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A High Speed Dilatometer and the Transformational Behavior of Six Steels

(BuShips Welding Test No. 329)

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ABSTRACT

This report describes a high speed dilatometer capable of measuring transformations in steel with cooling rates up to 500°C per second. The transformational behavior of six commercial steels is presented; two of these steels were high tensile steels of the manganese-titanium treated type of almost identical chemical composition. A careful study of the weldability of these two steels indicated that one of the steels was more weldable than the other; the transformational diagrams obtained indicate that the most weldable steel requires more drastic cooling in order to obtain a martensitic structure. Additional information regarding the transformational characteristics of steels of this type will be highly desirable in the evaluation of the effect of minor constituents on any such property as weldability.

INTRODUCTION

AUTHORIZATION

1. This problem was authorized by BuShips ltr. QP/W&C-(4)-(2)(334-692) of 4 April 1944 (BuShips Welding Test No. 329).

STATEMENT OF PROBLEM

2. In studies of weld and under bead cracking, the feasibility of matching the transformations of weld and base metals as to temperature and magnitude and the possibility of establishing a definite relationship between these transformations and weld cracking have been suggested (See NRL Report No. M-2277 of 15 April 1944). Attempts have been made to use the information obtained from standard dilatometric equipment which determine transformation temperatures at relatively slow cooling rates. However, in order to observe the behavior of steels and weld deposits at cooling rates obtained in actual welding the use of high speed equipment is essential.
3. Much information regarding the transformational characteristics of a steel as it cools in a known manner from the austenitic state can be obtained from dilatometric studies. This method has received more and more attention because of its great flexibility, accuracy and ease of interpretation. The method is entirely applicable to any rate of heating and cooling although the experimental procedures for high rates of cooling have offered some difficulties. This report presents a description of equipment employing cooling rates up to 500°C per second, far beyond those encountered in welding; the results of dilatometric observations on six commercial steels are included.

KNOWN FACTS RELATED TO THE PROBLEM

4. French and Klopsch (1) studied the cooling transformations of a series of carbon steels using the thermal arrest method. This was followed by the systematic investigation of Esser and co-workers on the effect of carbon (2) and other alloying additions (3) in which the transformation temperatures were determined by magnetic induction measurements indicated by a string galvanometer. Emphasis was placed on determining Ar' transformations and the critical cooling rates for these steels. The magnetic measurements were used in an effort to obtain more accurate data at higher cooling rates. Thermal arrest measurements were also made to check the validity of the magnetic indications and good correlation was found between the two, although Esser's Ar" temperature measurements on iron carbon alloys were about 200°F lower than the measurements of French and Klopsch. A notable contribution of Esser was the introduction of gas quenching.

5. Wever and Rose (4) used a moving coil galvanometer in the study of the Ar' and Ar" of iron carbon alloys by the thermal arrest method and obtained somewhat lower Ar" temperatures than

those observed by Esser below 1 per cent carbon and considerably higher above. They concluded that the cooling rate had little influence on the Arⁿ temperatures above 0.4 per cent carbon although Esser had observed a decrease in the Arⁿ temperature with increasing cooling rate with similar steels.

6. Digges (5,6,7) determined the Arⁿ temperature for a series of high purity iron carbon alloys and close agreement with his results was obtained by Greninger (8). The thermal arrest method together with metallographic observation used in both these studies represents the latest investigations of this type. Any investigation by thermal analyses is dependent on the heat of reaction and the specific heat of the reaction products. In many instances this method does not yield satisfactory data. It is not only desirable to measure the temperature ranges during which the austenite decomposition takes place when cooled in a known manner but also to obtain a good indication of the amount of austenite that has transformed. Any investigation of cooling transformation behavior by magnetic induction measurements is necessarily dependent on the magnetic properties of the transformation products and such extraneous effects on magnetic induction as may be produced by stresses set up during the transformation. Neither the magnetic nor thermal arrest method lends itself readily to obtaining quantitative information regarding transformational behavior on cooling.

