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Thin Film Ceramic Strain Sensor Development for Harsh Environments

Interim Report on Identification of Candidate Thin Film Ceramics to Test for Viability for Static Strain Sensor Development

John D. Wrbanek, Gustave C. Fralick, and Gary W. Hunter
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December 2006

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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.

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Executive Summary

Instrumentation technologies to advance knowledge in fundamental aeronautics and develop technologies for safer, lighter, quieter, and more fuel efficient aircraft are being developed by the National Aeronautics and Space Administration (NASA) in support of its mission to pioneer the future in space exploration, scientific discovery, and aeronautics research. The Sensors and Electronics Branch of NASA Glenn Research Center (GRC) has an in-house 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 which will enable critical vehicle health monitoring and characterization of components of future space and air vehicles.

The use of sensors made of thin films has several advantages over wire or foil sensors. Thin film sensors do not require special machining of the components on which they are mounted, and, with thicknesses less than 10 μm, they are considerably thinner than wire or foils. Thin film sensors are thus much less disturbing to the operating environment, and have a minimal impact on the physical characteristics of the supporting components.

The need to consider ceramic sensing elements is brought about by the temperature limits of metal thin film sensors in propulsion system applications. In order to have a more passive method of negating changes of resistance due to temperature, an effort is underway at NASA GRC to develop high temperature thin film ceramic static strain gauges for application in turbine engines, specifically in the fan and compressor modules on blades. Other applications include on aircraft hot section structures and on thermal protection systems.

The near-term interim goal of this research effort was to identify candidate thin film ceramic sensor materials to test for viability and provide a list of possible thin film ceramic sensor materials and corresponding properties to test for viability. This goal was achieved by a thorough literature search for ceramics that have the potential for application as high temperature thin film strain gauges, reviewing potential candidate materials for chemical and physical compatibility with NASA GRC's microfabrication procedures and substrates.

Based on results of research given in this report, further efforts will focus on the application of zirconium nitride, titanium nitride, and titanium diboride strain gauges, doped with gold. The microfabricated thin film strain sensors will be developed using MEMS-based fabrication techniques in a class 1000 clean room at NASA GRC using physical vapor deposition and photolithography technologies. Technical metrics such as sensitivity, stability, repeatability, interference and durability will determine whether the sensor is ready for testbed qualifications for qualifying parts or structural elements and verify numerical codes, towards application in turbine engines.

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1. Introduction

1.1 Overview

To advance knowledge in fundamental aeronautics and develop technologies for safer, lighter, quieter, and more fuel efficient aircraft, instrumentation technologies are being developed by the National Aeronautics and Space Administration (NASA) in support of its mission to pioneer the future in space exploration, scientific discovery, and aeronautics research. These technologies also enable the capabilities for long duration, more distant human and robotic missions for the Vision for Space Exploration.

The Sensors and Electronics Branch of NASA Glenn Research Center (GRC) has an in-house 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 which will enable critical vehicle health monitoring and characterization of components of future space and air vehicles (ref. 1).

The use of sensors made of thin films has several advantages over wire or foil sensors. Thin film sensors do not require special machining of the components on which they are mounted, and, with thicknesses less than 10 μm, they are considerably thinner than wire or foils. Thin film sensors are thus much less disturbing to the operating environment, and have a minimal impact on the physical characteristics of the supporting components.

1.2 Technology Description

The need to consider ceramic sensing elements is brought about by the temperature limits of metal thin film sensors in propulsion system applications. Longer-term stability of thin film sensors made of noble metals has been demonstrated at 1100 °C for 25 hours (ref. 2). The capability for thin film sensors to operate in 1500 °C environments for 25 hours or more is considered critical for ceramic turbine engine development (refs. 3 and 4). For future space transportation vehicles, temperatures of propulsion system components of at least 1650 to 3000 °C are expected (ref. 5).

