

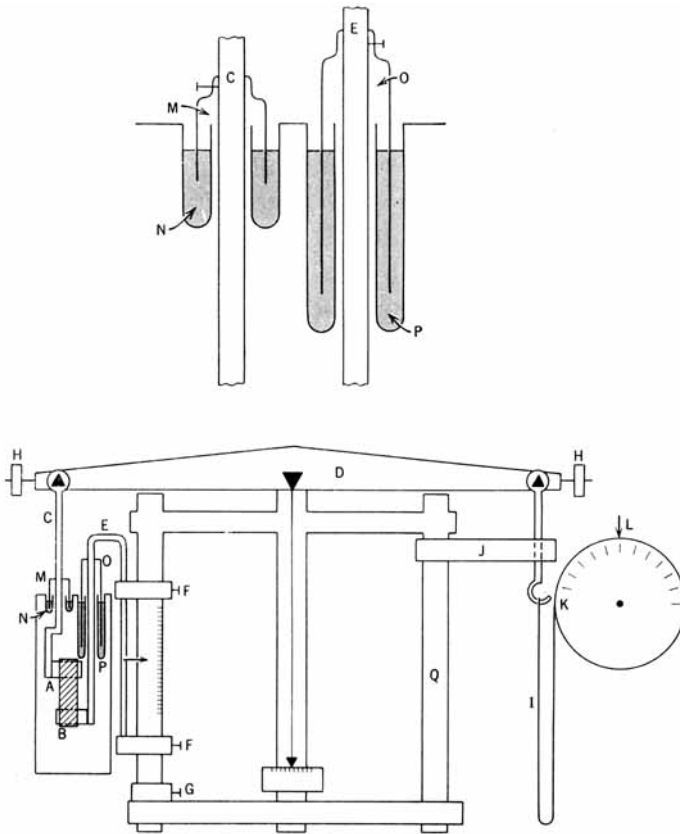


Fig. 1. Schematic diagram of stress relaxation apparatus.

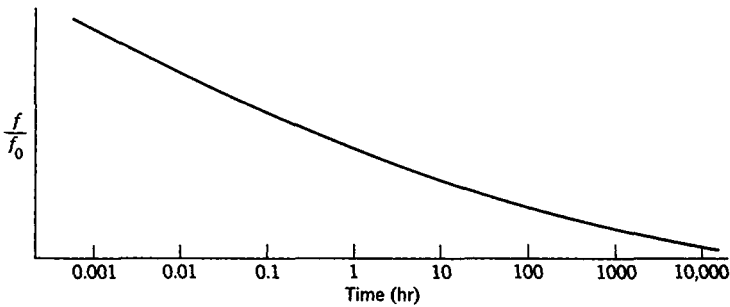


Fig. 2. Stress relaxation curve.

imum vertical motion of each end of the beam is 0.05 cm., which corresponds to an elongation of 0.5% based on the unstretched length of the band. A set of electrical indicators J is mounted on the column M so that the chainomatic weight is wound up the motor of the dial when the right end of the beam drops. An indication of the dial was taken as the balance against stress extended by the test piece band. The initial length of the test piece band was taken as the average circumference of the ring.

Very smooth stress relaxation curves have been obtained using this apparatus.

References

1. A. Kishimoto and H. Fujita, *Kolloid-Z.*, **150**, 24 (1957).
2. H. Fujita and A. Kishimoto, *J. Polymer Sci.*, **28**, 547 (1958).
3. A. Kishimoto and H. Fujita, *J. Polymer Sci.*, **28**, 569 (1958).
4. A. Mercurio and A. V. Tobolsky, *J. Polymer Sci.*, **36**, 467 (1959).
5. M. Nisizawa, preprint of the Annual Meeting of the Japanese Society of Scientific Fisheries, April, 1966.

MIZUHO NISIZAWA

Department of Chemistry
Defense Academy
Yokosuka, Japan

Received September 21, 1966