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APPARATUS FOR DETERMINING LINEAR THERMAL
EXPANSIONS OF MATERIALS IN VACUUM OR
CONTROLLED ATMOSPHERE
S. D. Fulkerson

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METALLURGY DIVISION

APPARATUS FOR DETERMINING LINEAR THERMAL EXPANSIONS OF
MATERIALS IN VACUUM OR CONTROLLED ATMOSPHERE

S. D. Fulkerson

DATE ISSUED

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APPARATUS FOR DETERMINING LINEAR THERMAL EXPANSIONS OF MATERIALS IN VACUUM OR CONTROLLED ATMOSPHERES

ABSTRACT

An apparatus for determining linear thermal expansion data up to 1350°C for materials that cannot be heated in air is described. Expansion is not measured directly, but is derived from the differential expansion between the material being tested and the materials of construction of the dilatometer. The apparatus is calibrated against published data on the linear thermal expansion of both fused silica and polycrystalline tungsten metal. It can be operated either as a high-vacuum or as a controlled-atmosphere apparatus. Recording of data is either fully automatic or manual. The percentage error is believed to be no greater than $\pm 0.1-0.2\%$.

Original data for linear thermal expansion of the following materials are reported: French hot-pressed BeO, hafnium-free ZrO₂, zirconium-free HfO₂, INOR-8 alloy, siliconized silicon carbide, uranium dioxide, four different compositions of Be + BeO, four different determinations of CS 312 graphite, boron nitride, and tungsten metal.

INTRODUCTION

During the early evolution of the Ceramics Laboratory of the Metallurgy Division of the Oak Ridge National Laboratory, a need arose for determining the linear coefficients of thermal expansion of certain materials that could not be heated in air without decomposition. This need resulted in the development of the apparatus described in this report.

Since the publication of the first report¹ in which this apparatus was described, there have been furnace modifications as well as much new and unpublished data that justify a more formal report. Although many similar dilatometers, which have performed quite well within their individual capabilities, are described in the literature, it is believed that the subject apparatus has sufficient unique and desirable features and capabilities to make publication worthwhile.

¹S. D. Fulkerson, Apparatus for Determining Linear Thermal Expansion of Materials in Vacuum or Controlled Atmosphere, ORNL-CF 57-7-123 (July 30, 1957).

The instrument is quite versatile and results are dependable. It has been used successfully to obtain thermal expansion data on a wide variety of ceramic and metallic materials. Data curves are consistently reproduced on the same materials to within ± 0.00002 in./in., indicating good precision. The accuracy is somewhat more difficult to define. The calibration of instruments of this type is based on data obtained from some other instrument; consequently, the accuracy is no better than that of the calibration data used. In the case of this dilatometer, calibration was based on two materials whose thermal expansion characteristics are well known and understood: fused silica and polycrystalline tungsten metal. A reasonable estimate of the accuracy of the instrument is believed to be about 99%. Very close agreement is obtained with the published and accepted data for some of the better known materials.

DESCRIPTION OF APPARATUS

The apparatus can be described best by treating it in its major components: (a) the vacuum system, (b) the furnace and controls, (c) the mensuration and sensing devices, and (d) the recording devices.

(a) Vacuum System

The vacuum system is a very important component. Although this apparatus was designed primarily for samples that cannot be heated in air, it may be used advantageously with any material. More uniform heating of a sample is obtained in vacuum than in a gaseous atmosphere because convection currents are eliminated and the sample is heated by radiation only.

The components of the system are (1) the vacuum pumping system, (2) a Pyrex bell jar (13-in. dia x 24 in. long), (3) a water-cooled brass plate (1 in. thick x 24 in. square), and (4) a closed-end porcelain tube (1-in. ID x 1-1/4-in. OD x 18 in. long).

The vacuum-pumping system consists of a 4-in. oil diffusion pump (DPI), with a conventional oil vapor baffle spool, backed by a Welch DuoSeal mechanical pump. Since this system is made up of standard equipment, it is not described in detail nor illustrated; however, the equipment can be seen in Fig. 1 on the lower deck of the table. With most samples the system will maintain pressures as low as 0.05μ during the heating cycle.

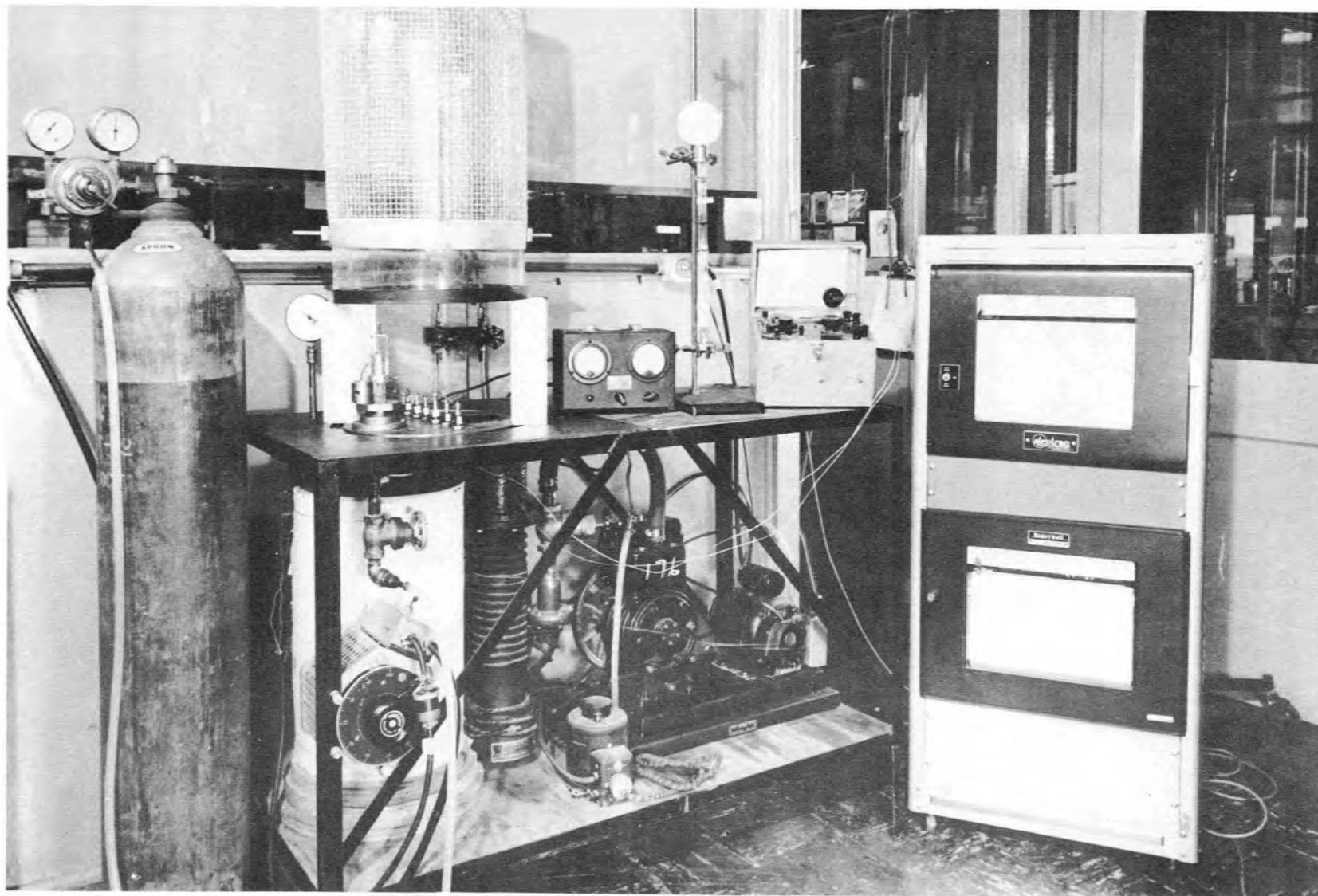


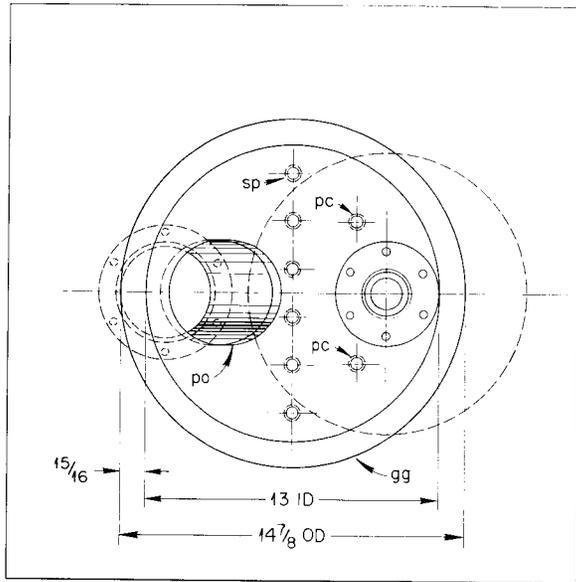
Fig. 1 Assembly of the Apparatus for Determining Linear Thermal Expansion of Materials in Vacuum or Controlled Atmosphere.

The bell jar, as shown in Fig. 1, is guarded by a wire cage and is fitted with a rubber boot gasket, L-shaped in cross section, stretched over the open end. The gasket provides a seal between the bell jar and the brass plate by means of a large groove in the upper face of the plate and is designated gg in Fig. 2. The groove was machined after all the soldering operations were completed to ensure a flat and true surface.

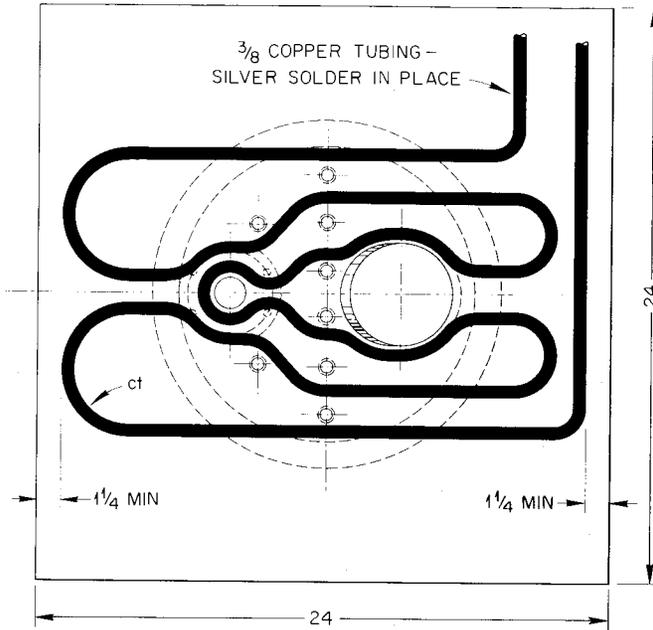
From the standpoint of construction, the brass plate of the vacuum system requires the greatest care because it is the part most vulnerable to leakage. All service connections to the outside pass through this plate. As shown in Fig. 2, there are six drilled and tapped holes for the spark plug connections, sp, to which the electrical leads to the sensing devices are attached. The two drilled and tapped holes, pc, are for 1/4-in. pipe nipples: one serves as a gas-inlet or let-down-to-air connection, while the other is a vacuum gauge connection. The 4-in.-dia pump-out nozzle and flange, po, serve as a suspension for the diffusion pump and baffle spool. The seams of the pump-out nozzle and flange were all hard soldered. A 1/4-in. copper tube, ct, which was symmetrically bent and distributed around the various connections, was hard soldered to the bottom face of the base plate. This affords water cooling to the plate which keeps all metal parts of the dilatometer head at a constant temperature.

The porcelain vacuum tube, vt, passes through the base plate and is held in place at the upper end with a vacuum-tight seal by means of a rubber ring packing gland and nut, pg, built into the base plate. When the apparatus is assembled, this vacuum heating chamber receives the sample end of the dilatometer unit. The 1-in. dia is sufficient to allow approximately 1/32-in. clearance between the walls and the dilatometer heat shields.

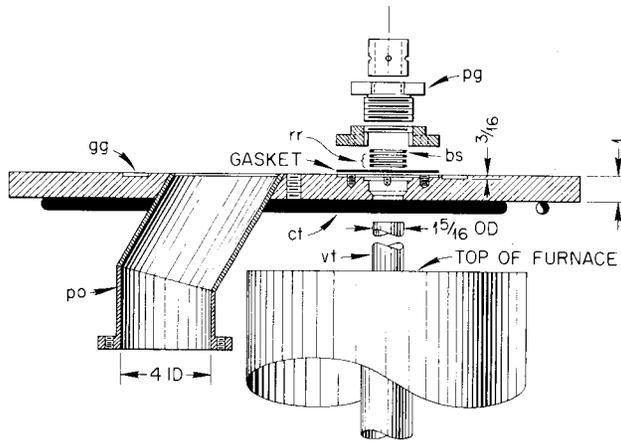
Shown in Fig. 2 (also shown in Fig. 5, p 10) is a thick-walled hollow brass cylinder, with four large holes through the sides, which is set between the instrument head and the gland nut. The purpose of this cylinder is to increase the mean free path for the escape of gas molecules from the vacuum heater tube and ensure a better vacuum around the specimen during the heating cycle. Without this cylinder the aluminum-foil heat shield would act as a lid over the porcelain tube in the furnace and greatly reduce the pumping speed.



BASE PLATE - PLAN



BOTTOM VIEW OF BASE PLATE - COOLING SYSTEM



BASE PLATE - SECTION

LEGEND

- sp = SPARKPLUG HOLES
- pc = 1/4" PIPE CONNECTIONS
- po = PUMP - OUT
- gg = GASKET GROOVE
- ct = COPPER TUBING
- vt = VACUUM TUBE
- pg = PACKING GLAND NUT
- bs = BRASS SLIP RING
- rr = RUBBER PACKING RINGS

ALL DIMENSIONS ARE IN INCHES

Fig. 2. Base Plate Detail.

