

THIN FILM THERMOCOUPLES FOR HIGH TEMPERATURE MEASUREMENT  
ON CERAMIC MATERIALS

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## SUMMARY

Thin film thermocouples have been developed for use on metal parts in jet engines to 1000°C. However, advanced propulsion systems are being developed that will use ceramic materials and reach higher temperatures. The purpose of this work is to develop thin film thermocouples for use on ceramic materials. The thin film thermocouples are Pt13Rh/Pt fabricated by the sputtering process. Lead wires are attached using the parallel-gap welding process. The ceramic materials are silicon nitride, silicon carbide, aluminum oxide, and mullite. Both steady state and thermal cycling furnace tests were performed in the temperature range to 1500°C. High-heating-rate tests were performed in an arc lamp heat-flux-calibration facility.

The fabrication of the thin film thermocouples is described. The thin film thermocouple output was compared to a reference wire thermocouple. Drift of the thin film thermocouples was determined, and causes of drift are discussed. The results of high-heating-rate tests up to 2500°C/sec are presented. The stability of the ceramic materials is examined.

It is concluded that Pt13Rh/Pt thin film thermocouples are capable of meeting lifetime goals of 50 hours or more up to temperatures of 1500°C depending on the stability of the particular ceramic substrate.

## INTRODUCTION

Thin film thermocouples have been available for some time for use on metal parts in jet engines to 1000°C (ref. 1-8). However, advanced propulsion systems are being developed that will use ceramic materials and have the capability of attaining higher temperatures in their operation. Newer thin film thermocouples have been tested on silicon nitride at 1000°C (ref. 9). Additional testing of thin film thermocouples on several ceramic materials up to 1400°C has been performed (ref. 10-11).

The purpose of this report is to describe the effort to develop thin film thermocouples for use on ceramic materials and determine the temperature range and operating conditions for which they are capable of being used. The thin film thermocouples used were Pt13Rh/Pt fabricated by the sputtering process, and the ceramic materials were silicon nitride, silicon carbide, aluminum oxide, and mullite. Both steady state and thermal cycling furnace tests were performed in the temperature range from 1000-1500°C. High-heating-rate tests were

also performed in an arc lamp heat-flux-calibration facility (ref. 12) to 1500°C.

## APPARATUS AND EXPERIMENTAL PROCEDURE

The ceramic materials that were used in this program were silicon nitride, silicon carbide, aluminum oxide, and mullite. Tab. I lists the purity of these materials, the fabrication procedures, and some of the physical properties. The low purity of the silicon nitride is caused by the addition of 13 percent yttria and 3 percent alumina as densification agents for the sintering process. The surface finish is of particular interest because the thin film thermocouple is deposited on the surface of the ceramic materials. Surface finish varied from about .025-.15  $\mu\text{m}$  for the aluminum oxide to about .5-.75  $\mu\text{m}$  for the silicon nitride.

Fig. 1 shows a schematic diagram for the fabrication of thin film thermocouples on ceramic substrates. For the electrically insulating ceramic substrates, the thin film thermoelements are deposited directly on the surface. For the electrically conducting ceramic substrate (silicon carbide), an insulating layer must be deposited between the sensor and the substrate. A two-layer approach was used, starting with a stable, adherent, thermally grown silicon dioxide, followed by a sputter-deposited layer of aluminum oxide of the thickness needed to obtain the required insulation resistance. Each of the two layers was 1-2  $\mu\text{m}$  thick. The thin film thermocouples are Pt13Rh/Pt and are 5-7  $\mu\text{m}$  thick. They were deposited using the RF magnetron sputtering process at a sputtering power of 800 watts and sputtering rate of 5  $\mu\text{m}/\text{hr}$ . Substrate heating of about 300°C was used for the Pt and Pt13Rh films, and oxygen-enhanced sputtering of Pt was used for the first .02 $\mu\text{m}$  of deposition to improve film adhesion. Sputtering parameters were chosen based on previous work (ref. 5) and experience at this laboratory.

Fig. 2 is a photograph of the test samples used in these experiments. The ceramic substrates are 15 cm long and 2.5 cm wide, and the thicknesses are 1.5 mm for aluminum oxide, 4.5 mm for mullite, and 6 mm for silicon nitride and silicon carbide. They were cemented to an aluminum oxide support plate using an alumina-based cement with no binders. The thin film thermocouple deposited on the test sample was at least 12.5 cm long with film widths of about 3 mm. Pt13Rh/Pt lead wires were attached to the thin films using the parallel-gap welding process described in detail in ref. 13. These wires were 75  $\mu\text{m}$  in diameter and were routed through ceramic tubing to connectors.

Two ceramic tube furnaces were used in these experiments. The furnaces had a maximum temperature capability of 1300 and 1700°C, respectively. Set-point control of the furnaces was about  $\pm 1^\circ\text{C}$  in the central core of each furnace. Reference thermocouples made from 0.5 mm Type R (Pt13Rh/Pt) material were used to monitor these temperatures. Negligible drift rates for these reference thermocouples were observed throughout the testing; they verified the stability of the furnaces. Thermoelectric potentials were measured with a digital voltmeter with a sensitivity of 1  $\mu\text{volt}$  and an accuracy of  $\pm 0.01$  percent + 5  $\mu\text{volts}$ . Cold junction temperature was recorded but not controlled, and the emf data were corrected to 0°C. Furnace testing of the thermocouples took place in steady state and thermal cycling modes. For steady-state tests, two types of test sample configurations were used. In one configuration, the test sample was only partially inserted into the furnace, resulting in a large temperature gradient along the length of the thin film thermocouple up to a maximum of

about 600°C. This temperature gradient is shown in Fig. 3 as a function of furnace temperature for silicon nitride and silicon carbide test pieces. In the other configuration, the entire test sample and part of the support plate was inserted into the furnace, resulting in a small temperature gradient along the length of the thin film thermocouple up to a maximum of about 100°C. These two test configurations were chosen to evaluate the effect of different temperature gradients on drift rate patterns in thin film thermocouple circuits. The steady-state tests were carried out in the temperature range from 1000-1500°C. Thermal cycling accompanied repeated steady-state tests of a particular test sample.

A total of 15 test samples was fabricated and tested for these experiments. Total test time was about 1000 hours. The lifetime goal of a sensor for advance propulsion system applications was about 50 hours. For laboratory testing, longer lifetimes would be desirable.

