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THERMOCOUPLE DESIGN AND TEST PROGRAM FOR REACTOR PROJECTS

J. T. DeLorenzo

OAK RIDGE NATIONAL LABORATORY
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UNION CARBIDE CORPORATION
for the
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ABSTRACT

Tests were made to determine the stability of swaged, 0.250 inch O.D. Inconel Sheathed, Chromel P-Alumel thermocouple material with MgO insulation in the range 1100 to 1800°F under conditions where the temperatures were static, slowly cycled and rapidly cycled. All tests were conducted for a minimum of 3000 hours. A reliable method for the fabrication of the closure weld using Heliarc was developed. The effects of bending, welding and brazing on the sheath were also investigated with a special, "traveling-gradient" furnace. Comparison tests on a substitute thermocouple system employing 0.250 inch O.D. Inconel tubing as the protection tube were also made. Various types of thermocouple wires including Chromel P-Alumel were investigated. A Chromel P-Alumel system using short wells made from 1/4 inch and 3/8 inch schedule 40 Inconel pipe was designed and tested under similar conditions.

THERMOCOUPLE DESIGN AND TEST PROGRAM FOR REACTOR PROJECTS

SUMMARY

It was believed that the Chromel P-Alumel thermocouple was the most suitable for the temperature measurements in the high-flux region of reactors. To permit its use in the various corrosive media of the reactor, the thermocouple wires would have to be encased in an Inconel sheath with an insulation scheme capable of withstanding many sharp bends. The commercially available swaged thermocouple material with magnesium oxide insulation appeared to be very promising for this application. With two pairs of thermocouples included in each sheath, a spare couple for each measurement could be obtained quite simply. A sheath with an outside diameter of 0.250 inch and 0.030 inch wall thickness was selected. The specified initial accuracy of the thermocouple material was ± 3/4 percent from 530°F to 2300°F.

There were several questions associated with this material that had to be answered before its performance could be predicted with any degree of reliability. The long term stability (for a minimum of 3000 hours) of the Chromel P-Alumel at the high operating temperatures (1100 to 1600°F) was not known. Also, the homogeneity of the material was not known. Non-homogeneous sections could produce serious error when subjected to severe temperature gradients. The effects of bending, welding and brazing of the sheath on the homogeneity of the material had to be determined. A technique would have to be developed to permit a reliable and easily reproduced closure weld on the material. This would be complicated by requiring that the thermocouple junction be fused into the closure weld. Even after exhaustive inspection (dye-check, X-ray, helium leak test), the integrity of the closure weld in sodium and fluoride salt would still be in some doubt.

To provide answers to these and other questions, an intensive testing program was started. Aging tests under conditions approximating reactor operating conditions were made. These tests, which were made in sodium and fluoride salt environments, also served as integrity tests on the sheath and closure weld. Also, aging tests under more ideal conditions were made in an attempt to define the ultimate stability of the material. Similar tests were made on a possible substitute system of more conventional design, consisting of two pairs of 20 AWG thermocouple wires in an Inconel protection tube (0.250 inch outside diameter with 0.025 wall thickness). A special four-hole, magnesium silicate bead (0.156 inch diameter, 0.125 inch long) was employed as insulation. The effects of different lengths of protection tube and titanium as an oxygen getter within the tube were also investigated. In addition to Chromel P-Alumel, Kanthal, Driver-Harris and A. C. Scott equivalent materials were tested as thermocouple wires.

A special furnace with an extremely sharp gradient was constructed to make homogeneity checks on samples of this sheathed material. The apparatus was designed to permit scanning by moving the furnace at different speeds along the sheath.

An investigation of the effects of nuclear radiation on the dc resistance of the magnesium oxide was started. Samples were irradiated for a three week period in HB-3 at the LITR.

A study was made of an in-pile test for the purpose of determining the effects of nuclear radiation on the thermoelectric properties of the material. A dynamic type of test, with either sodium vapor pressure or melting points of eutectic salts as temperature standards, was considered. Bench tests designed to prove the feasibility of either system have been made in the case of the eutectic salt and is in the final stages for the sodium vapor pressure scheme. The eutectic salt melting point apparatus was also used as a quick and accurate means of spot-checking standard thermocouples employed in all phases of the work.

A 50 kilowatt Megatherm induction heater was employed to test sample thermocouples made from this sheath material under high thermal shock.

The measurement of temperatures on the NaK (a sodium-potassium alloy) piping associated with the reactor vessel was also critical although under less severe conditions. Here there were no nuclear radiation or access problems. The range of temperature operation would vary from 1100 to 1500°F. Once again it was felt that a Chromel P-Alumel thermocouple would be most suitable for the measurement. All thermocouples, wherever possible, would be installed in wells mounted radially on the pipe. Surface measurements would be employed where wells could not be installed. All thermocouples would be welded to the bottom of the well to insure accurate placement.

There were two major problems associated with these well-type units. Again, as with the sheathed type, the stability of the Chromel P-Alumel wire for a minimum of 3000 hours of operation was in question. Also, a means of making a reliable and easily reproduced attachment to the well bottom had to be found. This same scheme could be employed to attach similar thermocouples for the surface measurements on the Inconel piping.

Aging tests under conditions approximating reactor operating conditions were made on sample well-type units.

A special calibration apparatus was constructed permitting the calibration of 12 sheathed thermocouples simultaneously. Another similar piece of apparatus was constructed which permits the simultaneous calibration of five well-type thermocouples.

The temperature standards employed in all testing and calibration work were thermocouples fabricated from high purity platinum-platinum, 10 percent rhodium wire. Identical units made from this wire were sent to the National Bureau of Standards for a certified calibration. All working thermocouples were calibrated against these NBS standards to within an accuracy of $\pm$ 0.9°F.

CONCLUSIONS

All aging data on the Inconel-sheathed Chromel P-Alumel material show that the initial calibration is accurate to within + 3/4 percent and minus zero over the range of 1100°F to 1600°F. The material was purchased with the initial specified accuracy of ± 3/4 percent from 530°F to 2300°F.

The aging data in air where the temperatures were held constant (ideal conditions) indicate that the sheathed thermocouples are more stable than conventional, beaded couples in 0.250 inch O.D. Inconel protection tubes one foot in length over the first 3000 hours of operation. Not only was the average drift less by almost a factor of two but the spread between individual assemblies was less. Beaded couples in three foot tubes showed greater drifts but downward in an opposite direction.

The effects of aging on sheathed thermocouples where the temperatures were slowly cycled (approximating reactor operation) differ from those where the temperatures were held constant. This, as will be explained in detail later, could have been due to contamination of the magnesium oxide during the fabrication of the closure weld. Some closure welds for the cycling tests had to be redone two and three times before passing dye-check and X-ray inspections. New aging tests are being prepared where an all-brazed closure scheme will be used, and all test samples will be carefully baked and pumped on with a vacuum pump prior to being inserted into the aging rigs.

In spite of the disagreement in the two types of aging tests, only one sheathed sample out of 44 (this excludes the units aged at 1800°F) drifted beyond the ± 3/4 percent limits during the first 3000 hours of operation. This does not mean, however, that such performance can be expected in general. This sheath material when employed where temperature gradients are more severe can quite possibly show greater drifts.

The aging data on the sheathed material do show a stability which exceeds that which one might expect from earlier work on Chromel P-Alumel wires. The aging performance of the beaded assemblies in the 0.250 inch O.D. protection tubes is more in keeping with the earlier data.

The aging data on 24 and 200 hour soaked Chromel P-Alumel sheathed material are quite similar, thus making the higher cost of the 200 hour material prohibitive.

The use of thermocouple materials such as Kanthal alloys, Driver-Harris 242-33 alloys, appear quite promising on the basis of the first few thousand hours of their performance in one foot protection tubes. The drift of Chromel P-Alumel wires in these tubes appears to be partially cured by inserting an oxygen getter material in tubes in the form of 30 AWG titanium wires. More data is required here before more positive conclusions can be made.

The homogeneity of the "as received" sheath material seems to be good. Bending of the sheath or welding and brazing on the sheath does not appear to induce any inhomogeneity of any great magnitude.

Many of the rejects of the Heliarc closure weld were caused by porosity. This appears to be caused by contamination associated with the Chromel P and Alumel wires. An all-brazed closure method effectively isolates these porosities from the outside surface of the sheath. Because of this and other reasons to be cited later, it is strongly recommended as a closure scheme. It is also strongly recommended that, in addition to dye-checks and X-ray, a helium leak test be included as part of the inspection procedure for closure welds on this sheathed thermocouple material.

Rapid heating over a few hundred cycles can ultimately produce failure in the thermocouple wires of the sheathed material when they are fused into the closure weld. Some form of expansion loop or bend in the wires might possibly eliminate this type of failure. This should be considered in any future installation involving this material.

The tightness of pack of magnesium oxide insulation has one outstanding advantage that had been overlooked. By accident, a test sample with a faulty closure weld was included in the sodium aging rig. After a few thousand hours, the weld developed a leak. The thermocouple was removed and cross sectioned after five thousand hours. The sodium had not penetrated more than about an inch into the magnesium oxide even though the couple had been immersed over six inches and a 10 psig pressure maintained on the sodium.

The stability of the well-type couples appears to be quite good over the first 2000 hours of operation. A slight upward drift is evident. It is safe to predict that these units will read temperatures with an accuracy better than ± 1/2 percent in the range of $1100^{o}F$ to $1500^{o}F$ for 3000 hours of operation. A major improvement can be made in the fabrication of these wells in the method of attaching the wires to the bottom of the well. The Heliarc technique is very demanding upon the skill of the welder. It is recommended that a Coast Metal 52 brazing scheme be considered for this in any future work with this type of well design.

The effects of nuclear radiation (approximately 1.8×10^{19} nvt) in HB-3 of the LITR on the insulation resistance of the Inconel-sheathed material does not appear to be great. Further work is needed at greater values of nvt.

The effects of nuclear radiation on the thermoelectric properties of Chromel P-Alumel at values of 10^{21} nvt and greater is in doubt and could represent a major source of error. In-pile tests are required and for the greatest value, both dynamic and "before and after" type tests are needed.

INTRODUCTION

The control and safety of reactors are dependent upon reliable temperature measurement. Structural materials will be operating only a few degrees below temperatures that could seriously impair the strength of the material. Excessive temperature gradients across portions of the reactor could induce stresses of sufficient magnitude to cause failure - here the measurement problem is particularly difficult because a Δ T measurement is required. Fuel freeze-up and "snow" problems are other serious consequences that place an additional burden on the temperature measurement scheme.

The work described in this report will only involve the most severe temperature problems associated with the reactor vessel and its NaK (sodium-potassium alloy) cooling system. The many other hundreds of temperature measurements do not require the high reliability and accuracy and will be handled in a routine manner.

The Reactor Vessel Temperature Problem

The temperature measurement problem in the reactor vessel is essentially aggravated by four factors:

1. The high values of temperature operation.
2. The presence of intense nuclear radiation in many areas of measurement.
3. The inaccessibility of the area of measurement.
4. The rapid cycling of temperatures.

The temperature within the reactor structure will vary between 1050°F and 1600°F at full power operation. At these temperatures, thermocouples appear to be the strongest competitor with resistance elements a poor second - wire and insulator stability, ruggedness, readout equipment required all being in favor of the thermocouple. Chromel P-Alumel and platinum-platinum, 10 percent rhodium are two outstanding choices of thermocouple material. The Pt-Pt, 10 percent Rh offers the possibility of a more accurate measurement by factors less than an order of magnitude. This is due to greater uniformity of the wire material and the higher reproducibility of the material from one batch to another. The Pt-Pt, 10 percent Rh material is considerably more expensive and by nature of its high purity must be handled with extreme care to give maximum results. The Chromel P-Alumel material being less uniform or homogeneous offers greater chances for error when subjected to temperature gradients. Also, literature(3,4,5) reports that Chromel P-Alumel wires, after prolonged exposure to high temperatures (800°F and greater), suffer metallurgical and chemical changes; and, when subjected to temperature gradients can produce large errors.

The nuclear radiation at full power would be extremely high in many areas of temperature measurement. The high neutron cross-section of rhodium of about 150 barns makes it extremely questionable as a thermocouple element even at only a few thousand hours of operation. Chromel P-Alumel appears to be better suited for operation in regions of high fluxes. Earlier work by C. Boorman(1) in B.E.P.O. at Harwell, claims that nickel and platinum resistance thermometers exhibited an increase in resistivity over the range of 20°C to 110°C after a total irradiation of 2×10^{19} n/cm^2. Chromel P-Alumel thermocouples showed no change. Consideration of gamma heating of the temperature sensor wires and assembly implies a system of optimum mass (corrosion versus heat generation) and maximum heat transfer between the sensor and the reactor system. Here, once again the thermocouple excels since it is possible to weld the thermocouple to the reactor structure; whereas, any resistance element must be insulated from the system.

The inaccessibility of the many areas of temperature measurement within the reactor necessitates long, twisted paths for sensor wires to traverse. This dictates a sensor assembly that can be bent freely, with no damage to the assembly. In addition, considerable Heliarc welding and brazing will be necessary along the containers of the sensor. In many cases, the bends occur where the assembly passes through bulkheads or shells across which quite large temperature gradients exist. Literature (2) cautions one against the effects of bends on thermocouple material particularly when they are located in regions of high temperature and steep gradients. Accelerated grain growth and possible formation of inhomogenieties jeopardize both the integrity and accuracy of the system.

The rapid temperature fluctuations within the reactor (with rates as high as 20°F sec. being possible) can produce abnormal stresses and ultimate failure in a temperature sensor due to different coefficients of expansion of the various components. Also, rapid temperature changes can possibly precipitate another alloy phase in alloys such as Chromel P-Alumel thereby inducing inhomogenieties which can cause erroneous readings if subjected to temperature gradients.

In a first appraisal of the above, a Chromel P-Alumel thermocouple contained in an Inconel sheath with hard-packed magnesium oxide insulation was chosen as the type of sensor for reactor vessel temperature measurements. The assembly length was required to be approximately 10 feet in length. The outside diameter and wall thickness of the material was selected as 0.250 inch and 0.030 inch respectively. Permitting a spare couple for each measurement, two pairs of thermocouples (20 AWG solid, oxide finish) were contained within the sheath.

In the tests on this sheathed thermocouple material, to be described in full detail later, two types of Chromel P-Alumel assemblies are involved. One type of sheathed Chromel P-Alumel had been subjected to a 200 hour soak period at 1350°F in an atmosphere of helium, the other type had undergone a 24 hour soak period by the thermocouple vendor. The specific reasons for the 200 hour soaking period are still not clear. It was felt that it could have some tendency to stabilize the Chromel P-Alumel wire metallurgically and chemically by driving off any air or moisture trapped in magnesium oxide.

For certain precision temperature measurements in non-nuclear versions of the reactor, identical sheath material with two pairs of platinum-platinum, 10% rhodium thermocouple wires was chosen and, for comparison, was included in tests on this sheath material.

The NaK Piping Temperature Problem

Associated with the reactor system are other critical temperature measurements with considerably less severe environmental problems. One of these involves the measurement of the temperatures on the NaK piping connecting the intermediate heat exchangers of the reactor to external heat exchangers. Here, there are no nuclear radiation or access problems. The cycling will also be less severe. The range of temperature operation will vary from 1100°F to about 1500°F.

High accuracies will be required as they will be indicating ΔT measurements across heat exchangers thereby providing valuable performance data.

All temperature measurements will be made on Inconel piping ranging from two and one-half inches to four inches in diameter. All thermocouples, wherever possible, will be installed in wells mounted radially on the pipe with an immersion length equal to 1/3 the inside diameter of the pipe. All thermocouples will be welded to the bottom of the wells to insure accurate placement of the thermocouple. Special techniques will be required to make a reliable attachment weld.

For the few cases where wells cannot be installed, the thermocouples will be Heliarc welded to the outside surface of the pipe. No new problems are envisioned here. The same attachment techniques will be applied as will be used to attach the thermocouple to the bottom of the well.

In these instances, the metallurgical and chemical stability of the Chromel P-Alumel couple is the major problem. All wells will be made as short as possible and of 3/8 inch, or in a few cases 1/4 inch, schedule 40 pipe. With four inches of insulation around the piping, a well will not be any longer than about six inches for the largest piping. Chemical changes, such as selective oxidation, of some of the Chromel P-Alumel alloying material appears to be prevalent in wells of limited oxygen supply. This effect should be minimized in the well described above.

A platinum-platinum, 10% rhodium thermocouple has been seriously considered for this NaK piping temperature measurement. The problems of welding to the well bottom and supplying an impervious ceramic liner for the well are not compatible. Tests on the stability of Pt-Pt, 10% Rh wires in Inconel protection tubes were considered for 3000 hours operation.

Objectives of the Design and Test Program

Upon appraising all of the above, an attempt was made to organize a program that would supply the answers to some of the questions involving Chromel P-Alumel and Pt-Pt, 10% Rh systems in various configurations (sheathed, conventionally beaded in 0.250 inch O.D. protection tubes). In addition, the program includes the investigation of other types of thermocouple materials in the event the Chromel P-Alumel thermocouple should prove unsatisfactory. Given below is an outline of the program. It should be stated that, with the exception of aging tests on well-type thermocouples, the entire program is aimed at evaluating 0.250 inch Inconel sheathed thermocouples and a possible substitute assembly consisting of two pairs of 20 AWG thermocouple wires with a special four-hole ceramic bead in a 0.250 O.D. Inconel protection tube.

1. 0.250 inch O.D. Inconel Sheathed Thermocouple

    A. To develop, in conjunction with the Metallurgy Division, a sound and easily reproduced technique for a junction and closure weld. Integrity tests, for a minimum of 3000 hours will be made in immersing mediums of fluoride salts and sodium.

    B. To age thermocouples made from this material containing either two pairs of Chromel P-Alumel or Pt-Pt, 10 percent Rh wires at temperatures ranging from 1100°F to 1800°F for a minimum of 3000 hours. The effects of static and slow cycling of temperatures will be resolved.

    C. To examine the uniformity of "as received" material and the effects of welding, brazing and bending on the uniformity of the material.

    D. To examine the effects of rapid heating on the accuracy and physical soundness of thermocouples made from this material.

    E. To evaluate the electrical resistance of the magnesium oxide insulation at elevated temperatures and in the presence of nuclear radiation.

    F. To determine the effect of nuclear radiation on the aging performance of this material.

2. Thermocouples in 0.250 inch O.D. Inconel Protection Tubes

    A. To age Chromel P-Alumel and other commercially available thermocouple material in these protection tubes under test conditions identical to those in 1(B). Special four-hole ceramic beads will be employed as insulator material.

3. Well-type Thermocouples for NaK Piping

    A. To design and fabricate well-type thermocouples employing short lengths of 3/8 inch and 1/4 inch schedule 40 Inconel pipe as well body material and Chromel P-Alumel wire (± 3/8 percent 530°F to 2300°F, 20 AWG, solid, bright finish). Here, it will be required to develop, in conjunction with the Metallurgy Division, a sound junction weld for 20 AWG Chromel P-Alumel wires and a simple and reliable attachment to Inconel surfaces.

   B. To devise an installation scheme which will preserve the accuracy of the well-type unit.

   C. To age sample units under conditions approximating reactor operation for a minimum of 3000 hours.

4. Chromel P-Alumel Thermocouples for Attachment to Inconel Surfaces

   A. To be fabricated with techniques identical to those employed for thermocouples used in the well-type units. Installation problems will be solved with the aid of the Metallurgy Division.

5. Calibration apparatus for Reactor 0.250 inch O.D. sheathed Thermocouples and NaK Piping Well-type Thermocouples.

6. Standards Employed in the Aging Tests and Calibration Work

Work on this program was started during the summer of 1956 and extended to December of 1957 at which time the program was terminated at ORNL. The program as outlined above was not completed. The work involving nuclear effects on the performance of the sheathed thermocouple was started but did not get beyond the "bench testing" stage. The effect of rapid heating and cooling on the accuracy of the sheath material was not investigated. Also, the aging tests involving the well-type thermocouple did not exceed the minimum limit of 3000 hours. It should be mentioned that much of the aging work involving the sheathed thermocouples and thermocouples in Inconel protection tubes was continued and is still in progress at the time of writing of this report. This additional work will be covered in future ORNL reports. Further work has also been done on the closure problem for the sheathed material, and "before and after" type tests are being conducted on Pt-Pt, 10 percent Rhodium thermocouples in the LITR and ORR. The result of this work will also be available in future ORNL reports.

0.250 INCH O.D. INCONEL SHEATHED THERMOCOUPLES (11,12)

Junction and Closure Weld

Considerable effort was expended to develop a technique to form the junction and closure weld on this sheathed thermocouple material. It was required, because of gamma heating considerations, to have the thermocouple wires welded into the closure weld. Figure 1 shows a sample of the sheath material with a four-hole Inconel filler tip, mounted in a water-cooled chill block prior to the Heliarc welding operation. The chill block minimizes gas expansion and subsequent "bubbling" as the filler tip is being welded to the sheath and thermocouple wires. Details of the filler tip, sheath preparation, Heliarc welding operation and chill block are given in ORNL Drawings (13)and (14). An S. S. White Airbrasive unit was employed to remove the hard-packed magnesium oxide. Work to be described later will show why extreme care must be exercised in the cleaning of the thermocouple wires. On the average, approximately 30 percent of all welds passed dye-check and X-ray inspections. The spread ranged from about 10 to 50 percent. All of the test samples used in tests to be described had junction closures made in the above fashion. Figure 2 shows a cross-section of a typical closure weld. Note the large void space below the closure.