Since 1991, there have been many investigations into the application of ceramic thin films for use as high temperature thin film strain gauges. One important property to determine the appropriateness of a material's application as a strain gauge is its gauge factor. The gauge factor (γ) of the strain gauge relates the sensitivity of the gauge to strain ($\varepsilon = \delta l/l$), as shown in equation (1).

$$\frac{\delta R}{R} = \gamma \frac{\delta l}{l} = \gamma \varepsilon \tag{1}$$

The apparent strain sensitivity to temperature ($\delta\varepsilon_a/\delta T$) is the temperature coefficient of resistance (TCR) divided by the gauge factor plus the difference in the substrate and the gauge material's coefficient of thermal expansion (CTE), as shown in equation (2). The difference in the CTE's is expected to be less than +5 ppm/°C based on the materials that we are exploring, and this will be left as an uncertainty in our apparent strain calculation.

$$\frac{\delta \varepsilon_a}{\delta T} = \frac{\mathrm{TCR}}{\gamma} + \Delta\mathrm{CTE} \tag{2}$$

A limitation of thin films used as sensors to measure strain is that their resistance changes as the temperature changes. This "apparent strain" can be falsely interpreted as actual strain on the component being monitored. For static strain applications for use on gas turbine engines, the current required accuracy is ±200 μin/in (με), approximately ±10 percent of full scale, with the goal of ±1 percent accuracy (ref. 6). The thin film palladium-chromium alloy strain gauge, developed at NASA GRC for high temperature strain measurement application, is stable to 1100 °C, but has a TCR of +135 ppm/°C and an apparent strain sensitivity ($\delta\varepsilon_a/\delta T$) of +85 με/°C, requiring temperature compensation for high temperature static strain measurements (refs. 2 and 6) Currently, this compensation is in the form of setting a "ballast" potentiometer in a bridge to perform first order elimination of the apparent strain at a particular temperature, but deviations from this matched temperature results in measured apparent strain (ref. 7).

A thin film strain sensor with thermal stability over a wide range of temperatures would allow high temperature static measurements as a more passive method of eliminating apparent strain without the need for a completion bridge. Ultimately, the goal is to be able to achieve the desired ±20 με accuracy of measured applied static strain being no less than 0.1 percent of a total strain measurement (= applied + apparent + drift strain), or ±20,000 με. The apparent strain limit of less than ±20,000 με limits the temperature sensitivity to be less than ±20 με/°C over the current temperature range. As this goal is approached in research efforts, the drift strain ("creep") will also need to be considered as part of the total strain measurement.

1.3 GRC Research Effort Objective

The objective of this task is to develop high temperature thin film ceramic sensors to allow the non-intrusive in-situ measurement of static strain characteristics of engine components at high temperatures. These sensors will be applied using Micro-Electro-Mechanical Systems (MEMS) based sensor processing technology to allow miniaturized instrumentation and enhance sensor reproducibility and redundancy. This group has a deep history in developing sensor systems which can be applied directly on the component using MEMS-based techniques. Due to their thin film nature, the sensors have minimal affect on the operation of the part or the surrounding air flow. The thin film sensors can be embedded directly on the surface of components and thus provide detailed information on surface conditions.

1.4 Milestones and Deliverables

The schedule of milestones and deliverables are given in table 1. The dates of the milestones are set by resources available for the work involved. Fabrication process optimization for ceramic sensors is limited by the ongoing institutional construction in the same building as NASA GRC's micofabrication facility and competition for resources with other projects utilizing the fabrication facility.

TABLE 1.—MILESTONES AND DELIVERABLES

Date	Milestone	Deliverable
June 2006	Identify candidate thin film ceramic sensor materials to test for viability	List of possible thin film ceramic sensor materials and corresponding properties to test for viability
September 2006	Preliminary testing of candidate thin film materials for high temperature strain measurement applications	Preliminary data on temperature and strain characteristics
May 2007	Identify viable thin film ceramic sensors	Demonstrate viable thin film ceramic sensors in low temperature tests
September 2007	Preliminary high temperature cycling tests of viable thin film ceramic sensors	Preliminary data on temperature and strain characteristics
September 2008	Identify thin film ceramic sensor viability for component qualifications	Demonstrate thin film ceramic sensors under high temperature cycling test