High-heating-rate tests were also performed, using an arc lamp heat-flux-calibration facility. Currents of 50-400 amps are generated in the lamp to produce heat fluxes in the range from about 0.1-5 Mw/m<sup>2</sup>.

## RESULTS AND DISCUSSION

The discussion of the results of these tests is divided into three main parts. First, the initial accuracy of the thin film thermocouple, as fabricated, will be discussed. Second, the subject of thermocouple drift will be discussed to illustrate the causes and effects of the change in thermocouple output with time. And last, an analysis will be made of the physical durability of the thin film thermocouples, which includes a discussion of the physical durability of the ceramic materials as well.

### Calibration of Thin Film Thermocouples

A calibration experiment was performed to determine the accuracy of the thin film thermocouple as fabricated by the sputtering process. The thin film thermocouple was fabricated on a silicon nitride substrate in the configuration shown in Fig. 2. A wire thermocouple made from the same 75- $\mu$ m-diameter wire used for the lead wires of the thin film thermocouple was cemented to the back of the test piece directly opposite the thin film thermocouple junction.

The test was performed in the configuration in which the test piece was only partially inserted into the furnace, resulting in the maximum attainable temperature gradient along the length of the thin film thermocouple. In a separate experiment, the value of this temperature gradient was determined as a function of furnace temperature; it is shown in Fig. 3. These data were obtained by cementing an additional wire thermocouple to the back of the test piece opposite the thin-film-to-lead-wire connection.

The calibration test results showed that the thin film thermocouple output was 3 percent less than the wire thermocouple. The result is expressed as percent of the temperature gradient applied to the thin film. The major uncertainty in the experiment is caused by the severe temperature gradient on the test piece and the inability of the cemented

reference thermocouple to indicate the exact test piece temperature at the exact location of the thin film thermocouple junction.

### Causes of Thin Film Thermocouple Drift

Thermocouple drift is defined as a change with time in the voltage vs temperature characteristic of a thermocouple. Suspected causes of thermocouple drift in these thin film thermocouples are oxidation of Rh in the Pt13Rh thermoelement, foreign material at the thin-film-to-lead wire connection, and chemical interaction or diffusion between the sensor and the substrate.

Preferential oxidation of rhodium in the Pt13Rh leg of the thermocouple would cause a change in the Pt/Rh ratio in that leg and result in thermocouple drift. This oxidation rate increases as temperature increases, but there is a conversion of the oxide back to elemental rhodium at a temperature of about 1000°C and above. Oxidation rate is also proportional to the surface area/volume ratio of the thin film sensor and lead wire geometry. The value of this ratio is at least 4 times greater for a 5 μm thin film compared to a 75 μm diameter lead wire. Finally, oxidation rate is dependent on the quantity of oxygen present in the gaseous environment surrounding the thermocouple. In these experiments, ambient air was the environment for all of the thermocouples.

The thin-film-to-lead-wire connection could be a source of thermocouple drift if a foreign material, such as a cement or paste, were introduced into the thermocouple circuit at this point to make the connection. But in these experiments, connections were made using the parallel-gap welding process, which eliminates this source of thermocouple drift.

Thermocouple drift could originate at the substrate-sensor interface if a chemical reaction were to occur at this interface or if diffusion of material into or out of the thermocouple were to occur that would change the thermoelectric characteristics of either thermoelement.

### Results of Thermocouple Drift

Drift-rate data for thin film thermocouples on ceramic materials are shown in Figs. 4 and 5 for steady state tests. The data are plotted as drift rate in °C/hr against the steady state temperature, and each point represents the average drift rate of a steady state test. Also shown on each figure is the temperature gradient across the thin film portion of the thermocouple circuit. In Fig. 4, the tests were performed on silicon nitride and silicon carbide substrates with a large temperature gradient of 500-600°C across the length of the thin film. This was accomplished by inserting only part of the test samples (shown in Fig. 2) into the testing furnace. With the hot junction of the thin film thermocouple at about 1000-1200°C in these tests, the lead wire end of the thin film thermocouple would be about 500-700°C; thus a large portion of the thin film would be in the temperature range where rhodium oxidation occurs. The result is a drift rate of about 0.5°C/hr. In these tests, the region of rhodium oxidation was easily seen by the formation of a dark deposit on the Pt13Rh

thermoelement. Tests were also performed where the temperature gradient along the length of the thin film was only about 100°C (Fig. 5). In these tests, the entire test piece and part of the support plate is inserted into the testing furnace. For a test where the hot junction of the thin film is at 1200°C, the lead-wire end of the thin film would be at about 1100°C; thus only the lead-wire portion of the thermocouple circuit would be in the temperature range where rhodium oxidation occurs. This results in a drift rate of less than 0.2°C/hr for the data in Fig. 5 between about 1000-1200°C. There is no dark deposit on the Pt13Rh thin film thermolement.

At temperatures greater than about 1250°C in Fig. 5, drift rates rapidly increase as test temperature increases. It is suspected that a sensor-substrate interaction is beginning to occur in this temperature range, either because of a chemical reaction or a diffusion effect. It should also be noted that the drift rate is not the same for each substrate material in this higher temperature range. Auger depth profiling analysis is being used to analyze these effects.

A thermocouple probe was fabricated completely from 75 µm lead wire in order to separately determine lead-wire drift rate. The drift rate was determined at three temperature levels. The drift rate was .03°C/hr at 1150°C, 0.1°C/hr at 1370°C, and 0.3°C/hr at 1500°C. These lead-wire drift rates are tabulated in Fig. 6 along with selected values of the drift rates of the thin film thermocouples on the four ceramic materials. Figs. 4-6 illustrate the complexity of thermocouple drift of thin film thermocouples, which are in actuality composite thin film/lead-wire thermocouple circuits. We can summarize the information contained in Figs. 4-6. Drift rate varies with: the absolute temperature level; the substrate material on which the thin film thermocouple is deposited; the temperature gradient distribution between the thin film and the lead-wire portion of the circuit; and the film thickness and diameter of the thin films and lead-wires, respectively. And for every application, some of these factors could well be different.

Fig. 7 is a plot of drift in °C against time for thin film thermocouples on two test pieces. One test piece was silicon carbide tested at 1100°C for 95 hours with a 500°C temperature gradient across the thin film portion of the thermocouple circuit. In this test, the primary cause of drift was expected to be oxidation of the rhodium in the Pt13Rh leg, and such oxide was visible at the conclusion of the test. It was also expected that the oxide growth rate would follow a parabolic rate law, because of the passivating effect of the rhodium oxide layer; and this in turn would cause a similar functional relationship between temperature drift and time, which can be seen to be the case in Fig. 7.