7. While studies have been made on a variety of steels, data are yet lacking for construction of complete diagrams of transformations on cooling. Grange and Kiefer (9) pointed out the experimental difficulties to be encountered in the study of the transformations of any but the most sluggish alloys and suggested that cooling diagrams be derived from isothermal data and presented an empirical method correlating transformational behavior on cooling with isothermal transformation behavior. Metallographic studies were made of specimens quenched after cooling for various predetermined times and good agreement between the estimated and the experimentally observed cooling behavior was found. The interrupted cooling method of Grange and Kiefer makes possible very satisfactory observation of the cooling behavior of sluggish steels but is not suited to the investigation of faster reacting steels.

8. After a careful survey of the literature it was felt that the most promising method of attack was to use a dilatometer capable of following the transformation of a small steel specimen as it cools rapidly from the austenitic state. With suitable dilatometric equipment it will be possible to obtain the information required for the construction of a diagram indicating transformations on continuous cooling. If the temperature ranges in which the various transformation products are formed can be determined for a series of different cooling rates, the problem of interpreting the microstructures will be very much simplified. The dilatometric method is of particular advantage in the field of welding where the actual dilation characteristics are important

in the study of transformation stresses and their effect on cracking tendencies. The present equipment is suitable for investigating the transformational behavior of a steel over a wide range of cooling rates. In this report, the transformations on continuous cooling are referred to as transformational behavior and the resulting diagrams are designated as transformational diagrams.

DESCRIPTION OF EQUIPMENT AND PROCEDURE

9. The necessity of indicating and recording the three variables; time, temperature, and dilation (volume change), over a wide range of cooling rates demands an instrument possessing very low inertia and good flexibility. The cathode ray oscillograph (10) fulfills these requirements ideally. By using the vertical deflection of the cathode ray beam as an indication of volume change, the horizontal deflection as an indication of temperature, and extinguishing the beam at suitable time intervals, a simultaneous plot of the three variables is possible. This plot can be recorded photographically.

10. From a functional standpoint, the equipment is best considered in four parts:

1. The mechanical arrangement of specimen, furnace and cooling device.
2. The measurement of dilation.
3. The measurement of temperature.
4. The measurement of time.

11. The mechanical construction of the dilatometer is similar to many of those now in use (Plate 1). A quartz rod with a quartz cap is inserted through a tubular steel specimen of approximately $3/8$ " inside diameter, $7/10$ " in length and $1/32$ " wall thickness with the cap in contact with the upper end of the specimen. A quartz tube is then slipped over the quartz rod until the upper end of the tube is in contact with the lower end of the specimen. Positive contact between the quartz and the specimen is assured by means of a small spring. Any volume change of the steel thus causes a relative displacement between the quartz tube and the quartz rod. The quartz tube is supported in a fixed position and the quartz rod is attached to the movable element of an electrical micrometer tube. Any expansion or contraction of the metal specimen thus produces a proportional displacement of the movable element of the tube.

12. The electrical micrometer tube (11) consists of an evacuated glass bulb (1), (see Plate 2), enclosed by a metal shell (2), containing a hot electron emissive filament (3), and two insulated plates (4,5) mounted on a tube arm (6) which extends outside of the tube. The tube arm is carried by an elastic metal diaphragm (7) sealed to the glass envelope. By this constructio

the plates may be moved with respect to the filament from outside the tube. Two fixed resistances and an adjustable resistance complete a bridge circuit with the plate resistance of the tube (see Plate 3). Any movement of the tube arm moves one plate closer to the filament and the other farther from the filament and changes the electrical balance of the bridge and causes a voltage to develop across the bridge. Within the limits specified for the tube (approximately 0.012" from the point of zero displacement) the voltage developed across the bridge is directly proportional to the displacement of the tube arm. This allowable displacement is within the limits of expansion of a one-inch specimen heated from room temperature to 1600°F.

13. A gas quenching fixture is mounted concentrically with the specimen and allowed to move freely in the axial direction so that it may be moved down away from the specimen while the specimen is being heated and moved up around the specimen when it is desired to cool the specimen. Rubber tubing is used to provide a flexible coupling between the quenching fixture and the pressure regulator of the supply tank. The pressure adjustment of the tank regulator establishes the severity of quench. Helium gas is used as the cooling medium, although other gases or mixture of gases such as hydrogen or nitrogen-hydrogen mixture may be used satisfactorily.

14. The specimen and quenching fixture assembly is placed within a protecting quartz cylinder through which a small flow of helium is introduced from the top to maintain an inert atmosphere. A resistance type heating coil is suspended around the outer quartz cylinder and mechanically arranged so that it may be raised away from the specimen when the specimen is being cooled. A photograph of the dilatometer assembly is shown in Plate 4.