Later work by the Metallurgy Division revealed an inherent source of contamination associated with the Chromel P-Alumel wires which was causing a large portion of rejects with the Heliarc technique. Their recommendation was an all-brazing operation using a four-hole Inconel cap to keep the four thermocouple wires joined together and to the sheath material. Then, a blank Inconel cover would isolate the porosities of four-hole braze from the outside of the sheath.(8) Figure 3 shows the details of the fabrication involving the sheath material, four-hole cap, the cover plug and the brazing material. Figure 4 shows a cross-section of the all-brazed closure. In addition to being relatively simple to fabricate, the brazing scheme has two other important advantages. First, it permits a minimum void space below the closure and secondly it produces an accurately positioned junction. The small void space is considered an advantage since it reduces the possibility of a selective oxidation effect. Additional integrity tests in sodium and fluoride salts have been proposed for this new closure scheme. The results on these tests will be given in a later ORNL report.

Other methods for making the closure weld were considered and were rejected for various reasons. An attempt was made to spin the sheath material down and over the wires, then making the Heliarc weld without a filler tip. It was not possible to spin the Inconel material without special annealing apparatus and the possibility of inducing metallurgical imperfections was too great. Another scheme involved fusing the four wires into a junction before welding it to a filler tip containing one center-drilled hole. The elimination of the excessive burning of the wires during the junction formation was a problem, and the fact that the junction would be further removed from the outside surface of the closure was another possible source of error when sharp gradients and gamma heating are considered.

Dye-checks and X-ray inspections are not foolproof. The integrity of the closure weld technique using the four-hole filler tip and Heliarc was tested by operating 19 samples of these closure welds in sodium at 1500°F. Sixteen samples were tested in a fluoride salt at 1500°F. All closures had previously passed dye-checks and X-ray examinations. After 8000 hours of operation, there was no evidence of any leak. It should be noted that approximately once a week, the temperature of the sodium and fused salt was reduced to 1300°F and 1100°F for the purpose of obtaining stability data on the thermocouple wires.

Aging Tests

One of the major causes of instability of Chromel P-Alumel thermocouples, as mentioned earlier, results from metallurgical and chemical changes occurring in the wires when operated at elevated temperatures in a limited oxygen supply. The aging tests to be described here were performed to observe the magnitude of these effects in the Inconel-sheathed thermocouple.

The aging tests that were conducted were of two distinct types. In one type, the aging conditions were slanted at actual reactor operating conditions. In the second type, aging conditions were more ideal in nature, tending to define the ultimate stability of the thermocouple.

The aging tests approximating actual reactor operating conditions were the same as those mentioned above providing information on closure integrity. Ninteen thermocouples made from Chromel P-Alumel sheath material that had been soaked for 24 hours in helium at 1350°F by the vendor were operated in sodium at 1500°F. Approximately once a week, the temperature was reduced to 1300°F and 1100°F for readings at these temperatures. This produced the effect of slow cycling. Also, another 16 test couples were aged in a fluoride salt in an identical fashion. Of these 16 couples, two were made from Chromel P-Alumel sheath material that had been soaked for 200 hours in helium at 1350°F by the vendor and three contained 2 pairs of Pt-Pt, 10 percent Rh thermocouple wires. The test couples were approximately 2 feet long and were immersed about 6 inches into the liquid media. The lead end of the couples were sealed by a deposition of Dow-Corning Number 4 silicone grease. Test results will be given later in this section.

In the second type of aging test, the couples were aged continuously at one temperature in air. Three different temperatures were considered - 1800°F, 1600°F and 1300°F. The 1800°F and 1600°F tests each contained six test couples made from 24 hour-soaked sheath material, six made from 200 hour-soaked material and three sheathed couples with 2 pairs of Pt-Pt, 10 percent Rh thermocouples. The 1300°F test contained 6 test couples made from the 24 hour-soaked sheath material. The test couples in these test were also approximately 2 feet long with the lead end sealed with Dow-Corning Number 4 silicone grease. A copper block jacketed with Inconel was employed as the test container here. The Inconel jacket was constructed to contain helium gas which prevented oxidation of the copper. Approximately nine inches of the sheath couple was immersed in the copper block. The test results will be given later in this section.

Figure 5 shows a photograph of the sodium, NaK and fluoride salt aging systems along with calibration facilities for sheathed and well-type thermocouples. The second cabinet from the left contains switch panel, L&N Model H (3 mode) controller, 12-point Brown recorder and furnace control panel for the Nak aging system for NaK piping well-type thermocouples. The three cabinets in the middle contain switch panels, controllers, multi-point recorders and furnace control panels for the sodium and fluoride salt aging tests. The operator is shown adjusting the thermocouple readout console consisting of a L&N K-3 potentiometer and Model 9835-B microvolt amplifier employed as an electronic galvanometer. The two cabinets on the far right are employed to operate a sodium filled system for calibration of well-type thermocouples and a copper block system jacketed with Inconel for calibration of 0.250 inch O.D. sheathed thermocouples. The cabinet on the far left (partially completed) will contain the controls for a melting-point system containing a NaF, CaF salt for spot checking (1497°F) the calibration of our standard thermocouples.

As expected, the drift of the couples in the sodium followed the same general pattern as the drift of the couples in the fused salt. Three typical trends appear to be evident over the first 8000 hours of operation. Seven of the 30 sheathed couples (24 hour soaked) displayed a slight upward drift throughout, four finally exceeding by about 2 degrees the vendors upper tolerance limit of ± 3/4 percent of reading at the 1100°F test point. Seventeen of the 30 displayed a slight downward drift initially, then reversing into a slight upward trend after 4000 to 5000 hours. Six displayed a sharp downward trend after a few thousand hours of a slight downward drift and all finally drifted slightly beyond the vendors lower tolerance rating of -3/4 percent at the 1500°F test temperature. Eventually, all but one reversed the downward trend and have drifted back into tolerance. The single unit continued its downward drift finally going well out of tolerance. Figures 6, 7 and 8 show these three typical trends at the three test temperatures of 1500°F, 1300°F, and 1100°F. Figure 9 shows the drift of one of the two 200 hour-soaked Chromel P-Alumel sheaths that were tested. The drift of the other unit was very similar. Figure 10 is typical of the drift of the three sheathed Pt-Pt, 10 percent Rh couples that were tested. Tables 1 and 2 summarize the aging data for the sodium and fluoride salt tests.

The drift of the sheathed couples aged continuously in air at three different temperatures were <u>all</u> observed to be in the upward direction. Figures 11, 12(6) show the drift of the 200 hour and 24 hour-soaked material. The curves show the drift of a typical couple, the drift of the mean of the 12 readings associated with the six assemblies (there were two couples per assembly), and the spread of the 12 readings. The 1800°F and 1300°F tests were terminated after 5000 hours to release the testing facility for aging tests on other thermocouple materials in 0.250 inch protection tubes. Samples of the sheath material removed from the 1800°F tests have been cross-sectioned and metallographic studies were made by the University of Tennessee under contract to ORNL(9). Figures 13 and 14 are photographs of some typical micro-structures. Both Chromel P and Alumel wires appeared very sound showing very little evidence of oxidation. Further details on these aging tests in air will be given in another ORNL report by J. F. Potts of the ORNL Instrumentation and Controls Division.

The explanation for the different performances of the couples when aged continuously and when slowly cycled is not clear at this time. It was considered earlier that a sodium or fluoride salt leak could be causing the downward shifts in drift. One sheath couple was removed from the sodium test after 5000 hours to verify this, but no evidence of a leak was found, the closure weld showing very little indication of attack. There is one explanation that appears plausible. The closure welds on the couples used in the sodium and fused salt aging tests were required to pass dye-check and X-ray inspection; consequently, at least 50 percent of them were remade two and three times. It is very possible that this additional handling and welding may have contaminated the sheath material with moisture. The closure welds for the aging tests that were conducted in air were not examined as critically - good fusion of wires to sheath being the only requirement - which meant less possibility for moisture contamination. The proposed new aging tests on the sheath material with the all-brazed closure will have all test samples carefully baked and pumped on with a vacuum pump. The lead end will then be sealed off with glyptal.

Uniformity Tests and Effects of Bending, Welding and Brazing

The bending of the sheath material could possibly induce localized inhomogeneities in the thermocouple wires. These, when subjected to sharp temperature gradients, could produce spurious voltages causing error. To test this material for these effects, a special gradient furnace was designed and constructed which could be moved along the sheath at uniform rates.(6) The maximum gradient pattern developed was symmetrical about a peak temperature of 1830°F with a slope of about 840°F per inch. This far exceeds any gradients that will be encountered in the ART reactor and auxiliaries. Tests at lesser gradients with 1600°F and 900°F peak temperatures were made to possibly reveal any annealing effect that might be induced by the test itself. In these tests the individual wires of the sheath were connected to an L&N 9835-B microvoltmeter which operated a conventional recorder. A perfectly homogeneous wire would produce no output as the sheath is scanned. Several samples of the sheath material "as received" from the vendor were scanned establishing the normal "background" of the thermocouple material. Then several samples that were bent in a U-shape over a one-inch mandrel and straightened out were subjected to the traveling gradient. Figures 15, 16 and 16A(6) shows typical response curves of the "as received" material, material that was bent and straightened, material that was annealed then bent and straightened. The response curves include the output from individual Chromel P-Alumel wires along with the response of the two wires made up as a thermocouple.

Samples of the sheath material with Inconel sleeves brazed (using Coast Metal 52 brazing alloy) and Heliarc welded to sheath were scanned with the above mentioned apparatus. No effects above the normal background of the material could be observed. This work with the traveling gradient furnace will be described in greater detail in another ORNL report by J. F. Potts.

Rapid Heating Tests

The rapid temperature fluctuations can produce abnormal stresses and ultimate failure in the sheathed thermocouple due to different coefficients of expansion of the sheath material and wires and the different masses involved. This is particularly severe in the sheathed thermocouple assembly since the wires are firmly welded into the closure and are firmly held at the other end of the hard-packed magnesium oxide. Also, stresses induced across the closure weld could produce cracks thereby reducing the integrity of the closure.

In addition to the two possible types of failure mentioned above, the accuracy of the sheathed Chromel P-Alumel couple can be influenced by the possible precipitation of other alloy phases by these rapid changes in temperature. Once again this would appear as an inhomogeneity and could cause large errors when subjected to gradients.

A 50 kilowatt Megatherm induction heater was renovated and employed to heat a single test specimen of sheath material which was submerged in an Inconel charge container filled with NaK. A Flexopulse timer was used to provide a variable "on-time" for the induction heater. With the masses of the charge container and NaK kept to a minimum value, the maximum rate of temperature rise obtainable was about 130°F per second. Arcing of the induction coil to the charge container prevented raising the output of the Megatherm unit to obtain a more rapid heating rate. The 130°F per second rate is still far in excess of anything possible within the reactor.

One sheath couple was tested and both Chromel P wires opened after 240 heating and cooling cycles between 800° F and 1200°F. An X-ray examination revealed that the closure weld was still sound. The two wires showed fractures at the point of juncture to the closure weld. Either a ductile-type break or a break caused by embrittlement were possibilities. A cross-section examination revealed that the break was ductile.

Another sample was cycled for 200 times and the wires were removed for investigation in the traveling gradient apparatus. The termination of the program at ORNL did not permit any further work on this or with this test facility. Figure 17 is a photograph of the facility. On the left is the instrument control cabinet with a haze indicator and alarm device mounted in the top panel. The haze indicator is used to monitor the temperature of the charge container by its color with a 930 phototube. The device would turn off the induction heater in the event the temperature of the charge container became too high. The Brown recorder mounted below the haze indicator is a special 1/4 second unit which monitors the NaK temperature through a 1/16 inch sheathed thermocouple or can be switched to readout the signal of the test couple. The bottom panel contains a Flexopulse timer which is used to cycle the induction heater and permits independent adjustments of the "on" and "off" time of the induction heater unit. The middle cabinet contains the induction coil, Inconel charge container and NaK expansion pot. The large cabinet on the right is the main control cabinet for the 50 kilowatt Megatherm induction heater, containing the r-f oscillator and associated circuitry.

Insulation Resistance of the Magnesium Oxide Insulation

Under Nuclear Irradiation

The effect of nuclear radiation on the resistance of magnesium oxide was checked by inserting three samples of the sheathed material each abour four feet long in HB-3 of the LITR(7). Two samples were maintained at a constant temperature of 1200°F and the third sample was not heated (gamma heating raised its temperature to 450°F). Approximately one foot of the samples was subjected to the elevated temperatures and high flux. The samples were irradiated for three weeks, and a study of the data reveals:

1. No significant, immediate effect was produced by the radiation - a 10 to 30 percent decrease in resistance was observed in going from zero power to full reactor power.

2. No significant long term drift in resistance was noted during the three week interval of the test. An upward drift during the first 48 hours of the test was believed to be a result of additional drying of the magnesium oxide.

Figure 18 shows the resistance versus time curve of the Chromel P-Alumel wire to wire resistance for the heated and unheated samples. A General Radio Type 544-B Megohm bridge with a battery power supply was employed for all resistance measurements.

The test work revealed the extreme susceptibility of the magnesium oxide for water absorption. Considerable baking was necessary to insure dryness of the material prior to the tests. Glyptal was employed as a sealant for the end of the sample in regions of low flux. A cap welded on with Heliarc was used to seal off the irradiated end of the test sample.

Sheath material with beryllium oxide insulation was fabricated at ORNL for irradiation tests. This work will be continued and will be covered in a future ORNL report. Sheath material with zirconium oxide insulation was not available and no attempt is being made at this time to include it in any irradiation tests.

THERMOCOUPLES IN 0.250 INCH O.D. INCONEL PROTECTION TUBES

At the outset of the ART thermocouple program, the stability of the Inconel sheathed thermocouple was an unknown quantity. It was felt advisable to parallel the testing of the material with a possible substitute thermocouple system in the event some intolerable characteristic of the sheathed material would be uncovered. This substitute system would consist of the following:

Protection Tube - Inconel tubing with 0.250 inch outside diameter and 0.0250 wall thickness.

Insulator Bead - Four-hole, magnesium silicate bead, 0.156 inch diameter, 0.125 inch long with 0.040 inch holes.

Thermocouple Wires - I. Chromel P-Alumel, 20 AWG, bright finish, with ± 3/8 percent accuracy from 530°F to 2300°F.

II. Same as above with two 30 AWG titanium wires as oxygen getter material.

III. British-made (A. C. Scott) Chromel P-Alumel with special vacuum anneal.

IV. Kanthal alloys with temperature-emf characteristics identical with Chromel P-Alumel.

V. Driver-Harris 242-33 alloys.

All of the work presented in this section will be covered in greater detail in another ORNL report by J. F. Potts.

Aging Tests in Air with One Foot Protection Tubes Using Type I Wires

The air aging tests were made simultaneously and in the same copper blocks that were used for the air aging tests on the sheathed material. Six units where extreme care in assembly and handling was exercised and six units where no special care was taken were inserted in each of the aging furnaces operating at 1800°F, 1600°F and 1300°F. The special care consisted of washing all ceramic beads with alcohol and distilled water then baking at 1500°F for one hour, washing the protection tube with alcohol and distilled water then baking for one hour at 400°F, and washing the wires with alcohol. All handling and assembly work was done with clean white gloves.

Figures 19 and 20(6) show the drift of these two types of assemblies. The curves show the drift of a typical couple, the drift of the mean of the 12 readings associated with six units (there were two couples per protection tube), and the spread of the 12 readings. The drift of all couples were in the upward direction. The spread in readings of the carefully handled units was less by a factor of two than the spread of the units that had received normal handling after 3000 hours of operation at 1600°F. The mean drift, however, was about 30 percent greater. The drift and spread of either type of beaded assembly was greater after 3000 hours than the drift of the sheathed couples. Compare Figures 19 and 20 with Figure 11 and 12.

Samples of the wire removed from the 1800°F test have been cross-sectioned and metallographic studies are being made by the University of Tennessee (17). Figures 21, 22, 22A and 22B(9) are photographs of some typical micro-structures. It is interesting to note the similiarity in grain structures between Chromel P-wires of the sheathed assembly, Figure 13, and the ceramic beaded assembly, Figure 21. The heavily oxidized alumel wire of the beaded assembly shown in Figures 22, 22A and 22B, is particularly amazing. There is no evidence of this in the Alumel wire of the sheathed assembly, Figure 14.

The drift of the couples in these tests did not agree with the results of Spooner and Thomas(5) where it is shown that the drift of Chromel P-Alumel thermocouples in confining protection tubes is downward. It is believed that the one foot protection tube was inadequate to produce the effect described by Spooner and Thomas. Indeed, ART thermocouple assemblies would necessarily be required to be at least ten feet long. Thus, it was decided to terminate the tests on these couples in the 1800°F and 1300°F furnaces after 5000 hours. The 1600°F furnace was allowed to continue until 10,000 hours had been logged. Additional tests to be described in the next section will contain test samples in three foot protection tubes.

Aging Tests in Air With Three Foot Protection Tubes Using Type I and II Wires

In these tests, an identical set of assemblies were tested in three separate aging furnaces utilizing the copper blocks described earlier. One furnace was kept continuously at 1800°F, another at 1600°F, and the third at 1800°F with weekly reductions to 1600°F and 1300°F. The set consisted of three tubes with two pairs of Chromel P-Alumel (± 3/8 percent accuracy, solid, 20 AWG bright), four tubes with one pair of Chromel P-Alumel (± 3/8 percent accuracy, solid, 20 AWG bright), and two 30 AWG titanium wires. In addition to these, three one foot assemblies with Chromel P-Alumel (± 3/8 percent accuracy, solid, 20 AWG, bright), were included to insure that test conditions similar to those described in the preceding section prevailed. Approximately 2000 hours of aging data has been accumulated and Figures 23 and 24(6) show the drift of the two types at 1800°F and 1600°F continuously.

These tests on the three foot protection tube quite clearly support the findings of Spooner and Thomas. It is too early to predict the trend of the assemblies with titanium wire although it appears that the oxygen getter material has partially cured the drift of Chromel P-Alumel over the first 2000 hours of operation.

Aging Tests in Air With One Foot Protection Tubes Using Type III, IV and V Wires

Aging tests operating in air under conditions identical to those described in the preceeding section on Driver Harris 242-33 alloys, Kanthal alloys, British-made (A. C. Scott) Chromel P-Alumel have been operating for over 2000 hours. Not enough data has been accumulated for any definite conclusions at this time. The Kanthal and Driver-Harris wires show considerable promise as being superior to Chromel P-Alumel. The A. C. Scott appears to be quite unsuitable at 1800°F, several samples failing after only a few hundred hours of operation.

WELL-TYPE THERMOCOUPLES FOR NaK PIPING

The temperature of high velocity (1400 gpm) and high temperature (1100°F to 1500°F) NaK in Inconel piping will be measured with Chromel P-Alumel thermocouples located in wells mounted radially on the piping.

Fabrication Details

The thermocouples (two per well) were fabricated from Chromel P-Alumel (± 3/8 percent 530°F to 2300°F, solid, 20 AWG, bright wire). The thermocouples were welded to the bottom of the well with Heliarc to insure accurate placement. All ceramic beads contain a minimum of 96 percent aluminum oxide. Three different beads were employed - a 1 1/2 inch long x 0.094 x 0.156 inch oval bead with two 0.040 inch holes, a 0.25 x 0.25 inch ball and socket bead with two 0.040 inch holes and a 0.125 x 0.125 inch ball and socket with a single 0.056 hole. Figure 25 shows a typical unit in the assembled and disassembled state along with the copper chill block and jig required for junction formation and attachment weld respectively. These will be covered in more detail later in this section.

The well immersion depth was arbitrarily chosen as one-third the inside diameter of the pipe line. The well body material for 2 1/2 inch and greater pipe-line was selected to be 3/8 inch schedule 40 Inconel pipe and 1/4 inch schedule 40 pipe for a two inch pipe line. The immersed portion of the body was turned down to 0.050 inch wall thickness. This well body design was tested for pressure drop in a 3 1/2 inch pipe on a water loop at velocities up to 1200 gpm. The permanent pressure drop was less than 2.0 psi at 1200 gpm.

The lagging length of the well body was made as short as possible in an attempt to minimize heat losses and any selective oxidation effect that is common in long narrow wells at elevated temperatures. With four inches of the insulation around the piping, the longest well (for 4 inch piping) will only be about 6 inches.

To insure a reliable and easily reproduced attachment of the couple junction to the well bottom(10), a technique was developed which permitted the coating of the thermocouple junction with Inconel without any undue burning of the wires. This "wetting" of the junction simplifies the subsequent Heliarc weld to the well bottom. With very little practice, the welder is capable of controlling the diameter of the spherical junction to within ± 0.005 inch. The four wires with ends carefully cleaned of any oxide are set up in one of the countersunk holes of the copper chill block(15) shown in Figure 25 with wires flush with the surface of the block. The four wires are then fused with Heliarc and without removing the gas flow, a small quantity of Inco Number 62 weld rod is added, filling the countersunk hole. It was discovered that a careful cleaning of the thermocouple wires of any oxide leads to more reliable junction. This is shown in Figures 26 and 27. Here, cross-sections of the Chromel P-Alumel junction reveal that the carefully cleaned wires were free of chromium oxide deposits at the junction point of the Chromel P-Alumel wire and thermocouple junction. These deposits can very easily lead to fractures under thermal cycling. Cleaning of the wires with an S. S. White airbrasive unit using aluminum oxide grit is recommended, although cleaning by hand using abrasive paper with aluminum oxide grit is equally as effective with a little more care.

