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TURBINE BLADE TEMPERATURE MEASUREMENTS USING THIN FILM TEMPERATURE SENSORS

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FOREWORD

The work described in this report was accomplished by the Commercial Products Division of Pratt & Whitney Aircraft Group, United Technologies Corporation, under the National Aeronautics and Space Administration Contract NAS3-20831. Mr. Raymond Holanda of the NASA Lewis Research Center was the Project Manager for the program.

This report was prepared by Mr. Howard P. Grant, the Pratt & Whitney Aircraft Program Manager, Mr. John S. Przybyszewski, and Mr. Richard G. Claing.

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SECTION 1.0

SUMMARY

The development of thin film temperature sensors is being conducted by Pratt & Whitney Aircraft under a three-phase NASA program. Phase I, previously completed under Contract NAS3-20768, consisted of the analysis, design, development, fabrication, and testing of a basic temperature sensor system and the development of reliable thin-film-to-lead-wire connections. The thermocouple systems were applied to flat plate specimens of four turbine blade and vane materials and to simulated turbine blades and tested in a high temperature, low velocity exhaust gas environment. The results of this first phase of the program are documented in NASA CR-159782 (PWA-5526-31), Reference 6.

The efforts during Phase II, the subject of this document, under Contract NAS3-20831 consisted of applying the basic Phase I technology to actual turbine blades, reducing the sensor size, and extending the test conditions to higher temperatures and gas velocities. The problem of thin film adherence to the alumina (α-Al_2O_3) insulating layer, originally identified during Phase I, was extensively investigated under Phase II, and a procedure was established which increases the probability of achieving successful thin film sensors.

Problems of adapting fabrication procedures to turbine blades were uncovered and improvements were recommended. Test were performed in a combustor exhaust gas flow at 1250K, Mach number = 0.5 for 60 hours and 71 thermal cycles. Mean time to failure was 47 hours for six thin film thermocouple systems. Calibration drift was about 0.1 percent per hour, attributable to oxidation of the rhodium in the thin films. An increase in film thickness and application of a protective overcoat are recommended to reduce drift in actual engine testing.

The objective of the on-going Phase III of the program, under Contract NAS3-22002, is to improve the accuracy of the temperature sensor systems and to test these systems on actual-engine rotating turbine blades, with an intercomparison of turbine blade temperatures as indicated by optical pyrometers, reference wire thermocouples, and thin film thermocouples.

SECTION 2.0

INTRODUCTION

2.1 BACKGROUND

Turbine blade temperature measurement by blade-mounted sensors is presently accomplished by embedding thermocouples in the blade wall. The uncertainty in this method of measurement can be as high as 50K because of heat path distortion and uncertainty of exact location of the thermocouple junction. As blade wall thicknesses are decreased because of complex blade cooling schemes, the presence of embedded thermocouples becomes more adverse both from a structural and a measurement viewpoint. It is desirable to have a surface-mounted temperature sensor which does not require removal of any blade material and, at the same time, is of such minimum mass and thickness that it causes no serious heat path distortion within the blade or gas flow distortion over the blade. A thin film temperature sensor would be compatible with these considerations.

Using various thin film techniques (plating, vacuum evaporation, vacuum sputtering), a sensor can be formed by depositing layers of electrical insulating materials and metals whose thickness is only a few micrometers (μm). Successful surface thermocouples were obtained in this way by Burger in 1930 (Reference 1), Harris in 1934 (Reference 2), and Benderskey in 1953 (Reference 3). On metal parts, the technique was limited to surface temperatures below 800K due to the lack of a stable thin film insulating layer to provide reliable electrical isolation from the base metal at higher temperatures. Layers of quartz, SiO and Al_2O_3, deposited by either vacuum evaporation or sputtering, were found to be too brittle to withstand the severe thermal strains (up to 3.5×10^9 Pascals; 500,000 pounds per square inch) induced by the mismatch of temperature coefficients of expansion between the insulating layer and base metal.

Recent work at Pratt & Whitney Aircraft is summarized in Table I. The work described in columns 1 and 2 was performed by Dils (Reference 4), using solid FeCrAlY test pieces oxidized in air to form an insulating layer of aluminum oxide. The thermocouple legs were then sputtered on this insulator, and tests at 1350K were performed. This combination of fabrication methods and materials was able to withstand high temperatures. Thermal electromotive force was within 1.5 percent of standard reference Type S wire thermocouples, while a 600K temperature gradient was imposed along the length of the thin film. Drift was generally downward at less than 0.01 percent per hour at constant temperature, over a period of 150 hours (Reference 5).

The work described in columns 3, 4, 5, and 6 of Table I was performed by Przybyszewski (unpublished). In furnace tests on flat specimens (column 3), a typical turbine blade material (MAR-M-200 plus hafnium) was coated with a typical alumina-forming protective material (NiCoCrAlY); then platinum/platinum-10 percent rhodium thermocouples

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TABLE I

REVIEW OF PRATT & WHITNEY AIRCRAFT EXPERIENCE WITH SPUTTERED THIN FILM SENSORS ON MCrAlY SUBSTRATES AT HIGH TEMPERATURES

	1	2	3	4	5	6	7
Date	1972 - 75	1975	1975 - 76	1976	1976	1977	1979
Sputtered Sensor Type	Thermocouples	Thermocouples	Thermocouples	Strain gage	Erosion samples	Thermocouples	Thermocouples
Sputtered Film	Pt/Pt-10% Rh	Pt/Pt-10% Rh	Pt/Pt-10% Rh	Platinum	Platinum	Pt/Pt-10% Rh	Pt/Pt-10% Rh
Substrate Coating	---	---	NiCoCrAlY	NiCoCrAlY	NiCoCrAlY	NiCoCrAlY	NiCoCrAlY CoCrAlY
Base Material	FeCrAlY	FeCrAlY	MAR-M-200 + hafnium	Hastelloy X	B1900 + hafnium	B1900 + hafnium	MAR-M-509 B1900 + hafnium IN100 MAR-M-200 + hafnium
Lead-Wire-to-Film Connection	Strap weld	Strap weld	Fired platinum paste	Fired platinum paste	None	Fired paste	Hot compression bond
Configuration	Erosion bars, rods & airfoils	Cylinder	Flat bar	Strain bar	JT9D 1st-stage turbine blades	JT9D 2nd-stage turbine vanes	Flat bars
Test Facility	Lab combustor	Lab combustor	Lab furnace	Lab furnace	Operating engine	Operating engine	Furnace and torch
Time Above 1250K, Each Sensor	100 hours	180 hours	170 hours	32 hours	150 hours	60 hours	60 hours
Cycles to 1250K, Each Sensor	100	20	6	1			30
Reference Publications	(4) (5)	(5)	---	---	---	---	(6)
Remarks	20 specimens tested	Instrumented specimen for proof of calibration stability	Instrumented specimen for proof of calibration stability	Strain test specimen	Engine erosion test	Metal temperature and heat transfer coefficient	Developed lead wire attachments and ran extended hot calibrations of thin film thermocouples in hydrocarbon flame

were sputtered on a furnace-grown Al_2O_3 layer and tested to 1250K to demonstrate the feasibility of use on standard engine hardware. Film strain gages were also tested (column 4), and film erosion samples were prepared and tested (column 5) to further evaluate possible applications and assess potential problems. The strain gage tests demonstrated the feasibility of miniaturization of instrument sensor systems with pattern line widths down to 75 m, and the erosion tests proved the capability of thin film metal coatings to survive under engine conditions without prohibitive deterioration. Finally, an engine test of thin film thermocouple devices was performed with sensors mounted on turbine vanes (column 6). Although problems occurred with the thin-film-to-lead-wire connections, the sensors on the vanes survived engine operating conditions for 60 hours in most cases. No calibration data was obtained from this experiment.

The work in column 7 of Table I was performed under the Phase I contract, NAS3-20768, (Reference 6), of the on-going three-phase NASA program. Reliable lead wire attachments were developed, feasibility of sputtered thermocouples on four different turbine blade alloys was demonstrated, and eight sputtered thermocouple systems on flat bars were cycled 30 times to 1250K, with total time of 60 hours at 1250K for each, in a low velocity oxyacetylene flame. Average life was 42 hours, with less than 4 percent drift in calibration. Initial thermal electromotive force was between 1.4 percent above and 3.6 percent below reference Type S wire thermocouples, including effects of temperature gradients of up to 400K along the length of the thin films. Drift was generally downward at less than 0.1 percent per hour. The primary failure mode was loss of adherence of platinum films. A literature search and some preliminary experiments resulted in several recommendations for improving adherence.

2.2 OBJECTIVES

The objectives of Phase II, completed under the present Contract NAS3-20831, were as follows:

- o Determine the best method to improve adhesion of the thermocouple films to the oxide insulator.
- o Extend the flat surface technology developed in Phase I to the installation of a thin film temperature sensor on the intricately curved surface of an actual turbine blade.
- o Miniaturize the ensor slightly.
- o Adapt the lead-wire-to-thin-film connection to the turbine blade application.
- o Extend the test conditions to higher temperatures at the thin-film-to-lead-wire connection and higher velocities in the exhaust gas flow.

The objective of Phase III is to test sensor systems on actual engine rotating turbine blades, with intercomparison of three temperature measurement techniques: optical pyrometer, wire thermocouple, and thin film thermocouple. Test conditions include metal surface temperatures up to 1250K, blade tip speeds up to 400 meters per second, wheel diameters up to 95 centimeters, centripetal accelerations up to 35,000 g's, ten start-up and shutdown cycles, and 50 hours of total engine running time.

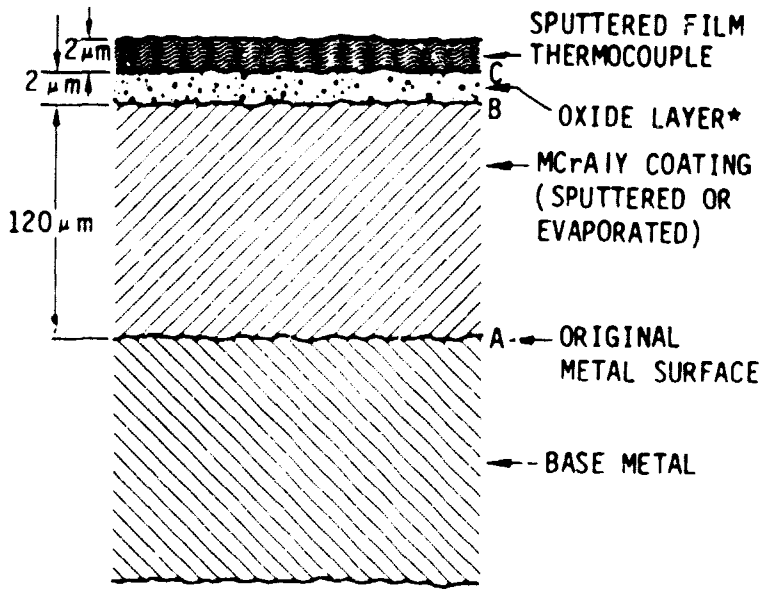
The goals throughout the three phases of the program are: 75 percent survival rate for the 50-hour steady-state portion of the test program, $\pm$ 1.2 percent initial calibration accuracy of a thin film thermocouple, and drift rates of less than 4 percent in 50 hours from the initial calibration value.

SECTION 3.0

TECHNICAL APPROACH

3.1 THIN FILM SENSOR

The thin film thermocouple structure selected, based on the experience described in Section 2.1, is illustrated in Figure 1. A section through one leg of the thermocouple is shown.



* THE STABLE ADHERENT Al_2O_3 INSULATING LAYER IS OBTAINED BY AT LEAST 50 HOURS OXIDATION (AT 1300K) OF THE COATING, FOLLOWED BY Al_2O_3 SPUTTERING.

Figure 1 Thin Film Thermocouple Cross-Section

The thermocouple legs are platinum and platinum-10 percent rhodium. This thermocouple is designated Type S by ANSI Standard MC 96.1 and is metallurgically the simplest of those recommended for service up to 1800K.

The key ingredient in the structure shown in Figure 1 is the alumina forming "MCrAlY" coating. The coating composition is typically 18 percent Cr, 12 percent Al, 0.5 percent Y, and the balance "M", where "M" is selected for best compatibility with the turbine blade material.

"M" may be Fe, Ni, Co, or a combination of Ni and Co. The attractive property of this coating is that at high temperatures in air, a dense layer of Al_2O_3 forms on the surface. When properly grown and filled by sputtering an additional amount of Al_2O_3 onto the surface, this Al_2O_3 coating is an excellent insulating material with high dielectric breakdown voltage. In addition, this oxide layer is mechanically tough and adherent, resisting erosion and spalling through extended temperature cycling and exposure to high velocity contaminated gas flows. When the oxide layer eventually spalls or erodes, a new protective oxide soon grows to take its place. It is this property that provides extended corrosion protection. The useful life of a thin film sensor sputtered onto the oxide is ended when the first oxide spalling occurs

The MCrAlY coatings, when properly formulated, are ductile, so that severe mechanical and thermal strains are not transmitted to the hard oxide layer. The strains within the Al_2O_3, therefore, remain within the elastic and buckling limits.

The strong bond at "B" in Figure 1, between the oxide layer and the MCrAlY material, has been the subject of extensive study and may be explained by the presence of the yttrium which combines with other constituents of the coating to form yttride "pegs" extending into the grain boundaries of the coating.

The bond at "C" in Figure 1, between the oxide layer and the noble metal of the thin film thermocouple, is not inherently strong because the platinum and platinum-10 percent rhodium sensor films do not readily react chemically with, or chemically bond to, Al_2O_3. In order to obtain an adherent sensor film, a high energy vacuum sputtering process is employed that mechanically embeds the sensor metal molecules in a slightly roughened oxide surface. The sensor is ductile compared with the Al_2O_3, and the sensor temperature coefficient of expansion matches that of Al_2O_3 reasonably well. Therefore, the sensor is expected to withstand temperature cycling stresses with only a gradual long-term shift in electrical resistance or change in thermocouple electromotive force.

The bond at "A" in Figure 1, between the MCrAlY coating and the turbine blade material, is partially chemical in nature and is enhanced by heat treatment to promote diffusion and solid solution in the interface region.

No overcoating of the thin film thermocouples was planned because the platinum and platinum-10 percent rhodium films were thought to be resistant to corrosion and erosion, based on results obtained during previous engine experiments (Table I, columns 5 and 6). However, these results did not include calibration accuracy comparisons.

3.2 THIN-FILM-TO-LEAD-WIRE CONNECTION

In the engine test of sample sputtered films, lead-wire connections had been fabricated by oversputtering the sensor lead films onto lead wires embedded in insulated platform holes and reinforcing by application of fired platinum paste (Engelhart Industries No. 6869). These lead-wire connections failed early. Subsequently, during the Phase I development program (Reference 6), a number of alternative techniques for lead-wire attachment were investigated, and a successful hot compression bonding technique was developed. This technique was then used throughout the Phase I and Phase II tests of sample thermocouple systems (furnace cycling, vibration tests, and combustor exhaust gas-stream tests) with excellent results. This bonding technique is described in Reference 6.

SECTION 4.0

SENSOR SYSTEM REQUIREMENTS

During the design of the thin film temperature sensor systems to meet the overall goals of the program, five major specifications were given special consideration. These specifications are defined in the contract and include size, accuracy, reliability, environment, and blade material. This section describes specifications of the contract.

4.1 PHYSICAL SIZE

The maximum permissible value of four physical dimensions of the thin film sensor system are specified in the contract. These are: sensor measurement-zone (hot junction) size; film width from the measurement zone to the film-to-lead-wire connection; thickness of the overall thin film portion of the sensor assembly, including insulator; and lead wire diameter. The following tabulation compares these maximum allowable dimensions with those actually selected for the sensor system.

Dimensions	Maximum Permissible Value in Contract Specification (mils)	Maximum Permissible Value in Contract Specification (mm)	Actual Value (mm)
Measurement-Zone Size	50	1.27	1.27
Film Width	50	1.27	1.09
Film Thickness	0.6	0.015	0.004
Lead Wire Sheath Diameter	32	0.813	0.813

The specification also required that the lead-wire cable assembly be capable of withstanding a temperature of 1140K (1600°F). The lead wire cable used in these experiments has a temperature capability of 1800K (2800°F).

4.2 ACCURACY

The following accuracy specification goals are defined in the contract (the percentages are based on temperature expressed in degrees Farhenheit):

1. Accuracy of reference wire thermocouples, $\pm$0.75 percent.

2. Reference wire thermocouple maximum drift, 1 percent during the full testing program.

3. Agreement of thin film thermocouples with reference wire thermocouples, as fabricated at start of testing program $\pm$1.5 percent.

4. Thin film thermocouple maximum calibration shift goal, 5 percent during the full testing program.

The reference thermocouples used in the experiments exceeded the accuracy specifications of items 1 and 2. The achieved accuracy of thin film thermocouples (items 3 and 4) will be discussed in Section 6.0, Results.