15. The output of the micrometer bridge is about 1.5 volts and is amplified to approximately 200 volts by means of a direct coupled single stage push-pull amplifier to give full scale deflection of the cathode ray beam. The 5" DuMont cathode ray oscillograph, type 208, which a deflection sensitivity of approximately 50 volts per inch is used with the output of the amplifier directly coupled to the vertical deflector plates of the cathode ray tube. By operating the amplifier tubes on the linear portion of their characteristics curves the deflection of the cathode ray beam will be to all practical purposes directly proportional to the dilation of the specimen. The adjustable resistance of the micrometer bridge provides a means of vertical positioning of the cathode ray beam.

16. The temperature of the specimen is measured by means of a No. 28 B & S gage chromel-alumel thermocouple spot welded to its surface. The output of the thermocouple is about 40 millivolts at 1700°F. In order to make possible a more accurate temperature measurement on the oscillograph, the temperature

scale has been doubled by using the full viewing screen width to indicate the temperature range from 0 to 20 millivolts. At 20 millivolts, the trace then reverses in direction to indicate the temperature range from 20 to 40 millivolts. To do this it is necessary that the 20 millivolts be amplified to approximately 200 volts to give the full scale deflection on the screen. This would not be difficult if a resistance coupled amplifier could be satisfactorily used. However, the drift inherent in resistance coupled amplifiers demanded constant recalibration of the temperature scale and for this reason was unsatisfactory. In order to use a resistance-capacitance coupled amplifier the thermocouple current is interrupted by an electrical vibrator having make-and-break contacts that operate at a repetition rate of 500 cycles*. This supplies a square wave signal, the amplitude of which is alternately zero and the full thermocouple voltage; this can be amplified by a standard two stage resistance-capacitance coupled amplifier. The amplified signal is then full wave rectified, filtered free of undesired ripple and applied to the horizontal deflector plates of the cathode ray tube.

17. A bucking potential of 20 millivolts is put in series with the thermocouple with the result that as the specimen is heated the potential applied to the amplifier starts at minus 20 millivolts, gradually becomes zero, and then assumes a positive value as the thermocouple voltage rises above 20 millivolts. The reversal of the trace occurs when the potential is zero. Adjustment of the bucking potential can be used to compensate for the cold junction temperature. Satisfactory horizontal positioning of the cathode ray beam is obtained by placing two 45 volt dry cells in series with one of the leads from the amplifier to the horizontal deflector plates.

18. The viewing screen of the cathode ray tube is provided with a transparent face cover upon which uniform grid lines have been ruled. The grid lines have been calibrated with respect to temperature and a calibration curve is shown in Plate 5. No calibration was made of the electrical micrometer since it is only necessary to know the relative dilation, although, a calibration could easily be made by means of a micrometer screw.

19. An indication of the time rate at which the temperature and volume changes of the steel are taking place is obtained by extinguishing the beam which traces the pattern at pre-established time intervals. The oscillograph is provided with a beam switch which operates to increase the modulating-electrode to cathode bias to beyond cutoff. By replacing the switch with a small magnetic relay and exciting the relay by means of a metronome in series with a dry cell, the beam can be momentarily extinguished at rates from every 0.3 second to every 3.0 seconds

* A converter tube from a Brown potentiometer pyrometer although designed for 60 cycle operation gave fair operation on 500 cycles. A change in the circuit so that the vibrator could be operated at 60 cycles would be desirable.

depending upon the adjustment of the metronome. Slower cooling rates are timed by manual operation of the beam switch but can be done electrically if desired. It is important that the relay be carefully shielded to prevent disturbance of the cathode ray beam by the magnetic field of the relay.

20. A block diagram of the dilatometer assembly is shown in Plate 6. Examples of photographic records are shown in Plate 7.

