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LONG-TERM DRIFT OF SOME NOBLE- AND REFRACTORY-METAL THERMOCOUPLES AT 1600 K IN AIR, ARGON, AND VACUUM

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Long-Term Drift of Some Noble- and Refractory-Metal Thermocouples at 1600 K in Air, Argon, and Vacuum

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Thermal emf changes (drift) for 38 pairs of noble- and refractory-metal thermocouples using high-purity alumina insulators were determined in air, argon, and vacuum environments at 1500 to 1600 K for time periods up to 10 000 h. Wire sizes ranged from 0.3 to 1.1 mm diam with 0.5 mm being the most common size. In air, the maximum drift of 87Pt13Rh/Pt thermocouples, for 10 000 h, was -5 K (lower emf). In argon, 87Pt13Rh/Pt and 95W5Re/74W26Re pairs with 0.5 mm wires drifted about -22 K for 10 000 h; 70Pt30Rh/94Pt6Rh pairs of the same diameter drifted about -12 K; and smaller diameter (0.33 mm) noble-metal pairs behaved erratically, with drifts of up to -72 K. In one vacuum test system most noble-metal thermocouples failed (by open circuit) within a few thousand h, after less than -4 K drift; in a second test system, 87Pt13Rh/Pt pairs (0.5 mm diam) drifted -3 K at 3700 h without failures. Testing of the W-Re alloy thermocouples in a vacuum was terminated at 3800 h by failure of tantalum test capsules; at 3800 h, drift was -16 K. Test-induced chemical and physical changes of the materials were determined by emission and mass spectrometry, photomicrography, hardness tests and electrical resistivity measurements.

INTRODUCTION

There are several areas of aerospace-engineering research involving material studies, component testing, and complete systems operation that require the use of thermocouples[1] over long periods of time in air, inert gas, or vacuum. One such example is a space-power system designed to operate continuously for 10 000 h.

Under these conditions, thermoelectric stability becomes important because the investigator has to distinguish between actual temperature change and the thermal electromotive force (emf) change at constant temperature, commonly called thermocouple drift. This drift may be associated with change in the chemical composition or metallurgical structure of the thermocouple materials when exposed to a thermal gradient.

Chemical composition changes occur by exchange of material between the wires and also between the wires and the surrounding media. Some of the mechanisms involved are diffusion, chemical action, selective evaporation of an alloy constituent, or evaporation from one element and subsequent deposit and combination with the other. Structural differences, related to changes in the amount of lattice defects produced by cold-working[2] can occur, as well as increased grain growth resulting from exposure at elevated temperatures.

Among the factors affecting drift are insulator and sheath materials, assembly geometry, fabrication method, gas environment, rate of thermal cycling, and nuclear radiation environment, if present.

The present investigation was intended not to study the effect of all possible operating conditions, but rather to establish the drift that might result when the thermocouple-assembly materials, construction, and usage were selected, in advance, to minimize drift. Selection was based on the

best judgment that could be exercised from results previously reported by others. To this end, for example, thermocouple wires and insulators were of the best grade commercially available. Construction and test installation were such that mechanical strain on the wires was minimized. Additional precautions taken will be described in a later section.

This report compares the relative merits of some commerically available thermocouple wires in high-purity Al_2O_3 insulators exposed to air, argon, and vacuum at 1500 or 1600K for up to 10 000 h. The thermocouples tested were 87Pt13Rh/Pt, 70Pt30Rh/94Pt6Rh, and 95W5Re/74W26Re. These thermocouples were chosen because the first is a common type with a wide background of published information, the second should potentially have better drift characteristics than the first, and the third is commercially available and, although usually used at a temperature level higher than that of the present test, appears to be a likely candidate for long-term use at the lower temperature level.

The tests were conducted in two separate facilities which shall be identified as Facility I and Facility II. All three wire types were tested in Facility I, which exposed them to environments of air, argon, and vacuum at 1600 K for up to 10 000 h. A total of 30 pairs of thermocouples were tested; drift measurements were made periodically during the course of the test period, without disturbing the thermocouples. A second test, intended to provide some redundancy, exposed 8 pairs of 87Pt13Rh/Pt thermocouples to a high-vacuum environment at 1530 K for 3700 h. This test was conducted in Facility II. Drift was determined by calibrating the test thermocouples in air, in a separate calibration furnace, before and after the 3700-h exposure.

During testing, in both facilities, gas analyzers were used to monitor the gas composition of the thermocouple environments. Post test analysis

were also made to determine the compositional and structural changes in the thermocouple wires.

PREVIOUS INVESTIGATIONS

Walker, Ewing, and Miller[3] indicated that the major cause of drift in noble-metal thermocouples using alumina insulators involved contamination of the wires by iron transferred from the insulator. The magnitudes of the changes were directly related to the amount of the impurity present in the various grades of insulators. One test in their series of experiments was performed on 87Pt13Rh and platinum thermocouple wires fired in various grades of alumina insulators in argon at 1650 K for 120 h. The wires were then removed from the test furnace, and the thermal emf change for each wire was determined by recalibration in a separate calibration furnace. In this furnace the wires were subjected to a gradient extending from the junction at a temperature of 1130 K to room temperature. From the results, one can calculate that, for a low-purity insulator, the maximum change for an 87Pt13Rh/Pt thermocouple pair was -96 K, and, for a high-purity insulator, the change was a negligible -2 K. (A negative sign represents a drop in emf).

It has also been reported[4, 5, 6, 7] that Si contamination, particularly in the pure Pt leg of a Pt-Rh alloy/Pt thermocouple, causes both thermal emf change and mechanical failure. Mechanical failure is explained by the fact that, in pure metals, large grains will grow when the material is subjected to high temperature for long periods of time, resulting in structural weakening. In the case of Pt, when Si is present, a Pt-Si eutectic can form and enter the grain boundaries. This attack in the grain boundaries affects the output emf as well as accelerating structural failure. Zysk[4] states that

some investigators observed erratic output signals prior to an open-circuit failure.

Metcalfe[8] postulated that the addition of Rh to the pure Pt leg would make a thermocouple less susceptible to contamination, and he successfully developed and used an 87Pt13Rh/99Pt1Rh thermocouple. This approach eventually led to the introduction in Germany of commercially available 70Pt30Rh/94Pt6Rh thermocouples. The National Bureau of Standards,[9] did extensive work in preparing calibration tables and qualifying the 70Pt30Rh/94Pt6Rh thermocouple for more general acceptance.

Some recent studies[10] of 90Pt10Rh/Pt and 70Pt30Rh/94Pt6Rh thermocouples in high-purity alumina insulators at 4×10^{-8} torr and 1720 K for 1000 h showed both thermocouple pairs to be stable to within ±10 K.

Others[11] have also reported on the drift of these two thermocouple pairs, with alumina insulators, in air at 1820 K for 400 h. The change in emf of the 90Pt10Rh/Pt thermocouple was -75 K, and the 70Pt30Rh/94Pt6Rh thermocouple drifted -38 K. This is a rather severe deviation for an air environment test; unfortunately the report gives no indication of the purity of the insulators. However, one may note that the drift of the 70Pt30Rh/94Pt6Rh thermocouple was approximately one half the drift of the 90Pt10Rh/Pt thermocouple.

When aerospace and nuclear technology advanced to the state of requiring measurements at temperatures and in environments not suitable for the use of noble-metal thermocouples, investigation of refractory-metal thermocouples was accelerated. This led to the development and commercial production of W-Re alloy thermocouples. A great deal of investigation was done in this area,[12, 13, 14, 15, 16, 17] but, since much of it dealt with material studies and applications not specifically relevant to the scope of the

present report, only that reported material that appears pertinent will be presented. Perhaps one of the most significant conclusions to be reached from the studies is that it takes a determined effort to control the purity and processing of the thermocouple assembly to achieve reliability and accuracy at the higher temperatures.

