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An Apparatus for the Measurement of Dynamic Mechanical Properties of Polymers in a Gas Medium with the Vibron Viscoelastometer

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Synopsis

The Vibron Viscoelastometer is useful for obtaining dynamic mechanical properties of film and fibers in atmospheres at 0 and 100% relative humidity. No apparatus has previously been available for obtaining dynamic mechanical properties in a gas medium; however an apparatus and procedure have been developed for measuring these dynamic properties in a gas medium using the Vibron instrument. This technique will be useful in studies on the effects of different gases on polymers in the process and on the effects of gas treatments on materials. A new gas cell which is adaptable for other mechanical testing instruments such as the Instron, Tensometer, and Torsion Pendulum is also discussed.

INTRODUCTION

The Rheovibron Viscoelastometer (Toyo Measuring Instruments) is useful for obtaining dynamic mechanical properties of films and fibers over a wide temperature range. Numerous studies of dynamic mechanical properties have been made in the temperature range of -160 to 250°C in an atmosphere at 0% relative humidity.

Studies on dynamic mechanical properties at 100% relative humidity and in a liquid medium have been reported by several investigators.[1-6] However, no studies exist which show the measurement of dynamic mechanical properties of materials in a gas medium using the Rheovibron instrument.

In this paper, a new method for investigating dynamic mechanical properties of materials in a gaseous medium is presented, which shows the change in viscoelastic properties during HCl sorption of nylon 66 fiber.

EXPERIMENTAL

The Rheovibron applies a sinusoidal tensile strain to one end of a sample and measures the stress output at the other end. The instrument operates at frequencies of 3.5, 11.0, 35.0, and 110 cps. Two transducers are used to read directly the absolute dynamic modulus $|E^*|$ (the ratio of maximum stress amplitude to maximum strain amplitude) and the phase angle δ between stress and strain. From these two quantities, the real part E' (dynamic modulus)

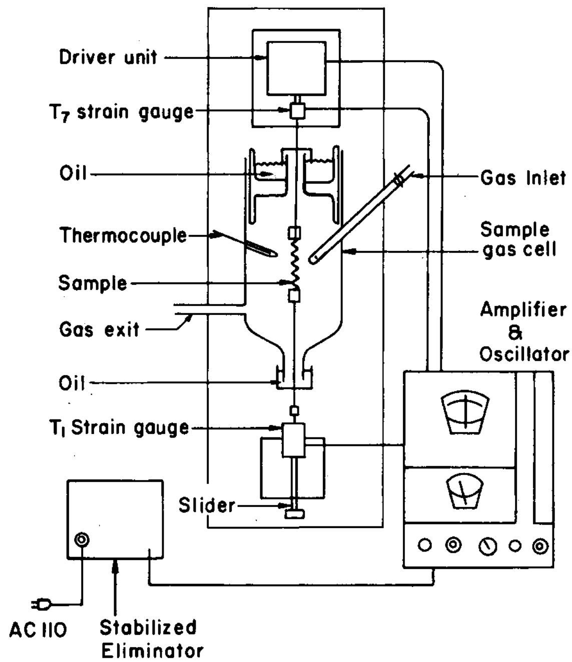

Fig. 1. Schematic of apparatus.

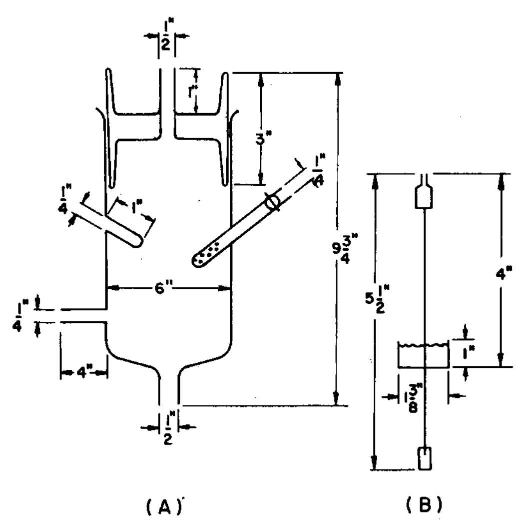

Fig. 2. Gas cell (A) and clamp with liquid seal (B).

and the imaginary part E'' (loss modulus) can be calculated from the complex dynamic tensile modulus $|E^*|$. The principles of this direct-reading method and instrument are described in detail by Takayanagi.[4]

To measure the dynamic properties in a gas medium it was necessary to devise an apparatus to keep the sample immersed in gas and the transducers outside the gas, yet aligned with the sample. This was accomplished by using the sample gas cell with two liquid seals. The driving unit, T_7 strain gauge, the sample gas cell, T_1 strain gauge, and slider were mounted vertically on a stand.