4.3 RELIABILITY

The contract specifies the failure rate goal for the thin film thermocouple system, due to any failure which renders the system inoperative, as 25 percent or less during the full testing program. The achieved reliability will be discussed in Section 6.0, Results.

4.4 ENVIRONMENT

The contract established six environmental conditions for evaluation of the thin film thermocouple systems:

1. Maximum operating temperature, 1250K (1800°F);

2. Test temperature profile as defined by Figure 2;

3. Fuel source for the exhaust gas flame, hydrocarbon fuel;

4. Twenty complete thermal cycles from room temperature to the 1140 to 1250K (1600 to 1800°F) temperature range;

5. Exhaust gas conditions of nominally 1 atmosphere pressure and a Mach number range of 0.5 to 0.7.

6. Peak vibrational accelerations of up to 8 g's in any or all axes over a frequency range of 100 to 400 Hertz.

The following tabulation compares these contract requirements with the actual conditions during the test program.

	Contract Specification	Actual Test Condition
Maximum Operating Temperature	1250K (1800°F)	1250K (1800°F)
Temperature Profile	See Figure 2	See Figure 2
Gas Flame Source	Hydrocarbon Fuel	Hydrocarbon Fuel
Thermal Cycling:		
Room Temp. to Max. Temp.	1140 to 1250K (1600 to 1800°F)	1250K (1800°F)
Number of Cycles	20	71

	Contract Specification	Achieved Condition
Exhaust Gas Conditions:		
Nominal Pressure	1 atmosphere	1 atmosphere
Mach Number	0.5 to 0.7	0.5
Vibration Testing:		
Level/Frequency	8 g's/100 to 400 Hertz	8 g's/100 to 400 Hertz
Axes	Any or All; 1 hour on each axis	Each of Three Axes, Mutually perpendicular; 1 hour on each axis

4.5 BLADE MATERIAL

The contract defines the preferred blade material as PWA 1422, a modified MAR-M-200 nickel base alloy. The blade material selected for use was MAR-M-200 + hafnium.

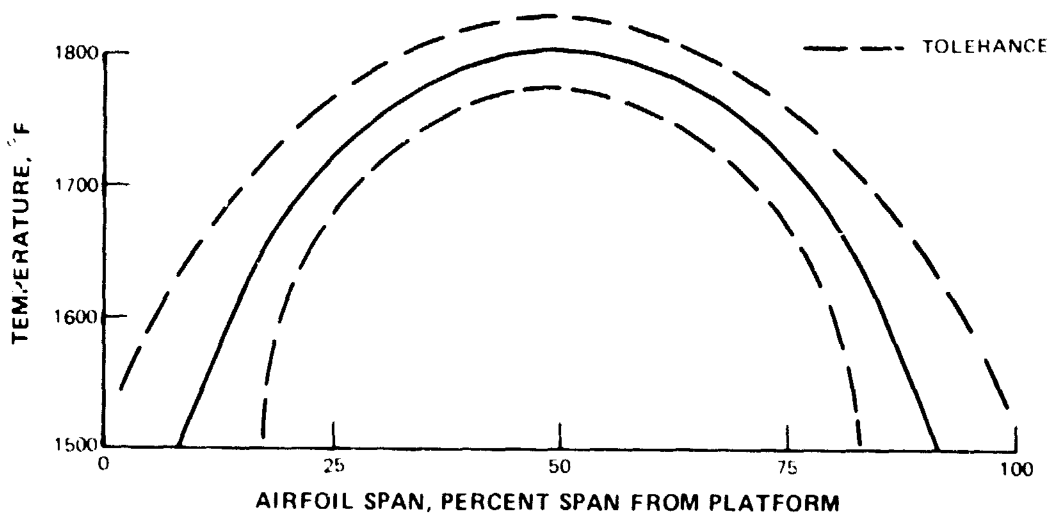


Figure 2 Contractually Specified Surface Temperature Profile - Blade surface temperatures during the test program were within the tolerance band.

SECTION 5.0

FABRICATION AND TEST PROCEDURES

Two sets of turbine blades were instrumented with thin film thermocouples for two different purposes. One set was prepared for a "Performance Evaluation Program" in which the performance of the thin film thermocouples was evaluated in simulated engine conditions. The other set was used in an "Adherence Evaluation Program" to evaluate factors affecting adherence of the thin film thermocouples to the insulating layer. The fabrication and test procedures for these two programs are described below.

5.1 PERFORMANCE EVALUATION PROGRAM

5.1.1 Description of Test Pieces

Six thin film thermocouple sensor systems and four reference wire thermocouples were fabricated on each of three standard production turbine blades (designated Blades No. 1, No. 2, and No. 3) as shown in Figure 3. The blade material was MAR-M-200 plus hafnium. Three thin film thermocouples and one reference wire thermocouple were installed on each side of the airfoil. The airfoil reference thermocouples were installed as shown (Thermocouple A), centered within 5 mm of the three thin film thermocouple junctions. The other reference wire thermocouples were installed on the blade platform top surface near the thin-film-to-lead-wire connections (Thermocouple B).

Lead wire pigtails 2-cm long of 0.075-mm O.D. platinum or platinum-10 percent rhodium wire were hot compression bonded to the thin films on the top of each platform. (In an actual engine installation, the film would extend around the platform edge to the underside of the platform, and connections would be made in that region to ensure centripetal loading in the safe direction.) One end of a 60-cm length of 0.81-mm O.D. platinum-10 percent rhodium sheathed dual-thermocouple extension wire was strap welded to the root fir tree. Each extension wire contained two 0.127-mm O.D. conductors (one platinum and one platinum-10 percent rhodium) in magnesium oxide ceramic insulation. These two conductors were tweezer welded to the 0.075-mm O.D. pigtails of the thin film sensor system. The exposed lead wires were then embedded in ceramic cement (Cerama-dip No. 538). A thermocouple connector was used at the other end of the 60-cm long sheathed cable to connect long Type S extension wires to a temperature recorder.

The reference wire thermocouples were grounded Type S wire (formed from additional 60-cm lengths of the 0.81-mm O.D. high-temperature sheathed cable) spot welded at locations "A" and "B" shown in Figure 3 to provide reference measurements of surface temperature. The thermocouple wire used was Type S reference grade ($\pm$ 2.5K accuracy at 1300K).

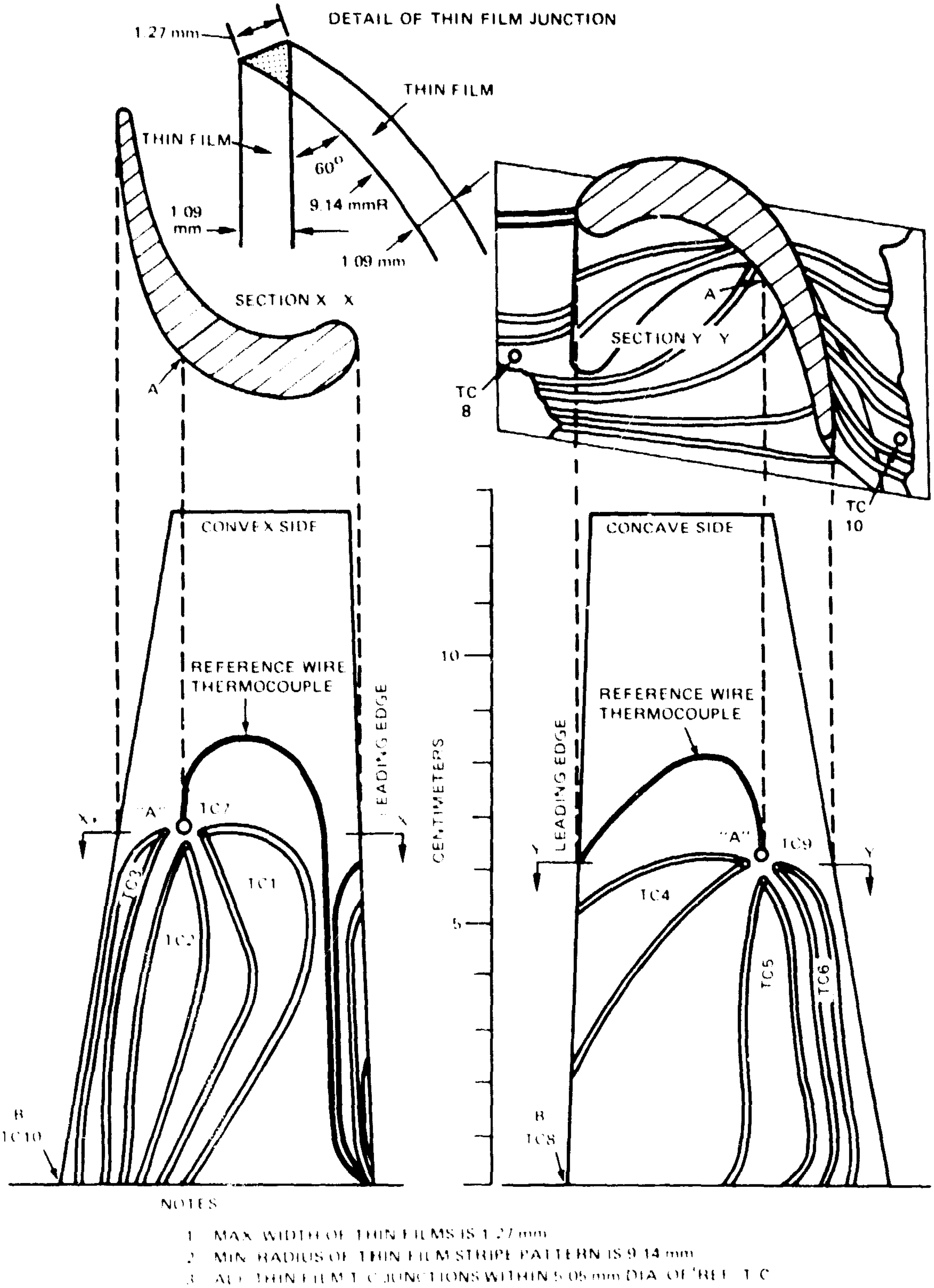


Figure 3 Thin Film Temperature Sensor Pattern on Turbine Blade No. 1 and Detail of Thin Film Junction.

5.1.2 Fabrication Procedures

The basic fabrication procedure is outlined in Table II. A narrative description of each step is provided following the table for a clear understanding of the principal requirements in fabricating thin film thermocouple systems on coated turbine blades. The procedure is similar to that described previously in Reference 6, with the additional specification that laboratory dew point be maintained below 282K (48^{o}F) during Steps 9 through 15, and a requirement for scanning electron microscope examination of a sample of each batch of blades (Step 11).

TABLE II

OUTLINE OF FABRICATION PROCEDURE FOR
THIN FILM THERMOCOUPLE ELEMENTS

1	Polish and clean the turbine blade surface
2	Vapor hone
3	Electron beam vapor deposition coat with MCrAlY
4	Dry glass bead peen the coating
5	Polish the coating
6	Grit blast the coating
7	Heat treat at 1350K for 4 hours in dry hydrogen
8	Oxidize in air at 1300K for a maximum of 50 hours
9	Sputter Al_2O_3
10	Age at 1250K for 1 hour
11	Examine surface, using scanning electron microscope. Accept or Reject
12	Mask for platinum
13	Sputter platinum
14	Mask for platinum-10 percent rhodium
15	Sputter platinum-10 percent rhodium
16	Attach leads by hot compression bonding
17	Age at 1250K to stabilize films and eliminate short circuits

1. After casting and machining, the turbine blades were mechanically polished on all surfaces to be deposited with thin films to a surface finish of 0.25 $\pm$0.1 micrometers (μm) measured on a Talysurf profilometer. All test pieces were then degreased in methylene chloride, cleaned in an ultransonic Alconox detergent bath, rinsed in boiling deionized water until no detergent residue remained, rinsed in hot isopropyl alcohol, soaked in a hot ultrasonic Freon 12 bath, and given a final hot freon vapor rinse. After cleaning, the test pieces were handled with cotton or nylon gloves to prevent oil or grease contamination, and were stored in a dessicant chamber here and between all subsequent operations.

2. Each piece was vapor-honed (grit-blasted with a water spray) just before coating.

3. A 120 $\mu m \pm 25 \mu m$ NiCoCrAlY coating was then applied by the electron beam vapor deposition process by evaporation from a molten pool of the coating material in a vacuum chamber. per PWA 270 specifications. A three-orientation process was employed to coat the desired areas.

4. The finished coating was dry glass bead-peened to increase its density.

5. The coating surface was repolished mechanically to a 0.25 $\pm$0.1 μm finish.

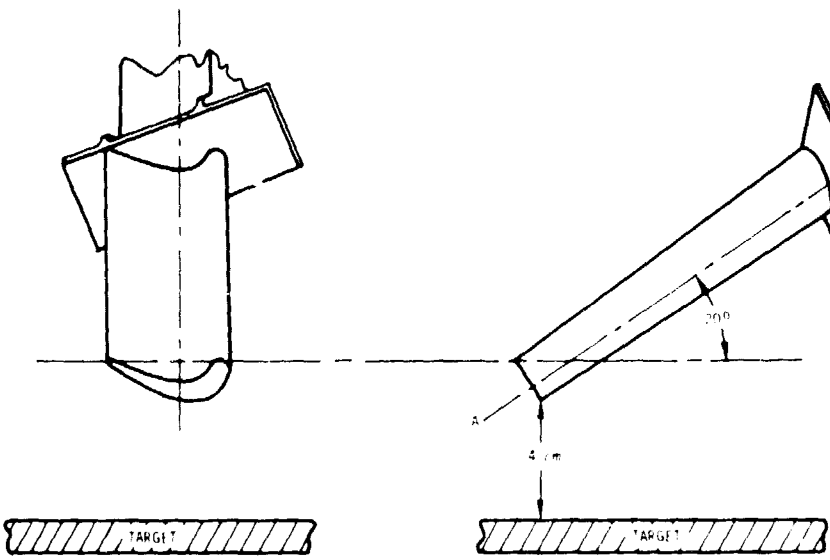
6. The surface was then lightly grit blasted (320 grit Al_2O_3 at 150 to 300 kPa pressure at about 45-degree incidence angle and 8-cm distance) to produce a controlled surface roughness for adherence of platinum films. (The grit blasting does not change the measured arithmetic average roughness, but alters the texture from a mirror like surface to a dull gray.) The entire cleaning procedure was repeated after polishing and again after grit blasting.

7. The turbine blades were then heat treated at 1350K (1975^{o}F) in dry hydrogen at a pressure of 100 kPa for four hours. The environment during this hydrogen heat treatment is extremely critical to the ultimate success of the thin film sensor system. The purpose is to stabilize the coating, to start the migration of aluminum toward the surface, and to start the gradual growth of a dense, hard, and adherent layer of Al_2O_3 in an oxygen-poor environment. The partial pressure of oxygen should be less than 10^{-5} Pascal (Pa). Excessive oxygen results in the appearance of poorly adherent blue oxides of chromium and cobalt, as well as spinels (Cr-Al-O compounds). It is believed that an argon atmosphere or a vacuum would produce as good a result as hydrogen, but these alternatives were not explored in this program. Contamination by more than 0.1 ppm of gaseous water, chlorine, sodium, or hydroxyl radical during the heat treatment can result in a poor Al_2O_3 structure such as the stringy reticulated whiskers of low adhesion described in Reference 7.

8. All turbine blades were cleaned in a Freon 12 rinse and oxidized at 1300K for 50 hours in air in a clean oxidation furnace. The furnace used was a Marshall oven with high-purity aluminum oxide muffle, dedicated to this oxidation process and never used for any other purpose.

9. From this point on during the fabrication procedure, care was taken to expose the blades to an atmosphere with a dew point no higher than 282K (48^{o}F) to avoid the formation of absorbed layers of water molecules which were found to be detrimental to platinum film adherence. At higher humidities, surface condensation of moisture can occur readily during cleaning procedures employing volatile solvents such as Freon, for example. The turbine blades were cleaned in a Freon 12 rinse and sputtered with 1.0 to 1.5 μm of

Al_2O_3. A Materials Research Corporation 15-cm diameter MARZ grade Al_2O_3 planar target (99.992 percent purity) was used, and the sputtering was done in a an r.f. diode sputtering system at 1100 VDC (self bias) for about 9 hours in an argon-5 percent oxygen gas mixture at 5×10^{-1} Pa. The location and orientation of the blade relative to the Al_2O_3 source target are shown in Figure 4. The blade was held stationary during sputtering. Therefore, two sputtering runs were required to coat all surfaces. The concave surface was always sputtered first. It was found that if the insulation resistance was over 100 ohms at this point, then the subsequent heat treatments associated with aging the sputtered Al_2O_3, lead wire bonding, and aging the platinum and platinum-10 percent rhodium films brought the resistance above 20 megohms due to the growth of additional Al_2O_3 at interface B of Figure 1. In this way, during the fabrication procedure the total exposure time of the turbine blades in air to temperatures in the 1100 to 1300K range was ordinarily below 75 hours.