One test[18] measured the drift of several 95W5Re/74W26Re thermocouples using high-purity Al_2O_3 insulation in a 99Cb1Zr sheath that was exposed to an inert-gas environment at 1370 K for 10 000 h. The resultant drift averaged -10 K.

Another test of these thermocouples[10] in high-purity Al_2O_3 insulators at 4×10^{-8} torr and 1720 K for 1000 h found them to be stable within ±10 K.

A third investigation[19] reported drift rates of -0.9 K per 1000 h for 95W5Re/74W26Re thermocouples in Ar at 1370 to 1870 K for periods of 1000 to 4000 h.

It becomes readily apparent from a review of work relating to drift studies that many variables are involved and any single investigation, being limited in scope, does not reveal a general solution, but merely supplies limited information usually relating to a particular application. Furthermore, failure to completely define the variables involved results in apparent contradictions and causes difficulty in correlating the results of several investigators.

FACTORS AFFECTING THE STABILITY OF A THERMOCOUPLE

The selection of a thermocouple assembly that is to be stable over long periods of time involves considerations of the many factors that influence the magnitude of emf change. Some of these factors present choices in the design selection, and others are related to the operating conditions.

A general list of factors relating to thermoelectric drift may be grouped as follows:

(1) Design selection

- (a) Wire type
- (b) Wire diameter
- (c) Sheath material
- (d) Insulator
- (e) Assembly geometry
- (f) Fabrication method and its control

(2) Operating conditions

- (a) Temperature level
- (b) Time
- (c) Temperature gradient
- (d) Environmental gas
- (e) Pressure level
- (f) Thermal cycling
- (g) Nuclear radiation

The selection of the type of wire is usually made on the basis of the application temperature range and previous experience of the drift characteristics of the wire.

Wire diameter is often compromised by the size and geometry of the assembly necessary to fit the application. However, it is reasonable to conclude that if some impurity in the environment is available to cause degradation, then for the same conditions, a smaller wire will be degraded more than a larger wire, the surface-to-volume ratio being greater for the smaller wire. One investigation[3] has shown such a size-stability relation for some environments.

If the thermocouple assembly includes a sheath, then the sheath must be compatible with the external environment as well as with the components it contains.

The type and purity of the insulator have been shown by many investigators to be of prime importance, as previously discussed in this paper.

Although the geometry of the thermocouple assembly is usually dictated by size and mechanical considerations, possible variations in insulator geometry, clearance between wire and insulator and between insulator and sheath, and use of metallic foil barriers[20] can be considered for preventing or decreasing contamination of the wires and their junction.

Quality control during fabrication is necessary to prevent the introduction of contaminants into the assembly.

Exposure time and temperature level are both important factors contributing to the magnitude of drift for a particular system. The importance of the location of the thermal gradient in the application and in post-test evaluation for drift has been previously emphasized.[21,22]

The nature of the environmental gas, namely, whether it is oxidizing, neutral, or reducing, will govern the kinds and degree of compositional changes that occur. Even in the case of a vacuum application, possible backstreaming of hydrocarbons from the pumping system must be prevented by proper cold trapping, and the release of constituents in the system due to outgassing at high temperatures must be considered as part of the reactant environment.

The pressure level affects the rate of change in composition by controlling the diffusion away from the surface.

One investigation[23] has indicated an emf change related to thermal cycling rate for bare and insulated 95W5Re/74W26Re thermocouples.

The final consideration in the list of factors involves the effect of nuclear radiation on the wire composition. Compositional changes can occur by transmutation resulting from the production of a radioactive isotope, which decays into an isotope of a different element. The effect of this change in material on the drift is, however, dependent on the magnitude and location of the thermal gradient in the application.[21]

The present investigation involves consideration of the aforementioned factors with the exception of the last two items, namely, thermal cycling and nuclear radiation.

APPARATUS

Facility I was the principal means for the drift studies. Three thermocouple combinations and three environments were used, for test durations up to 10 000 h, with continuous monitoring of drift. Facility II was used as a backup for Facility I, to provide an independent check of results. A single thermocouple combination was used, in a high vacuum environment, with a single measurement of drift made at the end of a 3700 h run. Termination of this test was due to a failure of the heater power supply.

Facility I

Figure 1 is a schematic of Facility I, which is composed essentially of the following subsystems and their instrumentation: (a) furnace, (b) test capsules, (c) vacuum system, (d) argon system, (e) cooling system, and (f) temperature measurement system. A more detailed description of the test system components and their operation can be found in a previous publication.[24]

Furnace

The furnace was a muffle-type using a vitreous refractory

mullite tube, 20 cm diam., with a 0.95-cm wall, and 168 cm long. A 29-kg cylindrical Mo block was placed in the center of the furnace to provide a uniform temperature zone; the block had 18 holes (nine in each end) for insertion of thermocouple test capsules. The mullite furnace tube was sealed at both ends by end plates which in turn contained compression fittings to hold and seal individual test capsule assemblies. After the test capsules were inserted, the mullite tube was evacuated and back-filled with high-purity Ar. An absolute pressure of 1.2×10^5 N/m^2 (1.2 atm.) was maintained in the system during the test period. A water cooling system supplied the diffusion pump, furnace end plates, and expansion joint clamps, as well as, two heat shields hung adjacent to the sides of the furnace to absorb heat that would have normally been rejected to the room.

The furnace used SiC rods for resistance heating, and used approximately 10 kVA at the operating temperature of 1600 K. The SiC heating rods were distributed in the furnace cavity to form three controlled heating zones.

Although it has been reported that Pt-Rh thermocouples are very stable in an air environment, it was anticipated that the control thermocouples which were exposed to air would be subject to a small degree of drift over the 10 000 h exposure time and that a set-point adjustment would have to be made to correct for control-thermocouple drift.

To accomplish this, an air-exposed test thermocouple was periodically removed (about once every 1000 h) from the center of the Mo block and replaced by a calibrated 87Pt13Rh/Pt working standard. This working standard remained in the block just long enough (about 20 min.) to insure accurate test-block temperature measurement; at other times it was carefully stored in a clean tube at room temperature. The furnace-control set

point could then be adjusted whenever the working standard indicated a change in block temperature from the initial set point. This refinement of maintaining test-block temperature to within a few degrees was introduced because it was easy to do and it provided a check on the control system.

It will be noted later, however, that the drift measurements of the test thermocouples involved only temperature-difference measurements, so that the results would be independent of the exact block temperature level, provided the approximate temperature level were maintained.

Test Capsules

Two types of test capsules to contain the thermocouple assemblies were used to allow a choice in the gas environment surrounding the thermocouple assembly. A thermocouple assembly consists of a calibrated pair of thermocouple wires in a two-hole insulator. One type of capsule (Fig. 2(a)), which exposed a Ta-tube plenum in the hot zone, was used to contain either a vacuum or Ar environment. Each capsule of this type contained one or two thermocouple assemblies. The other type of capsule (Fig. 2(b)) used a high-purity-alumina closed-end tube and was vented to the atmosphere, to afford an air environment for its thermocouple assembly. Each capsule of this type contained one thermocouple assembly. With this arrangement, assemblies exposed to air, argon, and vacuum environments could all share the same isothermal block. All ceramic exposed to the test environments was recrystallized Al_2O_3 of not less than 99.5 percent purity. In the test capsules that contained two thermocouple assemblies, care was taken to use random lengths of ceramic so chosen that the interface between two pieces of ceramic of a particular thermocouple assembly did not lie adjacent to the interface of the other thermocouple assembly sharing the

capsule. This was done to prevent possible line-of-sight exchange of wire material, by evaporation, from one thermocouple assembly to the other.

