NASA Technical Memorandum 106714

1N-35 20314 14 P

Applications of Thin Film Thermocouples for Surface Temperature Measurement

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Prepared for the Conference on Spin-Off Technologies for Commercial Sensors and Scientific Instrumentation cosponsored by the National Aeronautics and Space Administration and the Society of Photo-Optical Instrumentation Engineers San Diego, California July 24–29, 1994



National Aeronautics and Space Administration

(NASA-TM-106714) APPLICATIONS OF THIN FILM THERMOCOUPLES FOR SURFACE TEMPERATURE MEASUREMENT (NASA. Lewis Research Center) 14 p N95-11249

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Applications of thin film thermocouples for surface temperature measurement

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ABSTRACT

Thin film thermocouples provide a minimally intrusive means of measuring surface temperature in hostile, high temperature environments. Unlike wire thermocouples, thin films do not necessitate any machining of the surface, thereby leaving intact its structural integrity. Thin films are many orders of magnitude thinner than wire, resulting in less disruption to the gas flow and thermal patterns that exist in the operating environment. Thin film thermocouples have been developed for surface temperature measurement on a variety of engine materials. The sensors are fabricated in the NASA Lewis Research Center's Thin Film Sensor Lab, which is a Class 1000 Clean Room. The thermocouples are platinum-13% rhodium vs platinum and are fabricated by the sputtering process. Thin film-to-leadwire connections are made using the parallel-gap welding process. Thermocouples have been developed for use on superalloys, ceramics and ceramic composites, and intermetallics. Some applications of thin film thermocouples are: temperature measurement of Space Shuttle Main Engine turbine blade materials, temperature measurement in gas turbine engine testing of advanced materials, and temperature and heat flux measurements in a diesel engine. Fabrication of thin film thermocouples is described. Sensor durability, drift rate, and maximum temperature capabilities are addressed.

1. INTRODUCTION

Thin film thermocouples (TFTCs) have been developed for surface temperature measurement on several material systems for various aerospace applications. Surface temperature data is needed in engine systems in order to provide experimental verification of computational models that have been developed for fluid mechanics and structures. Unlike wire thermocouples, thin film thermocouples provide a minimally intrusive means of measuring surface temperature. Wire thermocouples must be either mounted on the surface or set into machined grooves in the surface. In an engine environment, surface mounted wire thermocouples create a disruption of the gas flow over the surface, thereby changing the environmental conditions at that surface. Wire thermocouples set into machined grooves in the surface prevent disruption of the gas flow; however, this method compromises the structural integrity of the component. As a result, the surface thermal profiles provided by wire thermocouples do not accurately reflect the true operating conditions. Thin film thermocouples are sputter deposited directly onto the surface and have thicknesses on the order of a few micrometers. Therefore, TFTCs create minimal disturbance of the gas flow over the surface and do not require that the surface be structurally altered. Consequently, TFTCs add negligible mass to the surface and have minimal impact on the temperature distribution. TFTCs were developed for superalloy materials used in jet aircraft engine applications through contracts¹⁴ and grants⁵⁶ that were sponsored by NASA Lewis Research Center. The results of this work were also adapted for use in reusable liquid propulsion applications such as the Space Shuttle Main Engines (SSME).⁷⁻⁸ Advanced propulsion systems currently being developed have resulted in the need for materials that have the capability of attaining higher operating temperatures. This has led to the development of TFTCs for ceramic components for jet engines.⁹⁻¹¹ The sensors have been fabricated on superalloys, ceramics, and composites. These have undergone furnace tests as well as tests in harsh environments, including gas turbine³ and hydrogen/oxygen engine environments under both low and high pressure conditions,⁷⁻⁸ a high heat flux facility,⁹⁻¹⁰ and a diesel engine environment.¹²

Given here is a description of the fabrication of the sensor on the range of materials. Preparation for testing in SSME simulated environments and the results will be provided in detail. Results of extensive high temperature testing of TFTCs on ceramic materials will be presented. These include thermocouple drift and durability as well as heat flux. Recent work with advanced materials will be briefly discussed. Finally, a TFTC application for diesel engine testing is mentioned.

