SPORIAN MICROSYSTEMS, INC

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Phase II Contract #: DE-SC-0008269

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

Authors: Yiping Liu (PI), Laurel Frediani, Michael Usrey (Presenter)
Contact: Michael Usrey, Email: musrey@sporian.com,
Phone Number: 303-516-9075 x19

Sporian Microsystems, Inc. (www.sporian.com)
515 Courtney Way - Suite B, Lafayette, CO 80026-8821

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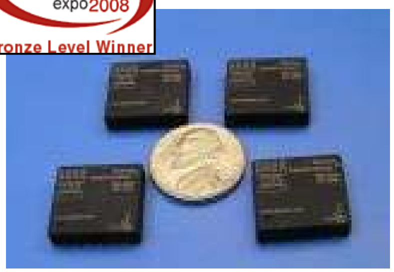
Overview

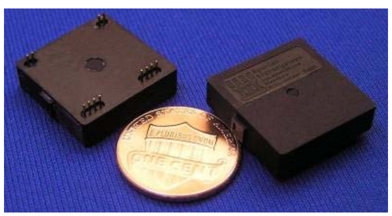
- Sporian Introduction
- Project Motivation
- Prior, Related Work <1400C
- Current Effort Progress Update
- Discussion/Questions

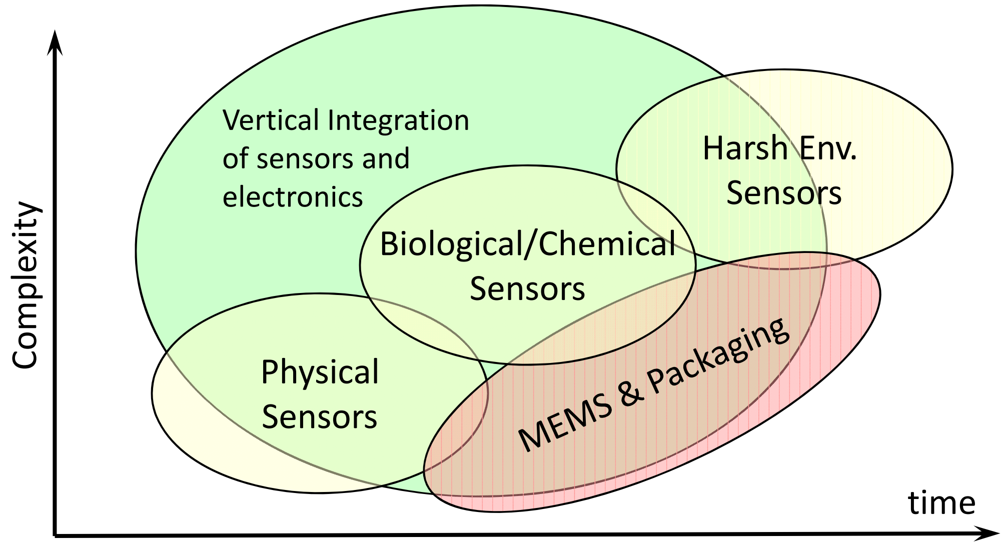
Sporian Introduction

- **3 primary development efforts at Sporian:**
  - Physical sensor suites – shock, humidity, temperature, strain, etc.
  - Photonic-based wireless chemical/bio-sensors.
  - Harsh Environment/High Temperature sensors & packaging
    - Pius: Systems integration (signal processing, logging, storage, communications)

