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Thermal Expansion Measurements of High-Temperature Carbon/Carbon Materials from Room Temperature to 3000 K[1]

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The thermal expansion of carbon/carbon material has been measured to 3000 K using a variety of methods. Coupon samples have been measured in a direct-view dilatometer and an automatic recording dilatometer. Cylindrical or hoop samples, 5 to 50 cm in diameter, have been measured by a fiber-wrap technique. The direct-view or, as it is sometimes referred to, the twintelemicroscope method is generally used when good accuracy is required for measurements to elevated temperatures. The change in length is observed with the use of telemicroscopes aligned on fiducial marks machined on opposite ends of the specimen. An Apple IIe computer controls the automatic recording dilatometer and also provides an output to an X–Y recorder, giving a continuous curve of thermal expansion percentage versus temperature through the range from RT to 300 K. Circumferential thermal expansion is defined as the change in circumference per unit of initial circumference. The change in the circumference of the hoop is measured by wrapping the hoop with a graphite yarn and measuring the change in length with a linear variable differential transformer.

KEY WORDS: carbon/carbon materials; dilatometry; high temperatures; thermal expansion.

1. INTRODUCTION

When heat is added to or removed from a body, so that there is a change in its temperature, there is a corresponding change in its length. Exceptions occur, however, in some specially prepared alloys and composite materials in which the various components have dissimilar or unique expansion

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characteristics. Frequently, thermal expansion is related to temperature through a coefficient (α) such that as a body is heated from T_1 to T_2, its length change, L_1 to L_2, is expressed by the following:

$$\alpha = \frac{(L_2 - L_1)}{L_1(T_2 - T_1)} \tag{1}$$

where L_1 is the length at T_1 and L_2 is the length at T_2. More generally, the change in length of a body as heat is added or removed is expressed as a percentage expansion for a specific temperature change.

Many methods have been developed for measuring thermal expansion, which may be grouped as either *relative*, in which expansion of the material is measured relative to the expansion of a reference material, or *absolute*, in which expansion of the material is measured directly. We describe variations to a number of standard techniques, for automatic control, as well as a unique method for measuring changes in circumference of a cylindrical sample.

2. COUPON SAMPLES

Coupon samples, as defined here, are specimens of a uniform cross-sectional area and 5 cm long. These samples have been measured in the dilatometer as described in the following sections.

2.1. Quartz Tube Dilatometer

In this procedure, illustrated schematically in Fig. 1, the specimen is supported between members of a quartz frame and pushrod assembly. The assembly is inserted into a furnace capable of uniformly heating the

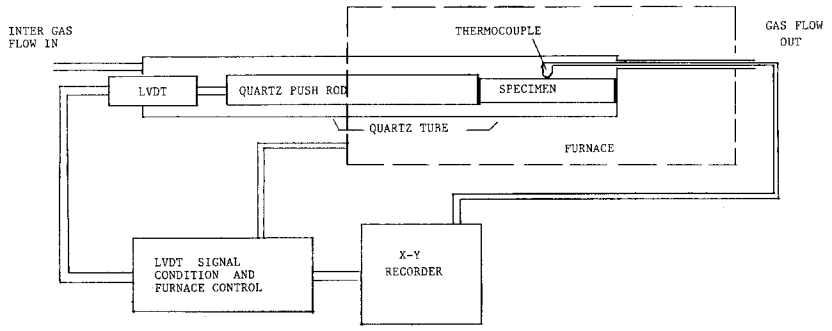

Fig. 1. Quartz dilatometer.

specimen zone. As the spcimen's temperature is changed, changes in its length result in relative displacement of the quartz push-rod and frame assembly. The amount of displacement is sensed by a linear variable differential transformer (LVDT) and recorded on one scale of an X–Y plotter. Specimen temperature, sensed by a thermocouple, is recorded on the other scale. Thus, a continuous record of dilation versus temperature is produced.

Ideally, the specimen is a 5-cm-long rod nominally 0.6×0.6 cm. However, many other sizes and configurations can be accommodated by appropriate selection of control and boundary conditions.

The quartz dilatometer can be used for thermal expansion measurements through the range from 80 to over 100 K. This is the range over which the expansion characteristics of quartz have been well defined by the U.S. National Bureau of Standards. Actually, the measurement range can be extended to over 1200 K, providing the devitrification temperature of the fused silica is not reached.

