2016 NETL Crosscutting Research Review Meeting

April 18-22, 2016

SBIR Phase IIA Project: DOE 12-14C

Contract #: DE-SC-0008269

Advanced Ceramic Materials and Packaging Technologies for Realizing Sensors Operable up to 1800 Celsius in Advanced Energy Generation Systems

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Acknowledgement

This material is based upon SBIR work supported by the Department of Energy under Award Number DE-SC0008269.

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Overview

- Sporian Introduction
- Project Motivation
- Prior, Related Work
- Current Effort Progress Update

About Sporian Microsystems

- Sporian develops advanced sensors and sensor systems for a range of applications.

Core Technical Competencies

- Novel Materials Science
- Leading edge signal Conditioning & Smart Electronics
- Advanced Electronics & Hardware Packaging

Advanced Sensor Technologies

Biological & Chemical

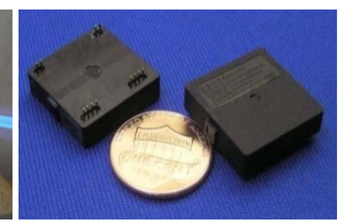
- Water Quality
- Gas Composition
- Biomedical
- Hyperspectral Imaging

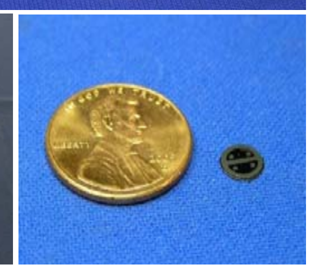
Energy & Aerospace

- Very High Temperature
- Harsh Environments
- Asset monitoring
- PHM





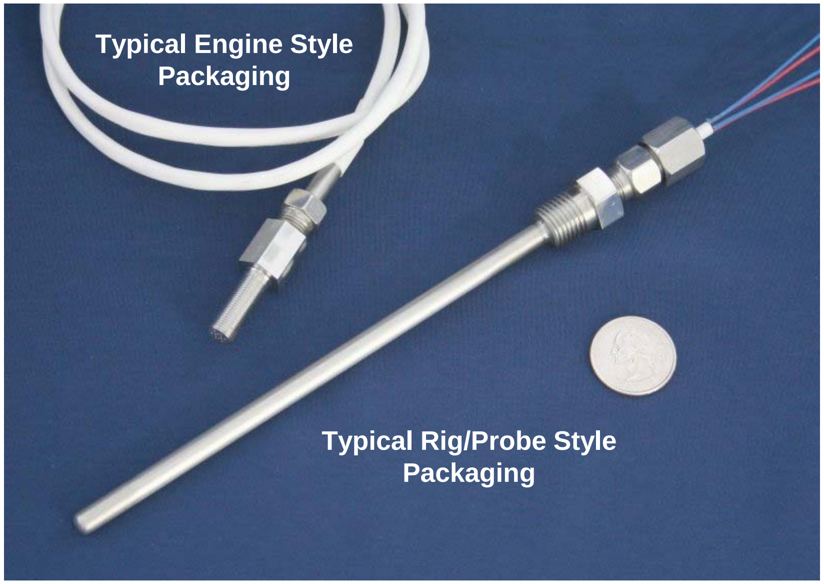





Overview of Sporian's Harsh Environment/High Temperature MEMS Sensors and Packaging

R&D focus area on **high-temperature sensors** and **packaging**

- Directly monitor the most harsh/costly sections of equipment
  - Pressure, temperature, flow, flame ionization, strain, etc.
  - Packaging a critical enabler
- Started with **DOE-funded basic science** SBIR 2003
- Aerospace (turbine engines)
  - Air Force, Navy, NASA funded
- Energy generation (gas turbines, coal gasification, nuclear, CSP, etc.)
  - **DOE funded**



- Prior work predominantly focused on <1400 °C application
- **Current DOE project focusing on extending capabilities to 1800 °C**

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Motivation

- Higher turbine efficiencies achievable at higher combustion temperatures (≤1800 °C depending on fuel).
- Existing thermocouples (TCs) for combustor monitoring are expensive and short-lived
  - Practical only in design phase of turbine life-cycle.
- TCs used at turbine exhaust (lower temp) to infer combustor temperature -- limits efficiency
- Additional efficiency gains possible with dynamic pressure measurement.

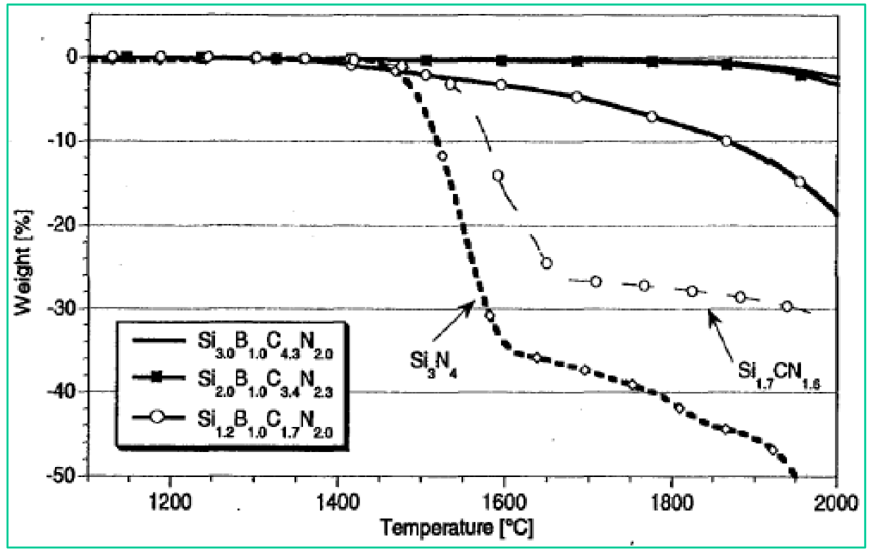
Ultra-High Temperature SiCN Ceramics

- SiCN has shown excellent HT thermo-mechanical properties.
- Sporian existing polymer-derived ceramic (**PDC**) SiCN formulations can work safely under 1350 °C
- SiBCN is thermally stable up to 1800 °C