RESULTS OF TESTS

21. It has been found desirable first to obtain a continuous record of the transformational characteristics of a sample and then to follow this with a time interrupted record. This procedure has been necessary in order to adjust the cooling rate as well as to provide an accurate interpretation of the transformational behavior. A replot of the observations can be made from the photographic records giving a family of curves similar to that shown for SAE 4130 steel (Plate 8). From the time-temperature cooling curves plotted from the photographic record, the transformation diagram (Plate 9) may be constructed. The interpretation of these curves will be assisted by supplementing the dilatometric study with microscopic observations and hardness measurements. Details, especially in the complex "split transformation" range will be clarified by skillful interpretation of the dilatometric observations. It is felt that the present equipment is a tool which will enable the metallurgist to obtain direct fundamental data regarding the transformational behavior which up to this time has been available only through cumbersome indirect methods which could be skillful and experienced workers in the field.

22. The cooling curves show a diminishing cooling rate as the temperature is lowered. This may be objectionable for some fundamental studies although it more nearly approaches cooling conditions in actual practice. No difficulty was encountered in obtaining the desired range in cooling velocity by means of the gas quench although care had to be taken to prevent decarburization of the thin specimens from the oxygen present as an impurity in the quenching gas.

23. Of practical interest in connection with the transformations on continuous cooling are the cooling curves for normalized sheet steel ranging in thickness from 0.020 to 0.250 inches. The time to cool from 1600°F to 400°F in these tests ranged from 60 to 600 seconds. The preliminary observation of the transformation of this steel (Plate 9) would indicate a more or less similar type of transformation for the whole range of thickness in the continuous air cooled or normalized condition.

24. The information obtained from a study of a sample of NE 8630 (Plate 10) indicates that the transformational characteristics are much more complicated than those observed for the

SAE 4130 type of steel. The completion of the austenite to bainite transformation is not clearly defined. Here again nearly isothermal transformations are encountered just preceding the martensitic type of reaction. The opportunity of observing the finish of the martensitic reaction as well as the start of this reaction will be useful in practical heat treatment.

25. A study of a highly alloyed steel such as SAE 4340 (Plate 11) indicates a similarity between the transformational diagram and the isothermal diagram presented by Davenport (12). In the present studies, however, the start and finish of the martensitic reaction can be determined. The constancy of the start and finish of the martensitic reaction is to be noted.

26. The transformational diagrams were determined for two samples of manganese-titanium treated high tensile steels whose weldability was being studied. From these diagrams (Plates 12 and 13) it is shown that steel 386 has a higher hardenability than steel 384. That such is the case is supported by the fact that the ductility of the heat affected zone under bead welds was lower for steel 386 than for steel 384. The maximum Vickers hardness in the heat affected zone was 322 for steel 386 compared with 277 for steel 384. The hardenability for steel 386 was slightly higher than steel 384 as measured by the end quench hardenability test. The chemical compositions of the two steels, however, were almost identical. Additional information regarding the transformational characteristics of steels of this type will be highly desirable in the evaluation of the effect of minor constituents on any such property as weldability.

27. The transformational diagram presented in Plate 14 for an SAE 1025 steel illustrates the range of measurements made possible by the use of the present equipment. The time ordinate has been expanded as the information given is that which occurs within two seconds after quenching.

