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A vitreous silica tube dilatometer for the measurement of thermal expansion of solids from −195 to 1000° C

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A vitreous silica tube dilatometer to measure the thermal expansion of solids from − 195 to 1000° C in air is described. Two tests are made to cover the temperature range − 195 to 1000° C, one from room temperature to − 195° C using a cold chamber and liquid nitrogen as the coolant, and one from room temperature to 1000° C using a resistance-wound furnace to heat the specimen. The specimen is $\frac{1}{4}$ in. diameter and 2 in. long. Data accurate to ± 2% can be obtained during routine testing.

1. Introduction

The thermal expansion of materials is an important physical property to consider when temperature changes or temperature gradients are expected in a design. Without its consideration, thermal stresses large enough to destroy the part may result. Undesirable deformation can occur as a result of thermal mismatches, as can failure of bonded joints. For these reasons, it is important to know the thermal expansion of the materials in a design. The thermal expansion often cannot be calculated analytically; it must be measured. It is therefore necessary to have equipment to measure the thermal expansion accurately.

Most industrial designs operate in the temperature range −195 to 1000° C. For this reason, equipment was designed to measure the thermal expansion in this temperature range. In this system two runs are necessary; one from −195° C to room temperature, nominally 25° C, and one from room temperature to 1000° C. A vitreous silica tube, which is stable at temperatures up to about 1000° C, is used to hold the specimen.

A cooling chamber is used to cool the specimen from room temperature to −195° C and a resistance furnace is used to heat the specimen from room temperature to 1000° C.

The dilatometer described here has been used to test several thousand specimens for Westinghouse engineers. Many improvements have been added over the past several years, so that it is now a fast, efficient, reliable instrument, and is used routinely on a daily basis.

Other thermal expansion systems for higher temperature testing in air or vacuum are described in [1] and [2].

2. Description of dilatomer elements

The dilatometer, shown in Figs. 1 and 2, is composed of a furnace or cold chamber which is used respectively to heat or cool the specimen, a fused silica tube and rod assembly, and a transducer system to measure the expansion of the specimen.

In this dilatometer, the specimen rests in the horizontal position at one end of the tube as shown in Figs. 1 and 2. The dilatometer is constructed on a steel rail base $14\frac{1}{4}$ in. wide by 38 in. long to which are attached two pedestals. One pedestal supports the furnace or cold chamber and the other the transducer and tube and rod assembly. The furnace pedestal is fixed at one end of the base by two screws. The transducer pedestal can slide on the base at the other end to move the tube in and out of the furnace or cold chamber. The furnace and chamber are controlled with a program controller and the temperature and thermal expansion of the specimen are recorded on an $X-Y$ recorder.