Selected Literature Review of SiBCN

Empirical Formula	Maximum Stable Temperature	Selected Reference from More than 100 Papers/Reviews
$Si_{2.9}B_{1.0}C_{14.0}N_{2.9}$ $Si_{5.3}B_{1.0}C_{19.0}N_{3.4}$	2200°C-30min	Wang and Riedel, 2001
$Si_{3.0}B_{1.0}C_{4.3}N_{2.0}$	~2000°C	Riedel, 1996
$Si_{1.0}B_{1.0}C_{1.6}N_{2.4}$	~1785°C	Wilfert and Jansen, 2012
$Si_{1.0}B_{1.0}C_{1.7}N_{2.3}$	~1700°C	Weinmann, 2008
$Si_{2.0}B_{1.0}C_{3.4}N_{2.3}$	~1600°C	Zhang, 2011
$Si_{1.0}B_{1.0}C_{2.0}N_{2.8}$	>1400°C	Tang, 2009

Weight Loss at High Temperatures (in UHP He)



Challenges:

- Synthesis of new precursors
- Viscosity control for workability/patternability
- UV cure capability to make useful devices
- Optimized pyrolysis processing
- Contamination and defect control for thermal stability

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Prior, Related Work <1400 °C - PDCs

Features, Advantages and Benefits

Features	Advantages	Benefits
Polymer-derived ceramic (**PDC**) materials	• Operating temperature >1000 °C w/o liquid cooling or fiber routing • Pressures ≥1000 psia • High oxidation/corrosion resistance • Thermal shock resistance • Low creep rate & diffusion rate	• Lower weight, smaller size • Lower cost, low-maintenance • Higher durability • Higher operational availability
Temperature / pressure sensor suite	• Improved T-compensation of pressure measurements • Opportunity for redundancy and/or multi-sensor package	• Lower weight, smaller size • Higher accuracy
Immersion sensing at source	• Eliminate stand-off tubes • Avoid tube moisture collection	• Lower cost, higher accuracy • Reduced weight • Improved dynamic response • Reduced latency • **Avoid failure mechanism**
Electronics-based	• Compatible with existing controls & CBM	• Lower cost

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Prior, Related Work <1400 °C
Performance

Specification	PIWG* Target	Achieved by Sporian
Pressure Range (**psi**)	25-750	Atm-1000
Operation Temperature (**°C**)	700-1350	25-1350
Natural Frequency	>100 kHz	TBD
Internally Compensated Temp. Range (**°C**)	700-1350	700-1350
Length (**in**)	1.25-3.00	1-10 (modifiable)
Diameter (**in**)	<0.25	0.25
Sensitivity/Combined Uncertainties	≤ 1% FS	≤ 1% FS
Power (**VDC**)	5-10	12 V (modifiable)

***Propulsion Instrumentation Working Group**

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Prior, Related Work <1400 °C

Demonstrations (various projects)

Asset	Station	Hours *	Max T (°C)	Max P (psi)
Laboratory	N/A	-	1400	1000
Mult. OEM Burner Rigs	N/A	535	**	**
DOE Burner Rig	N/A	150	1000	30
Honeywell HTF 7000	P3	24	**	**
GE (NAVAIR) T700	P3	200	**	**
OEM Engine	P3, P4, P4.5	100	**	**

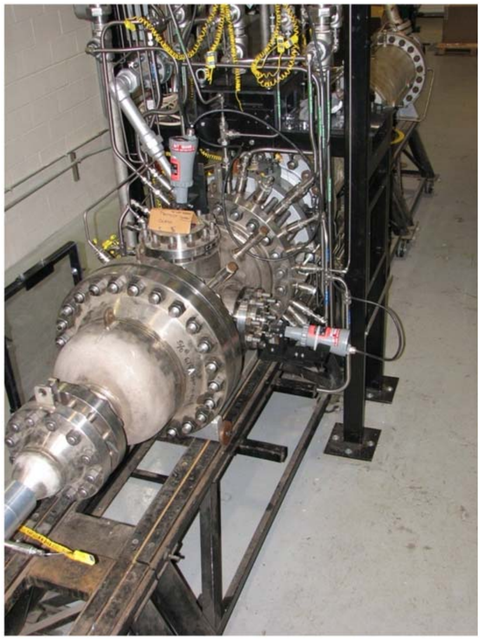
Asset	Type	Hours *	Max T (°C)	Max P (psi)
Sandia Nitrate Salt Soak	Flow/P/T	500	300	N/A
UW Chloride Salt Soak	Flow/P/T	500	750	N/A
UW Nitrate Salt Soak	Flow/P/T	500	500	N/A
Skyfuel Molten Salt Loop	Flow/P/T	80	300	50
USGS: Neutron 10^{19} n/cm^2	various	N/A	N/A	N/A