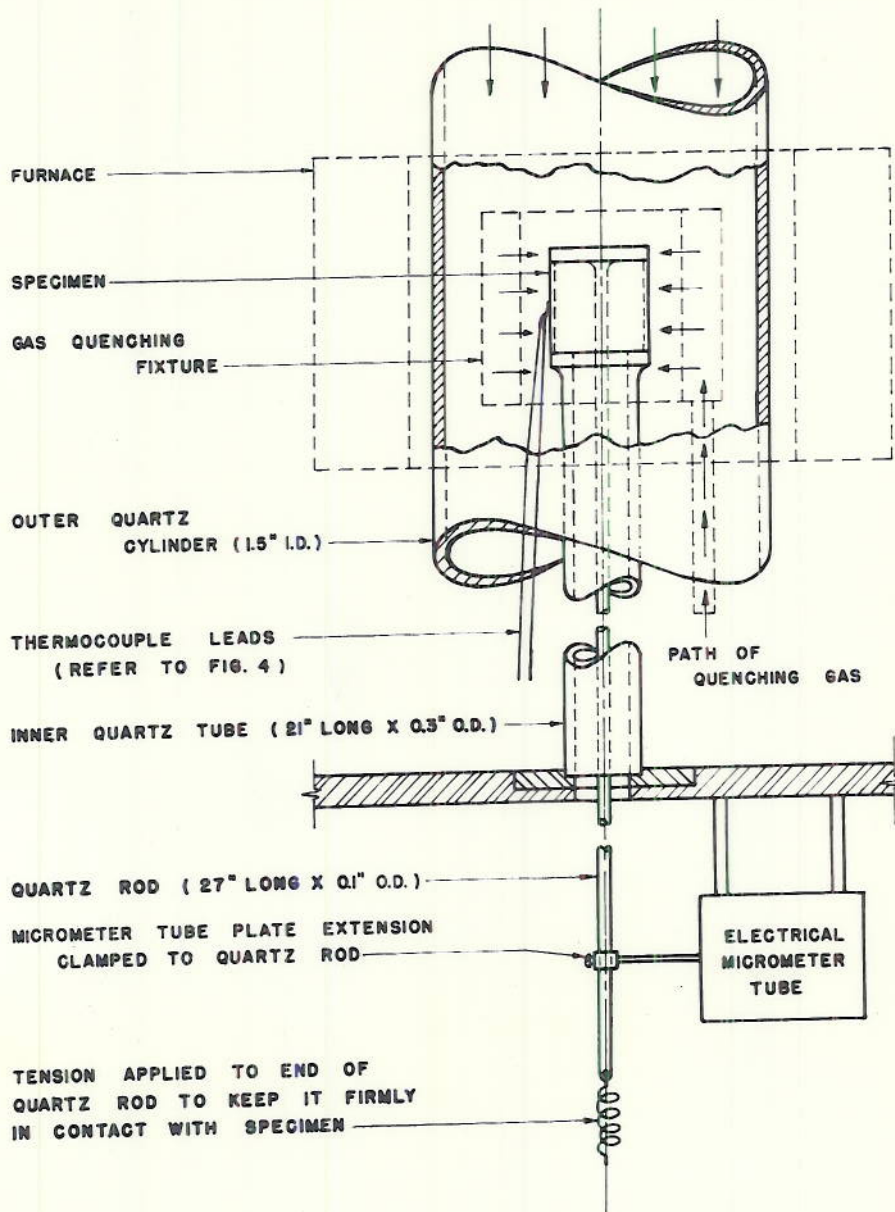
SUMMARY AND CONCLUSION

28. A high speed dilatometer has been described which has sufficient range and accuracy so that it will be useful in many metallurgical problems which depend upon continuous cooling behavior. A limited number of observations are presented showing the wide range and flexibility of the method. It is hoped that such measurements will prove directly applicable in a fundamental interpretation of the behavior of steels as affected by the process of welding. The information may be further useful in other metallurgical studies of quenching stresses and properties of structures which undergo non-uniform cooling.

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GAS FLOW FOR MAINTAINING A
NON-OXIDIZING ATMOSPHERE

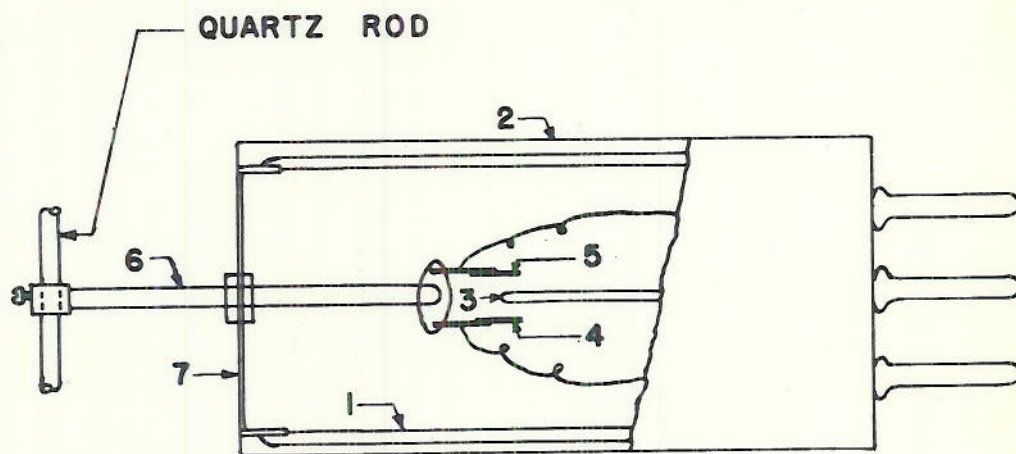


FURNACE AND QUENCHING FIXTURE MAY BE RAISED AND LOWERED INDEPENDENTLY OF EACH OTHER OR THEY MAY BE RAISED AND LOWERED SIMULTANEOUSLY.