2. SENSOR FABRICATION

Figure 1 shows a schematic diagram of the layers in the thin film thermocouple for both electrically insulating and

electrically conducting substrates. Materials onto which TFTCs have been fabricated include nickel based superalloys, ceramics, including silicon nitride, silicon carbide, mullite and aluminum oxide, ceramic based composites, and intermetallics. For superalloy materials, an MCrAIY coating is first deposited onto the substrate by electron beam vapor deposition or by sputter deposition. M can represent Fe, Co, Ni, or a combination of Co and Ni. With heat treatment, this coating forms a stable, adherent, electrically insulating aluminum oxide layer. An additional layer of aluminum oxide is sputter deposited to fill any pinholes or cracks that may be present in the grown oxide. Electrically conductive ceramic materials such as silicon carbide are thermally oxidized to form a stable, adherent silicon dioxide layer which is followed by a sputter deposited layer of aluminum oxide of the thickness needed to obtain the required insulation resistance. The thickness of the thermally grown and sputter deposited alumina layers are approximately $1-3 \mu m$ each.

The thermocouple legs are sputter deposited onto the electrical insulator; in the case of electrically insulating ceramic materials such as silicon nitride, the TFTC is deposited directly onto the surface. The legs are patterned with stenciled shadow masks. The thermocouple is platinum-13% rhodium vs platinum (Pt13Rh vs Pt) and is approximately 5 μ m thick. For those applications that require one, a protective overcoat of aluminum oxide is then sputtered deposited onto the sensor to a thickness of approximately 5-8 μ m thick. Platinum-13% rhodium and platinum leadwires of 75 μ m diameter are attached to the films via the parallel gap welding process.

The sensors are fabricated in the NASA Lewis Research Center's Thin Film Sensor Lab which is a Class 1000 Clean Room. The state-of-the-art facility includes several thin film deposition systems, wire bonding systems, etching systems, equipment for photolithography processes, and a surface profiler.

3. SPACE SHUTTLE MAIN ENGINE APPLICATION

3.1 Test of flat substrates in hydrogen/oxygen rocket test facility

Flat substrates of a nickel based superalloy have been fabricated with TFTCs for testing under conditions that approach that of the Space Shuttle Main Engine (SSME) high pressure fuel turbopump. The purpose of these tests was to determine the adherence of the films in a high temperature, hydrogen-oxygen environment. Flat substrates of two sizes were tested: $2.5 \times 5.1 \text{ cm}$ and $3.5 \times 12.5 \text{ cm}$. Figure 2 shows a $3.5 \times 12.5 \text{ cm}$ specimen with TFTCs prior to testing. These specimens were tested in a rocket lab facility located at the NASA Lewis Research Center (Figure 3) that approximates the thermal shock conditions of the SSME turbopump,¹³ but lacks the high pressure. Hydrogen-oxygen combustion gases ranging in temperatures from about 1000°C (1800°F) to about 2700°C (5000°F) are obtainable at combustion chamber pressures up to 4 MPa (600 psi). For these tests, the engine was operated at fuel-rich ratios resulting in temperatures of approximately 900-1100°C. The samples were mounted downstream of the exhaust nozzle (at atmosphere) and the tests were five seconds in duration. Figure 4 shows a specimen mounted in the test facility. The TFTCs proved to be highly adherent and durable by surviving the duration of testing through ten cycles. This was considered sufficient for this application since the operating time of the SSME turbopumps is of short duration.

3.2 Test of TFTC on SSME turbine blades

Thin film thermocouples have also been fabricated on SSME turbine blades for testing in a high temperature, high pressure turbine blade test facility located at NASA Marshall Space Flight Center. Figure 5 shows one of six turbine blades that were instrumented with TFTCs for testing in this facility. Each blade was fabricated with two TFTCs with the junctions and leadwire attachments located on the suction side airfoil and the shank area, respectively. The welds were coated with a ceramic adhesive which was subsequently covered with a metal strip to protect the weld area during testing. A chromel vs alumel wire thermocouple was attached to the shank on the pressure side of each blade. The blades were mounted into two wired blade holders, each of which held three blades, for testing in the turbine blade test (TBT) facility. Figure 6 shows the instrumented blades in a blade holder ready for testing.