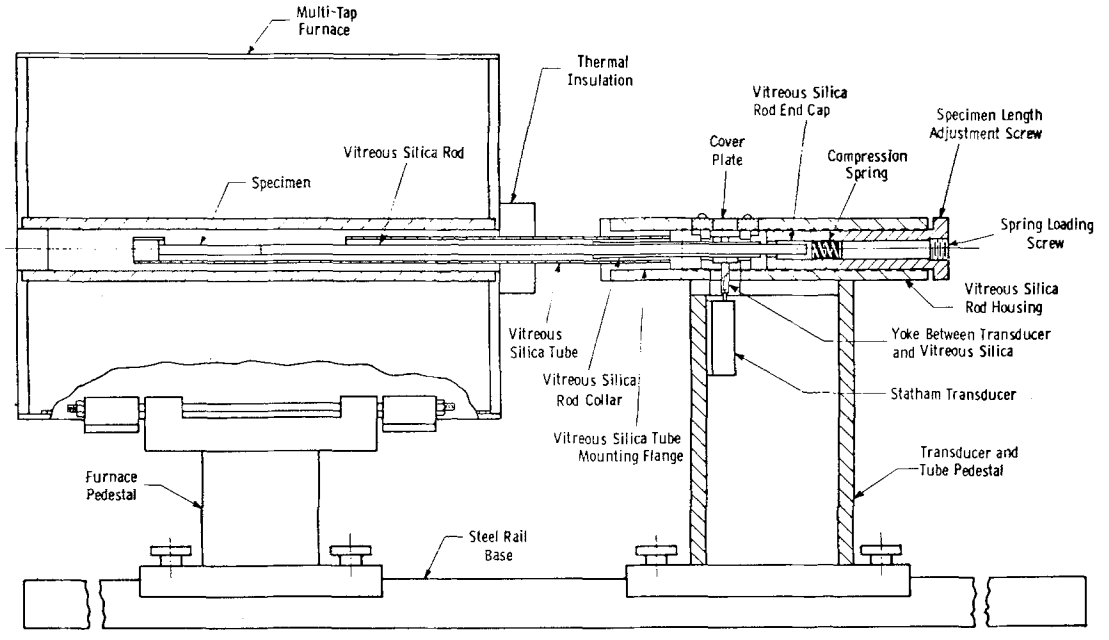


Figure 1 Cutaway view of vitreous silica tube dilatometer.

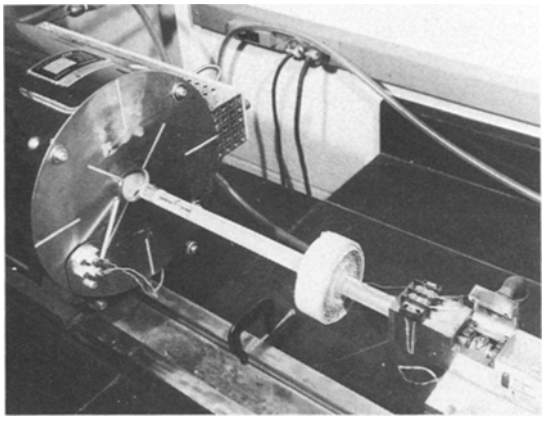


Figure 2 Specimen in vitreous silica tube.

2.1. Pedestals

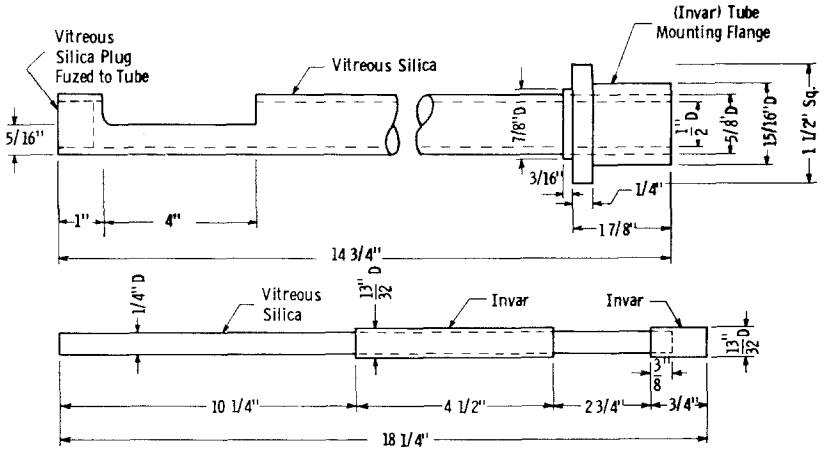
The furnace and cold chamber pedestal is a heavy wall tube with a plate welded to one end and a channel welded to the other end. The channel rests on the steel base and is attached to it with screws. Two lugs are welded to the plate, one at each side, and each lug is made with a centre hole parallel to the base. Four lugs, with centre holes, are attached to the furnace; when the furnace is placed on the top plate of the pedestal the plate lug centre holes are in line with the furnace lug centre holes. Two steel rods, one on each side of the plate, are passed through centre holes to hold the furnace to the pedestal. Similarly, the holes in the mounting plates of the cold chamber are used to fix it to the pedestal.

The transducer and tube pedestal is a stainless steel box made from $\frac{3}{8}$ in. thick plate. The box is mounted on a channel with screws and the unit slides on the base, as shown in Figs. 1 and 2. The housing through which the vitreous silica rod passes is attached to the top plate of the pedestal. This housing also contains the specimen length adjustment screw, the spring loading screw and spring and the Invar tube mounting flange, shown in Fig. 1. The measuring transducer is mounted on the inside of the front plate of the pedestal as shown in Fig. 1 and can be removed for repairs, since screws are used to hold the plates together.