As shown in Figs. 2 and 3, the porcelain vacuum tube is coaxial with the furnace core and extends well past the center of the heating zone.

(b) Furnace and Controls

The furnace is a platinum resistance wound, vertical tubular type, closed on the bottom end and was custom built by Marshall Products Company, Columbus, Ohio. Using platinum for the winding limits the specimen temperature to a 1350°C maximum. Although temperatures as high as 1400°C have been attained, extended operation at these high temperatures would seriously decrease the service life of the furnace. The furnace winding is equipped with a series of taps used to adjust the zone temperatures by making use of external shunt resistors of various resistance values. Also the furnace is equipped with a built-in compressed-air tubular manifold that is used to control the rate of cooling when the furnace temperature approaches that temperature where natural cooling becomes too slow due to the low-thermal conductivity of the furnace insulation.

This latter feature gives the new furnace a decided advantage over the older model used with this equipment. Quite often it is very desirable to run a cooling curve on a material in order to fix a temperature range over which a transformation or phase change takes place or to make a hysteresis determination.

Power requirement for the furnace is not over 2 kw and is supplied from a 110-v, a-c, 20-amp circuit. The furnace stands vertically on a base beneath the instrument proper, as can be seen in Fig. 1. A diagrammatic section of the furnace is shown in Fig. 3.

Controls are quite simple and, in conjunction with the appropriate mensuration and sensing system described in the next section, runs can be made either manually or automatically.

When making a run by manual control, a variac is used, and the temperature is raised by stepwise increments in power, turned up by hand. If the increments are at all sizable, it is necessary to wait until furnace and sample reach temperature equilibrium before an accurate dilatation reading can be recorded.

When automatic control is desired, a program controller is used and the specimen temperature is raised continuously at a rate not to exceed 3°C/min. Continuous heating is made possible by the use of very small specimens, which can be either 1-in.-long by 1/4-in.-round or 1-in.-long by 1/4-in. by 1/4-in.

LEGEND	
<i>VT</i>	VACUUM TUBE
<i>R</i>	REFRACTORY
<i>I</i>	HEAT INSULATION
<i>FS</i>	FURNACE SHELL
<i>Pt</i>	PLATINUM WINDING
<i>S</i>	SPECIMEN
<i>CA</i>	COOLING AIR TUBES
<i>T</i>	WINDING TAPS
<i>SR</i>	SHUNT RESISTORS

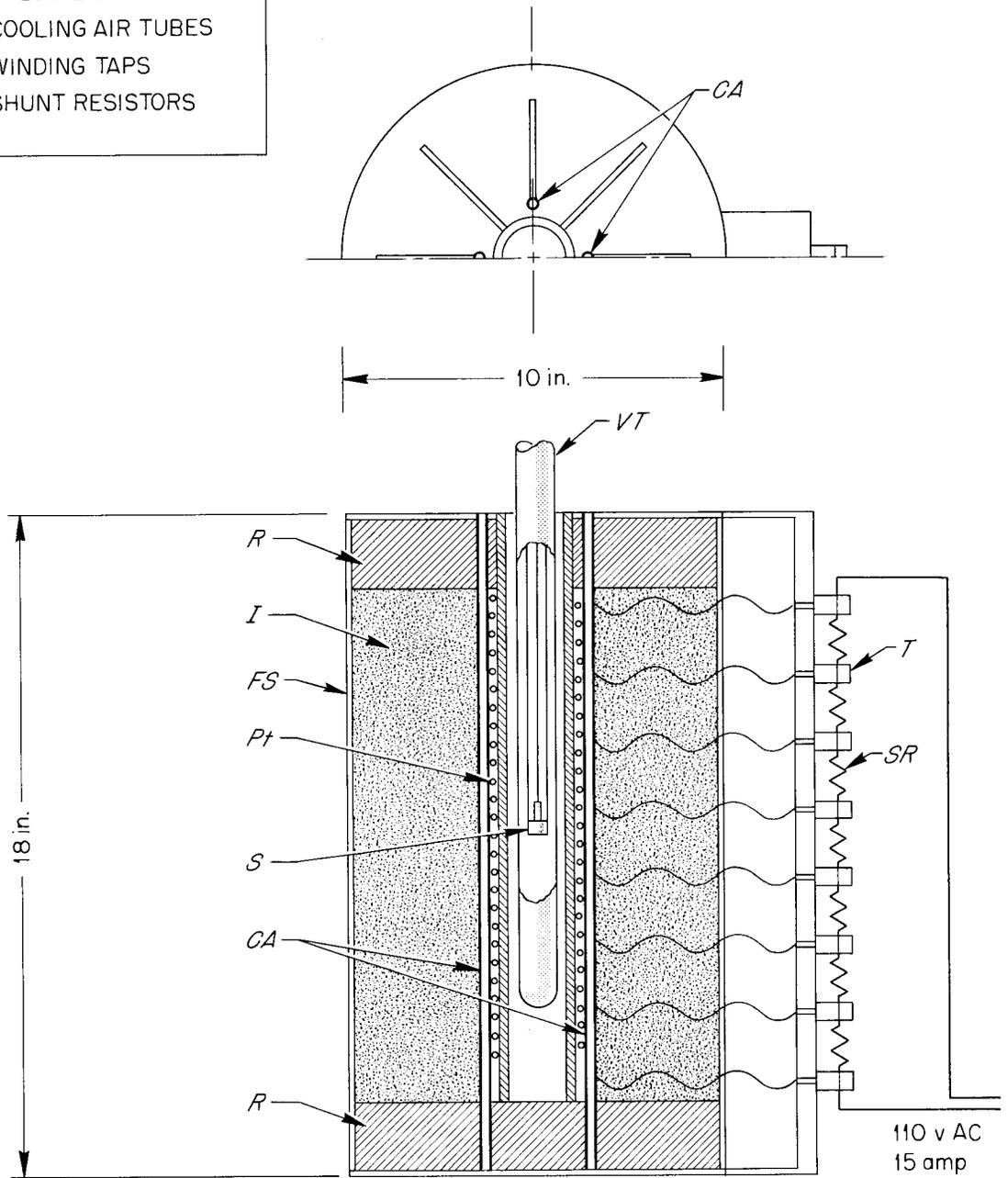


Fig. 3. Cross Section of Furnace.

1/4-in.-square section. It was determined experimentally that no readable temperature difference is observed in any portion of the sample as long as the heating rate does not exceed 3°C/min. Therefore, only one thermocouple is necessary to measure the specimen temperature.

When a program controller is used the apparatus is completely automatic; dilatations are recorded continuously and simultaneously with temperature, and the power is shut off at the end of a run. When a cooling curve is to be determined, at least one more automatic control is desired: cooling rates have to be maintained by hand manipulation of the power input and by the manipulation of a compressed air valve, making it difficult to hold a steady cooling rate. However, this drawback is not too serious as a reliable cooling curve can be obtained even though it is necessary to wait, in some instances, for temperature equilibrium conditions.

(c) Mensuration and Sensing Devices

There are two measuring and sensing units which are used with the apparatus: (1) a manually operated unit and (2) an automatic unit. Both appear to yield precise results and one can be used as a check against the other. Neither differs radically from a host of others now in use. The literature abounds with descriptions of this type of dilatometer.

The basic construction of both instruments is the same, as can be seen in Fig. 4, with one exception: the dial gauge used on the manually operated unit has been replaced on the automatic unit by a differential transformer sensing assembly. A better view of construction details common to both dilatometer units is shown in Fig. 5.

The anvil on which the specimen stands was fabricated from No. 38-900 fused alumina powder compacted isostatically, soft fired, machined, and then fired to 1800°C. Next, the piece was very meticulously surface ground on the upper side in order to provide a flat, smooth surface, perpendicular to the stay-rod holes which go all the way through the anvil. A cement consisting of a mixture of fine-grained fused alumina with 30% fine silica was used to rigidly secure the anvil to the lower ends of the stay rods. This mixture sinters well at a temperature of about 1500°C.

The three stay rods and the push rod are single-crystal synthetic-sapphire rods all centerless ground to a diameter of 0.121 in. and perfectly straight.

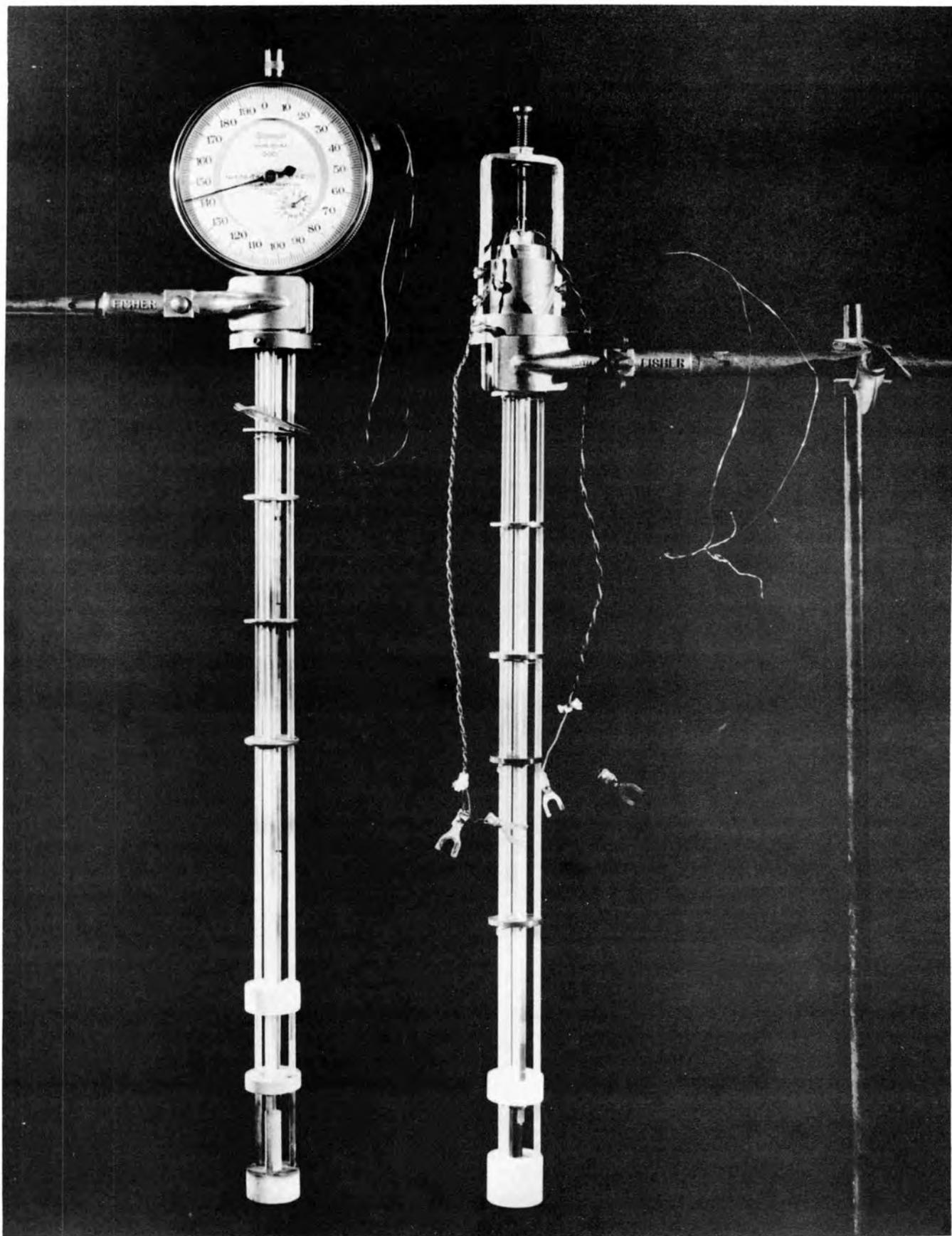


Fig. 4 Dilatometer Units - Manual and Automatic.

