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

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FROM: S. D. Fulkerson

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I. INTRODUCTION

During the early evolution of the Ceramics Department of the Oak Ridge National Laboratory Metallurgy Division, a need arose for an apparatus for determining linear thermal expansion of materials that could not be heated in air. This need resulted in the development of the apparatus about to be described. The apparatus was designed, built, and put into operation some four years prior to this writing. A description of this apparatus now seems necessary because there has been some original data obtained on it that has recently been published, both for ceramics and alloys. Furthermore, it is believed that this apparatus has some very desirable attributes worthy of publication, although there is nothing new to offer by way of either instrument design or improved accuracies over some of the now existing ones.

Results are quite reproducible on the same materials and very close agreement is had with the already published and accepted data for some of the better known materials. As is the case with many other mechanical devices of this type, it is impossible to estimate the percentage accuracy. A reasonable estimate of the precision of this instrument would be 99%. It is realized that no thermal expansion data are any better than the instrument on which it is obtained, assuming that all other variables are held constant.

II. DESCRIPTION OF APPARATUS

The apparatus can best be described by treating it in its major component parts:

- (a) The furnace and controls.
 - (b) The vacuum system.
 - (c) The mensuration and sensing devices.
 - (d) Recording devices.
- (a) Furnace and Controls - The furnace is a platinum-40% rhodium resistance wound, shop-made type and has served very well the purpose for which it was intended. However, if this instrument is ever duplicated, it is suggested that a furnace of wider temperature limits be used, and that some provision be incorporated for a controlled rate of cooling. The upper specimen temperature limit is about 1350°C, due to the large ΔT between the heating element and the specimen. Also, due to the amount of heat insulation required to back up the heating element, cooling becomes extremely slow as room temperature is approached.

A cross section of the furnace is shown in Fig. 1. The shell, fs, is a cylinder that has been formed of 18-gauge stainless sheet and is 18-in. x 12-in. in dimensions. The ends of the shell are closed by means of 1/2-in. thick circular transite boards t. The transite boards t have been cut slightly larger than 12-in. dia in order that a shoulder could be machined on the circumference, over which the furnace shell fits and is secured by screws. There is also a circular groove 1/4-in. deep machined in each of the transite covers that is concentric with the core. This provides a means of holding in place the 4-in. ID fused-quartz tube, q. This tube keeps the high-temperature insulation, hi, which is bubbled alumina, separate from the low-temperature insulation, li, which is "Sil-o-cel", or diatomaceous earth. The furnace core, fc, consists of an alumina tube, grooved in a helix from end to end, and measures 1-1/2 in. ID x 1-7/8 in. OD. It too, is held in place by means of a shoulder machined into each of the transite ends, as shown. The heating element, Pt, is a 20-mil platinum 40% rhodium wire of about 6.5 ohms resistance. It is wound in the helical groove of the core for a distance of 12 in. The leads are of this same wire, but doubled, which pass through two small holes in the quartz tube and thence to the spark-plug insulators, sp, through the side of the metal furnace shell. The furnace stands vertically on a wood base which is protected by another transite board, and is mounted underneath the instrument proper, as can be seen in Plate 1.

Controls are quite simple and are determined by the method chosen to run a particular sample. It might be well to point out here one of the advantages the apparatus has over some of the others. It is built and calibrated for a very small specimen which offers the advantage of heating at a continuous rate, rather than heating up to a given temperature, and soaking for a period to assure a uniform specimen temperature. The specimen, which is a machined cylinder or square bar, 1-in. x 1/4-in., is small enough so that heating rates up to three degrees per minute can be used with no readable difference in temperature being observed in any portion of the specimen. This was determined experimentally by thermocouple placements, hence, only one couple is necessary to measure specimen temperature. It might also be well to mention one of the disadvantages of a short specimen. An error in reading a single dilatation is apt to be greater, percentage wise, than it would be on a longer one; however, the error would tend to average zero on numerous successive readings.