NOTES: 1) BLADE IS STATIONARY DURING SPUTTERING.
2) ORIENTATION SHOWN IS FOR SPUTTERING OF CONVEX SIDE. FOR CONCAVE SIDE, BLADE WAS REPOSITIONED BY 180° ROTATION AROUND AXIS A-A.

Figure 4 Orientation of Turbine Blade During Sputtering

10. After sputtering, each turbine blade was aged at 1250K for one hour to ensure the formation of α-Al_2O_3 structure in the sputtered Al_2O_3.

11. The Al_2O_3 surface was then examined with a scanning electron microscope to ensure that a surface structure reasonably conducive to adherence of platinum had been formed.

12. The turbine blades were then masked for platinum film, using the tape plus positive photoresist method. Strips of 3M No. 218 tape were applied directly to the surface to cover areas which would become platinum films. The entire surface was then painted with liquid photoresist (Shipley No. AZ-1350-J), a material which hardens to a protective layer when baked at 470K, but which can be easily removed later with a solvent. After baking, the tape was removed to expose the film pattern. The surface was then ultrasonically cleaned in a Freon 12 bath at room temperature to remove traces of tape residue. The Freon 12 ultrasonic bath temperature was raised to 320K, and the cleaning was continued (for several hours in some cases) until a water break test indicated no residue. In the water break test, deionized water is placed on the surface. If it flows into a film over the entire surface, with no breaks, the cleanliness is acceptable. This cleaning procedure was found to be critical to success, since any tape residue severely degraded the adherence of the platinum films.

13. Platinum was then r.f. diode sputtered to a thickness of 2.0 μm using a 15-cm diameter, 99.98 percent platinum plane target in a pure argon gas at 5 x 10^{-1} Pa at 800 VDC for 4.5 hours. Blade orientation (Figure 4) and sequence (concave side first) were the same as for Al_2O_3 sputtering. After sputtering, the photoresist was removed by washing with Shipley AZ-1350 Remover solvent. Any remaining traces of photoresist were removed by charring in an oven at 700 to 800K.

14. and
15. The turbine blades were remasked and sputtered with platinum-10 percent rhodium films to the same thickness range as the platinum films, using thermocouple quality target material. All sputtering parameters were the same as those used for platinum. After photoresist removal, all films were then tape tested for adherence, using Dodge Industries No. 2045-5 tape pressed firmly in place over test patterns and then removed by pulling vertically. If more than 1 percent of the film area was removed, the film adherence was judged not acceptable.

16. The hot compression bonding procedure of Reference 6 was used for fabricating lead wire attachments.

17. After fabrication and lead wire attachment, the assembly was aged in air for one hour at 1250K to stabilize the platinum and platinum-10 percent rhodium films. If resistance to ground was found to be less than 20 megohms at this stage, the assembly was aged in air for additional periods of 25 hours at 1250K to grow additional Al_2O_3 under the lead wire junction. If the resistance could not be increased to 20 megohms after a total of 150 hours in air at 1250K, the assembly was rejected.

5.1.3 Oven Calibration Tests

Initial calibration of thin film sensor systems on blades was conducted in a laboratory oven with a chamber size of 10 cm x 10 cm x 23 cm. The opening to the oven was closed except for a slot large enough to allow the airfoil of the blade to be inserted with the blade chord in a horizontal position. The root of the blade was cooled by blowing air over the root fir tree. The thermocouples were connected to a multichannel thermocouple chart recorder containing reference cold junctions and calibrated for ANSI Type S thermocouples from 811K (1000°F) to 1366K (2000°F) with an accuracy of ±10K. In addition, a Lewis thermocouple switch permitted manual selection of any one thermocouple reading on a precision digital temperature indicator with traceable accuracy of ±5K with ANSI type S thermocouples.

The usual oven calibration test procedure consisted of a 5-hour cycle to 1250K conducted as follows:

- 2-hour warm up to 1250K (as indicated by the highest-reading reference wire thermocouple on the blade) with cooling air blowing over the root;
- 2-hour soak at 1250K with cooling air blowing over the root;
- 1-hour cool down to room temperature with cooling air blowing over the root.

The object of these tests was to compare the output of the thin film thermocouple to that of its adjacent reference wire thermocouple. Note that a temperature gradient was imposed on the blade. This gradient was necessary to evaluate the thermal electromotive force generated by the thin films. In the absence of a temperature gradient, no thermal electromotive force would be generated by either the thin film or reference thermocouple systems on the blades, and the electromotive force recorded would be that generated in the extension leads from the platform to the recording system cold junction. The recorded information in the absence of a temperature gradient between the platform and airfoil thermocouple locations would provide no information on thin film thermocouple calibration.

Any anomalies in the thin film thermocouple, such as variations in film composition along its length or leakage to ground through the Al_2O_3 insulating layer, would, in the presence of a temperature gradient, result in a temperature indication different from that of the actual junction temperature by an amount dependent upon the temperature gradient.

These oven calibration tests were also performed at any other time in the testing program that a calibration verification was required.

Additional oven tests wre performed on Blade No. 2 to study long-term calibration drift. The same equipment and instrumentation was used, but the time at 1250K was extended to 50 hours.

5.1.4 Combustor Tests

Two of the fully instrumented turbine blade specimens (Blades 1 and 3) were subjected to an exhaust gas environment test by exposing each specimen in a combustor rig flow, as shown in Figure 5. The exhaust gas was directed toward the blade midspan leading edge, simulating a typical engine zero incidence angle. The exhaust gas Mach number was approximately 0.5, as determined from measurements of air supply total volume flow and combustor exit nozzle flow area. Figure 6 shows three photographs of Blade No. 1 during the combustor test.

The following test sequence was conducted for Blades 1 and 3:

- o Fifty hours at nominal temperature profile (see Figure 2), including 50 cycles from ambient to maximum to ambient temperature. Each cycle consisted of 1 hour at 1250K as indicated by the highest reference thermocouple reading on the blade airfoil surface. Test sensors and reference wire thermocouples were monitored.
- o Twenty cycles from ambient to nominal temperature profile to ambient temperature. Time at maximum temperature was 20 minutes in each cycle. Test sensor and reference thermocouples were monitored.
- o Three hours at nominal temperature profile. Test sensor and reference thermocouples were monitored.

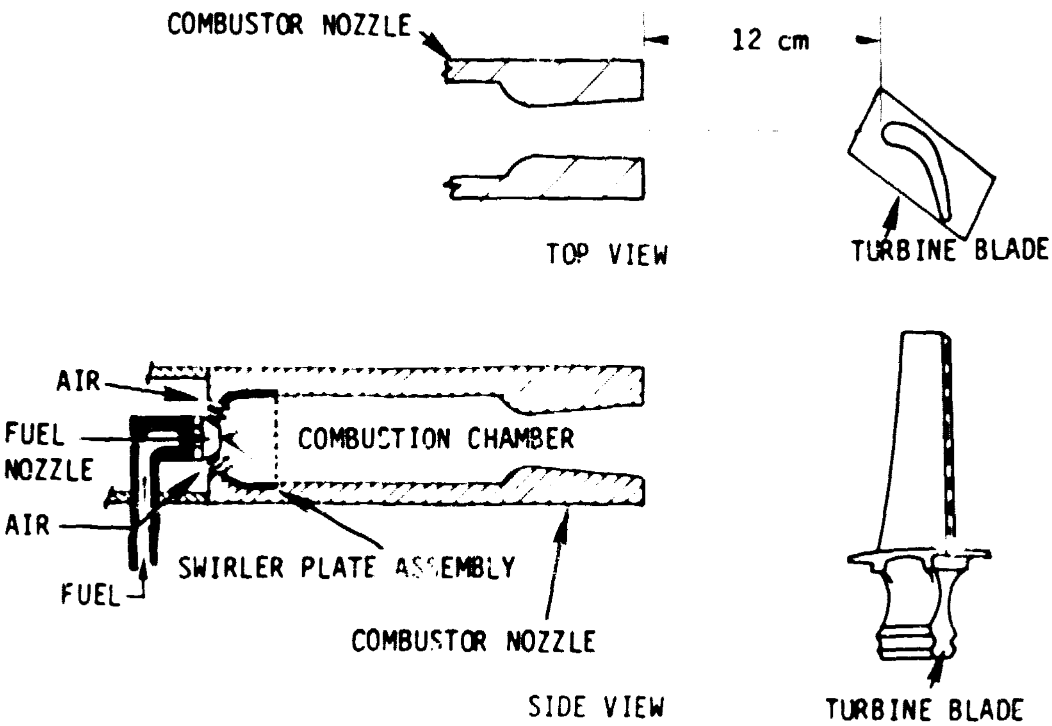


Figure 5 Simulated Combustor Exhaust Gas-Stream Test Arrangement

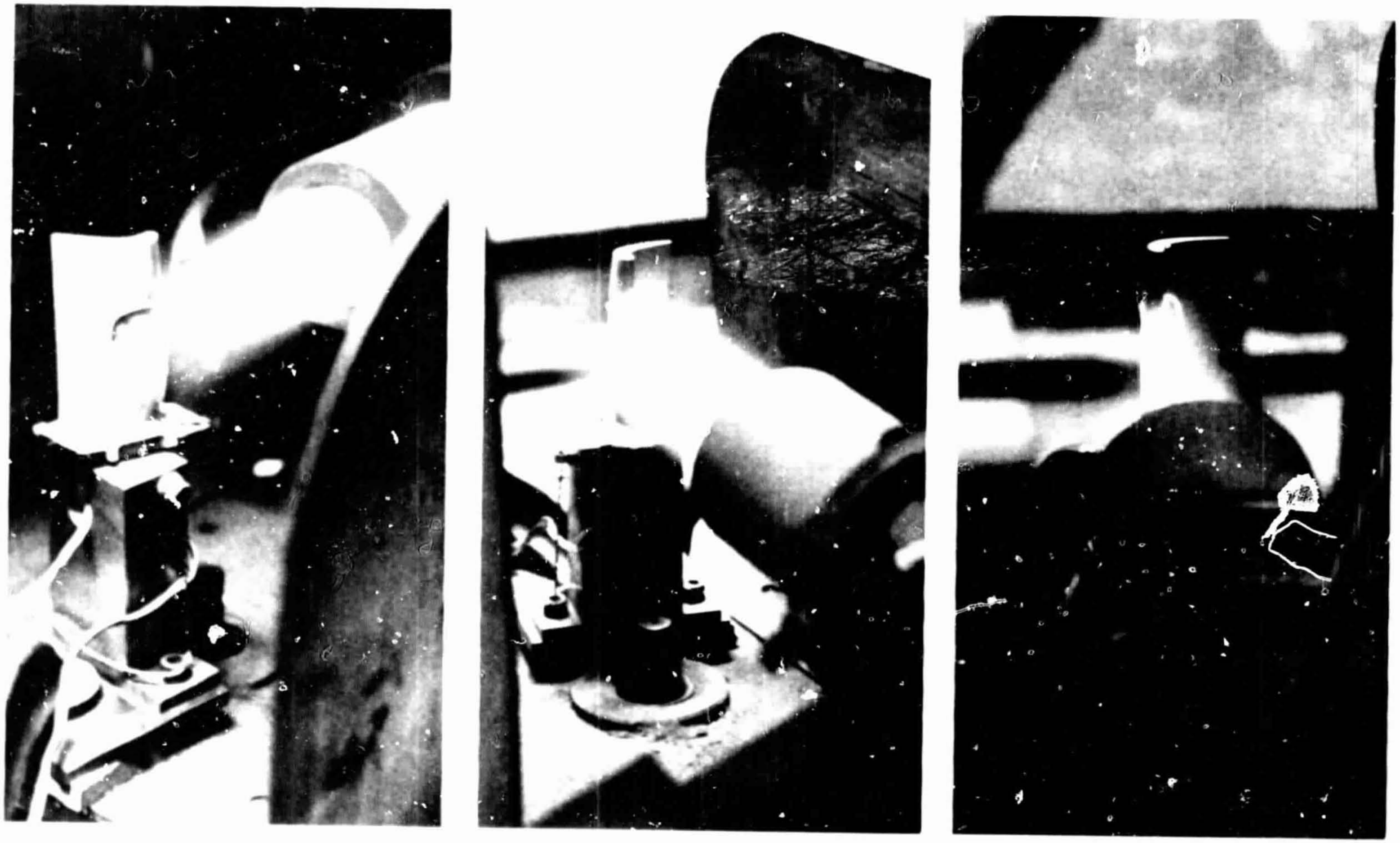


Figure 6 Three Views of Blade 1 During Combustor Exhaust Gas-Stream Testing - Luminous particulates in the flame are most clearly visible in the view at the left. (80-441-0517-A, -B, and -C)

Thus, the total time at maximum temperature in hot gas flow was at least 59.7 hours for each specimen tested.

The instrumentation for these tests was the same as that used in the oven calibrations. A continuous chart recording of all operational test sensor and reference thermocouples was obtained. In addition, readings were taken at selected times using the precision digital temperature indicator.

The temperature error of the reference wire thermocouple on the airfoil during the combustor tests due to aerodynamic effects not present during oven tests was estimated to be less than 5K. The possible error arises from conduction of heat into the grounded thermocouple junction from the individual thermocouple lead wires. These lead wires are on or slightly above the blade surface in the hot flow and therefore at a temperature above the local blade surface temperature. (Note that the glowing sheath of the thermocouple extension wire is easily visible in Figure 6). The 5K estimate is based on the observation that the area for heat conduction into the junction (lead wire cross section area) is about 100 times smaller than the area for heat conduction out of the flattened grounded junction and into the blade surface. Since the path lengths and thermal conductivities are about equal, then the temperature difference between blade and junction is expected to be 100 times smaller than the temperature difference between the junction and the lead wire. Even if the latter is on the order of 500K, the former is only on the order of 5K. It should further be noted that no cooling air was supplied to the internal cooling passages in the blade. Therefore local temperature gradients normal to the blade surface within the blade were small.

5.1.5 Vibration Tests

Vibration testing of two turbine blade specimens (Blades No. 1 and No. 3), completely instrumented with thin film thermocouples, reference wire thermocouples, and extension leads, was performed using a Ling model A300-B electrodynamic vibration exciter having a force rating of 15,500 newtons (3500 pounds force) and a frequency range of 5 to 3000 Hertz.

Each blade was attached to the exciter table using F-88 dental cement. Testing was performed with the blade oriented in each of three mutually perpendicular axes. In each axis, the blade was subjected to a peak vibration level of 8 g's for 1 hour at a frequency sweep range of 100 to 400 Hertz and a rate of one octave per minute.

Vibration testing of Blade 3 was performed prior to thermal testing. Vibration testing of Blade 1 was performed between the last two items of the combustor test sequence.

Electrical continuity and insulation resistance of the sensors were measured before and after vibration testing.

5.2 ADHERENCE EVALUATION PROGRAM

The physics and chemistry of surface adherence were discussed in the Phase I report (Reference 6). Seven factors were identified that could affect platinum film adherence on turbine blades. These factors are:

1. Alumina-forming NiCoCrAlY coating
2. Humidity effects
3. Masking techniques
4. Sputtering parameters
5. Reactive sputtering
6. Precoats
7. Field-assisted bonding

Fabrication methods and test procedures were developed to evaluate these seven factors.

5.2.1 Description of Test Pieces

Four turbine blades were chosen for instrumentation and testing to investigate adherence effects. Two blades (A and B) were coated with NiCoCrAlY by the Chromalloy Corporation, and the other blades (C and D) were coated by Pratt & Whitney Aircraft.

The pattern of thin film thermocouples shown in Figure 7 was sputtered on one side of each blade. This pattern consists of a common platinum-10 percent rhodium film element intersecting four platinum film elements to form four thermocouple junctions. The four platinum elements were sputtered using the following four different sputtering techniques thought to affect adherence:

- o Film No. 5 - Standard platinum, that is, platinum sputtered in argon;
- o Film No. 3 - Standard platinum plus sputtered chromium precoat, 500 Angstroms thick;
- o Film No. 2 - Standard platinum plus sputtered platinum-10 percent rhodium precoat, 500 Angstroms thick; and
- o Film No. 4 - Standard platinum reactively sputtered (50 percent oxygen plus 50 percent argon for 5 minutes, followed by standard sputtering in argon).
- o In addition, an extra platinum film (No. 6) was sputtered for field-assist tests.

Two extra test patches of each material were sputtered on each blade to permit tape testing and pull testing to destruction for adherence evaluation before and after flame testing. The location of reference wire thermocouples is also shown in Figure 7.