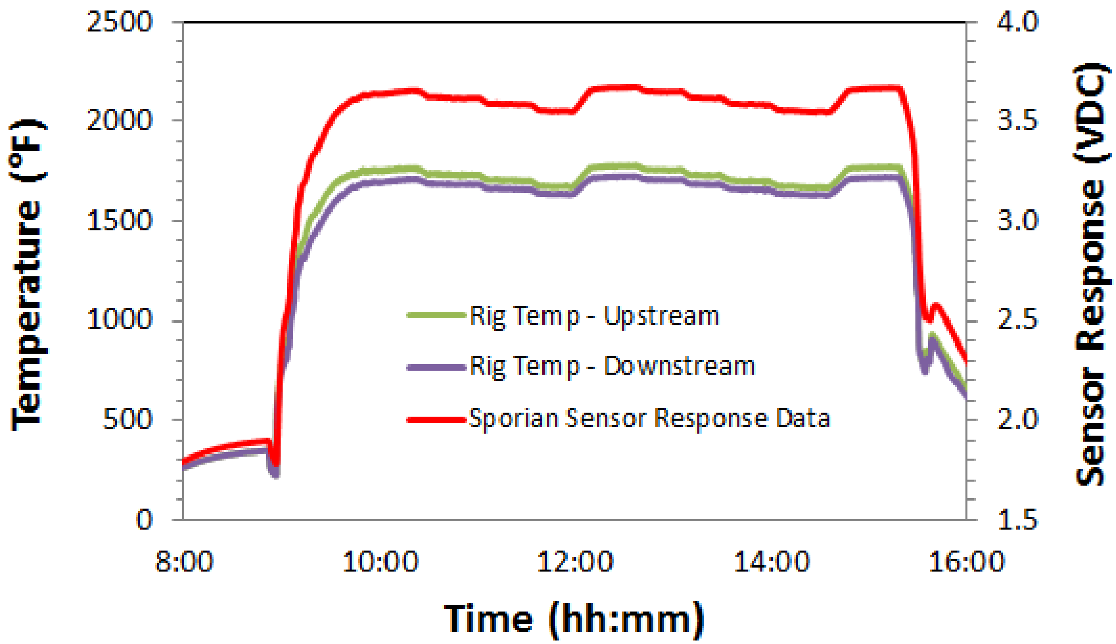
*** Test durations dictated by budgets. All sensors were fully operational after test completion.**

**** Proprietary**

NETL Rig Testing Results

Aerothermal Rig

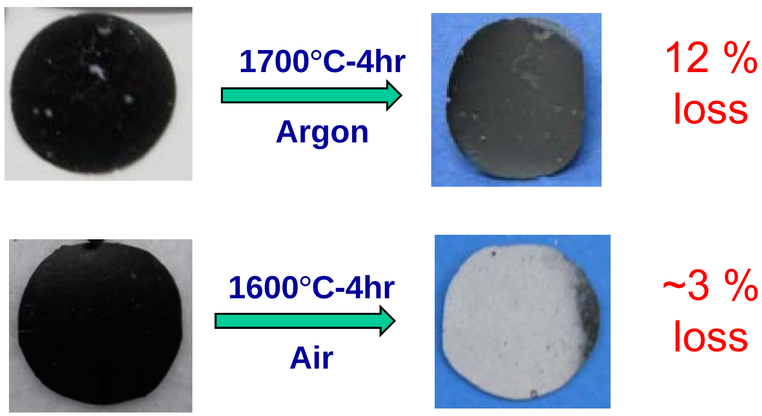


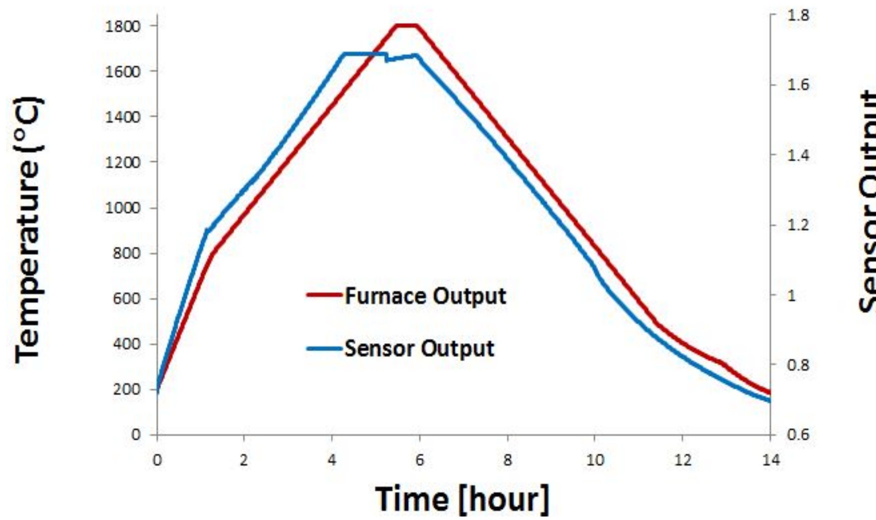


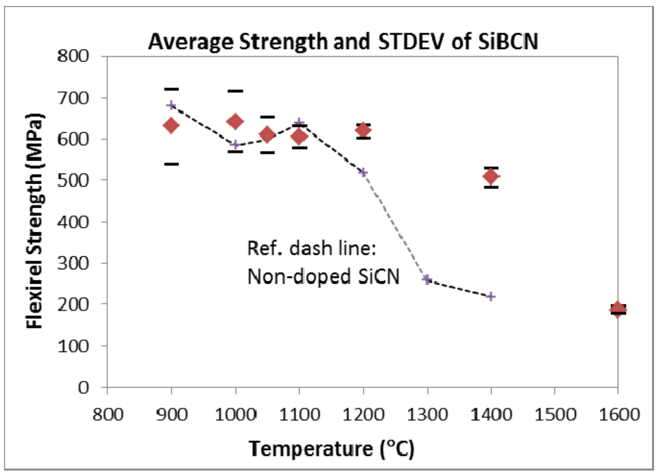
2014 Preliminary Results:

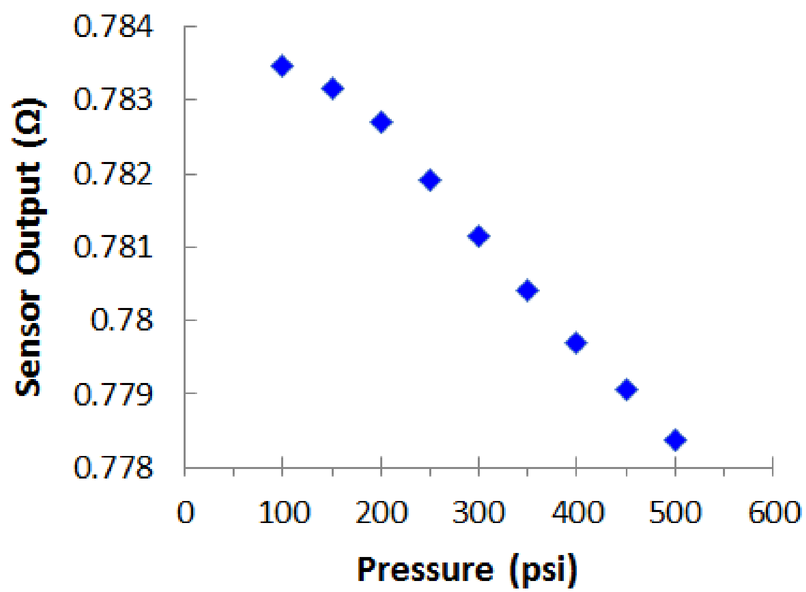
- Testing date: 10/29, 11/5, 11/12/2014
- 3x test cycles
- Maximum T : 1100 °C
- Total duration: 30 hours
- **Stable response and performance**

Phase II In Brief









- Developed sensors and packaging capable of 1800 °C operation
- Conducted pressure response testing on 1600 °C-capable probe

Current Effort Progress Update

- Extend Sporian's Existing Ceramic Sensors and Packaging Technology to Ultra-high Temperatures (UHT): 1600-1800°C
- Build on PII tasks for sensors, packaging, electronics to push capabilities to 1800 °C

1. Work with OEMs to guide the design and development of UHT sensor technology: Commercialization and transition efforts.
2. Continue optimizing PDC precursor formulation and device fabrication to further extend capability to 1800 °C
3. Develop improved UHT P/T sensors, packaging, and drive/conditioning electronics
4. Rigorous lab-scale testing of optimized sensors/packaging to promote post Phase IIA transition, **emphasize reliability assessment**
5. Revise sensor suite designs based on test results, construct next generation prototypes, and demonstrate a full prototype sensor in stakeholder test systems

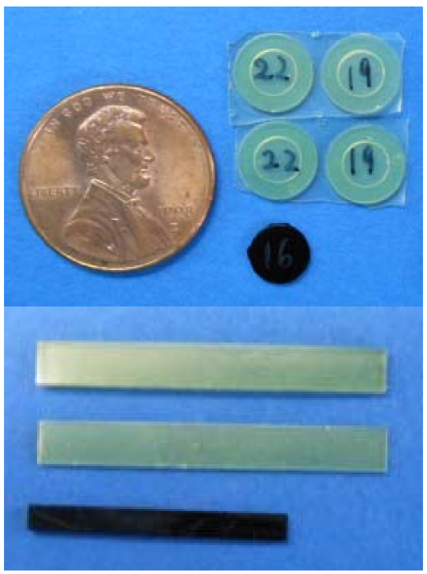
OEM Collaboration/Coordination

- Strong interest, requirements, and some in-kind support from:
  - Turbine OEMs
  - Controls/CBM OEMs
  - **Industry Research Institutions & Consortia**
  - Academic Institutions
  - Established sensor OEMs

Synthesis, Evaluation of Fully Dense SiBCN

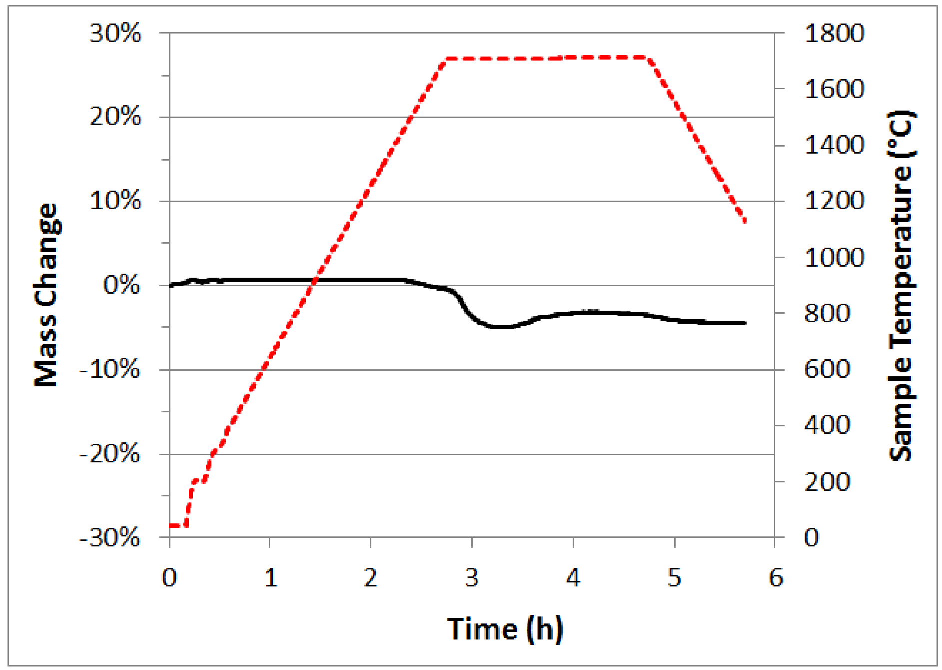
- Synthesized boron-doped polysilazane with good workability/stability
- Incorporated UV-curability to polyborosilazane precursors
- Achieved dense SiBCN ceramic materials and defect-free parts

UV-Curable Precursor and Fired SiBCN



B-doped Polymer and SiBCN Sensor and Coupons

Thermogravimetric Analysis of SiBCN



2 h at 1700 °C in air: ~ 5 % loss

- **Sensor-ready material**
- **In line with best-case literature (powders)**

Sporian SiBCN in Phase IIA

- B-doped composition focus – optimize precursor process, green part fabrication and handling, and fully-dense part and device processing
- Increase thermal stability in application-relevant environments
- Evaluate mechanical and chemical properties at increased temperatures
- Incorporate into sensor packaging for 1800 °C temperature and 1600 °C pressure sensor suites