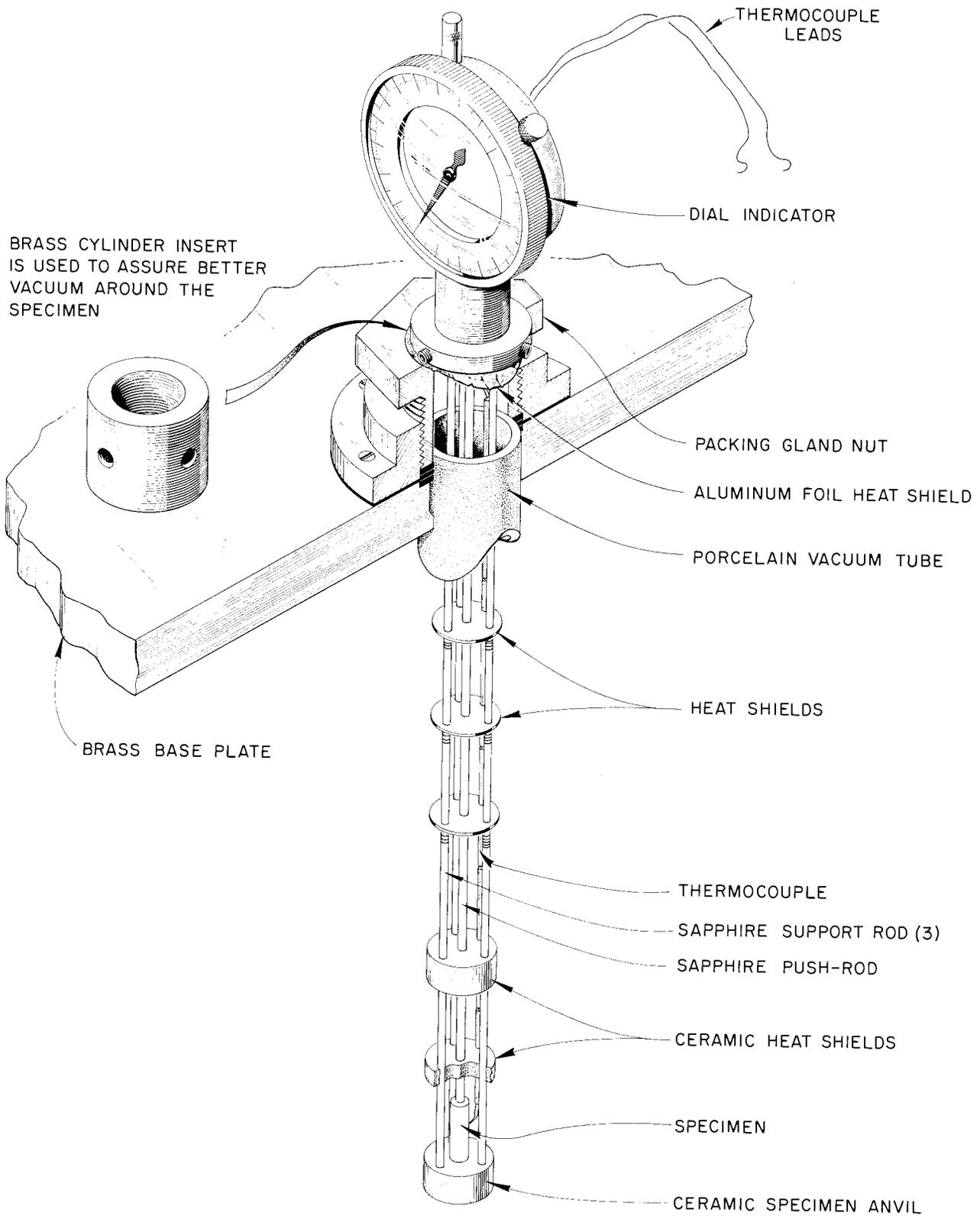


Fig. 5. Dilatometer — Showing Section Through Base Plate. Ref. R.E. Clausing, Thermal Expansion Studies on High-Temperature Brazing Alloys (to be Published)

The stay rods were cut 15-1/2 in. long while the push rod is one inch shorter. In the manufacture of these rods, the angle at which the c-axis of the crystal is oriented with the longitudinal axis of the rod may vary slightly from rod to rod. It is well to use matched sets if they can be obtained. However, this is not essential, because the error caused by unequal linear expansion of the rods is mostly eliminated in the calibration of the instrument.

Ceramic sample holders and the thick ceramic heat shields shown in Figs. 4 and 5 were fabricated in the same manner as the anvils. As seen in Fig. 4, the manual (dial gauge) unit has two ceramic heat shields while the automatic unit has only one. This difference makes the former operable to a higher temperature because it has no metal (Inconel) heat shields in the hot zone.

All holes in the Inconel and ceramic heat shields and the sample holders were drilled on the same pattern. The three outer holes that accommodate the stay rods are 120 deg apart and the hole for the thermocouple was drilled midway between two of these stay-rod holes. The heat shields and the sample holder are free enough to be slid easily up or down on the stay rods and the thermocouple insulator tubing. In operation, they rest on a turn of small platinum wire wrapped around the stay rods. A hole was drilled in the very center of each of the shields, large enough to permit free vertical movement of the sapphire push rod, yet small enough to prohibit any lateral movement. The heat shields serve a second important purpose: they act as guides and stays for all the rods and give rigidity to the long and otherwise flimsy assembly.

The upper ends of the stay rods are secured in place by means of rubber-tipped set screws through the flange of the gauge holder.

The dial gauge used is a Starrett No. 656-T4, range 0-200 divisions of 0.0001 in. each, with a total push-rod travel of 1/2 in. It is fixed in place by set screws through the hub of the brass gauge holder fitting that is covered by the ring stand clamp in the picture. It can be read directly to the fourth decimal place and estimated in the fifth. There is a certain amount of mechanical slack and sticking in the linkage of a gauge of this type, which makes data unreliable on the start of the heating cycle while it is getting set in its bearings. The same is true at the start of the cooling cycle when the compressive stresses, due to spring loading, begin to relieve themselves. In practice, these problems are, to some extent, alleviated by slapping the base plate with the palm of the hand prior to taking a reading.

The brass gauge holder fitting has a small center hole through the flange that guides the upper end of the push rod and exactly centers it on the ball end of the gauge push rod. The push rod is loaded by the spring in the gauge. The gauge holder also serves as a suspension base for the dilatometer to rest upon after it is lowered into the furnace. The unit rests flatly and squarely on top of the heavy brass cylinder, which in turn rests flatly and squarely on the water-cooled packing gland nut. Thermal contact to the water-cooled base plate is good and the metal part of the instrument heads do not vary any more in temperature than does the tap water. Further refinement in precision could be made here by circulating water from a constant temperature reservoir.

The Platinum-10% Rhodium thermocouple for measuring specimen temperature is preferably placed in a small hole drilled to the center of the specimen and located midway of the length. However, for a small specimen, 1 in. x 1/4 in. x 1/4 in., this placement of the couple makes little or no difference from a placement directly on the outer surface of the specimen, providing the heating or cooling rate is not greater than 3°C/min. Of course, the rate at which a sample can be uniformly heated is also a function of its thermal conductivity. The thermocouple signals leave the vacuum system by way of spark plug connections through the water-cooled base plate.

When automation is desired, the unit with the automatic sensing head shown on the right of Fig. 4 is used. It consists of a small differential transformer with a linear response over a range of ± 0.008 -in. armature travel, slip fitted into a metal sleeve, which in turn is mounted in the heavy flanged adapter fitting. The sleeve fits loosely enough in its mooring so that push-rod alignment is brought about by laterally shifting the sleeve, which is finally fixed in the correct position by the three adjusting set screws through the hub of the adapter.

The upper end of the sapphire push rod is fitted with a metal extension. The joint is a long, close, thimble fit cemented in place. The upper end of the extension is machined to a diameter that will just allow the tubular armature to slip over it without straining. The armature is then secured firmly with plastic cement in the correct position.

In construction of the sensing head, care must be taken to ensure that none of the parts are made of a magnetic material. In this particular case, all metal parts are brass.

As with the dial gauge unit, the flange of the heavy adapter fitting serves the dual purpose of securing the upper ends of the sapphire stay rods and provides a suspension base for the dilatometer unit when the apparatus is assembled. Again, all metal parts of this sensing unit are kept at a constant temperature through good thermal contact with the water-cooled base plate.

(d) Recording Devices

The principles involved in both the sensing and recording devices are basically those of a differential transformer and a Wheatstone bridge. The primary of the differential transformer is energized by a 3-v, a-c current. The secondary winding is essentially two coils in series, each having the same number of turns, but wound in opposite directions. When the armature of the transformer is in exact dead null position, the emf induced in each of the secondary windings is the same but opposite in sign, resulting in no flow of current in the secondary circuit. If, for any reason, the armature is forced past null position in either direction, the emf in the winding closest to the armature becomes the greater of the two. Then there is a flow of current in an amount directly proportional to the amount of displacement.

This linear relation between displacement and current makes possible the recording of changes in length of the test specimen. The recorder used for this purpose is an "Atcotran," made by Automatic Temperature Control Company, Incorporated. Any vertical movement of the push rod, no matter how slight, is sensed by the transducer and the resulting induced emf in the secondary is amplified and becomes a voltage supply for one arm of a bridge circuit. The voltage supply for each of the other three arms of the bridge comes from three larger transducers, which are built into a servo system within the strip chart recorder. The armature of one of these three is attached to a manually operated zeroing screw which remains fixed during operation. The other two armatures are mounted on a cam-operated lever, and are shifted automatically by the servo system to bring about a bridge balance when, and if, the bridge is thrown out of balance by the expanding specimen. The recording pen is driven across the

chart through a series of gears and linkages operated by this automatic armature shifting mechanism. An interior view of the recorder is shown in Fig. 6.

A conventional strip-chart temperature recorder, which receives its signal from the specimen thermocouple, is mounted in the same cabinet with the differential expansion recorder. The two signals, temperature and change in length, are recorded independently of each other; however, an automatic or manual check-run switch serves both recordings. This switch is a timing mechanism which simultaneously puts a check mark on each recording at fifteen-minute intervals for the purpose of correlating dilatations with corresponding temperatures. The switching mechanism can be seen in the upper left-hand corner of the instrument case, Fig. 6.

CALIBRATION - THEORY AND PRACTICE

Dilatometers of this type do not read or sense the actual dilatation of the test specimen, but rather a differential expansion between one inch of sapphire and one inch of specimen. Theoretically, if one inch of single-crystal synthetic sapphire were used as the test specimen, the observed differential expansion should be zero over the entire temperature range. In practice, this is never the case because of various factors inherent to this type of unit. Some of the causes for the discrepancies are the difference of expansion of the alumina anvil and the sapphire stay rods, the stretch in the stay rods and compaction of the push rod due to the spring loading of the push rod, and distortion in the stay rods caused by expansion of the metal heat shields.

Before this type of apparatus can be used to determine coefficients of linear thermal expansion with any degree of accuracy, it must first be calibrated against one or more materials whose expansion characteristics are known and accepted. Probably the one material which adapts itself best to this purpose is fused silica, and the calibration curve for this particular apparatus, along with a host of others similar to it, is based on this well-understood material.

Fused silica, "quartz glass," by definition, can have no preferred orientation of molecules and, therefore, possesses the property of expanding equally in all directions when heated. Its expansion is quite low and closely approximates linearity with temperatures from room up to as high as 1000°C. Very few other materials have this degree of linearity over this range.

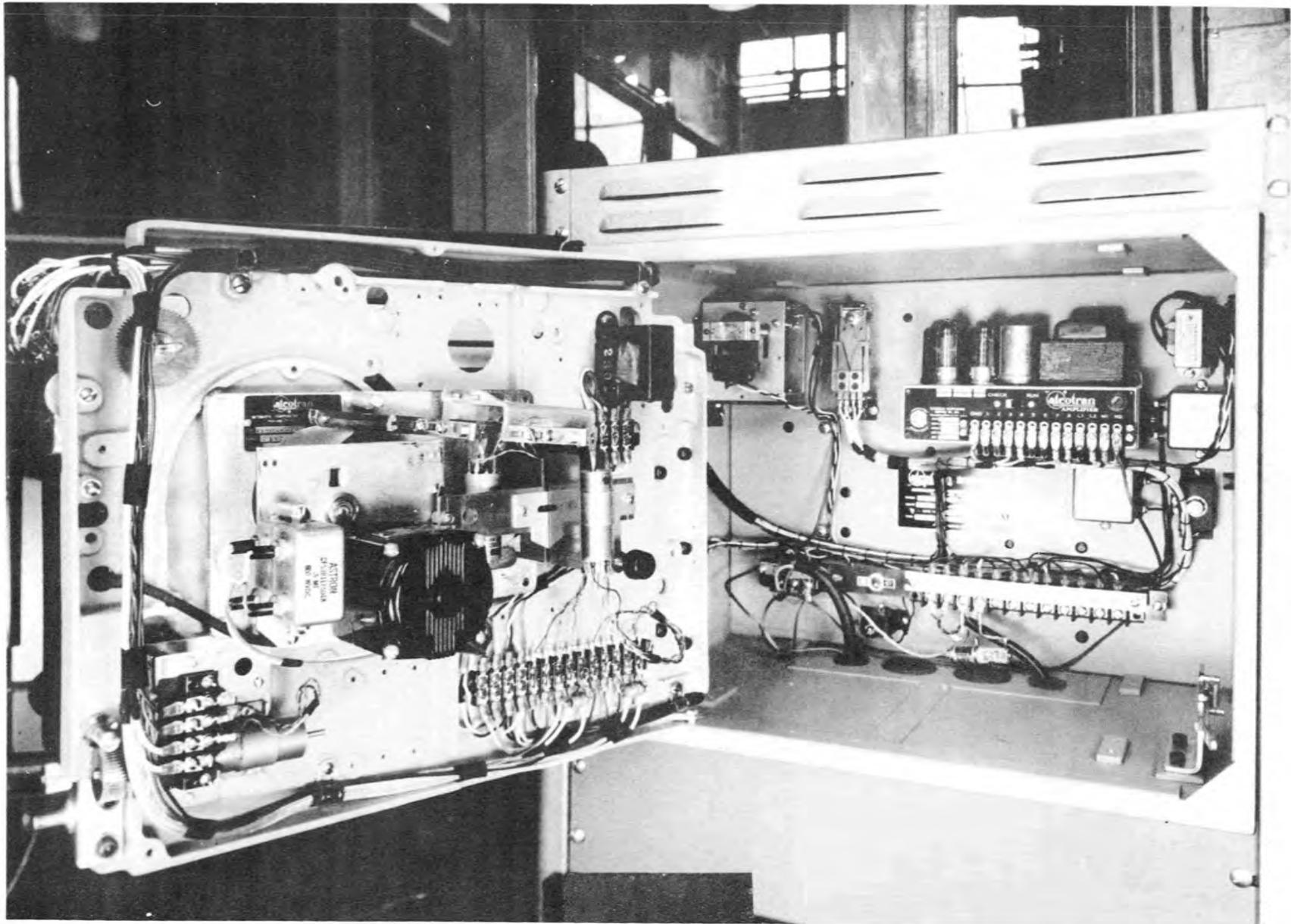


Fig. 6 Interior View of "Atcotran" Recording Unit.

From the literature,² there are sufficient accurate data to plot a reliable thermal expansion curve for fused silica and it is on such a curve that the calibration of this apparatus has been developed. Using a 1-in. x 1/4-in single-crystal synthetic-sapphire rod specimen, the final calibration curve has been checked with good agreement up to 1350°C. Such a specimen, however, cannot be used reliably for a calibration base. Allowances must be made for the inclination of the c-axis to the rod axis which can vary from rod to rod due to either the machining or the manufacture of the specific piece used.

