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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

AN AUTOGRAPHIC STRAIN-GAGE DILATOMETER FOR PLUTONIUM AND ITS ALLOYS

Printed in USA. Price 75 cents. Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

LA-2175
INSTRUMENTS
(TID-4500, 13th Ed., Suppl.)

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REPORT WRITTEN: June 1952

REPORT DISTRIBUTED: February 24, 1958

AN AUTOGRAPHIC STRAIN-GAGE DILATOMETER FOR PLUTONIUM AND ITS ALLOYS

by

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Contract W-7405-ENG. 36 with the U. S. Atomic Energy Commission

ABSTRACT

A vacuum dilatometer for use in determining the expansion characteristics of plutonium and its alloys is described. The dilatometer consists of a vertical silica tube containing the specimen and a silica push tube which transmits specimen expansion to a cantilever beam. Strain gages cemented to the beam convert linear displacement of the specimen into an electrical signal that is fed to a Leeds and Northrup X-Y recorder. Specimen temperature, as determined by a thermocouple, is also fed to the recorder. Heating of the specimen is obtained by means of a split-tube furnace.

The principal advantages of this dilatometer over the popular silica-tube and dial-indicator dilatometer include light loading of the specimen and continuous autographic recording of expansion-versus-temperature data. Experience has shown this dilatometer to be simple in operation, reliable and adaptable to determining the expansion characteristics of a large number of plutonium alloys.

INTRODUCTION

The determination of thermal expansion curves has been found to be one of the most valuable methods used in metallurgical research for establishing solid-phase transformation temperatures in pure metals and alloys. Use of this method was outstandingly successful in the early investigations of Martin and Selmanoff,[1] which showed the existence of five allotropic modifications in plutonium. They used a manually operated silica-tube and dial-indicator type of dilatometer[2] that had been modified for vacuum and inert gases as described by Walters and Gensamer.[3]

As a result of these early dilatometric investigations on plutonium, the technique became firmly established as a useful means for conducting phase transformation studies on plutonium and plutonium alloys. At the same time it was evident that the silica-tube and dial-indicator apparatus had several inherent limitations which would eventually need to be eliminated if precise measurements were to be obtained. The equipment required the constant attention of a skilled technician, and plotting the dilation-versus-temperature data was laborious. Another objection was that frequently readings could not be made fast enough to follow the true changes in length of a specimen. The stepwise transformation of delta plutonium to gamma while cooling at 3°C/min is a good example of this situation.

The limitations on the silica-tube and dial-indicator dilatometer seemed to justify the use of a recording dilatometer which could automatically make a continuous record of dilation versus temperature. In addition to the continuous recording feature, other basic requirements which were established for dilatometric research on plutonium alloys included the following:

1. Loading limit to be a minimum in order to permit dilatometric measurements to be made on very soft phases at elevated temperatures
2. Minimum total range to be 0.120 in.
3. A high-vacuum system capable of being evacuated to pressures of 5×10^{-5} mm of Hg or lower to be provided
4. Hood or enclosure suitable for work with alpha-active materials to be provided
5. Temperature range of operation from 0° to 1000°C; heating and cooling at rates on the order of 3°C/min

Unfortunately, a commercially available dilatometer could not be found that would meet the requirements mentioned above. It became necessary to analyze the equipment requirements and to design and construct the apparatus. This resulted in the development of an autographic dilatometer (designated Model SGD-1) based on the concentric-silica-tube principle.

DILATOMETER CONSTRUCTION

General Description

The general features of the strain-gage dilatometer and accessory equipment are shown in Figs. 1 and 2. The basic operating principle