Thermocouple junctions were formed by arc welding in an inert-gas atmosphere. Copper chill blocks were used to hold the W- Re alloy thermocouples during welding to reduce the length of the embrittled heat-affected zone.[19]

Tantalum tubing used in capsule plenum construction was degreased in trichloroethylene vapor, pickled in an aqueous solution of nitric and hydrochloric acid, flushed with distilled water, and dried with hot air. During fabrication and assembly, thermocouple wires, insulators, and refractory-metal components subjected to the test environments were handled with clean-room-type nylon gloves and were stored in polyethlene bags.

Table I lists the wire diameter and number of each type of thermocouples tested in the various environments. All types included pairs of 0.51-mm diam wires, so that a common size could be used for intercomparison of results in the various environments.

Vacuum System

The primary vacuum pumping system was a 10-cm oil diffusion pump using silicone oil with a freon-refrigerated cold trap held at 250 K. A secondary 15-cm ion pump operated in parallel with the primary diffusion pumping system. The main purpose of the ion pump was to hold system pressure in the event of nonelectrical failure of the primary system. If such an event occurred during testing, a pneumatically operated valve was arranged to automatically isolate the primary pump from the rest of the vacuum system. The vacuum pumping stations were connected to a manifold ring positioned around the external portion of the furnace tube by 10-cm diam stainless-

steel piping. Thermocouple-type thermal conductivity gages and Bayard-Alpert type ionization gages were used at various locations to monitor pressures during pumpdown and steady operation. Steady pressure in the manifold was about 4×10^{-6} torr equivalent N_2 pressure. An electrostatic mass spectrometer tube was also fitted into the manifold to monitor gas composition in the ring. Individual test capsules were connected to the manifold by flexible metal tubing with appropriate isolation valving. All the test capsules connected to the vacuum system were inserted at one end of the furnace tube, while all of the argon-environment capsules were at the opposite end. Capsules vented to the air were inserted in both end plates.

Argon System

The bottled Ar used, nominally contained less than 2 ppm of O_2 and less than 5 ppm of H_2O. A metal-diaphragm pressure regulator reduced the pressure to 1.2×10^5 N/m^2 (1.2 atm.). The main branch of the Ar system was connected to two cylindrical manifolds located adjacent to the external portion of the furnace tube. These manifolds, through flexible tubing and appropriate valving, fed the individual test capsules containing an Ar environment. The Ar was essentially dead ended in the test capsules, the only circulation being due to free convection. A second supply line branched off from the main line to supply an Ar cover gas inside the muffle-furnace tube. Two sampling lines, one originating near the supply-manifold pressure regulator, and the other coming from the capsule distribution manifold, were connected to a galvanic-cell-type O_2 analyzer for on-the-line monitoring of O_2 content in the Ar system. During the 10 000 h test period, O_2 content in the manifold did not exceed 1 ppm.

Temperature Measurement System

Thermocouple wire was run from the test-capsule connector blocks to

stirred constant-reference-temperature oil baths (Fig. 1). These connecting wires were from the same lots or spools as the test-capsule wire to which they were attached. The wires were joined directly to each other at the connector block; the block itself was of insulating material and merely furnished mechanical support. Thus, the thermocouple circuit extending from the junction to the reference bath was composed entirely of calibrated pairs of thermocouple-grade wire. Shielded Cu wiring was then brought out of the baths, through an emf-suppression system and into two 24-point, zero-center, 2-millivolt span, strip-chart recorders.

The emf-suppresion system, referenced to an unsaturated standard cell, suppressed most of the generated emf of each thermocouple, leaving the last millivolt or fraction thereof to be recorded by the strip-chart recorder to a limit of error of 7μV ($\approx$0.5 K).

Facility II

The test chamber of Facility II was a 150-cm stainless-steel cross mounted directly on top of a 15-cm ion pump. All flanges were water-cooled and copper-gasketed. One side flange was connected to a mechanical roughing pump through a bakeable valve and liquid-nitrogen cold trap. The other side flange held a Bayard-Alpert type ionization gauge and an electrostatic mass spectrometer.

The top flange held the thermocouple test assembly, mounted on tantalum supports. All wires, insulated with alumina tubes, passed through the flange and were cemented into a feed-through with a low-vapor-pressure epoxy cement to form a hermetic seal. Thereby, the test-thermocouple wire material extended, without break, to an emf-measuring potentiometer.

Details of the test section are shown in Figure 3. An externally-threaded 99.5 percent-pure alumina tube, 1.5-cm i.d. by 12.5 cm long,

was wound with cleaned W wire to form a heater. Inside this tube a central closed end Pt tube, 0.5 cm i.d. by 30 cm long, extended through the top flange to form an air-exposed well for the insertion of a reference 87Pt13Rh/Pt thermocouple to monitor temperature. The Pt tube was Au-Ni welded to the flange to form an airtight seal.

Eight test thermocouples surrounded the central Pt tube and almost filled the inside diameter of the heater. Each thermocouple consisted of a 87Pt13Rh/Pt pair, using 0.05-cm diam wires, in 2-hole high-purity Al_2O_3 insulators. A Pt wire wrapped tightly around the 8-thermocouple assembly promoted intimate thermal contact with the central thermocouple well. A cylinder of Pt sheet lined the inner wall of the heater, to minimize any direct contamination of the test thermocouples by vapors emitted from the Al_2O_3 heater.

The end of the heater tube was open, to promote maintenance of a high vacuum. However, spaced and staggered radiation shields (the inner one of Pt, the others of Ta) reduced radiant heat loss from the end of the heater. Similar cylindrical shields were used around the side of the heater. Additional details are presented in a separate publication.[25]

FACILITY OPERATION AND PERFORMANCE

Facility I

Furnace and Reference Temperature

The furnace of Facility I was brought to the test point temperature at a rate not exceeding 20 K per hour. This heating rate was necessary to prevent thermal-shock rupture of the large ceramic furnace tube.

Thermocouple drift was determined by the indicated change in temperature difference between the reference block and each test thermocouple, as

a function of time. Since reference-block temperature was periodically checked, this method eliminated the need of keeping the block at one fixed temperature. Nevertheless, by readjustment of furnace control set point (3 times in the 10 000 h period) block temperature was always held within ±2 K of 1593 K. About 300 data points were taken for each test thermocouple that completed the 10 000-h test period.

The reference-block temperature existing at the time a set of data (30 points) was recorded was taken to be the corrected temperature of one of the 87Pt13Rh/Pt, air-exposed test thermocouples, which was used as a reference. This reference thermocouple was subject to a systematic drift amounting to -4.8 K at the 10 000-h end point; however, its drift was determined directly by periodically comparing its output with the output of the calibrated 87Pt13Rh/Pt working standard that was placed in the test capsule just long enough to measure true block temperature. Thus, after completing all the tests, a drift curve was generated for the reference thermocouple; then this curve was used as a correction curve to establish block temperature at any previous time.

Vacuum Level and Composition

The mean nitrogen-equivalent pressure of the vacuum manifold ring was monitored with the ionization gage during the tests. After initial vacuum-system pump down and furnace warmup to the test temperature, the pressure in the manifold ranged between 3×10^{-6} and 5×10^{-6} torr. At about 3100 h test time, the pressure in the manifold was observed to be slowly rising with time. A systems check indicated that one of the tantalum plenums of a test capsule was leaking. This individual capsule had to be isolated (see isolation valves, Fig. 1) from the pumping system after 3480 h; subsequently, as other tantalum plenums failed, they also were

valved off, with the last vacuum capsule shut down at 4080 h. The capsules were always isolated before vacuum manifold pressure exceeded 8×10^{-6} torr.