Since expansivity determinations were to be made for some of the more refractory materials at temperatures up to 1350°C, the upper-temperature limit of the furnace, a rod of polycrystalline tungsten was used as a second standard for calibrating the instrument. Polycrystalline tungsten was chosen for its high melting point and known expansion characteristics,³ and because it does not require an extrapolation of the calibration curve to the temperatures involved. It has neither the limitation of single-crystal sapphire discussed above nor that of fused silica which softens at approximately 1000°C.

The method for deriving the instrument calibration curve is shown in Fig. 7 and is described as follows: making use of average data from the literature, a curve of the linear thermal expansion of fused silica was plotted over the range from room temperature to 1000°C; average experimental data were taken from several runs using a strain-free specimen of fused silica rod carefully ground to the finished dimensions, 1 in. x 1/4 in.; a second curve, which lies below the first curve, was obtained by plotting these experimental data on the same sheet of linear graph paper with the literature data. The total difference of expansion between the push and stay rods is theoretically that for one inch of sapphire. It is quite obvious that this difference registers on the sensing devices as a negative quantity. At the same time, the expansion of the fused-silica test specimen will register as a positive quantity. Therefore, the

²W. Souder and P. Hidnert, "Measurements on the Thermal Expansion of Fused Silica," Sci. Papers, NBS No. 524 (1926).

³Values for α , the coefficient of linear thermal expansion up to 1800°C, were supplied by the U. S. Bureau of Standards in personal communications.

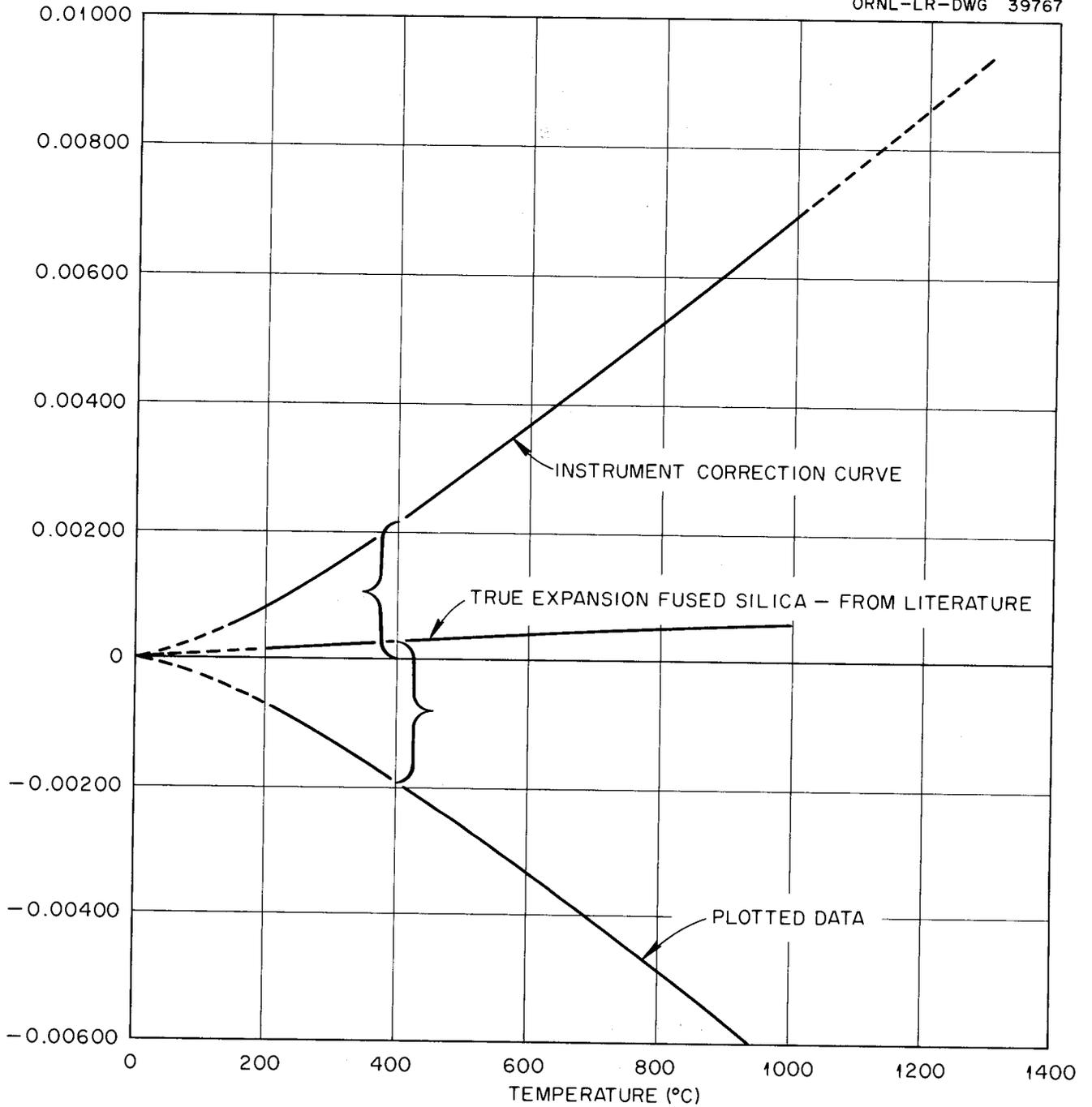


Fig. 7. Method for Deriving the Instrument Calibration Curve.

algebraic difference of the two curves is the correction curve due to the net expansion within the instrument. This difference includes all the inherent built-in errors mentioned earlier in this report.

Since the slope of the curves, expressed in in./in./°C, is the coefficient of thermal expansion, the property being measured, the curves can be moved up or down the ordinate scale until their extrapolated zeros coincide, without changing any values in slope. This shifting of the two known curves makes plotting of the unknown, whether it be the calibration curve for the instrument or the true expansion curve of an unknown material, quite simple. It is accomplished easily and quickly by using a pair of draftsman's dividers and stepping off the correct sum of the two known ordinates. Once a good calibration curve is established, it is used in future determinations of unknown thermal expansions.

In reporting the coefficient of linear thermal expansion of a material, it cannot be assumed that α is constant for any temperature range. If the expansion of a material happens to be perfectly linear with temperature, then α would represent the true coefficient over the entire temperature range. No known material has such a linear expansion. By using the observed data and integrating small increments between limits, an equation for that portion of the curve can be developed. If expressed in the form $l = f(t)$, then $(\frac{dl}{dt} = \tan \phi = \alpha)$ is the instantaneous rate of change in l at a specific temperature. Coefficients reported in this way would be entirely correct, but for all practical purposes, average values of α for short temperature ranges can more conveniently be read from the curve and are quite satisfactory. It is the opinion of the writer that data of this type are most conveniently reported in the graphic form.

The calibration of the "Atcotran" recorder was accomplished by making use of suitable combinations of optical flat gauge blocks, wrung together in stacks to give stepwise increments in thicknesses of 0.001 in. These blocks were placed flat on the specimen anvil of the dilatometer and the push rod was brought to bear on the upper surface of the stack. To make the recorder register the correct thickness, a high-resistance helipot (variable resistance) was placed across the secondary leads from the transducer of the dilatometer, and adjusted accordingly. This procedure was carried out for both plus and minus limits of the transducer. After the shunt resistance was accurately set

by the helipot, this resistance value was then determined by placing the helipot in a milliohm bridge. A resistance of this value was then made up and permanently placed across the transducer secondary. This resistor can be seen in the back and lower part of the instrument case shown in Fig. 6.

During the calibration, it was interesting to note that air-film thicknesses between the flat-ended push rod and the upper surface of the gauge block stack were recorded on the chart in sufficient magnitude to be read easily by the unaided eye. By wringing the push rod down firmly, the last trace of air film was squeezed out from between these two surfaces and only then did the recorder give a constant reading. This experience brought to light one precaution to be used in loading the instrument for a determination - the push rod must be firmly seated on the end of the test specimen.

ILLUSTRATIVE EXAMPLES OF METHODS

Figures 8, 9, and 10 are illustrative of work that has been done with the apparatus. Figure 8 not only shows the true expansion curve for French hot-pressed beryllium oxide, perpendicular to the direction of pressing, but also illustrates the method of determining this curve which is the algebraic sum of the correction curve and the data curve.

In Fig. 9 a comparison of two materials, hafnium-free zirconium dioxide and zirconium-free hafnium dioxide, is made. As was expected, the well-known inversion of ZrO_2 is clearly shown between 1100 and 1200°C where it changes from the monoclinic to the tetragonal crystal form. Although not shown here, this phase change is reversible and could be shown if data were obtained on a sample during cooling at a slow rate. The data indicate that HfO_2 does not go through a similar change of crystal form over this temperature range. However, high-temperature x-ray data indicated a change of this nature takes place well up the temperature scale, progressing rapidly at 1850°C. (ref 4)

⁴C. E. Curtis, L. M. Doney, and J. R. Johnson, "Some Properties of Hafnium Silicate, Calcium Hafnate, and Hafnium Carbide," J. Amer. Ceram. Soc. 37(10) (1954).

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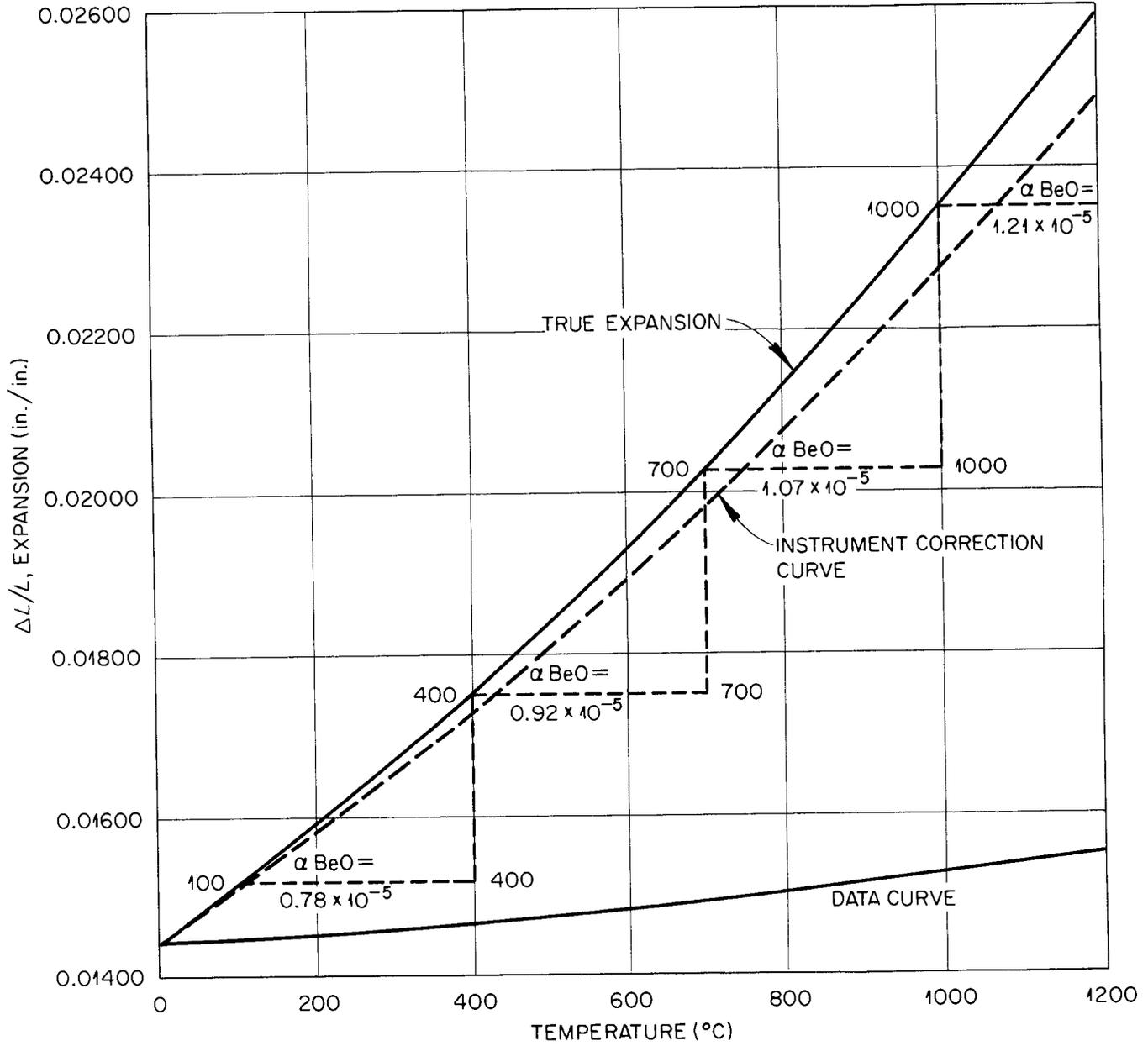


Fig. 8. Thermal Expansion of French Hot Pressed BeO.