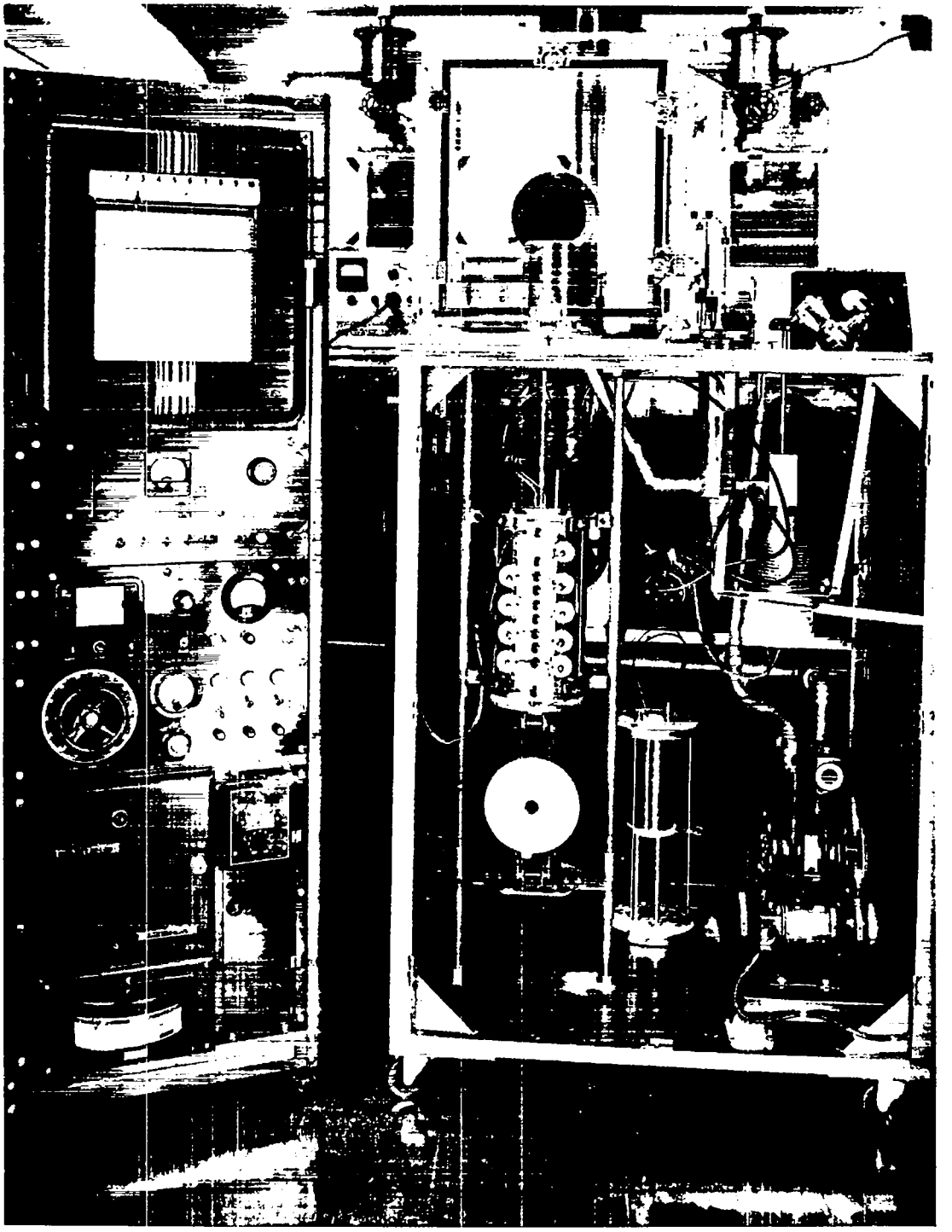

Fig. 1 Autographic Strain-Gage Dilatometer, Model SGD-1.