LEGEND

- t = Transite Board
 fs = Furnace Shell
 q = 4" D. Quartz Tube
 pt = Heating Element
 s = Specimen
 vt = Porcelain Vacuum Tube
 fc = Furnace Core
 sp = Sparkplug Insulators
 li = Low Temperature Insulation
 hi = High Temperature Insulation

CROSS SECTION OF FURNACE
 FIGURE 1.

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Since the power requirements for the furnace are never over 2 kw, an ordinary 110-volt variac of 20-amp capacity is sufficient when making runs where stepwise increments in sample temperature are used. If a continuous heating rate method is used, nearly any program controller of this same rating can be employed.

Automation is incorporated into this apparatus, a program controller is used, and dilatations are recorded simultaneously with temperature. The controller shuts off the power at the end of the run and the human element has been minimized in the determination. Opposing this very desirable characteristic is a very serious drawback - the furnace cannot be cooled at a controlled rate - but there is no reason why a furnace and controls could not be built that would do this and be substituted for the present one.

(b) Vacuum System - A very important part of this particular apparatus is the vacuum system. Although it was designed primarily for samples that could not be heated in air, it adapts itself equally well to any material. A more uniform heating of the sample is obtained in vacuum than in a gaseous atmosphere because convection currents are eliminated and the sample is heated by radiation only.

The component parts of the system are as follows:

The 13-in. dia x 24-in. long Pyrex bell jar, the 1-in. thick x 24-in. x 24-in. water-cooled brass base plate, a 1-in. ID x 1-1/4-in. OD x 18-in. long closed-end porcelain tube which passes through the brass plate and extends down into the heating zone of the furnace, and the vacuum pumping system.

The vacuum pumping system consists of a 4-in. oil diffusion pump (DPI), with the conventional oil vapor knockout baffle spool, backed by a Welch DuoSeal mechanical pump. Since these are all standard equipment they have neither been sketched nor described here. They can be seen in Plate 1 on the lower deck of the table. With some samples the system will maintain pressures as low as 0.05 microns during the heating cycle.

The bell jar shown in the photograph is guarded by means of a wire cage and is fitted with a rubber boot gasket, L-shaped in cross section, stretched over the open end.

From the standpoint of care in construction, the brass base plate is the part of the vacuum system which requires the most attention to detail. It is at this plate that the chances for a leak are the greatest. 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 electrical connections, sp, for the electrical leads to the sensing devices. Also, there are two other drilled and tapped holes, pc, 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. There is the 4-in. dia pump-out nozzle and flange, po, the seams of which are all hard soldered. It also serves the purpose of suspension for the 4-in. diffusion pump and baffle spool.

The large gasket groove, gg, which accomodates the bell jar, is machined in the upper face of the plate. It measures 13-in. ID x 14-7/8-in. OD x 3/16-in. deep. Since this groove must be flat and true, the machining was done after all soldering operations were complete.

A 1/4-in. copper tube, ct, which has been symmetrically bent and distributed around the various connections, is hard soldered to the bottom face of the base plate. This affords water cooling to the plate which in turn keeps all metal parts of the dilatometer head at a constant temperature.

The porcelain vacuum tube, vt, passes through the base plate and is coaxial with the furnace core. It extends well down past the center of the heating zone and is closed on the lower end. This is the vacuum heating chamber that receives the sample end of the dilatometer unit when the apparatus is assembled. It is held in place at the upper end and made vacuum tight by means of a rubber ring packing gland and nut, pg, built into the base plate. The 1-in. diameter is sufficient to allow for about 1/32-in. clearance between its walls and the dilatometer heat shields.

(c) Mensuration and Sensing Devices - There are two measuring and sensing units which are used with the apparatus; one, a manually operated unit and the other, an automatic. Both seem to yield equally 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. Due credit is hereby given to John Draghic of General Electric, Evendale, Ohio. The vacuum system and dilatometer unit of the present unit were copied from his apparatus. In the case of the automatic dilatometer, credit goes to Mr. S. M. Lang of the U. S. Bureau of Standards.