The other test results plotted in Fig. 7 are for a thin film thermocouple deposited on an aluminum oxide substrate, tested at 1500°C for 20 hours, with a 100°C temperature gradient across the thin film portion of the thermocouple circuit. In these test conditions, the cause of drift was suspected to be sensor-substrate interaction caused by chemical reaction or diffusion. The functional relationship of temperature drift with time is seen to be approximately linear.

### Durability of Thin Film Thermocouples

The four ceramic materials used in this research program exhibited significantly

different characteristics when exposed to high temperatures. The oxide ceramics, aluminum oxide and mullite, showed little visible surface deterioration when exposed to the entire temperature range of these experiments (1000-1500°C). The aluminum oxide was 99.6% pure and the mullite was a 98% pure mixture of aluminum oxide and silicon dioxide (60 to 38 ratio). Despite the lack of visible surface deterioration, the thin film sensors showed a significant increase in drift rate on these substrates above about 1300°C (see Fig. 5), indicating some form of sensor-substrate interaction. Very little degradation of the sensor structure occurred.

The non-oxide ceramics, silicon nitride and silicon carbide, exhibited visible surface changes during these tests. The silicon nitride was fabricated by the hot-pressed method using 13% yttria and 3% alumina as densification agents. It was observed during the testing process that this material formed a complex surface oxide, and that the rate of oxidation increased dramatically at temperatures above about 1250°C. As the oxide formation increased in magnitude, it caused a gradual bubbling and delamination of the thin film sensor material.

The silicon carbide was 99% pure and required an insulating layer to be superimposed between the sensor and the substrate because it is an electrically conducting ceramic. The insulating layer consisted of a thermally grown silicon dioxide layer plus a sputter-deposited aluminum oxide layer. The silicon carbide showed no visible deterioration during testing up to about 1250°C, but above this temperature, the surface morphology began to change to a glassy appearance over a portion of its surface, and other nonuniformities in structure appeared. This change in surface morphology caused delamination of the thin film sensor material to begin.

A total of 15 test samples with thin film sensors were used in these experiments. Thermal cycling accompanied repeated steady-state tests of the same test sample up to a maximum of five cycles. No sensor failures occurred as a result of thermal cycling. Steady state testing of a single test sample occurred for various times up to a maximum of 149 hours. No sensor failures occurred as a result of total test time. The only sensor failures occurred on silicon nitride and silicon carbide substrates and correlated with a deterioration of the ceramic substrate when tested beyond a critical temperature level in the range above about 1250°C.

Photographs of the thin film thermocouple hot junctions were taken at different stages of the testing process (Figs. 8 and 9). Also shown in these figures is the number of hours of testing time and the maximum test temperature of each specimen. Fig. 8 shows the sensors on aluminum oxide and silicon nitride substrates. The sensors on aluminum oxide substrates show negligible degradation up to 1355°C. Oxide formation can be seen on the silicon nitride surface at 1167°C, and a dramatic increase in the magnitude of the oxide formation is seen at 1343°C, causing a bubbling of the sensor material and leading to delamination. Sensors on silicon carbide substrates are shown in Fig. 9. Negligible degradation of sensor films is seen up to 1246°C, but at 1322°C, morphological changes in the substrate have begun to appear, leading to the start of sensor delamination.

Jacobson (ref. 14) discusses the durability of ceramic materials for use in advanced propulsion systems. He points out that the ceramic materials will degrade chemically by oxidation, vaporization, and interfacial reactions. For the oxide ceramics, such as aluminum oxide and mullite, vaporization is the major mechanism. In these experiments, this could lead to a slow, gradual deterioration of the bond between the sensor and the substrate.

For the non-oxide ceramics, such as silicon nitride and silicon carbide, all three

mechanisms of oxidation, vaporization, and interfacial reactions are at work. For silicon nitride, the oxidation of the silicon nitride not only forms an interface at the oxide-substrate boundary, but can lead to a complicated structural interaction with yttria and alumina present as densification agents in the ceramic. This was seen in Fig. 8 to result in the rapid formation of an irregular oxide structure above 1250°C leading to sensor delamination. In the case of silicon carbide, the interfaces were deliberately formed by thermal oxidation and sputtered alumina to form the insulating layer for the sensor. Above 1250°C, the formation of a glassy layer and other irregular structure in the surface layers of the ceramic could be caused by interfacial reactions, phase change, or further oxidation. This leads to a deterioration between the sensor-substrate bond, and eventual delamination.

Lifetime goals for thin film sensors of 50 hours or more are feasible at temperature levels where a particular ceramic substrate is sufficiently stable. Each formulation of a ceramic must be evaluated to determine this limit.

### Heat-flux-calibration Facility Tests

Another aspect of sensor durability is the ability of the sensor to withstand high heating rates accompanied by rapid temperature excursions from room temperature to the maximum operating temperature of the sensors. The arc lamp heat-flux-calibration facility is capable of concentrating a high, known heat flux over a small, well-defined area. Lamp currents from 30-400 amps are used to generate heat fluxes from about 0.1-5 Mw/m<sup>2</sup> over a 1 by 4 cm area. Fig. 10 shows a test piece with a thin film thermocouple deposited on the surface in such a way that the hot junction is at the center of the focal area of the lamp. The test piece is silicon nitride, and a black coating is applied to a portion of the surface to increase the absorption of the radiant energy. A second thin film thermocouple is mounted on the back surface directly behind the front sensor. Fig. 11 shows the temperature rise vs time for the hot-side thermocouple for different lamp currents. Heating rates from about 2-2500°C/sec were generated in these tests. Silicon nitride and mullite were used. Maximum temperature was 1500°C, and maximum  $\Delta T$  across a ceramic was 560°C. No sensor failures occurred during these tests, and a single test piece was subjected to a maximum of 20 test cycles. Note that in these tests the total test time is measured in seconds or minutes rather than hours and therefore the ceramics suffered very little surface degradation.