1.5 Organizational Approach

NASA GRC will lead the MEMS sensor system development, fabrication and characterization, interacting with outside centers and contractors as necessary. Responsible participants:

Task Manager: Gustave Fralick (GRC), (216)–433–3645, Gustave.C.Fralick@nasa.gov
Sensor Design and Testing: Gustave Fralick and John Wrbanek (GRC)
Thin Film Sensor Fabrication (TFOME Service Pool): Charles Blaha (Jacobs Sverdrup), José Gonzalez (Gilcrest Electric) and Kimala Laster (Sierra Lobo)

2. Research Efforts and Results

2.1 Overview

The near-term interim goal of the research effort was to identify candidate thin film ceramic sensor materials to test for viability and provide a list of possible thin film ceramic sensor materials and corresponding properties to test for viability. This goal was achieved by a thorough literature search for ceramics that have the potential for application as high temperature thin film strain gauges, reviewing potential candidate materials for chemical and physical compatibility with our microfabrication procedures and substrates.

2.2 Potential Ceramics as Thin Film Sensors

A summary of notable materials that have been applied by a variety of investigators as high temperature thin film strain gauges for use over 1100 °C (2000 °F) is given in table 2. Using the apparent strain sensitivity as a guide, aluminum nitride (AlN), indium-tin oxide (ITO), titanium boride (TiB_2), and doped and undoped tantalum nitride (TaN) and titanium nitride (TiN) are obvious candidates for use for static strain measurements based on previous work reported in table 2. For zirconium nitride (ZrN) and hafnium nitride (HfN), no gauge factors were reported, but are attractive since the TCR can be modified through the reactive sputtering process. Also attractive for static strain measurements are materials that can have TCR modified that have not yet been tested as to their applicability as strain gauges.

TABLE 2.—REVIEW OF HIGH TEMPERATURE THIN FILM STRAIN GAUGE APPLICATIONS

Gauge material	TCR (ppm/°C)	Gauge factor (γ) ($\delta R/R/\varepsilon$)	Apparent strain sensitivity (με/°C)	Maximum use temperature	Notes	Reference
Ni-20%Cr	290	2.5	116	700 °C	COTS standard	8, 9
Pd-13%Cr	135	2 to1.4	85	1100 °C	NASA standard	2
AlN	–1281 to 109	3.72 to 15	–344 to 29	>1100 °C	Al reacted with N	10
ITO	–469 to 230	–6.5 to –11.4	–35 to 72	>1100 °C	Oxygen doping	11
Al:ITO	–1200	8	–150	1280 °C	Aluminum doping	12
TiN	–143 to 588	---------------	------------	<2930 °C	Ti reacted with N	13
TiB_2	–50	1.4	–36	<2970 °C?	Nitrogen doping w/ no effect	14
TiB_2	36	---------------	------------	<2970 °C	Ti reacted with B_2H_6	13
ZrN	184 to 275	---------------	------------	<2980 °C	Zr reacted with N	13
TaON	–290	3.5	–83	<3000 °C?	Ta reacted w/N; 1% Oxygen	15
TaN	–80	3.5	–23	<3090 °C?	Ta reacted with N	16
Cu:TaN	–800 to 200	2.3 to 5.1	–348 to 87	<3090 °C?	Ta reacted w/N; Cu doping	17
HfN	90	---------------	------------	<3310 °C	From COTS target	13
HfC	–426 to –110	---------------	------------	<3890 °C	Hf reacted with C_2H_3	13

Table 3 gives a summary of ceramic mixes used in modifying the TCR in bulk, thick or thin films. Ruthenium oxide and derivatives are given for completeness sake, and sublime at too low of temperature for use as a high temperature strain gauge. Based on our experience, bulk material that can survive to ~1650 °C (3000 °C) can survive in film form to ~1100 °C (2000 °F). Doped titanium oxide (TiO), zinc oxide (ZnO), antinomy-tin oxide (ATO) and chromium-silicon oxide (CrSiO) films are attractive as static strain candidates based on the table 3 summary.