Both instruments are alike in their basic construction, as can be seen in Plate 2. The only real difference is that the dial gauge of the manually

BASE PLATE - PLAN

BASE PLATE - SECTION

BOTTOM VIEW OF BASE PLATE - COOLING SYSTEM

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
- br = Brass Slip Ring
- rr = Rubber Packing Rings

FIGURE 2. BASE PLATE DETAIL

operated unit has been replaced by a differential transformer sensing assembly for the automatic one. Neither one reads or senses the true dilatation of the test specimen, but rather a differential expansion between one inch of sapphire and 1-in. of specimen. In the construction of these units, as well as any unit of this type, there are always some causes of error introduced that no practical means can avoid. To mention some of them: there is the difference of expansion of the alumina anvil on which the specimen stands and the sapphire stay rods; the amount of spring loading on the push rod produces a like amount of stretch in the stay rods and compaction in the push rod; and, expansion of the metal parts produces some distortion in the stay rods. One cannot correctly assume that the true expansion of the test specimen is the sum of the dial-gauge increment and the expansion of one inch of sapphire. If one inch of single-crystal synthetic sapphire is used as a test specimen, theoretically the observed differential expansion should be zero all the way up the temperature scale, but this is not the case. There is always a slight difference in the negative direction due to these inherent built-in errors. Consequently, a calibration curve is necessary for each and every individual dilatometer unit of this type. The theories on which these general statements are based are expounded more fully under the heading: CALIBRATION-THEORY AND PRACTICE.

In the construction of these two units, the anvil on which the specimen stands is fabricated from No. 38-900 fused alumina powder compacted isostatically and soft fired to permit machining of the piece and later high fired to 1800°C. Prior to cementing the anvil to the sapphire stay rods, this 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. The cement used to rigidly secure the anvil to the lower ends of the stay rod consisted of a mixture of fine-grained fused alumina to which had been added 30% fine silica. This mixture sinters well at a temperature of about 1500°C. The sample holder is fabricated in the same way as was the anvil and the thick heat shield shown in the dial gauge picture, Plate 2. This difference in the units makes the dial gauge unit usable to a higher temperature because there are no metal heat shields in the hot zone.

All holes in the Inconel metal heat shields, as well as the ceramic heat shield and sample holders, are drilled on the same pattern. The three outer holes that accommodate the stay rods are 120° apart. A fourth hole, which

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PLATE 2

accommodates the two-hole thermocouple insulator tubing, is drilled midway between two of these holes. All these heat shields, as well as sample holder, are free enough to be easily slid up or down on the stay rods and thermocouple tubing. When in operation, they rest on a turn of small platinum wire wrapped around the stay rods. The hole in the very center of each of these shields is large enough to permit free vertical movement of the sapphire push rod, yet small enough to prohibit any lateral movement. The heat shields also serve another very important purpose. They act as guides and stays for all the rods and give rigidity to the extra long, and otherwise flimsy assembly.

From Plate 2 it can be seen that there is a large-diameter heat shield at the very top of the dial gauge unit. This is a disc of aluminum foil that fits snugly around all rods and serves the double purpose of stopping all radiation that escapes upward between the porcelain tube walls and the outer periphery of the heat shields down in the heating zone, and acts as a good thermal contact between the brass flange of the gauge holder and the water-cooled packing gland nut of the base plate when the instrument is lowered into place.

Shown in Plate 1 is a thick-walled hollow brass cylinder, with four large holes through the sides, set between the instrument head and the gland nut. Its purpose is to increase the mean free path for the escape of gas molecules from the vacuum heater tube and thereby assure 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.