### SUMMARY OF RESULTS

Pt13Rh/Pt thin film thermocouples were fabricated on ceramic substrates of silicon nitride, silicon carbide, aluminum oxide, and mullite using the sputtering process. They were tested in high temperature furnaces in steady state and thermal cycling modes in the temperature range from 1000-1500°C. The following results were determined:

1. The output of a Pt13Rh/Pt thin film thermocouple was lower than the output of a reference wire thermocouple by 3 percent of the value of the temperature gradient applied to the thin film.

2. A principal cause of thermocouple drift of the thin film thermocouple was determined to be rhodium oxidation of the Pt13Rh thin film thermoelement. A much smaller drift was caused by rhodium oxidation of the lead wire. Rhodium oxidation was confined to that portion of the thermocouple circuit below about 1000°C. Above this temperature the rhodium oxide dissociates. The rhodium oxidation proceeds at an approximately parabolic rate.
3. Above about 1250°C, thermocouple drift increased rapidly. The cause of this drift is presumed to be a chemical reaction or diffusion effect at the sensor-substrate interface.
4. Oxidation of the silicon nitride substrate was visible in tests above 1000°C and increased rapidly above 1250°C. Formation of this surface oxide led to bubbling of the thin film sensor and eventual delamination.
5. No physical change in the appearance of the silicon carbide was seen up to 1250°C. Above this temperature, the surface morphology changed to a glassy appearance accompanied by other nonuniform structural defects. These changes could be caused by interfacial reactions, phase change, or oxidation. The changes caused bubbling and delamination of the sensor to begin.
6. No physical change in the appearance of the aluminum oxide or mullite was seen in the temperature range of these experiments. Thin film sensors on these materials showed very little degradation.
7. Thin film sensors were tested for up to 149 hrs and five thermal cycles in furnace tests without failures attributable to these conditions alone. Lifetime goals for thin film sensors up to 50 hours or more appear feasible at temperature levels where a particular ceramic substrate is sufficiently stable.
8. Thin film sensors on silicon nitride and mullite were tested in an arc lamp heat-flux-calibration facility to a maximum temperature of 1500°C, heating rates from 2-2500°C/sec, and up to 20 thermal cycles, with no sensor failures.

## ACKNOWLEDGMENTS

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TABLE I- DESCRIPTION OF CERAMIC MATERIALS									
MATERIAL	FAB. METHOD	SURFACE FINISH, $\mu\text{m}$	THICKNESS, $\mu\text{m}$	DENSITY, $\text{gm}/\text{cm}^3$	THERMAL COND., $\text{W}/\text{m}\cdot\text{K}$	ELECTR. RES., $\text{ohm}\cdot\text{cm}$	MELTING POINT, $^{\circ}\text{C}$	TCE, $\alpha$ , $^{\circ}\text{C}^{-1}\times 10^{-6}$	PURITY, %
SILICON NITRIDE	SINTERED	.5-.75	6	3.28	30	$10^{14}$	1900	4	84
SILICON CARBIDE	SINTERED	.25-.5	6	3.1	125	10	2700	4	99
ALUMINUM OXIDE	TAPECAST	.075-.15	1.5	3.9	25	$10^{14}$	2040	8	99.6
MULLITE	HOT-PRESSED	.25-.5	4.5	3.6	4	$10^{14}$	1700	10	98

\*- Temperature coefficient of expansion

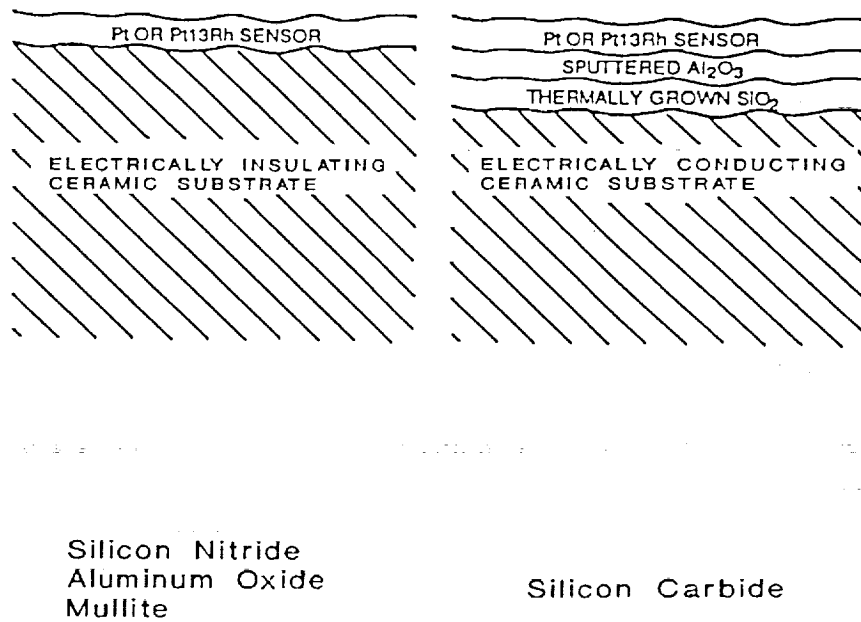


Fig. 1. Schematic diagram of thin film thermocouples on ceramic substrates.