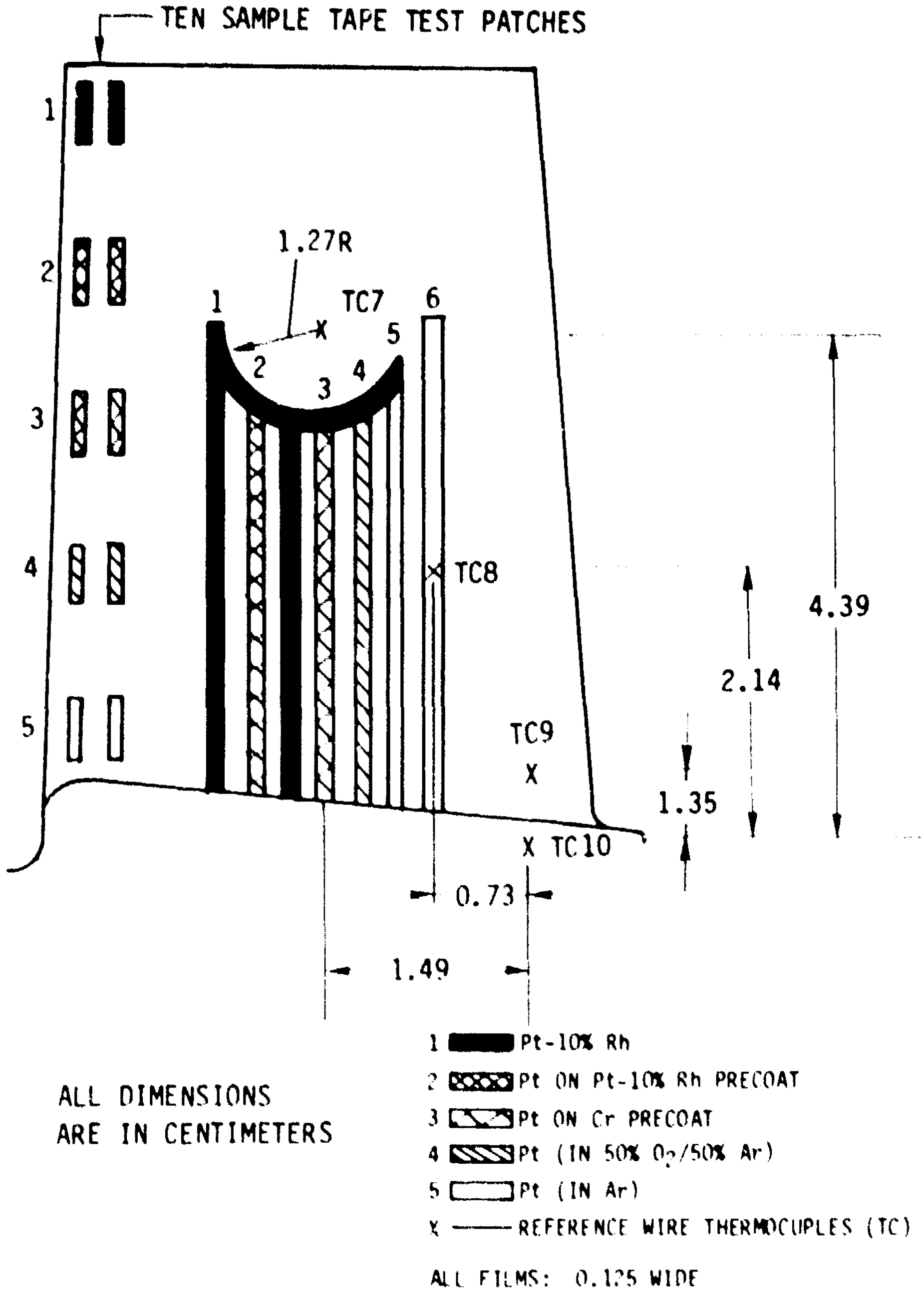


Figure 7 Thin Film Temperature Sensor Patterns for Adherence Tests on Blades A, B, C, and D.

5.2.2 Modification of Fabrication Procedures

The fabrication procedures outlined in Section 5.1.2 were modified in the following manner:

- Steps 1 through 11 - No change in the fabrication procedure.

- Steps 12 through 15 - Two types of masking procedures were used. One of these was the normal method of masking described in Section 5.1.2. In this case steps 12 through 15 would be unchanged, resulting in the normal sputtering of the standard platinum and platinum-10 percent rhodium legs. Then additional steps are added after step 15 to mask and sputter for the remainder of the legs.

- The other masking method is called crossed-tape masking and was tried in an attempt to simplify the masking steps. In this method, the entire pattern of Figure 7 was masked according to the normal procedure. Then, as each leg of the pattern was sputtered, special shadow masks were used to cover all parts of the pattern except the one being sputtered. These shadow masks were formed by tape doubled in such a way that no adherent sides were exposed or touched the surfaces to be sputtered. This is done to prevent contamination of clean surfaces. Thus, between sputtering runs, only shadow masks were moved to expose the next leg of the pattern. Crossed-tape masking was used in trials on three of the blades (A, C. and D), and normal tape masking was used on final fabrication of two blades (A and C).

- Steps 16 and 17 - These steps were performed in the manner described in Section 5.1.2.

An additional step was performed for the field-assist bonding described in Reference 7 for enhancement of adherence. With the blade at 1140K, a positive direct-current voltage was applied to the No. 6 film, and the voltage was increased until a current flow of 10 microamperes was observed. After 10 minutes, the voltage was removed.

5.2.3 Thermal Tests

Two of the four turbine blades (A and B) were subjected to an exhaust gas environment test by exposing each specimen to an oxyacetylene torch flame, as shown in Figure 8. The torch was directed toward the convex (suction) surface of the blade (opposite the test sensors) to avoid exposure of the thin film sensors and reference wire thermocouples to flame radiation temperature. The duration of each flame test was 50 hours at 1250K, as determined by the reading of reference wire thermocouple No. 7, and included at least 10 cycles to room temperature.

During flame tests of Blade A, temperature indications from three of the thin film thermocouples were monitored continuously. These three thermocouples were:

- platinum-10 percent rhodium versus platinum;

- platinum-10 percent rhodium versus platinum + chromium; and

- platinum-10 percent rhodium versus platinum + oxygen.

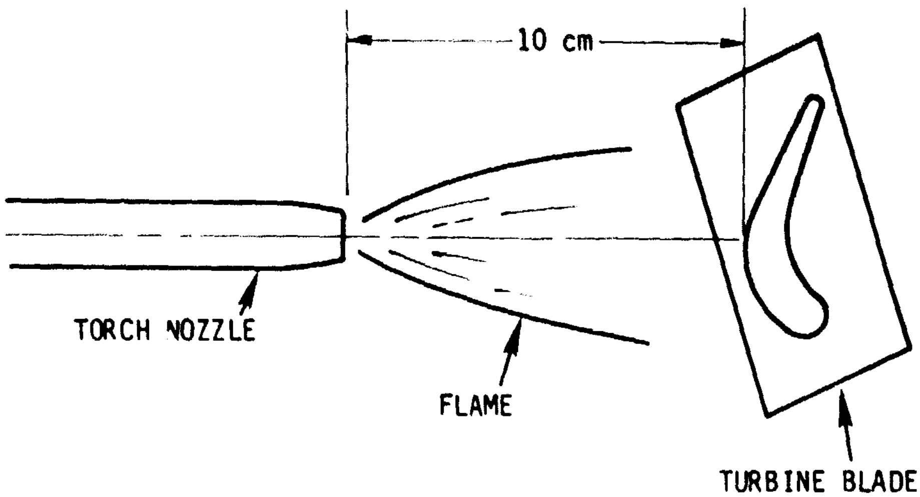


Figure 8 Oxyacetylene Torch Test Arrangement

The four Type S reference wire thermocouples and a scanning optical pyrometer measured the blade temperature distribution. A temperature gradient of about 400K (700°F) was maintained between the thin film thermocouple junctions on the airfoil and the lead-wire-to-film connections on the platform.

One or more oven calibration tests to 1250K were conducted before and after flame tests, as described in Section 5.1.3.

5.2.4 Adherence Tests

Tape adherence tests were conducted on the films and sample patches of each material before and after flame tests. The percentage of film removed by the tape was measured.

A wire-pull tensile test was conducted to evaluate adherence. A 3-mil diameter wire was epoxy cemented to each sample patch of sputtered material on the blade, and to the field-assisted platinum film near midspan on the airfoil, in an area of about 1.25-mm diameter. The tensile force was applied normal to the surface, and the amount of force required to pull the film from the surface, or break the wire, was measured.

Sections of Blade A and Blade C were sent to the United Technologies Research Center for examination by a scanning electron microscope (SEM) to determine whether the alumina surface morphology offered any clue to an explanation of platinum film adherence differences.

SECTION 6.0

RESULTS

In Section 5.0, the Performance Evaluation Program was described first, followed by the description of the Adherence Evaluation Program. This was done to present a general description of the fabrication procedure first, followed by variations of that procedure.

In this section, the Adherence Evaluation Program results will be presented first because they were performed first.

6.1 ADHERENCE EVALUATION PROGRAM

The results of tape testing on the films and patchs of the four turbine blades designated Blades A, B, C, and D (Figure 7) are shown in Table III. In the body of the table, the number represents the percentage of film remaining after the tape test. Information is presented on both films and patches and on both crossed-tape and normal masking. The data are summed up in a final Adherence Rating for each coating on each blade. (It should be noted that this Adherence Rating does not include data from the junction area. This will be discussed under Section 6.1.3, Masking Techniques.) The effects on adherence for each of seven selected factors are reviewed below.

6.1.1 Coating Structure

The most significant adherence factor was the source of the alumina-forming NiCoCrAlY coating. On two blades (A and B) the coating source was an outside vendor. On the other two blades (C and D), the coating was accomplished in-house. Both coatings (vendor and in-house) regularly meet the primary requirement of the PWA 270 coating which is the usual turbine blade corrosion protection used for in-service engines.

The Adherence Ratings in Table III show that all films sputtered on vendor-coated blades (A and B) using normal masking procedures survived the entire adherence test program, including tape tests, photoresist removal, oven cycling, 50-hour flame tests at 1250K surface temperature, and subsequent wire-pull tensile tests. All films sputtered on in-house coated blades (C and D) showed poor adhesion initially and did not survive tape tests or photoresist removal.

In one case, platinum films were sputtered simultaneously on one blade of each type (Blade A and Blade C). Even in this case, adherence was excellent on the vendor-coated Blade A and poor on the in-house coated Blade C.

TABLE III

SUMMARY OF ADHERENCE DATA

Percent of Film Remaining After Test

Coating Source	Vendor										In-House							Adherence Rating[4] On Blades A & B	Adherence Rating[4] On Blades C & D
Blade Designation	A						B				C				D				
Tape Mask Procedure[1]	I			II[5]			I				I		II[5]		I				
Adherence Test[2]	S	P_o	J	S	P_o	J	S	P_o	P_f	J	S	P_o	S	P_o	S	P_o	J		
Pt-10% Rh	100	100	0	99	100	100	100	60	100	50			30	0	40	30	50	100	25
Pt	100	100	0	100	100	100	100	90	99	50	60[3]	0	30	0	40	30	50	100	25
Pt + Pt-10% Rh	100	100	0	100	100	100	100	20	100	0			30	0	40	30	50	100	25
Pt + O_2	100	100	0	100	95	100	100	20	100	50	60[3]	0	30	0	100	30	50	99	40
Pt + Cr	100	100	0	100	100	100	100	90	95	50			30	0	100	30	50	99	40
Pt (for field assist)	100			100			100						30						

NOTES:

1. Procedure I: Crossed tape shadow mask at junctions.
   Procedure II: Photoresist remask at junctions.

2. S = Stripe area remaining after photoresist removal. Local losses near junction not counted in this rating.

   P_o = Patch tape test, as sputtered (first set of patches).

   P_f = Patch tape test, after 50-hour flame test (second set of patches).

   J = Junction area remaining after photoresist removal.

3. Fabrication was stopped after sputtering of Pt and Pt + O_2 because contamination in junction area was noticed. Photoresist was then removed and patches tape tested.

4. Rating = Average of S and P_f readings. (where P_f not yet available, P_o used).

5. A,II and C,II were oxidized simultaneously and then sputtered simultaneously.

The results of the wire-pull tensile tests of adherence on Blades A and B (after 50-hour flame tests), utilizing steel wires epoxy-cemented to the films, showed that film bond strength exceeded the 180 grams force (1.8 Newtons) breaking strength of the wire in every case. The tests were repeated on at least two different areas of every film.

The results of scanning electron microscope (SEM) examination of the Al_2O_3 surface structure on Blades A and C are shown in Figures 9 and 10, respectively. Blade A displays the expected dense fine-grained Al_2O_3 structure with some flat scales and surface re-entry features, believed to be conducive to good mechanical adherence of metal films, while Blade C displays a coarse patchy structure. The coarse structure was fragile and easily removed, and apparently explains the poor adherence of metal films on Blade C. The coarse structure is extremely unusual. Energy dispersive spectroscopy (EDS) showed no striking difference in atomic species present on the surfaces of the two blades except the presence of a significant amount of iron on Blade C, thought to be a trace of residual Fe_2O_3 polishing rouge. Salt or water vapor contamination during Al_2O_3 growth, which can produce a whiskery, poorly adherent oxide (Reference 7), is believed to be ruled out since no whiskers or chlorine were detected.

Further SEM examination of the Blade C oxide structure (Figure 11) reveals a corncob appearance with a demarcation plane within the corncob structure at the 6000-Angstrom depth. The corncob extends below this level to a depth of 11,000 Angstroms. Below this is a close-grained Al_2O_3 surface. It appears that the initial growth of Al_2O_3 was normal during heat treatment in hydrogen, and perhaps during part of the furnace oxidation in air. By the end of the oxidation process, the coarse nodular corncob structure had developed and was preserved during subsequent sputtering of Al_2O_3 and heat treatment in air. The literature reveals that various nodular, cellular, or columnar Al_2O_3 structures can be produced by growth on aluminum surfaces containing asperities or on aluminum surfaces pretreated with phosphoric or chromic acid in preparation for epoxy adhesive bonding of aluminum panels (Reference 9). The corncob structure is reminiscent of these structures but not exactly like any previously reported structure. Surface finish and oxidation were the same on all blades. In fact, Blade A and Blade C were oxidized in the same furnace at the same time. Therefore, it appears that the difference in structure may have stemmed from some difference in initial coating surface chemical composition.

The conclusion reached is that the oxide structure of Figure 9 is conducive to adherence of platinum film, and that the structure can be produced repeatably when the original electron beam vapor deposition coating process is carried out in the commerical vendor facility, following PWA 270 coating specifications. The repeatability of the

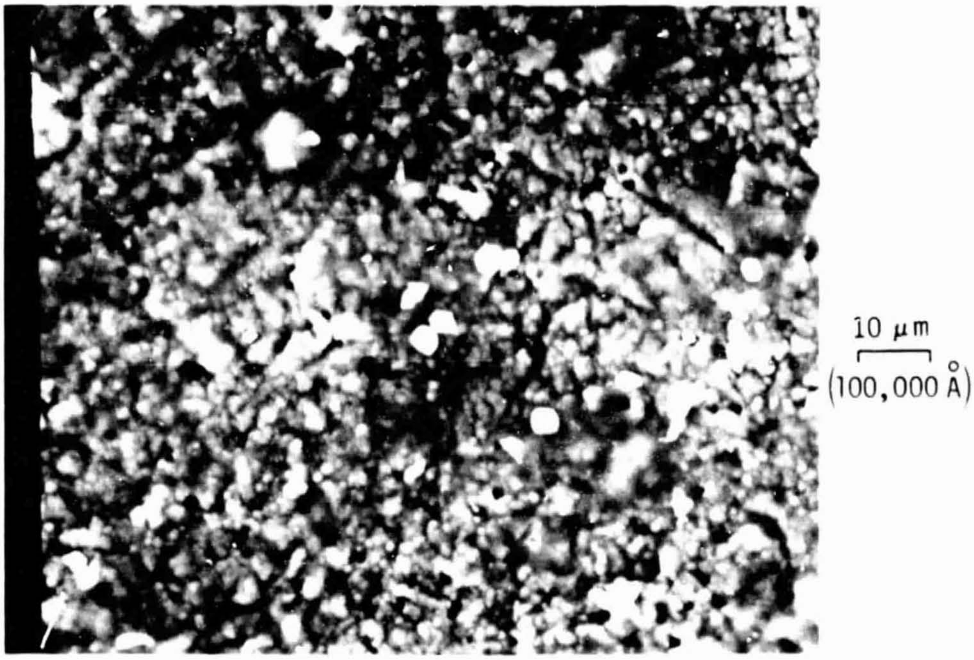


Figure 9 Scanning Electron Micrograph of a Good Alumina Surface, Favorable to Platinum Film Adherence (Adherence Specimen A, after 50-hour flame test).