TABLE 3.—CERAMIC MIXES USED TO MODIFY TCR IN BULK, THICK OR THIN FILMS

Ceramic	Base	Dopant(s)	Common name	Melting point	Reference
RuO2	Ru	O	Ruthenium Oxide	1200 °C (s)	18
M:RuO	RuO_2	Au, Pt, Pd	Ruthenium Oxide Cermet	1200 °C (s)	19
CuO:RuO	RuO_2	CuO	Ruthenium Cupric Oxide	1200 °C (s)	20
WAO	WO_3	AlOx	Tungsten Aluminum Oxide	1470 °C	21, 22
TiO	Ti	O	Titanium Oxide	1750 °C	23
ZAO	ZnO	AlOx	Zinc Aluminum Oxide	1800 °C?(s)	24
ZAON	ZnO	Al, N	Zinc Aluminum Oxynitride	1800 °C?(s)	25
CrSiO	Cr	Si,O	Chromium Silicon Oxide	1800 °C?	26
ATO	SnO	SbO	Antimony Tin Oxide	1900 °C?	27
N:ATO	ATO	N	Nitrogen doped ATO	1900 °C?	28
ITO	InO	Sn,O	Indium Tin Oxide	1900 °C	11
GITO	ITO	GaOx	Gallium-ITO	1900 °C	29
Al:ITO	ITO	AlOx	Aluminum doped ITO	1900 °C	12
CrTiN	Ti	Cr, N	Chromium Titanium Nitride	2900 °C?	26
AlN	Al	N	Aluminum Nitride	3000 °C	10
AuTaO	Ta	Au,O	Gold-Tantalum Oxide	3000 °C?	26
TaN	Ta	N	Tantalum Nitride	3090 °C	30, 31, 32
TaON	Ta	O,N	Tantalum Oxynitride	3090 °C?	33

2.3 Chemical and Physical Compatibility

The candidate ceramic thin films given above have the ability to be reactively sputtered (except for TiB_2) or co-sputtered to various doping levels as static strain gauges at NASA GRC. Because of the reactive nature of our fabrication processes, and the harsh chemical environments that the gauges are expected to be exposed to in operation, consideration of film hazards and stability is a concern. Table 4 gives the results of a search of various components that would be expected to be present in the candidate materials in The Merck Index (ref. 34), a reference of chemicals and chemical compatibility. Unfortunately, no quantitative reactivity is given in the index, and the description "attacked" in the text is assumed to be reactive or very reactive. No information was given on CrSi, TaN, TiN, TiB_2, or ZrN by Merck, but an extensive search indicates that these materials are believed to have substantial chemical resistance and do not pose a health hazard. {Aside: Conflicting MSDS information was found on TiB_2, with one considering it "an industrial poison" and another as "not a toxic hazard." Currently, boron compounds are considered an irritant to the eyes and throat, borates (derivatives of HBO_2) are toxic to insects but not mammals, and boranes (borohydrides of $(BH)_nH_4$) are deadly (refs. 35 and 36) As a safety precaution, we will not reactively sputter borides to prevent the formation of borates.}

TABLE 4.—REACTIVITY OF POTENTIAL MATERIALS (REF. 34)

Material	Hazards	Attacked by
Al	Powder Flammable	HCl, H_2SO_4, KOH, NaOH
AlN		water
Au		A.R.
Cr	Skin and Nasal Irritant	HCl, H_2SO_4
Cu		HNO_3, hot H_2SO_4, HBr
In_2O_3		hot acids
Pd		HNO_3, H_2SO_4, HCl
Sb_2O_3	Skin and Nasal Irritant	HCl, H_2SO_4, HNO_3
Si		HF
SnO_2		hot acids
Ta		HF
Ti	Flammable	hot HNO_3 (oxidizing)
TiO2		hot H_2SO_4, HF
W	Powder Pyrophoric	Steam (oxidizing), HNO_3, A.R.
Zn	Flammable, Toxic Fumes	HNO_3
ZnO	Toxic Fumes	KOH, mineral acids
Zr	Flammable	HF, *Aqua Regia* (A.R.), hot KOH