Failure and subsequent shut down of the vacuum system prematurely ended the vacuum-exposed refractory-metal thermocouple test. However, the vacuum-system failure did not essentially affect the noble-metal thermocouples exposed to the vacuum environment because the majority of this type failed physically, prior to vacuum-system failure. The thermocouples undergoing tests in the air and argon environments were not affected by vacuum-system shutdown and continued performing to 10 000 h or to their failure point, whichever came first.

During furnace warmup, molecular weights of 18 and 28 were the principal constituents below 1000 K. Hydrogen appeared and became the main constituent at 1360 K. It was still predominant when the furnace reached its operating temperature of 1600 K with small amounts of 18 and 28 also present. The compositional changes from this point on essentially amounted to occasional appearance of CO_2, the disappearance of the 28 indication, and a slow diminishing of the amount of H_2. The background finally stabilized into fixed quantities of H_2 and H_2O.

Accuracy of Drift Determination

One method of calibrating immersion temperature sensors at a given temperature is to place the devices in holes located in a high-heat-capacity isothermal block and compare their output with a reference device placed in another hole in the block. If the block is not truly isothermal this will introduce an error in the calibration.

If the test system is used to determine test-sensor drift over a long period of time at a given temperature level, it is not essential that the test

block be absolutely isothermal nor must the test-block temperature level remain at an absolute fixed value. It is only essential to accurately determine for each set of data (taken at a given time) the temperature of the reference hole and the change in the initial difference between this reference-hole temperature and the temperature indicated by each test device. The added provision is that the block temperature distribution remain constant with time and temperature level. These considerations, along with the long-term stability of the recording system, are involved in determining the accuracy of the experiment.

Applying these factors to the present tests, the specific considerations are the long-term stability of the temperature distribution in the high-temperature isothermal block, the constancy of the temperature in the cold-junction reference bath, the voltage stability of the emf-suppression device, and the readability and reproducibility of the recorders.

The test thermocouples were equally spaced on a 5-cm radius circle centered about the reference thermocouple in the isothermal block. At the starting time of the experiment, the difference in temperature between the center hole and the circumferential test holes varied between 0.4 and 0.3 K. A final check of temperature difference between one test hole and the center reference hole after 10 000 h showed that these differences were not changed by more than the readability of the strip-chart recorder ($\approx$0.2 K). These two tests thus showed that the initial spatial variation of temperature within the isothermal block was negligibly small and also indicated a negligible change after 10 000 h.

The probable error in temperature measurement, resulting from this and all other known sources of measurement uncertainy, is estimated to be 0.3 K.

Facility II

General Test Procedure

The test procedure consisted of the following basic operations:

(1) Samples of test-thermocouple-wire pairs were initially calibrated, up to 1735 K in air, against a NBS calibrated 87Pt13Rh/Pt thermocouple, in a separate calibration furnace.

(2) The empty test section was evacuated and cleaned, through baking, before the test thermocouples were installed. The test section was back-filled with dry N_2 after it had cooled.

(3) Test thermocouples were installed and held in the vacuum at the test temperature.

(4) A reference thermocouple was inserted periodically in the central hole, which was always exposed to air, to check the average test-thermocouple temperature. This temperature was held constant by appropriate heater-control adjustment.

(5) At the conclusion of the test, test thermocouples were cooled, removed from the test apparatus, and recalibrated in the same calibration furnace as used for operation (1).

Vacuum Level and Composition

Gas composition after 23 h of heater bakeout at 1400 K, and prior to installation of test thermocouples, was principally molecular weights 18, 28, and 44. Total equivalent N_2 pressure indicated by the ionization gage was 6×10^{-7} torr. After the thermocouples were installed, the test section was slowly brought up to the maximum temperature (1530 K), while a pressure below 5×10^{-6} torr was maintained. The pressure then decreased monatonically to an equilibrium value of 2×10^{-8} torr at 1000 h. At this time the gas

consisted principally of H_2 and H_2O. The pressure and gas composition data thus indicate that the environment provided for the test thermocouples was low in chemically active molecules. It should be noted that this final equilibrium gas composition namely, H_2O and H_2, were the same equilibrium gas constituents remaining in the vacuum system of Facility I.

Test Termination

After 3700 h of test-thermocouple exposure to an environment with a mean temperature of 1530 K and a pressure of 10^{-8} torr, the test was terminated because of a heater power supply failure. All eight thermocouples were then removed. The thermal emf-change data were then obtained by a recalibration in the separate calibration furnace. These calibrations were performed in air, with the temperature gradient in the calibration furnace being essentially the same as that in the test facility.

TEST RESULTS

Table II presents a summary of the drift-test results.

Figures 4 to 8 are plots of drift for various selected pairs, arranged to facilitate comparison of results. The curves presented in these figures are data from a single test thermocouple that has been selected as typical for the several thermocouples of each type tested. The data points plotted show the typical variation in the drift with time for each case. The 10 000 h plots each include about 300 data points.

Drift in Argon

Figure 4 shows the typical drift for 0.51-mm diam wire of the three types of thermocouples tested in argon. It is interesting to note the similarities and differences in the shapes of the curves generated over the 10 000-h period. The drift of the two noble-metal thermocouples can be described as

approximately linear for the first 4000 h and nonlinear thereafter. The converse is true for the 95W5Re/74W26Re thermocouple, which responded in a nonlinear manner for the first 4000 h but was approximately linear the remaining 6000 h.

The average magnitude of the total drift for both the 95W5Re/74W26Re and the 87Pt13Rh/Pt was about the same (averaging -22 K) at the end of 10 000 h. However, the consistency of the drift was higher for the refractory-metal thermocouples than for the noble-metal thermocouples. Table II shows that the four 95W5Re/74W26Re test thermocouples were at this time in closer agreement (1.4 K spread) with each other than were the three 87Pt13Rh/Pt thermocouples (8.8 K spread).

The drift (-13 K) of the typical 70Pt30Rh/94Pt6Rh thermocouple in Ar (Fig. 4(b)), was about one half the drift for the other two types. It should be noted, however, by again referring to table II, that the spread (10.9 K) among the three 70Pt30Rh/94Pt6Rh thermocouples in Ar was close to the spread among the three 87Pt13Rh/Pt thermocouples in Ar.

Drift in Vacuum

Figure 5 shows the drift for the same size of wire and the same thermocouple types as shown in Fig. 4, but for the case of a vacuum environment. The tests of the noble-metal types in Facility I were essentially ended by open-circuit failure. Up to the time of such failure, the 87Pt13Rh/Pt thermocouple in vacuum performed similarly to the same size and type in argon, but the 70Pt30Rh/94Pt6Rh thermocouple had a drift in vacuum approximately twice that of its like kind in Ar.

The 0.51 mm diam 87Pt13Rh/Pt thermocouples in Facility II were still functioning after 3700 h, where the test was terminated by a power-supply failure. The average drift (-2.8 K) of the six specimens was the same

as the average total drift of the three similar specimens in Facility I at the time that they became open circuited (average: 1800 h), and also the same as the average drift of those similar specimens in Ar at 3700 h. Using the Ar data to extrapolate the vacuum data leads to the prediction of -23 K drift in 10 000 h.

