NASA/TM—2002-211873

AIAA–2002–3731

A Multilayered Thin Film Insulator for Harsh Environments

John D. Wrbanek and Gustave C. Fralick
Glenn Research Center, Cleveland, Ohio

Charles A. Blaha
Akima Corporation, Brook Park, Ohio

A. Rachel Busfield
Rolls-Royce plc, Derby, United Kingdom

Valarie D. Thomas
Ohio Aerospace Institute, Brook Park, Ohio

September 2002

The NASA STI Program Office . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the Lead Center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized databases, organizing and publishing research results . . . even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI Program Home Page at ***http://www.sti.nasa.gov***

- E-mail your question via the Internet to help@sti.nasa.gov

- Fax your question to the NASA Access Help Desk at 301–621–0134

- Telephone the NASA Access Help Desk at 301–621–0390

- Write to:
  NASA Access Help Desk
  NASA Center for AeroSpace Information
  7121 Standard Drive
  Hanover, MD 21076

NASA/TM—2002-211873 AIAA–2002–3731

A Multilayered Thin Film Insulator for Harsh Environments

John D. Wrbanek and Gustave C. Fralick
Glenn Research Center, Cleveland, Ohio

Charles A. Blaha
Akima Corporation, Brook Park, Ohio

A. Rachel Busfield
Rolls-Royce plc, Derby, United Kingdom

Valarie D. Thomas
Ohio Aerospace Institute, Brook Park, Ohio

Prepared for the
38th Joint Propulsion Conference and Exhibit
cosponsored by AIAA, ASME, SAE, and ASEE
Indianapolis, Indiana, July 7–10, 2002

National Aeronautics and
Space Administration

Glenn Research Center

September 2002

Acknowledgments

The authors would like to acknowledge the assistance of Drago Androjna (Akima Corporation) and Dorothy Lukco (QSS Group, Inc.) for the EDAX and Auger analysis of the samples. This work was pursued under the Information Rich Test Instrumentation (IRTI) project, part of NASA's Advanced Space Transportation Program (ASTP). The authors would also like to acknowledge the support of the management of NASA Glenn Research Center, together with Rolls-Royce and Allison Advanced Development Company, for arranging this collaboration. Additional funding was provided by Rolls-Royce and the Royal Academy of Engineering.

This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

The Aerospace Propulsion and Power Program at NASA Glenn Research Center sponsored this work.

Available from

NASA Center for Aerospace Information
7121 Standard Drive
Hanover, MD 21076

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22100

Available electronically at http://gltrs.grc.nasa.gov

A MULTILAYERED THIN FILM INSULATOR FOR HARSH ENVIRONMENTS

John D. Wrbanek and Gustave C. Fralick
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Charles A. Blaha
Akima Corporation
Brook Park, Ohio 44142

A. Rachel Busfield
Rolls-Royce plc,
Derby DE24 8BJ, United Kingdom

Valarie D. Thomas
Ohio Aerospace Institute
Brook Park, Ohio 44142

Abstract

The status of work to develop a reliable high temperature dielectric thin film for use with thin film sensors is presented. The use of thin films to electrically insulate thin film sensors on engine components minimizes the intrusiveness of the sensor and allows a more accurate measurement of the environment. A variety of insulating films were investigated for preventing electrical shorting caused by insulator failure between the sensor and the component. By alternating layers of sputtered high temperature ceramics, a sequence of insulating layers was devised that prevents pinholes from forming completely through the insulator and maintains high electrical resistivity at high temperatures. The major technical challenge remaining is to optimise the fabrication of the insulator with respect to composition to achieve a reliable high temperature insulating film. Data from the testing of various potentially insulating thin film systems is presented and their application to thin film sensors is also discussed.

Introduction

As part of NASA Glenn Research Center's mission to develop critical technologies that enable safer, lighter, quieter, and more fuel efficient vehicles for aeronautics and space transportation, the Sensors and Electronics Technology Branch has an effort to develop thin film sensors for surface measurement in propulsion system research. The sensors include those for strain, temperature, heat flux and surface flow.

Rolls-Royce plc provides power systems for air and marine propulsion and industrial applications. The Instrumentation Engineering department is developing thin film sensor technologies to support the company's drive for designing and manufacturing systems having increased safety and durability while minimizing noise and emissions.