The accuracy of data produced by the quartz dilatometer is checked via measurements of reference standard materials acquired from the National Bureau of Standards.

2.2. Automatic Recording Dilatometer

This apparatus operates on the push-rod principle but measures the difference in dilation of two specimens of equal length as they are uniformly heated or cooled through the temperature range of interest. Twin push rods, which rest on the specimens, are fixed at their opposite ends to the core and the body of an LVDT. Knowledge of expansion characteristics of one specimen permits evaluation of the dilation in the other since the output is proportional to the difference in expansion. This difference in signal is electronically converted to percentage expansion and is recorded continuous, along with the specimen temperature, during the run. Temperature is measured by either a thermocouple or a radiometer. The major components of this facility are schematically illustrated in Fig. 2.

The preferred specimen geometry is a one-piece rod, 5 cm long, with either 0.7-cm diameter or 0.7-cm square cross section.

The apparatus utilizes a LVDT to sense and quantity dilation difference between the reference specimen and the unknown. This instrument provides a linear range of ± 0.250 cm and a sensitivity of 2.5 mV per 3×10^{-3} cm displacement. Displacements of 25×10^{-6} cm can be detected reliably.

Specimen temperatures are measured by either thermocouple or radiometer. If a thermocouple is used, it is selected on the basis of chemical

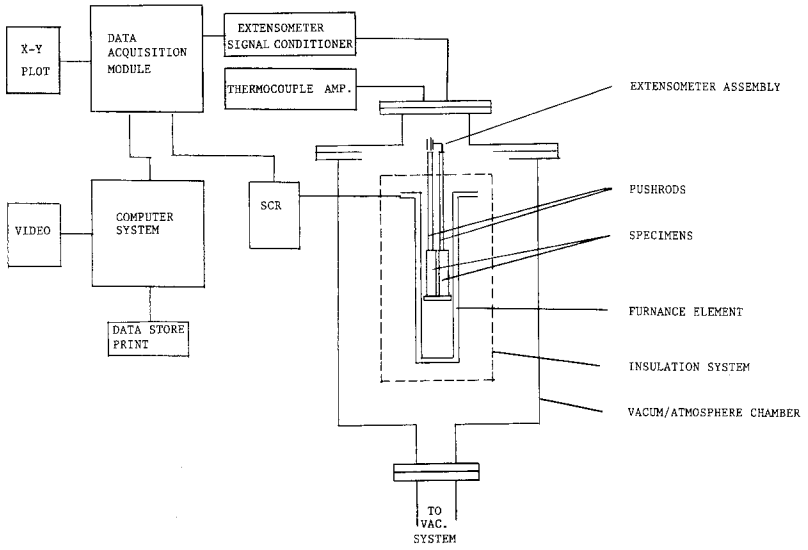

Fig. 2. Schematic diagram of automatic dilatometer.

compatibility with the environment, temperature range, and stability of performance in that range. Calibration must be within that specified by appropriate ANSI designation.

The specimen and its reference are uniformly heated inside a vertical tubular element which is powered by a controlled 27-kVA suply. The element is fabricated of a material suitable for the temperature range to be studied and compatibility with other components. Elements of graphite, tungsten, tantalum, molybdenum, or the nickel–chromiun alloys can be adapted.

The element and an apropriate insulation system are installed into an evacuable chamber. The two-stage vacuum pumping system is capable of achieving pressure in the low 10^{-5} Torr range within the chamber. Expansion measurements usually are made under inert gas.

A microcomputer is used to control all the various functions of the dilatometer. Prior to each run, the computer prompts the operator to select such test parameters as maximum temperature, heating and cooling rates, holds, and delayed start option. On signal from the operator, the computer applies heater power to start the run. A CRT displays indicate pertinent data during the run, including temperature in various scales, power input, specimen expansion percentage, and elapsed time, all at 1-s intervals. These data are recorded at selected intervals for printout. Closed-loop temperature feedback in software ensures that the heating ramp is following

the selected profile. The computer monitors signals from the sensor instruments and automatically makes range changes when necessary. When the desired peak temperature has been reached, the computer checks for any hold intervals and, if there are none, begins to lower the heater power at the preselected rate. When room temperature is reached, the run is terminated.