The testing of the 95W5Re/74W26Re thermocouples in vacuum was terminated between 3480 and 3860 h by test-capsule failures. However, the drift curve generated in vacuum up to the time of failure was practically identical in magnitude and shape to the drift curve for exposure to Ar. Therefore, although the tests were terminated prematurely, a first approximation for predicted drift at 10 000 h would be equal to the value (-22 K) for the typical example in Ar (Fig. 4(c)).

Drift of Noble-Metal Thermocouples in Air

Figure 6 shows the drift of three sizes of 87Pt13Rh/Pt thermocouples exposed to air for 10 000 h. These results show negligible drift for all three sizes with the average drift being less than the calibration tolerance (1/4%) for commercial special-grade wire of this type.

Drift of Smaller-Size Wire

Figure 7 shows the drift of some of the 0.33-mm diam 87Pt13Rh/Pt wires in Ar and vacuum environments. One of the thermocouples shown for the Ar case failed after 5660 h. The tests in vacuum ended between 3170 and 4080 h. In both of these cases, the drift rates to the point of failure were about twice those for the 0.51-mm diam wires of like type (Figs. 4(a) and 5(a)).

Figure 8 shows the drift of one of two other 0.33-mm diam 87Pt13Rh/Pt thermocouples in Ar, each of which acted erratically for a period of time during the 10 000 h test. Zysk[4] has reported possible erratic behavior of noble-metal thermocouples just prior to failure. It is noted, however, in

the present case, that after about 4000 h of erratic behavior, the output returned to a systematic trend, although the final drift rate was much greater than that displayed before the erratic behavior period. Zysk contends that the erratic behavior is a possible result of a low-melting-point eutectic that forms a film across the interfaces in the grain boundaries. If we apply this explanation to these tests, it appears that for the period of time that the film of eutectic is spreading through the grain boundaries, unstable, erratic behavior results. However, as this process continues, it appears possible that enough eutectic is formed finally to complete a stable circuit. The final emf produced, however, is strongly affected by the eutectic film, as indicated by the large departure from the original calibration (Fig. 8 and table II).

Summary of Expected Drift

Table III summarizes the results by indicating the drift rates to be expected in various environments for the thermocouple combinations tested. Qualifying annotations for some of the entries are indicated. Extrapolated values are used for the tests in which the thermocouple or test apparatus failed before 10 000 h of operation were reached.

Material Analysis

Post-test examinations involving photomicrographs, hardness testing, and mass spectrography were performed on some of the thermocouple assemblies.

Photomicrographs

Figure 9 shows a metallographic cross section of a 87Pt13Rh/Pt junction and the adjacent 87Pt13Rh wire. The large grain size at the junction is created at the time of junction manufacture and persists throughout the drift

test. Away from the junction, the wire initially has a relatively fine grain structure (Fig. 9(a)); the grains grow during the course of the drift test, and have the size shown in Fig. 9(b) at the end of the test.

All of the noble-metal wires used in both test facilities initially had a grain structure similar to that shown in Fig. 9(a); and all had a grain structure similar to that as shown in Fig. 9(b) at the termination of the drift test. The large grain structure existed in wires that lasted only 1800 h as well as in those that lasted 10 000 h; it also was found in wire samples taken from a portion of the wire that had been in the temperature gradient zone of the test capsules, at an approximate temperature level of 1250 K.

Figure 10 shows longitudinal sections of the 95W5Re wire, before and after the drift test. The appearance of the 74W26Re is very similar. There is only slight change in grain structure, associated with partial disappearance of drawing strains.

Table IV lists Knoop hardness numbers for several wires, before and after testing. The following deductions follow from Fig. 9 and Table IV.

1. Many of the large-grain boundaries form fairly direct paths across the diameter of the wire. Under these conditions, the probability of mechanical failure of the thermocouple wire is increased when the wire is stressed. Hence, methods of reducing wire stresses should be an important design consideration for long-duration use of noble-metal thermocouples at high temperatures.

2. The Pt exposed for 10 000 h at 1600 K in Ar shows high hardness, compared with negligible increase in hardness for Pt exposed in air. Both materials showed comparable grain growth. It was previously mentioned in the INTRODUCTION that large grains will result when the pure platinum leg is subjected to high temperature for long periods of time. The progressive

growth into a larger crystal, however, should not result in an increase in hardness, and, in fact, usually a decrease in hardness will be noted. If grain growth is accompanied by an increase in hardness, one may suspect other causes such as the introduction of new constituents. Figure 11, which compares the results of exposure in Ar and in air, tends to confirm this speculation, because the grains in Fig. 11(b), for air exposure, are freer of inclusions than the grains in Fig. 11(a), for Ar exposure. Metcalfe[8] indicated that the Pt leg of a Pt-Rh alloy/Pt thermocouple pair would be the most likely to take up impurities, and Walker, et al[3] showed it to be the leg most likely to change calibration.

The alloy with 6% Rh shows a smaller increase in hardness after 10 000 h in Ar and the alloys with 13% Rh and 30% Rh show only the reduction in hardness that might be expected from continued annealing and grain growth. Retention of ductility, clear grain structure and negligible drift of the 87Pt13Rh/Pt in air (Table III) collectively suggest that an air environment helps to prevent the contamination of Pt that might otherwise occur in the presence of Al_2O_3 and Ta.

Mass Spectrographic Analysis

Mass spectrographic analysis was made of samples of the alumina insulators and thermocouple wires used in Facility I.

Alumina: The specifications on the alumina insulators had stipulated a purity of not less than 99.5 percent. Examination verified this purity and, in fact, indicated a purity of 99.8 percent with the major impurities, in descending order of quantity present, being Mg, Si, Na, and Fe.

Thermocouple wire: Samples of thermocouple wires were examined before and after the 10 000-h exposure in the air and Ar environments. The

concentration of principal contaminants had always increased, by an amount ΔC, after the 10 000 h exposure. In descending order of ΔC, the contaminants of the noble-metal wires were Al, Fe, Si, and Mg; for the refractory-metal wires were Fe and Si. The noble-metal wires were found to be fused to the ceramic insulators in the length of ceramic extending from the junction area (1600 K) as far back as the 1300 K area. These wires were so firmly bonded to the insulators in the 1600 K region that the insulators had to be broken away from the wires so that wire samples could be examined. This procedure left particles of alumina embedded in wire samples and probably accounted for aluminum being the primary foreign constituent. Another recent investigation[26] produced similar results and concluded that, although Pt is exceedingly inert with respect to the more refractory oxides under normal atmospheric conditions, severe reactions can occur when the oxidizing potential of the surrounding atmosphere is reduced below a critical level. The refractory-metal wires, unlike the noble-metal wires, did not bond to the ceramic and were easily removed from their insulators. Also, unlike their noble-metal counterparts, the post examination indicated no increase in Al content.

The Fe and Si impurities were the main impurities commonly picked up by both the noble and refractory wires and appeared in like quantities, ranging from 200 to 2000 ppm.

Emission Spectrographic Analysis

The results of a spectrographic analysis of wires used in Facility II showed that Fe was the impurity in the thermocouple wires which had the highest concentration increase at the junction and the largest concentration gradient near the junction. Ni and Cu showed as the next lower concentration change along the wire. Ag and Si remained uniformly distributed throughout

the wire length. Hence, on the assumption that concentration changes or gradients produce drift, it was concluded that Si was not the prime cause of drift, while the Fe was, and furthermore that the most likely source of the Fe impurity was the Al_2O_3 insulator. The result that Fe is the prime impurity affecting drift agrees with the conclusions derived from the tests in Facility I and with the conclusions of an earlier investigation.[27]

Resistance Measurements

The total resistivity of a pure nonmagnetic metallic element can be represented to a good first-order approximation by the sum of two resistivity terms, only one of which is temperature dependent (Matthiessen's rule). Subtraction of the temperature-dependent term from the total resistivity yields the residual resistivity, which is due to the impurities and strains in the metal.[28] At 4.2 K, the total resistivity of platinum represents the residual resistivity. The residual resistance R(4.2 K), as well as the room-temperature resistance R(300 K), was determined for several test samples of Pt used in the tests in Facility II. The value of R(4.2 K)/R(300 K) was higher in the vicinity of the junction than it was for the unheated wire, suggesting that the Pt had acquired some contaminants during the drift tests. The direction of the resistance changes matches the direction of the thermal-emf changes.

