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**A DIFFERENTIAL DILATOMETER FOR RAPID
DETERMINATION OF THERMAL EXPANSION
COEFFICIENTS NEAR ROOM TEMPERATURE**

By
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July 14, 1953

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Metallurgy Division
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July 14, 1953

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INTRODUCTION

A simple, rugged, and inexpensive differential dilatometer has been developed for the determination of thermal expansions over the range from room temperature to approximately 100°C. The dilatometer was designed and built jointly by the Remote Control and Metallurgy Divisions of Argonne National Laboratory. The instrument specifications and requirements were dictated by the nature of the work planned.

INSTRUMENT TYPE AND REQUIREMENTS

A continuing program at Argonne involves the metallurgical study of a multiplicity of specimens from 1 in. to 2 in. long and from 3/8 in. to 1/2 in. diameter. A rapid convenient means for measuring their linear thermal expansivities over the temperature range of approximately 30° - 100°C was desired. It was estimated that the average change in length would be about 0.001 in. and that it would be desirable to measure this excursion with an accuracy of $\pm 2\%$. For rapidity and simplicity it was decided to use liquid immersion in hot and cold ethylene glycol to establish the temperature difference.

Simple devices for magnifying small displacements include measuring microscopes; optical leverages; mechanical leverages, some of which are commercially available as "dial indicators"; and interference systems.

Measuring microscopes, which may be considered to be optical leverages using lenses as pivots, are simple and compact devices for greatly improving visual acuity. With a filar eyepiece, measurements may be made with an accuracy of 0.0001 in. or less. Such resolution permits very accurate determinations when long gauge lengths are available. For this application, however, not only additional magnification but also a means of transferring the displacement from the submerged specimen to a convenient remote point is required.

Optical levers may be designed to produce a very large magnification of displacement. Such a system, however, becomes space-consuming and usually finds application either where the indication is to be autographic,

as with the spot of light falling on a rotating film drum, or where the test might be time-consuming and the operator would not wish to be confined to an eyepiece for the duration of the measurement.

Mechanical leverages must be designed and used with discretion if very small displacements are to be amplified. If constructed with freely pivoting parts, such as an ordinary geared dial indicator, starting friction and lost motion may mask the desired motion. If designed with "flexure pivots," the spring constant of the suspension must be kept adequately low. Such arrangements are thermally sensitive unless built of materials with low coefficients of thermal expansion or designed for thermal compensation.

Even with the most suitable mechanical or dial indicator systems there are apt to be other limiting features. A fused quartz tube and dial indicator dilatometer,⁽¹⁾ while accurate to a possible $\pm 2\%$, is fragile, requiring careful handling. To obtain the higher accuracy mentioned, a long sample of considerable size is required (20 cm in length). In another similar instrument⁽²⁾ a 4 in. sample is specified. As simple as these instruments are, it is entirely possible that a work program can be interrupted with considerable replacement expense due to the breakage problem. A slightly different type of instrument⁽³⁾ may obtain suitable accuracy with a shorter specimen due to the incorporation of a magnifying lever in conjunction with the dial gauge. All of these instruments may be used over a wide range of temperatures, in many cases over a considerably wider range than that anticipated for the work planned at this Laboratory.

Systems which measure displacements in units of wave lengths of light, using interference phenomena, are regarded as being basically the most accurate. Their use usually requires an operator to count the transit of interference fringes. In this case the counting of a large number of fringes at the high rate resulting from immersing the specimen in hot liquid discouraged use of the method.

Based upon a review of available methods it was decided to construct a dilatometer in which a large part of the magnification would be accomplished with a measuring microscope. A one-stage lever magnification of about 50:1 was planned to improve the resolution to the desired degree of accuracy.

