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An Environmental Instrument for Measuring Creep and Stress Relaxation in Polymers

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Synopsis

The construction and operation of an instrument for measuring tensile stress relaxation and creep, particularly of polymers, is described. The instrument is comparatively inexpensive to build and enables measurements to be carried out in vacuo or in a controlled atmosphere of gas or vapor. The design is based on principles used for some earlier stress relaxometers modified to enable characterization of samples having a very wide range of moduli either as stress relaxation or, additionally, as creep measurements. The instrument can therefore be used to evaluate material properties of hard plastics or of soft rubbers when exposed to selected environments.

INTRODUCTION

The presence of imbibed solvent or penetrant can profoundly affect the stress relaxation and creep behavior of a polymer. The amount of penetrant sorbed at a given time depends on the physical and chemical nature of the polymer and penetrant and on the interdiffusion coefficient for the system. Hence, studies of transient mechanical behavior in the presence of penetrant vapor can yield valuable information on the interrelation of mechanical and transport properties.

To carry out such work we have developed an environmental stress relaxometer and creep instrument. The design is based on a stress relaxometer originally used by Berry[1] and subsequently employed and developed by a number of workers[2–4] for material studies on elastomers. We have modified and extended the design in order to study polymer samples having a much wider range of moduli and to carry out creep as well as stress relaxation measurements.

DESCRIPTION OF INSTRUMENT

The interior of the environmental chamber is illustrated in Figure 1. The essential feature is that a strip of polymer film J is held in series with a music wire tension spring N of known spring constant. The film is held at its lower end in the clamp I and connected to the spring by the aluminum upper clamp K with adjustable frame L and hook M, the latter being free to

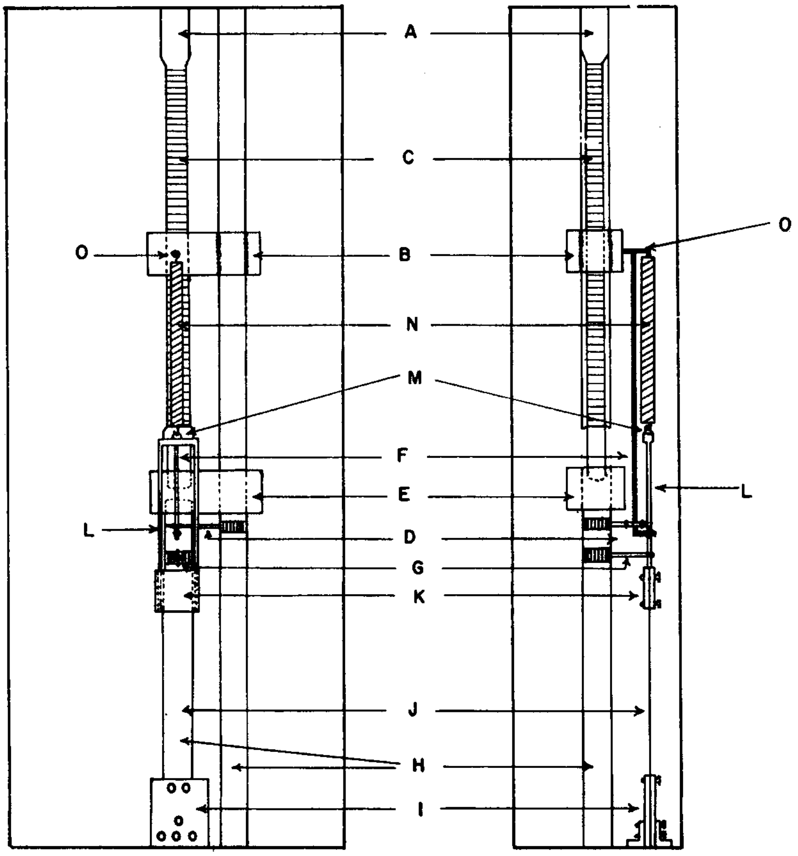

Fig. 1. Fundamental arrangement of instrument components inside environmental chamber. Letters as referred to in text.

rotate. The upper support O for the spring is screwed into a brass block B which is tapped to take the shaft A in its threaded region C. The block B slides on one of two steel support rods H_1, friction being reduced by the use of a Teflon insert. The shaft is mounted in a bearing in the steel block E which is in turn supported by the second rod, H_2. Hence rotation of A will produce vertical linear motion of B and stretch or relax the spring, thus exerting a calculated stress on the polymer sample J.