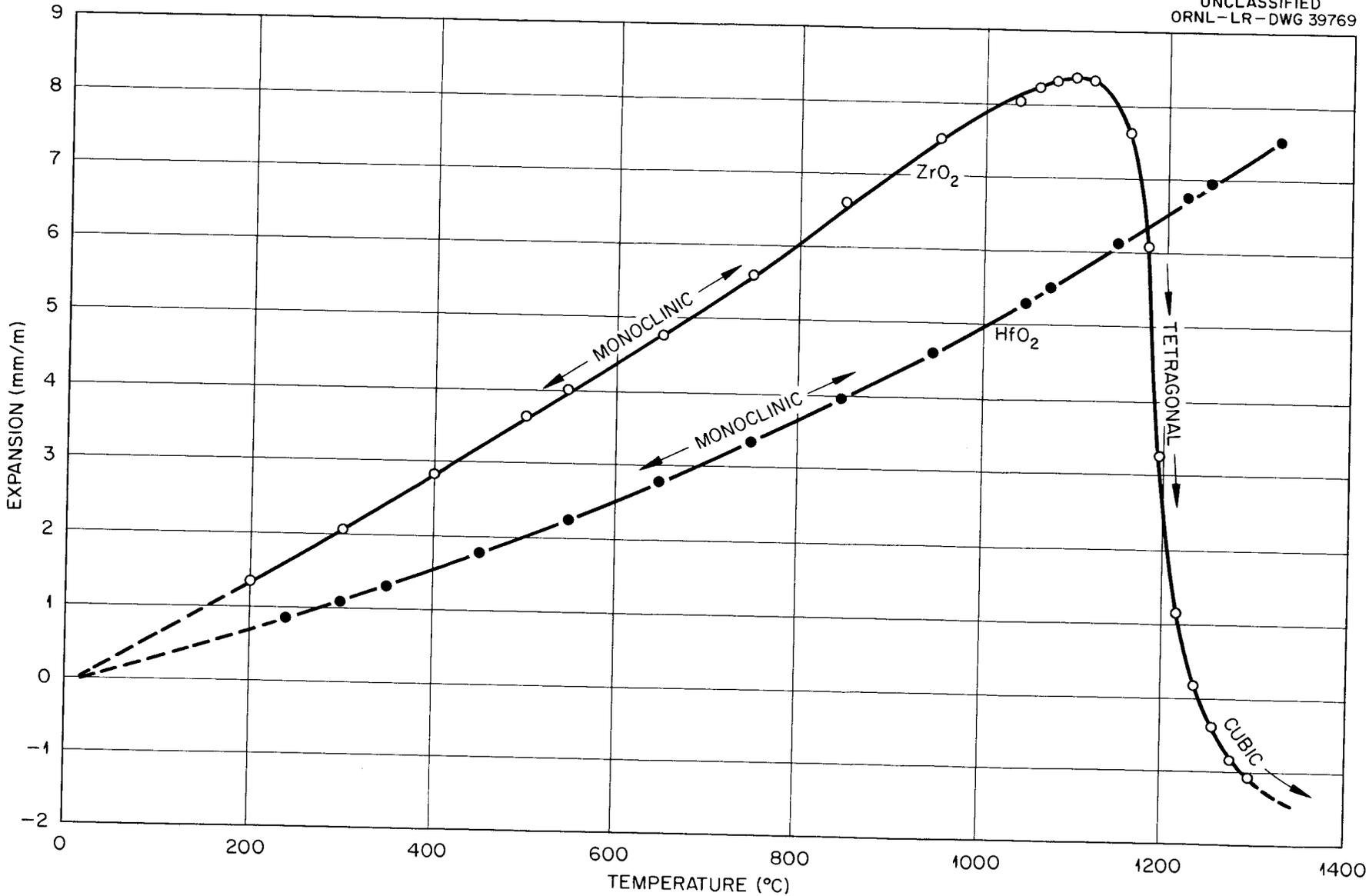


Fig. 9. Comparison of Expansions of ZrO₂ and HfO₂.

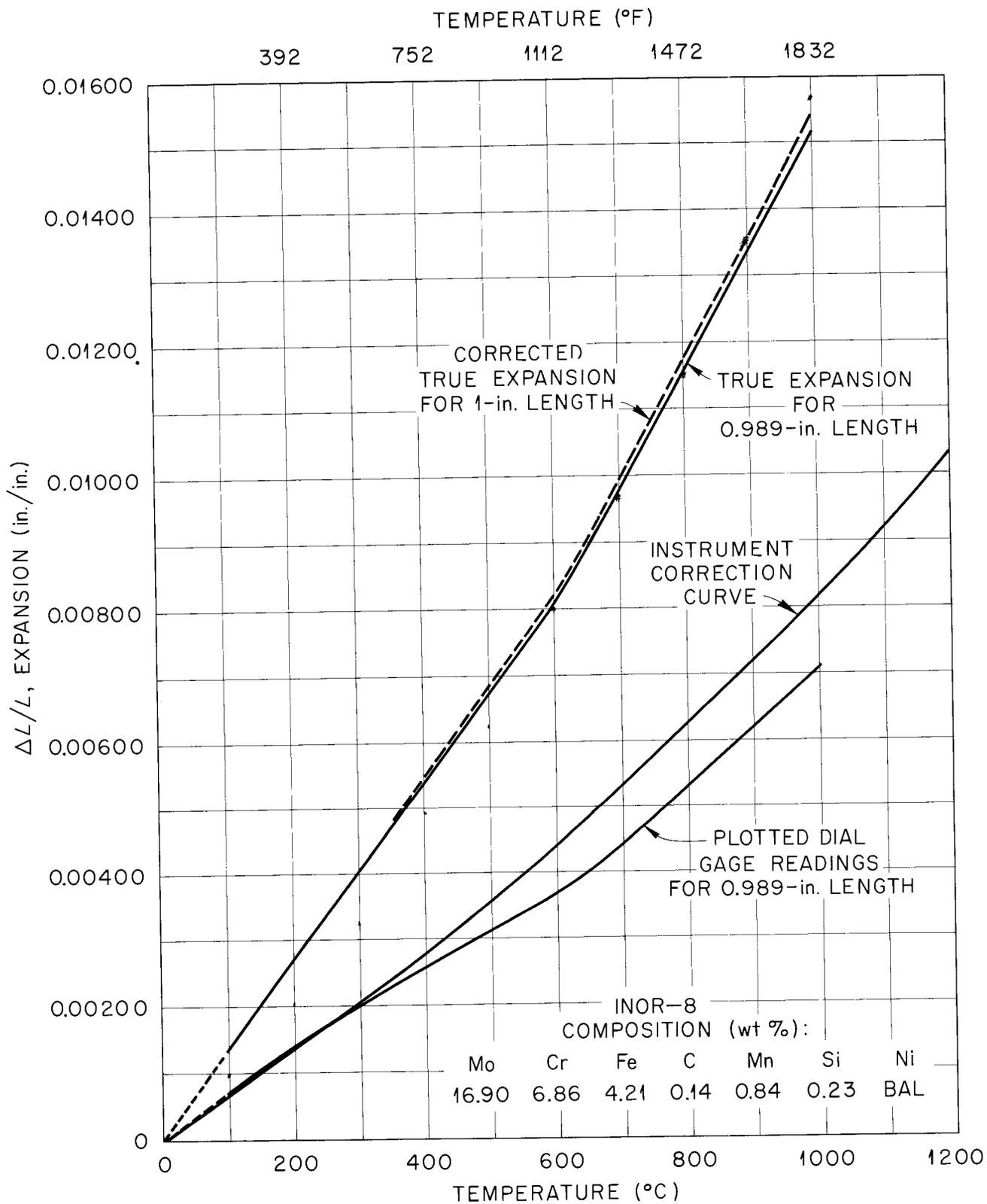


Fig. 10. Linear Thermal Expansion for INOR - 8 Alloy. Sample used was only 0.989 in. Long and Data was Interpolated to a Value for a 1-in. Length.

Figure 10 is illustrative of the method used when the test specimen was slightly shorter than the 1 in. for which the instrument was calibrated. In this particular case the specimen (INOR-8) was 0.989 in. long. The curve, captioned "True Expansion for 0.989-in. Length," is in slight error due to its derivation from the plotted data which were based on a 1-in. specimen, for this length and the correction curve for the instrument. In magnitude, it amounts to the difference of expansions between 0.011 in. of INOR-8 and 0.011 in. of sapphire and is negligibly small. In plotting the corrective curve, expressive of the expansion of a 1-in. specimen, a correction in ΔL at any given temperature was added. This correction was determined from the ratio $\Delta L/0.989$.

ORIGINAL DATA

Although the data shown in Figs. 8, 9, and 10 are used in this report to illustrate methods of operation, they can also be included as original data along with Figs. 11-22. It is believed that these data are sufficiently accurate to be safely used in engineering design work. The calibration curves have been adjusted so that samples of standard materials of fused silica, single-crystal sapphire, and tungsten (Fig. 11) all yield results that are in very close agreement with the already published and accepted data. This agreement in every case has been to within ± 0.1 to 0.2% for these particular materials.

However, for some other materials in which changes can occur in composition and fabrication techniques, such as those materials whose expansions are shown in Figs. 11-22, there can be appreciable variances in α values.

Values of α for four slightly different compositions of Be + BeO, varying only by 1/2 to 1% additions of oxide, are compared in Figs. 12-15.

Values of α for four samples cut from the same log of graphite but at different locations, both perpendicular and parallel to the direction of extrusion, are compared in Figs. 16-19. The total expansion of boron nitride is shown in Fig. 20, the linear thermal expansion of siliconized silicon carbide is given in Fig. 21, and the linear thermal expansion for isostatically pressed UO_2 can be seen in Fig. 22.

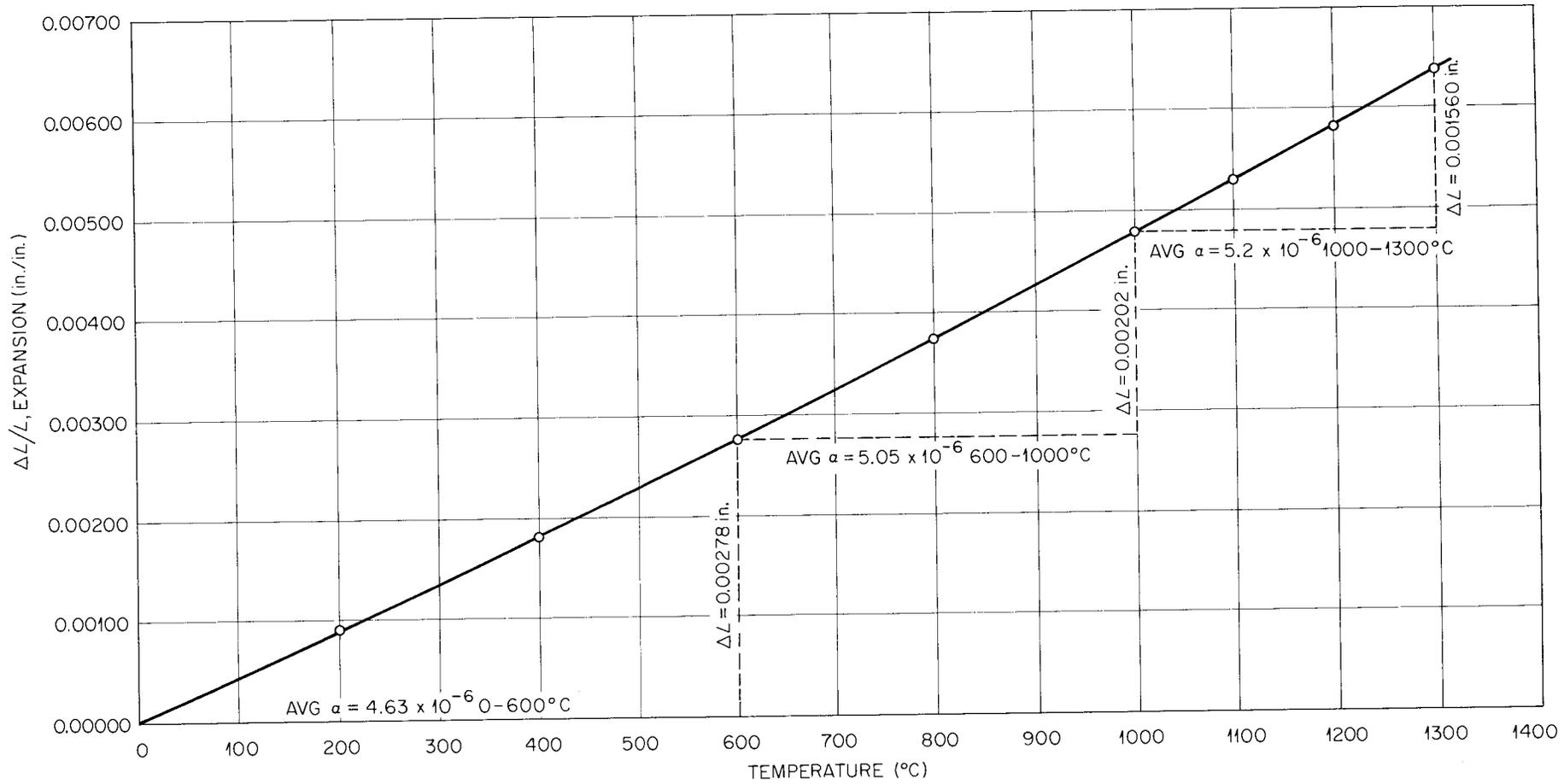


Fig. 11. Linear Thermal Expansion Data for a 1 x 1/4 in. Rod of Tungsten Metal.