Although the dilatometer was designed primarily for testing materials in air, provision is made to flow an inert gas such as argon into the tube, for those cases where some protection against oxidation is desired. The transducer cover plate, shown in Fig. 2, is made with a pipe connection through which the inert gas can be intoduced. The gas flows through the vitreous silica rod housing down the tube to the specimen and out into the furnace.

2.2. Vitreous silica tube and rod assembly

The tube, which is $\frac{7}{8}$ in. o.d. × $\frac{1}{2}$ in. i.d. × $14\frac{3}{4}$ in. long, is mounted horizontally on one pedestal. One end of the tube has a cut-out 4 in. long so that the specimen can be inserted; the opposite open



Note: All Invar Parts Cemented to Vitreous Silica Rod with Micro-Measurements, Inc. 610 Cement

Figure 3 Vitreous silica tube and rod dimensions.

end has an Invar flange cemented to it which is used to attach the tube to the square housing above the transducer, as shown in Fig. 1. An Invar tube $\frac{13}{32}$ in. o.d. by $4\frac{1}{2}$ in. long is cemented to the $\frac{1}{4}$ in. diameter × $18\frac{1}{4}$ in. long rod $10\frac{1}{4}$ in. from one end and an Invar cap is cemented to the opposite end of the rod as shown in Fig. 3. In the tube, the end of the silica rod contacts the free end of the specimen; a spring load of about three ounces is applied to the capped end to keep the rod, specimen, and bottom of the tube in good contact at all times. A yoke from the measuring transducer is fixed to the Invar tube (which is cemented to the rod) with a screw as shown in Fig. 1. As the specimen expands, it pushes the rod; the rod movement is transmitted to the measuring transducer through the yoke.

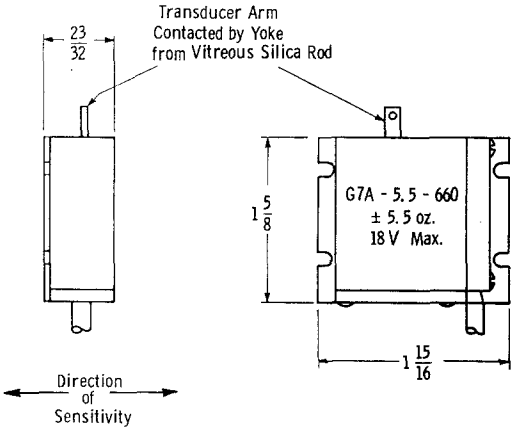


Figure 4 Model G7A-5.5-660 Statham displacement transducer for thermal expansion measurement.

2.3. Transducer, recorder and thermocouple

The transducer used to measure the thermal expansion is a Statham displacement transducer, Model 67A-5.5.5-660, whose dimensions are given in Fig. 4. The transducer is excited by a d.c. power supply and the excitation voltage level determines the sensitivity of the transducer. The transducer is mounted on the inside wall of the transducer pedestal, as shown in Fig. 1. The excitation voltage ranges from 0 to 17 V d.c. in 1 V increments. The output of the transducer is recorded on one axis of a Hewlett-Packard *X–Y* Recorder Model 7001A. The temperature of the specimen is measured with a chromel–alumel thermocouple; its output is recorded on the other axis of the recorder. The resultant curve is a continuous plot of temperature versus thermal expansion.

2.4. Furnace and controller

The furnace is a multitap ten-zone Marshall furnace with a Nichrome resistance heating element. It is 10 in. diameter by 16 in. long with a 1 in. diameter centre hole in one end for the vitreous silica tube. The other end of the furnace is closed. During testing, the centre of the specimen is located at the centre of the furnace, which has an 8 in. long uniform temperature zone. The furnace temperature is controlled by means of an L & N Controlall by which the heating rate, soak time, and cooling rate can be regulated. The specimen is normally heated and cooled at a rate of about 3° C min^{-1}.