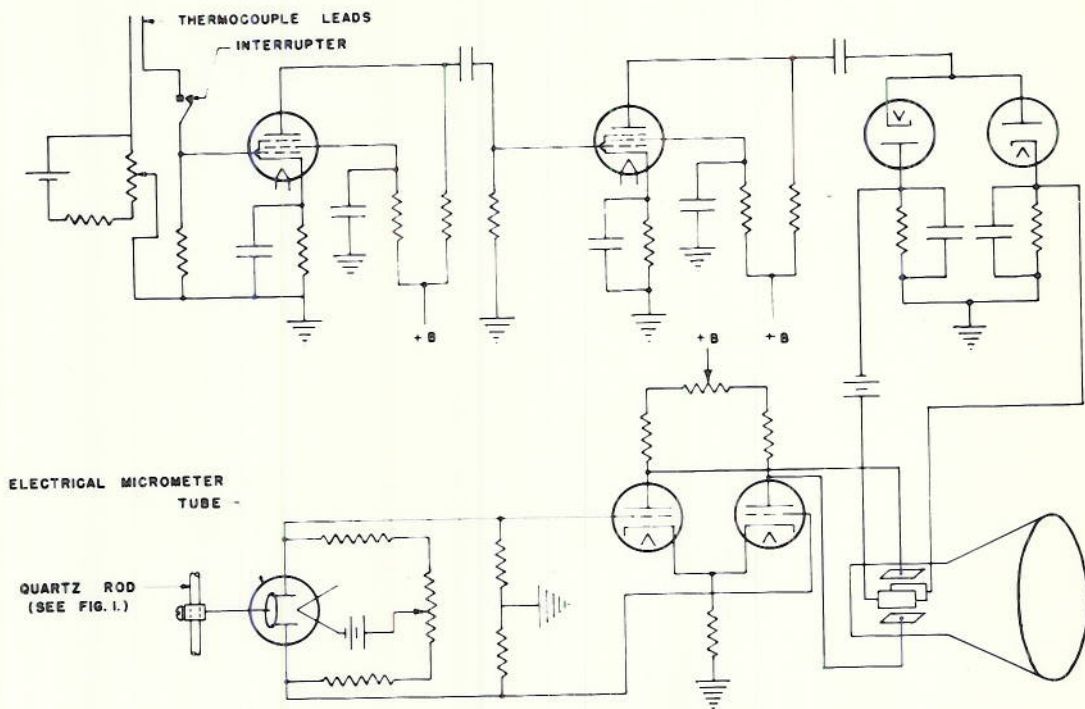
WHEN THE FURNACE IS LOWERED AND IS HEATING THE SPECIMEN, THE QUENCHING FIXTURE IS LOWERED.

WHEN THE SPECIMEN IS BEING QUENCHED, THE FURNACE IS RAISED AND THE QUENCHING FIXTURE IS IN THE POSITION SHOWN.