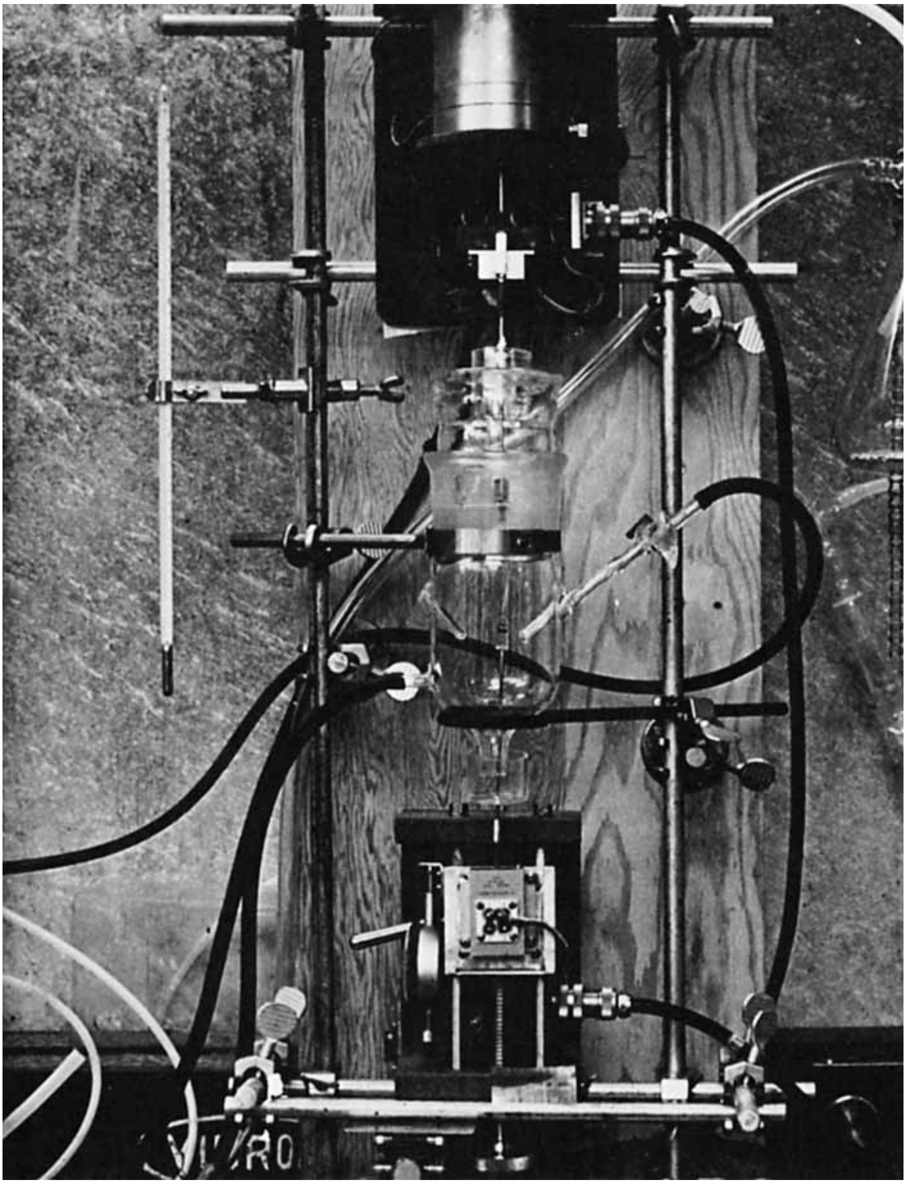

Fig. 3. Illustration of gas cell.

A sketch of the apparatus is shown in Figure 1. The amplifier with oscillator and the stabilized eliminator are connected to the strain gauges and the driving unit.