The jig(16) in Figure 25 is a 3/16" flat Inconel plate with a 0.625 diameter dish, 0.063 inch deep centrally located. This plate, mounted horizontally in a vise, is used to hold the well bottom while the four thermocouple wires are being attached. The attachment proceeds as follows, a small button, using Inco Number 62 weld wire, is fused to the bottom piece (about 3/16 inch diameter, 1/8 inch high) then, with the button held molten with the arc, the Inconel-coated junction is slowly pushed in with wires held horizontally. Two or three jabs may be necessary to heat the junction to a temperature where it will fuse. The two Alumel wires with its lower melting point should be adjacent to the well bottom.

Extreme care and very meticulous cleaning was taken in the assembly of the well couples. Justification for this is cited in considering the results of the aging tests in air on ceramic beaded couples in 0.250 inch O.D. protection tubes mentioned earlier in the report. All ceramic beads were carefully washed with methyl alcohol and distilled water and baked at 1500°F for one hour. All wires were washed with alcohol and air dried. All well body pieces (body and bottom tip) were degreased, washed in methyl alcohol and baked at 400°F for one hour. All handling of pieces was done with clean white gloves.

Serious consideration was given to a technique involving the attachment of the wires to the well bottom using Coast Metal 52 brazing alloy. This would eliminate the hand welding technique described above. Time did not permit further pursuit of this, but its possibilities are strongly recommended for any further work of this nature.

Installation Detail

The well (with thermocouples attached) will be installed in the NaK piping by Heliarc welding it into a close fitting Inconel riser - 3/4 inch schedule 40 pipe will be used as riser material for the 3/8 inch well and 1/2 inch schedule 40 pipe risers will be used for the 1/4 inch well. No consideration was given to welding effects on the thermocouple wires on the basis of the small effects observed by welding on the sheath material described earlier.

Each well thermocouple, upon its installation, will be provided with a clean, flexible extension consisting of a coiled spring made from 0.109 inch Inconel wire. This will eliminate any possibility of sharp bending of the thermocouple wires near regions of high temperature gradients.

Aging Tests

Five sample units were constructed for aging tests in a NaK pot. The units were welded into risers on the bottom of the pot in a manner identical to that which will be used on the actual pipe installation. Four inches of insulation was used to lag the wells, thus duplicating the final installation scheme.

The wells were maintained at 1500°F and approximately once a week the temperature was reduced to 1300°F and 1100°F for readings at these temperatures. This is identical to the aging procedure employed for the sheath couples in sodium and fused salt.

The first 2000 hours of aging data on one of the units are shown in Figure 28. The trend of drift of the other four units was very similar. The aging data of all units at 400 hour intervals is given in Table 3. The extremely close agreement between the two couples in a given well in all five cases is startling. This does not appear consistent with the larger deviations in readings between wells. The reason for the inconsistency is undoubtedly explained by the large axial temperature gradients known to exist in the pot and the unfortunate variation in immersion depth of the wells (as much as 1/4 inch) due to improper installation. All readings were still close to the ± 3/8 percent accuracy quoted for the wires.

CHROMEL P-ALUMEL THERMOCOUPLES FOR SURFACE ATTACHMENT(11)

Where the installation on the reactor and auxiliaries requires an attachment of a Chromel P-Alumel thermocouple to Inconel or stainless steel surfaces, the technique will be employed as described above in attaching the thermocouple to the well bottom. Back-up gas requirements and other details have been considered and have been resolved by the Metallurgy Division. The couple will have an Inconel-coated junction and will employ the same ceramic beads as used in the assembly of the well-type couple. The details of junction formation will be identical as to those described in the section on well-type thermocouples with the exception that one pair of thermocouple wires is involved instead of two.

CALIBRATION APPARATUS FOR REACTOR 0.250 INCH O.D. SHEATHED THERMOCOUPLES AND NaK PIPING WELL-TYPE THERMOCOUPLE

A copper block 3 1/2 inches in diameter and 14 inches long with 14 penetrations, each nine inches deep, was fabricated and completely jacketed with Inconel for use as a calibration system for 0.250 inch O.D. sheathed thermocouple. Twelve penetrations were for the couples under calibration, one for a Pt-Pt, 10% Rh standard, and one for a furnace control thermocouple. The jacket was made tight enough to contain helium in order to prevent oxidation of the copper. The block was heated by a Marshall gradient furnace (16 inches long with a 3 1/2" bore) which has a tapped heater permitting the adjustment of furnace gradient with resistor shunts. With no shunts applied, and the block centrally located in the vertically mounted furnace, the first inch above the bottom of the penetration had less than one degree F of gradient. The maximum radial gradient from the center hole to any of the outer holes was also less than one degree F. This effectively eliminated any positioning error within the calibration block. This meant, however, when one considers the Law of Intermediate Metals, that we are essentially calibrating the thermocouple wire one inch above the junction and the calibration does not include any irregularities that exist at or near the junction. If it is postulated that the wires are perfectly homogeneous, then the calibration is valid. Results of the traveling gradient tests on "as received" material described earlier reveals that this is approximately true. A five point calibration was to be made on each sheath couple at 100° intervals from 1100°F to 1500°F. The time constant of the furnace permits a calibration point every hour and a half.

The calibration system for the well-type thermocouples consisted of a sodium filled Inconel pot with five penetrations permitting the calibration of five well-type units simultaneously. Two additional penetrations were included for a standard Pt-Pt, 10% Rh couple and a furnace control couple. In this system, the gradient in the first inch of the tube was also less than one degree F. Radial variations were less than one degree F. Once again, the calibration does not include any irregularities that may exist at or near the junction, but if the wires are considered everywhere homogeneous, the calibration is valid. A three point calibration was considered adequate in the case of these units.

STANDARDS EMPLOYED IN THE CALIBRATION WORK AND AGING TESTS

All standards employed in the work described in this report were fabricated from the highest purity of platinum and platinum, 10% Rhodium wire obtainable. Three couples made from this material were sent to the National Bureau of Standards for a certified calibration. Several working standards were made from the same wire and were calibrated with these standards at ORNL by the Instrumentation and Controls Division Standards Room to an accuracy of ± 0.9°F from 700°F to 1800°F.

All standards are given a weekly spot check by inserting them in a eutectic salt (NaF, CaF_2; 68-32 Mol. percent) melting point apparatus. The melting point as measured by these standard thermocouples is 1487.0°F and is easily reproducible to within ± 1.0°F. Figure 29 shows two sets of melting point curves made by two standards with a time interval of one month separating the readings. These standards and the melting point apparatus had been in service several times during this interval.

All cold junctions employed ice made from tap water. Mercury U-tubes were used to connect the thermocouple wires to high purity copper wires going to the potentiometer.

All readings were taken with a Leeds and Northrup K3 potentiometer with a Leeds and Northrup 9835-B microvoltmeter employed as an electronic galvanometer. An Eppley unsaturated cell was used as the voltage reference.

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(12) ORNL Specification 7444-2: Sheathed Thermocouple For Use on 7503 Project

(13) ORNL Drawing Q-1719-1: 0.250 O.D. Sheathed Thermocouple Tip and Welding Details

(14) ORNL Drawing Q-1719-3: 0.250 O.D. Sheathed Thermocouple Chill Block for Closure Weld

(15) ORNL Drawing Q-1719-117: Project 7503 Thermocouple Well-Junction Formation Chill Block Details

(16) ORNL Drawing Q-1719-121: Project 7503 Thermocouple Well-Tip Holder for Thermocouple Attachment Details

(17) ORNL Specification 7444-1: Magnesium Oxide

TABLE 1

Results of Aging Test of 0.250 Inch O.D. Sheathed Thermocouples in Sodium

Time (hrs.	Nominal Test Temp. (°F)	Chromel P-Alumel Sheathed Assembly[a] Deviation[b] (°F.)	Spread[c] (°F.)
0	1500	5.3	3.0 to 6.6
1000	1500	4.9	-0.2 to 6.6
2000	1500	5.1	0.8 to 7.4
3000	1500	2.9	-6.5 to 7.0
4000	1500	1.5	-11.5 to 7.8
5000	1500	2.4[d]	-10.0 to 8.5
6000	1500	1.9[d]	-17.0 to 11.5
7000	1500	0.8[d]	-20.0 to 8.0
8000	1500	0.7[d]	-27.5 to 9.9
0	1300	7.3	6.0 to 8.8
1000	1300	8.1	5.0 to 10.0
2000	1300	5.7	2.0 to 8.0
3000	1300	5.6	-3.8 to 8.5
4000	1300	5.8	-8.5 to 8.0
5000	1300	3.8[d]	-6.0 to 9.7
6000	1300	2.7[d]	-10.5 to 9.0
7000	1300	3.5[d]	-14.4 to 10.1
8000	1300	0.9[d]	-23.0 to 9.2
0	1100	6.8	5.5 to 8.1
1000	1100	6.9	4.8 to 8.2
2000	1100	6.2	3.4 to 8.0
3000	1100	6.9	1.5 to 8.5
4000	1100	4.2	-14.0 to 9.5
5000	1100	4.9	-4.0 to 10.0
6000	1100	4.6	-5.0 to 9.5
7000	1100	4.2	-13.0 to 10.6
8000	1100	4.2	-3.1 to 10.5

a. Nineteen assemblies were aged and each assembly contained two thermocouples. All units were made from material that had been soaked for 24 hours in helium at 1350°F by the vendor.

b. The deviation of the average of 38 readings from the test temperature.

c. The maximum spread of the 38 readings.

d. Represents aging data on 18 assemblies.

TABLE 2

Results of Aging Test of 0.250 Inch O.D. Sheathed Thermocouples in Fluoride Salt

		Chromel P-Alumel[a.] Sheathed Assemblies		Chromel P-Alumel[d.] Sheathed Assemblies		Pt-Pt, 10% Rh[e.] Sheathed Assemblies	
Time (Hr.)	Nominal Test Temp. (°F.)	Deviation[b.] (°F.)	Spread[c.] (°F.)	Deviation[b.] (°F.)	Spread[c.] (°F.)	Deviation[b.] (°F.)	Spread[c.] (°F.)
0	1500	1.9	0.5 to 4.0	2.9	2.6 to 3.0	-1.0	-2.5 to 2.5
1000	1500	2.7	0.0 to 6.0	5.1	4.6 to 5.6	0.4	0.0 to 1.0
2000	1500	2.2	-11.0 to 6.5	7.3	7.0 to 7.7	-0.6	-4.0 to 1.0
3000	1500	0.2	-12.0 to 6.5	8.4	8.0 to 8.8	-1.4	-4.2 to 1.0
4000	1500	0.7	-14.5 to 7.5	8.0	7.5 to 8.4	0.7	-5.0 to 2.5
5000	1500	2.0	-10.5 to 7.6	8.2	7.6 to 8.8	0.4	-1.1 to 1.8
6000	1500	1.4	-10.0 to 6.0	9.2	8.9 to 9.5	1.0	-3.0 to 1.2
0	1300	7.0	5.0 to 8.0	6.9	6.9 to 7.0	-0.1	-1.0 to 1.7
1000	1300	4.3	-0.5 to 7.6	6.9	6.5 to 7.4	-1.2	-2.3 to -0.2
2000	1300	2.5	-6.2 to 8.0	7.8	7.6 to 8.0	-0.6	-2.7 to 1.5
3000	1300	1.9	-12.0 to 8.5	8.8	8.5 to 9.2	-0.8	-3.0 to 1.2
4000	1300	3.8	-5.5 to 8.2	8.8	8.2 to 9.4	-0.6	-3.5 to 1.8
5000	1300	3.6	-8.5 to 8.4	9.1	8.5 to 9.6	0.3	-1.5 to 2.1
6000	1300	2.3	-9.0 to 7.3	8.4	8.0 to 8.8	0.1	-1.2 to 2.5
0	1100	4.2	3.0 to 5.4	4.6	4.5 to 4.8	-2.1	-4.2 to -0.2
1000	1100	5.2	1.4 to 7.8	6.4	6.0 to 6.8	-0.3	-0.9 to 0.4
2000	1100	2.8	-5.0 to 8.2	7.7	7.2 to 8.2	-0.5	-1.5 to 1.2
3000	1100	2.4	-10.5 to 8.0	8.3	8.0 to 8.6	-0.3	-2.0 to 1.2
4000	1100	2.7	-9.5 to 7.8	8.1	7.5 to 8.6	1.0	-1.0 to 2.8
5000	1100	4.7	-6.5 to 8.5	8.7	7.5 to 10.0	1.5	0.2 to 2.9
6000	1100	6.1	-4.5 to 10.2	10.6	9.7 to 11.4	3.0	3.3 to 5.4

a. Eleven assemblies were tested and each assembly contained two thermocouples. All units were made from material that had been soaked for 24 hours in helium at 1350°F. by the vendor.

b. Deviation of the average of the thermocouple readings from the test temperature.

c. The maximum spread of all thermocouple readings.

d. Two assemblies were tested and each assembly contained two thermocouples. Both units were made from material that had been soaked for 200 hours in helium at 1350°F. by the vendor.

e. Three assemblies were tested and each assembly contained two thermocouples.

TABLE 3

Results of Aging Test in NaK of Five Well-Type Thermocouples[a]

Test Time (Hr.)	Nominal Test Temp. °F.	Well No. 1 Deviation (°F.)		Well No. 2 Deviation (°F.)		Well No. 3 Deviation (°F.)		Well No. 4 Deviation (°F.)		Well No. 5 Deviation (°F.)	
		TC 1	TC 2	TC 1	TC 2	TC 1	TC 2	TC 1	TC 2	TC 1	TC 2
0	1500	1.8	2.6	-2.4	-2.0	1.2	0.6	-2.6	-4.6	-0.4	0.2
500	1500	1.5	2.2	-3.3	-3.0	-0.4	-0.8	-4.8	-5.6	-0.4	-0.6
1000	1500	1.0	1.9	-3.6	-4.4	0.4	0.4	-4.6	-5.8	-1.4	-1.4
1500	1500	1.0	1.7	-4.4	-4.4	2.0	2.0	-3.2	-3.8	1.2	1.6
2000	1500	3.1	3.6	-2.8	-2.8	3.6	3.0	-1.6	-1.6	2.8	2.8
2500	1500	4.0	5.0	-1.6	-1.6	4.0	3.4	0.0	0.6	3.4	3.4
0	1300	-2.8	-2.8	-6.8	-6.8	-5.4	-5.8	-6.4	-6.0	-1.6	-1.2
500	1300	-2.7	-3.3	-2.0	-2.0	-4.2	-4.4	-5.6	-6.0	-4.0	-4.2
1000	1300	-2.5	-3.3	-0.4	-0.8	-0.4	-0.6	-7.4	-8.4	-1.6	-2.0
1500	1300	-2.2	-3.1	-1.2	-1.6	3.0	2.8	-2.4	-2.6	2.0	2.0
2000	1300	0.2	0.6	-0.8	-1.0	3.2	2.8	-1.6	-2.0	2.4	2.4
2500	1300	3.6	4.2	-0.5	0.8	3.8	2.8	-2.4	-3.0	2.8	2.8
0	1100	-1.5	-0.3	-4.2	-3.8	-2.0	-2.0	-2.2	-2.6	-0.8	0.6
500	1100	0.5	0.8	-4.0	-3.6	-3.4	-3.4	-3.4	-4.2	-3.6	-3.6
1000	1100	2.2	2.7	0.4	0.8	3.4	2.8	-3.2	-3.6	1.6	1.6
1500	1100	2.4	3.2	0.8	-0.2	3.6	3.2	-1.4	-2.4	3.0	2.8
2000	1100	3.6	4.2	1.0	0.4	3.6	3.2	-1.4	-2.0	3.0	2.8
2500	1100	4.6	5.1	1.2	1.2	3.6	3.2	-1.4	-1.8	3.0	2.6

[a] Each well contained two thermocouples made from Chromel P-Alumel wire (± 3/8 percent 530°F to 2300°F, solid, 20 AWG, bright finish).

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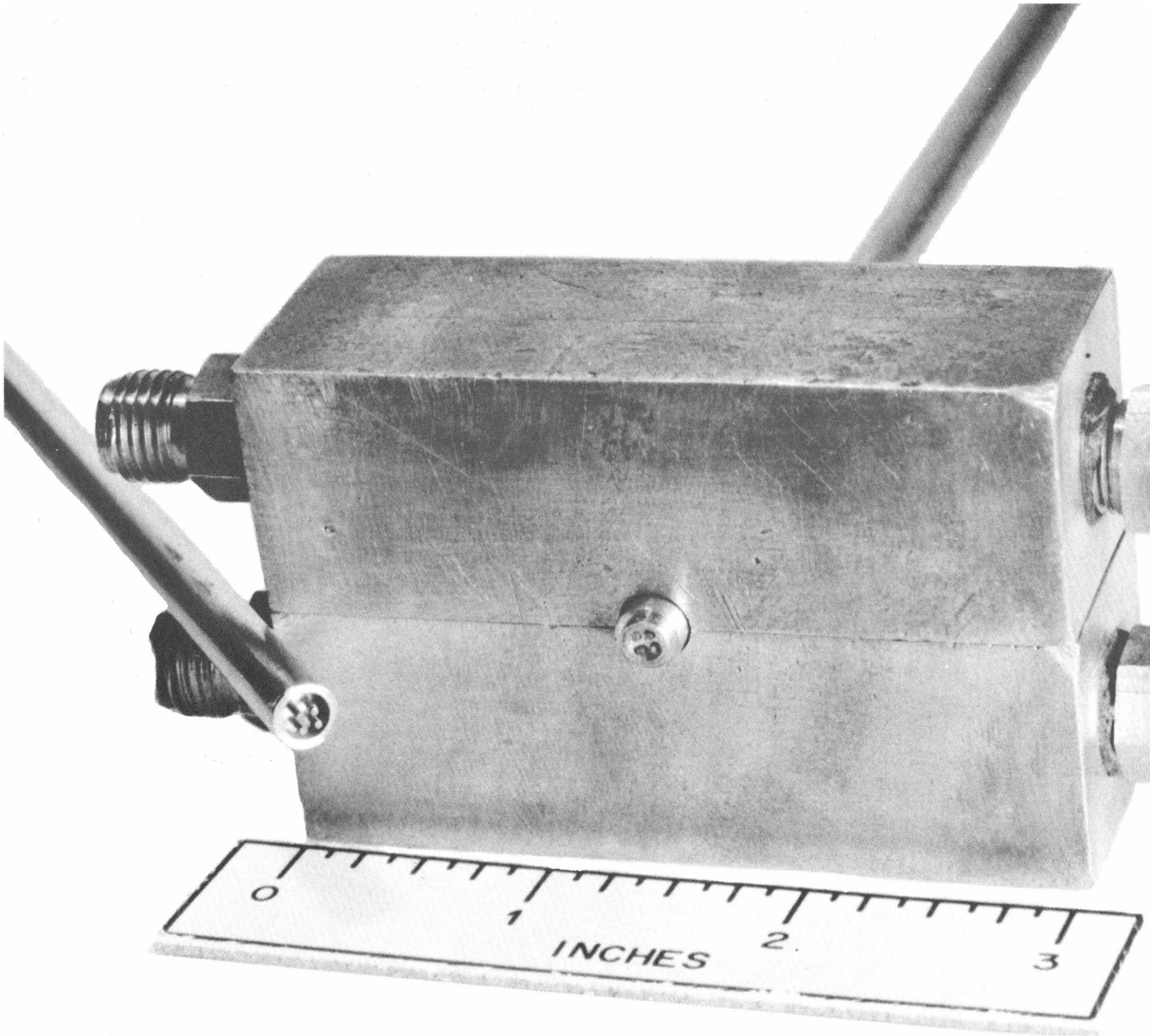

Figure 1. Setup for the Heliarc Closure Weld on 0.250-in.-O.D. Inconel-Sheathed Thermocouple Material Shown Mounted in a Water-Cooled Copper Chill Block with Inconel Filler Tip. (Also shown is the end of a piece of the material prepared for the welding operation.)