CONCLUDING REMARKS

This report has presented the drift characteristics and post-exposure material analysis of some thermocouple assemblies. These assemblies consisted of commercially available thermocouple wires in high-purity alumina insulators, which were exposed to air, argon, and vacuum environments at 1600 K for up to 10 000 h. The thermocouple wire pairs tested were 87Pt13Rh/Pt, 70Pt30Rh/94Pt6Rh, and 95W5Re/74W26Re in wire sizes

ranging from 0.33 to 1.12 mm. The analysis of test results yields the following conclusions:

1. In argon, the 0.5-mm diam noble-metal thermocouples drifted very little in the first few thousand h, but the drift rate increased subsequently. After 10 000 h, the average total drift was -22 K for the 87Pt13Rh/Pt pair and -13 K for the 70Pt30Rh/94Pt6Rh pair. A negative value represents a drop in emf.

The refractory-metal-thermocouple drift, on the contrary, was more rapid initially, resembling a decaying exponential. After 10 000 h, the average drift was -22 K.

2. Although the drifts for both the 95W5Re/74W26Re and the 87Pt13Rh/Pt in argon were about the same (-22 K) at the end of 10 000 h, the several 95W5Re/74W26Re test thermocouples were in closer final agreement with each other (1.4 K total spread) than the several 87Pt13Rh/Pt thermocouples (8.8 K spread).

3. Because of the initial shape of the noble-metal drift curve, it would be difficult to predict the long-term drift of the noble-metal thermocouple on the basis of short-term results.

4. A comparison of the behavior of the 0.33-mm diam with the 0.5-mm diam noble-metal wire in argon and vacuum shows about twice the drift for the smaller wire. This reinforces the belief that the drift is caused by insulator impurities that contaminate the wire, since the surface-to-volume ratio is 1.7 times greater for the smaller wire. In air, these impurities are converted to harmless oxides, and the drift is very low.

5. The 87Pt13Rh/Pt thermocouple drift in vacuum was like that of the same size and type in argon, up to the time of test termination. The 70Pt30Rh/94Pt6Rh thermocouple had a drift in vacuum (up to the time of failure) of

about twice that of its like kind in argon.

Vacuum tests of the W-Re alloy thermocouples were terminated by failure of the Ta tubing test capsules. However, the drift curve of the W-Re thermocouple up to the time of capsule failure (≈3500 h) was identical to the drift curve for argon exposure, so that it appears safe to predict that the similarity would have been maintained to 10 000 h, resulting in a drift of approximately -22 K.

6. Thermocouple pairs of 87Pt13Rh/Pt wires in air showed a maximum drift of -5 K in 10 000 h.

7. There was negligible grain growth of the W-Re wire after the 10 000 h exposure, whereas the noble-metal wire exhibited large grain growth.

8. Fe and Si impurities were the main impurities commonly picked up by both the noble and refractory wires and appeared in like quantities, ranging from 200 to 2000 ppm, with Fe having the highest concentration increase at the junction and the largest concentration gradient near the junction.

9. Resistance measurements of the Pt leg of a noble-metal thermocouple pair show that the direction of the resistance change matches the direction of the thermal-emf change to be expected when the wire becomes contaminated.

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TABLE I. - THERMOCOUPLE WIRE PAIRS TESTED

Thermocouple pair	Wire diam, mm	Number of thermocouples tested		
		In air	In vacuum	In argon
87Pt13Rh/Pt	0.33	-	3	3
	.51	1	3	3
	[a].51	-	6	-
	.81	1	-	-
	1.12	1	-	-
70Pt30Rh/94Pt6Rh	0.51	-	3	3
95W5Re/74W26Re	0.51	-	5	4

[a]In test Facility II.

TABLE II. - SUMMARY OF DRIFT TEST RESULTS, FACILITY I

Thermocouple pair	Environment	Wire diam, mm	Time tested, hr	Final drift, K	Cause of termination
87Pt13Rh/Pt	Argon	0.51	10 000	-18.8 to -27.6 (3 couples)	Test complete
		0.33	5 600	-19.2	Open circuit
			10 000	-58.8	Test complete
			10 000	-71.8	Test complete
	Vacuum	0.51	1 610	-1.5	Open circuit
			1 760	-2.6	Open circuit
			1 970	-4.7	Open circuit
			[b]3 700	-2.0 to -3.6 (6 couples)	Power failure
		0.33	3 170	-6.8	Open circuit
			3 430	-8.0	Open circuit
			4 080	-8.5	Capsule failure
	Air	1.12	10 000	2.9	Test complete
		.81	10 000	-.1	Test complete
		.51	10 000	-4.9	Test complete
70Pt30Rh/94Pt6Rh	Argon	0.51	10 000	-7.2 to -18.1 (3 couples)	Test complete
	Vacuum	0.51	2 780	-3.2	Open circuit
			2 950	-4.0	Open circuit
			4 110	-4.7	Capsule failure
95W5Re/74W26Re	Argon	0.51	10 000	-21.1 to -22.5 (4 couples)	Test complete
	Vacuum	0.51	3 480	-14.3	Capsule failure
			3 480	-15.3	Capsule failure
			3 700	-15.9	Capsule failure
			3 860	-15.4	Capsule failure
			3 860	-15.7	Capsule failure

[a]Erratic signals during test.

[b]In test Facility II.

TABLE III. - SUMMARY OF EXPECTED DRIFT

Thermocouple pair	Wire diam, mm	Expected drift per 10 000 hr, K		
		In air	In vacuum	In argon
87Pt13Rh/Pt	0.33	----	[a]-65	[b]-65
	.51	-5	[a]-23	-23
	.51	----	[c]-23	----
	.81	-0.5	----	----
	1.12	3	----	----
70Pt30Rh/94Pt6Rh	0.51	----	[a]-26	-13
95W5Re/74W26Re	0.51	----	-22	-22

[a]Open-circuit failure expected after 2000 to 4000 h. The values shown assume the same shape curve as in argon.

[b]Erratic behavior expected after 4000 h.

[c]Result of test in Facility II, terminated after 3700 h. Same extrapolation as for preceeding line entry.