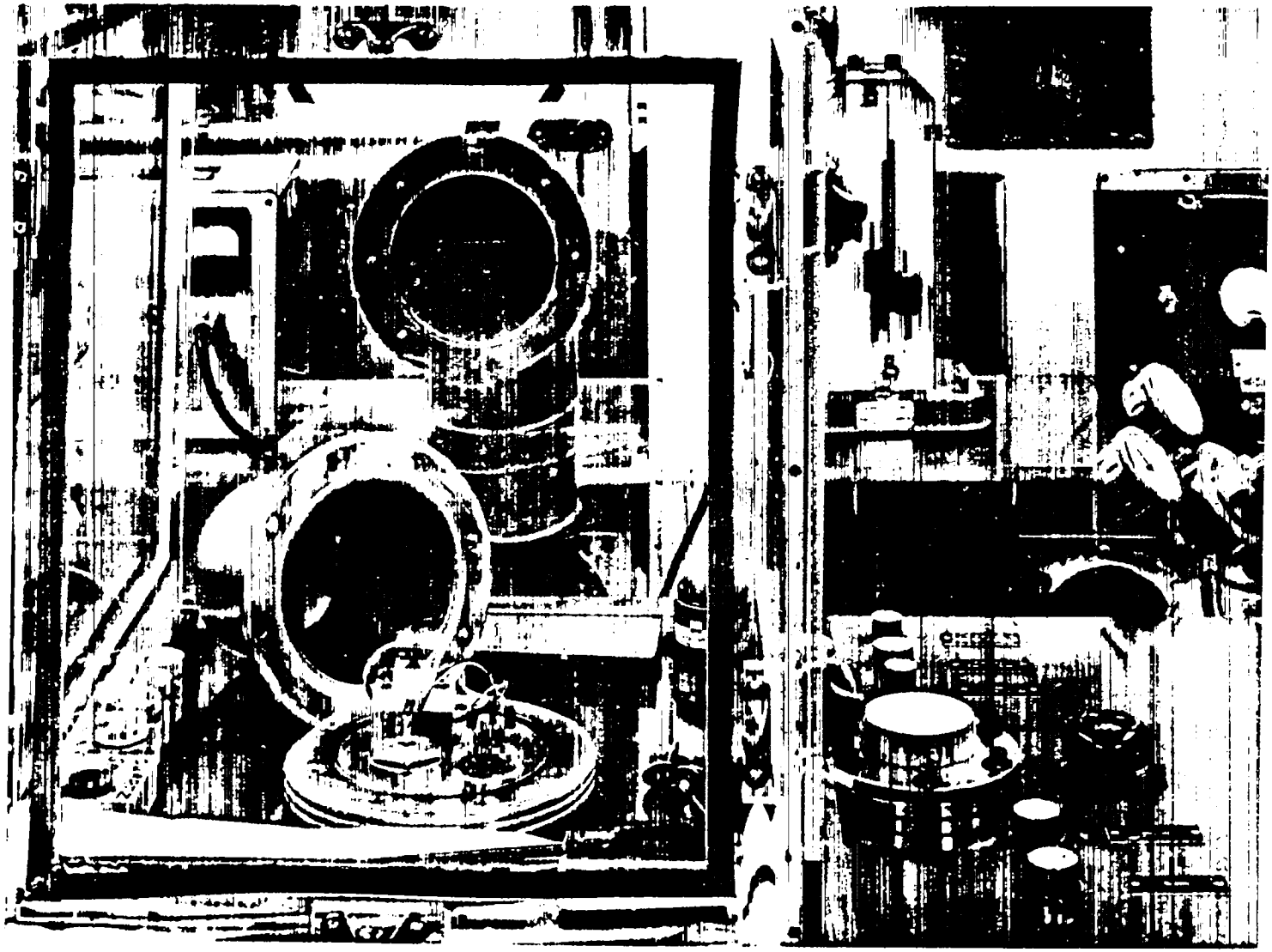

Fig. 2 Base-Plate Assembly Enclosed in Lucite Housing.

of the concentric-tube dilatometric method is described elsewhere in considerable detail.[2] A flat silica-glass plug, polished with No. 600 carborundum, is sealed into the bottom of an outer silica-glass tube, 0.3125 ± 0.001 in. I.D., and provides a flat, horizontal surface to support the specimen. Specimens ranging in diameter from 1/8 to 1/4 in., and any length between 1/4 and 2 in., can be tested. Specimens up to 1/2 in. in diameter can be tested by replacing the outer transparent silica tube with one having a larger diameter. An inner movable silica tube (push rod) extends upward from a molybdenum cap, which helps to center the specimen, to a point above the open end of the outer tube, where it makes contact with the cantilever beam of the strain-gage sensing unit and transmits movement caused by specimen expansion or contraction to the strain-gage beam (See Figs. 3 and 4). Four SR-4 (type A-7) strain gages are mounted near the root of the small cantilever beam; two of these gages are cemented on each side of the beam. As the beam bends, the strain gages on the side of the cantilever corresponding to the outer radius of bending are elongated. This stretches the wire and results in increasing the electrical resistance of the gage. The strain gages on the other side of the beam are compressed, thereby resulting in a reduction in resistance. By connecting the strain gages on opposite sides of the cantilever beam around the diamond of a Wheatstone bridge, a voltage output which is linearly proportional to the beam deflection is obtained. The variable resistance of the bridge is the X component slidewire of a Leeds and