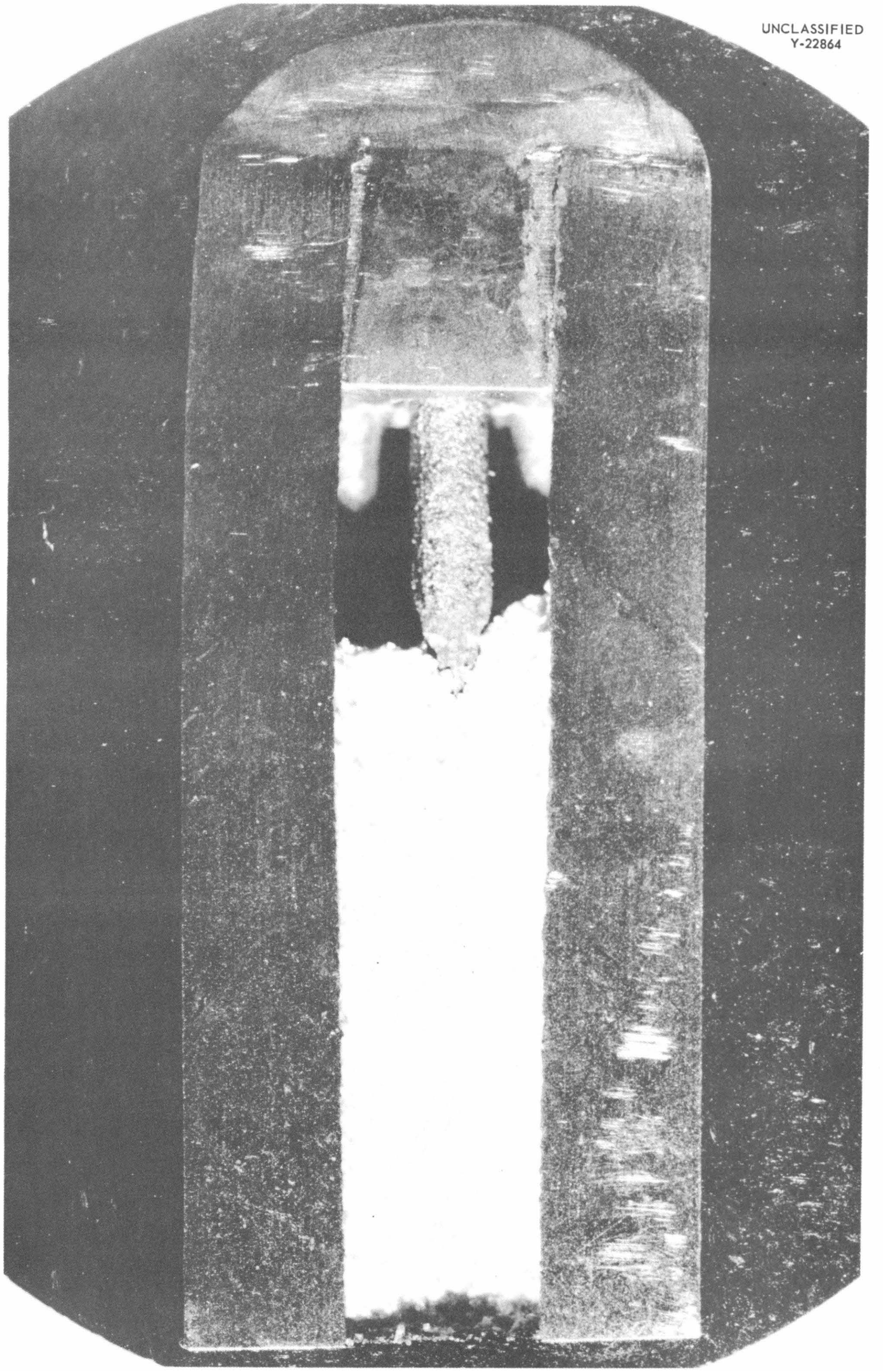

Figure 2. Cross-Section of Heliarc Closure Weld for 0.250-in.-O.D. Inconel-Sheathed Thermocouple Showing Inconel Filler Tip, One of Four Wires, Void Space and Magnesium Oxide Insulation.

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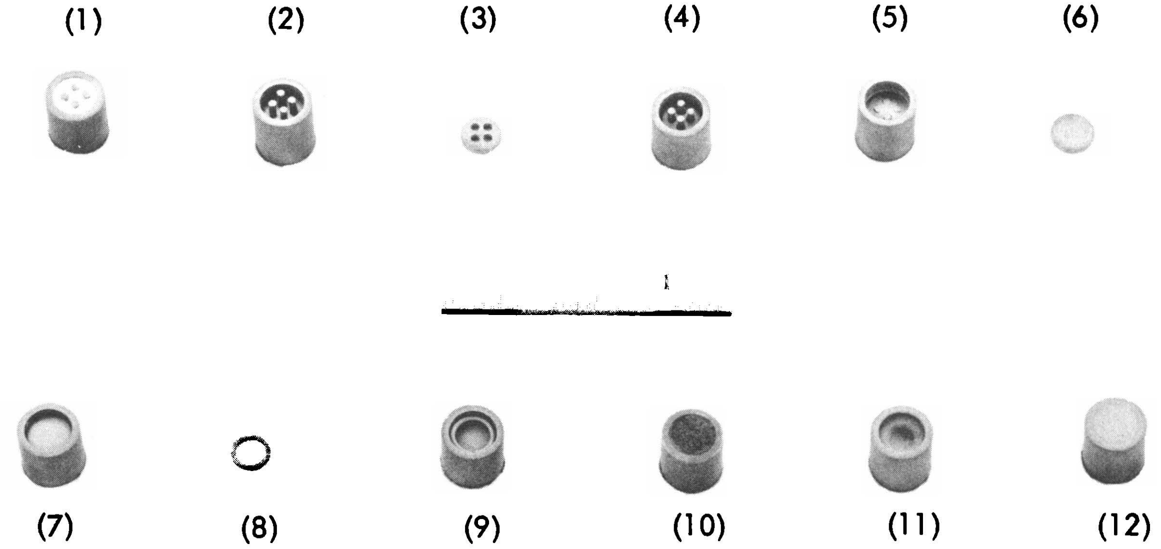

Figure 3(8). Steps in Fabrication of CM 52 Brazing Closure for 0.250-in.-OD Inconel-Sheathed Thermocouple (Top Row, Left to Right): (1) Cut, (2) Excavated, (3) Four-Hole Cap, (4) Cap Assembled, (5) Bored, (6) Cover; (Bottom Row, Left to Right): (7) Cover Assembled, (8) CM 52 Brazing Ring, (9) Ring Assembled or (10) CM 52 Powder, (11) After Brazing, (12) Ground to Cover.

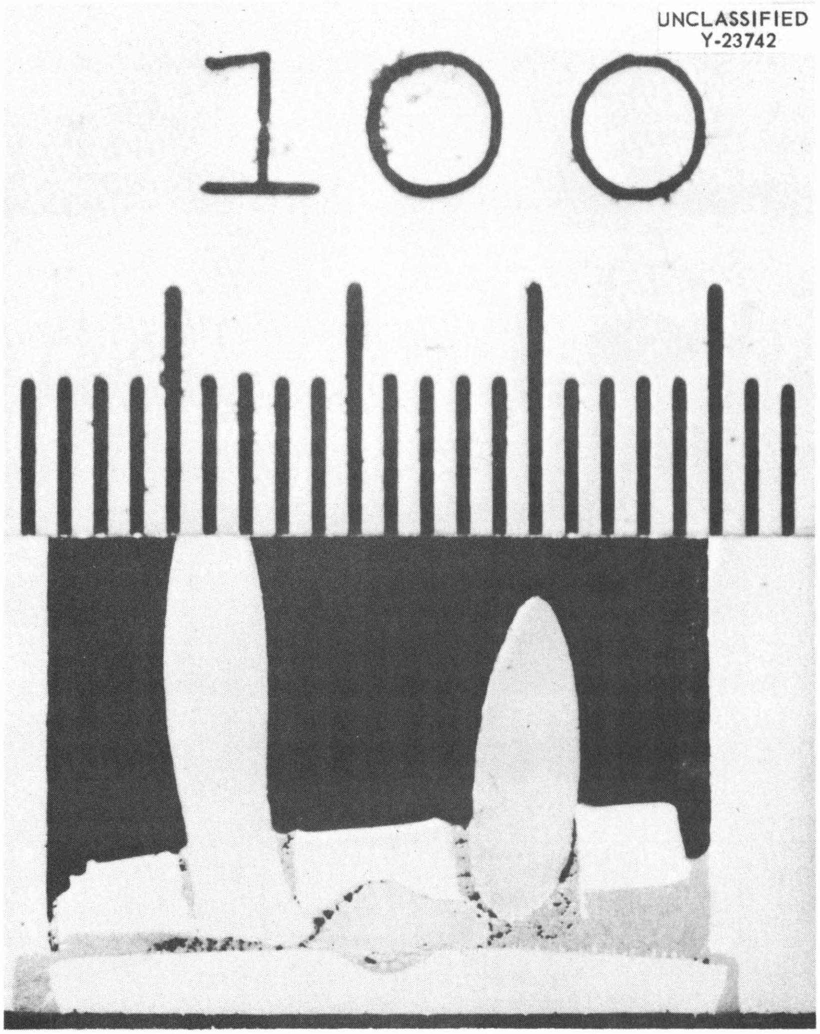

Figure 4(8). Cross-Section of CM 52 Brazed Closure for 0.250-in.-O.D. Inconel-Sheathed Thermocouple Showing Cover, Brazing Alloy, Four-Hole Cap, Two of Four Wires, Magnesium Oxide. (Note lack of void space and porosities around the wires.)

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Figure 5. Facility for NaK, Na, Fluoride Salt Aging and Calibration of 0.250-in.-O.D. Sheathed and Well-Type Thermocouples. (Uncompleted cabinet will contain the controls for a eutectic salt melting point apparatus.)

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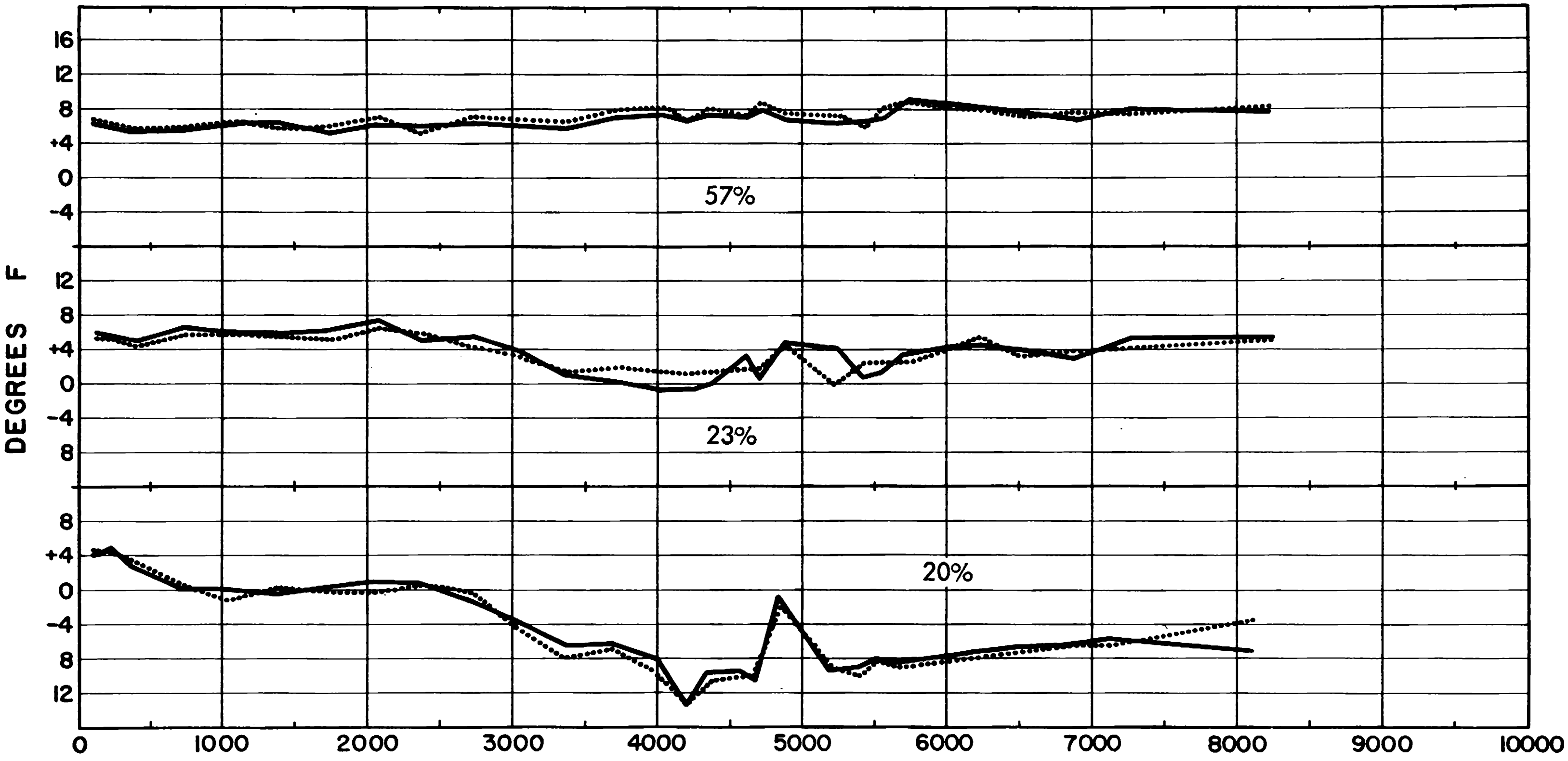

Figure 6. Typical Drift of 24-hr Soaked 0.250-in.-O.D. Inconel-Sheathed Chromel P-Alumel Thermocouple Material in Sodium and Fluoride Salt at 1500°F. (Note double curves represent the two couples contained in the sheath.)

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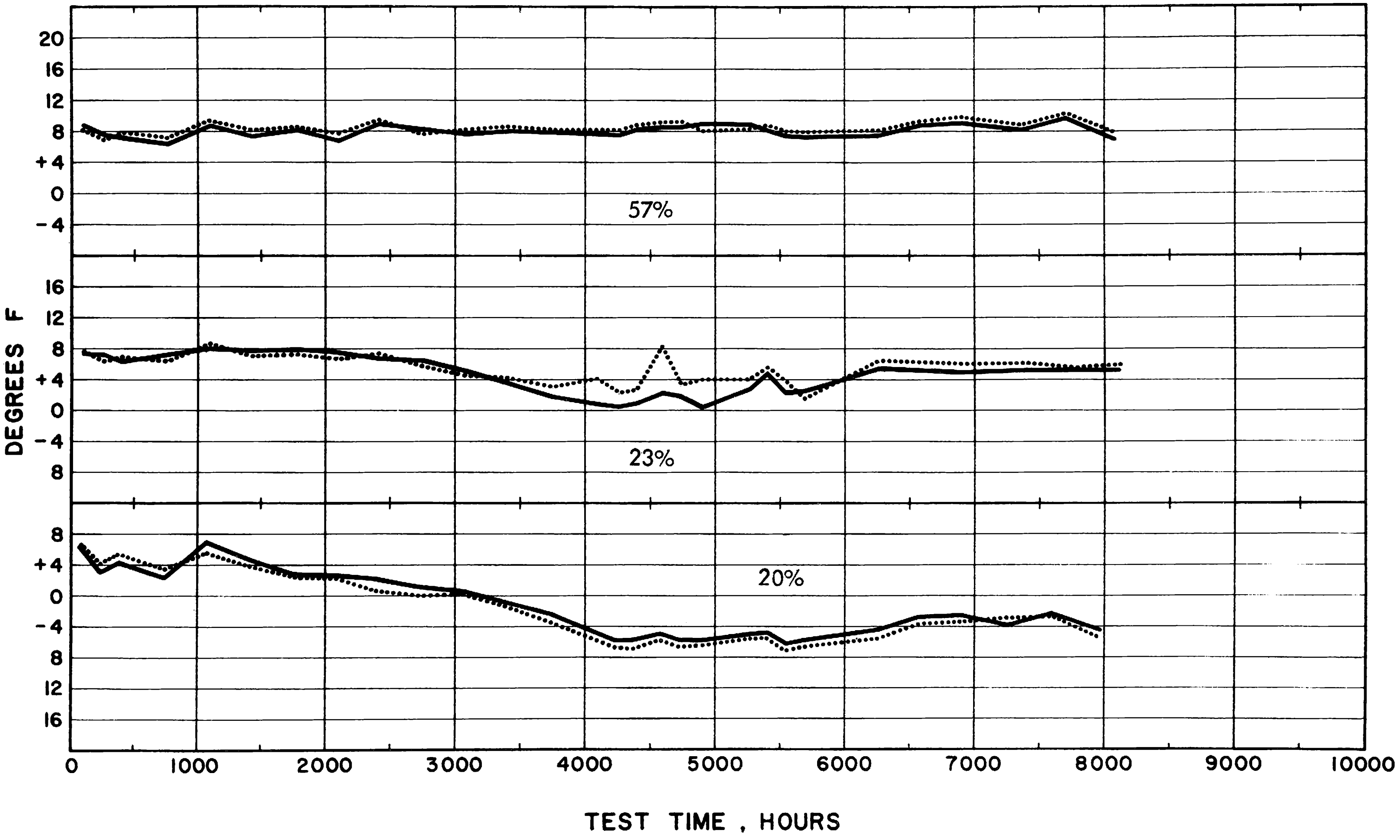

Figure 7. Typical Drift of 24-hr Soaked 0.250-in.-O.D. Inconel-Sheathed Chromel P-Alumel Thermocouple Material in Sodium and Fluoride Salt at 1300°F. (Note double curves represent the two couples contained in the sheath.)

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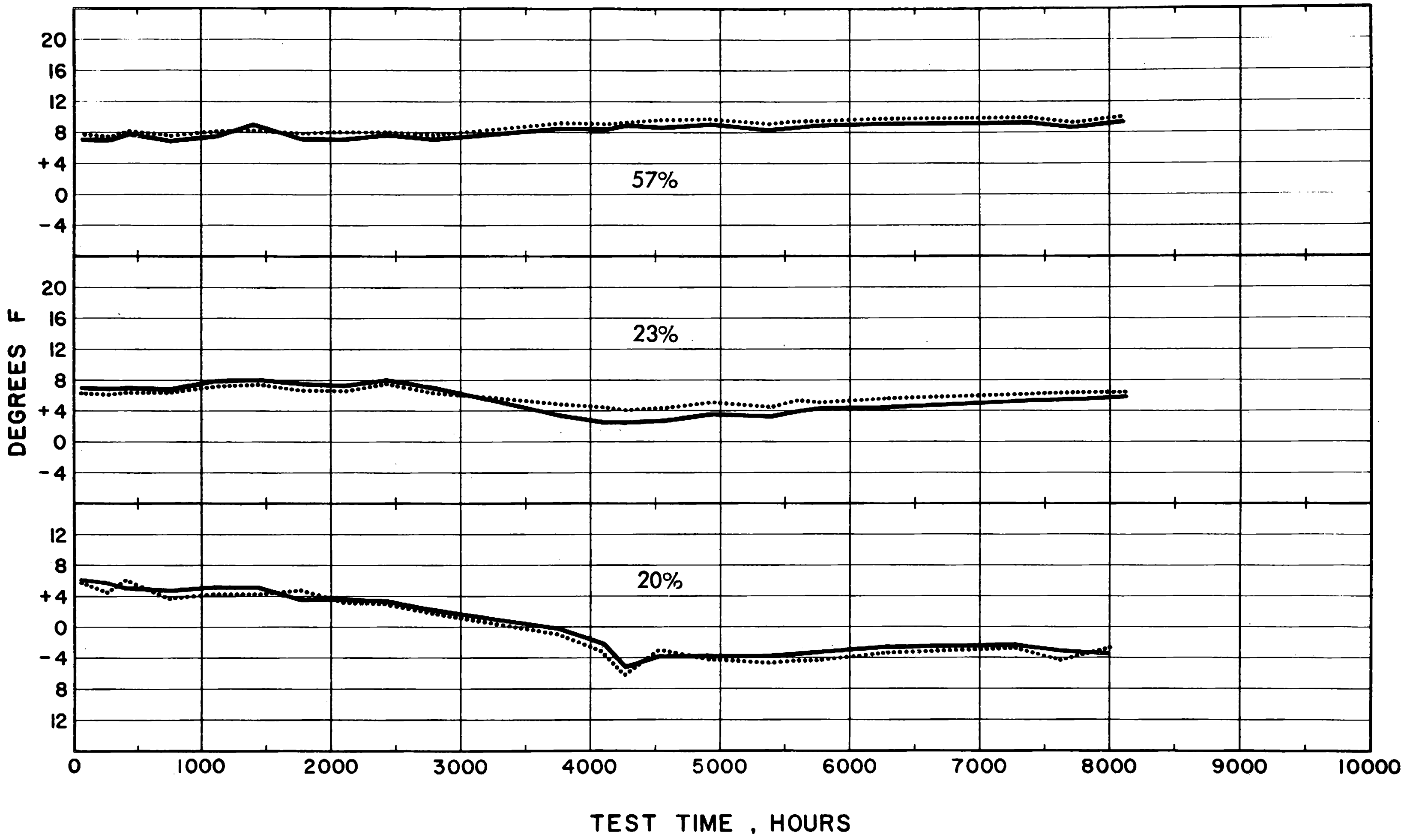

Figure 8. Typical Drift of 24-hr Soaked 0.250-in.-O.D. Inconel-Sheathed Chromel P-Alumel Thermocouple Material in Sodium and Fluoride Salt at 1100°F. (Note double curves represent the two couples contained in the sheath.)

