JOURNAL OF APPLIED POLYMER SCIENCE VOL. IV, ISSUE NO. 10, PAGES 34-37 (1960)

A Modified Helical Spring Stress Relaxometer for Automatic and Manual Operation

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Rubber elasticity theory predicts equality between the fractional decrease in stress during aging of an elastomer extended to a constant length and the fraction of the elastically active network chains which have been cut.[1] An instrument which measures this stress relaxation is valuable both in studying the mechanism of network breakdown and in comparing the efficiency of antioxidants in preventing network degradation.[2]

A simple device for this purpose, wherein the force in an extended strip of rubber was balanced by that in an extended helical spring and the proportional decrease in spring extension required to keep balance during aging was equated to the proportional force decrease, was described by Berry.[3] Berry's instrument for measurement of continuous stress relaxation in which the sample is held under continuous extension so that crosslinking during aging does not contribute to the stress has been somewhat modified to render it more versatile and convenient in use, the latest form being described below.

An automatic version of this apparatus has been constructed in which an escapement device is used which is similar in principle to that used by Bell[4] in a self-recording dilatometer. Reduction in the stress in the rubber strip as it ages allows one of the clamps holding it to move slightly, thereby closing a pair of contacts, completing an electrical circuit, and operating a device which reduces the spring extension by a definite amount. The closing of the contacts also causes actuation of a timing device. In this way are obtained a measured series of time intervals during each of which a constant decrement in the tension has occurred. From these measurements a complete stress–time curve of high accuracy can be constructed.

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CONSTRUCTION OF THE RELAXOMETER

Samples for examination in this instrument take the form of strips, 0.5 cm. wide, 0.02 cm. thick and 4 cm. long, cut from larger sheets by means of a special cutter already described.[3]

The strip R under test is gripped between stainless steel clamps E and F (Fig. 1), each consisting of two flat plates the front one of which is rounded so as to obviate premature strip rupture. The upper clamp E is attached through a swivel to the lower end of a 5-cm.-long helical steel spring S, the force in which is used to balance the stress in the extended strip. The upper end of S is attached to a capstan on the block D, whose position on the hollow brass framework AB may be adjusted by turning a steel screw Z, having 40 turns/in. by means of the slotted head H. The lower clamp F is fixed to a block whose position on the framework may be adjusted by rotation of the knurled head of steel screw X. Such an adjustment is necessary in order that relaxation may be examined at any required strip extension.

The balance point between the stress in the strip and the force in the spring is differently indicated, according to whether the instrument is automatically or manually operated. For automatic operation this is indicated by contact between a round-headed platinum stud, soldered face upwards on a tongue in front of the stirrup carrying E, and a flat platinum stud, soldered face downwards onto a copper bar protruding through a hole in the stirrup and screwed into block G. G is made of nylon so as to insulate the contact from the remainder of the apparatus and its position on the framework AB may be adjusted by rotation of steel screw Y by means of the knurled head. In manually operated instruments, balance is indicated by coincidence between a pointer P on the stirrup carrying E and a hairline on block G which may, as in this case, be made of brass.

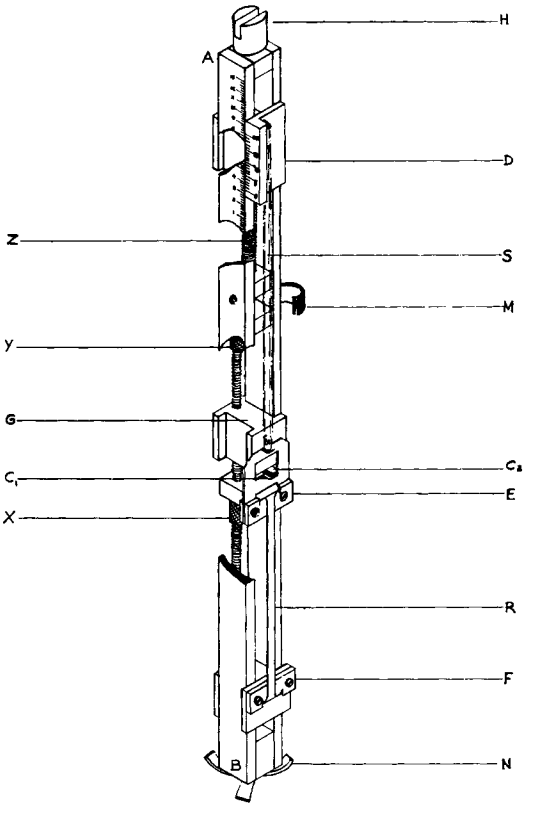

Fig. 1. Diagram of automatically operated stress relaxometer.