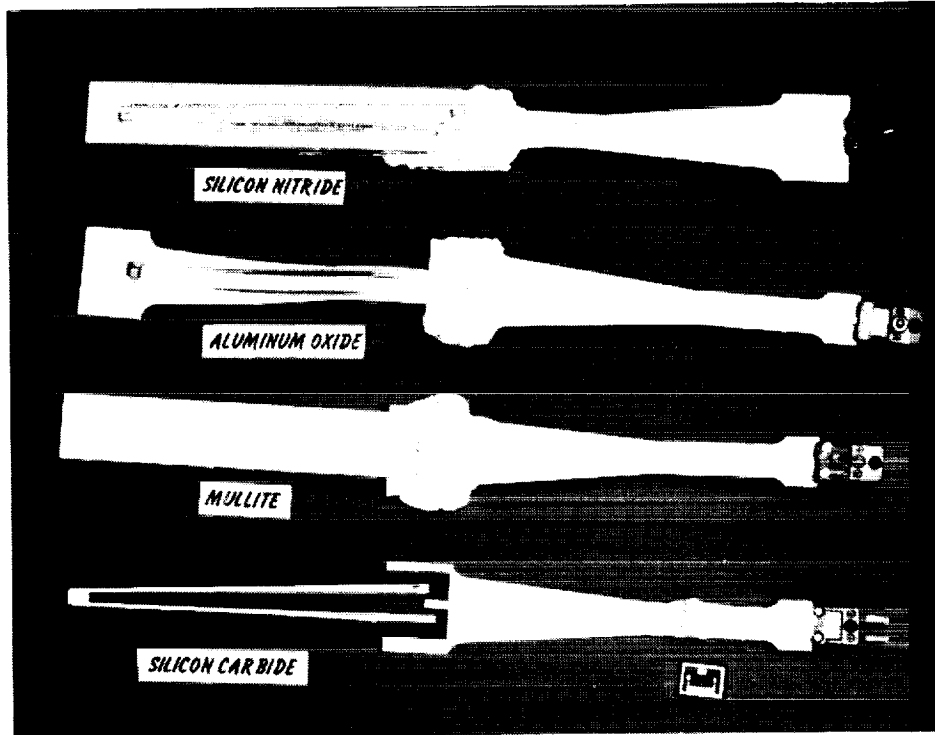


Fig. 2. Thin film Pt13Rh/Pt thermocouples on ceramic materials for high temperature furnace tests.

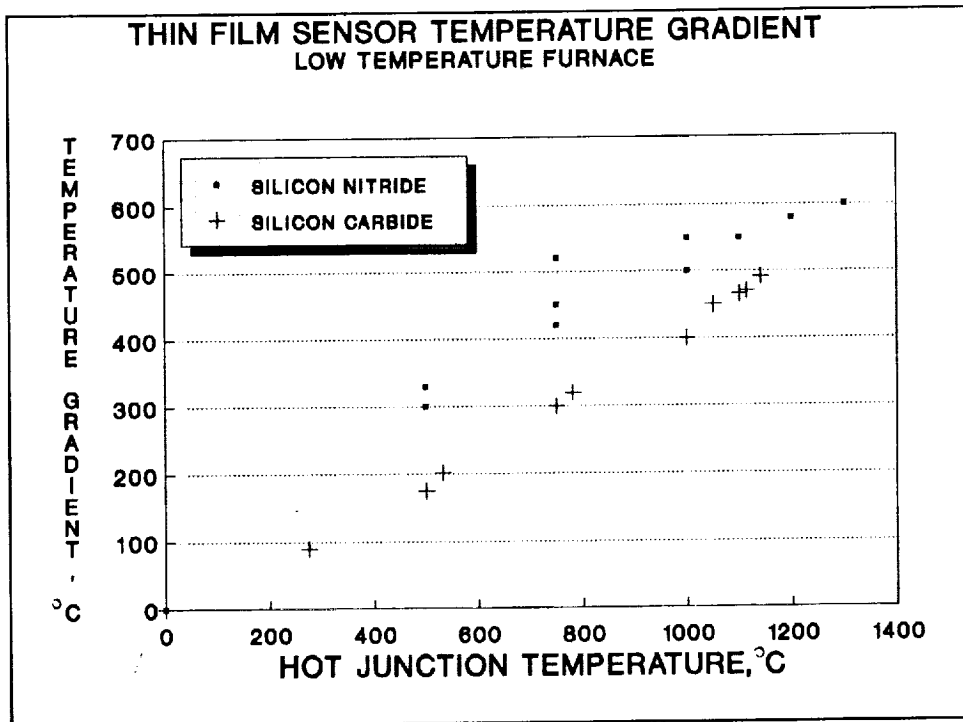


Fig. 3. Measurement of temperature gradient across thin film portion of thermocouple circuit on silicon carbide and silicon nitride substrates- high gradient configuration.

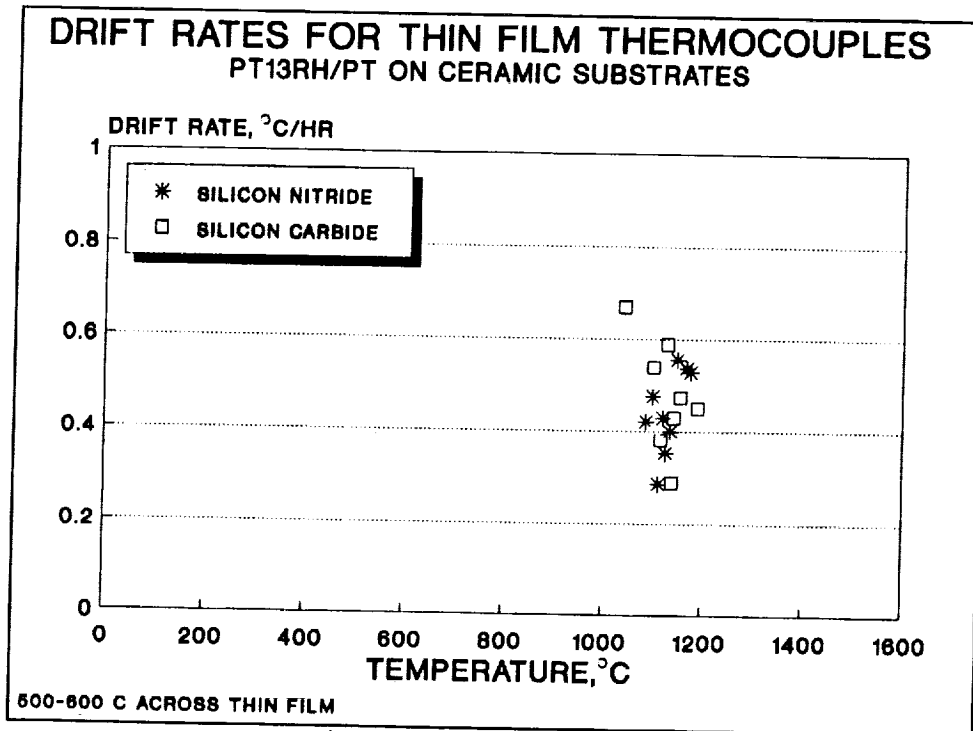


Fig. 4. Drift rates of Pt13Rh/Pt thin film thermocouples on ceramic substrates with 500-600°C temperature gradient across thin film portion of thermocouple circuit.