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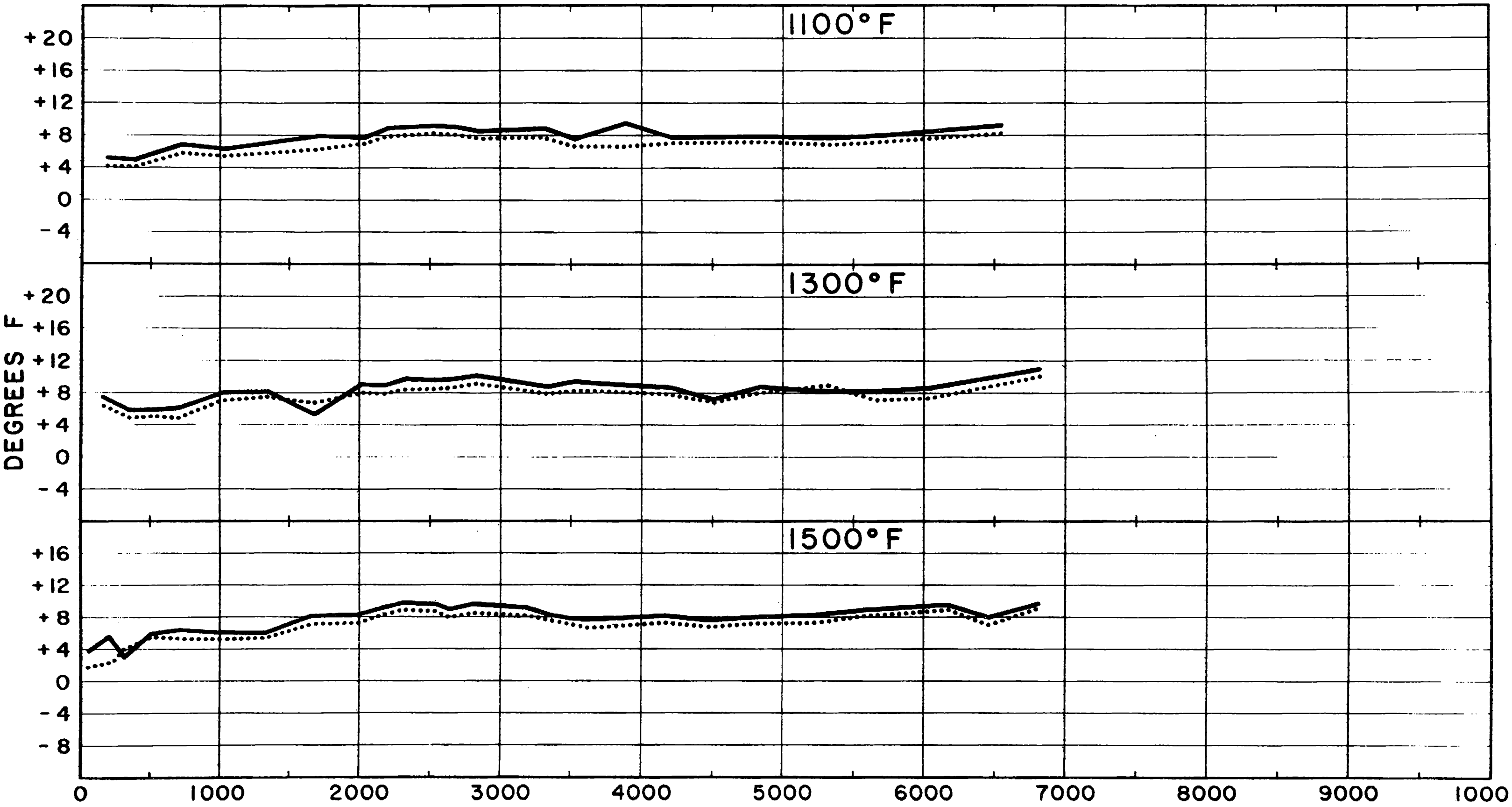

Figure 9. Typical Drift of 200-hr Soaked 0.250-in.-O.D. Inconel-Sheathed Chromel P-Alumel Thermocouple Material in Fluoride Salt at 1500, 1300, and 1100°F.

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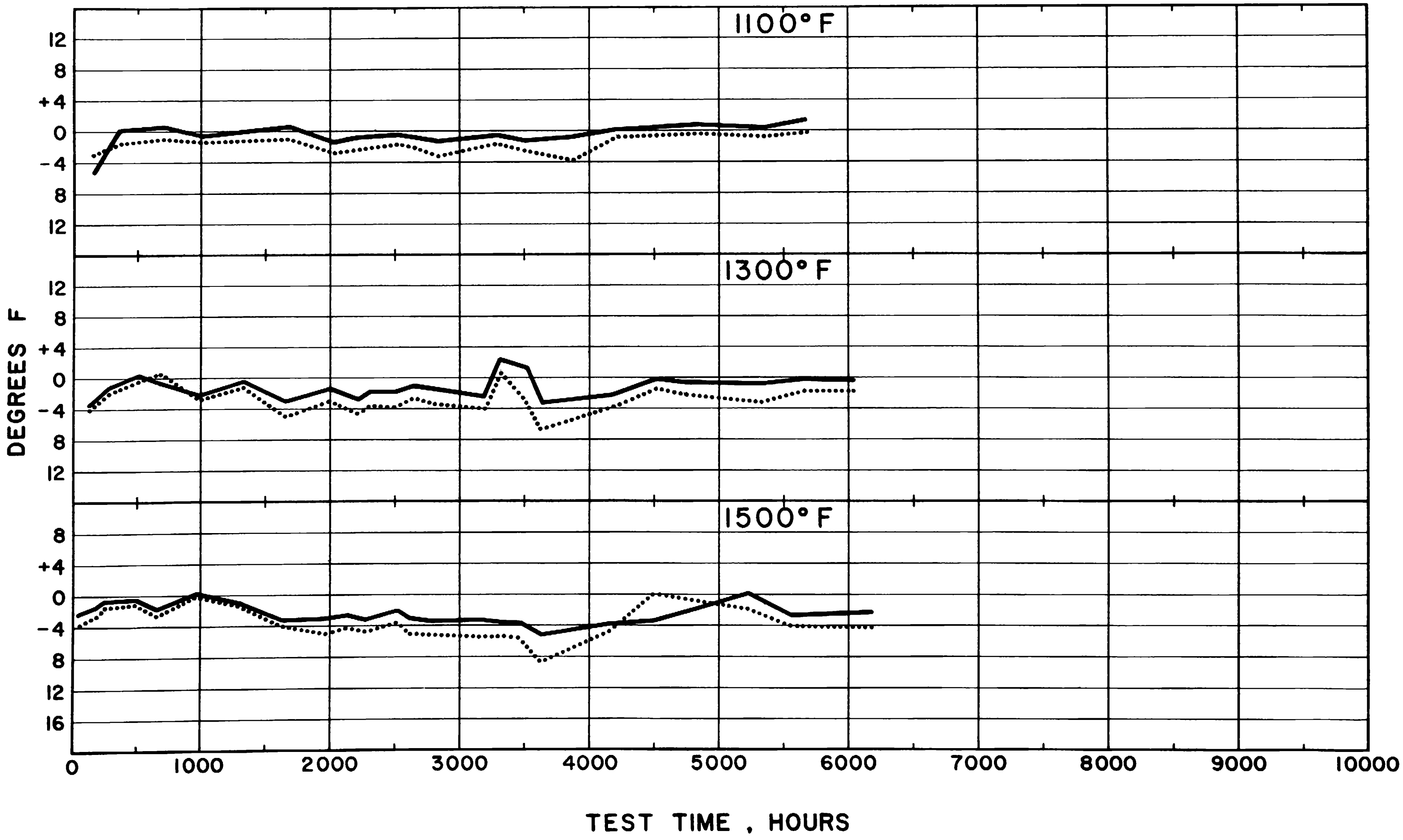

Figure 10. Typical Drift of 0.250-in.-O.D. Inconel-Sheathed Pt-Pt, 10% Rh Thermocouple Material in Sodium and Fluoride Salt. (Note double curves represent the two couples contained in the sheath.)

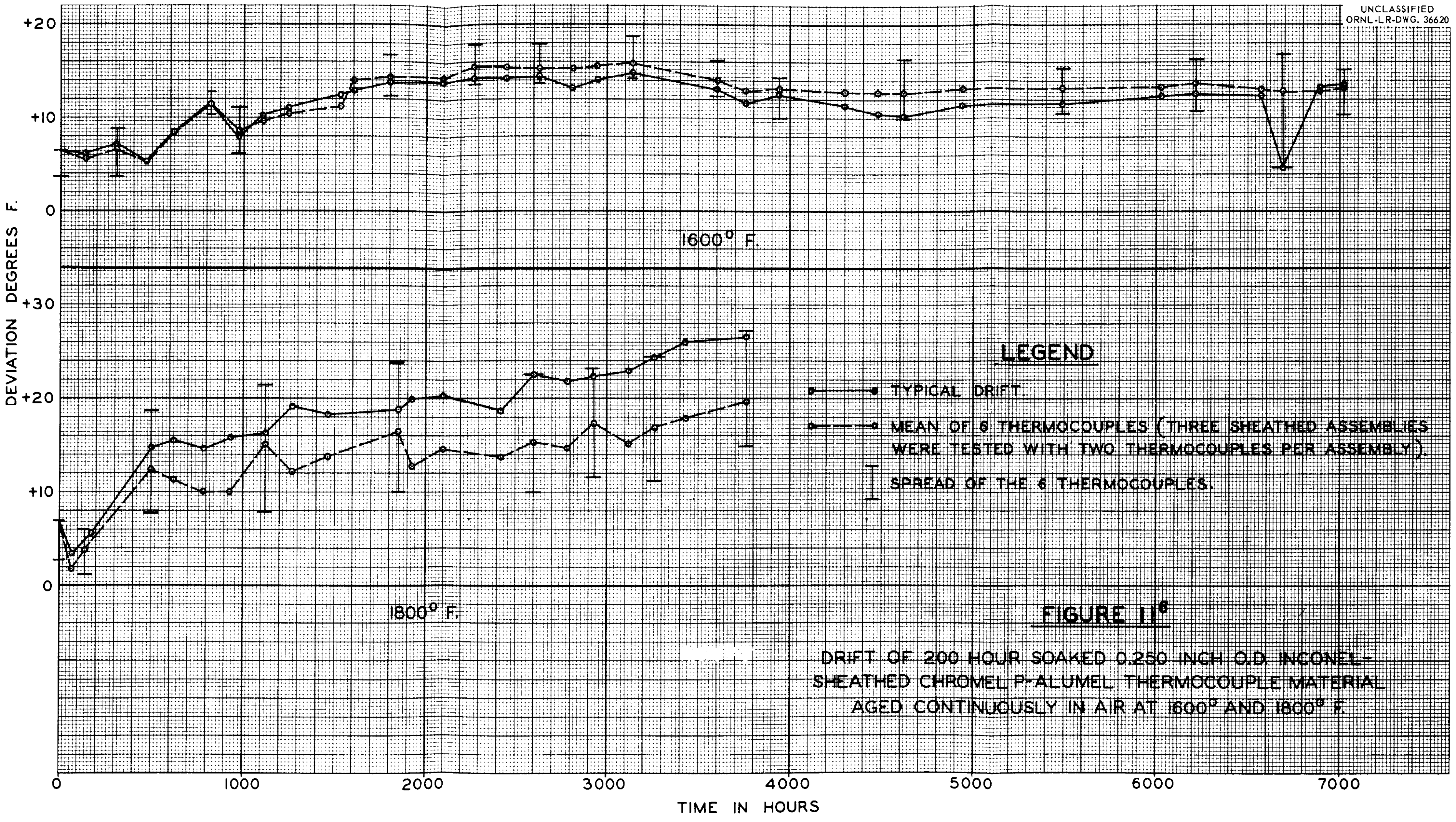

FIGURE 11[6]

DRIFT OF 200 HOUR SOAKED 0.250 INCH O.D. INCONEL-SHEATHED CHROMEL P-ALUMEL THERMOCOUPLE MATERIAL AGED CONTINUOUSLY IN AIR AT 1600° AND 1800° F.

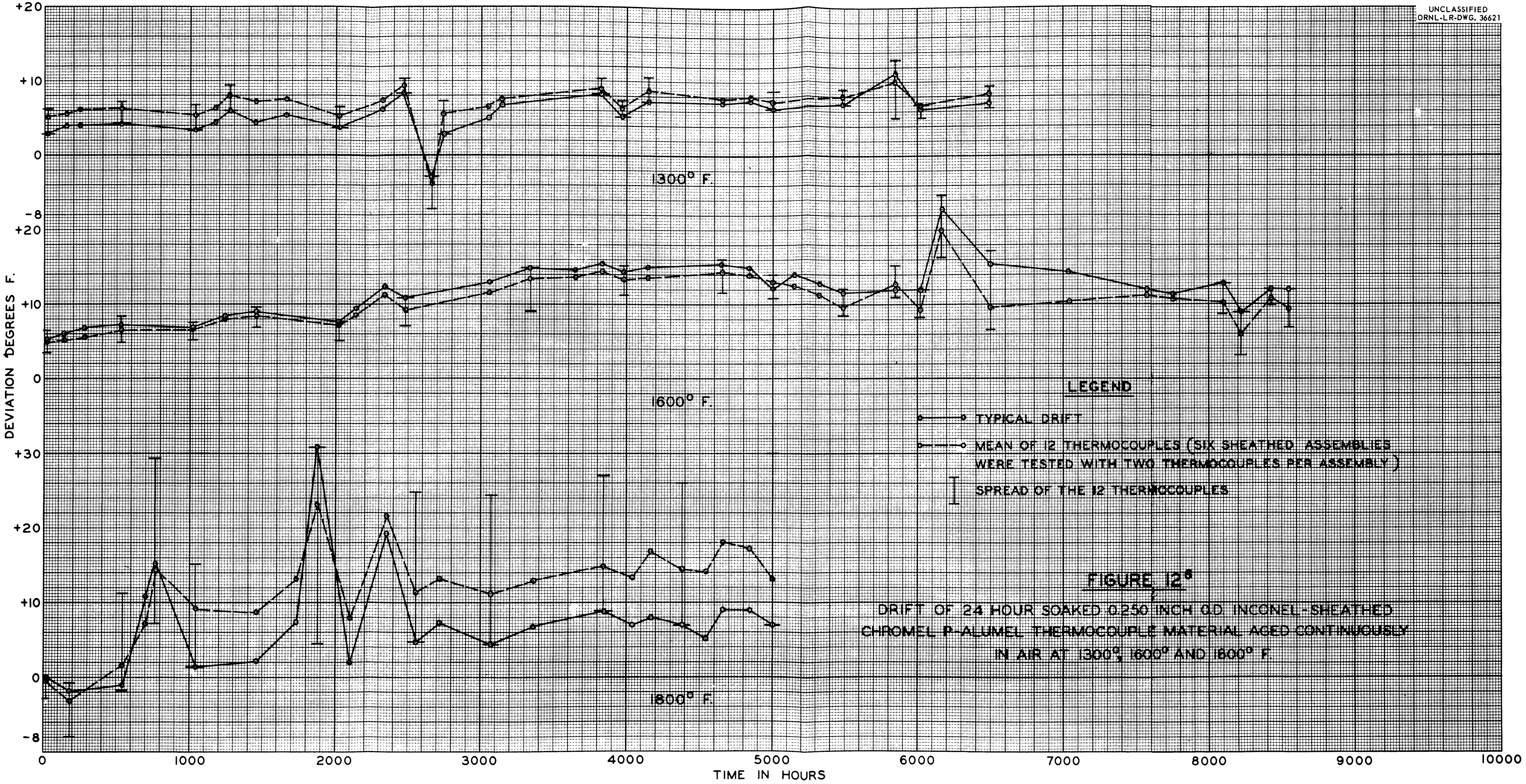

FIGURE 12^{B}

DRIFT OF 24 HOUR SOAKED 0.250 INCH O.D. INCONEL-SHEATHED CHROMEL P-ALUMEL THERMOCOUPLE MATERIAL AGED CONTINUOUSLY IN AIR AT 1300°, 1600° AND 1800° F.

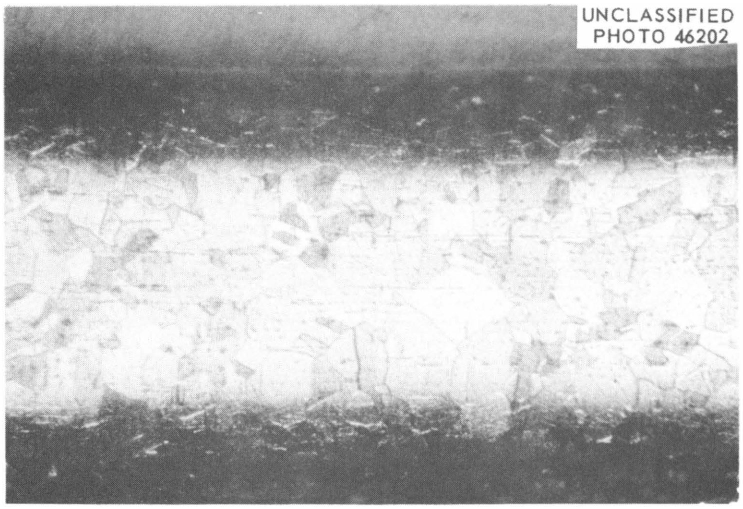

Figure 13(9). Cross-Section of Chromel P Wire from 0.250-in.-O.D. Inconel-Sheathed Thermocouple After 5000 hr at 1800°F. UT Sample No. 125. Etchant: $CuCl_2$ - aqua regia solution. 100X.

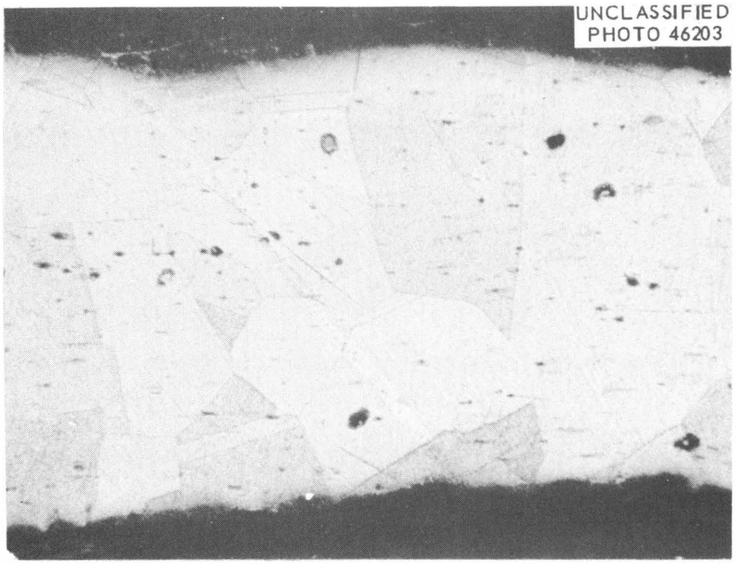

Figure 14(9). Cross-Section of Alumel Wire from 0.250-in.-O.D. Inconel-Sheathed Thermocouple After 5000 hr at 1800°F. UT Sample No. 124. Etchant: Chromic acid solution - $CuCl_2$ - aqua regia solution. 100X.

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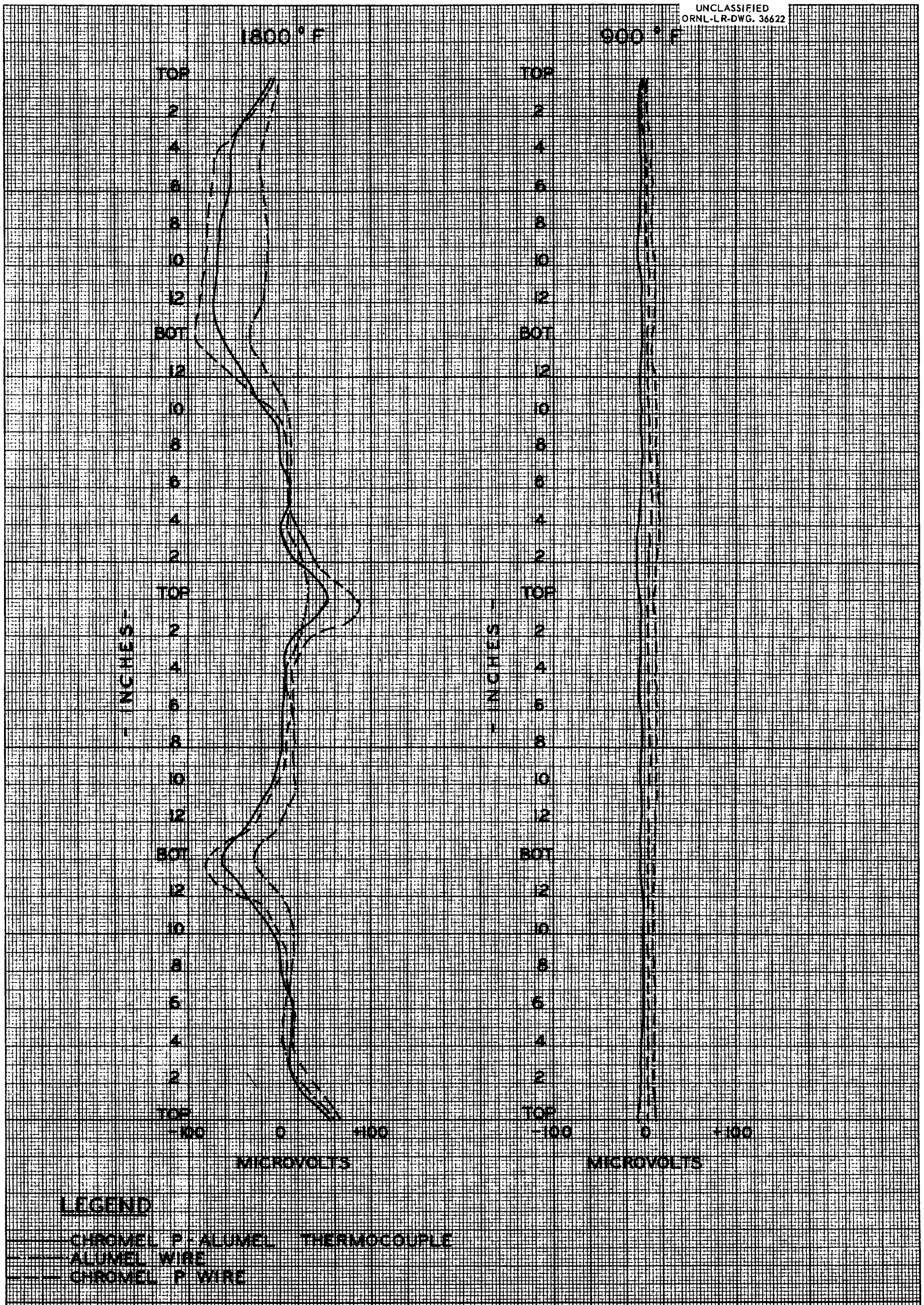

RESPONSE CURVES OF "AS RECEIVED" 24 HOUR SOAKED INCONEL-SHEATHED THERMOCOUPLE MATERIAL TO TRAVELING GRADIENT WITH 900°F AND 1800°F PEAK TEMPERATURES.