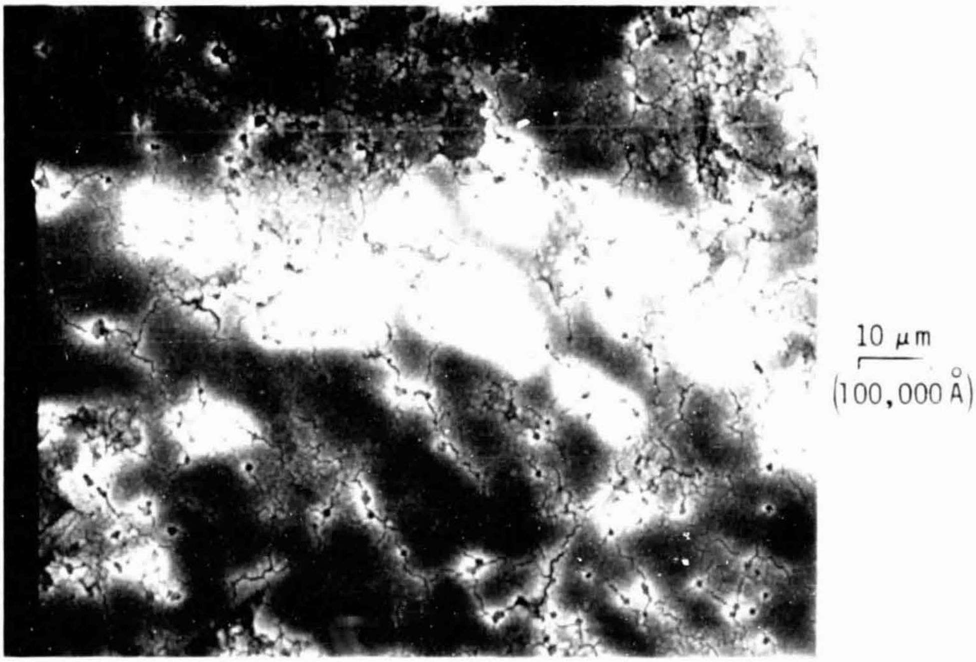


Figure 10 Scanning Electron Micrograph of a Poor Alumina Surface, Not Favorable to Platinum Film Adherence (Adherence Specimen C, not flame tested).

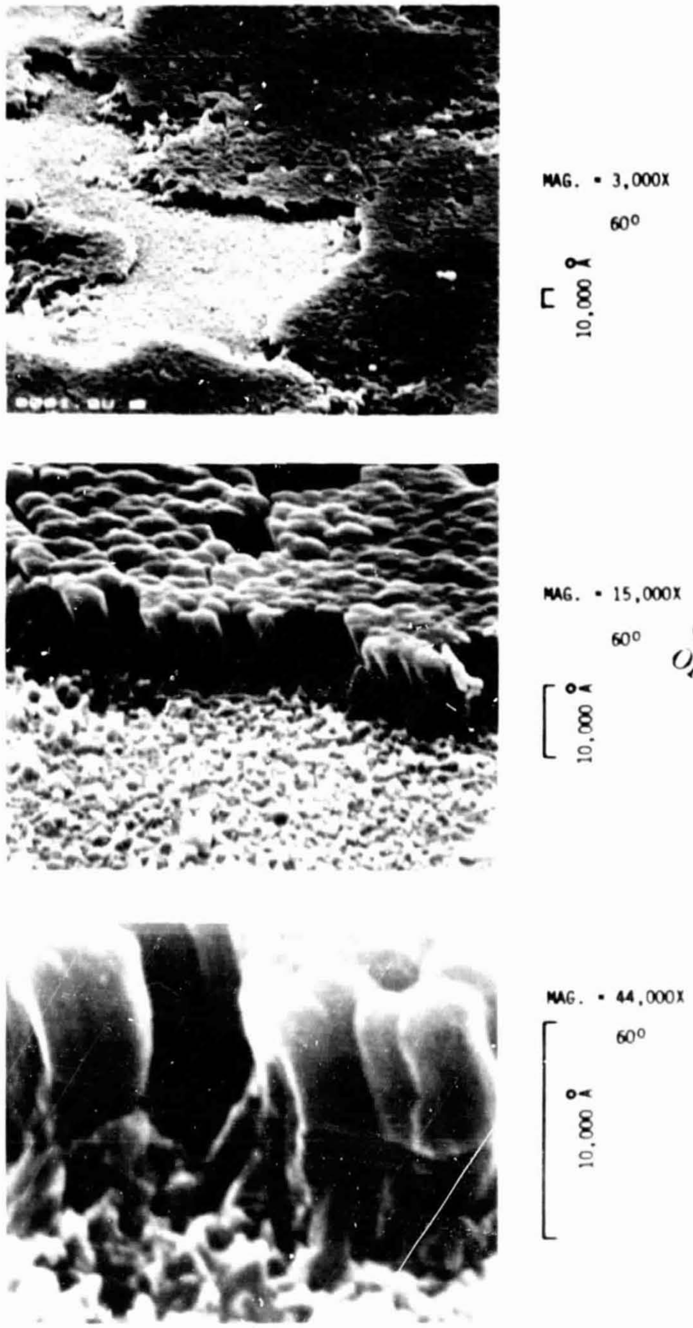


Figure 11 Scanning Electron Microscope Views of the Aluminum Oxide Structure on Blade C, Resulting in Poorly Adherent Platinum and Platinum-10% Rhodium Films - The "corncob" outer structure of the oxide is clearly visible at the higher magnifications. The fine-grained structure underlying the corncob (at a depth of 11,000 Angstroms) is also aluminum oxide. This is the same blade as that shown in Figure 10.

process was later confirmed by coating a second set of turbine blades for use in the combustor exhaust gas-stream tests. Figure 12 shows the surface structure of Blade No. 1 from this second batch, used successfully in combustor exhaust tests. The oxide structure is similar to the "good" structure in Figure 9. Further study of the coating process and alumina-forming process would be required to identify the cause of the difference in surface structure.

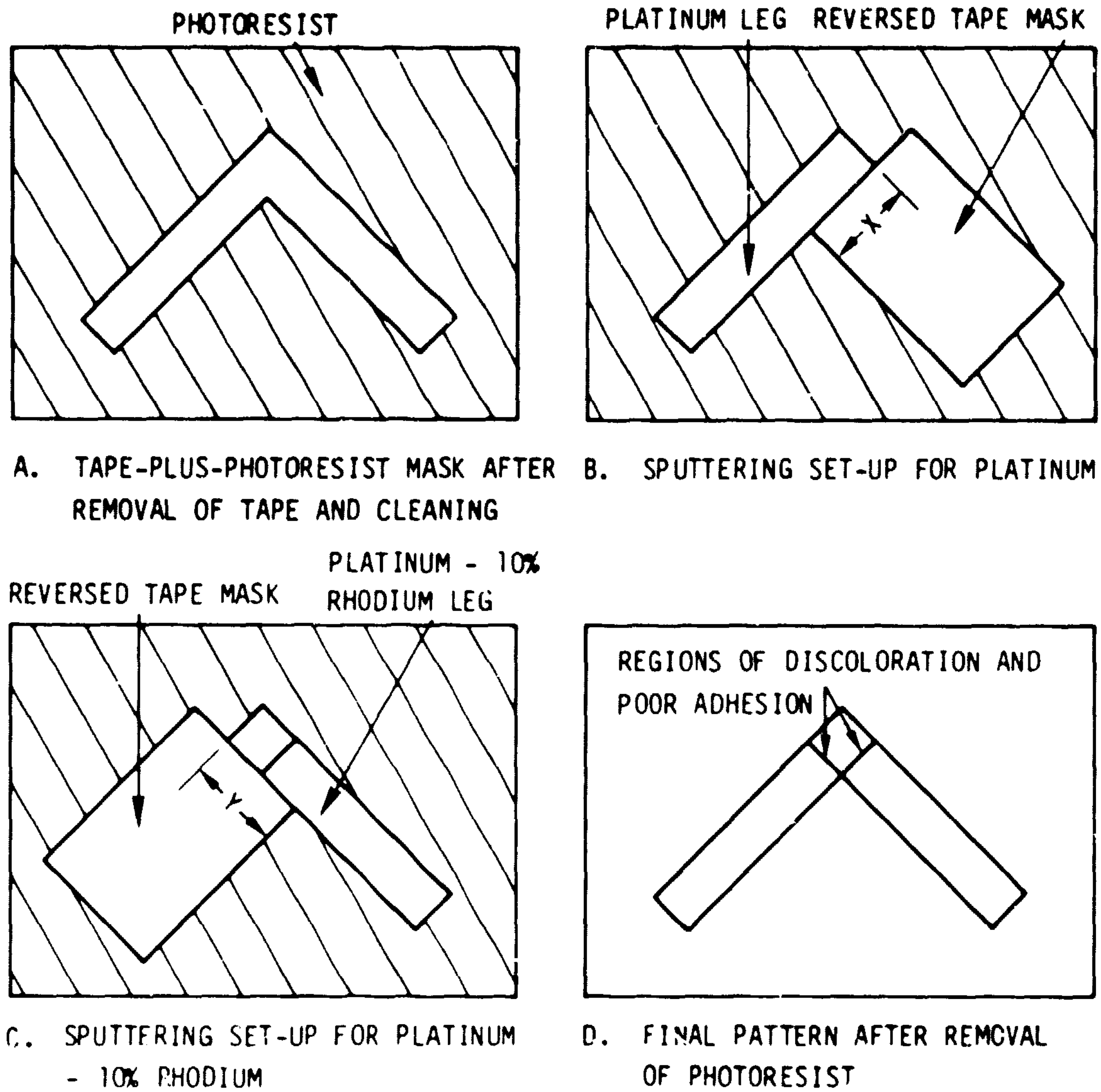
Figure 12 Scanning Electron Microscope View of Aluminum Oxide Surface (Darker Area at Left) and Platinum Film Surface on Blade No. 1 Before Combustor Tests - Adherence of platinum and platinum-10% rhodium films was generally good. The aluminum oxide structure is similar to that of Blade A, Figure 9, previously identified as a "good adherence" surface.

6.1.2 Humidity Effects

Dew point was maintained below 48°F during masking and sputtering of Blades A, B, C, and D. The importance of low humidity was emphasized in Phase I where it was concluded that a few minutes exposure to air with a dew point over 48°F can result in adsorption of moisture sufficient to prevent adhesion during subsequent sputtering, particularly when volatile cleaning agents which can produce rapid evaporative cooling and condensation are employed.

6.1.3 Masking Techniques

The crossed-tape masking procedure described in Section 5.2.2 was developed to reduce fabrication time. Only one application and removal of photoresist masking material is required in this procedure, compared with at least two applications and two removals in the standard procedure. The reduction in the number of handling and recleaning operations could also potentially reduce quality control problems. The results of several trials revealed that the crossed tape procedure introduced new problems of local failure of adherence in the region where the second film overlapped the first film to form a thermocouple junction. In these regions, the sputtered films were discolored and poorly adherent. A typical sequence of masking and sputtering steps, and the resulting region of poor adherence, is diagrammed in Figure 13.



A. TAPE-PLUS-PHOTORESIST MASK AFTER REMOVAL OF TAPE AND CLEANING

B. SPUTTERING SET-UP FOR PLATINUM

C. SPUTTERING SET-UP FOR PLATINUM - 10% RHODIUM

D. FINAL PATTERN AFTER REMOVAL OF PHOTORESIST

Figure 13 Crossed-Tape Masking Procedure, Showing Region of Poorly Adherent Films at Junction.

It was concluded that two difficulties contributed to the poor result. First, where the tape shadow mask forms one edge of a sputtered region (that is, at X or Y in Figure 13) the nearby bare Al_2O_3 surface on which the sputtered film was being deposited could become contaminated by accidental sputter etching of the tape itself. Second, firm contact of the tape shadow mask with the Al_2O_3 surface was difficult to achieve in region X or Y, and overheating and "burning" of the sputtered film could occur locally due to rf potential field concentrations in these areas. In the case of Blades A and B, where adherence of films was always better than on Blades C and D, some success was achieved with crossed tape masking, but results were always better with standard masking (Table III). In the case of Blades C and D, where adherence was poor with either procedure, the worst results were obtained with the crossed tape masking procedure.

6.1.4 Sputtering Parameters

There are several excellent reviews (as for example, Reference 10) describing the effect of sputtering process variables on the quality of the resulting thin film. The platinum films in the Phase II program are sputtered in an argon environment in diode systems from 15-cm diameter flat targets. In these diode systems, increasing the power increases the sputtering rate and might be expected to increase the impact energy and enhance adherence, but increasing the sputtering rate also increases the heating of substrates. The positive photoresist used for masking begins to decompose at 50[illegible], setting an upper limit on the sputtering rate of about 5000 Angstroms per hour. A more heat-resistant negative photoresist has been tried in another program, permitting sputtering rates up to 15,000 Angstroms per hour. The resulting platinum films (on vendor-coated blades) show the same excellent adherence and the same calibration properties. The current conclusion is that, on vendor-coated blades, adherence and calibration are not sensitive to sputtering power over this energy range. The positive photoresist is preferable to the negative photoresist because it is easier to remove completely with routine cleaning procedures.

It should be remarked that various degrees of sputter etching prior to deposition of platinum were employed during the NAS3-20768 contract work (Reference 6) and that none enhanced adherence. In fact, excessive sputter etching appeared to degrade adherence, possibly through decomposition of the photoresist. No etching was used in Phase II fabrication.

6.1.5 Reactive Sputtering

The use of 50 percent oxygen/50 percent argon as the sputtering gas during the first few minutes of sputtering of platinum, in the hope of enhancing adherence through formation of chemical bonds (platinum oxide to aluminum oxide), did not produce significant improvement. On vendor-coated blades, where adherence of all kinds of films was excellent, the adhesion of the reactively sputtered platinum films was about the same as that for all other films (Table III). On in-house coated blades, where adherence of all kinds of films was poor, the adherence of the reactively sputtered films was among the higher values, but far below the values obtained on vendor-coated blades.

6.1.6 Precoats

Sputtering a 500-Angstrom layer of an oxide-forming transition metal, selected from those forming solid solution with platinum, in the hope of enhancing adherence through formation of a chemical bond with the alumina and a local alloy with the platinum, did not produce significant improvement in adherence. Chromium and platinum-10 percent rhodium were tried. Table III shows that adherence of the platinum films was not significantly different with and without precoats.

6.1.7 Field-Assisted Bond

Applying the field-assist technique after sputtering of platinum did not measurably improve the adherence. As shown in Table III, the adherence of pure platinum was about the same with and without field assist. It is concluded that the adherence of platinum on these blades is largely due to mechanical interlocking, depending more on surface texture than on chemical or electrostatic potentials.

6.2 PERFORMANCE EVALUATION PROGRAM

Six of the 12 thin film thermocouple systems fabricated on turbine Blades No. 1 and 3 experienced failures during fabrication and initial oven calibration. The remaining six proved to be quite durable during the combustor test program, with an average life of 47 hours. Table IV summarizes the failure modes observed during the entire program. Figure 14 shows detailed post-test photographs of the 12 thermocouple systems on the two blades. The early failures and combustor test failures are discussed separately below. Finally, a comparison of the two turbine blades is presented.

6.2.1 Failure Analysis

6.2.1.1 Fabrication and Oven Test Failures

Four thin film thermocouple systems (TC4 and TC5 on each blade) were rejected during fabrication because of poor adherence of the platinum film on the airfoil surface. In two of these four cases (TC4 and TC5 on Blade No. 3) the platinum-10 percent rhodium film also showed poor adherence. The poor adherence was characterized by spontaneous lifting or spalling of the film along part of its length. The arrows in Figures 14a (Photo A) and 14b (Photo A) indicate the failure areas. The lifted and missing portions of film are easily visible. There was no significant further deterioration of these particular films during the combustor tests. Two features seem significant. First, all four failures occurred on the concave side of the blade. This is the side sputtered first in the fabrication sequence on each blade and therefore is the side with the least time of exposure to high vacuum and dry high-purity argon gas flow before sputtering. It is suggested that the longer exposure of the convex side to these conditions may have improved the surface cleanliness (by removal of latent moisture or other contaminants) and enhanced film adherence. Second, each of the two platinum films which passes across the leading edge of the blade (TC4 on each blade) suffered adherence failure in the sharply curved region near the leading edge on the concave side. It is suggested that the structure of the NiCoCrAlY coating or the alumina may be unusual in such regions. In addition, the average angle of incidence during sputtering between the flux of platinum atoms and the turbine blade approaches zero in this region, resulting in low incident energy normal to the surface to enhance adherence.