The ability to pattern fine line sensors would require the use of NASA GRC's in-house sacrificial-copper lift-off process, limiting for consideration non-toxic materials not reactive to HNO3 and water: Ta, Cr, Al, Au, TiO and ITO, as well as (it is assumed) CrSi, TaN, TiN, TiB_2 and ZrN. Pure Zr was eliminated from consideration due to its reactivity in engine-like environments, and reactively sputtering Hf due to the expense compared to similar metals such as Zr. To limit the scope of this study, gold is apparently the most stable metal dopant, and nitrogen the most stabilizing gas dopant.

Another characteristic to take in account in the application of thin film sensors is the coefficient of thermal expansion (CTE) and the matching of the film expansion to the substrate expansion due to temperature. The bulk properties of candidate materials and (for reference) alumina (Al_2O_3 - a common electrical insulator for superalloys) are given in table 5 (refs. 37 and 38). Though the properties of thin films may vary significantly from their bulk form, a general indication of relative properties of films can be gained from the bulk properties. Note the large bulk TCR for several of the materials compared to the thin film TCR reported in table 2. Also, no resistance data is given since in bulk the oxides are insulators. Of the ceramics identified, TiO, ITO, CrSiO, ZrN, TiB_2 and TiN appear to have good thermal expansion matches for alumina, in the range of ±30 percent (5.6 to 10.7 ppm/°C) to be considered compatible (ref. 39). To leverage off of NASA GRC's recent successes fabricating ceramic films by reactive sputtering (ref. 40), the ceramics TiB_2, TiN and ZrN optimized with gold and nitrogen doping can be examined for application of static strain gauges for the purpose of further development in this AFRL task.

TABLE 5.—BULK PROPERTIES OF HIGH TEMPERATURE CONDUCTIVE CERAMICS FOR POSSIBLE USE AS STRAIN SENSORS (REFS. 37 AND 38)

Ceramic	Density (g/cc)	Melting point (°C)	Resistivity at 20 °C (μΩ-cm)	TCR (ppm/°C)	CTE (ppm/°C)
TiO	4.0	1800	n/a	n/a	7 to 8
ITO	7.1	1900	n/a	n/a	7 to 8
CrSiO	3.7	2000	n/a	n/a	5 to 8
Al_2O_3	3.9	2100	n/a	n/a	8 to 8.2
ZrN	6.97	2980	21	4300	7.24
TiB_2	4.45	2980	14.4	2780	8.10
TaN	13.8	3090	128	30	3.60
TiN	5.20	3205	25	2480	9.35

3. Research Lessons Learned

3.1 Effectiveness of Research Techniques

The techniques used to identify candidate thin film ceramic sensor materials in creating a list of possible thin film ceramic sensor materials achieved their stated objective. In the literature search, materials were identified that were previously both known (such as tantalum nitride) and unfamliar (such as titanium diboride) to the GRC researchers for this application. The review of the potential materials for chemical and physical compatibility revealed known issues such as gold's susceptibility to attack by *Aqua Regia*, but also the susceptibility of aluminum nitride to attack by water. The lack of detailed information on the chemical resistance of several nitrides and titanium diboride was disappointing, and actual experience may result in a further down-select of ceramic sensor materials. We are confident that our technique has produced a valid list of thin film ceramics to investigate for the application as static strain gauges on turbine engine components.

4. Summary and Future Efforts

4.1 Results

The need to consider ceramic sensing elements is brought about by the temperature limits of metal thin film sensors in propulsion system applications. In order to have a more passive method of negating changes of resistance due to temperature, an effort is underway at NASA GRC to develop high temperature thin film ceramic static strain gauges for application in turbine engines, specifically in the fan and compressor modules on blades. Other applications can be on aircraft hot section structures and on thermal protection systems.