FIGURE 15B

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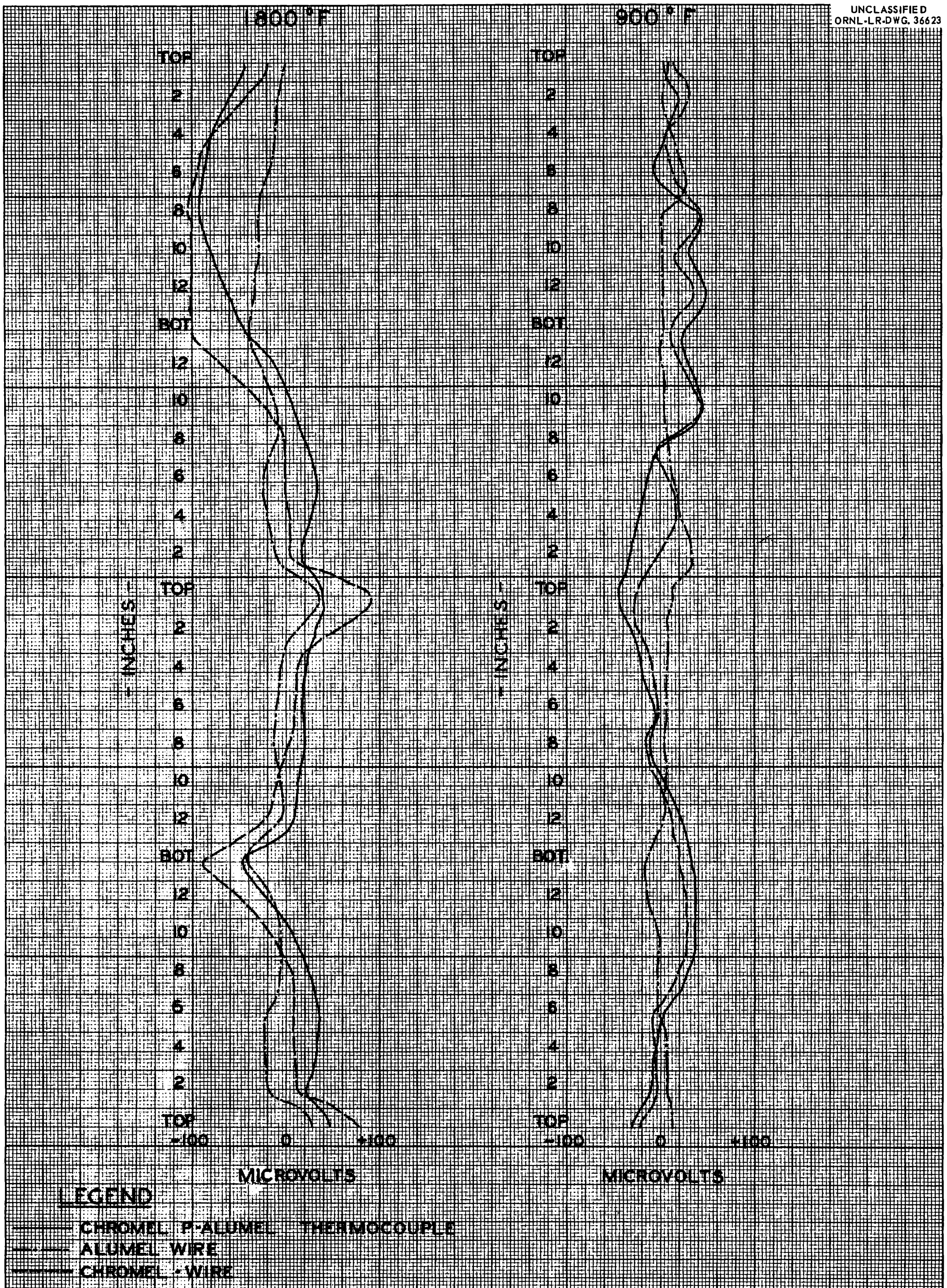

RESPONSE CURVES OF BENT 24 HOUR SOAKED 0.250 O.D. INCONEL-SHEATHED THERMOCOUPLE MATERIAL TO TRAVELING GRADIENT WITH 900 F AND 1800 F PEAK TEMPERATURES (MATERIAL WAS BENT 3 INCHES EITHER SIDE OF CENTER).

FIGURE 16^{a}

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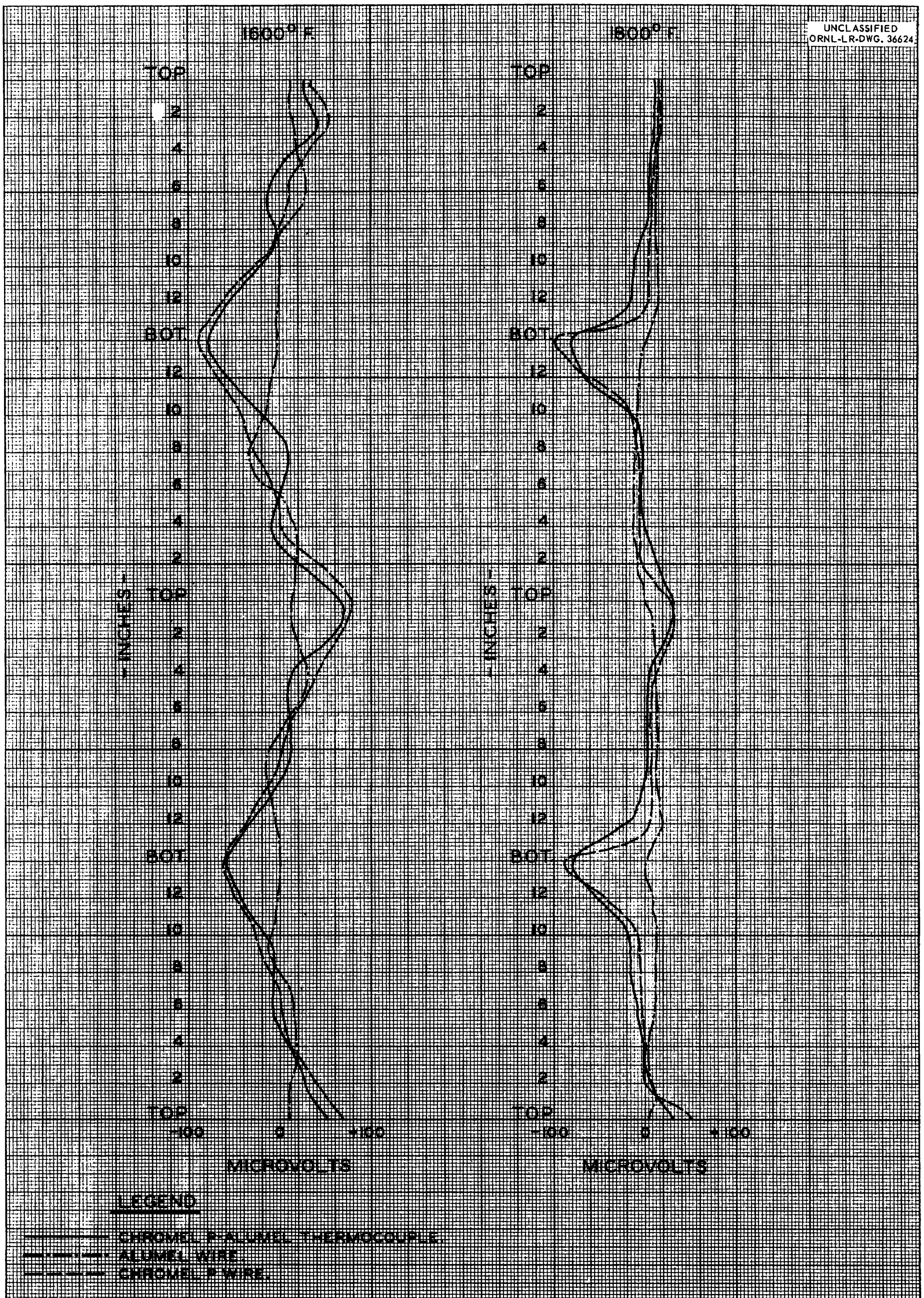

RESPONSE CURVES OF BENT AND ANNEALED 24 HOUR SOAKED INCONEL-SHEATHED THERMOCOUPLE MATERIAL TO TRAVELING GRADIENT WITH 1800° F. PEAK TEMPERATURE.

FIGURE 16 A[6]

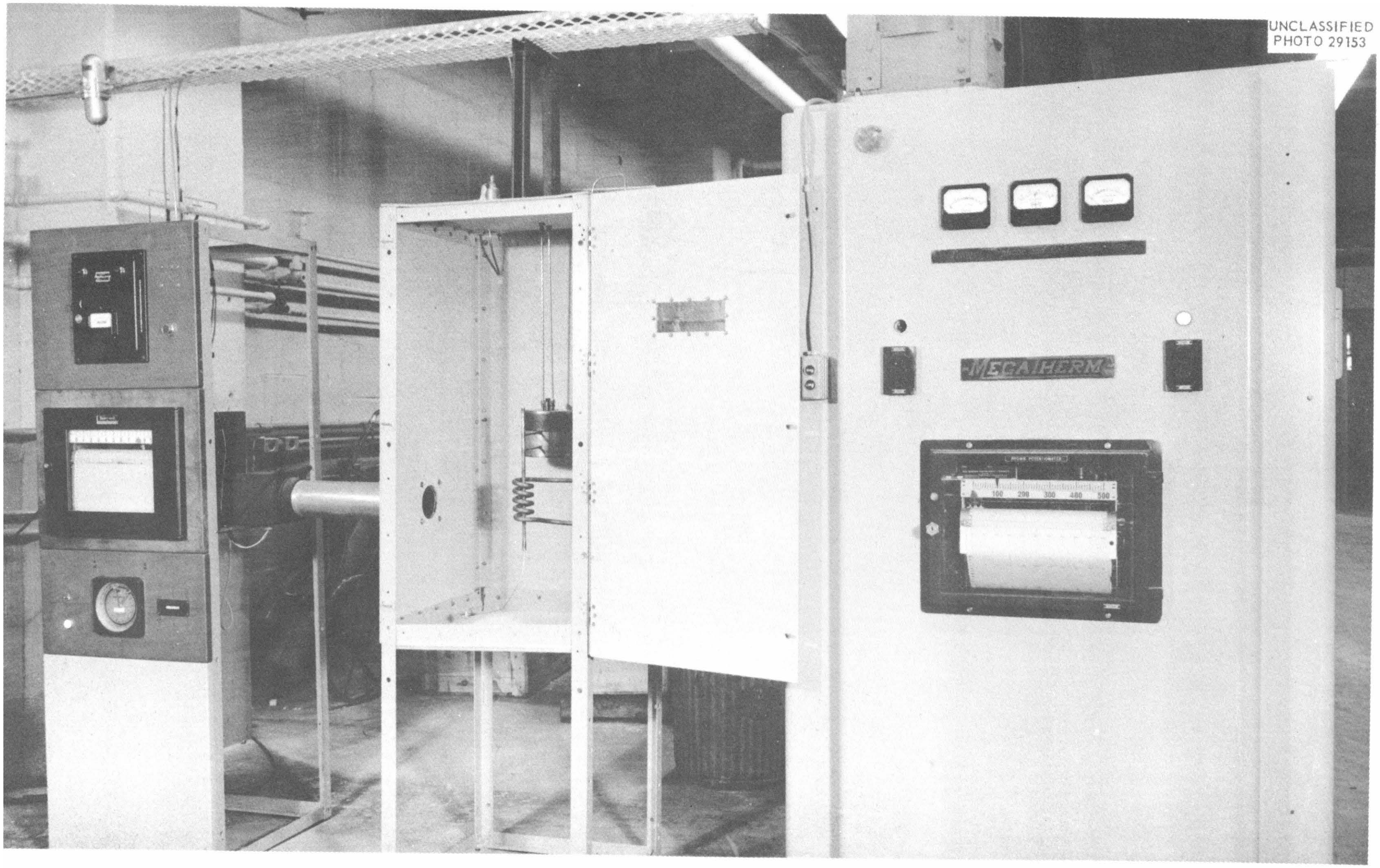

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Figure 17. Induction Heating Facility for Rapid Heating of 0.250-in.-O.D. Inconel-Sheathed Thermocouples Showing Instrument Control Cabinet on the Left, Test Cabinet in the Center and Main Control Cabinet on the Right.

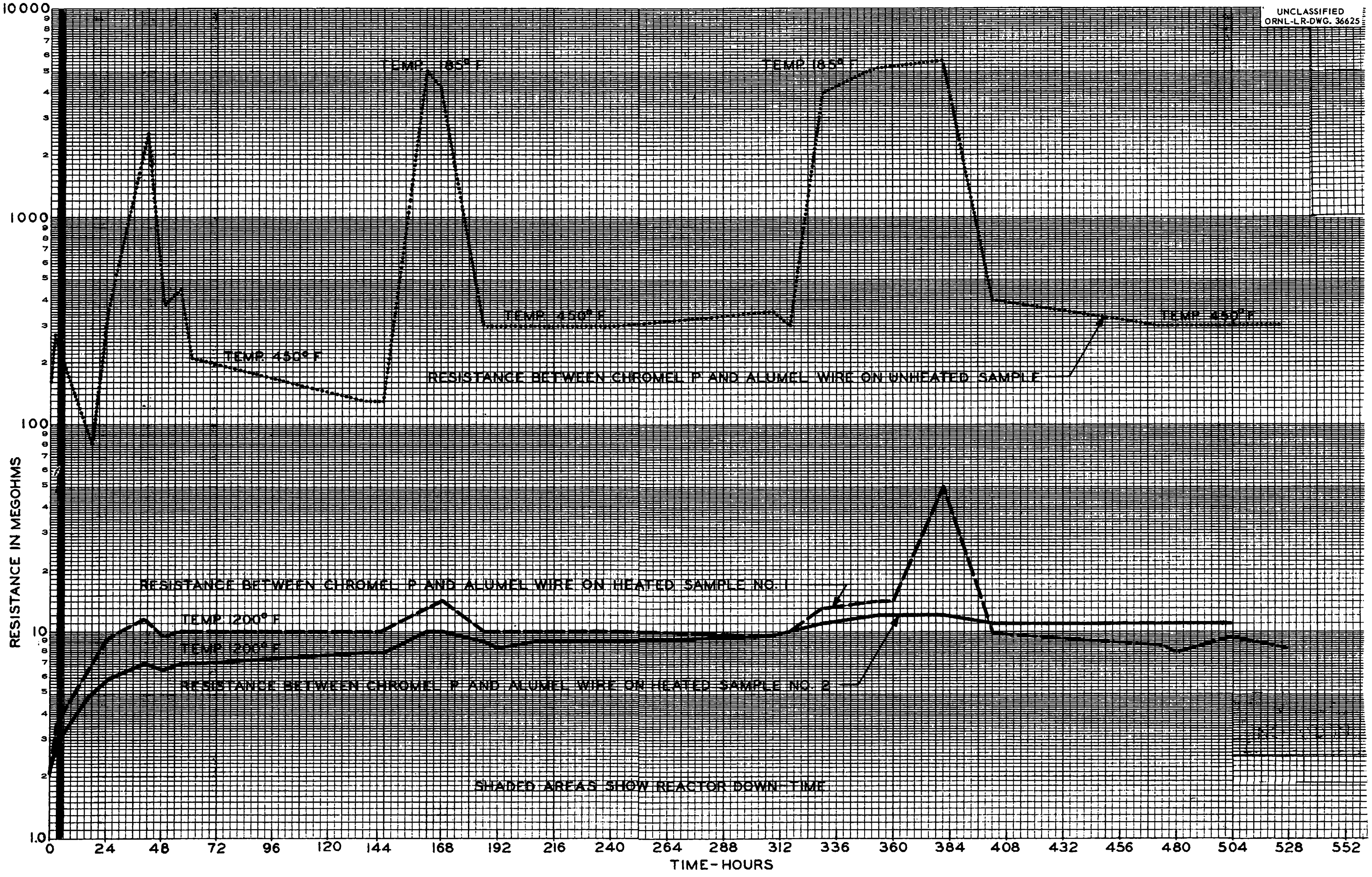

Figure 18[7]. Magnesium Oxide Insulation Resistance of 0.250-in.-O.D. Inconel-Sheathed Thermocouple Material vs Irradiation Time in HB-3 of the LITR.

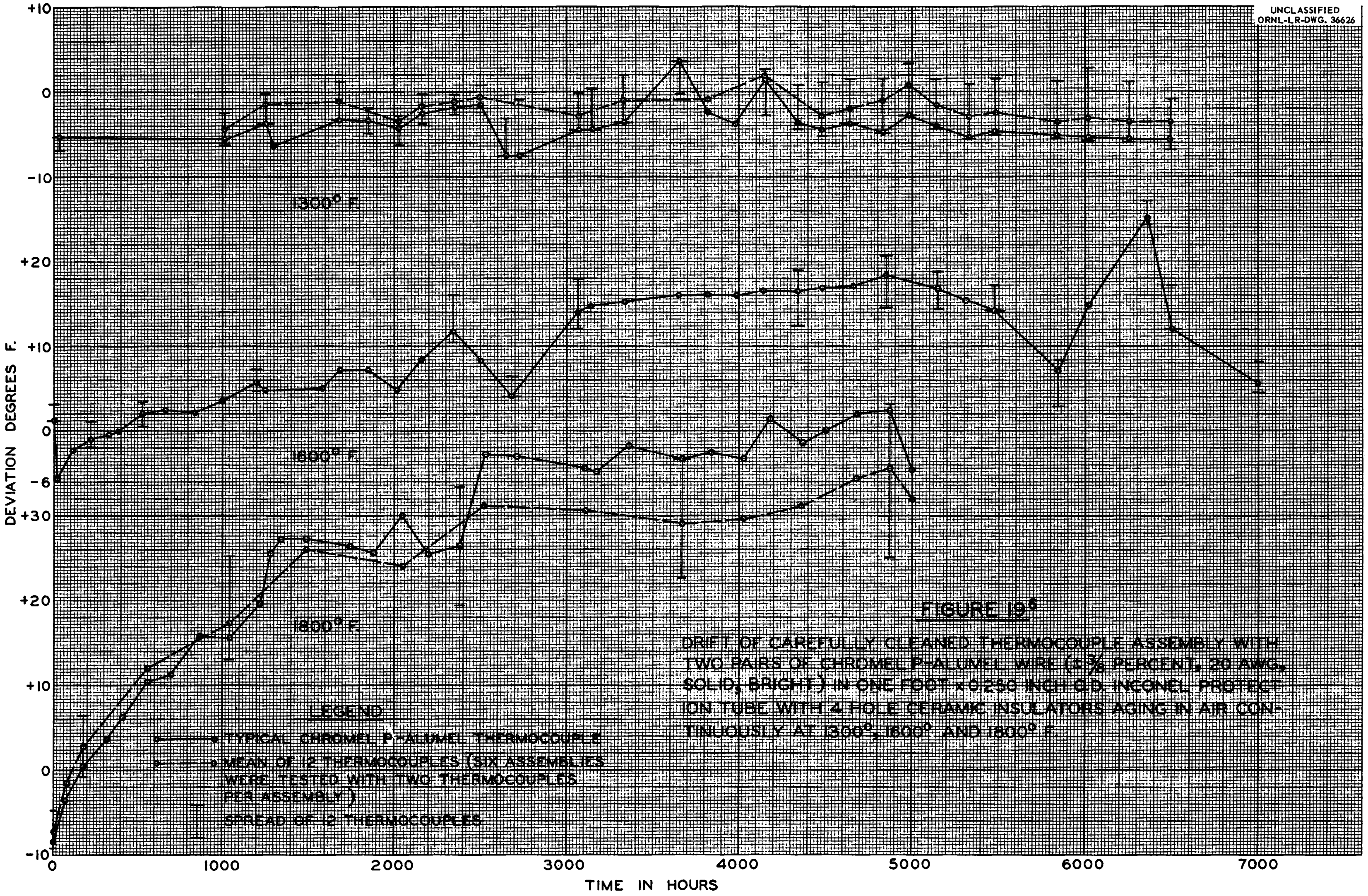

FIGURE 19[6]

DRIFT OF CAREFULLY CLEANED THERMOCOUPLE ASSEMBLY WITH TWO PAIRS OF CHROMEL P-ALUMEL WIRE (± 3/8 PERCENT, 20 AWG, SOLID, BRIGHT) IN ONE FOOT x 0.250 INCH O.D. INCONEL PROTECTION TUBE WITH 4 HOLE CERAMIC INSULATORS AGING IN AIR CONTINUOUSLY AT 1300°, 1600° AND 1800° F.