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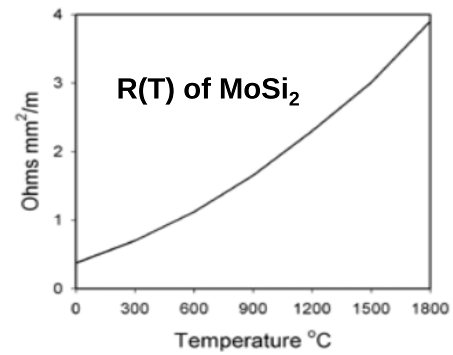
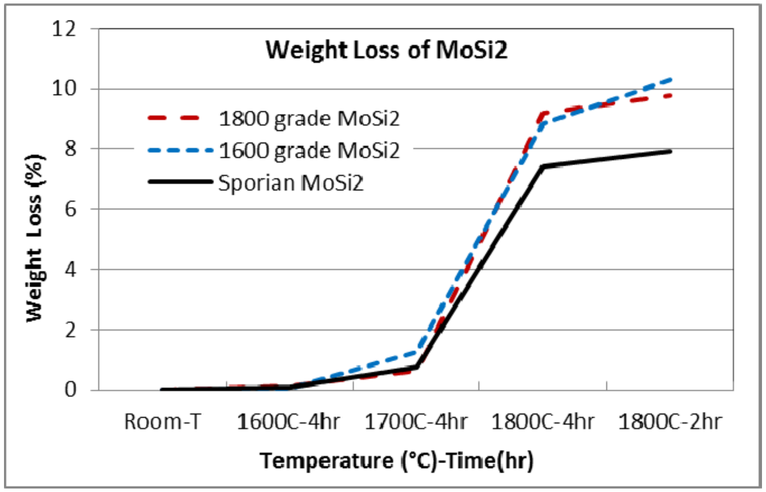
Ultra-High Temp $MoSi_2$–based Sensors

Sporian Sensor Materials, Prototypes:

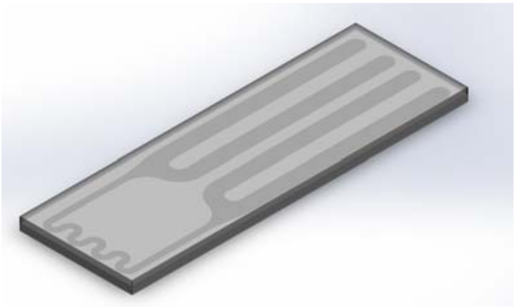
- Re-shapeable and stackable green tape, thick film inks as well
- Micro-fabrication and laser machinability
- High density (98 %) and high strength (351 MPa)
- Thermal stability and oxidation resistance at 1800 °C
- Comparable to the commercially available UHT grade (heater elements)
- Compatible CTE with alumina substrates and tapes





Sintered Structures and Packaged $MoSi_2$ Temp Sensor Element

Embedded MoSi2 RTD Temp Sensor – design and results

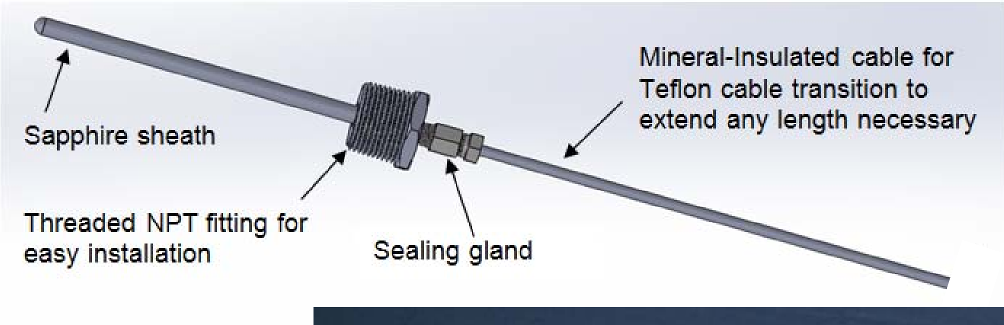


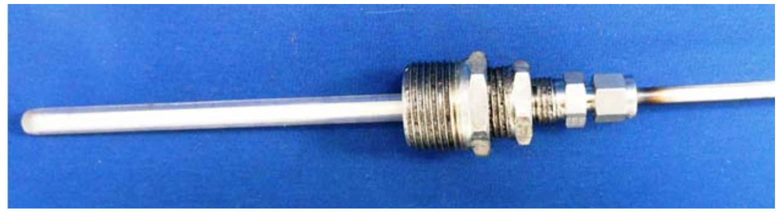
Challenges:

- Thermal stability
- Optimizing embedding layers for sensor efficiency, accuracy
- Interconnects to sensor electronics

Current Prototype 1800 °C Temperature Sensor
(Designed for NETL Rig Testing)

Sporian Sensor Packaging Design and Probe Assembly





‘Smart’ Signal Conditioning Electronics

Features:

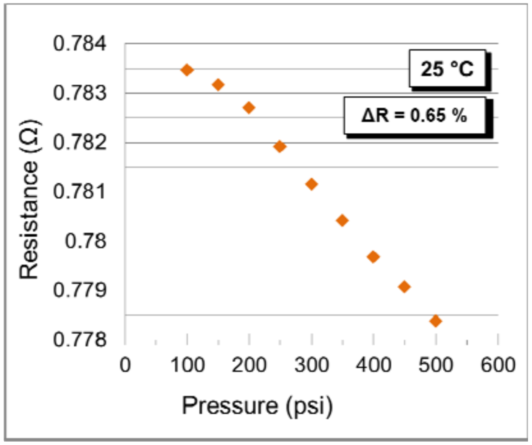
- Sapphire-sheathed UHT sensor packaging.
- Probe suitable for high pressures, high temperatures and particulate exposure.
- “Smart” signal conditioning electronics can drive the sensor over its entire operational range and measure the response.