The use of thin film sensors has several advantages over wire or foil sensors. The thin film sensors do not require special machining of the components on which they are mounted, and, with thickness less than 10 μm, they are considerably thinner than wire or foils. The thin film sensors are thus much less disturbing to the operating environment, and have a minimal impact on the physical characteristics of the supporting component. Several thin film sensors have been tested up to 1100 °C in different engine conditions at NASA Glenn Research Center.[1,2]

Fabricating metal sensors directly on metal components requires the use of an electrically insulating layer between the component and the sensor. Flame-sprayed insulators provide good insulating capability, but the thickness of the coatings (300 μm and greater due to the coating porosity) lessens the advantage of the thin film sensor. NASA Glenn Research Center has been examining the use of thin film insulators as an alternative. One process developed utilizes a 125 μm thick NiCoCrAlY base coat on which alumina can be thermally grown and vapor-deposited.[3] Rolls-Royce thin film sensors use a reactively-sputtered layer of alumina to provide the electrical insulation.

The application of alternative thin film dielectric insulators has been examined to further minimize the insulation thickness. However, ceramics lose their electrical insulation exponentially with increasing temperatures. Short circuit paths at grain boundaries and film defects can further reduce the insulation properties from the bulk values at high temperatures.

To further this research, NASA Glenn Research Center and Rolls-Royce have pursued a joint investigation of utilizing multilayered thin film dielectrics as a reliable insulator for use in harsh environments. The use of a multilayered scheme is thought to be promising for the fabrication of electrically insulating thin films. A major cause of conduction in thin film dielectrics is the presence of defects, such as pinholes, that propagate clear through the film to the underlining substrate surface. By alternating the insulating material, a new growth pattern would eliminate the previous one. Thus, direct pathways for conduction to the substrate are eliminated. The requirements for such an insulator are driven by the environments of aerospace applications, which currently require insulation of 100 kΩ or better to 1100 °C in oxidizing conditions.

Sample Preparation

The film depositions were conducted in the Microsystems Fabrication Cleanroom facility at NASA Glenn Research Center. Several samples of multi-layered insulators were generated using alumina and stainless steel shims as the test substrates. The alumina substrates provide readily available test beds that remain intact for the severe heating required of these films, up to 1700 °C if necessary. The stainless steel substrates can be annealed to 1000 °C, and the thermal expansion of the steel should produce stresses similar to those that the film would experience on an engine component at that temperature.

After surfactant cleaning, the test shims were rinsed in acetone and then methanol. A schematic of the typical layer sequence of the test samples is illustrated in Figure 1. A layer of platinum 1 μm thick was sputter deposited over the shims. On the alumina substrates, the platinum base coat insured the detection of a conduction path if one were to exist in the insulator. On the stainless steel substrates, the platinum base coat assisted in the bonding of the dielectrics to the metal. The dielectrics were then deposited with a total thickness of 5 μm. On top of the dielectric, several 4.75 mm diameter platinum pads were sputter deposited to simulate thin film sensors. The pads were deposited 5 μm thick to insure that any cracks or pinholes were sufficiently filled to reveal any conduction paths. A room temperature resistance check was then performed to verify insulating capability of the sample.

Lead wires were connected to the platinum pads on the insulating samples to afford high temperature testing in an annealing oven. The platinum wires were parallel-gap welded to the pads, which were then soldered to leads outside of the test oven. The oven was ramped at approximately 450 °C/hour while the resistance between the pads and the temperature measured by a type K thermocouple near the sample was monitored. The maximum resistance readable was 100 MΩ. One cycle was performed to stabilize the sample, and the data from the second ramp was used for insulator evaluation.

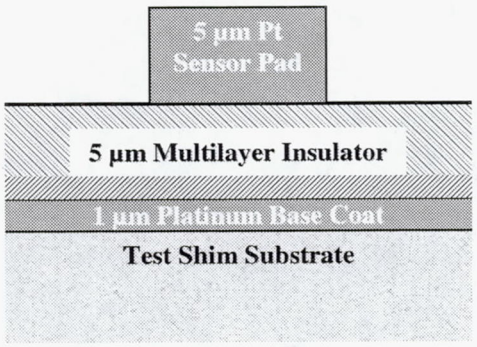

Figure 1.—Schematic of Insulator Test Sample

Analysis

A lettering scheme was used to identify specific samples of the study. The results of our sample tests are shown in Table 1. The runs were lettered as they were planned, and, as failures and successes dictated, some runs were skipped. The first three runs of samples set the baseline for the work.