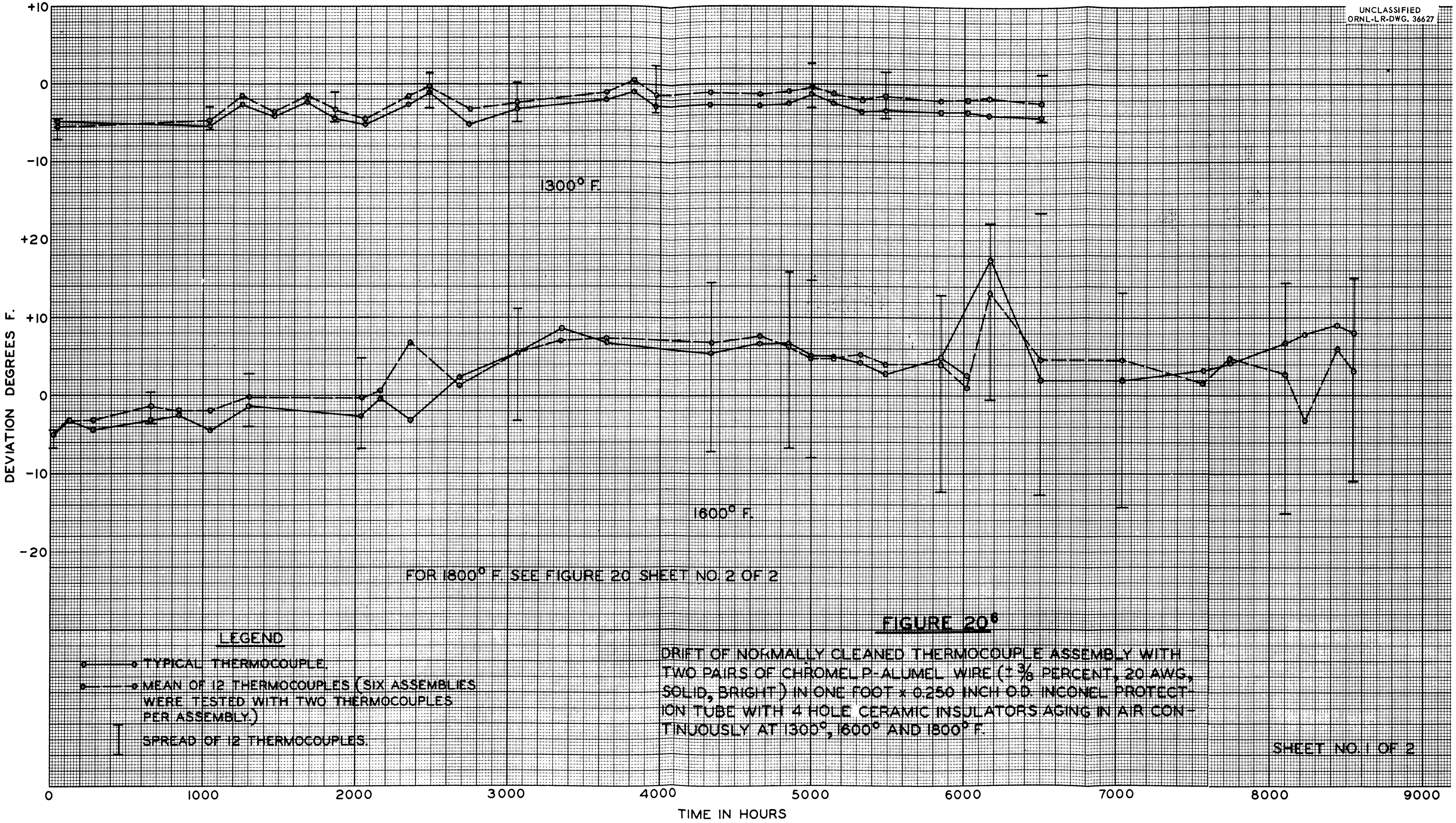

FIGURE 20[6]

DRIFT OF NORMALLY CLEANED THERMOCOUPLE ASSEMBLY WITH TWO PAIRS OF CHROMEL P-ALUMEL WIRE (± 3/8 PERCENT, 20 AWG, SOLID, BRIGHT) IN ONE FOOT x 0.250 INCH O.D. INCONEL PROTECTION TUBE WITH 4 HOLE CERAMIC INSULATORS AGING IN AIR CONTINUOUSLY AT 1300°, 1600° AND 1800° F.

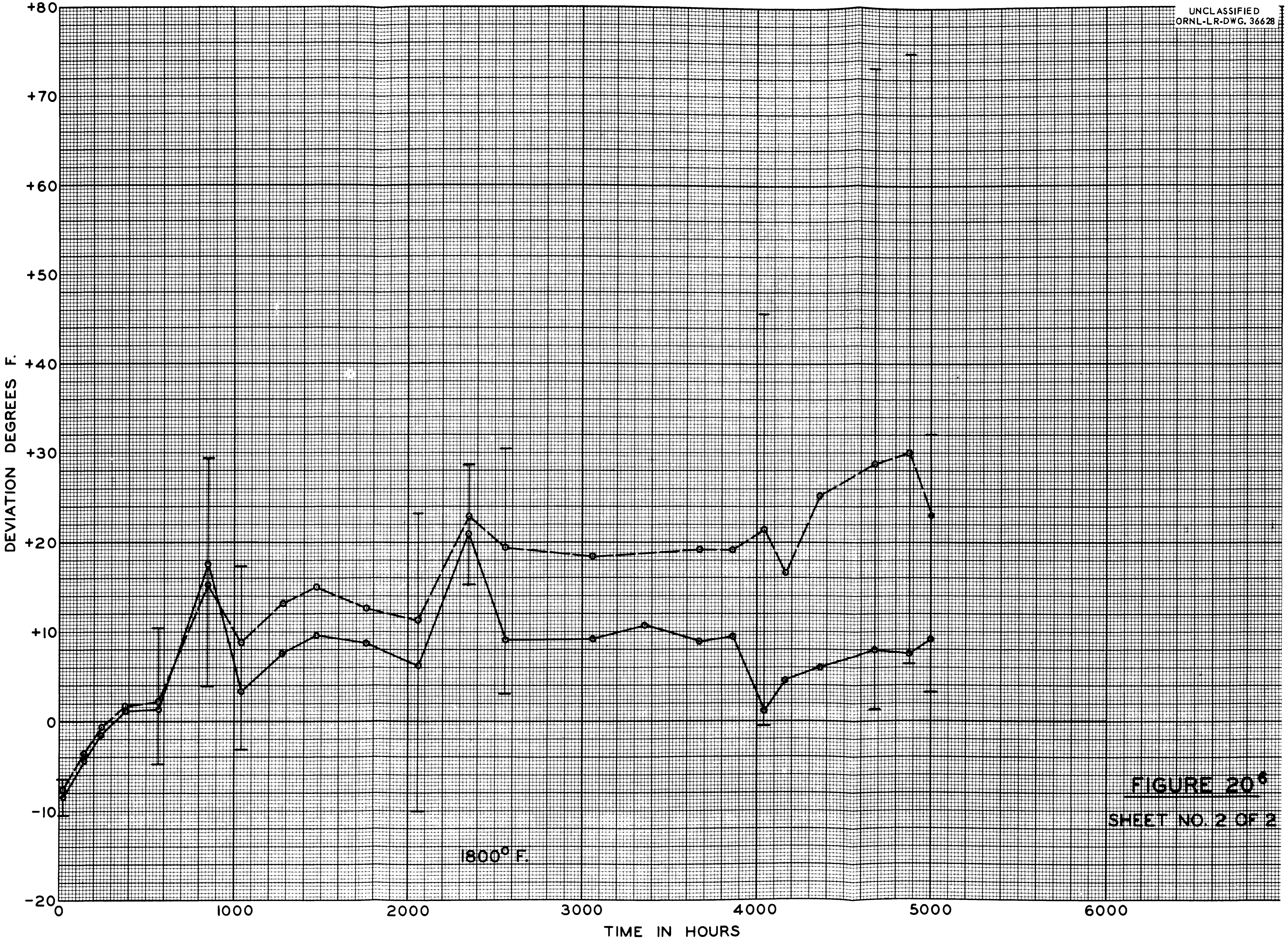

FIGURE 20^{6}

SHEET NO. 2 OF 2

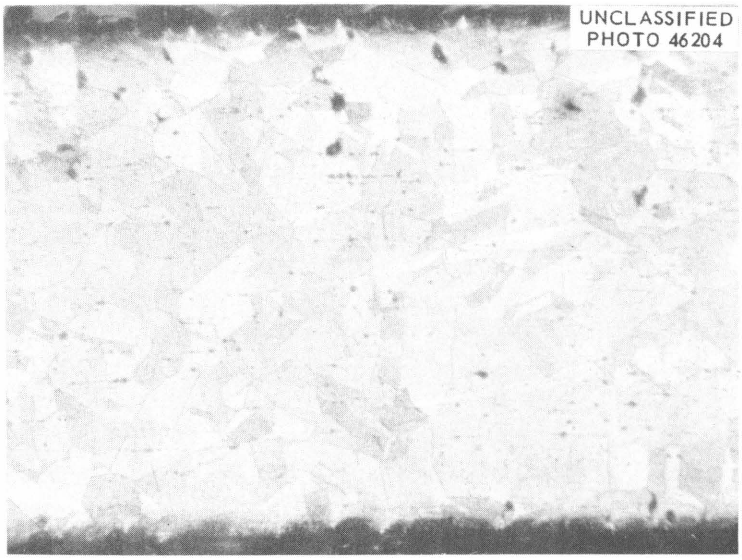

Figure 21(9). Cross-Section of Chromel P Wire Taken from Ceramic Beaded Assembly in One Foot, 0.250-in.-O.D. Inconel Protection Tube After 5000 hr at 1800°F. UT Sample No. 123. Etchant: $CuCl_2$ - aqua regia solution. 100X

Figure 22(9). Cross-Section of Alumel Wire (With Adhering Heavy Layer of Alumel Oxide and Ceramic Bead) Taken from Ceramic Beaded Assembly in One Foot, 0.250-in.-O.D. Inconel Protection Tube After 5000 hr at 1800°F. (Note oxide growth at the refractory break, unusual wire surface effects and the unusual oxide appearance.) UT Sample No. 122. Etchant: Chromic Acid Solution - $CuCl_2$ - aqua regia solution. 30X.

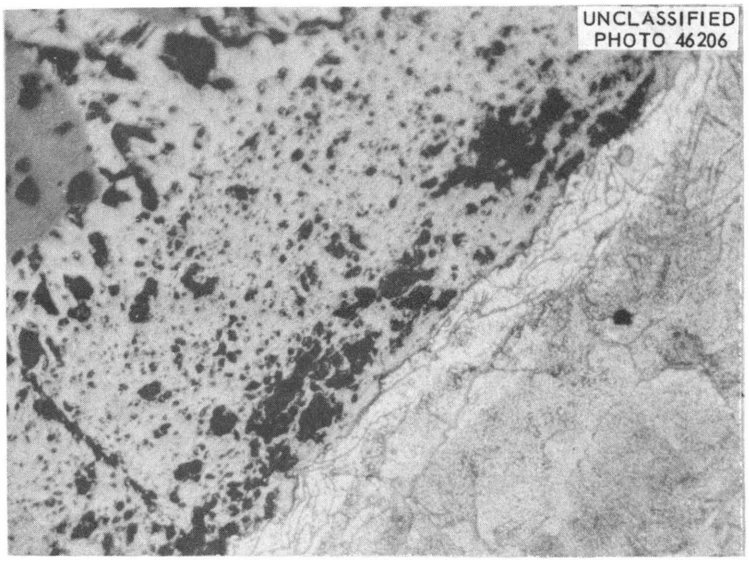

Figure 22A(9)

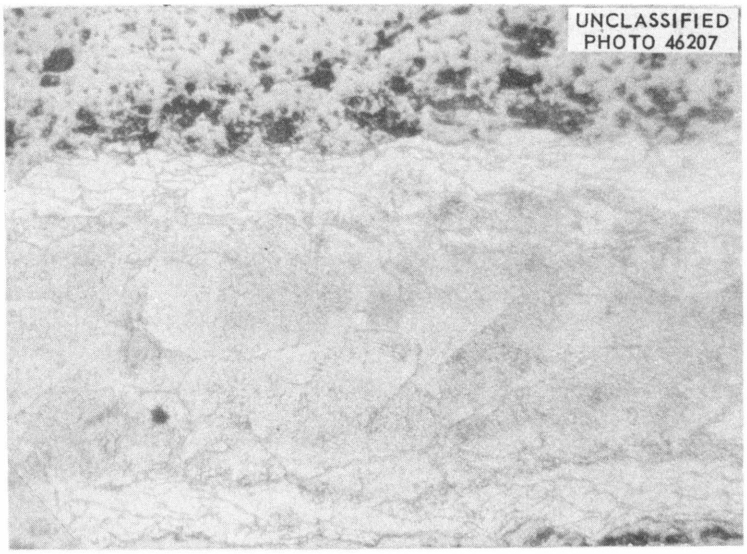

Figure 22B(9)

Greater Magnification of Cross-Section Shown in Figure 22 Showing the Alumel Wire and Alumel Oxide Interface. (Note the oxide metal penetration and the differences in metal microstructure and oxide structure.) 150X.

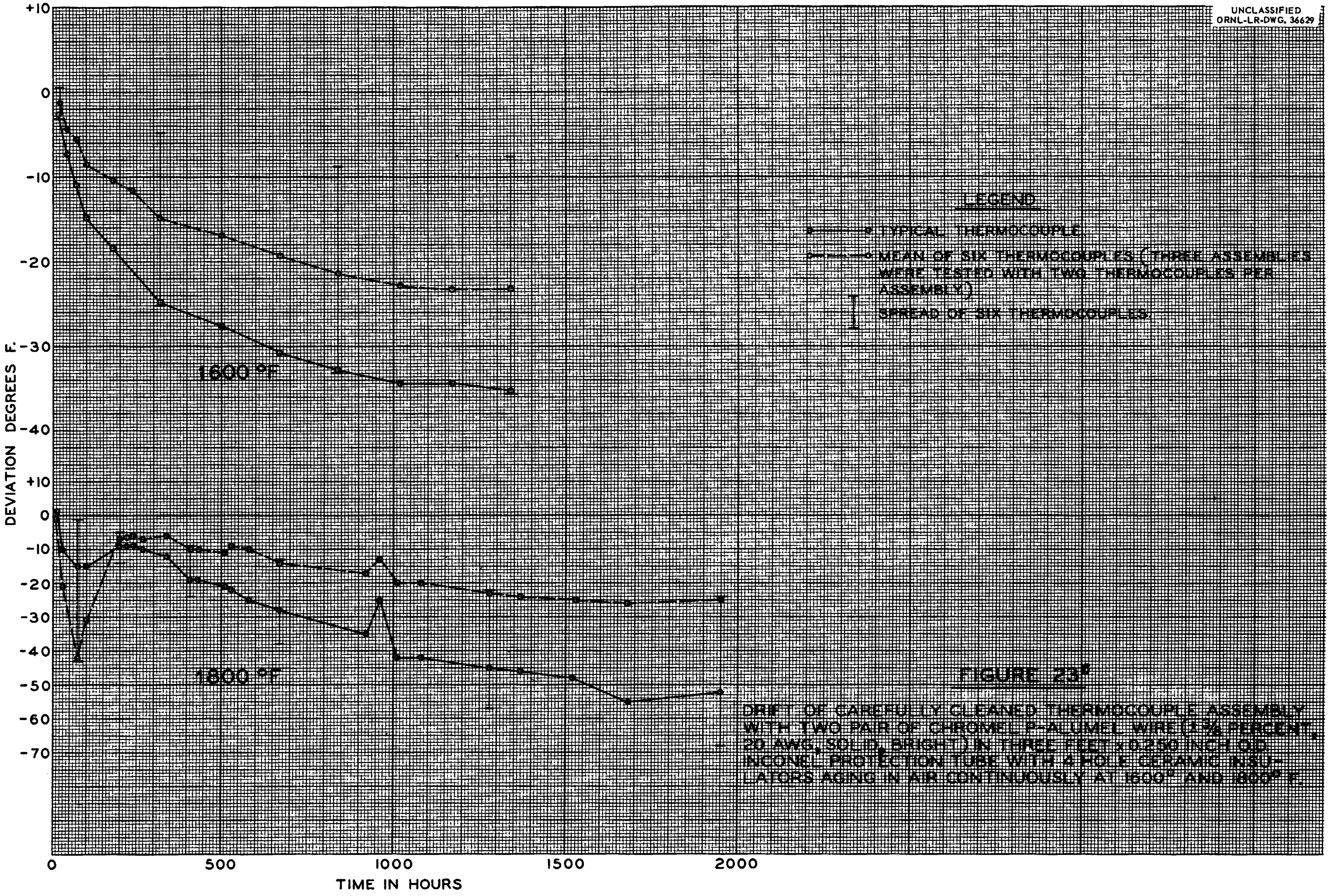

FIGURE 23

DRIFT OF CAREFULLY CLEANED THERMOCOUPLE ASSEMBLY WITH TWO PAIR OF CHROMEL P-ALUMEL WIRE (1¾ PERCENT, 20 AWG, SOLID, BRIGHT) IN THREE FEET x 0.250 INCH O.D. INCONEL PROTECTION TUBE WITH 4 HOLE CERAMIC INSULATORS AGING IN AIR CONTINUOUSLY AT 1600° AND 1800° F.

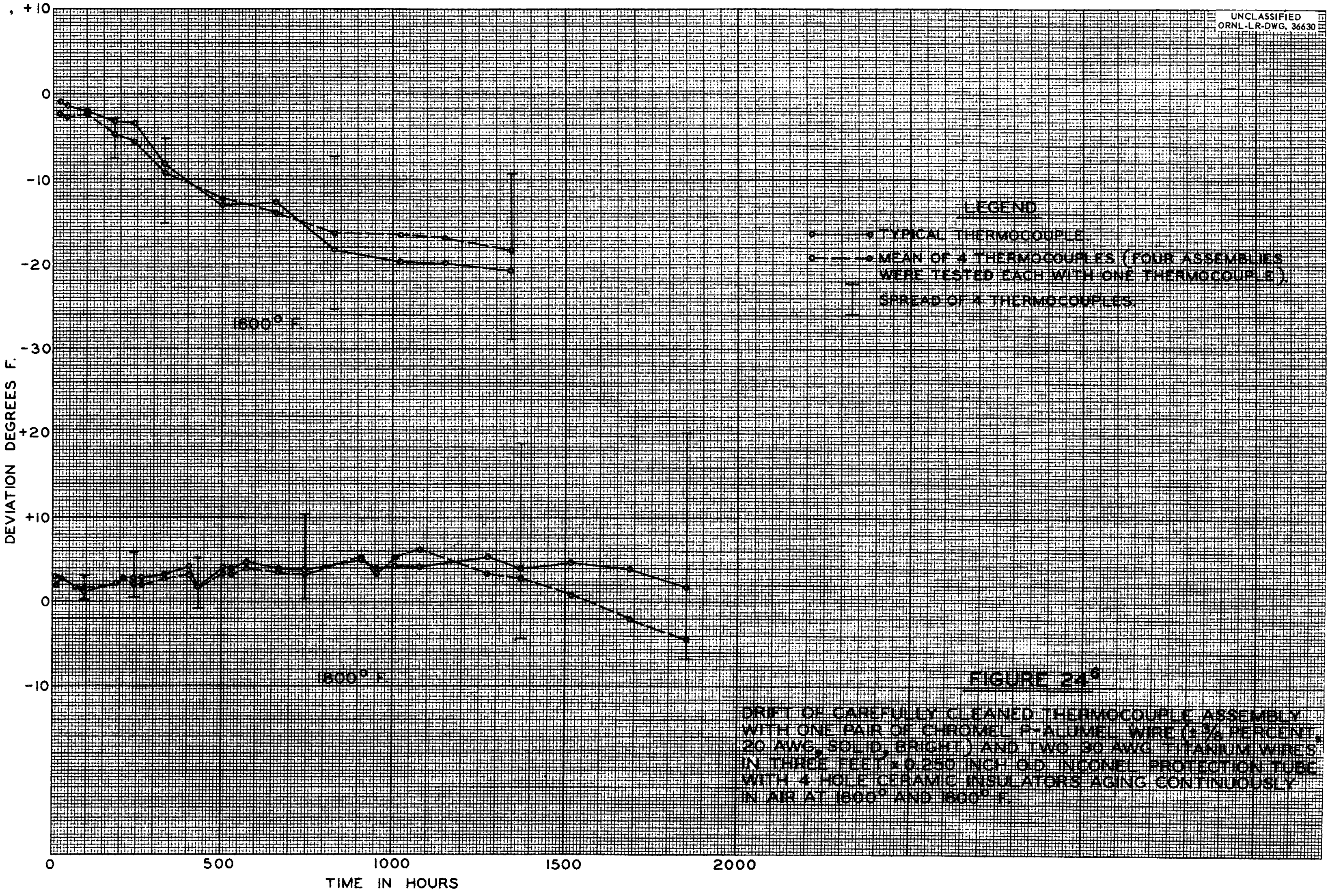

FIGURE 24[6]

DRIFT OF CAREFULLY CLEANED THERMOCOUPLE ASSEMBLY WITH ONE PAIR OF CHROMEL P-ALUMEL WIRE (± ¾ PERCENT, 20 AWG, SOLID, BRIGHT) AND TWO 30 AWG TITANIUM WIRES IN THREE FEET x 0.250 INCH O.D. INCONEL PROTECTION TUBE WITH 4 HOLE CERAMIC INSULATORS AGING CONTINUOUSLY IN AIR AT 1600° AND 1800° F.