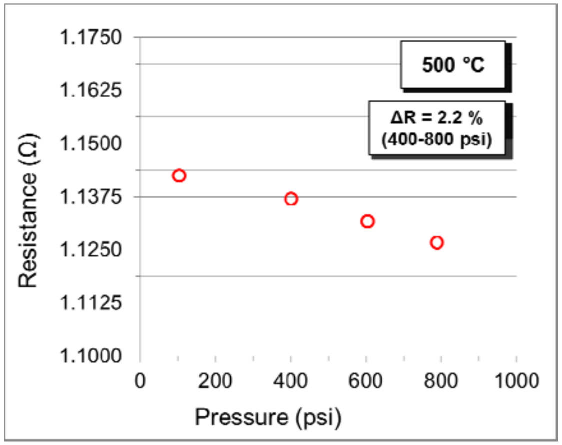
HT Testing of Sporian Prototypes

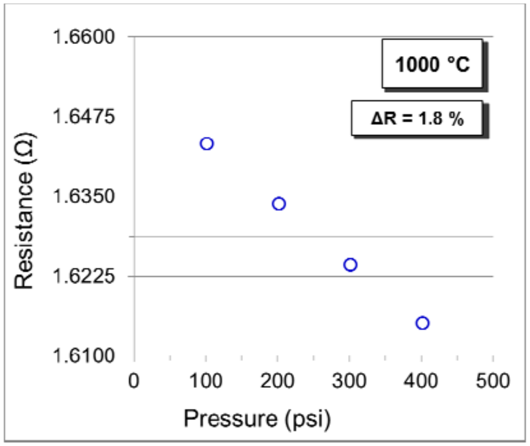
Test under Pressure at Temp:

- Exterior reference TC to track temperature
- 25, 500, and 1000 °C
- 15 – 800 psi
- Sensor response increased with increasing pressure
- Sensor and package stable, no degradation

Sporian In-House Pressure Test







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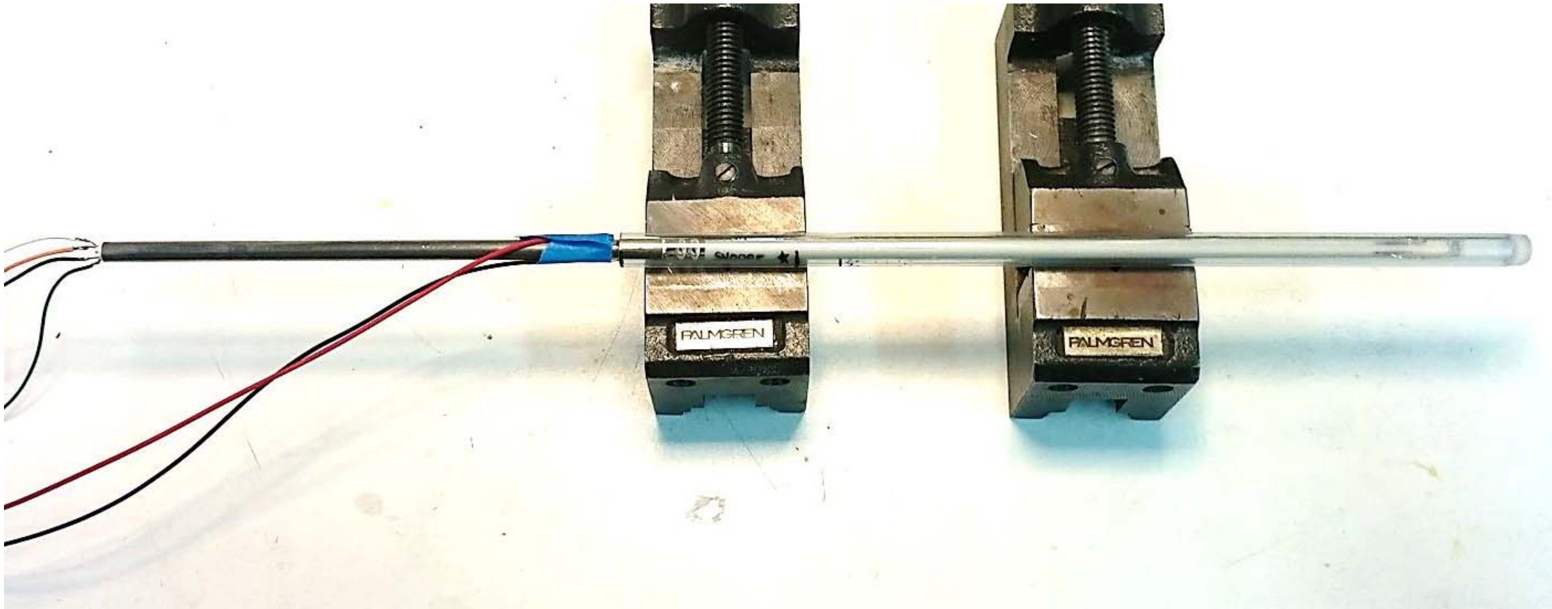
Current HT Stability Testing
(Designed for SwRI Rig)