The single-crystal synthetic sapphire rods are all centerless ground to a diameter of 0.121 in. and are perfectly straight. 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 and it is well that matched sets be used if they can be obtained; however, this is not essential, as the error encountered, due to unequal linear expansion in the rods, is mostly eliminated in the calibration curve for the instrument.

The three stay rods are cut 15-1/2 in. long while the push rod is one inch. shorter. The upper ends of the rod 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 slap 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 strains, due to spring loading, begin to relieve themselves. In operation, the tendency towards stepwise increments is, to some extent, eliminated 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 unit to rest upon after it is lowered into the furnace. It 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 than does the temperature of the tap water. Further refinement in precision could be made here by circulating water from a constant temperature reservoir.

The platinum-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 specimen this small, 1-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° per minute. 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 Plate 2 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 rod 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. It is then secured firmly in the correct position by means of a plastic cement.

In its construction, care must be used that none of the parts of the sensing head are made of a magnetic material. In this particular case all metal parts are of 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 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 device are basically those of a differential transformer and of a Wheatstone bridge. The primary of the differential transformer is energized by a 6 v, a.c. current. The secondary winding is essentially two coils in series, each of the same number of turns, but wound in opposite directions. When the armature of the unit 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 that 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 Plate 3.

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 checkrun switch serves both recordings. This switch is a timing mechanism which every five minutes simultaneously puts a check mark on each recording 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.

III. CALIBRATION - THEORY AND PRACTICE

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 of the materials whose expansion characteristics are known and accepted. Probably the one which adapts itself best to this purpose is fused silica, and it can be said that the calibration curve for this particular apparatus, along with a host of others similar to it, is based on this well-understood material.

"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. It can safely be said that very few other materials have this degree of linearity over this range.

From the literature⁽¹⁾, 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 safely be used for a calibration base. The danger lies in the fact that

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PLATE 3

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allowances must be made for the inclination of the c-axis to the rod axis which could vary from rod to rod due to either the machining or the manufacture of the specific piece used.

The instrument calibration curve shown in Fig. 3 is for the automatic unit, but the method used in its construction is typical for any similar unit. It was arrived at in the following manner: first, 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; next, average experimental data were taken from several runs using a strain-free specimen of fused silica carefully ground to the finished dimensions, 1-in. x 1/4-in. A second curve, which lies much 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 quartz test specimen is going to register as a positive quantity. Therefore, the algebraic difference of the two curves is the correction curve due to the total expansion within the instrument. It 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, which is the desired property being measured, the curves can be moved up or down the ordinate scale until their extrapolated zeros coincide without changing any values. 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 all future determinations of unknown thermal expansions.

In reporting the coefficient of linear thermal expansion, α , 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 making use of the observed data, if one were to use small enough increments on the curve and integrate

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METHOD OF ARRIVING AT INSTRUMENT CALIBRATION CURVE
FIGURE 3

between limits, an equation for the curve could 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. In the opinion of the writer, data of this type can only be correctly reported in the graphic form.

The calibration of the "Atcotran" recorder was accomplished by making use of the right combinations of optical flat gauge blocks, rung together and stacked up, to give stepwise increments in thicknesses of 0.001 in. each step. These blocks were placed flat on the specimen anvil of the dilatometer and the push rod 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 + and - limits of the transducer. When this shunt resistance was accurately set by the helipot, its value was then determined by placing the helipot in a milli-ohm bridge. A resistance of this value was then made up and permanently placed across the transducer secondary. It can be seen in the back and lower part of the instrument case, Plate 3.

During this 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 would be recorded on the chart in sufficient magnitude that they could easily be read by the unaided eye. By ringing the push rod down firmly, the last trace of air film would be squeezed out from between these two surfaces and then only would 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.

IV. ILLUSTRATIVE EXAMPLES

Figures Nos. 4, 5, and 6 are illustrative of work that has been done with the apparatus. Figure No. 4 not only shows the true expansion curve for French hot-pressed beryllium oxide, which is perpendicular to the direction of pressing, but also illustrates the method of determining this curve. In this case it is the algebraic sum of the correction curve and the data curve.