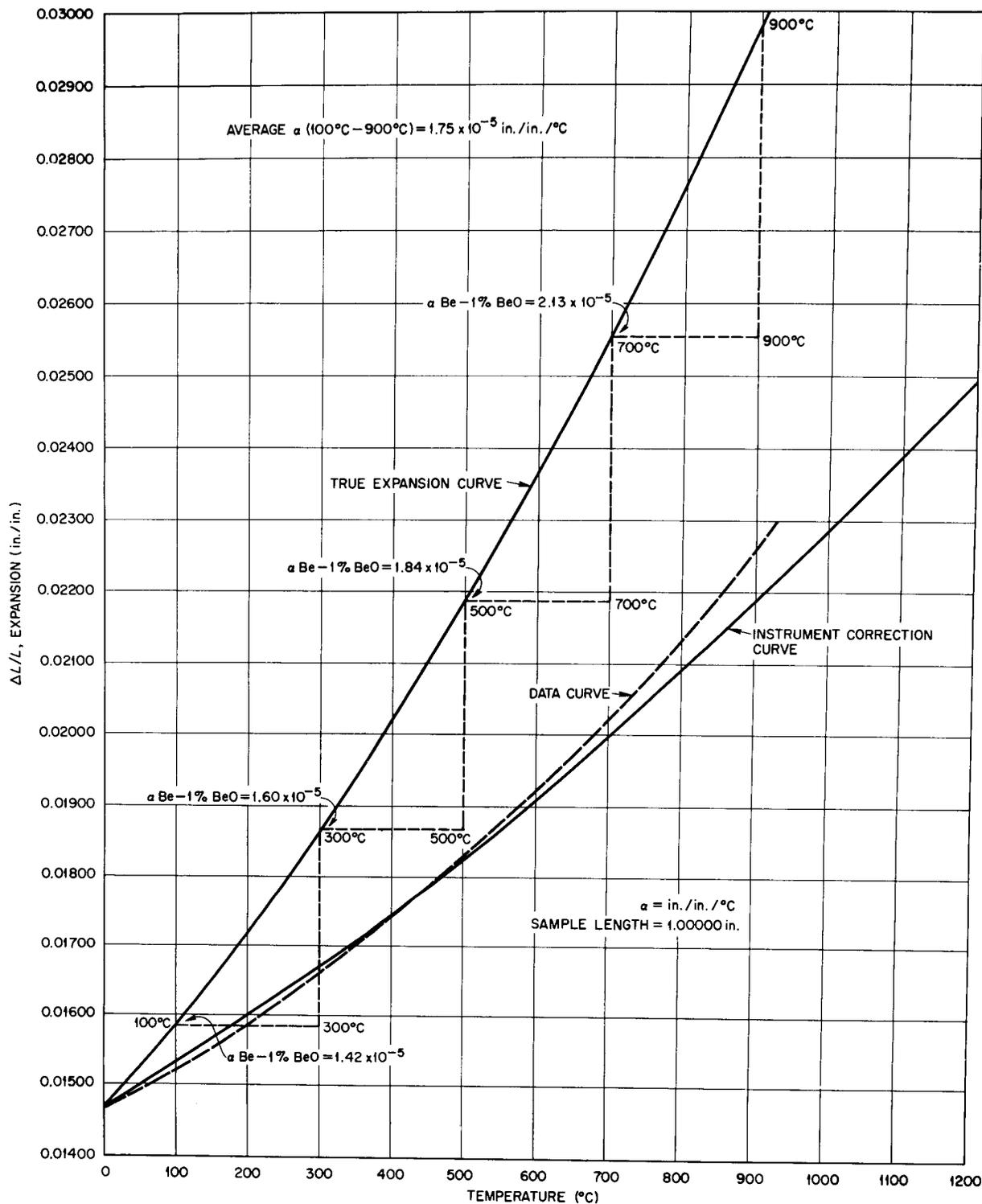


Fig. 12 Thermal Expansion of Be + 1% BeO. Ref. J. E. Barton and S. D. Fulkerson, Linear Thermal Expansion of Four Different Compositions of Be + BeO, ORNL-CF 56-11-59 (Nov. 7, 1956).

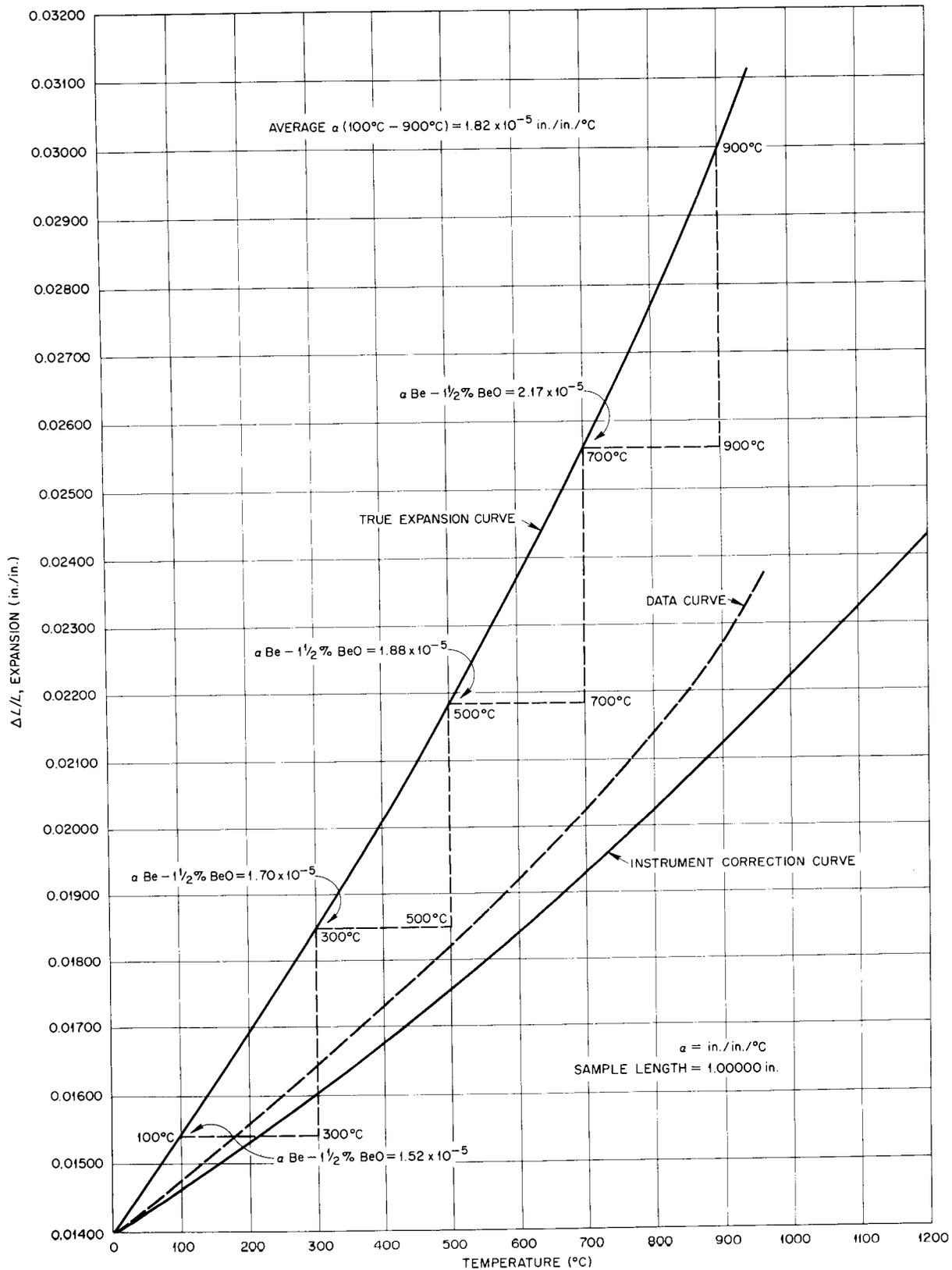


Fig. 13 Thermal Expansion of Be - 1-1/2% BeO. Ref. J. E. Barton and S. D. Fulkerson, Linear Thermal Expansion of Four Different Compositions of Be + BeO, ORNL-CF 56-11-59 (Nov. 7, 1956).

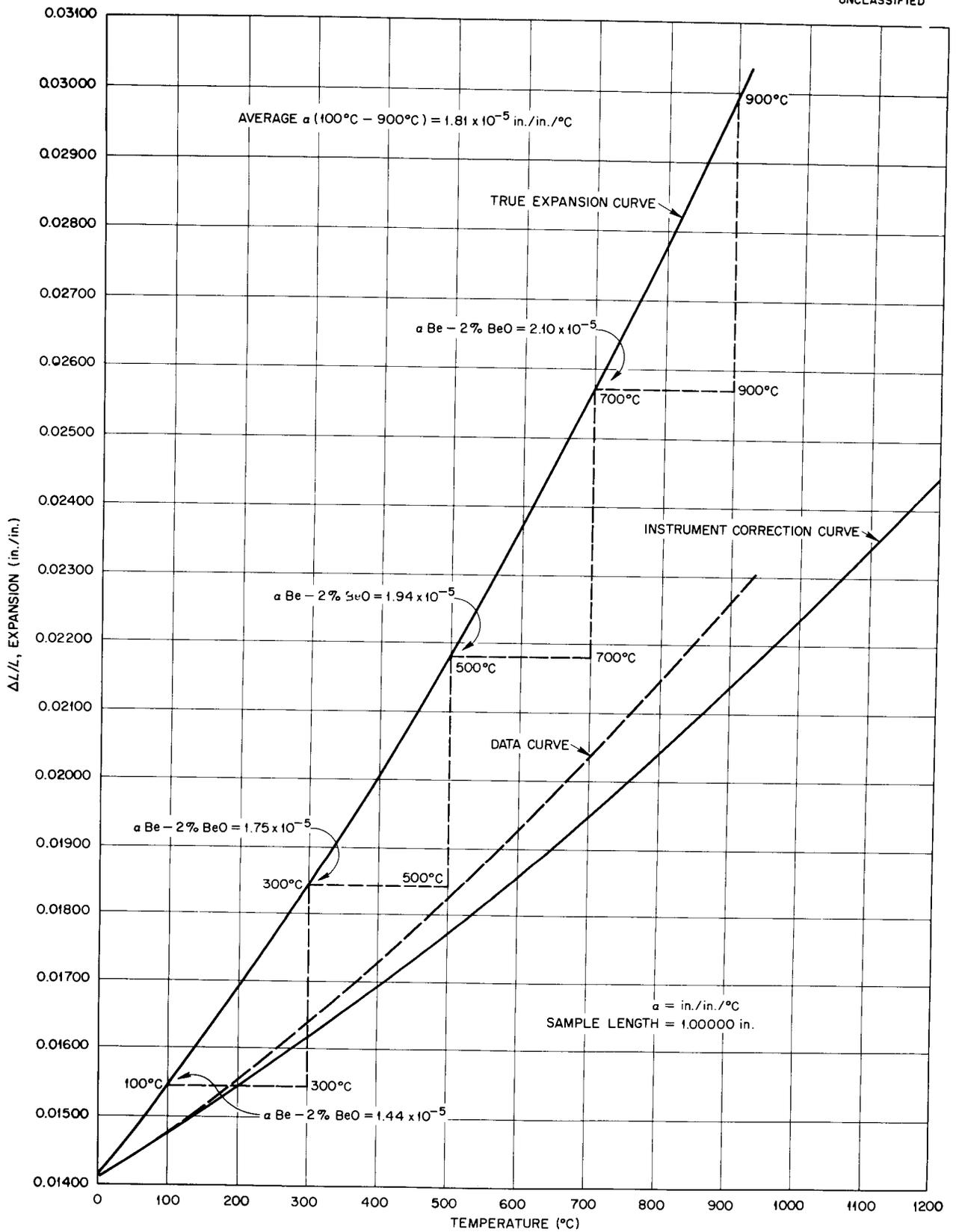


Fig. 14 Thermal Expansion of Be + 2% BeO. Ref. J. E. Barton and S. D. Fulkerson, Linear Thermal Expansion of Four Different Compositions of Be + BeO, ORNL-CF 56-11-59 (Nov. 7, 1956).

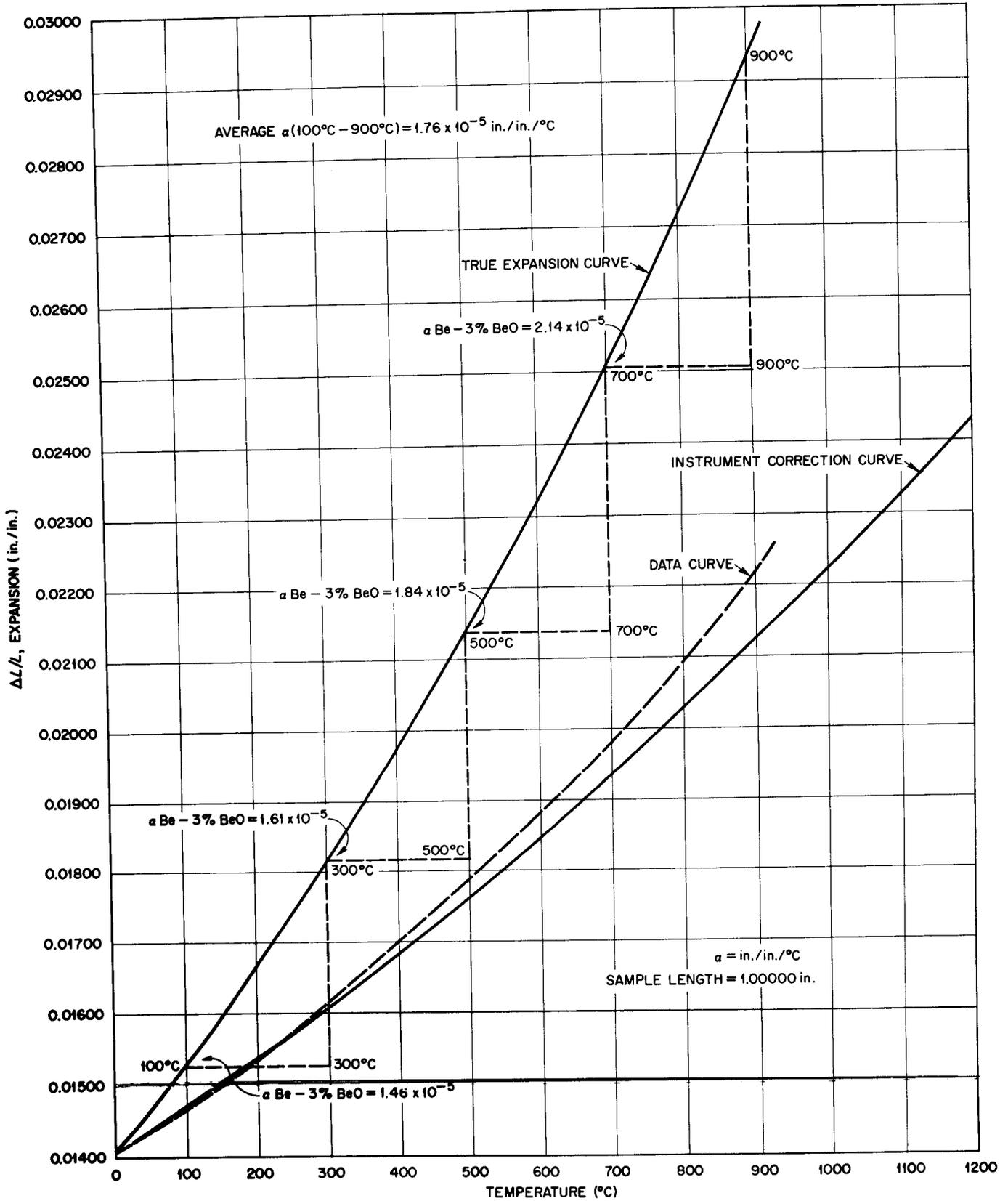


Fig. 15 Thermal Expansion of Be - 3% BeO. Ref. J. E. Barton and S. D. Fulkerson, Linear Thermal Expansion of Four Different Compositions of Be + BeO, ORNL-CF 56-11-59 (Nov. 7, 1956).