2.5. Cooling chamber and controller

The liquid nitrogen cooling chamber, shown in

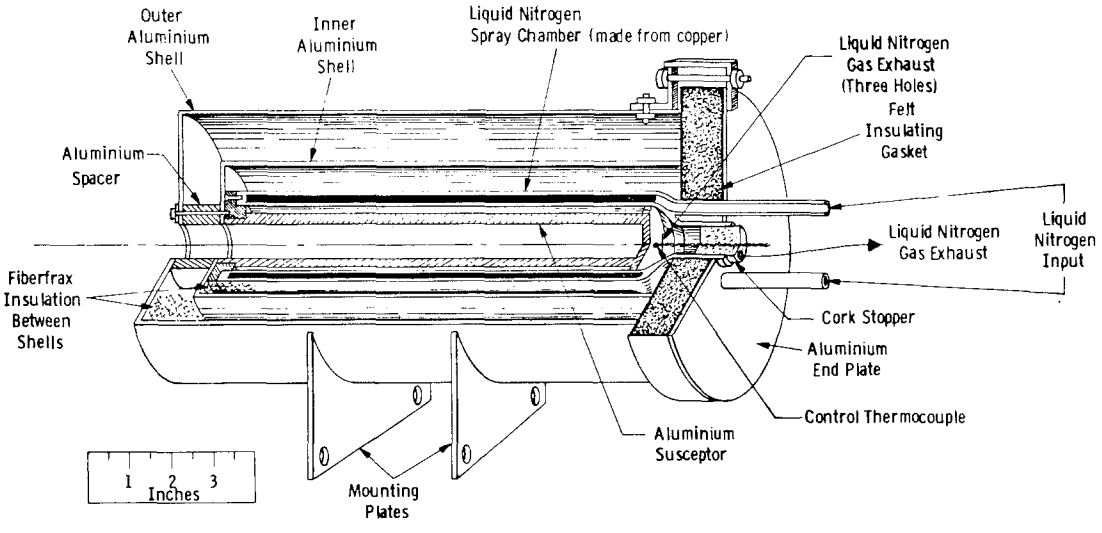


Figure 5 Liquid nitrogen cooling chamber for thermal expansion tests.

Fig. 5 consists of inner and outer cylinders held together concentrically by means of an aluminium spacer. A double-walled spray chamber made from copper is positioned within the inner cylinder, and a cylindrical aluminium susceptor fits inside the spray chamber, immediately surrounding the fused silica tube holding the specimen. The inner wall of the copper spray chamber contains a large number of 0.020 in. diameter holes through which liquid nitrogen is sprayed onto the susceptor. The susceptor, in turn, keeps the specimen and tube and rod assembly at a uniform temperature. Fiberfrax insulation between the chambers keeps heat losses to a minimum.

The temperature of the specimen is programmed using the L & N Controlall described earlier for contolling the furnace. A chromel–alumel control thermocouple is attached to the back end of the susceptor with a screw. The thermocouple enters the chamber through a hole in the cork pressed into the opening in the copper cooling chamber. The programme controller opens and closes a solenoid-operated liquid nitrogen valve attached to the liquid nitrogen tank, which is pressurized to about 15 psi.* As the programme calls for a lower temperature, the solenoid valve is opened allowing liquid nitrogen to flow into the copper chamber; when the control temperature is reached, as determined by the control thermocouple, the valve is closed. The rate of flow of liquid nitrogen through the solenoid valve is controlled by a hand set needle valve. The solenoid valve is a Valcor Engineering Model No. 94P19C6.

*10^3 psi $\equiv$ 6.89 N mm^{-2}.

3. Transducer and system calibration

The Statham transducer used to measure the thermal expansion must be calibrated at different excitation voltages so that the optimum sensitivity of the transducer can be chosen for the material being tested. After the transducer is calibrated, the entire system must be calibrated to take into account any other system errors that would result in inaccurate data. The transducer is calibrated using a micrometer or a Tuckerman gauge and the overall system is calibrated with a platinum standard in accordance with ASTM specifications.

3.1. Transducer calibration

As indicated in Fig. 6, the calibration of the Statham transducer can be made using a Tuckerman optical strain gauge graduated to 4×10^{-6} in., or a micrometer graduated to 0.0001 in. It is generally easier to use the micrometer but the Tuckerman gauge is more sensitive. The special mounting head shown in Fig. 6 is used in conjunction with these gauges.