DESCRIPTION OF DILATOMETER AND ACCESSORY EQUIPMENT

A full section view of the completed dilatometer is shown in Figure 1. The whole unit is relatively simple and compact, consisting of the microscope measuring unit on the left and the support arm, push rod and lever system on the right. The push rod and support arms are of Invar, eliminating the fragile nature of quartz systems but at the same time imposing more of a differential nature upon the system. The long lever arm provides a large magnification of specimen length changes which are then measured by the

(20X) Gaertner Filar Micrometer Microscope (M-112A), capable of measuring to 0.00005 in. per smallest division of the micrometer drum. (Actually the eyepiece resolution was determined to be ± 0.00015 in. or ± 3 small divisions on the drum.)

Lost motion in the mechanical lever system is overcome through a torsional spring suspension, as shown in the upper center of Figure 2. The torsion spring supports are also of Invar. One support and spring is stationary while the other is free to move with the push rod. The two knurled locking screws on the side of the aluminum support block provide adjustment of the instrument to specimen lengths ranging from about $7/8$ in. to $2-3/8$ in. The main Invar support bar (center) may be locked to the support block or to the differential screw (left), passing vertically through the block. In the latter instance the differential screw allows for careful lowering of the push rod onto the specimen.

The corrugated diaphragms that provide partial support for the push rod may be seen in Figure 3. These diaphragms allow high compliance for vertical motion and low compliance for transverse forces. A specimen about 1 in. long by $3/8$ in. diameter is shown resting on the stainless table. The specimen end of the push rod terminates in a $1/8$ in. diameter stainless steel ball.

Accessory equipment includes a thermocouple and potentiometer set up for temperature measurement. Two 800-ml beakers containing ethylene glycol are used, one at room temperature, the other at approximately 100°C . The hot glycol is held at temperature by a Chromalox ring type (A-O) 115V-175W heater resting on the bottom of the beaker. Temperature is controlled by a small Variac (General Radio Co. #200-C). By this method of heating there is little extraneous heat loss to the equipment during a typical test run. An elevator table has replaced the ring support shown in Figure 1, providing for easier manipulation of the glycol beakers.

CALIBRATION AND OPERATION

The first operation involved in the calibration of the instrument consists of determining the nature of the relationship of lever arm movement with change of specimen length. This relationship may be obtained by two methods: (1) a load-deflection plot of the spring suspension, or, (2) a varying specimen length-deflection plot. A characteristic load-deflection plot is shown in Figure 4. This plot was obtained by raising the push rod with a weighing system and measuring the lever arm deflection in eyemils.* A linear relationship exists over the largest deflection anticipated for the material to be tested. A similar type of linear plot was obtained using a series of cylinders of varying length (length vs. eyemils), all specimens being at room temperature.

* Defined as the movement of the lever arm of 0.001 in. as measured with the micrometer microscope.

Several other requirements must be satisfied before proceeding further with the calibration. The instrument must be insensitive to small changes in ambient temperature that might occur during an actual test. The use of Invar in critical locations, particularly in the upper part of the push rod and lever system, was intended to prevent any eyemil drift resulting from a slight change in temperature in uncompensated areas. No significant eyemil change was noted on an Invar test specimen after holding for approximately 20 minutes at 100°C.

For any test run only two temperature readings are used, the initial (RT) and final (approx. 100°C). Temperature equilibrium is required in either case if satisfactory results are to be obtained and "closure" is a further prerequisite. Results of a large number of tests indicate that 5 minutes at temperature will establish uniformity and a 10-minute reading will serve as a check. In all cases where the temperature has been allowed to return to the initial value with the specimen in place, the eyemil readings have checked within the resolution limits of the eyepiece (± 0.00015), i.e., "closure" has been obtained.

Having satisfied the initial requirements the second phase of calibration or determination of the lever arm magnification may be undertaken. This may be accomplished by using carefully prepared specimens of varying length, as mentioned earlier, or by the use of specimens of known thermal expansion characteristics. Both methods were used, and the lever arm magnification was found to be about 56 times by both methods. Values obtained by the first method will not be listed in detail. The calibration by standards will be discussed in detail because of the determination of corrections to be used later.