Both sets of instrumented blades were simultaneously installed into the TBT facility for testing. The turbine blade test facility, shown schematically in Figure 7, simulates the SSME high pressure fuel turbopump environment with the combustion of oxygen and hydrogen at approximately 930°C (1700°F) and 16 MPa (2400 psi). The facility was instrumented with pressure transducers in the main, combustion and blade chambers. Wire thermocouples were located in the combustion and blade chambers. The facility operated through one heat up/cool down cycle and was shut down. The test cycle time

from ignition to shutdown was six seconds. It then operated through two consecutive cycles before final shut down.

Thermal output was provided by one thin film thermocouple during the first thermal cycle. The remaining TFTCs provided unstable data throughout the test. The data from the operating TFTC are shown in Figure 8a which also includes thermal output from a wire thermocouple on the blade shank and a facility wire thermocouple measuring gas temperature in blade chamber B. The facility wire thermocouple in blade chamber A, in which the operating TFTC was located, was not operating during these tests. Also shown in Figure 8a are the facility pressure data for the combustion chamber and blade chamber A. The TFTC output was unstable for approximately two seconds during the rapid rise in temperature and pressure before it settled down into a similar pattern as that for the wire thermocouple data. Figure 8b shows the temperature data limited to the range of -300 to 1100° C (-500 to 2000° F) to allow closer comparison between the TFTC data and the wire data. The TFTC gave a higher temperature than the shank thermocouple and the facility thermocouple located in the downstream blade chamber. It is reasonable to expect that the blade airfoil would reach the higher temperature before the shank area since the airfoil was positioned more directly in the gas flow whereas the shank was shielded by the holder and the other blades. The facility thermocouple was located downstream from the TFTC blade, therefore, it was exposed to the hot gases later than the TFTC blade. Visual inspection of the blades were intact. However, many of the leadwire attachments had failed and the fragile fine wires emerging from the ends of the respective cables were gone.

During the two subsequent firings, the operating TFTC demonstrated unstable behavior during the transient heat up and cool down portions of the test but gave the expected output during the remainder of the test (Figures 9a and 9b). At the close of the third cycle, many of the thin films were gone from the blade surfaces.

The cause of the instability of the TFTC output during the heat up and cool down portion of the test is not clear; however, it may have been due to poor contact between the thin film and the leadwires. Comparison of the TFTC output with the pressure data indicates unstable TFTC output during changes in pressure (Figure 8a and 9a). Thus, the vibrations of the facility during start up and shut down may have disrupted contact between the wire and the film during these times thereby resulting in the unstable output.

4. TFTCs ON CERAMIC MATERIALS

4.1 Experimental Procedure

The ceramic materials that were used in this program were silicon nitride, silicon carbide, aluminum oxide, and mullite. Table I lists the purity of these materials, the fabrication procedures, and some of the physical properties. The low purity of the silicon nitride is caused by the addition of 13 percent yttria and 3 percent alumina as densification agents for the sintering process. The surface finish is of particular interest because the thin film thermocouple is deposited on the surface of the ceramic materials. Figure 10 shows the test samples used in these experiments. The ceramic substrates were 15 cm long and 2.5 cm wide. The specimens were cemented to an aluminum oxide support plate using an alumina-based cement. The thin film thermocouple deposited on the sample was at least 12.5 cm long with film widths of about 3 mm. Pt13Rh/Pt leadwires were attached and routed through ceramic tubing to connectors.

The samples were tested in ceramic tube furnaces under steady state and thermal cycling modes. The steady-state tests were carried out in the temperature range from 1000 to 1500°C for times up to 150 hours at ambient pressures. The effect of different temperature gradients on drift rate patterns in thin film thermocouple circuits was evaluated. The lifetime goal of a sensor for advanced propulsion system applications is about 50 hours. For laboratory testing, longer lifetimes would be desirable. High heating rate tests were also performed using an arc lamp heat flux calibration facility.