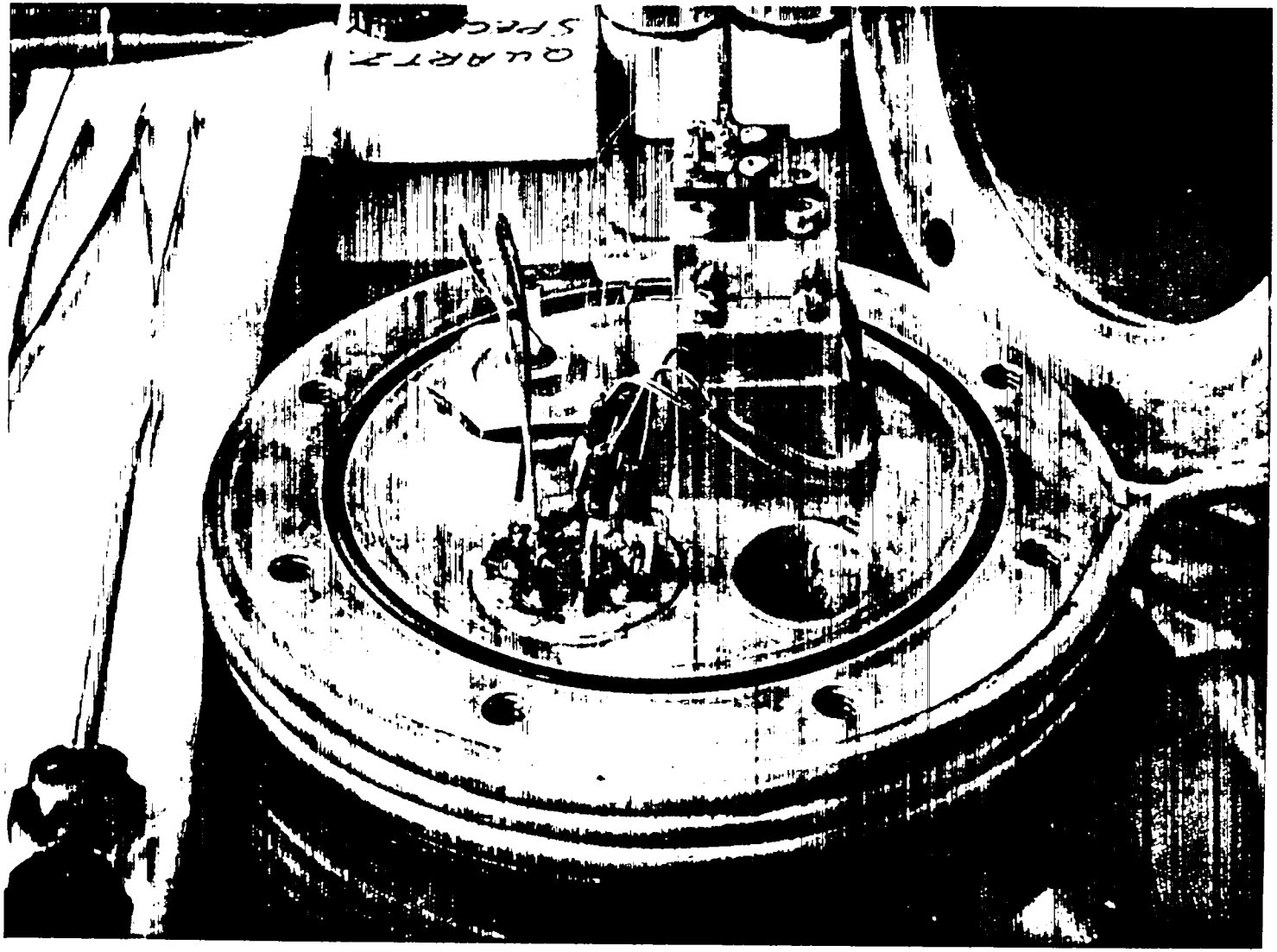

Fig. 3 Base Plate with Strain-Gage Sensing Element in Place (Side View).