For the transducer calibration, a compression spring and dummy specimen whose combined length is equal to the standard specimen length is placed in the tube. The spring is compressed by 25% more than the expansion it is desired to calibrate for, using the screw on the end of the

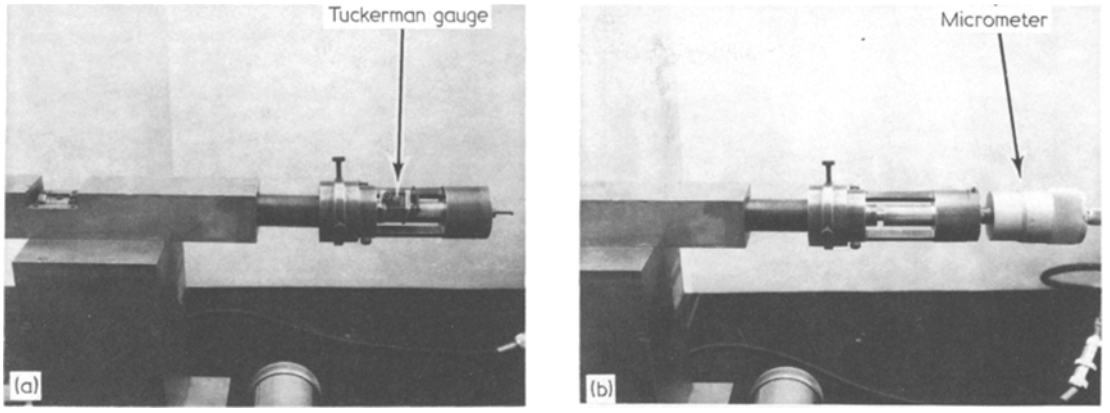


Figure 6 Statham transducer calibration with (a) Tuckerman gauge or (b) micrometer calibration system.

mounting head if the Tuckerman gauge is used or by advancing the micrometer in the direction of compression if the micrometer is used. The screw or micrometer replaces the spring loading screw of the basic dilatometer and pushes on the central plunger of the head which contacts the silica rod and hence the compression spring through the dummy specimen. The Tuckerman gauge must be read with an autocollimator. The micrometer has a graduated scale on its thimble and barrel and can be read directly.

During calibration, the output of the transducer is fed in to the X–Y recorder, with the excitation voltage to the transducer set at a predetermined value. The gauge, Tuckerman or micrometer, is then moved in increments in the direction simulating thermal expansion, and the deflection noted on the X–Y recorder. After the desired displacement range is convered by traversing the recorder in the expansion direction, the gauge movement is reversed, simulating specimen contraction, and incremental readings made. With this procedure the thermal expansion and thermal contraction are simulated at a fixed transducer excitation voltage and X–Y recorder setting. This same procedure is used for two X–Y recorder sensitivities at 1 V excitation voltage increments from 1 to 17 V. Six of the resulting 34 calibration curves are shown in Fig. 7. Using these curves the best thermal expansion range can be selected for any specimen. The Statham transducer has a range of 0.032 in. which is sufficient for most materials. If additional range is necessary, the transducer can be reset during a test.

The slopes of the curves are computed and summarized in Table I for easy daily reference in choosing the optimum thermal expansion range for a specimen. The data in Table I and the calibration curves are based on using millimeter graph paper on the X–Y recorder.

TABLE I Calibration summary for Statham transducer no. 934 used with vitreous silica tube dilatometer. HP autograph X–Y recorder model 7001A using Dietzgen graph paper no. 340D-M (d.c. power supply no. 1). Statham transducer model no. G7A-5.5-660

X–Y recorder 15 mV full scale		X–Y recorder 75 mV full scale	
Transducer excitation (V) d.c.	10^{-6} in. per chart division	Transducer excitation (V) d.c.	10^{-6} in. per chart division
1	97.2	1	–
2	49.2	2	–
3	32.7	3	168.5
4	24.6	4	125.0
5	19.6	5	99.3
6	16.4	6	82.6
7	14.1	7	70.9
8	12.3	8	62.1
9	10.9	9	55.6
10	9.9	10	49.8
11	8.9	11	45.4
12	8.2	12	41.6
13	7.5	13	38.6
14	7.2	14	35.9
15	6.6	15	33.6
16	6.3	16	31.4
17	5.8	17	–