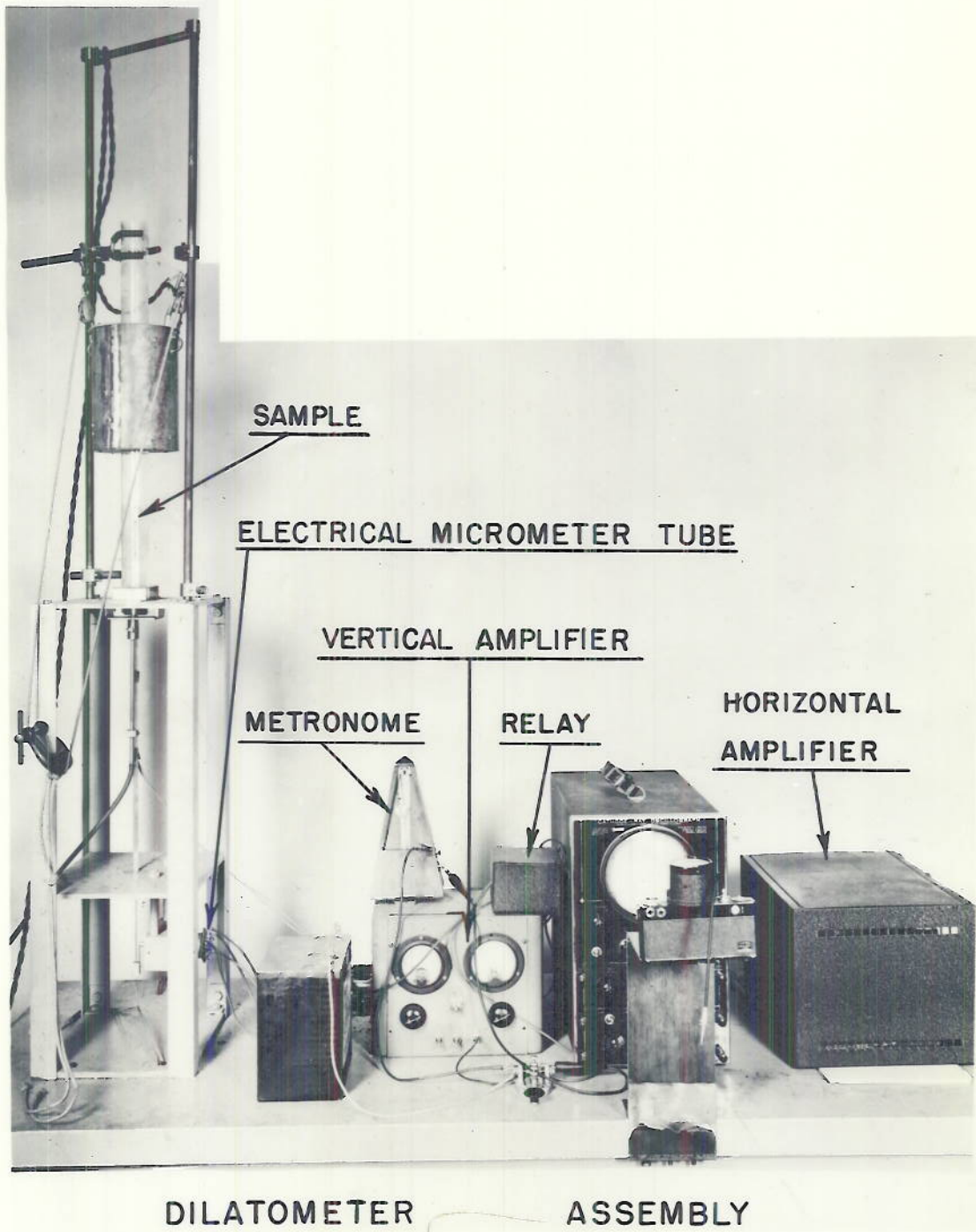
SPECIMEN ASSEMBLY



ELECTRICAL MICROMETER TUBE



CIRCUIT DIAGRAM FOR TEMPERATURE AND VOLUME MEASUREMENT



SAMPLE

ELECTRICAL MICROMETER TUBE