TABLE IV

SUMMARY OF FAILURE MODES

Blade No.	TC No.	Failure Mode During Fabrication	Failure Mode During Oven Test	Failure Mode During Combustor Test	Hours to Failure in Combustor
1	1			d(c)(e)	46
	2				60, f
	3			e	14
	4	a			-
	5	a			-
	6		d		-
3	1		b		-
	2				60, f
	3			d	51
	4	a, b			-
	5	a, b			-
	6			d	50

<u>Legend</u>

a = Poor adhesion of platinum film
b = Poor adhesion of platinum-10 percent rhodium film
c = Open bond, platinum-10 percent rhodium film to wire
d = Extension wire open or shorted to sheath
e = Erosion of film
f = No failure
() = Secondary failure, subsequent to first failure

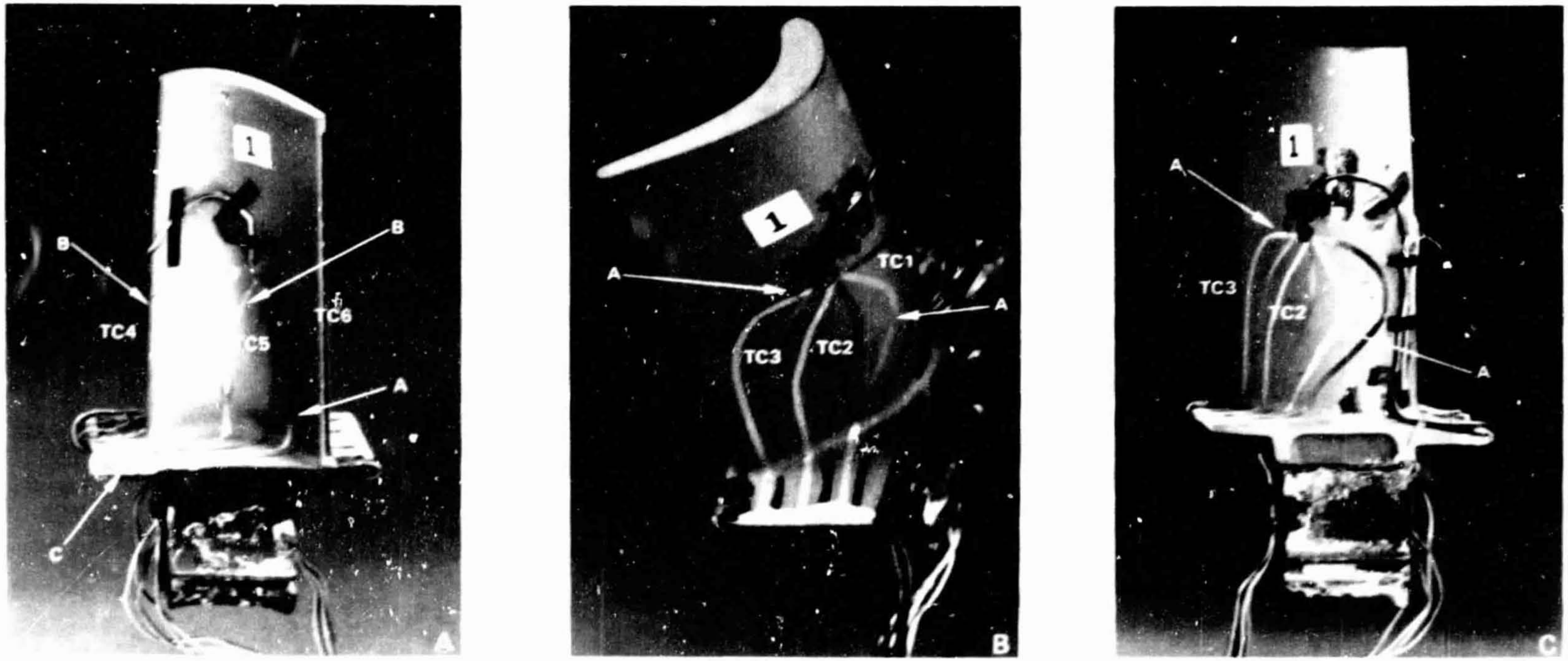


Figure 14a The Six Thin Film Thermocouples on First-Stage Turbine Blade No. 1 after Combustor Exhaust Gas-Stream Tests - The tests included 71 cycles to 1200K on the airfoil concave side, 1100K on the convex side, and 700 to 900K on the platform for a total time of 59.7 hours. Arrow A, erosion that occurred during combustor tests; Arrow B, poor initial adherence; Arrow C, lifting of cement due to excessive thickness in one location. (80-444-0462-E, -F, and -D)

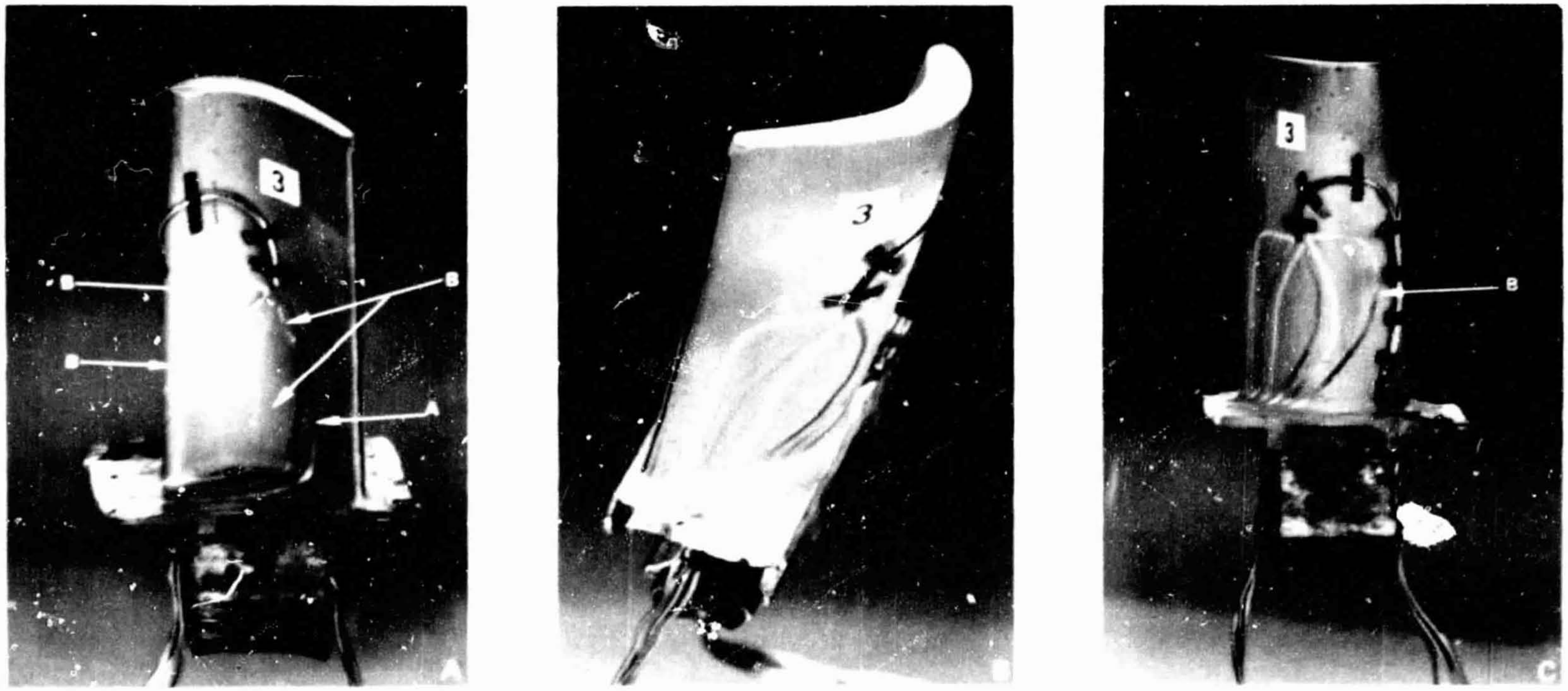


Figure 14b The Six Thin Film Thermocouples on First-Stage Turbine Blade No. 3 after Combustor Exhaust Gas-Stream Tests - The tests included 71 cycles to 1200K on the airfoil concave side, 1100K on the convex side, and 700 to 900K on the platform for a total time of 59.7 hours. Arrow A, erosion that occurred during combustor tests; Arrow B, poor initial adherence; Arrow C, lifting of cement due to excessive thickness in one location. (80-444-0462-B, -A, and -C)

Two additional thin film thermocouple systems (TC6 on Blade No. 1 and TC1 on Blade No. 3) were rejected during initial oven testing. Both systems provided stable calibration data during the early part of the oven test. Near the end of this test the adherence of the platinum-10 percent rhodium film of TC1 on Blade No. 3 failed in one spot on the airfoil near the leading edge on the convex side (Figure 14b, Photo C) resulting in an open circuit. The adherence failure was again in a sharply curved region of the airfoil near the leading edge where grazing incidence occurs during sputtering. The extension wires of TC6 on Blade No. 1 developed a short circuit to the extension-wire sheath, causing the thermocouple system to indicate platform temperature. The extension wire short circuit occurred at the point where the center conductors emerge from the sheath, and can be avoided by greater care in routing of the extension wire conductors and in cementing the extension wires to the platform.

6.2.1.2 Combustor Test Failures

After 14 1-hour exposures to the 1250K combustor exhaust gas stream, TC3 on Blade No. 1 failed due to local erosion of platinum (open circuit) near the thin film thermocouple junction at the center of the airfoil (Figure 14a, Photo B).

After 46 1-hour cycles, TC1 on Blade No. 1 failed due to shorting of extension wires to the sheath, causing the thermocouple system to indicate platform temperature. At some later time (undetermined), an open circuit developed at the platinum-10 percent rhodium film-to-lead wire bond. This was the only thin-film-to-lead-wire connection failure in the program. Some erosion of the platinum film is also evident in Figure 14a, Photo B.

After 50 1-hour cycles, TC6 on Blade No. 3 failed due to an open circuit in the extension lead wire.

After 50 1-hour cycles plus two 20-minute cycles, TC3 on Blade No. 3 also failed due to an open circuit in the extension lead wire.

The remaining two thermocouples (TC2 on Blade No. 1 and TC2 on Blade No. 3) survived the entire program of 50 1-hour cycles, 20 20-minute cycles, and one 3-hour cycle. It appears significant that these two thermocouple systems were the ones with films most favorably located on the airfoil from the point of view of sputtering geometry (flattest region and second side sputtered), temperature during combustor exhaust gas-stream tests (cooler side of airfoil), and erosion damage to films (the side of the airfoil away from the exhaust gas stream).

6.2.1.3 Summary of Failures by Location

Table V summarizes the failure analysis by location of the thin film thermocouple on the blade. Because of the unique shape of the turbine blade, failures can be correlated with location due to factors associated with fabrication as well as with testing.

TABLE V

SUMMARY OF THIN FILM THERMOCOUPLE FAILURE ANALYSIS
BY LOCATION ON THE BLADE

TC1: The thermocouple system failure mode was quite different for the two blades. On Blade No. 1, initial failure was late in the combustor program due to extension wire short circuit, followed by a later bond failure. On Blade No. 3, the failure occurred during initial oven testing due to poor adherence of the platinum-10 percent rhodium film near the blade leading edge. TC1 on Blade No. 1 suffered some erosion damage.

TC2: The thermocouples on both blades survived the entire program, with no adherence or erosion problems.

TC3: The thermocouple failure mode was different for the two blades. On Blade No. 1, failure was due to erosion of platinum during the 14th combustor cycle. On Blade No. 3, failure was due to a broken extension wire during the 52nd combustor cycle.

TC4: The thermocouples on both blades were rejected during fabrication because of poor platinum adherence and poor platinum-10 percent rhodium adherence.

TC5: The thermocouples on both blades were rejected during fabrication because of poor platinum adherence and poor platinum-10 percent rhodium adherence.

TC6: The thermocouples on both blades suffered only entension wire failures, on Blade No. 1 during oven pretest and on Blade No. 3 during the 50th combustor cycle.

Failures correlated with fabrication procedures can lead to improvements in these methods. It is recommended that the turbine blade be preconditioned under high vacuum, dry argon gas flow, and possibly mild temperature bake-out prior to initial sputtering to attempt to improve adherence. Another recommendation is to avoid angles approaching grazing incidence for sputtering of the thermocouple films. Finally, greater care should be exercised in lead-wire and extension-wire routing.

Failures associated with location during combustor testing suggest that film patterns should be selected to avoid areas of highest erosion. Figure 14 indicates that the areas of greatest erosion during the combustor tests are the central portion of the airfoil on the convex side (two film failures during combustor tests) and the rear half of the airfoil on the concave side (no failures, but obvious heavy impact of dark foreign matter, thought to be carbon, in this area).

6.2.2 Vibration Tests

Turbine Blade 1 was vibration tested prior to combustor exhaust gas-stream testing, and Blade 3 was vibration-tested after 56.7 hours of exhaust gas-stream testing. The test procedure, involving 1 hour of shake testing on each of three axes, is described in Section 5.1.5.

Post-test examination showed no deterioration of any kind. The lead-wire-to-thin-film connections and thin-film-to-alumina bonds remained mechanically sound. There was no change in electrical resistance to ground or in electrical continuity in the thin film thermocouple systems.

The negligible effect of vibration on sputtered thin films was to be expected, since the mechanical stresses involved are orders of magnitude smaller than the stresses arising from thermal gradients in oven tests and in the hot gas flow environment. The vibration tests were most important in establishing the durability of the thin-film-to-lead-wire connections.

6.2.3 Measurement Error Analysis

6.2.3.1 Correlation with Temperature Gradient

Indicated temperature error (T_{REF} - T_{SPUT}) is plotted against blade temperature gradient (T_{REF} - $T_{PLATFORM}$) in Figures 15, 16, and 17. T_{SPUT} is the temperature indicated by the thin film thermocouple, when the standard ANSI Type S (platinum versus platinum-10 percent rhodium) calibration table is used to translate thermocouple voltage to temperature. T_{REF} is the temperature indicated by the reference Type S wire thermocouple located adjacent to the thin film thermocouple junction. $T_{PLATFORM}$ is the temperature indicated by the reference Type S wire thermocouple adjacent to the extension wire connection to the thin films. The response of a thin film platinum versus platinum-10 percent rhodium thermocouple would lie exactly along the horizontal heavy dashed line seen in Figure 15 as T_{REF} - T_{SPUT} = 0. For rhodium in excess of 10 percent in the platinum-rhodium film, the response would lie along an upward sloped line. For no rhodium in the platinum-rhodium film, the response would lie along the 45^{o} downward sloped dashed line 0-Y.

Factors other than composition may affect the slope of the error line in Figures 15, 16, and 17. For example, if an electrical short circuit resulted in a direct connection between the platinum and platinum-rhodium films near the extension wire connections, then readings on the downward sloped line 0-Y would result.

Throughout this section, a straight line is drawn from the origin, 0, through a data point on the assumption that the error versus temperature gradient is a linear function. Although many nonlinear effects could occur (for example, a significant loss of insulation resistance in the insulating film with increasing temperature), no such effects were observed in these experiments.

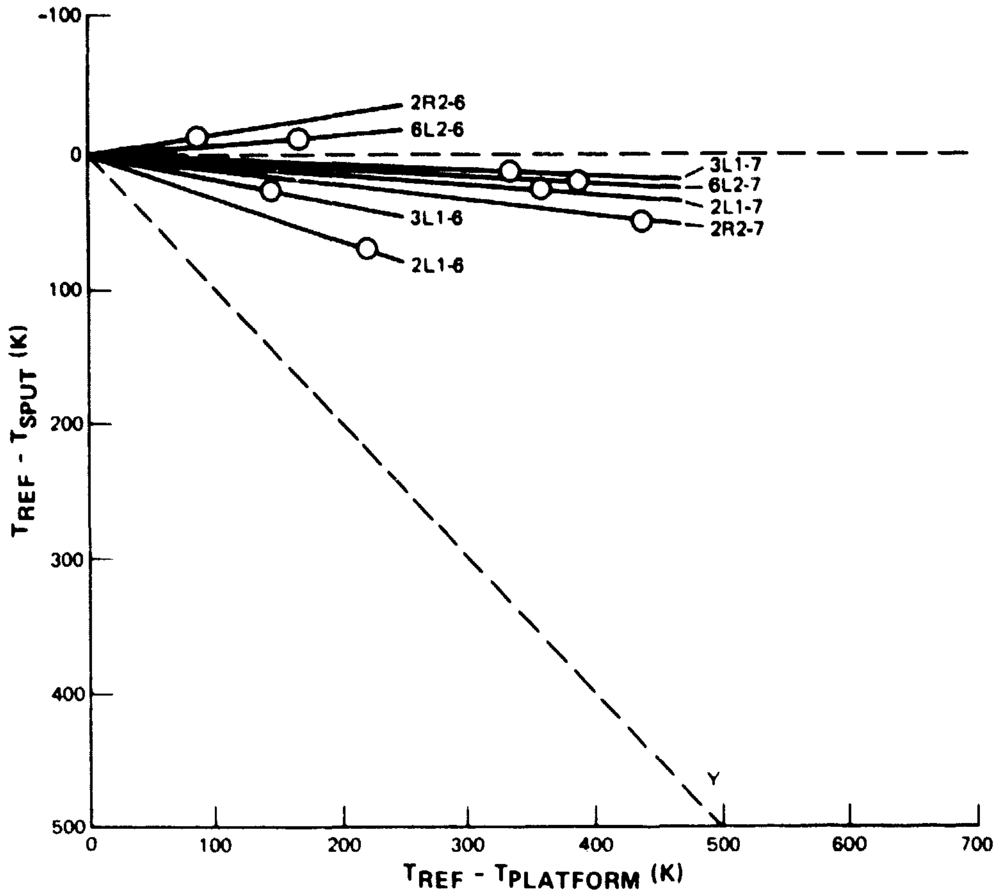


Figure 15 Calibration Data for Eight Thin Film Thermocouples on Phase I (Contract NAS3-20768) Simulated Blades - T_{REF} = actual junction temperature; $T_{PLATFORM}$ = actual platform temperature; T_{SPUT} = indicated junction temperature (assuming Type S response); the alpha-numeric symbol (for example, 2R2) is the serial number of the simulated blade; the suffix dash number is the thermocouple number on the blade. These data were obtained during initial oven tests or during the first hour of torch flame tests.