3.2. System calibration

After the Statham transducer is calibrated, the entire system is calibrated from room temperature to 1000° C and room temperature to −195° C using a chemically pure platinum standard whose thermal expansion is known and is documented [3, 4]. Chemically pure platinum is the standard

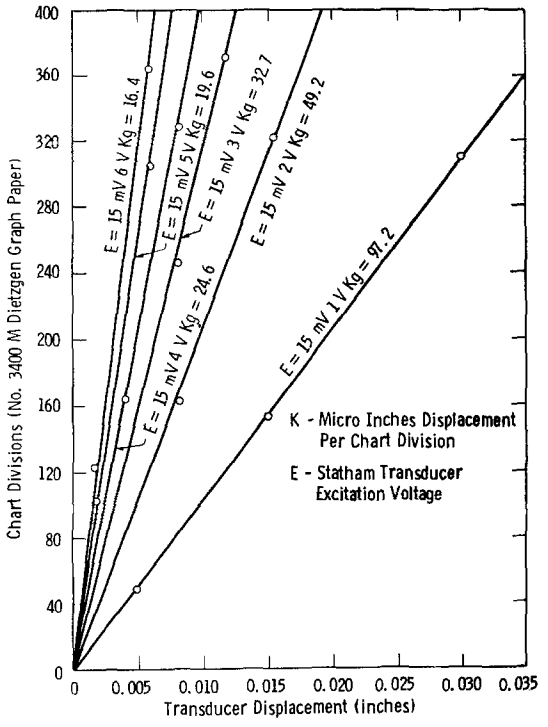


Figure 7 Typical calibration curves for no. 934 Statham transducer to 0.035 in. displacement on HP, $X-Y$ recorder, 15 mV range.