The samples from run A had a single layer of 5 μm alumina (Al_2O_3) sputter deposited at room temperature. The samples indicated a short at room temperature, verifying the need for a more complex insulating thin film structure to achieve the required insulating capabilities for a 5 μm film.

The next samples (run B) tested used an underlying layer of 1 μm thick silica (SiO_2) sputter deposited, with an upper layer of 4 μm alumina sputter deposited at room temperature. These samples showed insulating properties at high temperature, with the sample on stainless steel several orders of magnitude more conducting than that on alumina. Both samples showed significant cracking after heat treatment. The samples

also showed a severe hysteresis in their resistance curves during the heat cycle. Despite the poor insulation properties, the run validated the concept of using a multilayer structure to eliminate conduction paths to the substrate that caused the short in run A samples.

Samples in run C were composed of a bottom layer of 2 μm alumina, followed by 1 μm of silica, and then covered with 2 μm of alumina sputter deposited on stainless steel shims. These samples showed properties similar to those of the run B samples on stainless steel, but with more severe cracking of the surface. Figure 2 shows the cracking seen on a typical sample from run C on stainless steel.

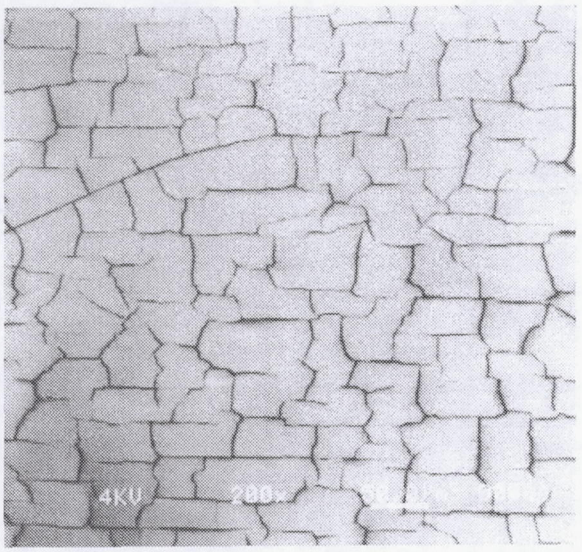

Figure 2.—Cracking on the surface of sample from Run C (Al_2O_3-SiO_2-Al_2O_3 Multilayer) after heat cycling to 500 °C. The width of the image is approximately 525 μm.

Several samples were prepared to further investigate the cause of the cracking. EDAX analysis of an annealed silica sample revealed that chromium and iron from the steel were migrating into the cracks formed in the films when heated. The deposition of the alumina by e-beam vaporization at 800 °C resulted in room temperature shorts. Surface preparation was altered to smooth the stainless steel by H_2SO_4/H_2O_2 cleaning. However, the poor film adhesion to the substrate surface and the high conductivity of the resulting film terminated this path.

Two alternatives to using silica as an underlying layer were tested and the results were promising. Run R used 1 μm of sputtered chromium carbide followed by 4 μm of e-beamed alumina, and run S used 1 μm of sputtered yttria-stabilized zirconia followed by 4 μm of e-beamed alumina. The platinum base coat was not deposited for run R since the chromium carbide proved to be an excellent bond coat between chromium-containing metals and oxide ceramics. Figure 3 shows the pinhole-free surface of a run R sample after heat-treating to 800 °C on a steel substrate. The temperature limit of both insulators for 100 kΩ of resistance appears to be about 1000 °C for the 5 μm of material deposited. The zirconia-alumina insulator was deposited on a steel substrate in run T, but the alumina failed before any data could be gathered.

The data from the temperature ramps of runs B, C, R, and S are plotted in figure 4. To give a quantitative comparison of the insulators, the data were fit to exponential decay curves. The results of the fittings are given in table 2, which gives the estimated 0°C values, decay constants, minimum use temperatures and uncertainties.