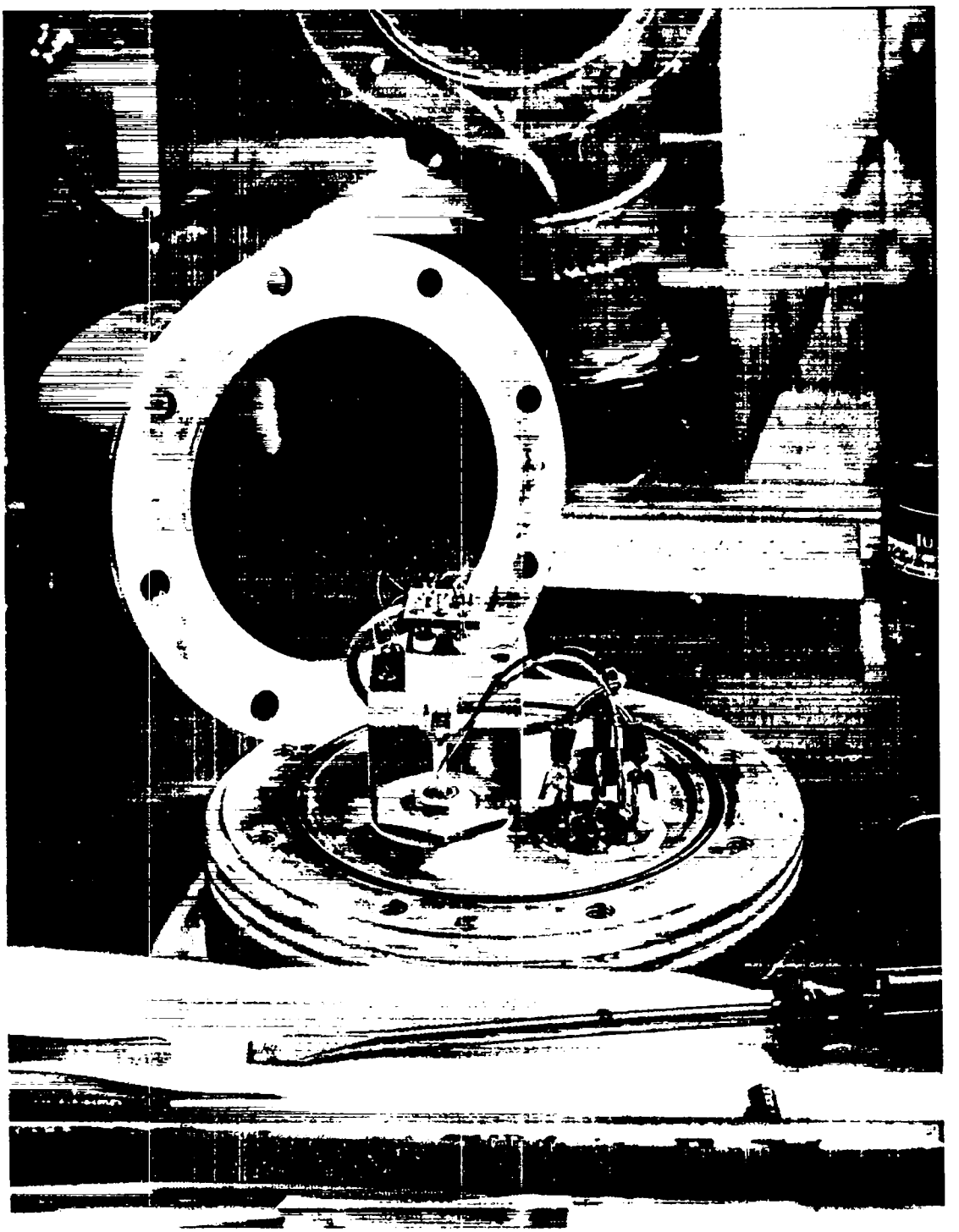

Fig. 4 Base Plate with Strain-Gage Sensing Element in Place (Front View).

Northrup Speedomax X-Y recorder. Balancing is accomplished by the mechanism of the recorder. The specimen temperature, as measured by a Chromel-Alumel thermocouple, is recorded on the Y component slidewire of the X-Y recorder.

The silica-tube assembly is supported from a circular brass plate. Hard de Khotinsky cement is used to attach the outer tube to a connecting gland which screws into the brass plate. A special semi-sliding "O"-ring seal used for joining this gland to the brass plate allows vertical positioning of the outer silica tube over a 1/2-in. range. In addition, a separate variable resistance on the bridge panel permits the instrument to be zeroed after the dilatometer has been adjusted.

Temperature Measuring System*

On top of the specimen is a molybdenum cap into which the thermocouple bead is peened. One thermocouple lead is threaded through the center of the inner tube and brought out at the top through a small groove at the upper end of the inner tube. The other thermocouple lead is taken out through a groove at the bottom of the inner tube and is threaded between the inner and outer silica tubes. These leads are brought outside the vacuum system through soft-solder connections to an

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*See item 1 of Addenda.

eight-wire Kovar seal on the circular brass plate. Chromel-Alumel thermocouple extension wires lead from the Kovar seal to the Y component slidewire of the Leeds and Northrup Speedomax X-Y recorder. The thermocouple e.m.f. is measured with reference to the melting point of ice.

Dilatometer Alignment

Precision-bore silica tubing having an inside diameter of 0.3125 ± 0.001 in. was used to make the outer dilatometer tube. A polished, flat silica-glass plug was sealed into the bottom of this tube with the flat surface normal to the principal axis of the tube. Alignment of this tube relative to the connecting gland during the cementing operation was carefully accomplished by using large V-blocks and a surface plate. The tube was cemented in place with its principal axis parallel to that of the gland.

A hole, about 0.060 in. in diameter, runs the entire length of the inner silica tube and serves to align this tube at the lower end, where it fits loosely over a centering stud on the molybdenum specimen cap. The upper end of the inner tube is self-aligned when its countersunk surface comes in contact with the small steel hemisphere on the free end of the cantilever beam of the strain-gage sensing element. The specimen cap also serves to center the specimen.

Furnace*

The function of a dilatometric furnace is to heat or cool the specimen at various predetermined constant rates while maintaining uniform temperature throughout the length of specimen. Although moderate rates of heating are generally rather easy to obtain, the corresponding rates of cooling are often difficult to obtain because of the slow rate of heat loss. Two furnace design features which tend to remedy this trouble are: (a) to include a means for conducting heat away from the furnace instead of depending only on heat losses to the atmosphere, and (b) to reduce the heat reservoir of the furnace by eliminating heavy insulation. The other consideration which must be included in both of these designs is minimization of temperature gradients along the length of the specimen.