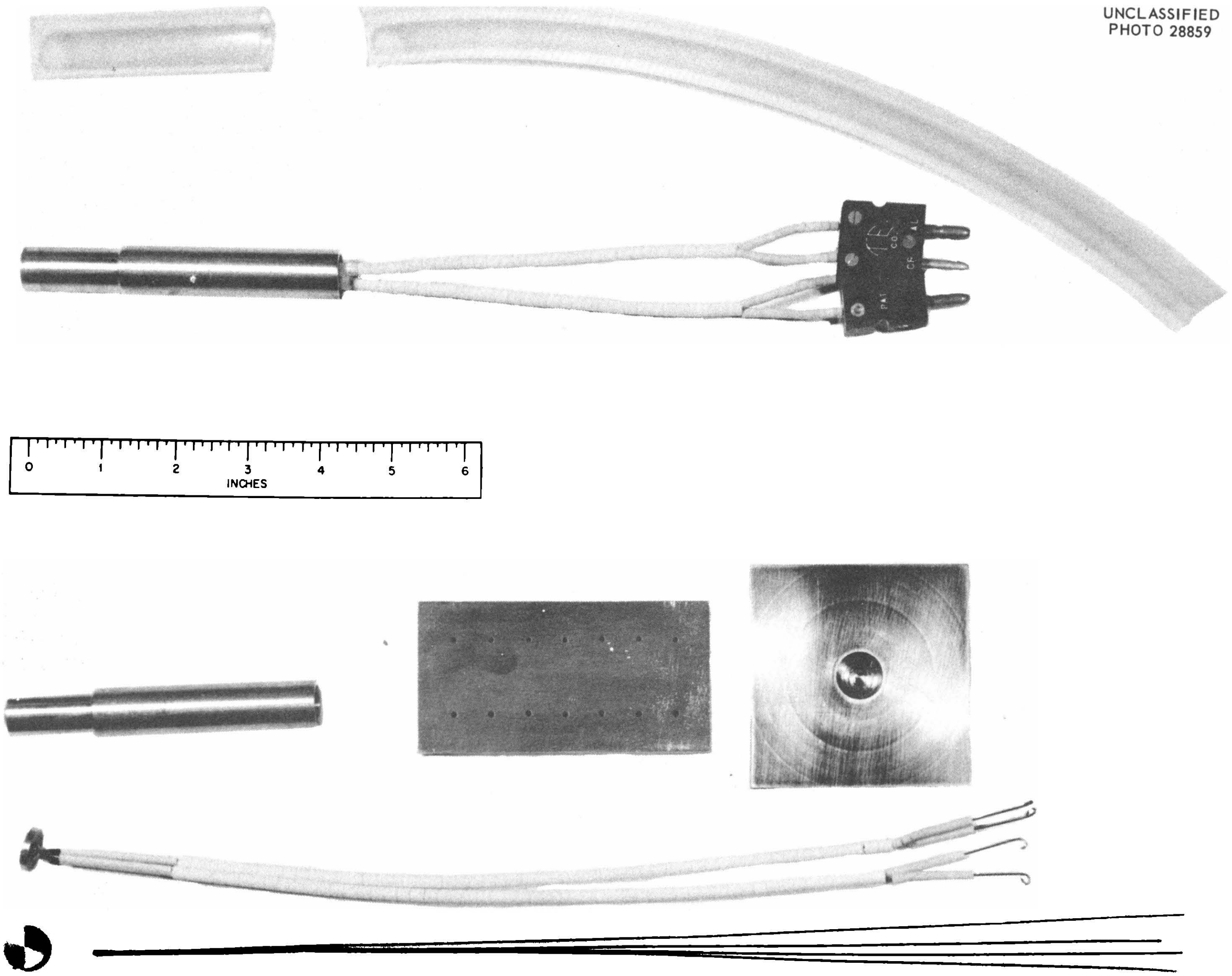

Figure 25. Disassembled and Assembled Views of Well-Type Thermocouple for NaK Piping Showing Copper Block for Fabrication of Inconel-Coated Thermocouple Junction, Jig for Attachment Weld to Well Bottom and Plastic Tubing for Protection of the Assembly During Handling.

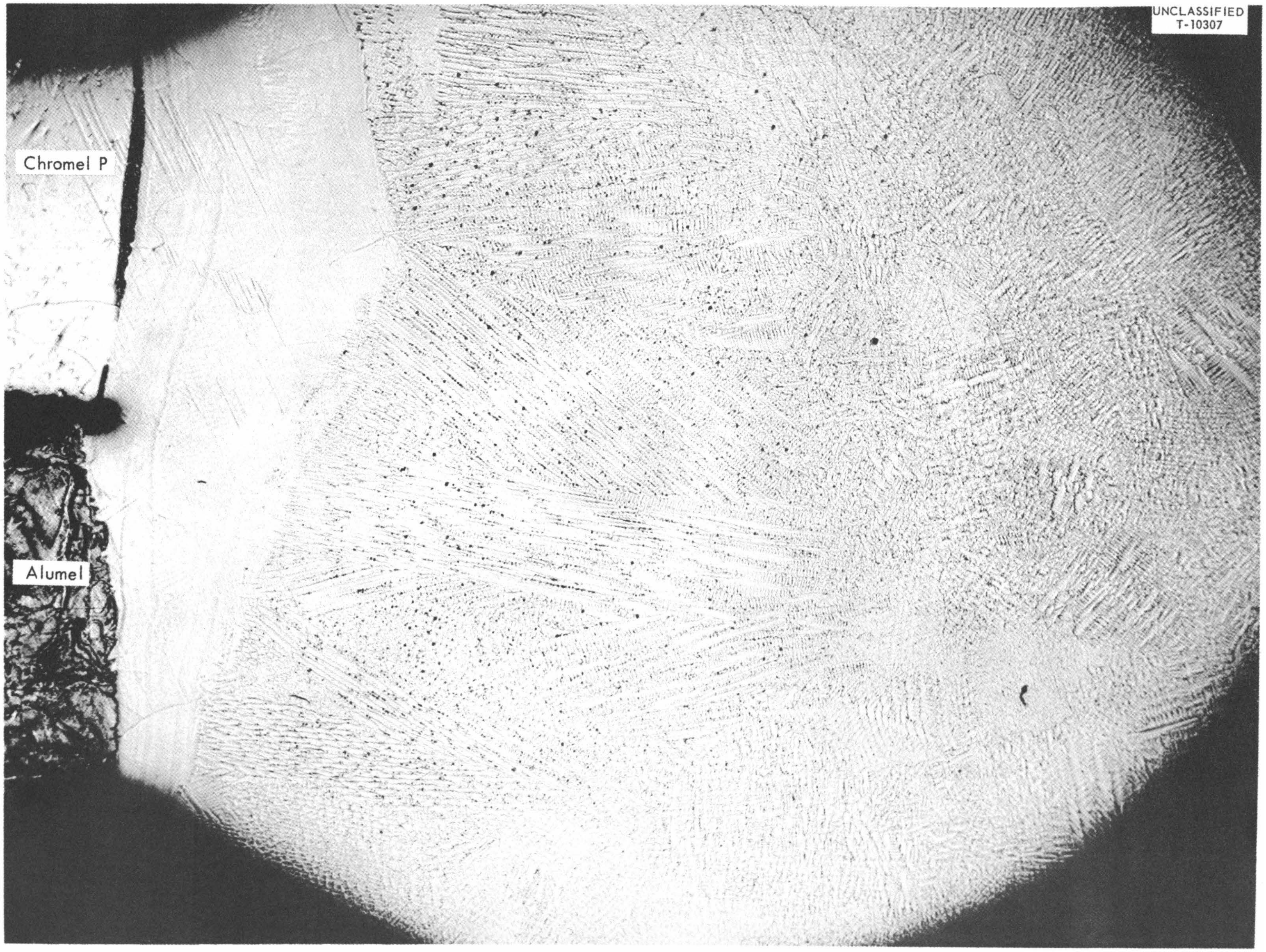

Figure 26. Cross-Section of Chromel P-Alumel Thermocouple Junction Where Wires Were Given a Normal Cleaning Prior to Junction Formation. (Note chromium oxide deposits in the Chromel P wire.) Reduced 25%.

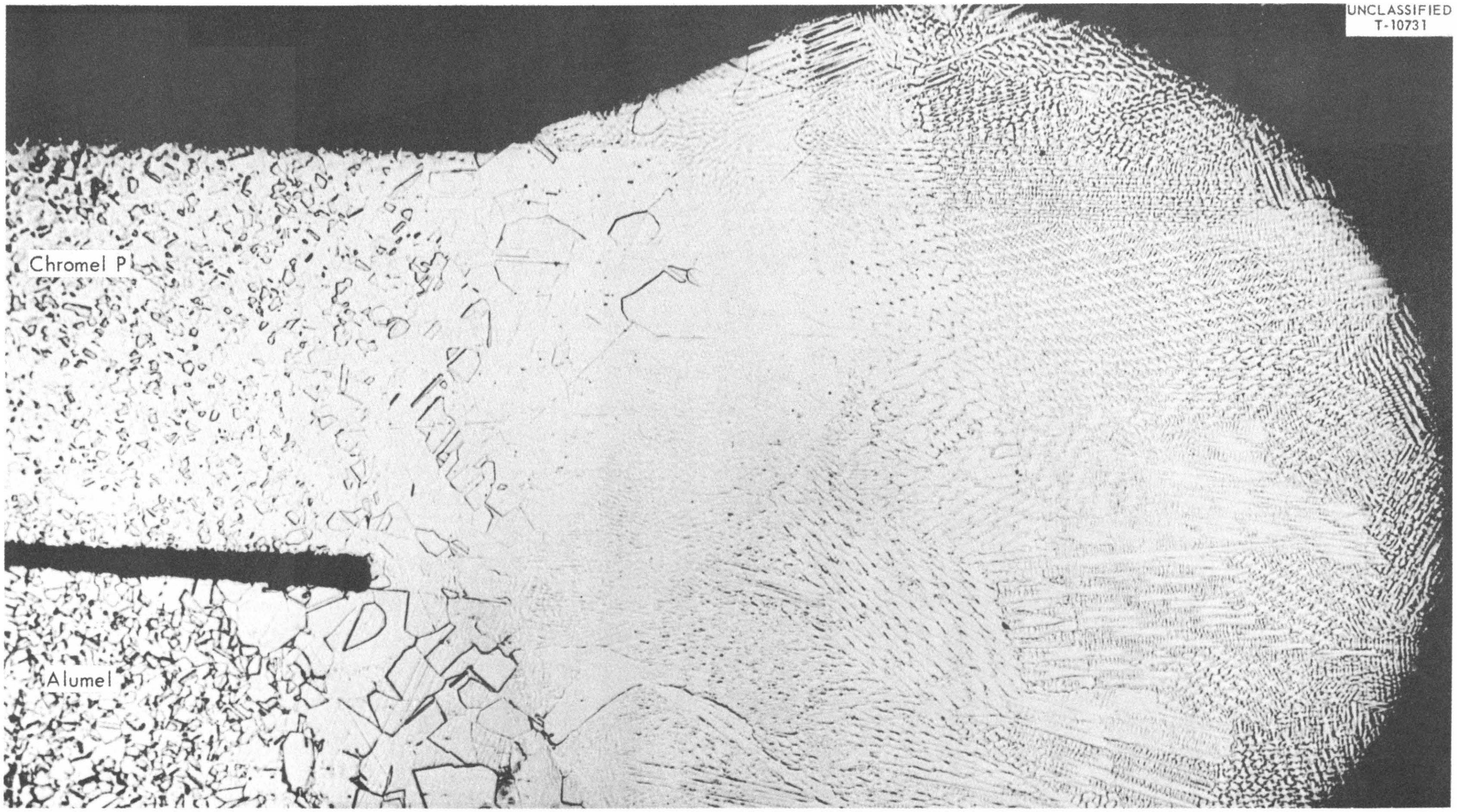

Figure 27. Cross-Section of Chromel P-Alumel Thermocouple Junction Where Wires Were Carefully Cleaned of Any Oxide Prior to Junction Formation. (Note absence of any deposits in Chromel P Wire.) Reduced 21%.

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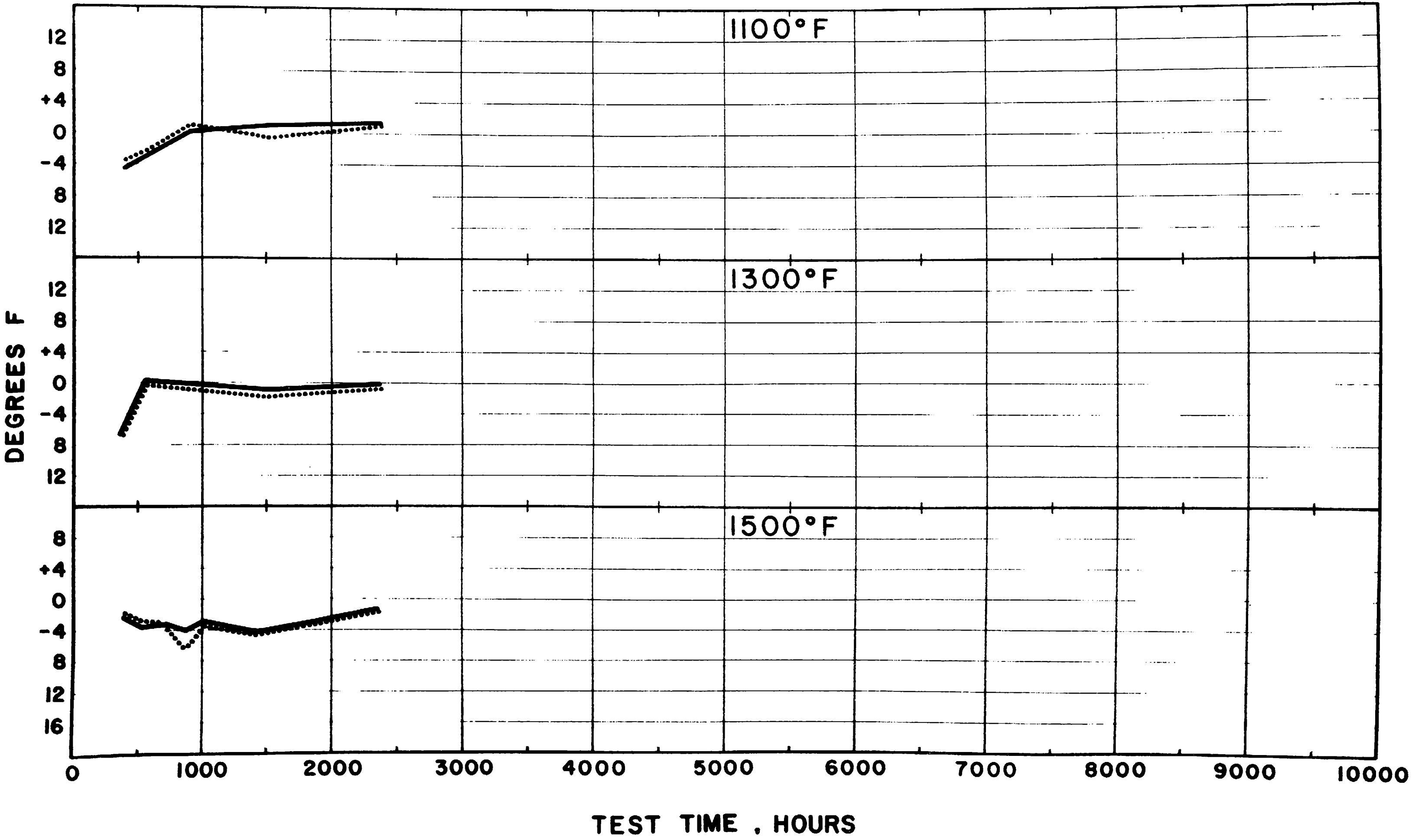

Figure 28. Typical Drift of Well-Type Thermocouple Assembly with Chromel P-Alumel Wires (±3/8% 530°F to 2300°F) in NaK. (Note double curves represent the two couples contained in the assembly.)

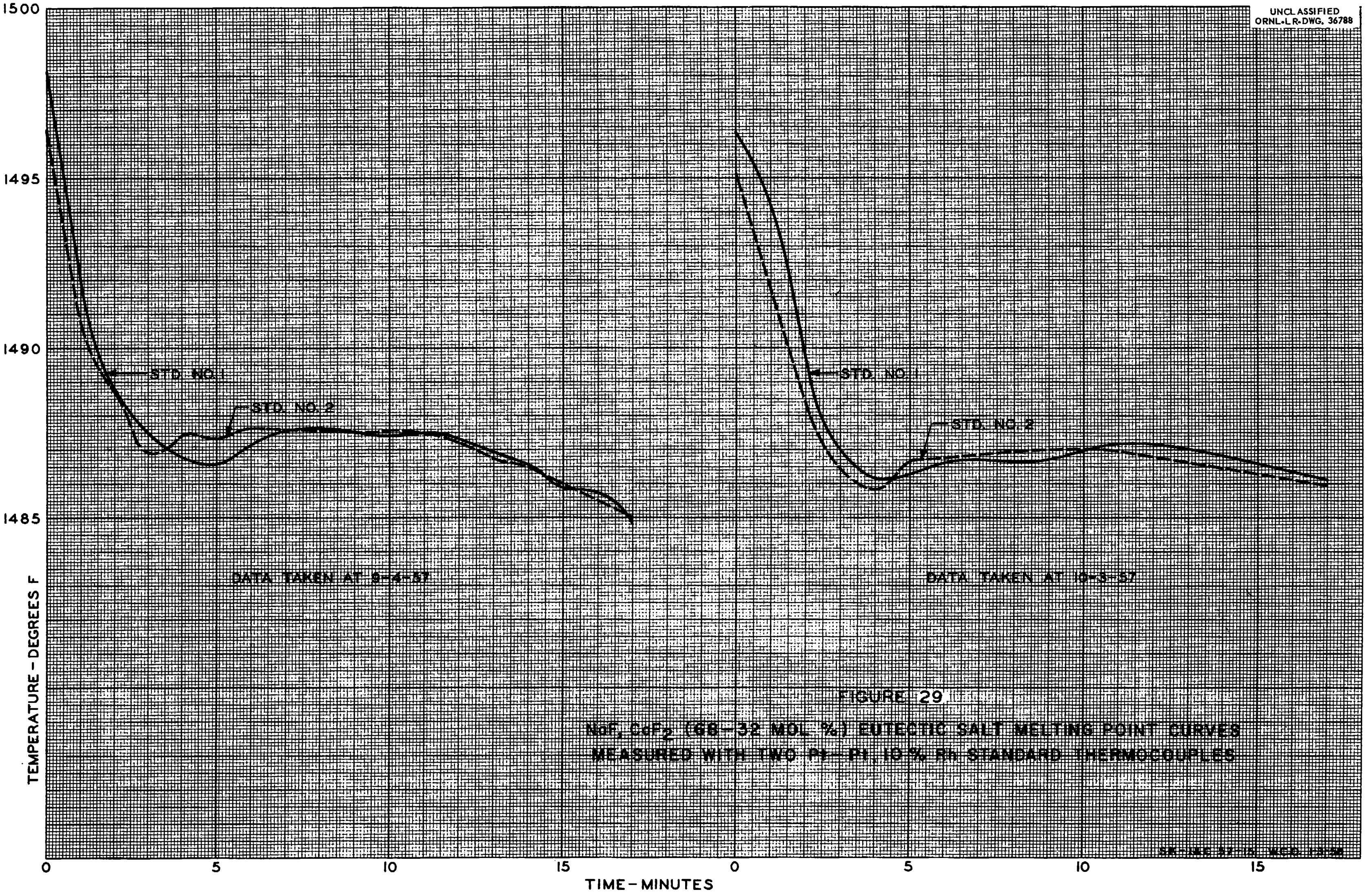

FIGURE 29

NaF, CaF$_2$ (68-32 MOL %) EUTECTIC SALT MELTING POINT CURVES
MEASURED WITH TWO Pt-Pt, 10 % Rh STANDARD THERMOCOUPLES

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