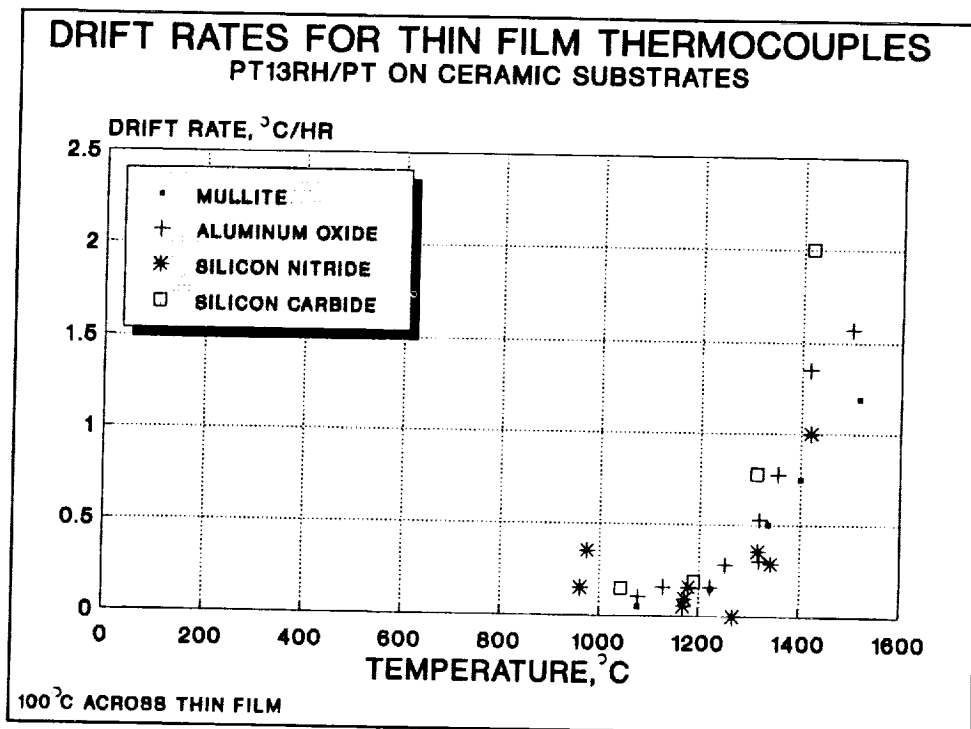


Fig. 5. Drift rates of Pt13Rh/Pt thin film thermocouples on ceramic substrates with 100°C temperature gradient across thin film portion of thermocouple circuit.

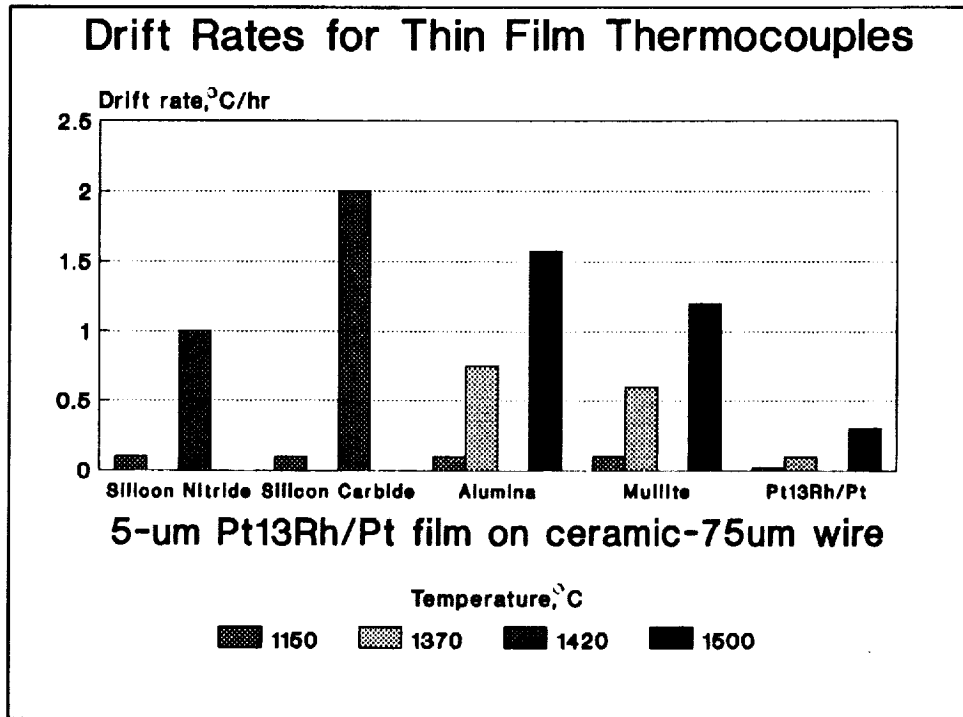


Fig. 6. Drift rates of 5  $\mu$ m Pt13Rh/Pt thin film thermocouples and 75  $\mu$ m lead wires at selected temperature levels.