The tubes F and G of brass and aluminum, respectively, are fixed with respect to O in the case of F and to H_2 in the case of G. These tubes surround Teflon-insulated electrical leads and have protruding electrical contacts positioned inside the frame L. The driving system for the shaft can be actuated electrically such that the contact from F (or from G) is maintained juxtaposed to L. This requires physically that the extension of the spring (or the polymer) is kept constant, and these are the requirements for creep (or stress relaxation) experiments. The positioning ring D can be used if necessary to keep the tube F in alignment. The polymer

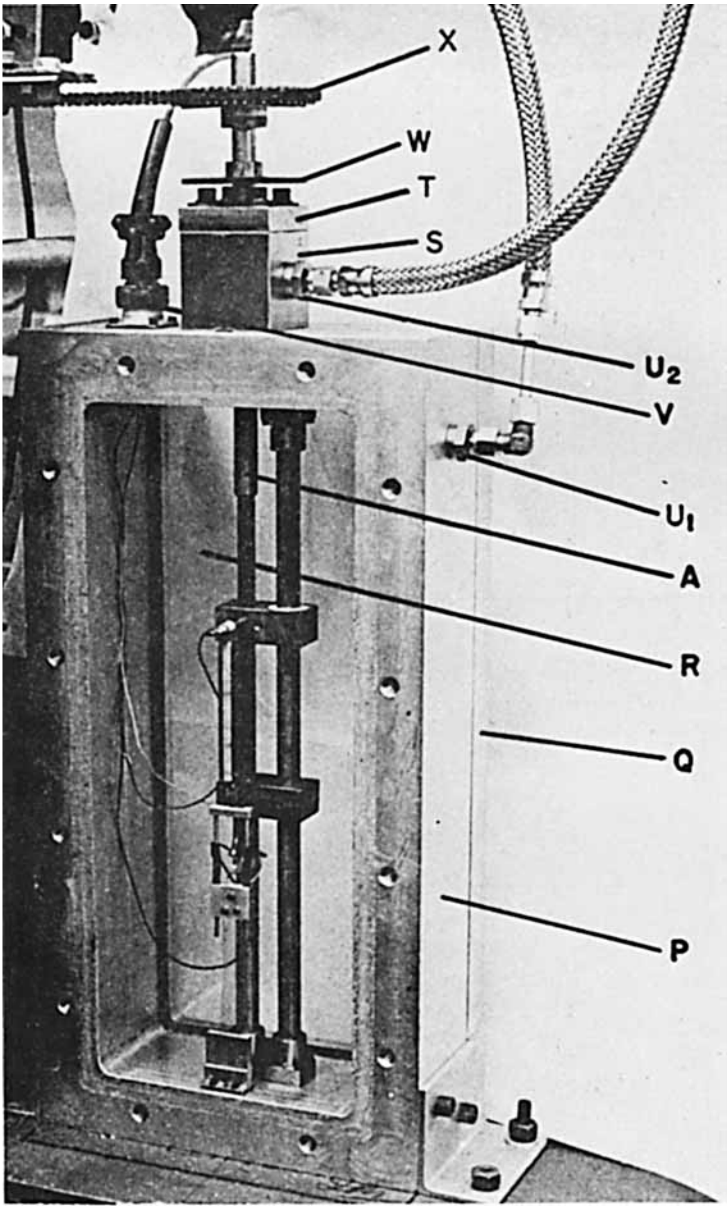

Fig. 2. Photograph showing housing construction, connections and accessories. Letters as referred to in text.

extension in stress relaxation is controlled by sliding G up or down H_2. The spring extension in creep is controlled by adjusting the height of L, which slides inside the clamp K, and of F, which in reality consists of two tubes one of which can slide inside the other. Large or small stresses can be applied by judicious choice of spring compliance.