TABLE IV. - KNOOP HARDNESS OF TEST SPECIMENS USED IN FACILITY I

	Prior to test	10 000 h 1250 K Ar	10 000 h 1600 K Ar	10 000 h 1600 K Air
Pt	<40	<40	201	<40
94Pt6Rh	108		156	
87Pt13Rh	141	125	126	87
70Pt30Rh	192		158	
95W5Re	480		419	

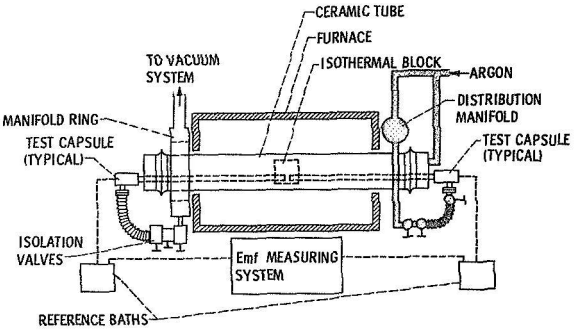
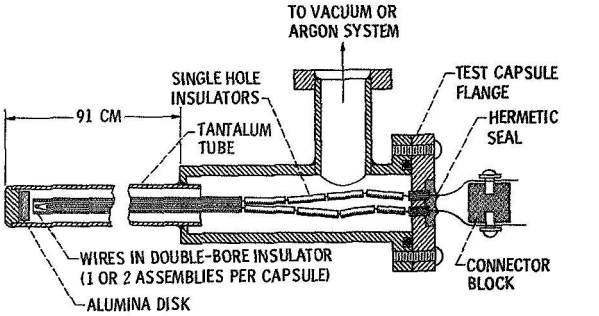
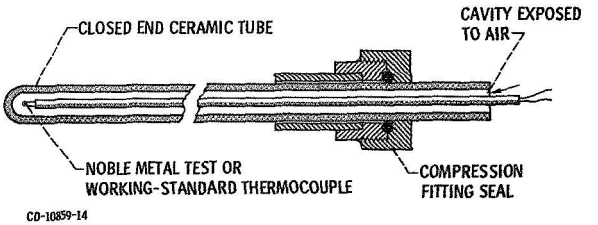


Figure 1. - Overall arrangement of Facility I. The cooling system is not shown.



(A) FOR ARGON AND VACUUM.



(B) FOR AIR.

Figure 2. - Test capsules for thermocouple assemblies of Facility I.

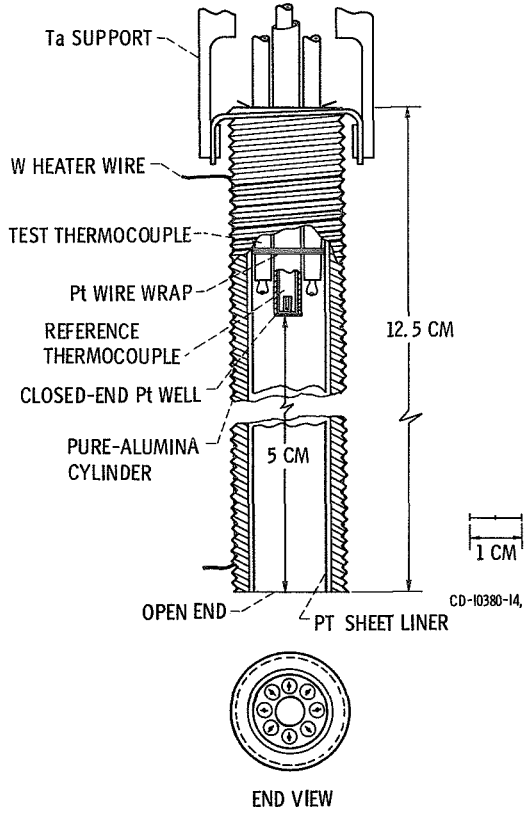


Figure 3. - Test section of Facility II.

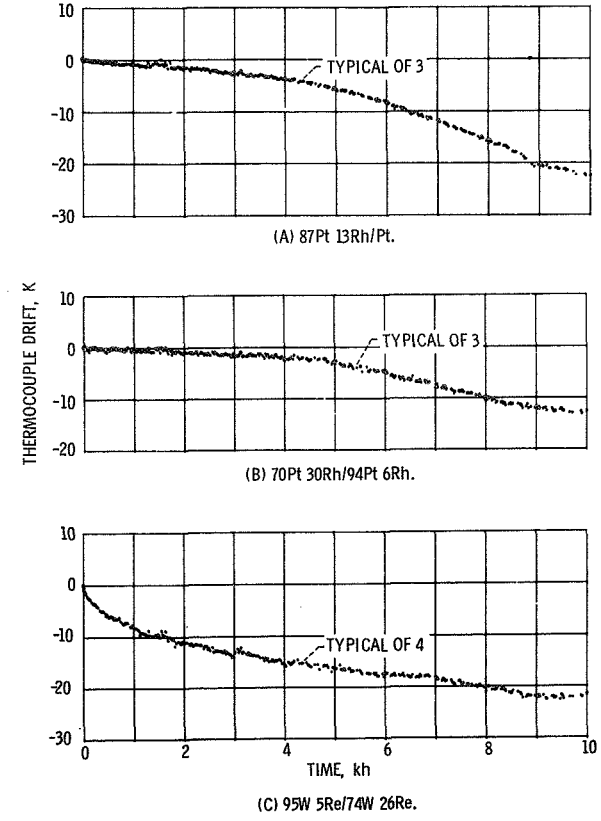


Figure 4. - Drift of 0.51-mm diam thermocouples in Ar at 1600 K for 10 000 h.

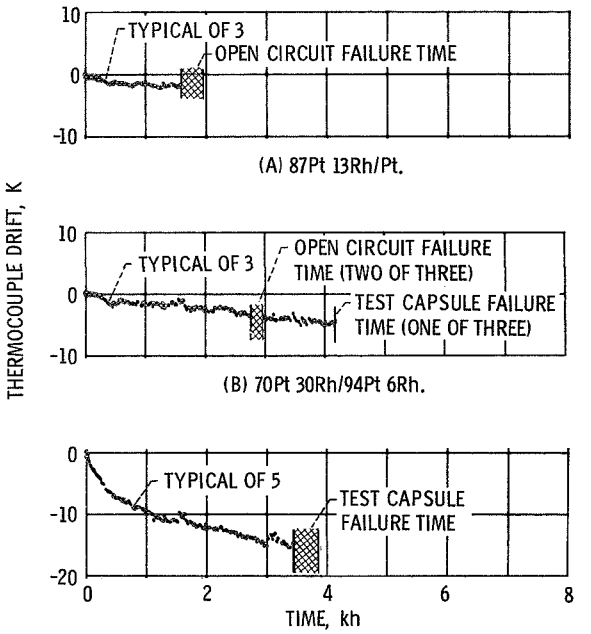


Figure 5. - Drift of 0.51-mm diam thermocouples in 10^{-6} torr vacuum at 1600 K.

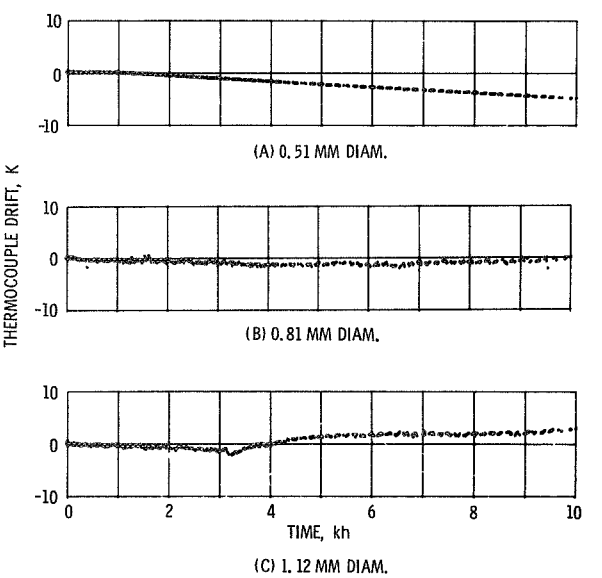


Figure 6. - Drift of various sizes of 87Pt 13Rh/Pt thermocouples in air at 1600 K for 10 000 h.