4.2 Thermocouple drift

Thermocouple drift is defined as a change with time in the voltage versus temperature characteristic of a thermocouple. Suspected causes of thermocouple drift in these thin film thermocouples are oxidation of rhodium in the Pt13Rh thermoelement, foreign material at the thin film-to-leadwire connection, and chemical interaction or diffusion between the sensor and the substrate. Preferential oxidation of rhodium in the Pt13Rh leg of the thermocouple would cause a change in the Pt/Rh ratio in that leg and result in thermocouple drift. This oxidation rate increases as temperature increases, but

there is a conversion of the oxide back to elemental rhodium at a temperature of about 1000°C and above. Oxidation rate is also proportional to the surface area/volume ratio of the thin film sensor and leadwire geometry. The value of this ratio is at least four times greater for a 5 μ m thin film compared to a 75 μ m diameter leadwire. Finally, oxidation rate is dependent on the quantity of oxygen present in the gaseous environment surrounding the thermocouple. In these experiments, ambient air was the environment for all of the thermocouples.

The thin film-to-leadwire connection could be a source of thermocouple drift if a foreign material, such as a cement or paste, were introduced into the thermocouple circuit at this point to make the connection. But in these experiments, connections were made using the parallel-gap welding process, which eliminates this source of thermocouple drift. Thermocouple drift could originate at the substrate-sensor interface if a chemical reaction were to occur at this interface or if diffusion of material into or out of the thermocouple were to occur that would change the thermoelectric characteristics of either thermoelement.

Drift rate data for TFTCs on ceramic materials are shown in Figures 11 and 12 for steady state tests. The data are plotted as drift rate in degrees Celsius per hour against the steady state temperature, and each point represents the average drift rate of a steady state test. Also shown in each figure is the temperature gradient across the thin film portion of the thermocouple circuit. In Figure 11, the tests were performed on silicon nitride and silicon carbide substrates with a large temperature gradient of 500 to 600°C across the length of the thin film. With the hot junction of the TFTC at about 1000 to 1200°C in these tests, the leadwire end of the TFTC would be about 500 to 700°C; thus a large portion of the thin film would be in the temperature range where rhodium oxidation occurs. The result was a drift rate of about 0.5°C/hr. In these tests, the region of rhodium oxidation was easily seen by the formation of a dark deposit on the Pt13Rh thermoelement. Tests were also performed where the temperature gradient along the length of the thin film was only about 100°C (Figure 12); thus only the leadwire portion of the thermocouple circuit would be in the temperature range where rhodium oxidation occurs. This resulted in a drift rate of less than 0.2°C/hr between about 1000 to 1200°C for the data in Figure 12. There was no dark deposit on the Pt13Rh thin film thermoelement.

At temperatures greater than about 1250°C in Figure 12, drift rates rapidly increase as test temperature increases. It is suspected that a sensor-substrate interaction is beginning to occur in this temperature range, either because of a chemical reaction or a diffusion effect. It should also be noted that the drift rate is not the same for each substrate material in this higher temperature range. Selected drift rates of TFTCs and leadwires are tabulated in Figure 13. Figures 11 to 13 illustrate the complexity of thermocouple drift of TFTCs, which are in actuality composite thin film/leadwire thermocouple circuits. As these figures demonstrate, drift rate varies with: the absolute temperature level; the substrate material on which the TFTC is deposited; the temperature gradient distribution between the thin film and the leadwire portion of the circuit; and the film thickness and diameter of the thin films and leadwires, respectively.

4.3 TFTC durability

Thermal cycling of the test samples accompanied repeated steady-state tests of the same test sample up to a maximum of five cycles. No sensor failures occurred as a result of thermal cycling. Steady state testing occurred for various times up to a maximum of 150 hours. No sensor failures occurred as a result of total test time. The four ceramic materials used in this research program exhibited significantly different characteristics when exposed to high temperatures. The oxide ceramics, aluminum oxide and mullite, showed little visible surface deterioration when exposed to the entire temperature range of these experiments (1000 to 1500°C). The aluminum oxide was 99.6 percent pure and the mullite was a 98 percent pure mixture of aluminum oxide and silicon dioxide (60:38 ratio). Despite the lack of visible surface deterioration, the thin film sensors showed a significant increase in drift rate on these substrates above about 1300°C (Figure 12), indicating some form of sensor-substrate interaction. Very little degradation of the sensor structure occurred.

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

The silicon carbide was 99 percent pure and required an insulating layer to be superimposed between the sensor and

the substrate because it is an electrically conducting ceramic. The silicon carbide showed no visible deterioration during testing up to about 1250°C, but above 1300°C, the surface morphology began to change to a glassy appearance over a portion of its surface, and other nonuniformities in structure appeared. This change in surface morphology caused delamination of the thin film sensor material to begin.