The sample gas cell is made from a 6-in. diameter, $9^3/_4$-in. length of glass. The upper part of the cell has a $^1/_2$-in. diameter, a 1-in.-high glass tube for the inlet of sample clamp, an oil deposit portion, and a ground glass tight seal about $1^1/_8$ in. The lower part of the cell consists of a $^1/_4$-in. diameter glass tube gas inlet, a $^1/_4$-in. diameter 1-in.-long gas outlet, a $^1/_2$-in. diameter 1-in.-long sample

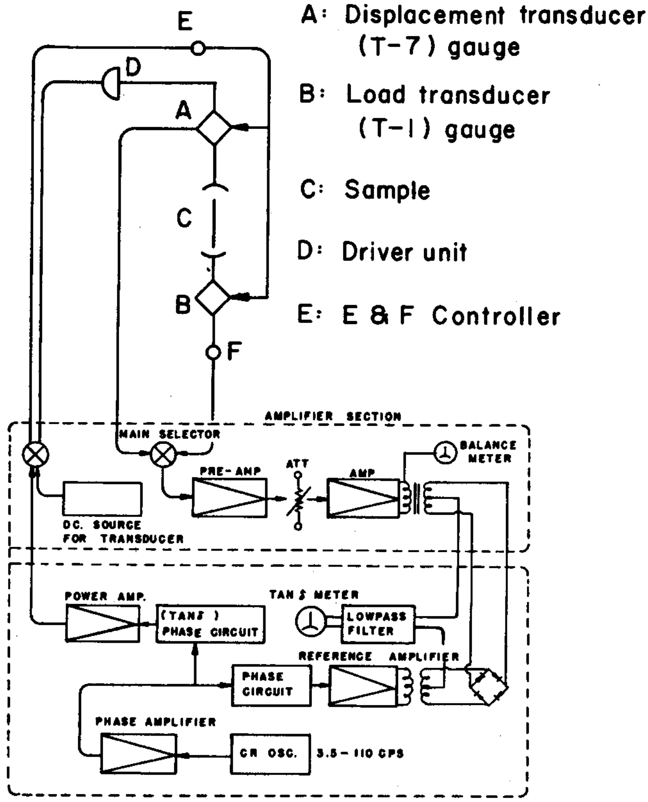

Fig. 4. Block diagram of apparatus.

clamp inlet, and a $^1/_4$-in. diameter 1-in. long thermocouple inlet. The gas cell is shown in Figures 2A and 3. The volume of the gas cell is about 1 liter.

In order to insert a sample into the cell without escape of gas from the inlet of the sample clamp, the liquid seal with clamp rod is designed as seen in Figure 2B. The clamp rod is connected to the liquid seal which is made from a polyethylene cap ($1^3/_8$-in. diameter, 1 in. high). One pair of clamp rods with a liquid seal is passed through a $^1/_2$-in. diameter inlet in the center of the gas cell, as seen in Figures 2 and 3.. The weight of the clamp with rod is controlled at 11 g. The total weight of the clamp and seal cap with oil is about 25 g. Fisher paraffin oil (15 ml) is used as an oil seal. The circuit of the Vibron is modified as shown in Figure 4. A new controller (E,F) is built to adjust the resistance of T_1 and T_7 gauges since the arrangement of the driving unit and clamps is changed.

RESULTS AND DISCUSSION

To demonstrate the apparatus and procedure, the dynamic mechanical properties of dry nylon 66 filaments were measured in three different gas mediums (100:0, 50:50, and 25:75 HCl/N_2 gas ratios) at 11 cps as a function of time at room temperature (25°C).

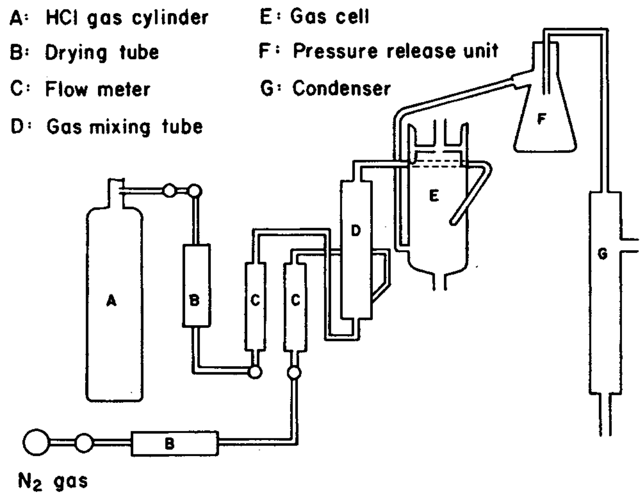

Fig. 5. Gas control system for sample gas cell.