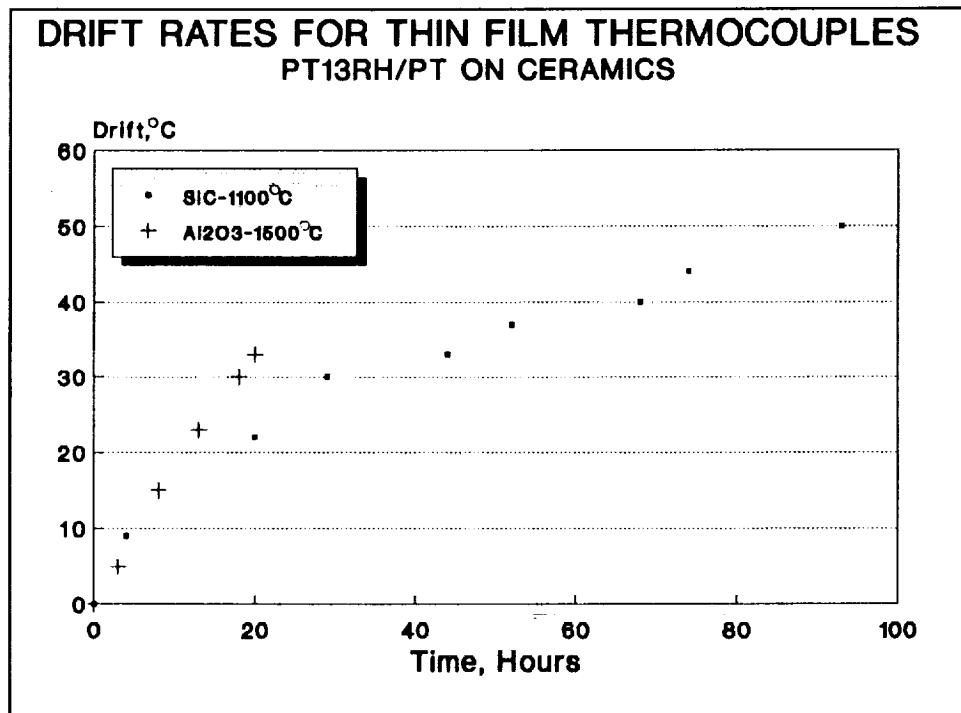
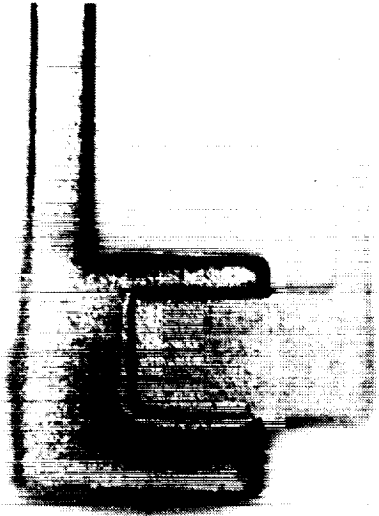
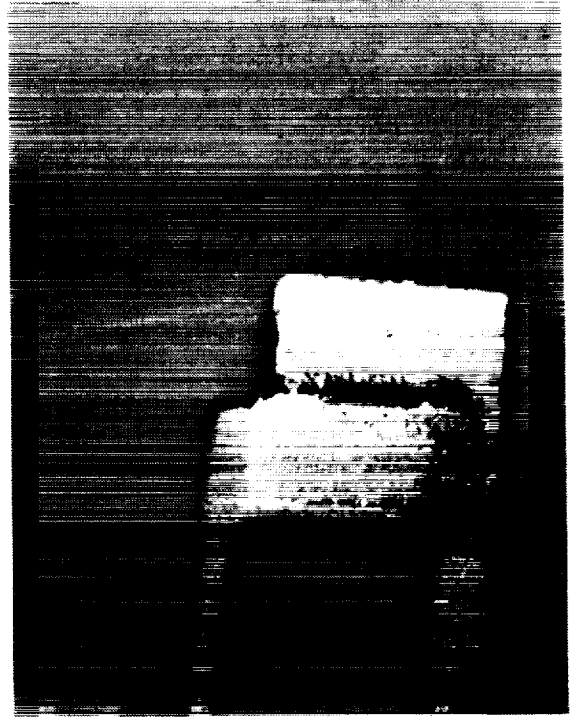


Fig. 7. Comparison of drift rates for Pt13Rh/Pt thin film thermocouples on silicon carbide at 1100°C and aluminum oxide at 1500°C.

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A. ALUMINUM OXIDE SUBSTRATE  
TEST TIME = 38 HOURS  
MAXIMUM TEST TEMPERATURE = 1216°C



B. ALUMINUM OXIDE SUBSTRATE  
TEST TIME = 94 HOURS  
MAXIMUM TEST TEMPERATURE = 1355°C



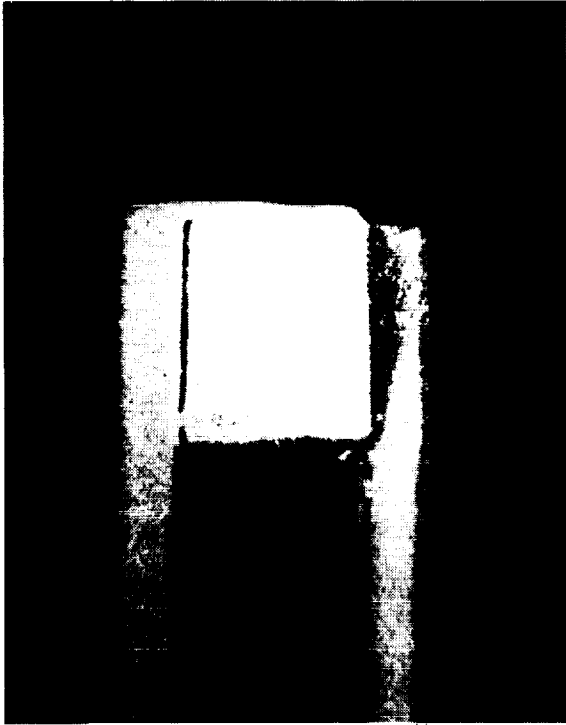
C. TEST TIME = 149 HOURS  
MAXIMUM TEST TEMPERATURE = 1167°C



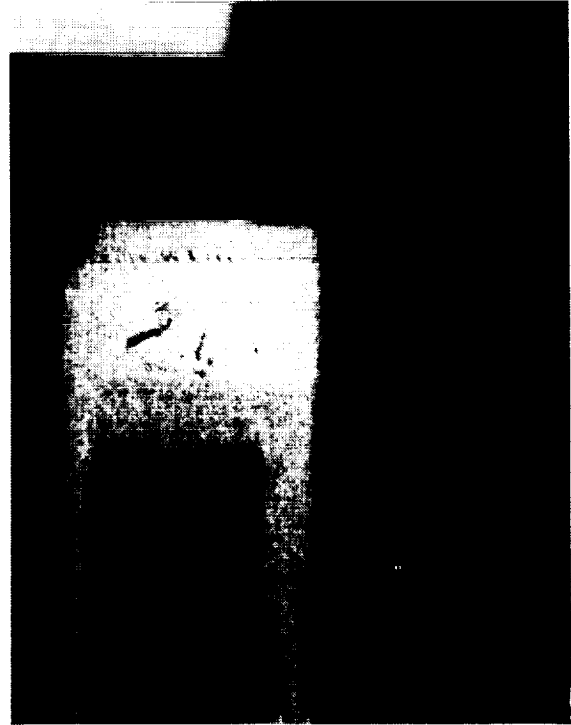
D. TEST TIME = 40 HOURS  
MAXIMUM TEST TEMPERATURE = 1343°C

Fig. 8. Pt13Rh/Pt thin film thermocouple hot junctions on aluminum oxide and silicon nitride at different stages of the testing process.