Ceramic materials will degrade chemically by oxidation, vaporization, and interfacial reactions.¹⁴ For the oxide ceramics, such as aluminum oxide and mullite, vaporization is the major mechanism. In these experiments, this could lead to a slow, gradual deterioration of the bond between the sensor and the substrate. For the nonoxide ceramics, such as silicon nitride and silicon carbide, all three mechanisms are at work. The oxidation of the silicon nitride not only forms an interface at the oxide-substrate boundary, but can lead to a complicated structural interaction with yttria and alumina present as densification agents in the ceramic. This resulted in the rapid formation of an irregular oxide structure above 1250°C leading to sensor delamination. In the case of silicon carbide, the interfaces were deliberately formed by thermal oxidation and sputtered alumina to form the insulating layer for the sensor. Above 1250°C, the formation of a glassy layer and other irregular structure in the surface layers of the ceramic could be caused by interfacial reactions, phase change, or further oxidation. This leads to a deterioration in the sensor-substrate bond, and eventual delamination.

Two additional materials were tested for sensor durability: a higher purity silicon nitride containing only 4 percent densification agents and a lower purity silicon carbide (94 percent purity). The TFTC on the high purity silicon nitride, which had a 1.5 μ m thick alumina protective coating, was successfully tested to 1400°C with negligible physical degradation. A thermal stress failure of the platinum leg of the sensor occurred at 1414°C. The TFTC on the low purity silicon carbide bubbled and delaminated during testing as compared to the TFTC on the high purity silicon carbide, which had negligible physical degradation. This demonstrates that the higher purity of these materials reduced the amount of substrate surface oxidation and interfacial reactions that can contribute to sensor failure.

4.4 Heat flux calibration facility tests

Another aspect of sensor durability is the ability of the sensor to withstand high heating rates accompanied by rapid temperature excursions from room temperature to the maximum operating temperature of the sensors. The arc lamp heat flux calibration facility is capable of concentrating a high, known heat flux over a small, well-defined area. Lamp currents from 30 to 400 A are used to generate heat fluxes from about 0.1 to 5 MW/m² over a 1 x 4 cm area. Figure 14 shows a test piece with a TFTC deposited on a silicon nitride surface in such a way that the hot junction is at the center of the focal area of the lamp. A black coating was applied to a portion of the surface to increase the absorption of the radiant energy. A second TFTC was mounted on the back surface directly behind the front sensor. Figure 15 shows the temperature rise versus time for the hot-side TFTC for different lamp currents. Heating rates from about 2 to 2500°C/sec were generated in these tests. Silicon nitride and mullite were used. Maximum temperature was 1500°C and maximum ΔT across a ceramic was 560°C. No sensor failures occurred during these tests, and a single test piece was subjected to a maximum of 20 test cycles. Note that in these tests the total test time is measured in seconds or minutes rather than hours and therefore the ceramics suffered very little surface degradation.

5. TFTCs ON ADVANCED MATERIALS

A TFTC has also been fabricated and tested on a silicon nitride ceramic matrix composite.¹¹ The sensor was successfully tested at 1000°C for 50 hours. TFTCs are currently being fabricated for testing on these materials in gas engine environments. In addition, an intermetallic matrix material was fabricated with a TFTC and tested successfully to 1260°C. Drift rates were measured to be about 0.1°C/hr with about 100°C gradient across the thin film portion of the circuit. This rate is similar to those observed on ceramic substrates in this temperature range.

6. DIESEL ENGINE APPLICATION

A thin film thermocouple has also been used as part of a heat flux sensor that was tested in the harsh, high temperature environment of a ceramic-insulated, low-heat-rejection diesel engine.¹² The sensor probe assembly was developed to provide experimental validation of heat transfer and thermal analysis methodologies applicable to the insulated diesel engine concept. The thin film sensor was installed on an iron plug and performed reliably during 6 to 10 hours of repeated engine runs at indicated mean surface temperatures up to 680°C.

7. SUMMARY

Thin film thermocouples have proven to be applicable to a range of materials and applications. The sensors have been demonstrated on superalloys, ceramics, ceramic composites, and intermetallics. Data has been obtained in furnace testing, under high heat flux conditions, and in harsh engine environments.