Two furnaces have been built with identical brackets so that they can easily be interchanged in the same mounting arrangement. The first of these consists of a Marshall tensile-test furnace, 2-1/2 in. I.D. x 7 in. O.D. x 12 in. long, equipped with a nickel equalization block in the core and a water jacket in the refractory brick.[4] The heating element of this furnace is composed of Chromel wire helically wound on the outside of a grooved Alundum tube. Ten binding posts provided on a furnace shunt panel permit localized control of temperature along the length of the furnace. By adjustment of the resistance between any two

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*See item 2 of Addenda.

binding posts the temperature of the furnace located between these binding posts can be varied considerably. By this means it is possible to attain any desired uniform temperature along the length of the furnace. A water jacket, consisting of 12 turns of 1/4-in. copper tubing with a 5-in. coil diameter, is provided in the refractory brick to increase the cooling rate.

Inside the Alundum core is a 2-in.-diameter, 6-in.-long nickel block which has a 7/8-in.-diameter hole concentric with its principal axis. The size of the block is such that it establishes considerable thermal capacity around the specimen. It has been shown that the temperature distribution obtained is uniform to within 1°C along the 4-in. central region of the furnace for the range from 70° to 800°C. Two Chromel-Alumel thermocouples, peened into opposite ends of a 1-in.-long stainless steel specimen, gave temperature readings within 1°C of each other over the range from 20° to 925°C. Eight 1/4-in.-diameter holes, parallel to and spaced evenly about the central 7/8-in.-diameter hole, were drilled in the block. These holes were connected in parallel and brought to a common entrance and a common exit at the top and bottom of the block, respectively. Connection to the entrance and exit is accomplished by means of 1/4-in.-diameter nickel tubing. This system is used to conduct a stream of hydrogen or other gas to the nickel block for controlling the cooling rate.

A Wheelco proportioning Potentiotrol program controller in conjunction with a motor-driven Variac provides the means for controlling

the heating and cooling cycles of the furnace. An arm on the cam shaft can be adjusted to operate a microswitch and turn off the controller at the end of the run. The Chromel-Alumel furnace thermocouple is located midway between the Alundum core and the nickel block. Such placing of the thermocouple offers close temperature control during the heating and cooling cycles.

The second furnace to be constructed was of a noninsulated type. This consists of a Nichrome heating element, 2 in. I.D. x 8 in. long, wound internally on a fire-clay core. Surrounding the core are two concentric radiation shields constructed of 0.010-in.-thick stainless steel sheet. The end supports are made of Transite and the whole assembly is held in place by three adjustable stainless steel rods. Constant rates of heating and cooling are obtained by means of a motor-driven Variac. This furnace was designed for dilatometric work with specimens approximately 1/2 in. long.

Both furnaces, which are described above, mount into the same bracket arrangement. Three screws at 120-degree intervals are located on both the upper and lower brackets. These screws permit lateral adjustment of the furnace so that the silica-tube assembly may be positioned exactly in the center of the furnace core. The brackets slide on two guide bars. An 11-in. vertical movement of the furnace is accomplished with an ordinary scissors jack mounted beneath the lower bracket plate.

The Enclosure*

A small enclosure, constructed of 1/2-in.-thick Lucite, houses the circular brass plate. Entry into the enclosure may be made by opening doors in front or on either side. These doors when closed are tightly sealed by means of a rubber gasket. A ventilating system draws air into the enclosure when the doors are open. This housing prevents the spread of alpha-active materials in the laboratory when making adjustments or changing specimens.

Vacuum System**

The strain-gage sensing element is enclosed by a small metal can which is supported by the circular brass plate. A rubber "O"-ring gasket forms a vacuum seal between the metal can and the plate. The brass plate is connected by means of metal tubing and Neoprene vacuum hose to a small VMF-20 vacuum diffusion pump and a mechanical backing pump. Vacua of the order of 5×10^{-5} mm Hg, as measured by a Philips ion gage, can easily be maintained with this system. It is possible to close the pump shut-off valve and introduce any desired gas into the system at known pressures.

EXPERIMENTAL PROCEDURE

The first step, after having decided to make a dilatometric analysis on a plutonium or plutonium-alloy sample, is to vacuum cast

*See item 3 of Addenda.
**See item 4 of Addenda.

a cylindrical ingot of the required composition. This ingot is then machined to its final cylindrical dimensions. The thermocouple is peened into the molybdenum specimen cap and one lead wire is threaded through the center of the inner tube. The specimen is gently slid to the bottom of the outer tube, followed by the specimen cap and inner tube. The silica-tube assembly is then threaded into the circular brass plate and adjusted for vertical position. A large lock nut on the connecting gland securely fastens the silica-tube assembly after it has been adjusted. The strain-gage sensing element is then placed in operating position, so that the recessed end of the inner tube comes in contact with the hemisphere on the free end of the cantilever beam, and is securely bolted to the brass plate. After tapping the apparatus to set the specimen in a stable position, the recorder is turned on and zeroed with the zero adjust knob on the panel. The thermocouple leads are then soft-soldered to their proper terminals.* The metal can is bolted to the circular brass plate and the system is evacuated.