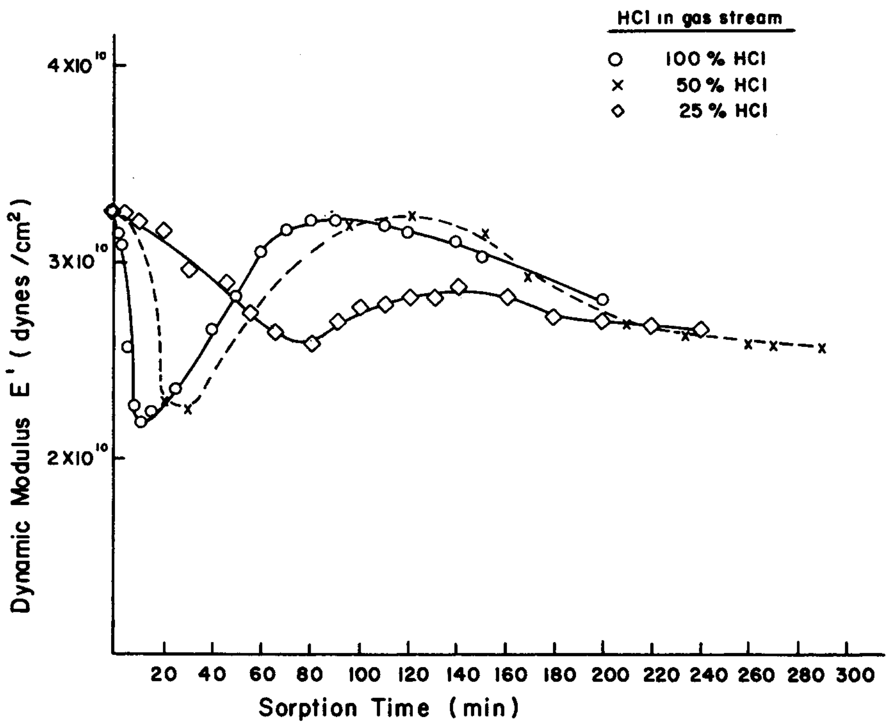

Fig. 6. Effect of HCl sorbed on the dynamic modulus of nylon fibers.

This new apparatus for the immersion of the test sample and the gas control system are placed in a hood to avoid the health hazard of HCl gas. The gas concentration is adjusted by the gas control system, as seen in Figure 5. Data for dynamic modulus are shown in Figure 6, and loss tangent (tan δ) in Figure 7. These results indicate that the tan δ and dynamic modulus (E') of dry nylon fiber gassed to equilibrium are identical at HCl concentrations of 25, 50, and

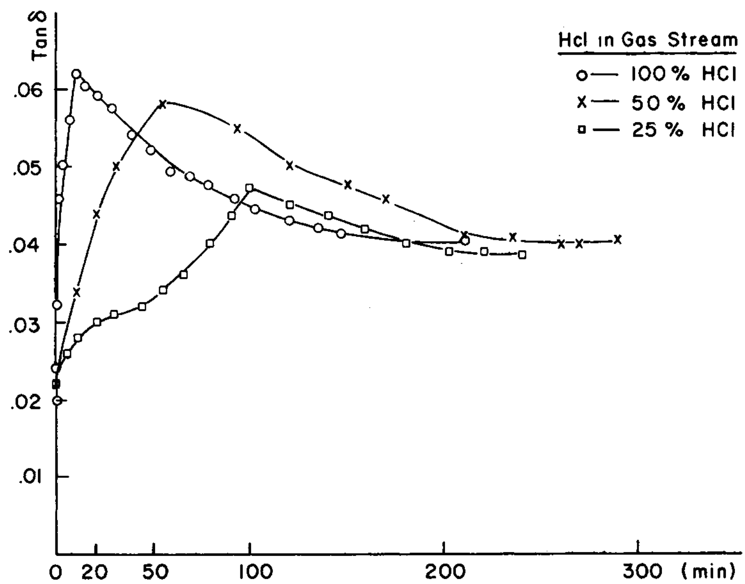

Fig. 7. Effect of HCl sorption on loss tangent (tan δ) of nylon 66 fibers.

100% HCl. Short-time gassing data indicate that the HCl sorption by nylon 66 is exothermic, and equilibrium sorption of HCl by nylon 66 doubles tan δ and reduces the dynamic modulus (E') by 20%.

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