best of sensors expo2008 Bronze Level Winner





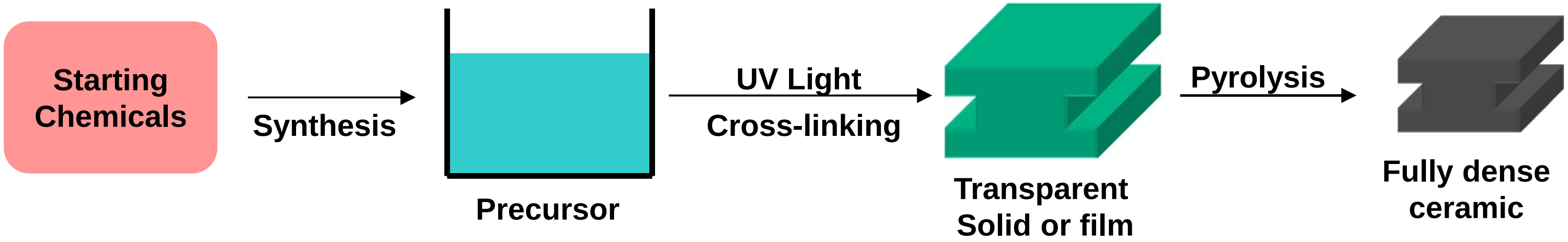


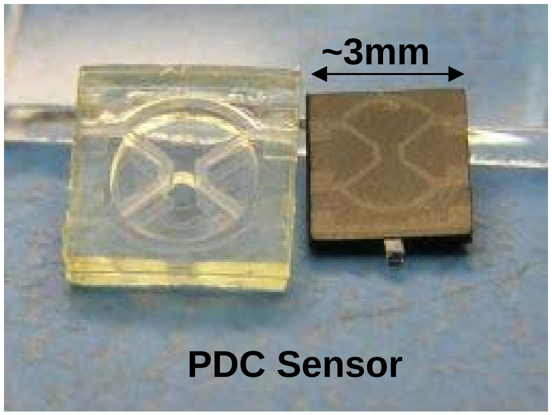
MERCURY 100 2013

Motivation

- Turbine efficiencies are driving combustion temperatures higher. Up to 1800C depending on fuel.
- Existing combustor thermocouples are expensive and short-lived. Useful only in design phase of turbine life-cycle.
- Efficiency lost due to use of thermocouples in exhaust to infer combustion temperatures in fielded turbines.
- Additional efficiency gains possible with dynamic pressure measurement.

Prior, Related Work <1400C





Wide Range, In Situ Pressure Sensor Suite for turbine engines

Prior, Related Work <1400C

Features, Advantages and Benefits

Features	Advantages	Benefits
Polymer derived ceramic materials	• Operating temperature >1000°C w/o liquid cooling or fiber routing • Pressures up to and beyond 1000 psia • Highly oxidation/corrosion resistant • Thermal shock resistant • 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 sensor fusion	• Lower weight, smaller size • Higher accuracy
Immersion sensing at source	• Eliminate stand-off tubes • Avoid tube moisture collection	• Lower cost, higher accuracy • Lower 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 <1400C

Performance

Specification	PIWG Target	Achieved
Pressure Range (psi)	25-750	Atm-1000
Operation Temperature (C)	700-1350	25-1350
Natural Frequency	> 100khz	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)

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Prior, Related Work <1400C

Demonstrations

Asset	Station	Hours *	Max T (°C)	Max P (psi)
Laboratory	NA	-	1400	1000
OEM Burner Rig	NA	8	**	**
OEM Burner Rig	NA	535	**	**
DOE Burner Rig	NA	150	1000	30
Honeywell HTF 7000	P3	24	**	**
GE (NAVAIR) T700	P3	200	**	**
OEM Engine	P3, P4, P4.5	100	**	**
Sandia Nitrate Salt	-	500	300	-
UW Chloride Salt	-	500	750	-
PNNL Gamma 10^8	-	-	-	-
USGS Neutron 10^{18}	-	-	-	-

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

**** Proprietary**

Current Effort Progress Update

- Extend Sporian's Existing Ceramic Sensors and Packaging Technology to Ultra-high Temperatures (UHT): 1600-1800°C

1. Work with OEMs to guide the development of a useful implementation of the proposed UHT sensor technology: Commercialization and transition efforts.
2. Synthesis of UV curable B-doped precursor formulations to realize SiBCN materials and sensors stable to target temperatures
3. Development of detailed designs and prototypes for a 1800°C capable temperature sensor, packaging, and associated drive/conditioning electronics
4. Development of designs and prototypes for a >1600°C capable pressure and temperature sensor suite, packaging, and associated drive/conditioning electronics
5. Rigorous testing of prototype sensors/packaging in lab scale environment to validate potential application suitability
6. Revise UHT ceramic materials, sensor fabrication techniques and packaging designs in order to build higher level hardware for testing.
7. Demonstration of UHT prototypes in application relevant testing system.