The recording dilatometer is now ready for use. The program controller is set for the predetermined rates of heating and cooling, and the arm on the cam shaft is adjusted to control at maximum temperature. Uniform rates of heating and cooling on the order of 1° or 2°C/min have been frequently used for phase transformation studies on plutonium alloys. Most of the program cams have been cut for the 0°

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*See item 1 of Addenda.

to 1000°C temperature range, and if the cooling cycle is to start before 1000°C is reached, manual switching is necessary. The furnace power and Variac drive are then turned on and no further attention is necessary except to start the cooling cycle if required. An accurate, continuous record is automatically plotted. The dilatometer can instantaneously follow extremely fast changes in expansion or contraction and reversals in slope, no matter how rapidly they may occur.

It was found that the apparatus did not require tapping during operation. The dilatometer will follow the expansion on heating and return to the original starting point on cooling with no tendency toward sticking. In a similar dilatometer, in which expansion was measured with a dial indicator, it was found that tapping was necessary before each reading on both heating and cooling cycles.

DISCUSSION

The dilatometer was checked in operating details by making practice runs with specimens of silica glass, aluminum, silver, and uranium. During these runs temperature checks and calibrations were made, and refinements were incorporated into the construction and instrumentation of the dilatometer. The data obtained on the various materials compared satisfactorily with published results. The sensitivity to dimensional changes of less than 0.0001 in. has proved most useful for determining the temperature at which a phase transformation begins. In addition, the specimen load has been greatly reduced.

Data obtained from runs made on a 1/4-in.-O.D. plutonium specimen did not indicate any plastic deformation even when the specimen was held overnight in the epsilon range at 550°C.

The apparatus has been successfully used for studying solid-state transformations in the plutonium-uranium alloy system. Data obtained were in good agreement with the results from X-ray and metallographic methods. Because of the sluggish rates of reaction of some of the transformations, relatively slow rates (about 1°C/min) of heating and cooling were used. A dilatometric record of a plutonium-uranium alloy containing 7 atomic per cent uranium, plotted while heating at 1.5°C/min, is shown in Fig. 5. The anomalous expansion which accompanies changes in crystal structure and the temperatures at which these changes occur are legibly and accurately recorded.*

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*See item 5 of Addenda.