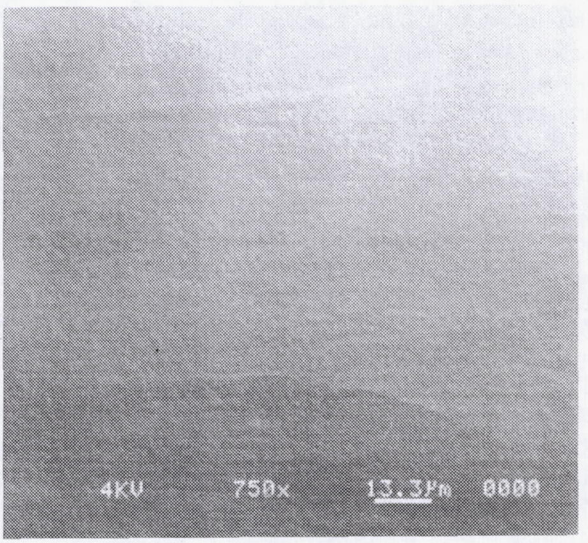

Figure 3.—Pinhole-free surface of sample from Run R (Cr_3C_2-Al_2O_3 Multilayer) after heat cycling to 800 °C. The width of the image is approximately 140 μm.

Conclusions

The results of this study indicate that the CTE mismatch between silica and metals inhibits its use as an effective thin film insulating layer or under layer for alumina. The CTE's of various materials used in these tests are given in the bulk properties listing in table 3. It is clear from these tests that a silica under layer does not expand freely enough to be used in a thin film

multilayer insulator sequence on metals, particularly when e-beam depositing the alumina at 800 °C. Even following a 500 °C annealing cycle, the silica cracking is sufficient to permit chromium and iron migration from the steel into the thin film insulation. In fact, under the 800 °C e-beam deposition temperatures of the alumina, the CTE mismatch between the steel and the silica is sufficient to cause cracking in the insulating film and adhesion failures.

The slightly lower CTE of yttria-stabilized zirconia may account for the stresses observed in the alumina thin film when the layers were applied to the steel substrate in run T. It should be noted that since most engine components are "super-alloys" similar to Hastelloy or Inconel with CTE's between 10 and 13 ppm/°C,[4] the zirconia-alumina multilayer may be applicable for use on those materials.

The success of run R is attributed to the graded interface between the chromium carbide and the chromium-containing metal. The intimate contact is believed to induce chemical-level bonding between the two materials. Thus, adhesion is promoted even at high temperatures. Further, the CTE of chromium carbide is intermediate between the alumina and the steel, thus grading the thermal stresses that would be produced during heating. Systematic Auger analysis of the chrome carbide chemistry is ongoing.

Ultimately, the goal is to use the multilayer insulator on a real engine component. The next challenge in this investigation is to optimize the chromium carbide-alumina multilayer on nickel-based "super-alloy" test shims for use on these engine components. Testing of the zirconia-alumina multilayer on these test shims may also give more insight on the thin film chemistry of these multilayer insulators.

References

1. J.F. Lei, L.C. Martin, H.A. Will, "Advances in Thin Film Sensor Technologies for Engine Applications," NASA TM–107418, *Turbo Expo '97*, Orlando, FL, June 2–5, 1997.

2. L.C. Martin, J.D. Wrbanek, G.C. Fralick, "Thin Film Sensors for Surface Measurements," NASA TM—2001–211149, *19th International Congress on Instrumentation in Aerospace Simulation Facilities*, Cleveland, OH, August 27–30, 2001.

3. L.C. Martin, G.C. Fralick, K.F. Taylor, "Advances in Thin Film Thermocouple Durability Under High Temperature and Pressure Testing Conditions," NASA TM—1999–208812, January 1999.

4. CRC Handbook of Chemistry and Physics, edited by R.C. Weast and M.J. Astle, CRC Press (1982).

5. S.W Bradstreet, "Oxide Ceramics," High-Temperature Materials and Technology, edited by I.E. Campbell and E.M. Sherwood, *The Electrochemical Society Series*, John Wiley & Sons, New York (1967), p. 235–303.

6. J. H. Westbrook and E.R. Stover, "Carbides for High-Temperature Materials," High-Temperature Materials and Technology, edited by I.E. Campbell and E.M. Sherwood, *The Electrochemical Society Series*, John Wiley & Sons, New York (1967), p. 312–348.

TABLE 1.—RUNS AND MULTILAYER SAMPLES GENERATED IN THIS EFFORT

Complete resistance data for runs B, C, R and S are plotted in Figure 4.