Two specimens of known thermal expansion properties were obtained from the National Bureau of Standards: An alloy specimen (DO), 1/4 in. diameter by 1 in. long, having a coefficient of expansion for the range from 25°C to 100°C of 17.5×10^{-6} in./in./°C, and a glass sample (DN), 1/4 in. by 1/4 in. by 1 in. long, having a coefficient of 9.2×10^{-6} in./in./°C for a temperature range from 20°C to 100°C. Each specimen was checked several times between about 25°C and approximately 100°C; the results are tabulated in Table I.

Table I
CALIBRATION TEST DATA

Test	Initial Temp. (°C)	Final Temp. (°C)	ΔT (°C)	Eyemils (5 min) (in. $\times 10^3$)
<u>Specimen DO (Alloy) 1.000 in. length</u>				
1	26.0	96.0	70.0	56.49
2	24.0	99.5	75.5	60.48
3	29.0	97.5	68.5	54.58
<u>Specimen DN (Glass) 0.9986 in. length</u>				
1	23.5	94.0	70.5	23.77
2	24.5	96.0	71.5	24.18
3	27.0	92.0	65.0	21.34

From the above readings on the two specimens and the linear relationship of eyemils vs. change in specimen length established previously, the lever arm magnification (M) and the instrument correction (K) for change in length of the Invar side arms may be determined as indicated below:

$$Y = M\Delta L_s(\text{specimen}) - M\Delta L_I(\text{Invar}) ,$$

where

$$Y = \text{Measured specimen } \Delta L \text{ (eyemils)}(\text{in.} \times 10^6)$$

$$M = \text{Magnification of lever arm}$$

$$\Delta L_s = \text{Specimen change in length}$$

$$\Delta L_I = \text{Invar side arm change in length} .$$

$$\text{Further } Y + M(L_I \Delta T \alpha_I) = M(L_s \Delta T \alpha_s) ,$$

where

$$L_I = \text{Initial Invar length}$$

$$\Delta T = \text{Temperature range}$$

$$L_s = \text{Initial specimen length}$$

$$\alpha_I = \text{Thermal expansion coefficient (TEC) of Invar}$$

$$\alpha_s = \text{TEC of specimen;}$$

but

$$L_I = L_s = L_0 \text{ of the specimen (Initial Length) .}$$

Letting $K = M\alpha_I$ and substituting ,

$$Y + K(L_0 \Delta T) = M(L_0 \Delta T \alpha_s) \quad (1)$$

Substituting in equation (1) the measured values given in Table I for the standard specimens DO and DN we have six equations and two unknowns which, when solved simultaneously by the method of averages, give

$$\left. \begin{array}{l} M = 0.05619 \times 10^6 \\ K = 0.1817 \end{array} \right\} \text{ 5-minute readings .}$$

Using a similar procedure and 10-minute readings,

$$\left. \begin{array}{l} M = 0.05584 \times 10^6 \\ K = 0.1783 \end{array} \right\} \text{ 10-minute readings .}$$

Putting these constants into equation (1) and solving for α , the thermal expansion coefficient, we have:

for the 5-minute reading:

$$\alpha = \left[17.797 \left(\frac{Y}{L_o \cdot \Delta T} \right) + 3.234 \right] \times 10^{-6} \quad , \quad (2)$$

and for the 10-minute reading:

$$\alpha = \left[17.908 \left(\frac{Y}{L_o \cdot \Delta T} \right) + 3.193 \right] \times 10^{-6} \quad , \quad (2')$$

where Y is the eyemil reading.