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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

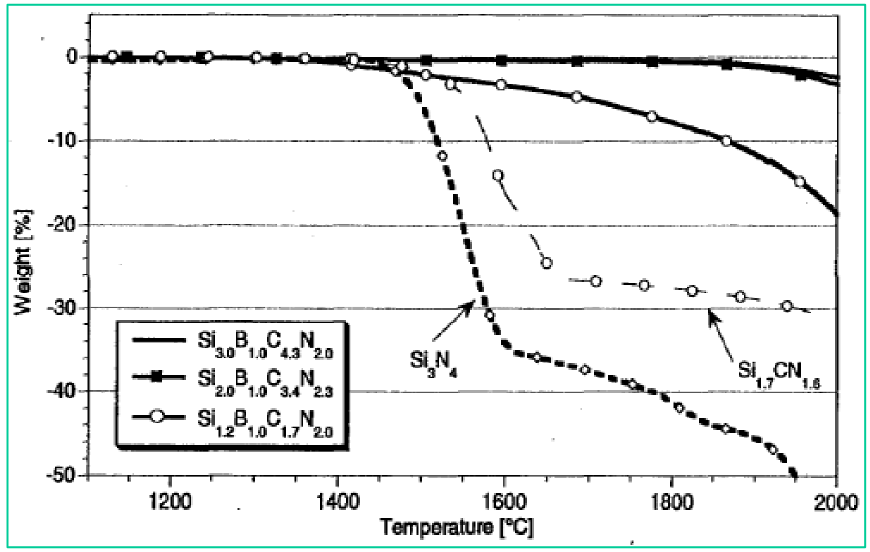
Ultra-high Temperature SiBCN Ceramics

- SiCN has shown excellent HT thermo-mechanical properties.
- Sporian existing SiCN formulations can work safely under 1350°C
- SiBCN is proven to be thermally stable up to 1800°C

Selected Literature Review of SiBCN

Empirical Formula of Ceramic	Maxima Temperature of Stability	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

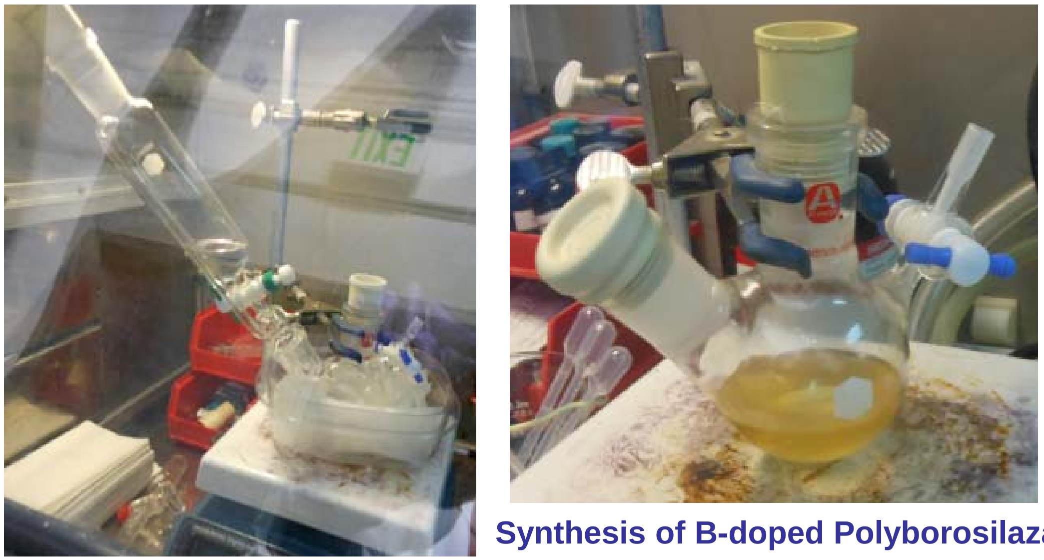


Challenges:

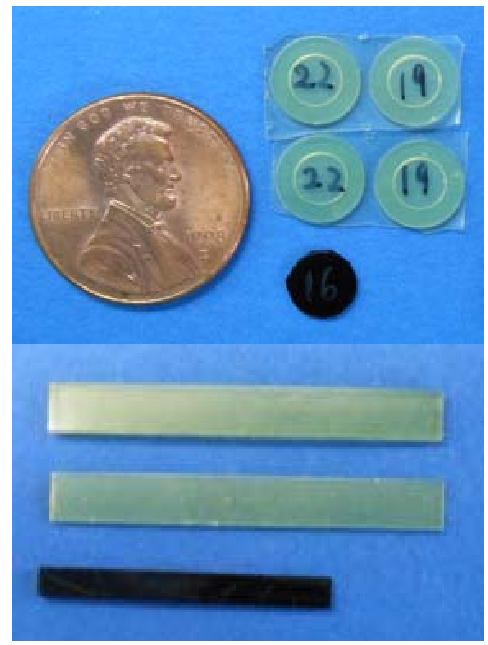
- Synthesis of new precursors
- Viscosity control for workability/patternability
- UV cure capability to make useful devices
- Survive pyrolysis
- Contamination control for thermal stability

Sporian Synthesis 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 devices



Synthesis of B-doped Polyborosilazane



B-doped Polymer and SiBCN Sensor and Coupons

UV Curable Precursor and B-doped SiBCN

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Thermal Stability of Sporian SiBCN

- Fabricated SiBCN Material Coupons Survived 1600°C-4hr in Argon

- 1600°C Thermal Test: weight loss ~1.3%
- 1700°C Thermal Test: weight loss ~12%

UV curable B-doped precursor and SiBCN:

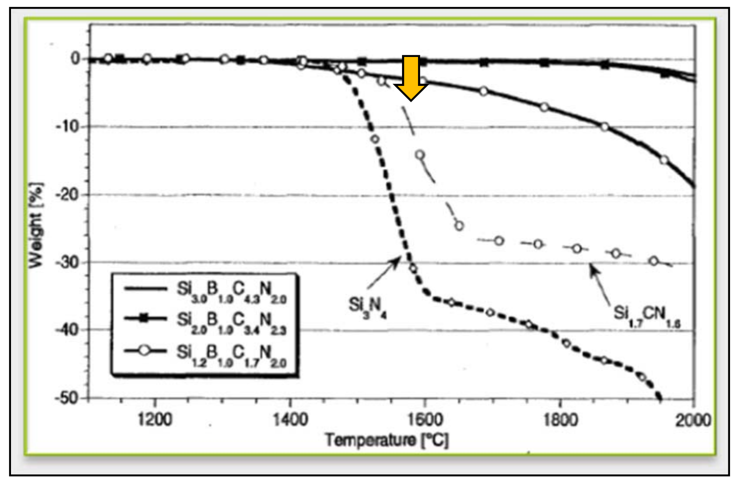
1400°C-4hr

Pyrolysis

1700°C-4hr

Argon

Data from literature (Argon)vs. where are we

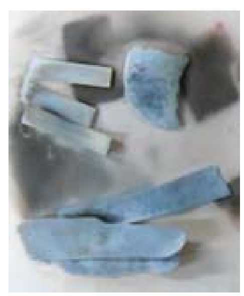


Reference: non B-doped SiCN

1700°C-4hr

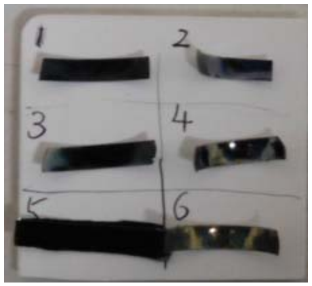
Argon

Decomposed



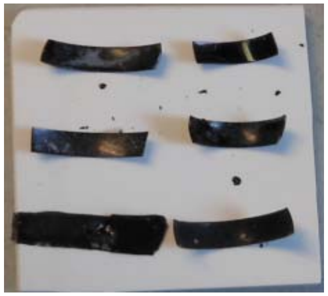
Oxidation Resistance of Sporian SiBCN

- SiBCN Coupons Showed Oxidation Resistance up to 1600°C in Air



1550°C-4hr

Air flow



1600°C-4hr

Air

1550°C Air Thermal Test Results:

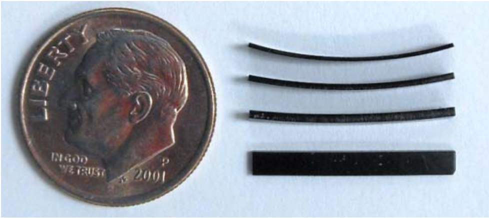
- *No Weight Change*
- *No Dimensional Change*