Run	Substrates	Preparation	Multilayer	Result
A	Alumina & SS Shims	Silica Polish	5 μm Al_2O_3 sputter deposited	Short @ RT
B	Alumina & SS Shims	Silica Polish	1 μm SiO_2 – 4 μm Al_2O_3 sputter deposited	Film Cracks after 500 °C Heat Treatment. Alumina Shim: 100 MΩ @ 720 °C, 1.78 MΩ @ 1000 °C SS Shim: 56 MΩ @ 41 °C, 1 kΩ @ 440 °C
C	SS	Silica Polish	2 μm Al_2O_3 – 1 μm SiO_2 – 2 μm Al_2O_3 sputter deposited	Film Cracks after 500 °C Heat Treatment 100 MΩ @ 17°C, 2 kΩ @ 500 °C
J	SS	Silica Polish	1 μm SiO_2 sputter deposited	EDAX after 800 °C anneal indicates substantial Cr & Fe migrating from the SS into the cracks
M & N	SS	Silica Polish	1 μm SiO_2 – 4 μm Al_2O_3 e-beam deposited	Short @ RT
P & Q	SS	H_2SO_4/H_2O_2 Cleaning	1 μm SiO_2 – 4 μm Al_2O_3 e-beam deposited	Film Adhesion Failures Short @ RT
R	SS	H_2SO_4/H_2O_2 Cleaning	1 μm CrC – 4 μm Al_2O_3 e-beam deposited	Good Insulator 84 MΩ @ 690 °C, 20 MΩ @ 750 °C
S	Alumina	Silica Polish	1 μm ZrO_2/Y_2O_3 – 4 μm Al_2O_3 e-beam deposited	Good Insulator 50 MΩ @ 690 °C, 17 MΩ @ 750 °C, 1.8 MΩ @ 900 °C
T	SS	Silica Polish	1 μm ZrO_2/Y_2O_3 – 4 μm Al_2O_3 e-beam deposited	Film Adhesion Failure

TABLE 2.—ESTIMATES OF THE THERMAL PROPERTIES OF SELECTED MULTILAYER INSULATORS

($R = R_0 * e^{-T/\theta}$ fit to Figure 4 data)

Run	Multilayer	Substrate	Resistance @ 0°C (R_0)	Decay Constant (θ)	Max Use Temp	±
B	SiO_2-Al_2O_3	Al_2O_3	3.85E+6 MΩ	65°C	1140 °C	42%
B	SiO_2-Al_2O_3	SS	184 MΩ	34°C	260 °C	17%
C	Al_2O_3-SiO_2-Al_2O_3	SS	164 MΩ	34°C	250 °C	1%
R	Cr_3C_2-Al_2O_3	SS	1.91E+9 MΩ	41°C	965 °C	1%
S	ZrO_2-Al_2O_3	Al_2O_3	2.18E+6 MΩ	64°C	1080 °C	2%

TABLE 3.—BULK PROPERTIES OF MATERIAL USED IN THIS STUDY [4,5,6]

Material	Density (g/cm^3)	Elastic Modulus (10^6 psi)	Coefficient of Thermal Expansion (CTE)	Resistivity (Ω-cm) @ 1100°C
Stainless Steel (SS) [4]	8.02	28	17 ppm/°C	$70x10^{-6}$
Alumina (Al_2O_3) [5]	3.65	56	8 ppm/°C	$1x10^6$
Silica (SiO_2) [4,5]	2.32	10	0.55 ppm/°C	$0.5x10^6$
Chromium Carbide (Cr_3C_2) [6]	6.7	>50	10 ppm/°C	$80x10^{-6}$
Yttria-stabilized Zirconia (ZrO_2/Y_2O_3) [5]	6.27	27	10 ppm/°C	50