The near-term interim goal of the research effort was to identify candidate thin film ceramic sensor materials to test for viability and provide a list of possible thin film ceramic sensor materials and corresponding properties to test for viability. This goal was achieved by a thorough literature search for ceramics that have the potential for application as high temperature thin film strain gauges, reviewing potential candidate materials for chemical and physical compatibility with our microfabrication procedures and substrates.

A variety of ceramics were identified as having potential as high temperature thin film static strain sensors that include aluminum nitride (AlN), titanium boride (TiB_2), and doped and undoped tantalum nitride (TaN), titanium nitride (TiN) and zirconium nitride (ZrN), as well as some conductive oxides that are being pursued for multifunctional sensor work under an internal NASA task. Considering the chemical and physical compatibility of the ceramics for microfabrication on alumina, a short list of TiB_2, TiN, and ZrN was generated. Co-sputtering candidates were identified as including tantalum, chromium, aluminum, copper, and gold. Of these, gold is the least reactive to our microfabrication processes.

4.2 Future Effort

The various microfabricated thin film strain sensors will be developed using MEMS-based fabrication techniques in a class 1000 clean room at NASA GRC using physical vapor deposition and photolithography technologies. Surface analytical tools such as scanning electron microscope, auger electron spectrometer, and x-ray photoelectron spectrometer will be used to characterize these thin film ceramics. Based on results of research given in this report, this effort will focus on the application of ZrN, TiN, and TiB_2 strain gauges, doped with gold. The fabrication matrix for the test sensors is identified in table 6. Each target/co-sputter group will be tested using variable gas mixtures. Testing the TCR at low temperatures to 200 °C on a hot plate will provide the first level of optimization for the sensors. The optimized candidate thin film ceramic sensors initially will be applied to alumina specimens and subjected to low temperature tests between 100 and 700 °C in a heater rig to determine the basic sensitivity to the thermal and mechanical characteristics to be measured. This test will identify those ceramic sensors that are viable for the high temperature cycling tests. Next, the thin film ceramic sensors will be applied to ceramic matrix composites specimens, and bench tested in a heater rig, cycling the temperature between 100 and 1300 °C for 200 cycles. This test is similar to what is run in validation of conventional flame spray instrumentation. Technical metrics such as sensitivity, stability, repeatability, interference and durability will determine whether the sensor is ready for testbed qualifications for qualifying parts or structural elements and verify numerical codes, towards application in turbine engines.

TABLE 6.—FABRICATION MATRIX FOR STATIC STRAIN GAUGE FABRICATION USING VARIOUS MIXES OF ARGON AND NITROGEN SPUTTERING GASSES AND GOLD CO-SPUTTERING

Target	No Co-Sputter	Gold (Au) Co-Sputter
TiB_2	Optimize Ar Pressure, Sputter Power	Optimize Ar Pressure, Sputter Power
Ti	Optimize N_2/Ar Mix, Pressure, Sputter Power	Optimize N_2/Ar Mix, Pressure, Sputter Power
Zr	Optimize N_2/Ar Mix, Pressure, Sputter Power	Optimize N_2/Ar Mix, Pressure, Sputter Power

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

The need to consider ceramic sensing elements is brought about by the temperature limits of metal thin film sensors in propulsion system applications. In order to have a more passive method of negating changes of resistance due to temperature, an effort is underway at NASA Glenn to develop high temperature thin film ceramic static strain gauges for application in turbine engines, specifically in the fan and compressor modules on blades. Other applications can be on aircraft hot section structures and on thermal protection systems. The near-term interim goal of the research effort was to identify candidate thin film ceramic sensor materials to test for viability and provide a list of possible thin film ceramic sensor materials and corresponding properties to test for viability. This goal was achieved by a thorough literature search for ceramics that have the potential for application as high temperature thin film strain gauges, reviewing potential candidate materials for chemical and physical compatibility with our microfabrication procedures and substrates.

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