The apparatus is zeroed with the upper end of the strip clamped in E. Block D is lowered to the zero position on the vernier scale engraved on the side of the instrument, and the position of G is then altered by rotating Y until the balance point is obtained. The lower end of the strip is then clamped in F, and by rotation of X it is extended by the required amount (usually 100%), which may be deduced by measurement of the distance between fine bench marks drawn on the strip (about 5 mm. from the clamps) by means of a solution of black rubber in benzene. Finally H is rotated until balance is restored, the vernier reading then being proportional to the force in the strip.

The zeroing of the instrument is so arranged that the vernier reads directly the spring extension, and, since the springs obey Hooke's law at the extensions employed, the ratio of the vernier reading at time t to that at zero time equals the ratio of forces exerted by the strip at time t and at zero time. Although calibration of the helical springs is not necessary in this apparatus, springs with a range of moduli are required so that samples of differing degrees of crosslinking may all be examined at a high initial vernier reading to ensure the maximum accuracy. A set of such springs has been prepared by Pocklington and Johnson Ltd.

The relaxometer is inserted into a Pyrex jacket (Fig. 2) in which it is centrally located by means of a pair of shaped phosphor bronze springs P. Rotation of the relaxometer is prevented by a y-shaped foot N engaging three pips inside the base of the jacket. The foot also allows for adjustment of the total length of the relaxometer, since its shank is threaded and provided with a locknut.

For experiments in oxygen or under vacuum the jacket may be closed by a special stopper containing a turnkey K the glass spade of which engages in H. The turnkey may conveniently be lubricated with a lithium stearate grease (kindly donated by Dr. I. E. Puddington),[5] since such a grease does not streak even after repeated turning of K. For investigation of thermal aging, the apparatus is set up in its jacket and placed in a silicone oil thermostat at the desired temperature. After it has been placed in the thermostat, the apparatus is evacuated and filled with oxygen to the desired pressure through stopcock T, which is attached to a side arm of the tube through a B24 joint.

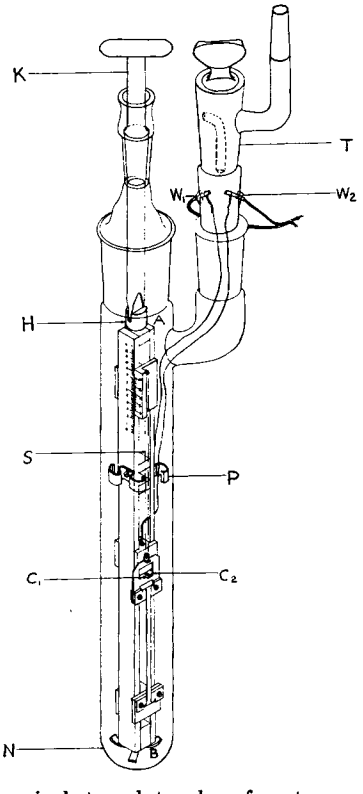

Fig. 2. Pyrex jacket and turnkey for stress relaxometer.

Since the force in the strip is proportional to the absolute temperature, the length of spring S has initially to be increased by turning H until thermal equilibrium is attained. The initial force is taken as the maximum value attained, and in manual experiments vernier readings are subsequently taken at 5-min. intervals. The instrument is suitable for use in investigating photochemical as well as thermal degradation, since the vernier engraved on the side of the instrument may be observed through a window in the thermostat at right angles to the incident light.

AUTOMATIC OPERATION OF THE RELAXOMETER

For automatically operated relaxometers, provision must be made for taking leads from C_1 and C_2 out of the relaxometer jacket (Fig. 2). To achieve this, tungsten wires W_1 and W_2 are sealed below stopcock T diametrically opposite each other.