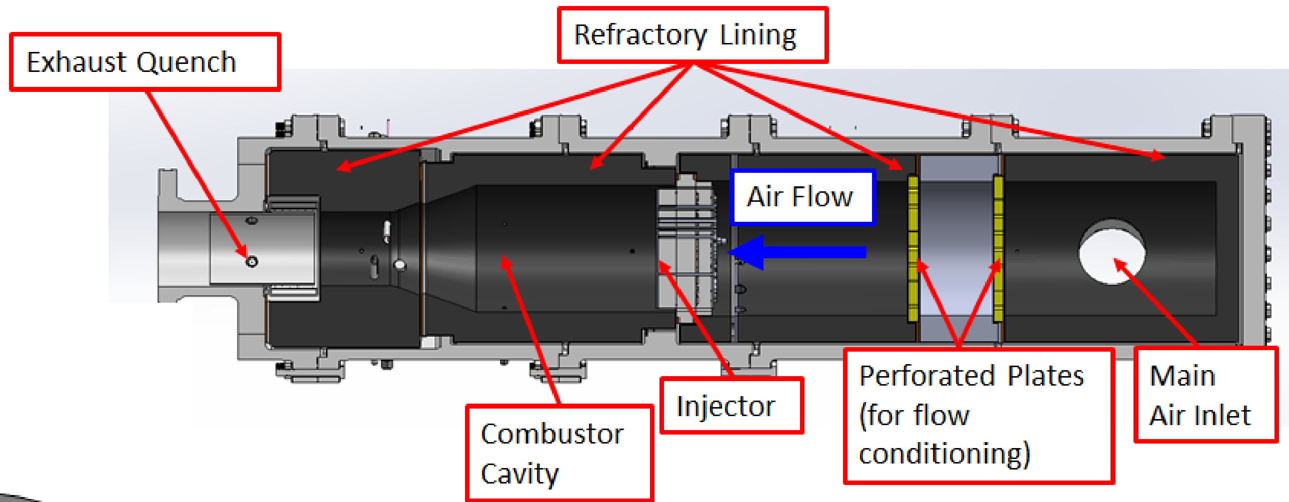


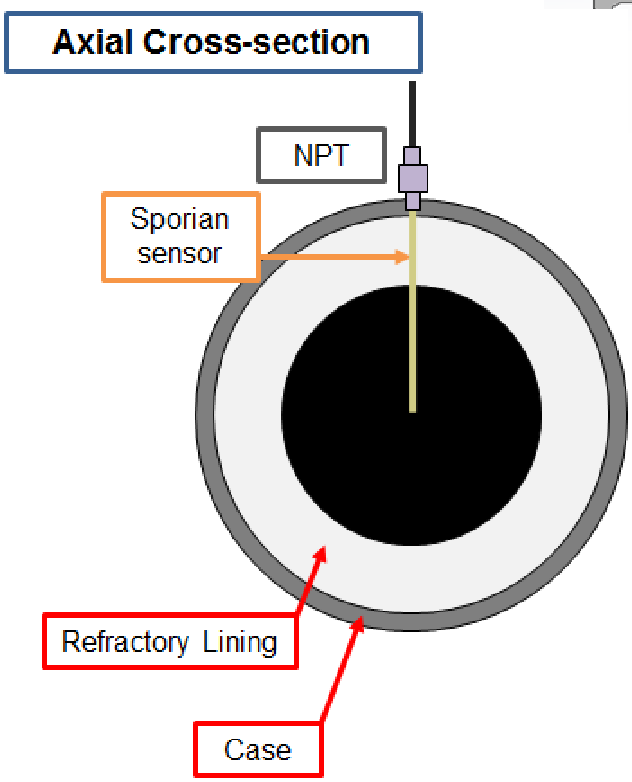
HT Test in-house:

- Heated to **1800 °C in air**
- 1 h hold
- Element and *in-situ* TC stable post-test
- Packaging stable – no cracking, warping, or degradation

SwRI PHTFF Rig Testing – Upcoming in PIIA

Pressurized High-Temperature Flow Facility (PHTFF)





Testing Objectives:

- Extended testing available with full access to rig's various sections
- UHT Temp Sensors (up to 1800 °C) **at Injector**
- UHT Pressure/Temp Sensors in **Combustor Cavity, behind Injector**

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Summary

1. Optimizing UV-curable/patternable PDC precursor materials and processing
2. Enhancing thermal, mechanical, and chemical stability of PDC materials and alternatives ($MoSi_2$) in sensor prototypes
3. UHT packaging temperature probes survived 1800 °C in lab and 1100 °C 30 h NETL Aerothermal Rig Test
4. Preparing for extended testing in SwRI PHTFF rig to evaluate 1800 °C temp sensors and 1600 °C pressure sensors

Thank you for your attention!

Questions?