In addition to the dilatometer control functions, the computer acquires data from the temperature and displacement transducers as the test proceeds. These raw data are reduced to engineering units in real time, and from them the computer provides a screen update in tabular form, a continuous hard-copy plot of expansion percentage versus temperature, and a floppy disk file of the data. On completion of the run, the disk file is used to generate report-quality tables and graphs of the data.

On confirmation that each instrument of the apparatus is functioning within its proper calibration limits, the apparatus accuracy is confirmed by thermal expansion measurement of a reference standard material. If a deviation from the reference data occurs during this run, and confirming runs, the deviation is used to apply a calibration correction.

Thermal expansion reference standards used in this instrument include Type ATJ graphite, fused silica, borosilicate glass, and copper. Samples of some of these were obtained from the U.S. National Bureau of Standards, while the others are considered reliable standards using published literature data.

3. HOOP EXPANSION

The need to establish thermal expansion properties of directionally oriented fiber-reinforced composites has accelerated the development of new measurement apparatus and methods, designed specifically to evaluate shapes and components of these structures in their as-fabricated condition. Obviously, a coupon specimen removed from a complex composite structure for property measurement cannot always represent the strucjture adequately. It becomes necessary to use whole sections of the structure as the specimen, in order to obtain useful data. One example is the circumferential thermal expansion measurement of a circumferentially reinforced carbon–carbon cyclinder used in rocket engine nozzles. Use of a section of the cylinder or hoop specimen is necessary to include of integral circumferential yarns.

3.1. Fiber-Wrap Technique

In this method, the circumferential expansion of a hoop specimen on heating is determined by measuring the effective length change of a yarn

wrapped around it. The yarn ends are attached to the cores of two linear variable differential transformers, which are in turn attached to a common fixed reference. Expansion characteristics of the yarn are measured separately. Output from the LVDTs thus provides information quantifying the change in hoop circumference.

The hoop specimen is heated by passing an electrical current directly through the sample as shown in Fig. 3. Specimen temperature is sensed by a radiometer. The specimen–yarn assembly, with electrodes and suitable insulation, is contained inside an evacuable chamber. The radiometer views the specimen through a port window.

Signals from the LVDTs and the radiometer are processed by a computer system, which scans and records their outputs at nominal 1-s intervals. All information i retained for later tabular and graphic printout of expansion percentage versus temperature.

The circumferential thermal expansion is calculated from the following equation:

$$TE_C = \frac{L_2 - L_1}{L_1} \Bigg| T_1 \text{ to } T_2 \tag{2}$$

where is TE_C is the circumferential thermal expansion, L_1 is the initial circumference at T_1, and L_2 is the circumference at T_2. The data are needed as a percentage change of the initial circumference.

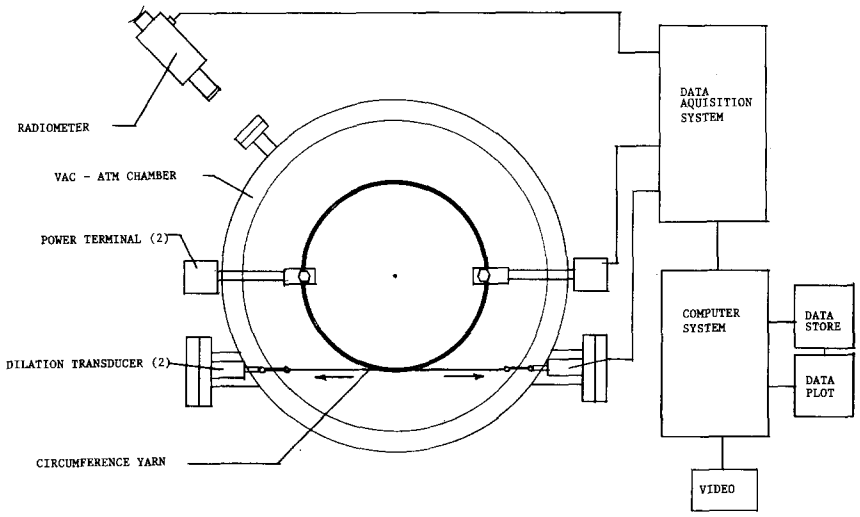

Fig. 3. Schematic diagram of hoop thermal expansion apparatus.