In Fig. No. 5 a comparison of two materials, hafnium-free zirconium oxide

THERMAL EXPANSION OF FRENCH HOT PRESSED BeO
FIGURE 4.

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COMPARISON OF EXPANSIONS OF ZrO_2 AND HfO_2
FIGURE 5.

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Linear Thermal Expansion for 1-in. Length of Inor-8 Alloy as Determined by Vacuum Dilatometer Using Direct Reading Dial Gauge. Sample used was only 0.989 in. long and data was interpolated to a value for a 1-in. length.

FIGURE 6

versus zirconium-free hafnium oxide, is made. For convenience of illustrating a point, the units in which these data are shown have been expressed in mm/m/°C. As was expected, the well-known inversion of ZrO_2 is clearly shown between 1100 and 1200°C where it changes over 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.

Data obtained with this apparatus indicate that HfO_2 does not go through this change of crystal form over this temperature range; however, when supplemented with high-temperature x-ray data this change was found to take place well up the temperature scale, progressing rapidly at 1850°⁽²⁾.

Since the instrument is calibrated for a 1-in. specimen, Fig. 6 is illustrative of the method used when the test specimen was slightly shorter than one inch. 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 for this length and the correction curve for the instrument, which was based on a 1-in. specimen. In magnitude, it amounts to the difference of expansions between 0.011 inch of INOR-8 and 0.011 inch of sapphire and is negligibly small. In plotting the corrected curve, expressive of the expansion of a 1-in. specimen, a correction in ΔL at any given temperature was added which was determined from the ratio $\Delta L/0.989$.

There is at least one other instance of reported data which were collected making use of this apparatus⁽³⁾, and much unpublished data.

V. SUMMARY

The above described apparatus should be of interest to those investigators who are studying the physical properties of materials; especially that of linear thermal expansion of materials that cannot be heated in air. In its favor some of the more desirable attributes are summarized. The apparatus adapts itself equally well to most any material from which a test specimen can be prepared. It yields quite dependable results; the error is believed to be no greater than ± 0.1 to 0.2% . The sapphire dilatometer unit itself is good up to as high as 1800°C. The apparatus is completely automatic and its cost of construction is reasonable. Controls are simple; the very small specimen size makes for a more uniformly-heated specimen, when a controlled rate of heating is employed. Data are reported directly in the graphic form and complicated calculations are avoided. In some cases the instrument can be used to determine phase

changes in a material, as indicated in Fig. 6.

Like many other pieces of laboratory equipment in which one strives to incorporate most of the desirable features into their construction, certain other undesirable features creep into it that have to be tolerated. For instance, the most plaguing of these drawbacks is the mechanical slap and sticking of the various linkages for transmitting motion. To mention some of the others, there is the difficulty of building a vacuum system at a reasonable cost. As it now stands, the instrument is limited to 1350°C specimen temperature by the moderate cost furnace which also prohibits a controlled cooling rate. True expansion of a specimen is not recorded directly but rather a differential expansion. The short specimen increases the chance for error in a single dilatation reading; however, this objection is not too serious since these mistakes usually disappear when the overall data are plotted. There are certain other built-in errors that have to be compensated for by making use of a calibration curve.

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REFERENCES

1. Souder, W. and P. Hidnert, "Measurements on the Thermal Expansion of Fused Silica," Sci. Paper, NBS. No. 524, (1926).
2. Curtis, C. E., L. M. Doney, and J. R. Johnson, "Some Properties of Hafnium Oxide, Hafnium Silicate, Calcium Hafnate, and Hafnium Carbide," Jour. of Amer. Ceram. Soc., Vol. 37 (10), (Oct., 1954).
3. Clausing, R. E., "Thermal Expansion Studies on High-Temperature Brazing Alloys," Intra-Lab. Correspondence.

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