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MASTER

AN AUTOMATIC RECORDING,
LOW-TEMPERATURE DILATOMETER

by

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RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

THE DOW CHEMICAL COMPANY
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GOLDEN, COLORADO 80402
U. S. ATOMIC ENERGY COMMISSION
CONTRACT AT(29-1)-1106

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ABSTRACT

Because many metal alloy phase transformations occur below room temperature, a low-temperature dilatometer is quite useful in the determination of phase diagrams. A dilatometer, having the temperature range of 50°K to 500°K, is described in this report.

INTRODUCTION

The dilatometer described in this report is simply constructed and easy to operate. Temperature is recorded directly in millivolts on the X axis of a direct-reading, X-Y, fixed-chart recorder while the change in length is simultaneously recorded on the Y axis. The result is a complete graph of the temperature and dilatation, which needs only the translation of the recorded voltage readings to useful values of length and temperature.

Control of the cooling cycle can be easily maintained at any rate (within desirable limits) between $0.5^{\circ}\text{K}/\text{min.}$ and $110^{\circ}\text{K}/\text{min.}$ Similar control of the heating cycle can be attained by conventional Variac control of the furnace element.

CONSTRUCTION

A Sanborn transducer control (Model No. 7DCDT050) is the heart of the dilatation sensing portion of this dilatometer. The transducer utilizes a differential transformer with coupled oscillator and demodulator to provide a d-c output from a d-c input. The manufacturer claims a linear output variation of less than 5 percent of total stroke range, which is 0.050 in. on this model. The transducer requires only a six volt dry cell battery for the input voltage. Calibration of the transducer is accomplished simply by mounting it in a calibration block as illustrated in Figure 1. The micrometer head, which reads to 0.0001 in., shows the linear movement of the core as the output voltage of the transducer varies.

Figure 1. TRANSDUCER CALIBRATION DEVICE

The transducer is easily zeroed to varying specimen lengths. A spring loaded, concentric mount is so constructed that rotation of the transducer body is prevented and fine control of vertical movement is achieved (Figure 2). The concentric mount consists of a cylinder which is threaded on the outside to accept a threaded cap. The top of the cap is bored so that the leads from the transducer can pass through, but the transducer body is held in a concentric position with a counter sunk ridge. A thin bearing of Teflon mounted against the ridge allows the cap to turn on its threads without rotating the transducer body, which is held against the bearing with a coil spring. As the cap is screwed on or off, the transducer body is positioned with relation to the core so that zero voltage output is attained.

Figure 2. TRANSDUCER, MOUNT, CORE TRANSMISSION TUBE, AND SPECIMEN CHAMBER

Figure 3. SCHEMATIC OF TRANSMISSION TUBE ASSEMBLY

The transducer core is connected to the top of a 4-mm diameter quartz transmission tube with a small brass cap (Figure 3). Another brass cap locates the transmission tube on top of the cylindrical specimen. Dilatation of the specimen is transmitted directly to the transducer core by the quartz transmission tube and read as a corresponding change in the voltage output of the transducer.

Temperature measurements are taken with a copper-constantan thermocouple which contacts the specimen through a hole in the specimen cap. The thermocouple runs through the transmission tube and exits through an angular hole in the brass cap which connects the transmission tube to the transducer core. A wide slit in the brass coupling which fixes the transducer mount to the main brass fixture plate provides a non-restraining exit for the thermocouple. This assures free movement of the transmission tube assembly.

The specimen chamber is a 9-mm diameter quartz tube with a flat, quartz plug fused to the bottom for the specimen to rest upon (Figure 2). The top of the quartz specimen chamber is cemented with de Khotinsky cement to a threaded, brass fitting. This fitting screws into the main, brass fixture plate. A silicon rubber gasket provides a seal between the fixture plate and the specimen chamber.

A bell jar fits over the transducer mount and forms a chamber with an "O" ring seal to the brass plate (Figure 4). An inlet through the brass plate into the bell jar chamber accommodates the use of vacuum and inert gas in both the bell jar and specimen chambers. The transducer and thermocouple leads leave the bell jar chamber through the fixture plate by means of a Kovar seal.