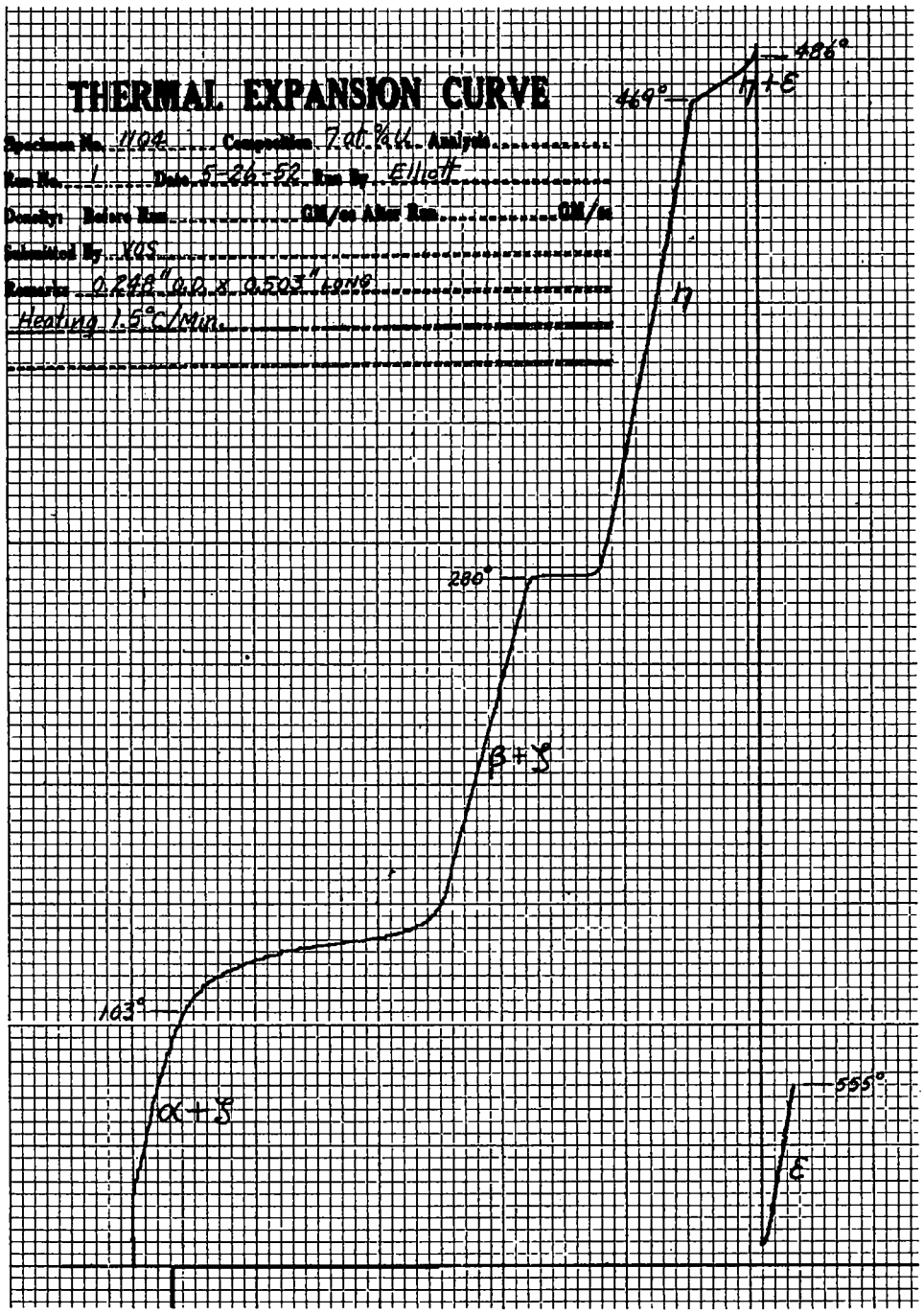

Fig. 5 Thermal Expansion and Transformations of a Plutonium Alloy Containing 7 a/o Uranium.

ADDENDA*

Since this report was written, a few modifications of the equipment and procedure have been made in order to obtain greater ease of operation of the dilatometer for routine tests. Throughout the report asterisks indicate the topics to which the modifications apply, and the following comments concerning such modifications are in the same order as the topics in the report.

1. The temperature measuring system has been modified slightly in that both thermocouple wires are now brought from the molybdenum cap (resting on the specimen) up through the longitudinal hole in the center of the silica push tube. One of the two thermocouple wires is encased in flexible silica sleeving to insulate it electrically from the other wire. Chromel and Alumel wires are brought into the dilatometer through glands in the brass base plate, and connections between these wires and the thermocouple wires are made by means of a standard Chromel-Alumel bayonet-type plug and receptacle. This arrangement allows easy replacement of push tubes to accommodate specimens of different lengths.

2. The Marshall tensile-test furnace originally used with the dilatometer provided for temperature uniformity along the length of the furnace. This furnace, however, required considerable headroom and also some raising and lowering mechanism in order to expose the dilatometer

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*August 15, 1957.

tube during specimen loading and unloading. At present, a split-tube furnace has replaced the Marshall furnace. During loading operations this hinged furnace may be opened, and visual examination of the specimen and push tube within the dilatometer tube may be made easily and quickly. If the specimen is placed near the center of the furnace and if the bottom of the furnace and its top, except for a hole to accommodate the dilatometer tube, are closed with Transite plates and asbestos plugs, any vertical temperature gradient in 1/2- to 1-in.-long specimens appears to be negligible in routine tests.

3. The enclosure has been replaced by a standard stainless steel furnace housing. This housing is sufficiently large to accommodate two dilatometers. Facilities such as circulating water, electrical power leads, thermocouple and strain-gage leads, and the vacuum and inert-gas lines are brought into the housing through glands or seals in the side of the housing. Access to the dilatometers is provided through a large Lucite door in the front of the housing. Use of this type of housing makes it possible to enclose the entire dilatometer and thus prevents any spread of alpha-active material should a dilatometer tube fail during operation.

4. Instead of the metal can which was first used to provide a vacuum chamber for the top of the dilatometer, a glass bell jar has been substituted. Because routine tests are made in vacuum, or in some few instances in an atmosphere of helium at pressure less than atmospheric, it has not been considered necessary to provide hold-down straps

for the bell jar. Use of the glass bell jar allows the operator to observe directly the top portion of the dilatometer during a test.

5. It is worth mentioning that this autographic strain-gage dilatometer, essentially the same as that described, has now been in use for several years. During that time hundreds of tests of plutonium and plutonium-alloy specimens have been made and consistently good results obtained.