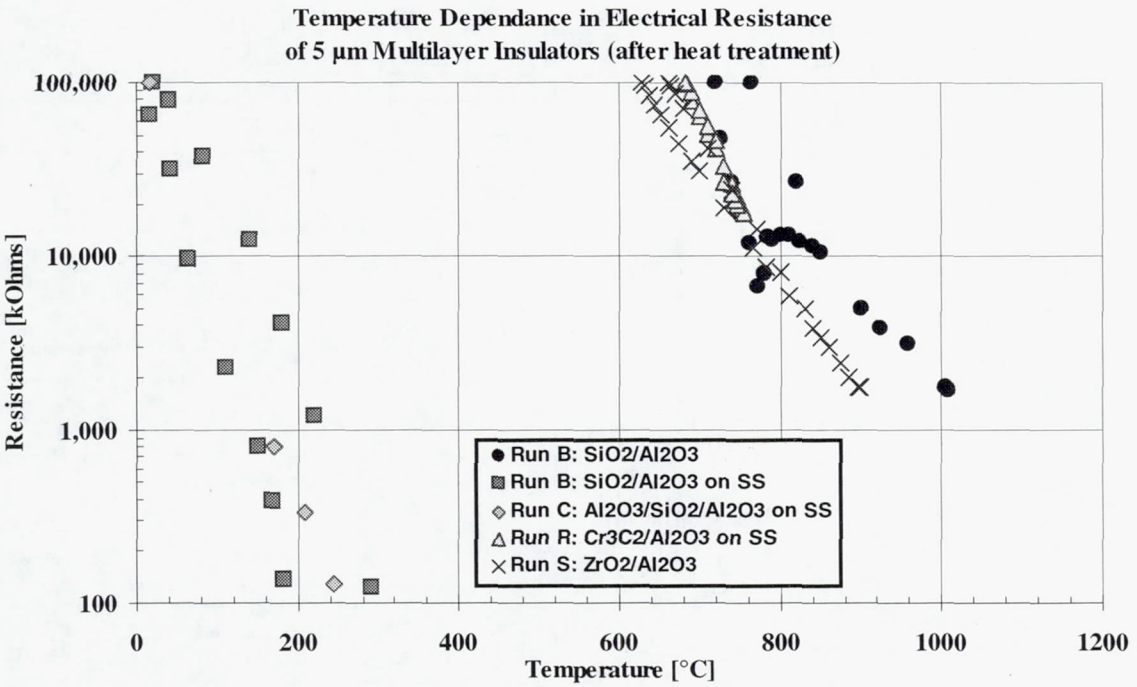

Figure 4.—Temperature dependence on electrical resistance for samples from runs B, C, R, & S. Note the large hysteresis shown by samples from Run B.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	September 2002	Technical Memorandum

4. TITLE AND SUBTITLE

A Multilayered Thin Film Insulator for Harsh Environments

5. FUNDING NUMBERS

WU–708–73–51–00

6. AUTHOR(S)

John D. Wrbanek, Gustave C. Fralick, Charles A. Blaha, A. Rachel Busfield, and Valarie D. Thomas

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
Cleveland, Ohio 44135–3191

8. PERFORMING ORGANIZATION REPORT NUMBER

E–13555

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NASA TM—2002-211873
AIAA–2002–3731

11. SUPPLEMENTARY NOTES

Prepared for the 38th Joint Propulsion Conference and Exhibit cosponsored by AIAA, ASME, SAE, and ASEE, Indianapolis, Indiana, July 7–10, 2002. John D. Wrbanek and Gustave C. Fralick, NASA Glenn Research Center; Charles A. Blaha, Akima Corporation, Brook Park, Ohio 44142; A. Rachel Busfield, Rolls-Royce plc, Derby DE24 8BJ, United Kingdom; and Valarie D. Thomas, Ohio Aerospace Institute, Brookpark, Ohio 44142. Responsible person, John D. Wrbanek, organization code 5510, 216–433–2077.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified - Unlimited
Subject Category: 35 Distribution: Nonstandard

Available electronically at http://gltrs.grc.nasa.gov

This publication is available from the NASA Center for AeroSpace Information, 301–621–0390.

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

The status of work to develop a reliable high temperature dielectric thin film for use with thin film sensors is presented. The use of thin films to electrically insulate thin film sensors on engine components minimizes the intrusiveness of the sensor and allows a more accurate measurement of the environment. A variety of insulating films were investigated for preventing electrical shorting caused by insulator failure between the sensor and the component. By alternating layers of sputtered high temperature ceramics, a sequence of insulating layers was devised that prevents pinholes from forming completely through the insulator and maintains high electrical resistivity at high temperatures. The major technical challenge remaining is to optimize the fabrication of the insulator with respect to composition to achieve a reliable high temperature insulating film. Data from the testing of various potentially insulating thin film systems is presented and their application to thin film sensors is also discussed.

14. SUBJECT TERMS

Insulator; High temperature; Thin film sensor; Instrumentation; Harsh environment

15. NUMBER OF PAGES

12

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	