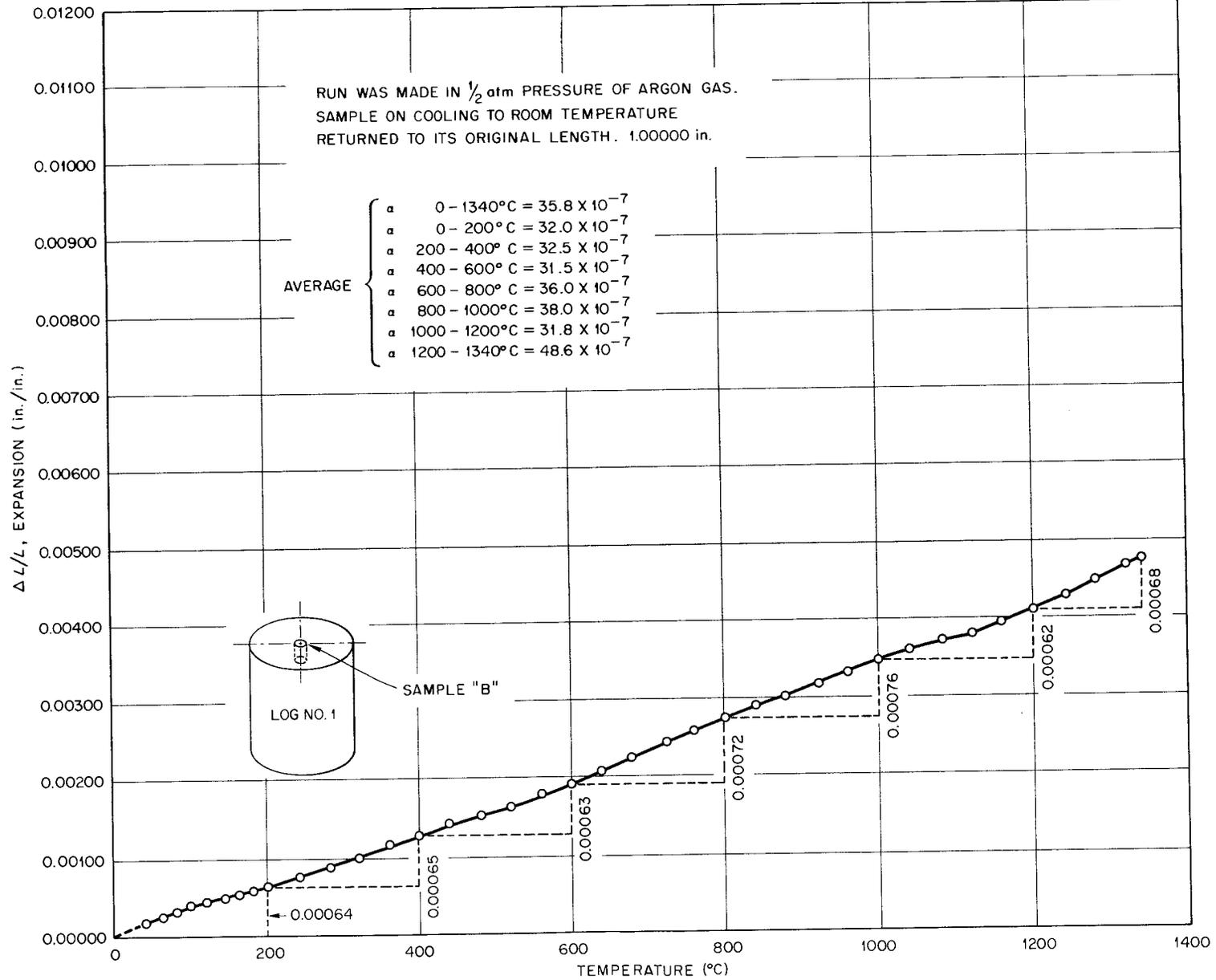


Fig.17. Linear Thermal Expansion of a $1 \times \frac{1}{4}$ in. Round Rod of CS 312 Graphite.
Sample Cut from Center of Log Parallel to Direction of Extrusion.

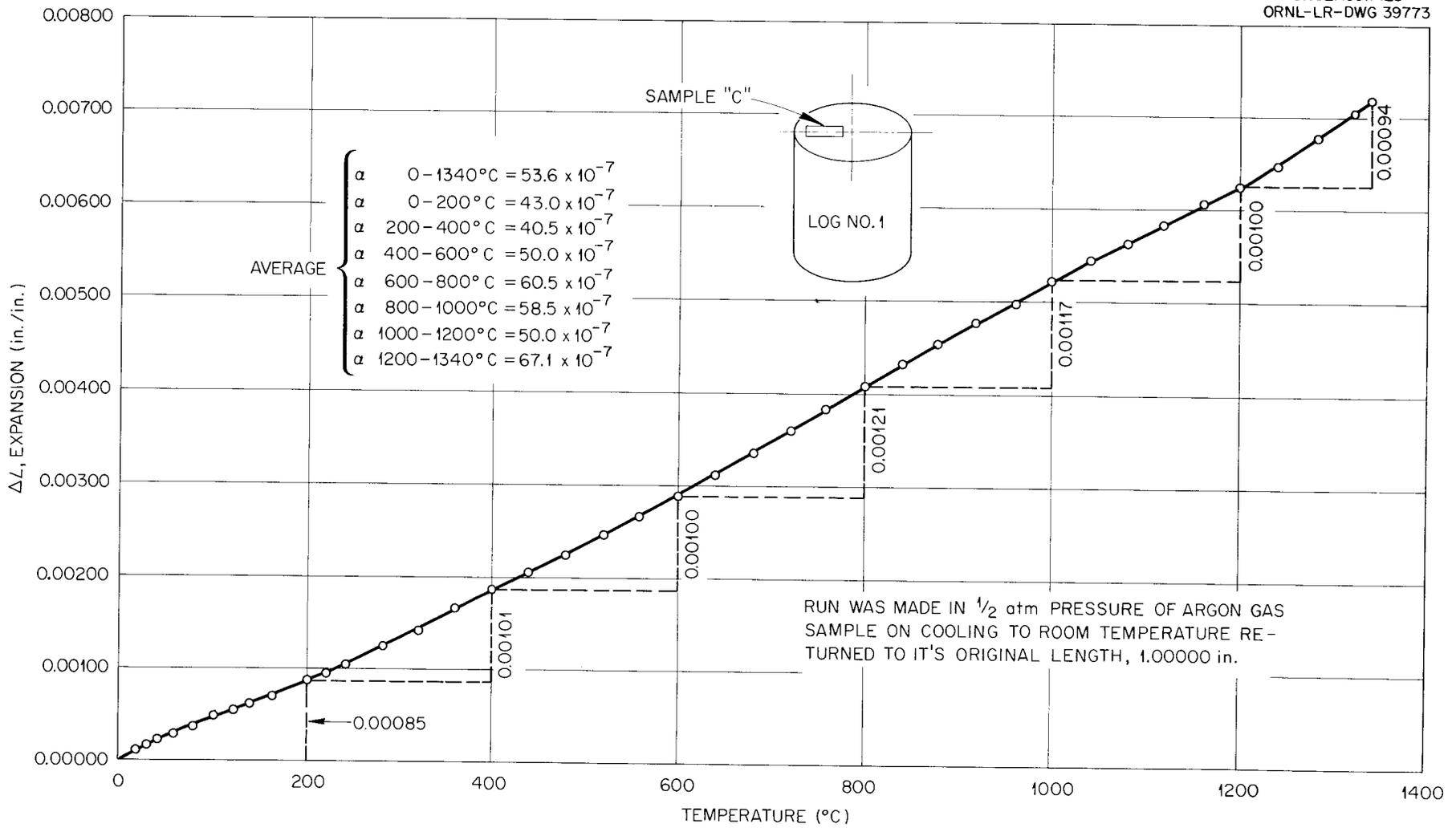


Fig. 18. Linear Thermal Expansion of a $1 \times \frac{1}{4}$ in. Round Rod of CS 312 Graphite.
Sample Cut from Outer Periphery of Log Perpendicular to Direction of Extrusion.

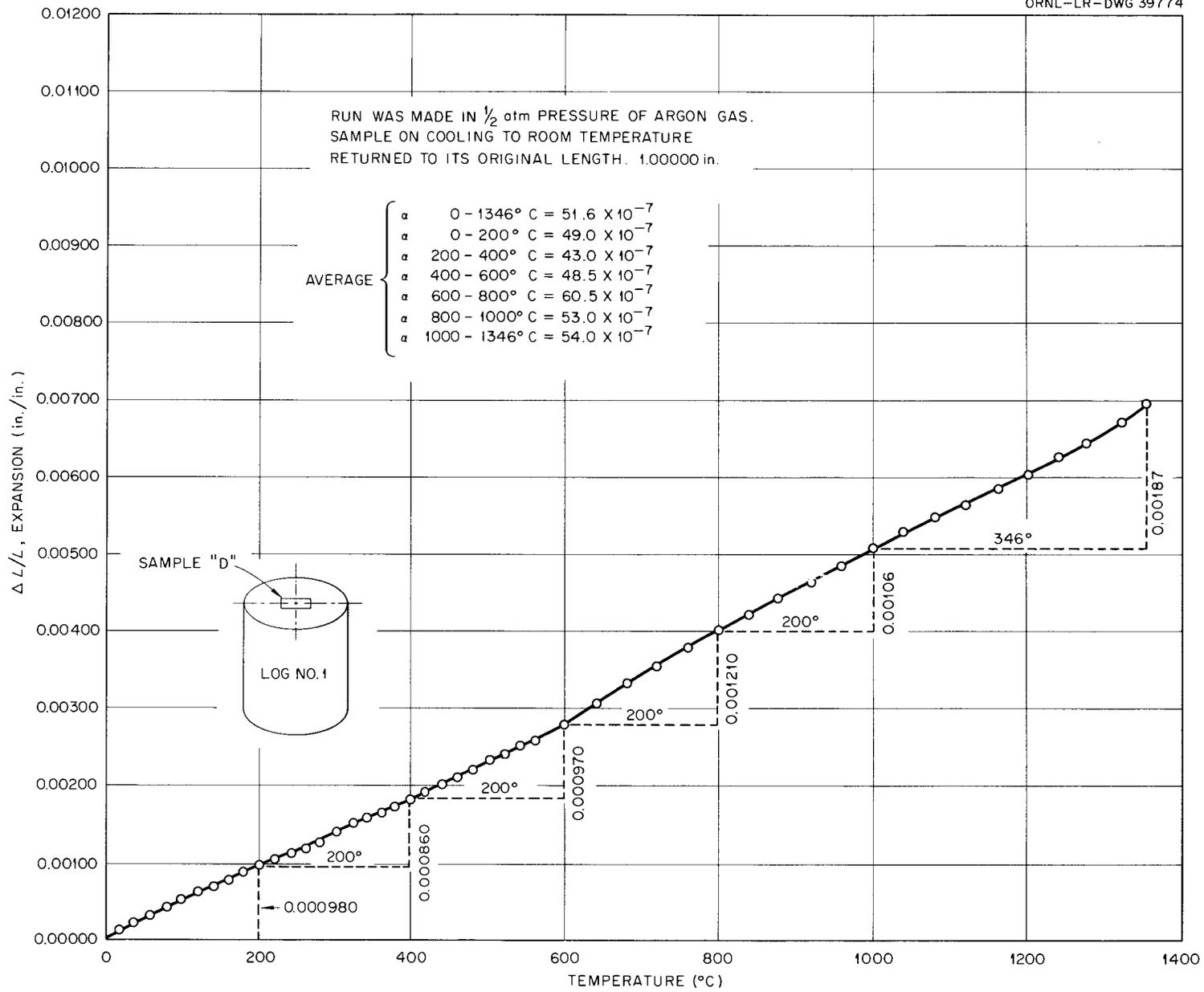


Fig. 19. Linear Thermal Expansion of a $1 \times \frac{1}{4}$ in. Round Rod of CS 312 Graphite.
Sample Cut from Center of Log Perpendicular to Direction of Extrusion.