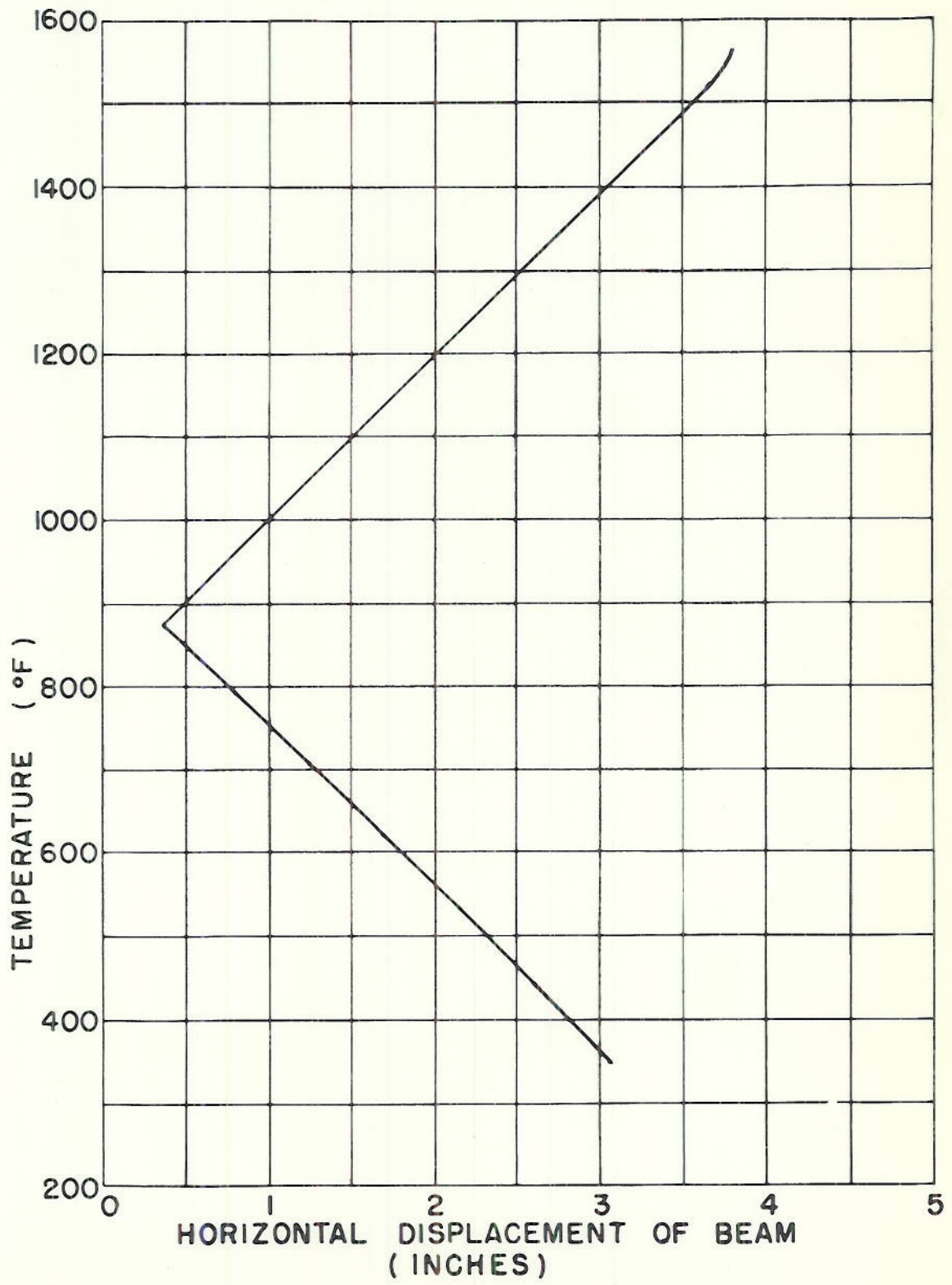
VERTICAL AMPLIFIER

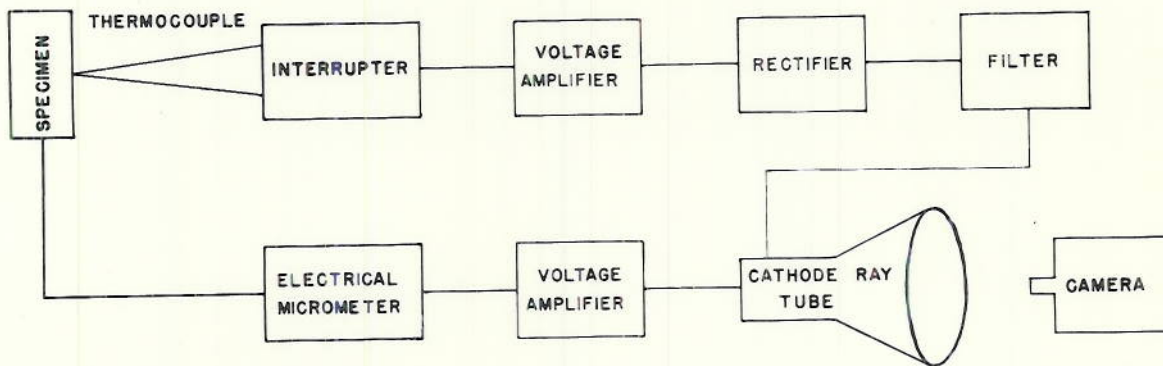
METRONOME

RELAY

HORIZONTAL AMPLIFIER

DILATOMETER ASSEMBLY





SCHEMATIC ARRANGEMENT OF DILATOMETER COMPONENTS