The information can also be given as the coefficient of circumferential thermal expansion expressed by the following equation:

$$\alpha_C = \frac{L_2 - L_1}{L_1(T_2 - T_1)} \tag{3}$$

where α_C is the coefficient of circumferential thermal expansion, L_1 is the circumference at T_1, and L_2 is the circumference at T_2.

The change in length of the hoop circumference, as measured by the graphite yarn, is corrected for the expansion of the yarn by the following equation:

$$\Delta L_C = \Delta L_{LVDT} + \Delta L_Y \tag{4}$$

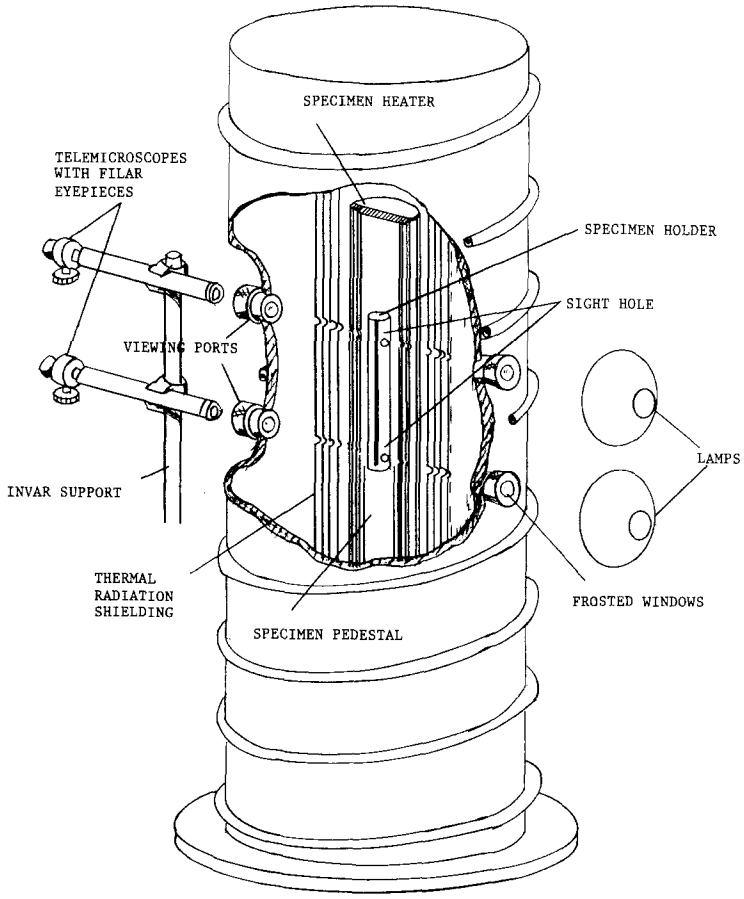

Fig. 4. High-temperature optical dilatometer.

where ΔL_C is the corrected thermal expansion, ΔL_{LVDT} is the expansion measured by the LVDTs, and ΔL_Y is the expansion of the yarn.

4. YARN EXPANSION

The expansion of the yarn is obtained by direct measurement in the optical dilatometer.

4.1. Optical Dilatometer

The procedure described here is sometimes referred to as the twin-telemicroscope method. In this procedure, illustrated schematically in Fig. 4, a yarn specimen is supported vertically on a pedestal inside a tubular furnace element, which is long enough to achieve a uniform temperature along the specimen length. Thin graphite washers are atached to the yarn, with graphite cement, 9 cm apart. The telemicroscopes are aligned with the washers and the L is measured using a calibrated filar eyepiece in the telemicroscopes.

The optical dilatometer is used routinely for measurements in the range 300 to 3000 K. For the range up to 1000 K, specimen temperatures are measured using a thermocouple. Above 1000 K, temperatures are measured using an optical pyrometer. Dilation data are recorded under conditions of thermal equilibrium. Changes in specimen length of the order of 20 μin. can be evaluated by this measurement technique. Verification is derived through calibration of the microscope micrometer filar eyepieces using a calibrated glass slide. Accuracy is confirmed by measurement of reference standard materials acquired from the National Bureau of Standards, including SRM-737, and various dense graphites, particularly type ATJ, for which good consensus data are available.