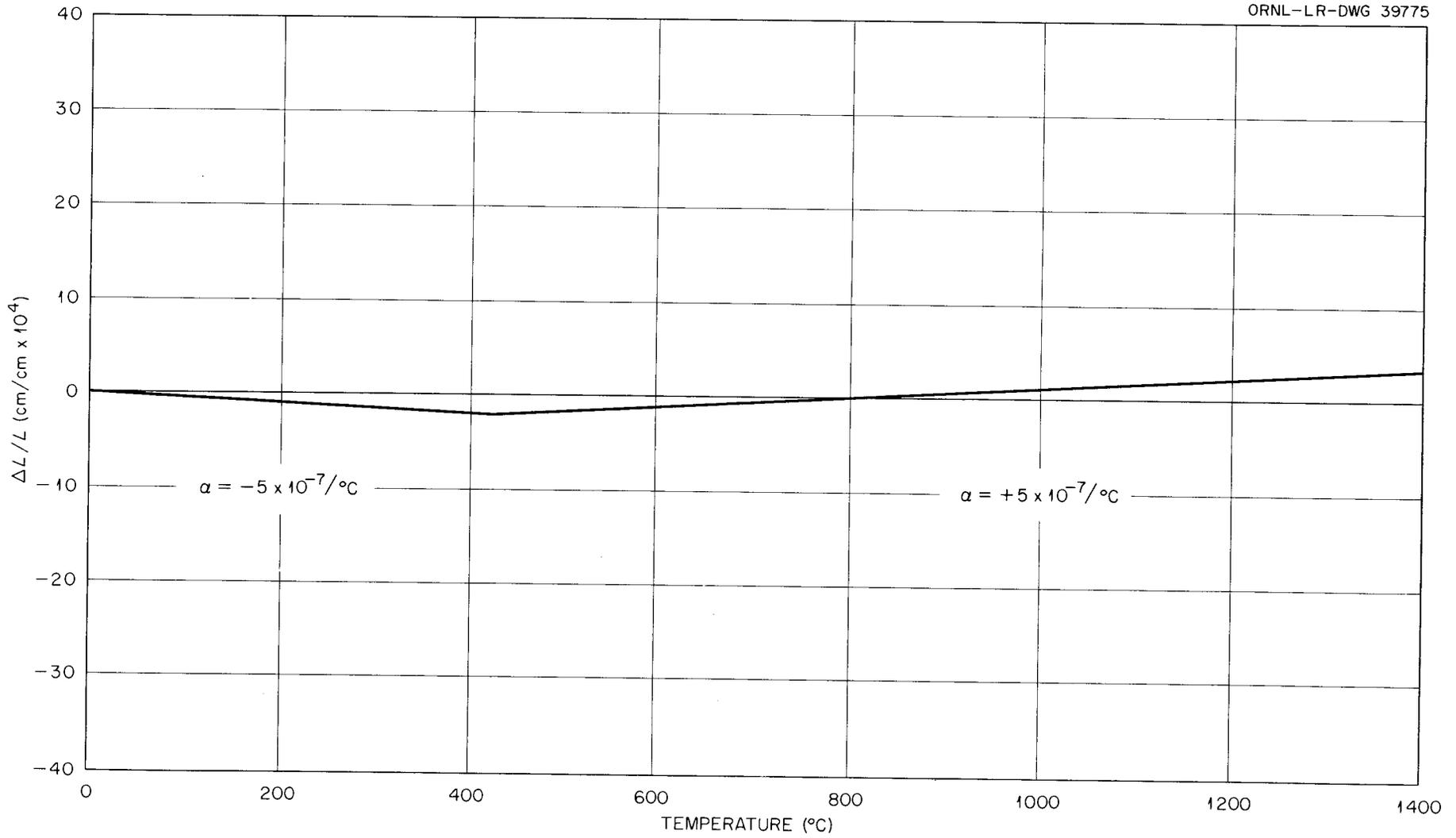


Fig. 20. Total Expansion, Boron Nitride.

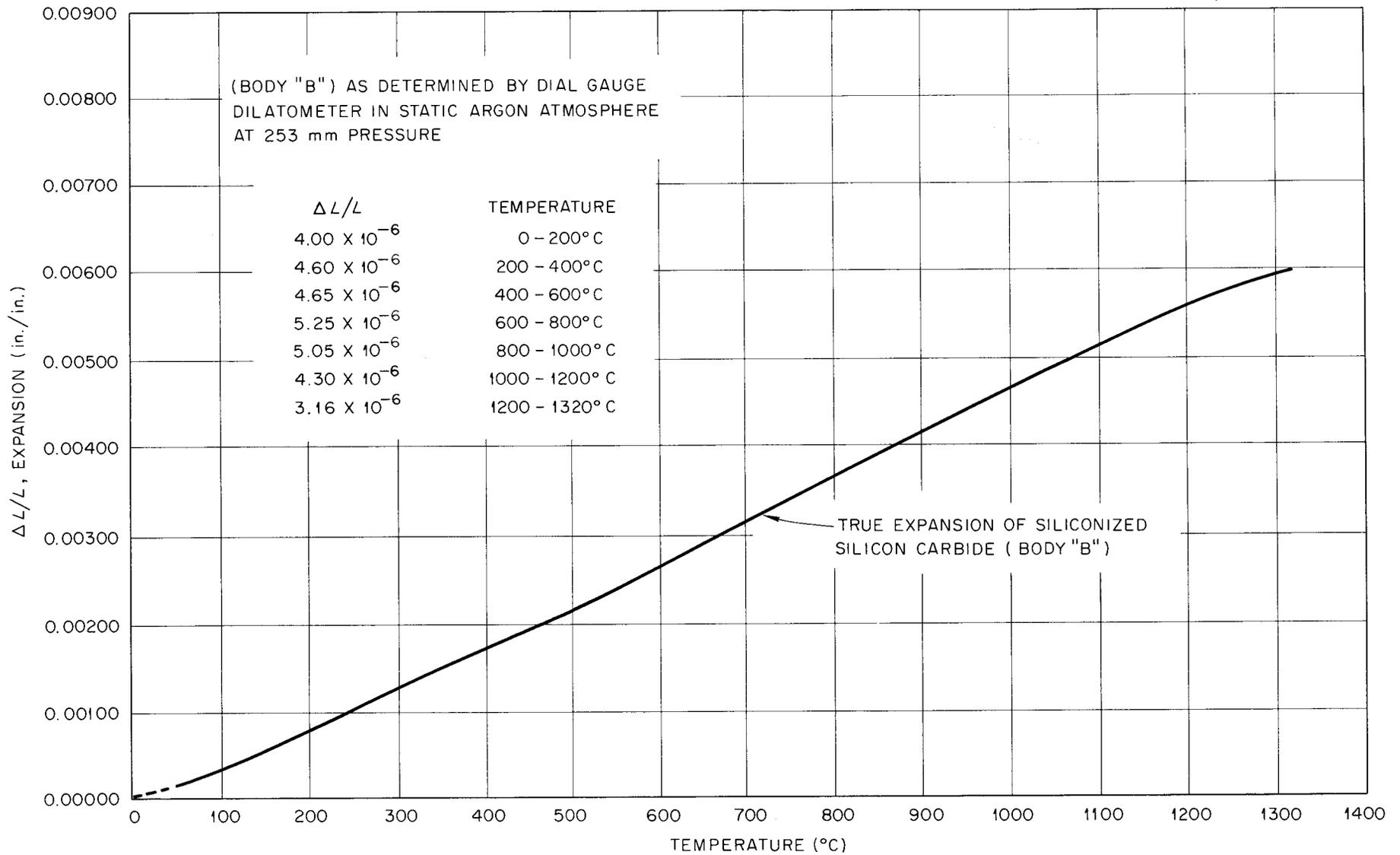


Fig. 21. Linear Thermal Expansion of a $1 \times \frac{1}{4} \times \frac{1}{4}$ in. Bar Specimen of Siliconized Silicon Carbide.

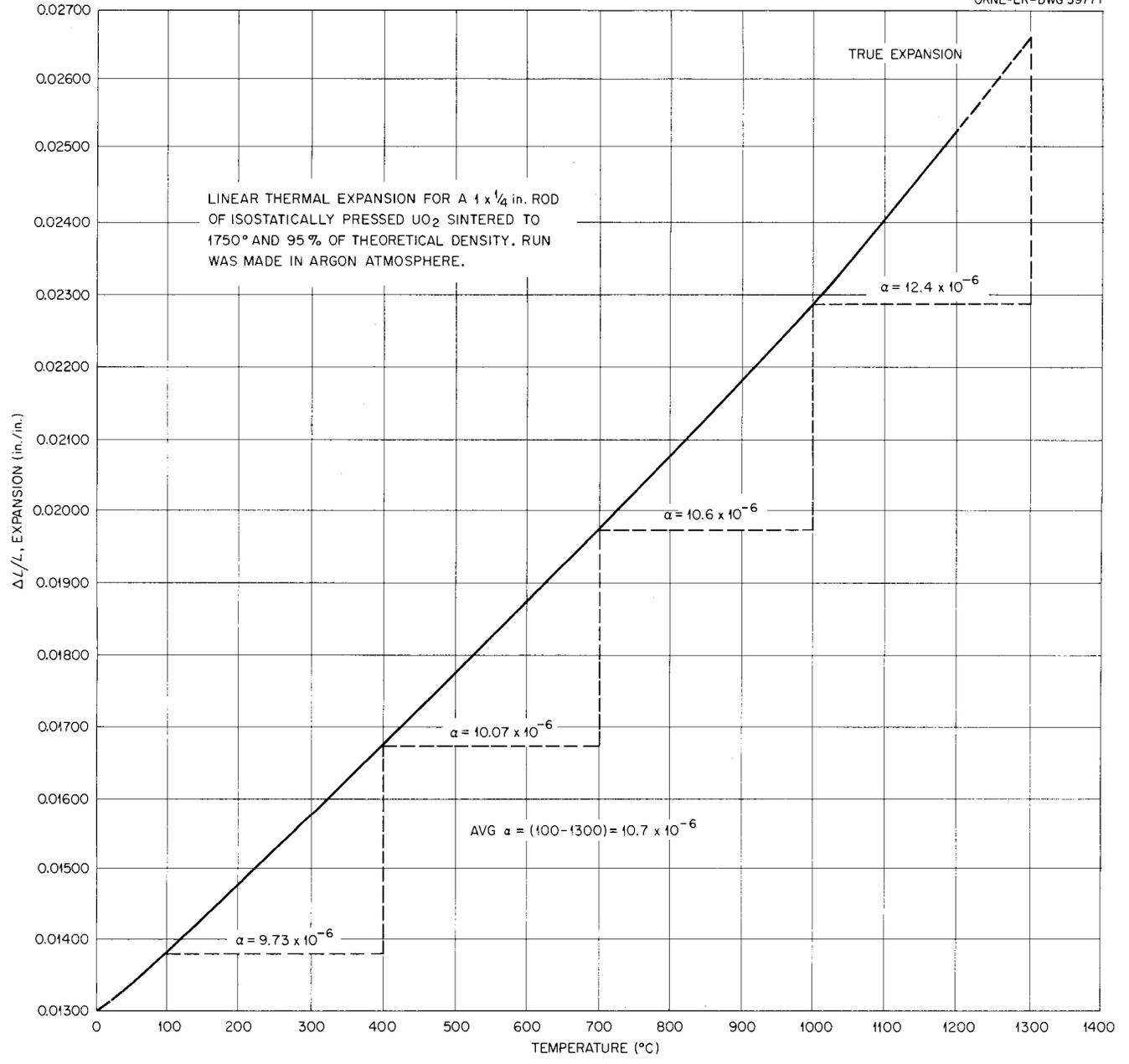


Fig. 22. Dial Gauge Determination.

There are a wealth of additional data to be found in the original manuscript of R. E. Clausing⁵ that further illustrates the work of the above described apparatus.

SUMMARY

The described apparatus should be of special interest to those investigators who are studying the physical properties of materials that cannot be heated in air. Some of its more desirable attributes are summarized. The apparatus adapts itself equally well to most of the materials from which a test specimen 1 in. x 1/4 in. x 1/4 in. can be prepared. There is no waiting between temperature increments for temperature equilibrium conditions if the rate of rise is not greater than three degrees per minute. The net result is a continuous curve, expressive of instantaneous coefficients of thermal expansion, and stepwise increments are eliminated. Results are found to agree very closely to some of the already accepted and published results for some of the better known materials, and the percentage error is believed to be no greater than $\pm 0.1 - 0.2\%$. The sapphire dilatometer units are usable up to as high as 1800°C. The apparatus is entirely automatic and its cost of construction is reasonable. Controls are simple, and the very small specimen size makes for a more uniformly heated specimen. In some cases the instrument can be used to determine phase changes in a material as illustrated in Fig. 9. Since the installation of a new Marshall furnace equipped with an air-cooling manifold, a fairly uniform rate of cooling can be maintained.

There are, of course, some undesirable features about the apparatus. The most plaguing of these drawbacks is in the mechanical linkage for the transmission of motion from the expanding specimen to the recorder pen. Mechanical slack and sticking on this system makes it necessary to tap the table each time a reading is desired. There is the difficulty of operating in a vacuum system. True expansion is not recorded directly but rather a differential expansion making

⁵R. E. Clausing, "Thermal Expansion Studies on High-Temperature Brazing Alloys," (publication pending).

a conversion curve necessary. The shortness of the specimen increases the chance for an error in any one single dilatation; however, this objection is not too serious for these errors tend to cancel out when many successive readings are plotted. The errors caused by the sticking of the mechanical linkage also tend to average zero when the over-all data are plotted.

ACKNOWLEDGMENT

The development of this apparatus was carried out in the Ceramic Laboratory of the Metallurgy Division. Much credit and thanks go to James R. Johnson, a former member of the Metallurgy staff and now with the Minnesota Mining and Manufacturing Company, St. Paul, Minnesota. His invaluable advice and calculations made possible the building and operation of this apparatus. Thanks are extended to John Draghic, now with the General Electric Company, Lockland, Ohio, and his colleague, Joseph Droher; during the building of the apparatus, both were with the NEPA Project at Oak Ridge. It was from their apparatus that much of the present apparatus was copied. Much credit also goes to both J. B. Wachtman and S. M. Lang of the U. S. Bureau of Standards, who contributed the ideas that made the apparatus automatic.



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