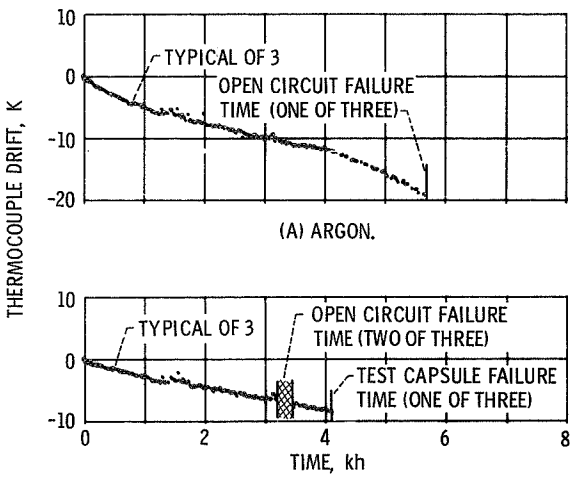


Figure 7. - Drift of 0.33-mm diam 87Pt 13Rh/Pt thermocouples in Ar and in 10^{-6} torr vacuum at 1600 K.

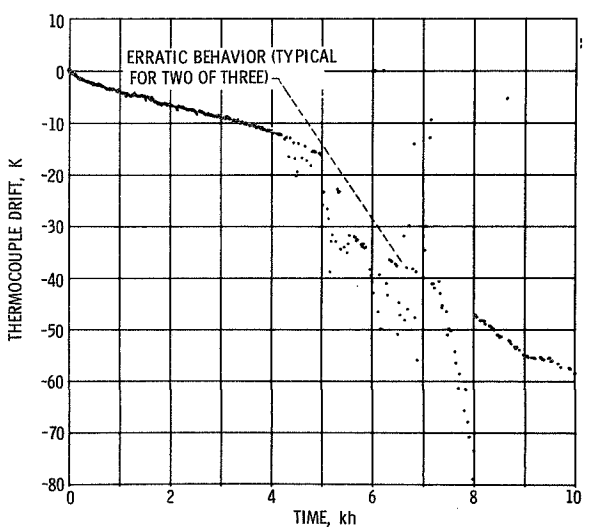
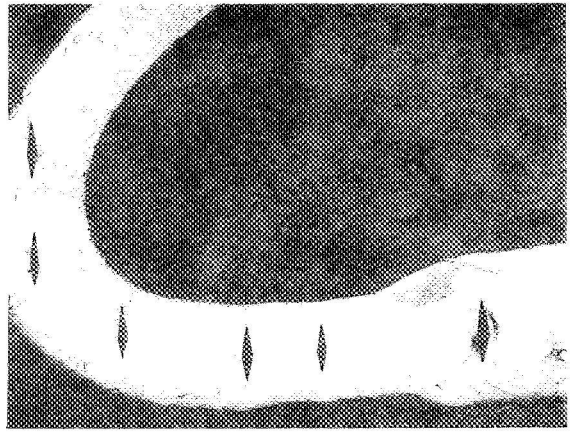
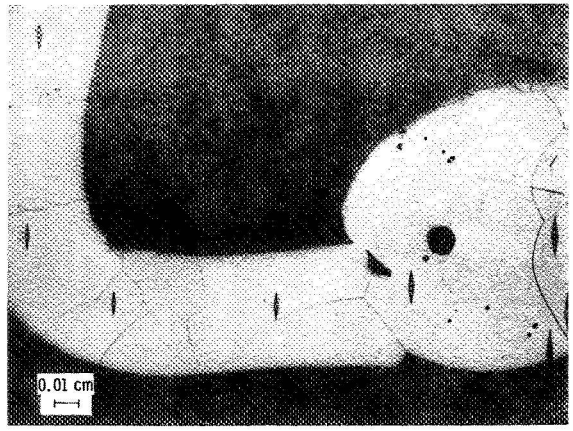


Figure 8. - Erratic drift of a 0.33-mm diam 87Pt 13Rh/Pt thermocouple in Ar at 1600 K.

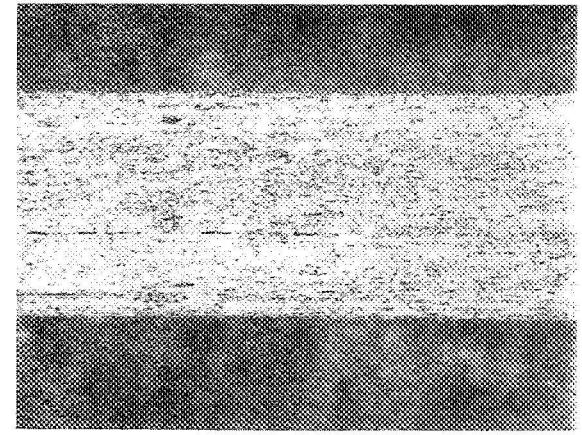


(a) Prior to test.

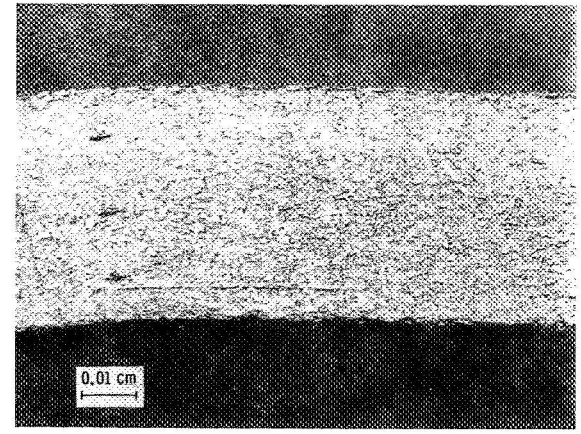


(b) After test.

Figure 9. - 87Pt13Rh/Pt junction and the adjacent 87Pt13Rh leg, before and after the drift test.

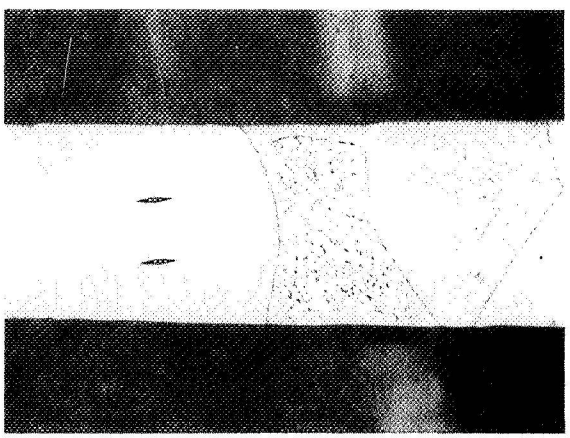


(a) Prior to test.

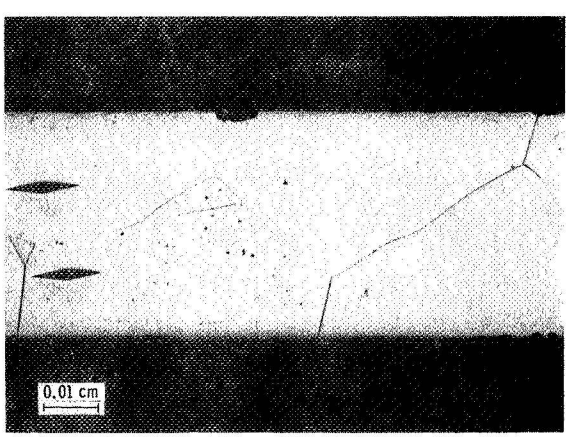


(b) After 10 000 h in Ar at 1600 K.

Figure 10. - Longitudinal section of 95W5Re thermocouple wires before and after 10 000 h test in Ar at 1600 K.



(a) After 10 000 h in Ar at 1600 K.



(b) After 10 000 h in air at 1600 K.

Figure 11. - Comparison of inclusion content of Pt wires in Ar or air, after 10 000 h at 1600 K.