In Figure 15, data from the eight thin film thermocouple systems on the simulated blades of the Phase I program (Contract NAS3-20768) are shown. The Phase I data shown were obtained early in the testing of each thin film thermocouple system. T_{REF} - T_{SPUT} generally increased as aging took place later in the Phase I program, indicating a decrease in emf of the thin film thermocouple with time at constant temperature.

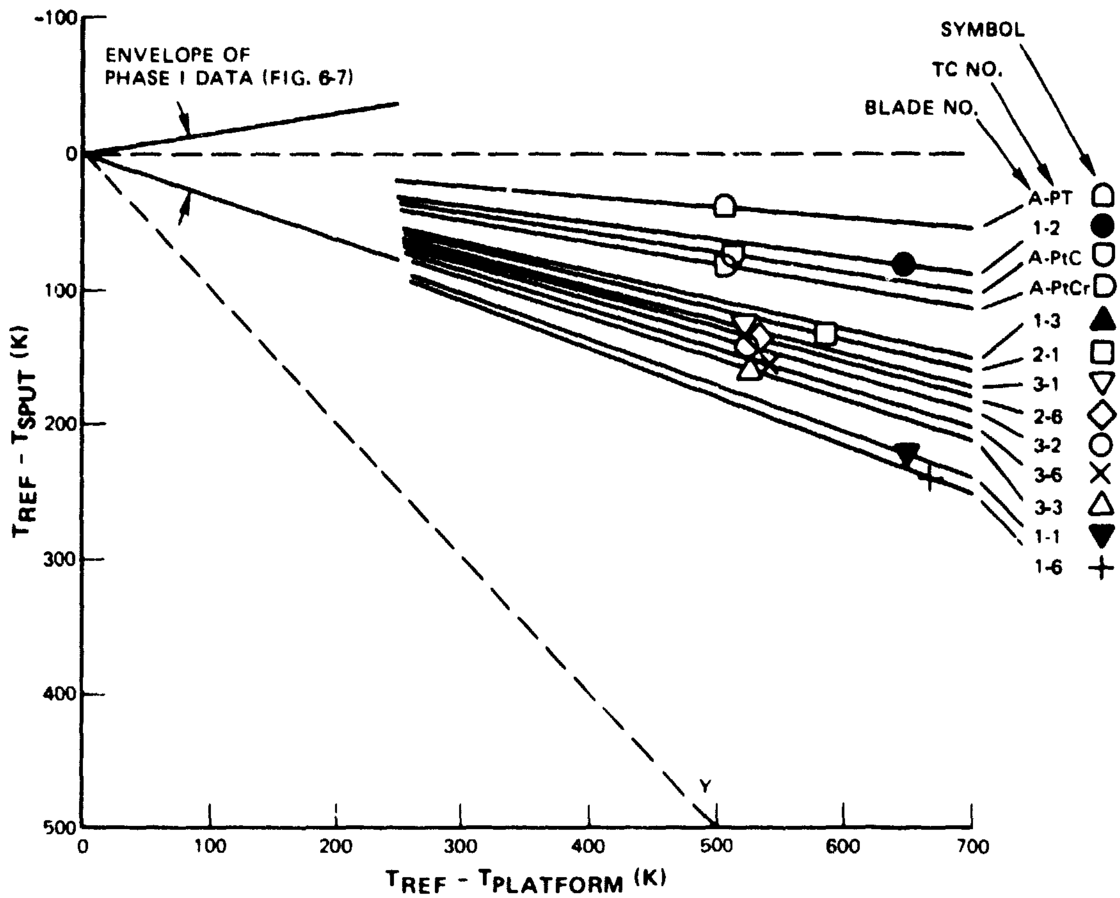


Figure 16 Calibration Data for the Thirteen Thin Film Thermocouples of Phase II (Contract NAS3-20831) - Temperature nomenclature is the same as that for Figure 15.

In Figure 16, data from the 13 thin film thermocuple systems of the Phase II program, reported in this document, are shown. For each of these systems, the initial oven calibration data point is presented. The envelope of the Phase I data of Figure 15 is shown for comparison.

In Figure 17, oven calibration data are presented for two thin film thermocouples on Blade 2 at two different times. The two lower curves are the initial oven calibration points, the same points as shown in Figure 16. It should be emphasized that, although these are the "initial" oven calibration points, the blades have been exposed for several hours to 1050K temperatures during lead wire bonding prior to this calibration. Note that these data points represent an error between reference thermocouple and thin film thermocouple of about 25 percent of the applied temperature gradient.

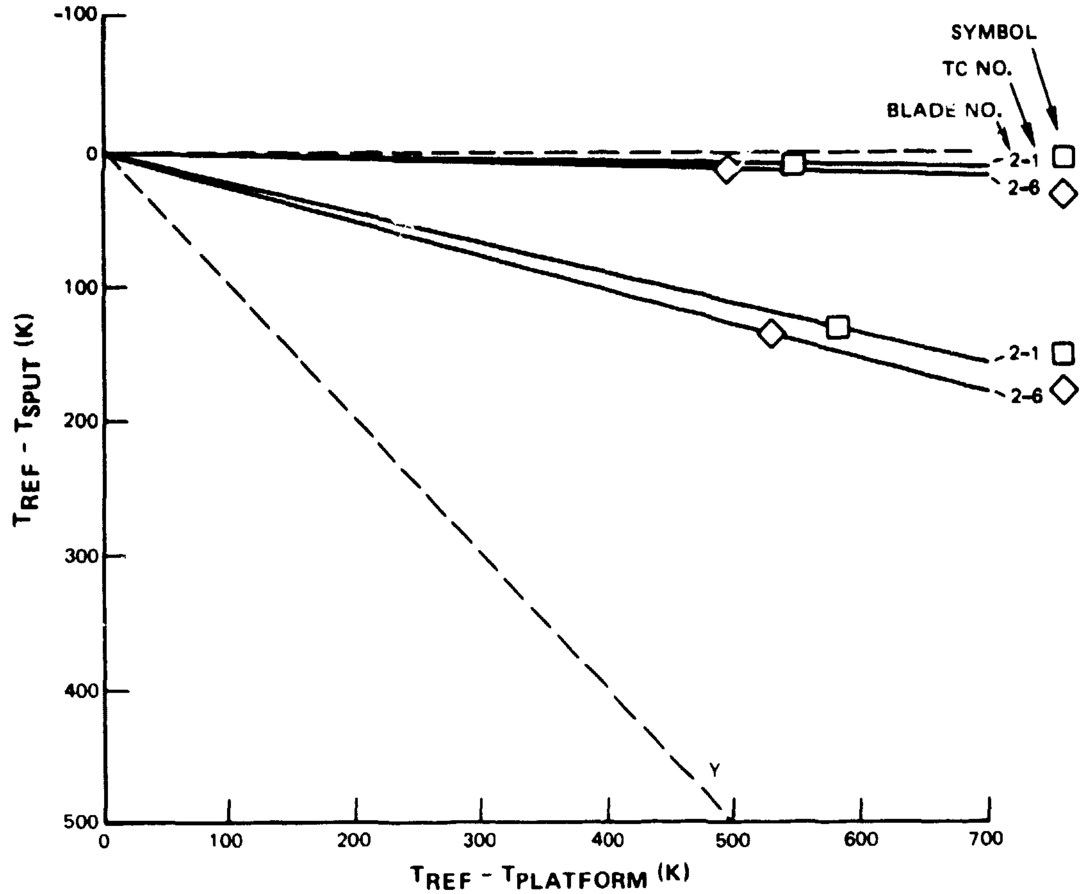


Figure 17 Calibration Data for the Two Thin Film Thermocouples on Blade 2 - Lower curves: as-fabricated; upper curves: after 45 minutes at 1366K. Temperature nomenclature is the same as that for Figure 15.

Immediately after this test, the blade was heat treated at 1366K for 45 minutes in an isothermal oven, following a suggestion resulting from a related in-house program at the Government Products Division of Pratt & Whitney Aircraft. Following this heat treatment, Blade No. 2 was subjected to a second oven test of 50 hours duration, with a large temperature gradient. The first data points of this run are included in Figure 17 as the upper two curves.

A study of Figures 15 through 17 and related calibration data taken throughout the testing programs resulted in the following observations:

1. Error is approximately proportional to temperature gradient. Wherever data at more than one temperature gradient are obtained over a short period of time, each individual thin film thermocouple system displays an error, $T_{REF} - T_{SPUT}$, increasing linearly with the temperature gradient imposed .., the thin film portion of the system.

2. The spread of all error curves is large. The error of the thin film thermocouple systems ranges from +14 to -36 percent of the temperature gradient on the airfoil.

3. The calibration errors in the systems having reactively sputtered platinum and chromium-precoated platinum films were near the average for all systems.

4. The spread of error curves on each blade is large. The spread of error among all thin film thermocouples on any one blade ranges from 6 percent of temperature gradient (four systems on Blade 3) to 24 percent (four systems on Blade 1). The spread is as large among systems on one side of the blade as from one side to the other.

5. The spread of error curves on flat simulated blades is large. Fabrication of thin film systems on the flat surfaces of Phase I did not result in smaller error spread than on the complex curved simulated blade surfaces of Phase II blades.

6. A large error spread occurred when the temperature profile along the blade was peaked at a point away from the junction. Thin film thermocouple systems No. 2, 4, 6, 8 on the four different simulated blades of Phase I were the only ones deliberately exposed for 50 hours to temperature profiles characterized by a temperature peak (far higher than the measured temperature) midway along the length of the film. The spread among these four systems is extreme (+14 to -30 percent of end-to-end temperature gradient).

7. Heat treatment at 1366K reduced the error on Blade 2 to near zero (Figure 17). The initial calibration points of the two thin film thermocouples on Blade 2 show average error of about 25 percent of temperature gradient. The two thin film thermocouples on this blade show an average error of only about 2 percent of temperature gradient immediately after the bake-out.

6.2.3.2 Correlation with Discoloration

The visual character of as-sputtered platinum films was a bright metallic luster while the platinum-10 percent rhodium films were slightly less bright and reflective. The pure platinum films retained a bright luster throughout the program, except in some local regions where foreign matter believed to be carbon particles became deposited during combustor tests. Portions of the platinum-10 percent rhodium films darkened to various degrees during lead wire bonding at temperatures from 1100K to 1200K. These films showed further changes in discoloration, darkening in some areas and becoming brighter in other areas, during oven tests, torch tests, and combustor tests with temperature gradients. The photographs of Blades 1 and 3 in Figure 6-6 show the final coloration of all films after the 60-hour combustor tests. Note that the right-hand film in each pair forming a thermocouple system is always the platinum-10 percent rhodium film and is often darkened.

In the controlled experiment on Blade 2 of the effect of heat treatment on the thin films, already mentioned in Section 6.2.3.1, the various degrees of darkening of the platinum-10 percent rhodium films during previous testing were completely reversed by bake-out at 1366K for 45 minutes, and the films attained a bright metallic luster. Subsequent oven calibration showed that the temperature readings from the thin film thermocouple systems were then within 2 percent of reference wire thermcouple temperature readings.

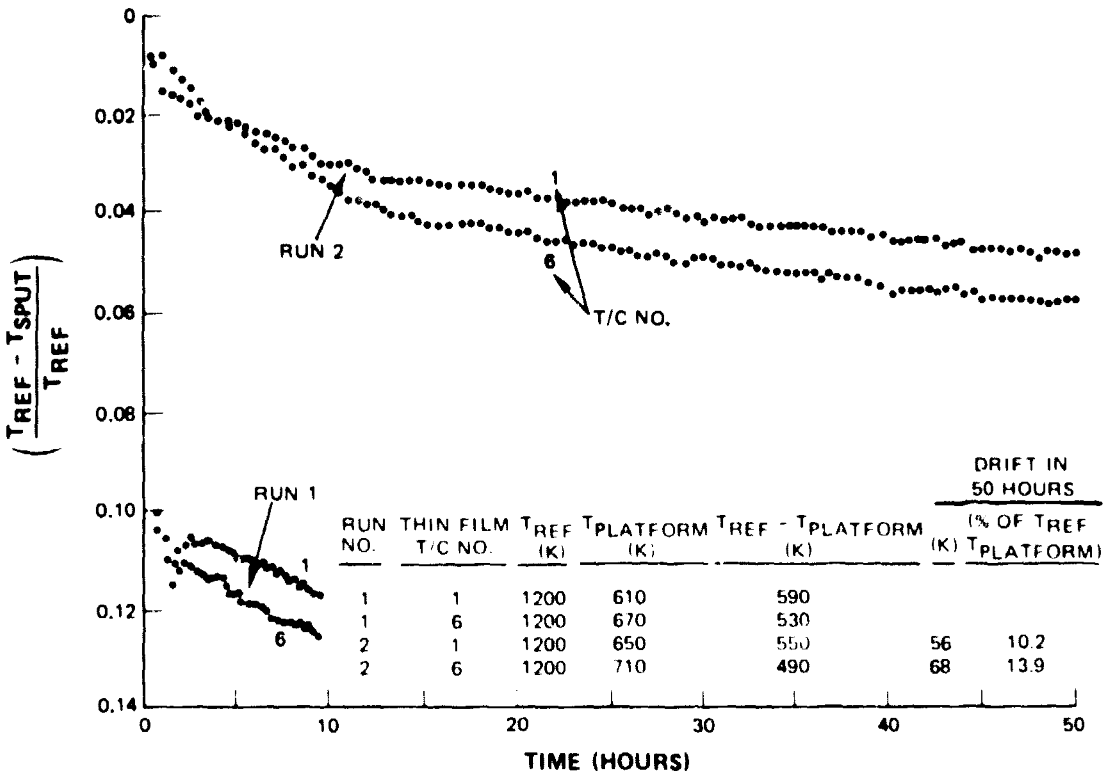
The discoloration behavior is consistent with that observed during selective oxidation of rhodium in platinum-10 percent rhodium wire thermocouples in engine testing. Rhodium is known to form dark oxides (Rh_2O_3 and RhO) when exposed to air at temperatures up to about 1166K, and the oxides are known to decompose at higher temperatures (Reference 11).

6.2.3.3 Correlation with Calibration Drift

The significant change in calibration of the thin film thermocouple systems with time has already been suggested in Figure 17. This behavior deserves more detailed examination, since the behavior throws more light on the reasons for the spread in initial calibrations in the Phase I and Phase II programs.

Drift behavior was found to be quite simple and systematic during steady-state oven calibration tests with a fixed temperature distribution along the film. In all such tests, the thin film thermocouple reading decreased slowly and smoothly with time. For example, Figure 18 shows data derived from continuous chart recordings of the Blade 2 oven calibration runs. Throughout the tests, the airfoil temperature (location "A" of Figure 3) was constant at 1200K on both sides of the airfoil, and the two platform reference locations were at about 600K. During run 1, which began with discolored platinum-10 percent rhodium films, the readings of the two thin film thermocouples decreased by about 4 percent of the temperature gradient in 8 hours, starting from an initial reading which was low by 25 percent of temperature gradient. During run 2, which began with bright metallic films, the readings decreased by about 6 percent of the temperature gradient in 8 hours, and 12 percent in 50 hours, starting from an initial reading which was within 1 percent of the correct reading.

The drift during cyclic exposures in torch tests and combustor tests was less simple and systematic, as shown in Figure 19. The error sometimes increases and sometimes decreases from cycle to cycle. It is important to note that the temperature distribution on the blade in these tests was usually constant during each cycle, but not closely repeatable from cycle to cycle, due to random variations in flame position with carbon build-up in the torch or combustor. The temperature at the center of the concave side of the airfoil was maintained within about 20K of 1250K throughout the tests of Blades 1 and 3, but the temperature at the center of the convex side of the airfoil was 1050K to 1150K from cycle to cycle. Platform temperatures were 750K to 850K from cycle to cycle.