Leads from C_1 and C_2 are attached to W_1 and W_2, respectively, by means of small brass collars on the end of helical steel springs. Outside the jacket, leads from W_1 and W_2 are connected to a relay which is energized whenever C_1 and C_2 make contact. The impulse from this relay operates a Ledex rotary solenoid (National Switch Factory Ltd.) (Fig. 3), which is attached to K by a grapple

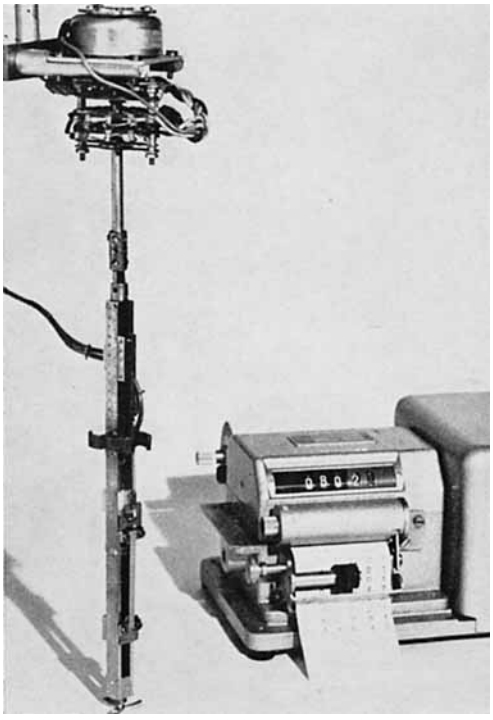

Fig. 3. Photograph of automatically operated stress relaxometer and time printer.

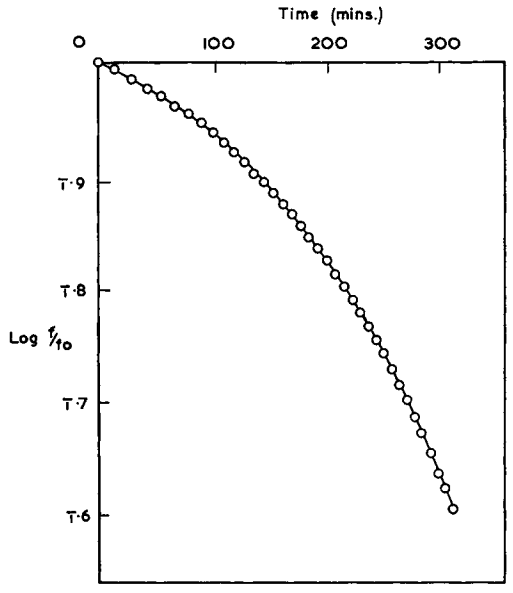

Fig. 4. Plot of log force vs. time for data on photochemical degradation obtained by use of automatically operated stress relaxometer.

and also energizes the pen of the operations recorder (Evershed and Vignoles Ltd.) or operates an electrically operated revolution counter with built-in synchronous motor which prints the time between operations to 0.01 min. (1Vo) time printer, Radiatron Ltd.). The Ledex circuit is so arranged that, once an impulse is received, the head H is rotated through 360°, representing a decrease of 0.085 in the vernier reading. Provided that the initial vernier reading is known, the entire relaxation curve may be reconstructed from the times of successive operations of the Ledex.

Very smooth relaxation curves have been obtained by use of the automatically operated relaxometer (Fig. 4), and the arrangement has the great advantage of being able to operate unattended for days if necessary, the system being adequately protected by inclusion of a 100-ma. fuse in the Ledex circuit.

We are indebted to Mr. B. Thorby of our Engineering department who prepared Figures 1 and 2.

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Synopsis

A relatively simple and inexpensive automatic and self-recording apparatus giving good accuracy has been developed for measurement of chemical stress relaxation in elastomer vulcanizates. The apparatus is based on a previously described helical spring relaxometer intended for manual operation. Several improvements applicable to this manually controlled instrument are also described.

Résumé

On a construit un appareil enrégistreuer relativement simple, bon marché, automitique et précis pour la mesure du relâchement de tension chimique dans les vulcanisats élastomères. L'appareil est basé sur un rélaxomètre à rèssort hélicoïdal manipulé à la main; on a également décrit plusieurs perfectionnements de cet appareil à contrôle manuel.

Zusammenfassung

Ein verhältnismässig einfacher und billiger, automatisch arbeitender und selbst-schreibender Apparat, der eine gute Genauigkeit leifert, wurde zur Messung der chemischen Spannungsrelaxation von elastischen Vulkanisaten entwickelt. Der Apparat beruht auf einem früher beschriebenen Spiralfeder-Relaxometer mit Handbedienung; einige Verbesserungen, die auf dieses von Hand bediente Instrument anwendbar sind, werden ebenfalls beschrieben.

Received January 23, 1960