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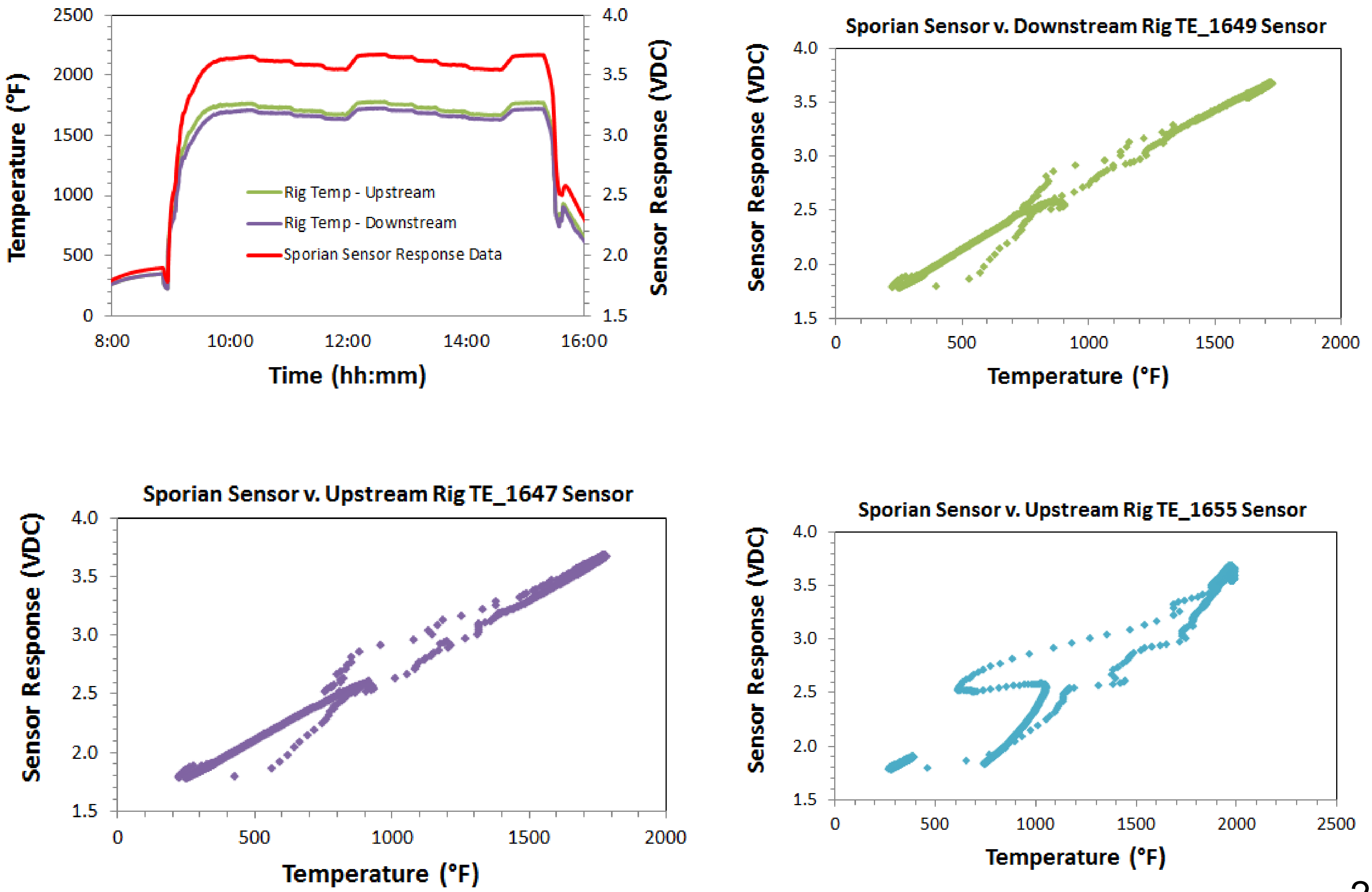
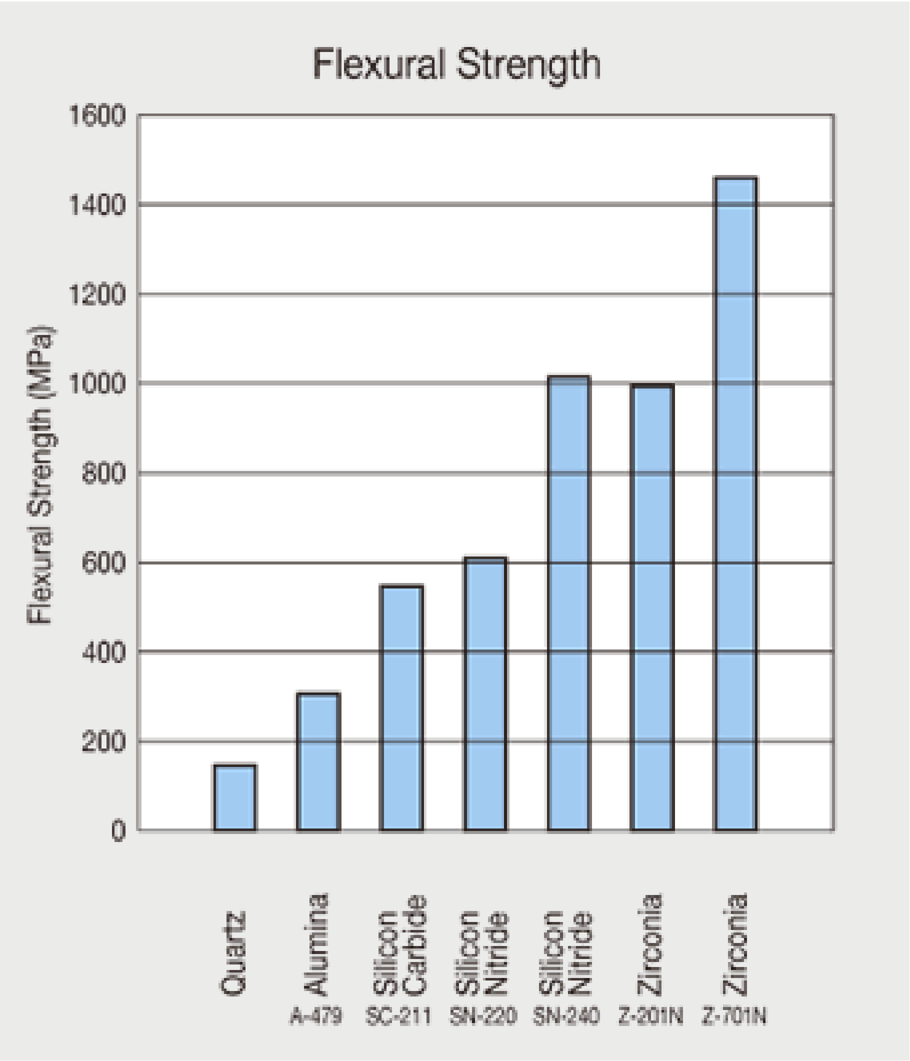
NETL Aerothermal Rig Testing Results




Flexural Strength
Flexural Strength (MPa)
1600
1400
1200
1000
800
600
400
200
0
Quartz
Alumina
A-479
Silicon Carbide
SC-211
Silicon Nitride
SN-220
Silicon Nitride
SN-240
Zirconia
Z-201N
Zirconia
Z-701N