The thermocouple and transducer leads connect to a Moseley Model 2D-2 X-Y recorder. The thermocouple signal is transmitted through a 273°K reference junction before entering the X portion of the recorder. The transducer signal is recorded on the Y axis.

Two Dewars are utilized to provide temperature control of the specimen (Figure 4). The inner Dewar is made with an unsilvered window on each side to aid in checking alignment of the specimen and transmission tube. Provisions are made so that the space between the walls of the inner Dewar can be either evacuated or back-filled with air or other gas to give control of the specimen cooling rate within desirable limits. The specimen chamber passes through a rubber stopper which seals the inner Dewar chamber from the atmosphere. A connection is made to the Dewar chamber for both vacuum and purge and allows evacuation of the space above the liquid nitrogen in the Dewar, effectively lowering the boiling point of the liquid nitrogen. Ball check valves on the nitrogen inlet port and in the vacuum outlet relieve any "bumping" pressure which might occur while pumping on the liquid nitrogen. The outer Dewar is used for obtaining slower cooling rates. In this instance the back-fill features of the valving manifold provide the use of various gases between the walls and in chamber of the inner Dewar. The desired cooling rates are achieved by using differences in the heat conducting properties of various gases.

A valving manifold connects a mechanical vacuum pump to the bell jar chamber, the inner Dewar chamber, and the space between the inner Dewar walls. The same manifold system is used to control the addition of inert gas to the specimen chamber as well as to the Dewar walls and chamber.

Elevated temperatures are obtained by removing the Dewars and using a small resistance furnace in their place.

Figure 4. SCHEMATIC DIAGRAM OF THE LOW-TEMPERATURE DILATOMETER

OPERATING PROCEDURE

The dilatometer is loaded with a machined, cylindrical specimen which is 0.250-in. diameter by approximately 1-in. long. The specimen is gripped with a special loading device which consists of a thin, slit collet on the end of a long piece of 1/8-in. tubing. A rod is pushed through the center of the tubing to dislodge the specimen in the specimen chamber. The transmission tube with connected specimen cap, thermocouple, and transducer core is positioned in the specimen chamber with the cap over the specimen. The unsilvered window slits, on the inner Dewar, aid in visual inspection of the loading process. The transducer body is carefully positioned over the core and the cap screwed into place to zero the transducer. The bell jar is placed in position and a vacuum is drawn on the specimen and bell jar chambers. A push button switch is used to short the thermocouple leads and a zero signal reference mark is made on the recorder chart (0°C).

Liquid nitrogen is used as the refrigerant in cooling the specimens. The outer Dewar is filled first when slower cooling rates are desired. Heat transfer is stepped up by evacuating the inner Dewar walls and chamber and back-filling them with helium which conducts the heat away from the specimen. The inner Dewar is filled with liquid nitrogen when the cooling rate has become too slow. When the specimen temperature reaches that of the nitrogen (75°K at this altitude) further cooling is accomplished by pumping the vapor until the liquid freezes (-50°K at altitudes in excess of 6000 feet above sea level).

Temperatures to 500°K can be reached simply by replacing the Dewars with a resistance furnace. Higher temperatures would require the use of an iron-constantan or chromel-alumel thermocouple and a different material for the specimen cap such as quartz or tantalum. Of course, the vacuum system would need to be of sufficient efficiency to prevent oxidation of the specimen.

A dilatometric trace of oxygen-free high conductivity (OFHC) copper is shown in Figure 5; the temperature range is 296°K to 50°K. The data points are taken from an actual run using the dilatometer described in this paper. (The actual data sheets are too large for enclosure.)

$$\text{CONTRACTION} = 3.667 \times 10^{-3} \text{ in } \frac{\Delta L}{L_0} = \frac{3.667 \times 10^{-3}}{1.0142} = 3.616 \times 10^{-3}$$

$$\left(\frac{\text{CONTRACTION}}{\times 10^3 \text{ in}} \right) \frac{\Delta L}{L_0} \frac{1}{\Delta T} = \frac{3.616 \times 10^{-3}}{233} = 15.5 \times 10^{-6} \% / ^\circ \text{C}$$

Figure 5. DILATOMETRIC TRACE OF OXYGEN-FREE, HIGH-CONDUCTIVITY COPPER

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