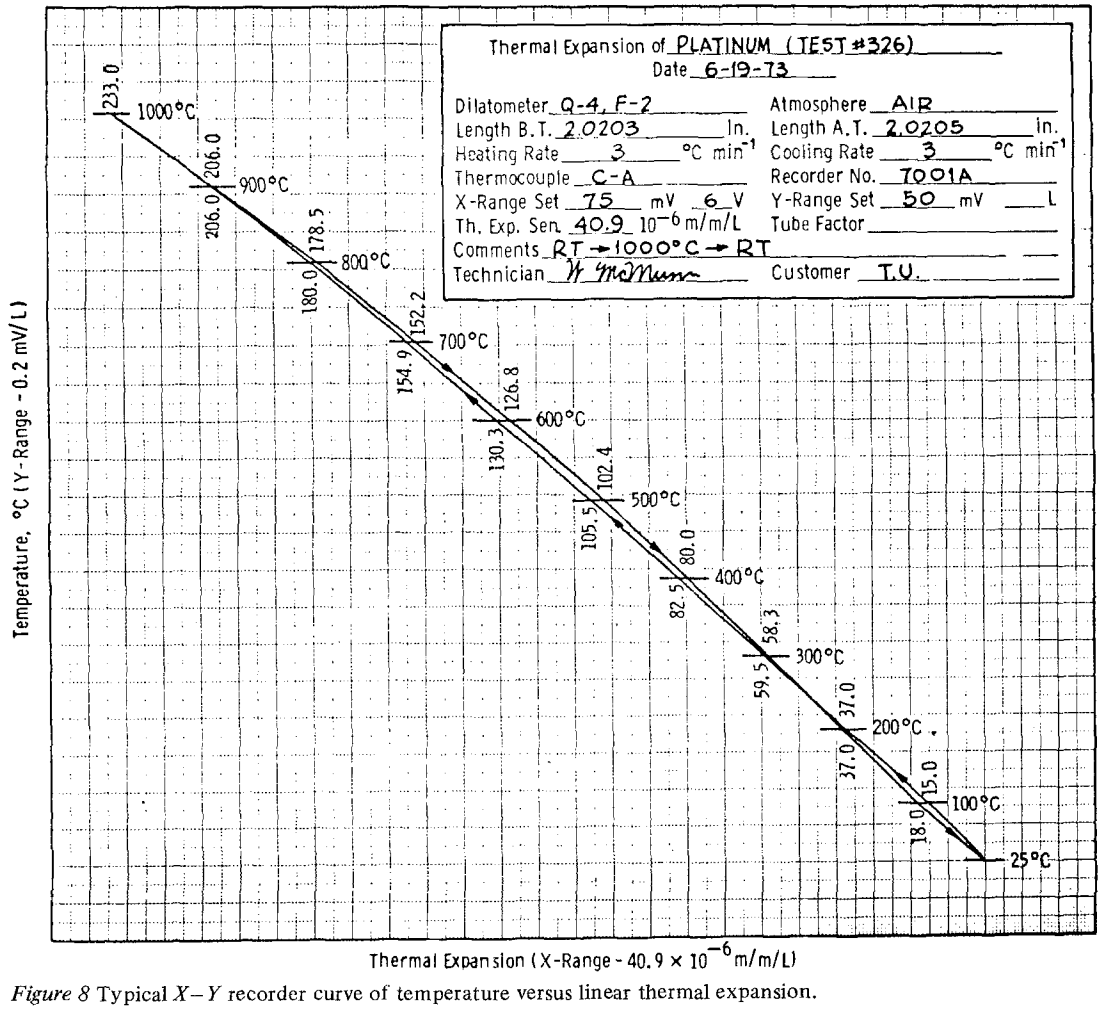


Figure 8 Typical $X-Y$ recorder curve of temperature versus linear thermal expansion.

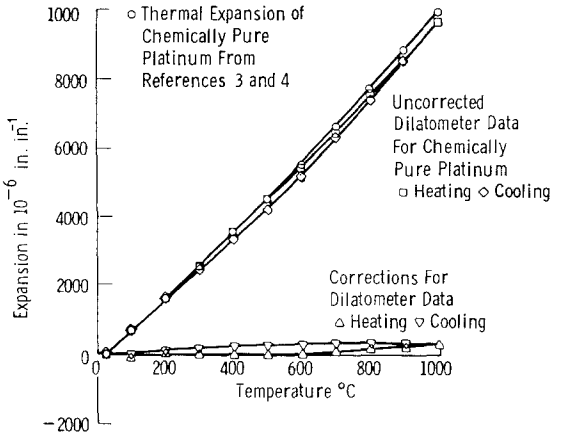


Figure 9 Thermal expansion of chemically pure platinum, uncorrected dilatometer data for chemically pure platinum, and corrections for dilatometer data over 25 to 1000° C temperature range.

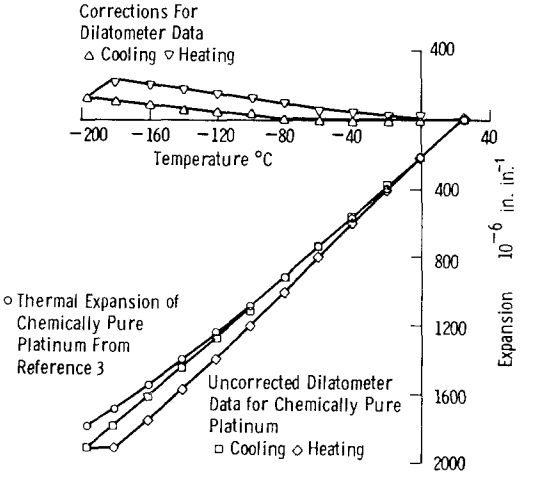


Figure 10 Thermal expansion of chemically pure platinum, uncorrected dilatometer data for chemically pure platinum, and corrections for dilatometer data over −195 to 25° C temperature range.

recommended by ASTM. The specimen is heated and cooled at an average rate of 3° C min^{-1}. Three calibration tests are generally made; the average of the three tests is used to establish the correction curve. A typical test curve is shown in Fig. 8 for the temperature range RT to 1000° C. A typical correction curve, uncorrected dilatomer data, and the expansion of chemically pure platinum are shown in Figs. 9 and 10 for the ranges 25 to 1000° C and 25 to −195° C respectively. The correction curve is obtained by determining the value that must be added to the uncorrected dilatometer data to make it identical to the expansion value of chemically pure platinum. The correction is made at increments of 100° C using room temperature as the starting temperature on both temperature ranges. These values are then plotted as shown in Figs. 9 and 10 and are used in subsequent thermal expansion tests to correct the test data. By this process the correction is made for the expansion of the vitreous silica tube as well as any other system errors that occur during the test. The reproducibility of the system is determined from the three calibration tests. It was found that the correction on heating is different from the correction on cooling in both temperature ranges as shown in Figs. 9 and 10. This is of no consequence as long as the test is repeatable.