A typical calculation is shown below:

From Table I, for Specimen DN (Glass) and test #1:

$$L_o = 0.9986 \text{ in.}$$

$$\Delta T = 70.5^\circ\text{C}$$

$$Y = 23.77 \text{ eyemils (5-minute reading) } .$$

Substituting these values in equation (2):

$$\begin{aligned} \alpha &= \left[17.797 \left(\frac{23.77}{0.9986 \times 70.5} \right) + 3.234 \right] \times 10^{-6} \\ &= [6.009 + 3.234] \times 10^{-6} = 9.24 \times 10^{-6} \text{ in./in./}^\circ\text{C} \quad . \end{aligned}$$

(The NBS value for this specimen was $9.2 \cdot 10^{-6}$ in./in./ $^\circ\text{C}$.)

The most satisfactory method for a rapid determination of thermal expansion is to place the specimen on the table and lower the push rod onto the specimen by means of the differential adjusting screw until a movement of about 25 to 30 eyemils is observed in the microscope. (This corresponds to an initial load of about 100 grams as shown in Figure 4.) The upper clamping screw (Figure 2) is tightened and the lower one loosened, locking the main support bar to the block instead of to the differential screw. With a thermocouple fastened to the specimen, the room temperature beaker of ethylene glycol is moved up so that the top of the solution is well over the top of the specimen. After several minutes temperature equilibrium is obtained and an initial eyemil reading is taken. The room temperature solution is replaced with the one at approximately 100°C , and eyemil and temperature readings are taken at one minute intervals up to 5 minutes, at 7-1/2 and at 10 minutes, after which the hot solution is removed and the test completed, unless a "closure" check is made. Calculations are made for both the 5 and 10-minute readings. The 10-minute reading is a check on the first reading and will further indicate whether the instrument is being adversely affected by time at testing temperature.

The operational procedure outlined above was used on all the calibration results discussed previously, 5-minute readings being used in all computations. All readings and calculations of a typical test run were recorded on a permanent record form, as shown in Table II.

RESULTS

For the designed purpose and with few exceptions, the instrument has given excellent performance. Reproducibility has been well within the range of $\pm 0.2 \times 10^{-6}$ in./in./°C in all cases where a series of tests were made on the same specimen by the same operator. The lever arm position can be determined within ± 0.15 eyemils. Considering a magnification of 56, this reading range represents approximately ± 2.15 microinches error in the actual length change of a specimen between room temperature and 100°C. The accuracy of temperature measurements, combined with other instrument characteristics as well as sample surface preparation and measurement, probably account for the larger part of the reproducibility range.

The over-all measurement accuracy is dependent upon the specimens used for calibration. In this case the calibration standards (DO and DN) were reported to be accurate to 0.1×10^{-6} in./in./°C.⁽⁴⁾ Based on these specimens the accuracy of a coefficient determination is estimated to be within 0.2×10^{-6} in./in./°C. Further tests on material of known characteristics will be required, however, to actually establish the limits of accuracy.

LIMITATIONS

The design of this instrument was based largely on the nature of work planned at this Laboratory; accordingly certain limitations are inherent so far as adaptation to other testing programs are concerned. Specimen size is limited by instrument design, which is not subject to easy modification. Temperature ranges near room temperature, other than the one used in this report, would generally require a separate calibration similar to the one reported. Under any circumstances, the useful temperature range is relatively small. For rapid thermal expansion analysis only the initial and final points are determined. Measurement of the change in length of a specimen with increments of temperature is unwieldy with this apparatus, and the results would probably not justify the laborious calibration that would be required. While the device is not affected by slight fluctuations in ambient temperature, the prolonged exposure to testing temperatures involved in this method of testing might produce undesirable test results.

With the present torsion spring assemblies the instrument is not recommended for use on soft materials because of the load involved (see Figure 4). This limitation would also apply to materials of extremely high

Table II

DILATOMETER TEST DATA SHEET

Material No. U 8-1

Page No. _____

Spec. Length (L_0) = 1.0254 in.