8. ACKNOWLEDGMENTS

The authors acknowledge the invaluable assistance of Keith F. Taylor, Gerald A. Danzey, and Terrian V. Nowden for the fabrication of the test specimens for testing, James Green for operation of the LeRC Rocket Lab Facility, Philip Best and John Wiley for operation of the MSFC Turbine Blade Test Facility, and Curt H. Liebert, William T. Dedula, and George W. Readus, Jr. for operation of the heat flux calibration facility.

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Material	Fabrica- tion method	Surface finish, µm	Thick- ness, mm	Density, gm/cm ³	Thermal conductivity, W/m-K	Electrical resistivity, Ω-cm	Melting point, °C	TCE ^a α, °C ⁻¹ x10 ⁻⁶	Purity, percent
Silicon nitride	Sintered	0.5 to 0.75	6	3.28	30	1014	1900	4	84
Silicon carbide	Sintered	0.25 to 0.5	6	3.1	125	10	2700	4	99
Aluminum oxide	Tapecast	0.075 to 0.15	1.5	3.9	25	1014	2040	8	99.6
Mullite	Hot- pressed	0.25 to 0.5	4.5	3.6	4	1014	1700	10	98

TABLE 1. - Description of ceramic materials

^aTemperature coefficient of expansion.



(a) Silicon nitride or aluminum oxide (b) Silicon carbide (c) Superalloy Figure 1. - Schematic diagram of thin film thermocouples on superalloy and ceramic materials.



Figure 2. - Flat superalloy substrate with thin film thermocouple prior to testing in the hydrogen-oxygen rocket lab.



Figure 3. - Schematic of Lewis hydrogen-oxygen rocket lab facility.



Figure 4. - Flat specimen of TFTC in Lewis rocket lab facility. Arrow indicates direction of gas flow.



Figure 5. - TFTCs on SSME turbine blade.



Figure 6. - SSME turbine blades assembled in blade holder.



Figure 7. - Schematic of MSFC turbine blade tester.



Figure 8a. - TBT cycle 1: Temperature and pressure output. (Comb P = Combustion chamber pressure; Blade P = Blade chamber A pressure; Facility TC = Blade chamber B temperature)



Figure 8b. - TBT cycle 1: Temperature output limited to -300 to 1100°C.







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Figure 10. - Thin film thermocouples on ceramic materials for high temperature furnace tests.



Figure 12. - Drift rates of TFTCs on ceramic substrates with 100°C temperature gradient across thin film portion of thermocouple circuit.



Figure 14. - TFTC on silicon nitride substrate for arc lamp heat flux calibrator test.



Figure 11. - Drift rates of TFTCs on ceramic substrates with 500-600°C temperature gradient across thin film portion of thermocouple circuit.



Figure 13. - Drift rates of TFTCs and 75 μ m leadwires at selected temperature levels.



Figure 15. - Heating rate curves for TFTCs on silicon nitride substrate in arc lamp heat flux calibrator test.

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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AN	D DATES COVERED					
	August 1994	Te	chnical Memorandu	ım				
4. TITLE AND SUBTITLE			5. FUNDING NUMBER	RS				
Applications of Thin Film T								
6. AUTHOR(S) Lisa C. Martin and Raymon	WU-505-62-5	0						
7. PERFORMING ORGANIZATION NA	8. PERFORMING OR REPORT NUMBER	GANIZATION						
National Aeronautics and Sp Lewis Research Center Cleveland, Ohio 44135–31	E-9080							
9. SPONSORING/MONITORING AGE	10. SPONSORING/MC AGENCY REPOR	DNITORING T NUMBER						
National Aeronautics and S Washington, D.C. 20546–0	NASA TM-100	6714						
Prepared for the Conference sored by the National Aeror San Diego, California, July 12a. DISTRIBUTION/AVAILABILITY S Unclassified - Unlimited Subject Category 35	to-Optical Instrume nization code 2510, 12b. DISTRIBUTION C	entation Cospon- entation Engineers, (216) 433–6468.						
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14. SUBJECT TERMS Surface temperature measu	vs; High	15. NUMBER OF PAGES 14 16. PRICE CODE						
				A03				
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFIC OF ABSTRACT	ATION 20. LIMITA	HUN OF ABSTRACT				
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