RUN NO.	THIN FILM T/C NO.	T_{REF} (K)	$T_{PLATFORM}$ (K)	$T_{REF} - T_{PLATFORM}$ (K)	DRIFT IN 50 HOURS (K)	DRIFT IN 50 HOURS (% OF T_{REF} $T_{PLATFORM}$)
1	1	1200	610	590		
1	6	1200	670	530		
2	1	1200	650	550	56	10.2
2	6	1200	710	490	68	13.9

Figure 18 Comparison of Thin Film Thermocouple Error and Drift in Two Oven Tests of Blade 2 - Run 1: blade in as-fabricated condition; Run 2: after Run 1 and after a 45-minute soak at 1366K. In each run, time is measured from the moment of insertion in an oven preheated to 1200K.

It can be seen from Figure 19 that the trend of long-term drift in the combustor tests was toward lower thin film thermocouple readings relative to the adjacent reference wire thermocouples on Blade A and Blade 1, but toward higher readings on Blade 3. The oven drift of thermocouple 6 on Blade 2, run 2, is superimposed on Figure 19 for comparison.

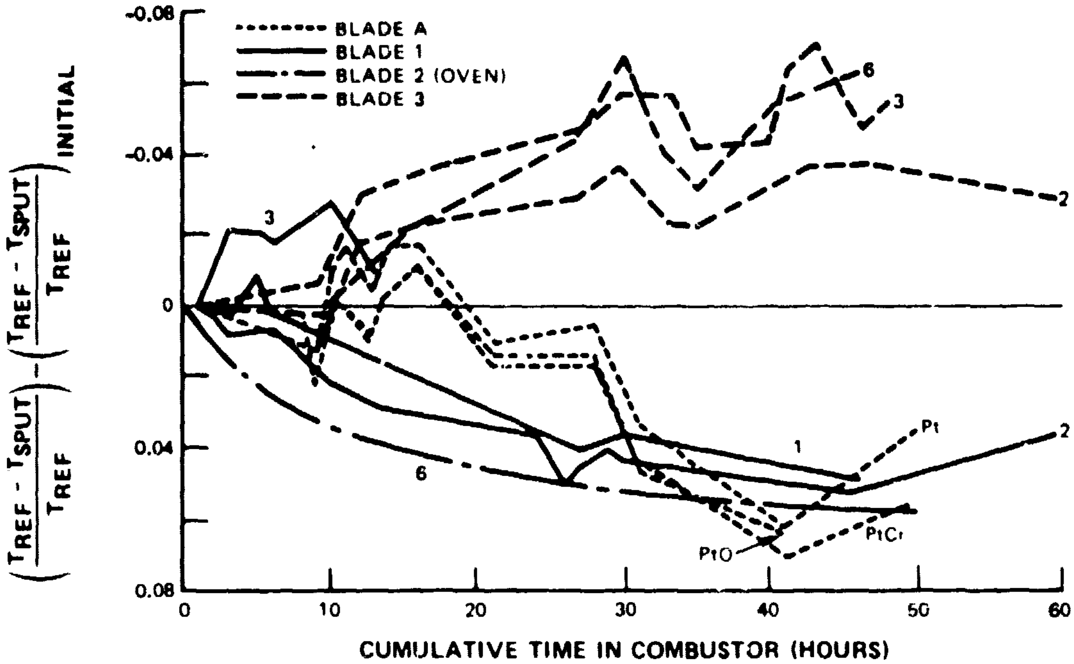


Figure 19 Relative Drift in Calibration of Thin Film Temperature Sensor Systems on First-Stage Turbine Blades During 1250K Tests in Combustor Exhaust Gas Stream - One 50-hour oven test is included for comparison (T_{SPUT} is temperature indicated by thin film thermocouple, assuming Type S calibration, and T_{REF} is the adjacent reference wire thermocouple temperature).

6.2.3.4 Correlation with Composition Measurement

Samples of the platinum-10 percent rhodium films as sputtered on glass slides were submitted to an analytical chemical laboratory (MERL) for determination of rhodium content, using atomic absorption photospectroscopy, on three occasions during the Phase I and Phase II programs. A sample sputtered during the period of fabrication of the Phase I simulated blades were reported to be 10 percent ±1 percent rhodium. Two samples sputtered at two different times during the Phase II program were reported to contain, respectively, 8.2 percent ±0.5 percent and 8.7 percent ±0.5 percent rhodium.

In these tests, the total rhodium content is reported by the analysis, regardless of whether the rhodium appears as an oxide or as an alloy with platinum. The measured rhodium content does not seem to correlate in any obvious way with observed trends in calibration results. It would appear that a better analytical technique would be required before useful correlation of chemical composition and thermocouple performance could be obtained.

SECTION 7.0

DISCUSSION OF MEASUREMENT ERROR

The results of the oven calibration tests and long-term drift tests in the oven, torch, and combustor environments (Figures 15 through 19) suggest strongly that the oxidation of rhodium in the platinum-10 percent rhodium alloy films governed both the "initial" readings and subsequent long-term drift behavior. These results are consistent with observations made in a related in-house program at the Pratt & Whitney Aircraft Group, Government Products Division. All of the observed behavior would be predicted on the basis of three assumptions: the rhodium content (a) is 9.9 $\pm$ 0.1 percent in the as-sputtered bright metallic films; (b) falls as low as 6.4 percent in films which have been darkened by oxidation at temperatures in the 1050K to 1300K range; and (c) returns to approximately 10 percent if the film is exposed to temperatures above 1166K (oxide decomposes, rhodium regained).

On the basis of these assumptions, the error in any particular thin film system is reversible by heat treatment, as observed on Blade 2, and the error depends upon the time-at-temperature history of each portion of each film and upon the temperature profile along the film at the time of the error measurement.

Note that low effective rhodium content, resulting from conversion of some rhodium to an oxide which does not display thermoelectric properties, can result in either positive or negative errors in the thin film thermocouple temperature measurement. For example, a number of hypothetical cases are illustrated in Figure 20, an emf-temperature diagram utilizing the gradient approach of Moffat (Reference 12). The thermocouple emf, E, generated by a temperature difference between a cold reference temperature, T_0, and a hot junction temperature, T_j, is determined by tracing a continuous path from (T_0, 0) around a thermocouple loop and back to (T_0, E). Table VI defines five possible loops illustrated in Figure 20. Note that a low rhodium content in a film exposed to a monotonic temperature profile results in a low emf, L, while a low rhodium content in some portion of a film exposed to a temperature profile with a maximum at some point between the hot and cold junctions may result in either a high reading, H, or a low reading, L, depending on whether the location of the film portion with the low rhodium content is to the left or right of the peak temperature.

If oxidation is the governing factor, then the drift rate would be inversely proportional to film thickness, that is, to volume-to-surface ratio. This expectation is borne out by a comparison of the 50-hour drift data of initially bright films in the current program test of Figure 16 and the Dils test of thicker films reported in Reference 5. Table VII lists the important parameters for each of these tests. Initial error (extrapolated to zero time) was less than 1 percent of temperature gradient in both tests. The error after 50 hours in the current program, from Figure 16, is seen to be about six times the error after 50 hours in the Reference 5 program, where six times thicker films were used.

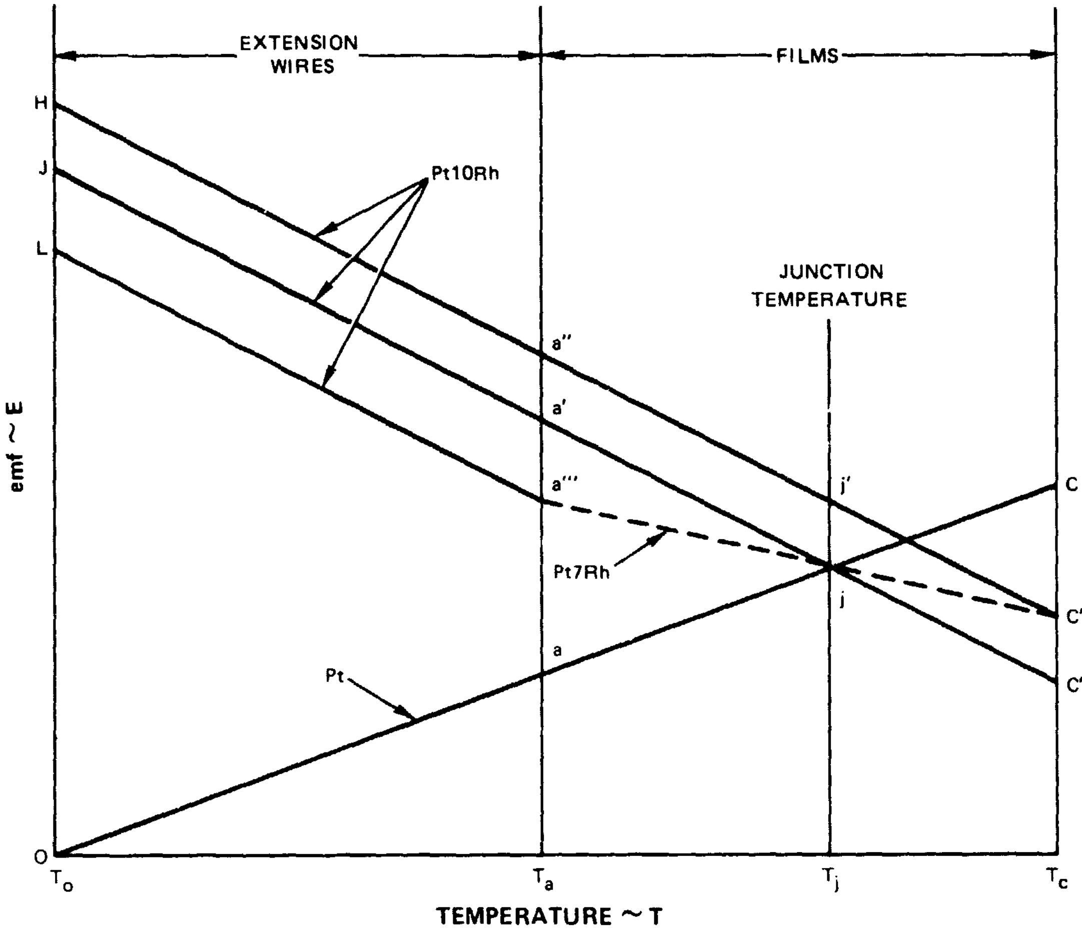


Figure 20 Illustration of Positive and Negative Measurement Errors Resulting from Low Rhodium Content.

TABLE VI

ERRORS RESULTING FROM LOW RHODIUM CONTENT

Path Description	emf Path in Figure 20	Final emf in Figure 20
Monotonic Profile (T_o to T_j):		
Type S	Oaja'J	J
Type S, except Pt7Rh T_j to T_a	Oaja'''L	L
Peaked Profile (T_o to T_a to T_c to T_j:		
Type S	Oajcjc'ja'J	J
Type S, except Pt7Rh T_j to T_a	Oajcjc'ja'''L	L
Type S, except Pt7Rh T_j to T_c	Oajcjc''j'a''H	H

TABLE VII

COMPARISON OF ERROR DATA

	Current Program	Ref. 5
Film Thickness (μm)	2	12.5
T_{REF} (K)	1200	1366
$T_{PLATFORM}$ (K)	700	700
Error after 50 hours, percent of (T_{REF} - $T_{PLATFORM}$)	12	1.8

The spread of the calibration curves in Figures 15, 16, and 17 and the erratic drift during cyclic combustor tests in Figure 19 appears to be caused by varying rhodium content due to oxidation during fabrication and test. Some thin film thermocouple systems were subjected to extra heat treatment at 1300K (above the rhodium oxide decomposition temperature) to increase the Al_2O_3 thickness after lead wire bonding, and these would be expected to show less initial error in oven tests. Some were cycled several times through the lead wire bonding procedure at 1100K (in the range where rhodium oxidizes rapidly) and these would be expected to show larger initial error. Finally, the metal film thickness undoubtedly varies considerably along the length of the film and from film to film due to variation in distance of various portions of the blade from the sputtering target. Those systems with thinnest films in regions of large temperature gradient are expected to show largest initial error and largest drift rates. Fabrication process records available in the present program are not detailed enough to permit correlation of error with thickness, temperature distribution, and time history for each thin film thermocouple. Warm-up times and cool-down times were not usually recorded, and film thickness distributions were not measured. In order to verify further the error and drift mechanism, it is recommended that Blades 1 and 3 of Figure 15 be retested after a 1366K heat treatment to decompose any rhodium oxide, as was done with Blade 2. If the spread of calibration curves collapses into a narrow band near zero error, then further explanation of the details of calibration spread becomes academic. If the spread does not collapse, then separate extension wires could be attached to each end of each film (platinum and platinum-rhodium) and the assembly tested in a temperature gradient to establish whether the observed behavior is peculiar to the platinum film, the platinum-10 percent rhodium film, or both.

To reduce the rhodium oxidation problem to reasonable limits during engine tests, thicker platinum-10 percent rhodium films should be employed in regions of largest temperature gradient along the film, and development of oxygen-barrier overcoating layers could be considered.

The accuracy goal of the current three-phase program is initial error less than 0.75 percent of Fahrenheit temperature and 60-hour drift of less than 5 percent of Fahrenheit temperature. If the largest gradient expected along the film is 50 percent of Fahrenheit temperature, then the goals translate to initial error less than 1.5 percent of gradient and 60-hour drift of less than 10 pecent of gradient.

It is believed that the initial-error goal of 1.5 percent of gradient has been attained (Figure 16) but that the drift is about 1.2 times the desired maximum rate of 10 percent of temperature gradient in 60 hours. Furthermore, if oxidation is indeed the major source of drift, then the rate of drift will increase in proportion to the partial pressure of oxygen. In a high-pressure turbine inlet flow in an operating engine, an oxygen partial pressure ten times that of sea level air may be expected. The drift rate of the present film systems may then increase to 1.2 x 10 = 12 times the desired rate. This may conceivably be overcome by increasing the film thickness alone. It is suspected that film thickness in the root area of the airfoil may be only on the order of 1 μm, and that increasing this thickness to 15 μm (the maximum originally specified to minimize alteration of blade temperature profiles on hollow thin-walled cooled blades) may result in acceptably small drift. Films of 15 μm thickness can be sputtered readily, and should be as durable as the present 1 μm films, based on the experience in Reference 4. An alternative approach would be a combination of increased film thickness and an oxygen-barrier overcoat, with total thickness of film and overcoat not exceeding 15 μm.

SECTION 8.0

CONCLUDING REMARKS

The technology developed under the previous Phase I program (Reference 6) for fabrication of sputtered thin film platinum versus platinum-10 percent rhodium thermocouples on turbine blades, for the purpose of measuring metal surface temperatures, was improved and extended.

Thin film systems were applied to actual turbine blades of complex shape, the size of the thin film systems was reduced slightly, and testing at 1250K was extended to a higher Mach number (0.5) in a combustor exit flow. The metal film thickness used was 2 micrometers (μm), as in the Phase I program.

Adherence of the platinum films was improved through a systematic study of effects of variations in fabrication techniques on four turbine blades. The deciding factor in attaining repeatably good adherence was found to be the morphology of the aluminum oxide surface on which the platinum was sputtered. Scanning electron microscopy revealed that excellent adherence was associated with a dense fine-grained alumina with some flat scales and surface reentry features, promoting bonding through mechanical interlocking. The favorable morphology and good adherence were repeatably attained by controlled oxidation of NiCoCrAlY coatings which had been applied in a vendor laboratory coating facility. Alterations in thin film fabrication procedures to promote additional chemical or electrostatic bonding had little effect on adherence. These alterations included reactive sputtering of platinum, sputtering of precoats of chromium or platinum-10 percent rhodium, and application of an electrical potential after sputtering (field assisted bonding).

Two additional blades were then fabricated, each containing six thin film thermocouples. Six of these 12 thin film thermocouples failed during the fabrication and oven calibration tests. Failures were attributed to poor film adherence, variability in fabrication techniques, and poor extension wire attachments. Appropriate modifications to these procedures are recommended.

The two blades were then subjected to a combustor exhaust gas test, consisting of 60 hours at 1250K, Mach number = 0.5, and 71 thermal cycles. A third blade was subjected to tests of long-term calibration drift under steady state, 1250K oven conditions with a 500K temperature gradient.

The average life of the thin film thermocouple systems in the high speed combustor flow was 47 hours, where failure was defined as either an open circuit or a 6 percent drift in calibration (percentage error based on absolute temperature).

An extensive study of the problem of calibration drift was conducted. The principal source of drift was determined to be oxidation of the rhodium in the platinum-10 percent rhodium film. An increase of film thickness to 15 μm is therefore recommended to reduce drift in actual engine testing. Protective overcoats are also recommended as a way to resolve the erosion and corrosion problems.

SECTION 9.0

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