Date _____

Time (Min)	Temp. (Millivolts)	Eyemils (in. $\times 10^3$)	5-Min Temp. °C	98.0
			Initial Temp. °C	<u>27.5</u>
0	1.42	+ 7.86		
1	4.67	- 29.52	5-Min ΔT	= 70.5
2	5.02	- 32.63		
3	5.12	- 34.01	10-Min Temp °C	99.0
4	5.16	- 34.51	Initial Temp °C	<u>27.5</u>
5	5.18	- 34.79		
7.5	5.23	- 35.29	10-Min ΔT	= 71.5
10	5.23	- 35.46		

5-minute results, equation (2):

$$\begin{array}{rcl}
 \text{Initial eyemils} & + & 7.86 \\
 \text{5-Min eyemils} & - & \underline{34.79} \\
 Y = & & 42.65 \\
 L_0 = & & 1.0254 \text{ in.} \\
 \Delta T = & & 70.5 .
 \end{array}$$

Substituting in equation (2):

$$\alpha = \left[17.797 \left(\frac{42.65}{1.0254 \times 70.5} \right) + 3.234 \right] \times 10^{-6} = 13.7 \times 10^{-6} \text{ in./in./}^\circ\text{C} .$$

10-minute results, equation (2):

$$\begin{array}{rcl}
 \text{Initial eyemils} & + & 7.86 \\
 \text{10-Min eyemils} & - & \underline{35.46} \\
 Y = & & 43.32 \\
 L_0 = & & 1.0254 \text{ in.} \\
 \Delta T = & & 71.5 .
 \end{array}$$

Substituting in equation (2):

$$\alpha = \left[17.908 \left(\frac{43.32}{1.0254 \times 71.5} \right) + 3.193 \right] \times 10^{-6} = 13.8 \times 10^{-6} \text{ in./in./}^\circ\text{C} .$$

Remarks: Second test on same specimen gave a 5-minute coefficient of $13.6 \times 10^{-6} \text{ in./in./}^\circ\text{C}$.

coefficients in which case the specimen length should be kept to a minimum. Either existing limitation may be overcome, in part at least, by adjusting the torsion spring assemblies to suit the work program, requiring a new calibration in each case. Even after adjustment, the instrument might not be suitable for extremely soft materials since a small push rod load is inevitable.

The differential nature of this instrument should not be considered a limitation, except perhaps in the case of extremely low coefficient material. Most dilatometers of a simple nature incorporate quartz or other low coefficient material in their make-up and similarly require calibration. It is therefore not practical to attempt to determine the coefficients of those materials which have coefficients near those of the materials used in the construction of the instrument.

The instrument is neither automatic in operation nor autographic. The only permanent record is that compiled by the operator through the course of a test, as shown in Table II.

CONCLUSIONS

This report has described the design, calibration, operation, results, and limitations of a mechanical type of differential dilatometer for rapid determinations of thermal expansion near room temperature. Excellent performance has been attained. The precision and accuracy are such that changes of expansion characteristics with metallurgical treatment may be easily analyzed, justifying the design and construction of the apparatus. It has been stated previously that this instrument cannot be classified as a universal testing device. Its design was based on work planned at this Laboratory and for this work it has proved extremely useful. Ease of handling, ruggedness and rapidity of testing are virtues not always present in comparable commercially available instruments.

ACKNOWLEDGEMENTS

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The multitude of operational test runs and calibration procedures were meticulously performed by R. J. Friddle of the ANL Metallurgy Division, who was also responsible for the design and construction of some of the auxiliary equipment.

Assistance in direction of the program was rendered by H. H. Chiswik, also of the ANL Metallurgy Division.

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Figure 1

Full Section View of Differential Dilatometer

Figure 2

View of Push Rod and Lever System Showing Torsional Spring Suspension

Figure 3

View of Push Rod and Support Arms Showing Corrugated Diaphragms

FIGURE 4

TYPICAL LOAD - DEFLECTION PLOT OF DILATOMETER FOR A GIVEN SET OF TORSION SPRINGS

MILLIMETERS (Deflection of lever arm in microscope)