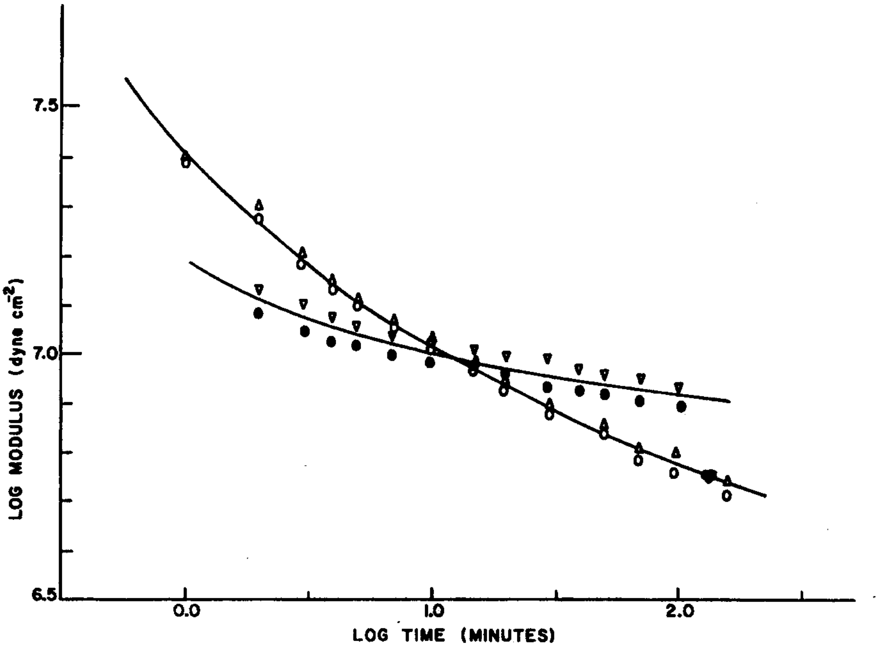

Fig. 3. Comparison of stress relaxation data. *Rhoplex AC-34 (23°C):* (▽) Instron tensile tester; (●) present instrument. *Vinyl acetate–dibutyl maleate copolymer (23°C):* (△) Instron tensile tester; (O) present instrument.

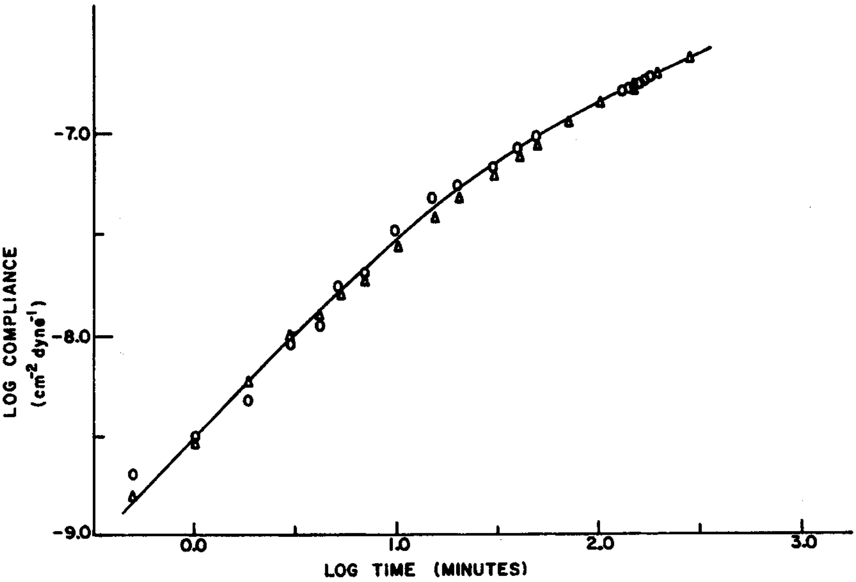

Fig. 4. Comparison of Creep data on vinyl acetate–dibutyl maleate copolymer (23°C): (△) regular laboratory creep measurement; (O) present instrument.