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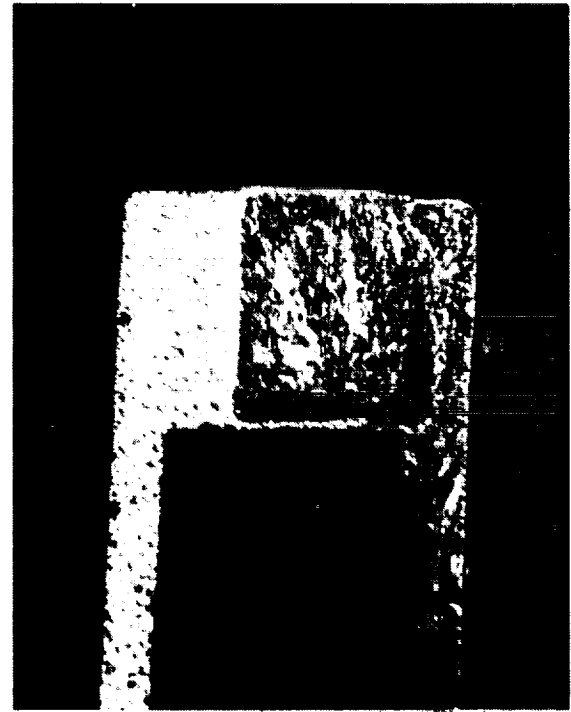
A. TEST TIME = 94 HOURS  
MAXIMUM TEST TEMPERATURE = 1142°C



B. TEST TIME = 93 HOURS  
MAXIMUM TEST TEMPERATURE = 1101°C



C. TEST TIME = 119 HOURS  
MAXIMUM TEST TEMPERATURE = 1246°C



D. TEST TIME = 56 HOURS  
MAXIMUM TEST TEMPERATURE = 1322°C

Fig. 9. Pt13Rh/Pt thin film thermocouple hot junctions on silicon carbide at different stages of the testing process.

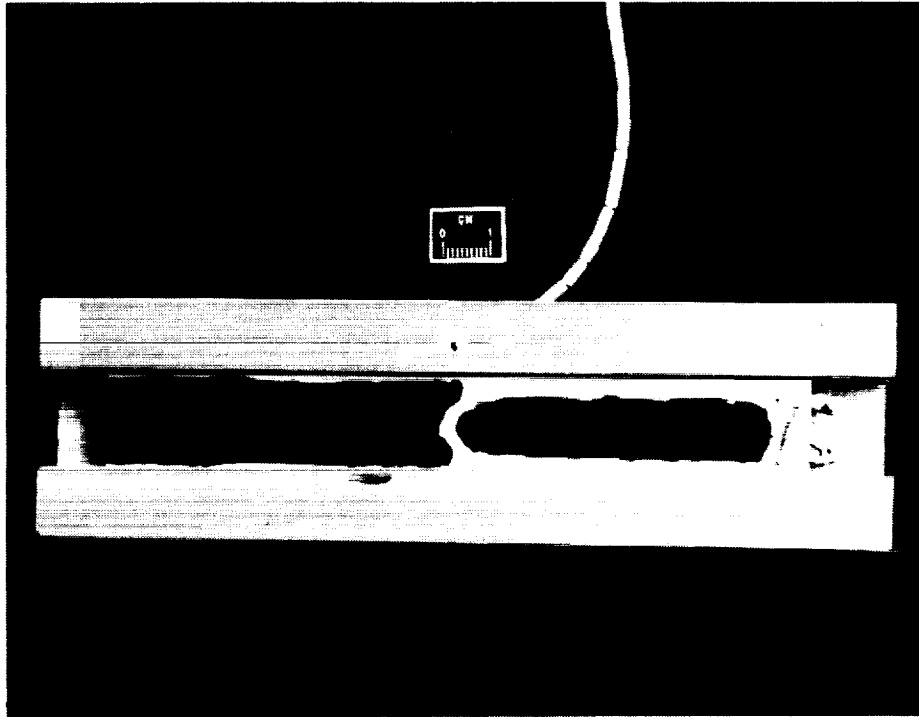


Fig. 10. Pt13Rh/Pt thin film thermocouple on silicon nitride substrate for arc lamp heat-flux-calibrator test.

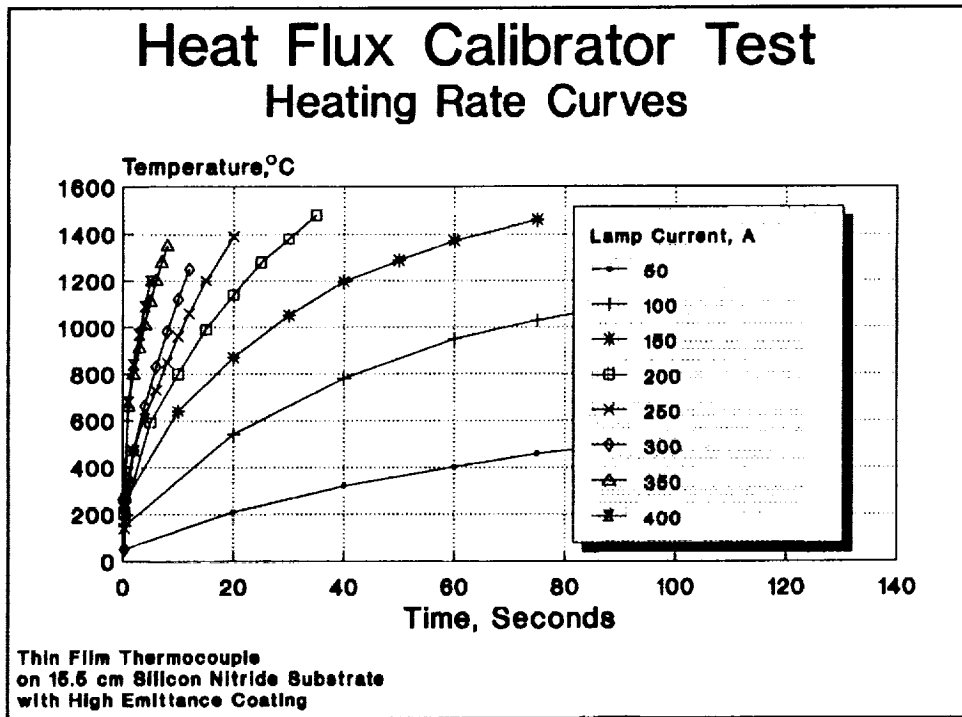


Fig. 11. Heating rate curves for Pt13Rh/Pt thin film thermocouples on silicon nitride substrate in arc lamp heat-flux-calibrator test.