4. Testing procedure

After the Statham displacement transducer and the dilatometer are calibrated, specimens can be tested and the data corrected. However, frequent checks of the $X-Y$ recorder must be made because its calibration can change. The $X-Y$ recorder is calibrated on both axes electrically before every test using an L & N No. 8662 precision potentiometer or an equivalent. This is accomplished by feeding millivolt signals from the potentiometer into each axis of the recorder. The $X-Y$ recorder is traversed incrementally over the entire chart in both directions to make certain that the calibration is linear. The expansion axis calibration is checked on either the 15 mV or 75 mV range depending upon the expected expansion and the temperature axis is calibrated on the 50 mV range to obtain the maximum deflection across the recorder for the 25° C (room temperature) to 1000° C temperature range, which represents a change of 40.31 mV. The $X-Y$ recorder is calibrated on the 10 mV range for tests from 25 to −195° C which represents a change of 6.68 mV.

5. Results

The data for two typical tests conducted with the vitreous silica tube dilatometer, given in Figs. 11 and 12 shows that the expansion results of yttrium aluminium garnet and Lucalox, a translucent alumina material, are essentially the same as that reported in the literature for each material. Additional information on these two materials is given in [5] and [6].

6. Conclusions

The dilatometer described here has been used to measure the thermal expansion of solids from −195 to 1000° C in air and flowing argon gas

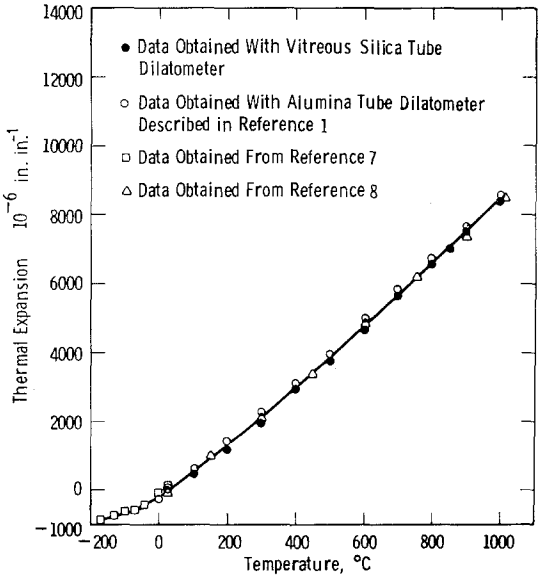


Figure 11 Thermal expansion of yttrium aluminium garnet ($Y_3Al_5O_{12}$).

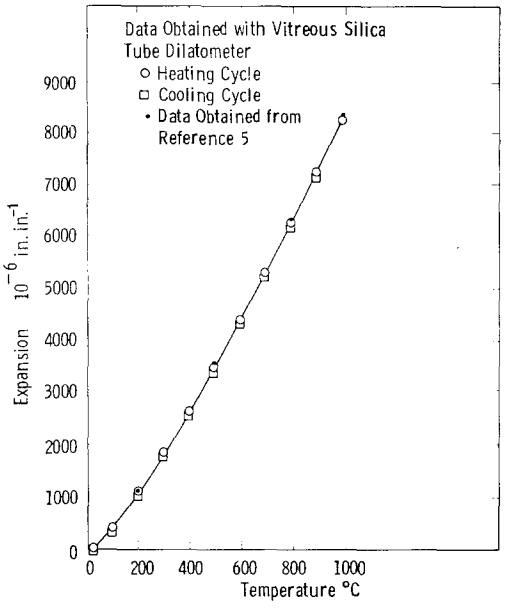


Figure 12 Thermal expansion of Lucalox from 25° C to 1000° C in air.

atmosphere. The measurement is accomplished in two tests: one from 25 to −195° C and one from 25 to 1000° C.

Acknowledgement

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