Figure 2 is a photograph showing the construction of the environmental chamber. P is a massive rectangular aluminum shell which serves as the housing and support for the parts shown in Figure 1. This type of housing and some other features of the chamber are similar to those utilized by Curran and co-workers[5] in a rather different type of stress relaxometer. The aluminum flanges Q (only one is shown in fig. 2 for the sake of clarity) hold slabs of plate glass R $^1/_2$ in. thick and are screwed into P. Neoprene gaskets $^1/_{16}$ in. thick form the vacuum seal between P and R, and gaskets of a foam rubber material $^1/_8$ in. thick are positioned between Q and R to distribute the stresses more evenly.

The rotary vacuum seal between the shaft A and the housing is accomplished by means of the drilled steel blocks S and T. Ethylene–propylene rubber O-ring seals are located in grooves cut between P and S and between S and T, and the blocks are screwed tightly on to the frame P. The chamber is connected to vacuum or to a vapor supply via U_1, which is an O-seal connector screwed tightly against the housing. A similar vacuum connection, U_2, is located on the block S between the rotary O-ring seals in order to improve the performance of these seals. Three electrical leads—from K, F, and G in Figure 1—are taken out of the chamber through a hermetically sealed connector V. The gear W drives a set of additional gears (not shown), which are used for measuring the number of shaft rotations.

The shaft is rotated from a chain drive around the sprocket X. A motor coupled with a clutch/brake unit make up the driving system. The maximum speed of rotation of the shaft was designed for about 600 rpm, but the electromagnetic clutch/brake unit (Stearns Electric Corp.) has a specially hardened armature so that low speeds can also be utilized. A relay serves to actuate either the clutch or the brake at any time and can be operated manually or automatically. Automatic operation enables the shaft to be driven or stopped whenever F or G makes contact with the frame L in Figure 1. Clutch operation when contact is broken will be referred to as relay setting 1, and when contact is made, as setting 2. The relay speed is fixed so that the speed of shaft rotation has to be chosen according to the degree of accuracy required in measurements of extension.

OPERATION

The polymer sample is screwed tightly into the upper and lower clamps, and the shaft is driven to position the block B such that the spring is in the equilibrium unstrained position. The contacts from F or G are adjusted to give the required spring or polymer extension according to whether a creep or stress relaxation experiment is to be performed. These adjustments can be made while viewing through a cathetometer. For creep, the relay setting 1 is used and the clutch is switched to automatic operation at zero time. The vertical travel of B is measured by rotation counting, and the polymer extension can be followed by viewing through a cathetometer. Creep recovery can be measured in a similar manner. For stress relaxation, the relay setting 1 is initially used. As soon as the polymer is strained, the relay setting is switched to 2 and the motor is reversed. The stress or spring extension is measured by rotation counting, and the consistency of the polymer strain can be checked by viewing through a cathetometer.

We have tested the accuracy of the instrument using two polymers: (1) Rhoplex AC-34 resin, a copolymer of esters of acrylic and methacrylic acid, supplied by Rohm and Haas Co.; and (2) a 75-25 vinyl acetate–dibutyl maleate copolymer supplied by Pittsburgh Plate Glass Company. Creep measurements were compared with those obtained from a standard laboratory creep test, while stress relaxation measurements were compared with results obtained from an Instron tensile tester. The results are shown in Figures 3 and 4 and indicate satisfactory agreement between the different methods of measurement. The largest differences were observed in stress relaxation for Rhoplex AC-34, where the Instron results yield values for the modulus about 10% higher than those from the environmental instrument. This is probably about the maximum experimental error involved, although in this particular case it may have been enhanced since different samples (from the same sheet, however) were used for the comparison.

The above results were obtained under atmospheric pressure. The ability of the instrument to hold a vacuum also has been tested. The chamber will pump down to and hold a vacuum of about 10^{-3} mm Hg for several hours. This is more than sufficient for many practical applications. The vacuum was scarcely affected by continuous or intermittent rotation of the shaft, so that the double seal utilized appears to be effective. Subsequent work has proved the continued effectiveness of the seal for a variety of organic vapors in the measuring chamber.

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