1600°C Air Thermal Test Results:

- Formation of surface passive oxide layer
- Small weight loss: 2~5%
- Small increase in dimension: 0~4%
- Depends on formulation and thickness

Mechanical Strength of Sporian SiBCN

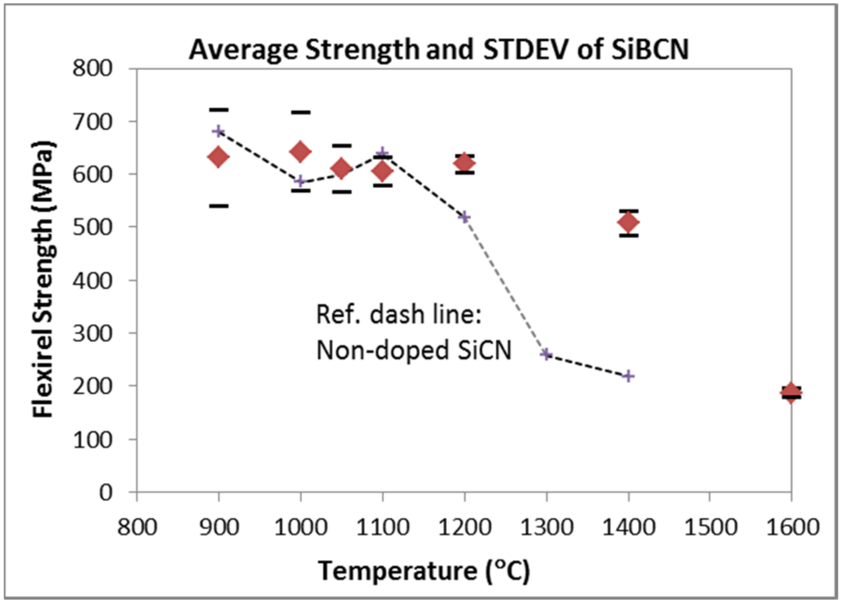
- SiBCN - Stable mechanical strength compared w/ non B-doped SiCN



Three-point Bending Test Coupons

Mechanical Testing System

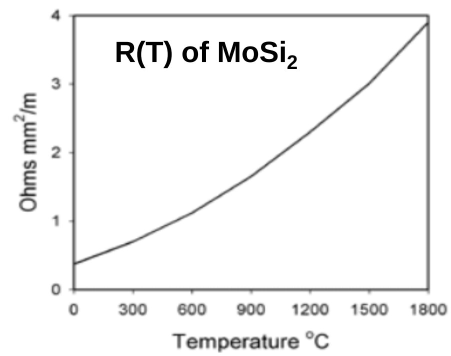
Three-point Bending Test Results



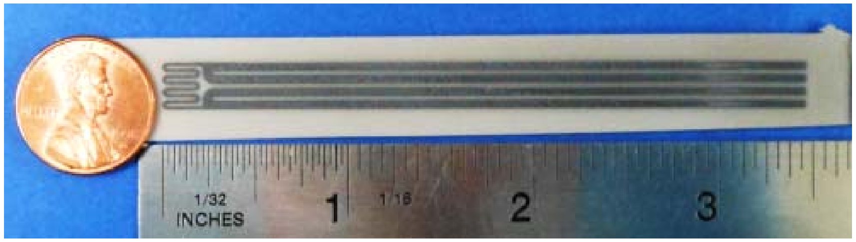
Development of Ultra-High T $MoSi_2$

Development of Sporian 1800°C $MoSi_2$ Sensor Materials:

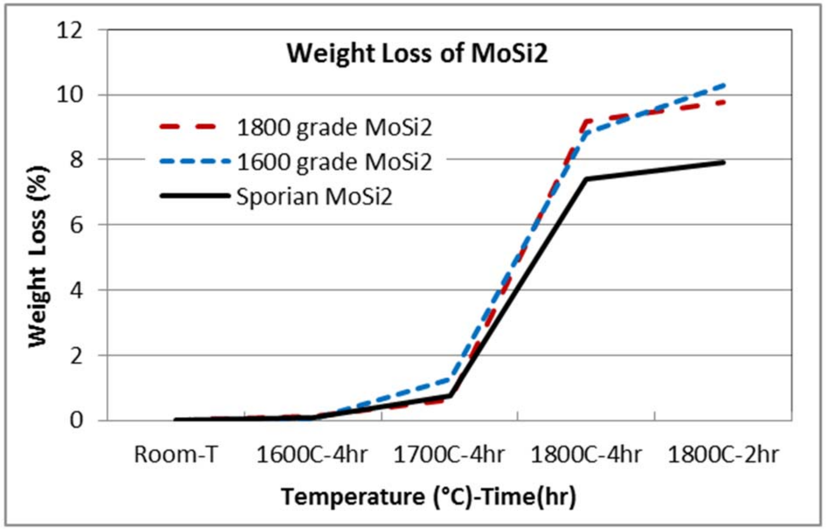
- Re-shapable and stackable green tape
- Micro fabrication and laser machinability
- High density (98%) and high strength (351 MPa)
- Thermal stability and oxidation resistance at 1800°C
- Comparable to the highest commercially available grade
- Compatible CTE with alumina substrates



Sintered Structures and Packaged $MoSi_2$ Sensor Element



Screen Printed $MoSi_2$ Paste/Ink on Alumina Substrate



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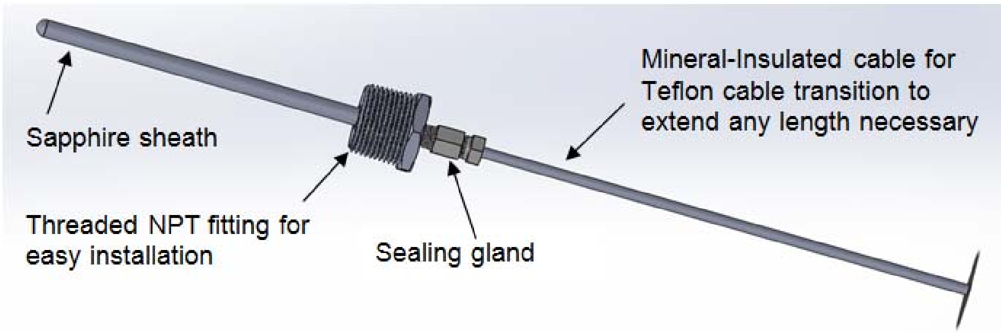
Current Prototype 1800ºC Temperature Sensor
(Designed for NETL Rig Testing)

Sporian Sensor Packaging Design and Probe Assembly

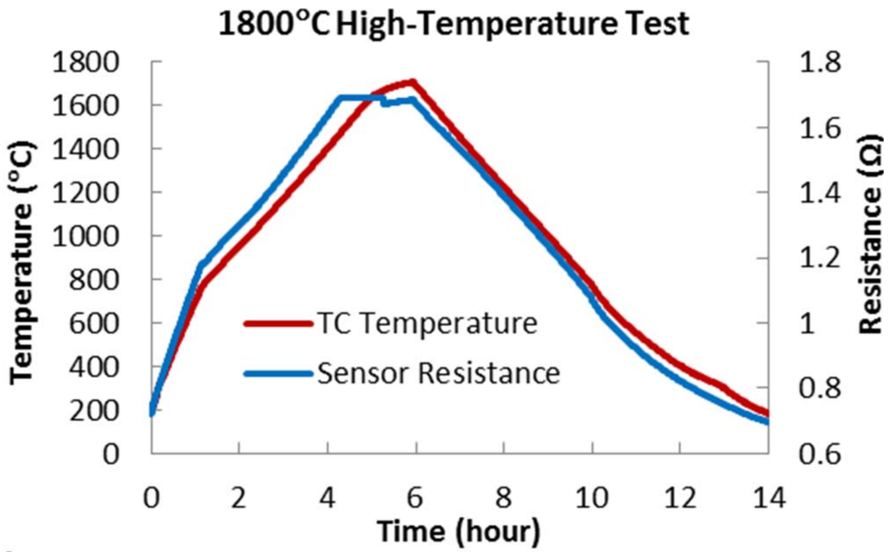


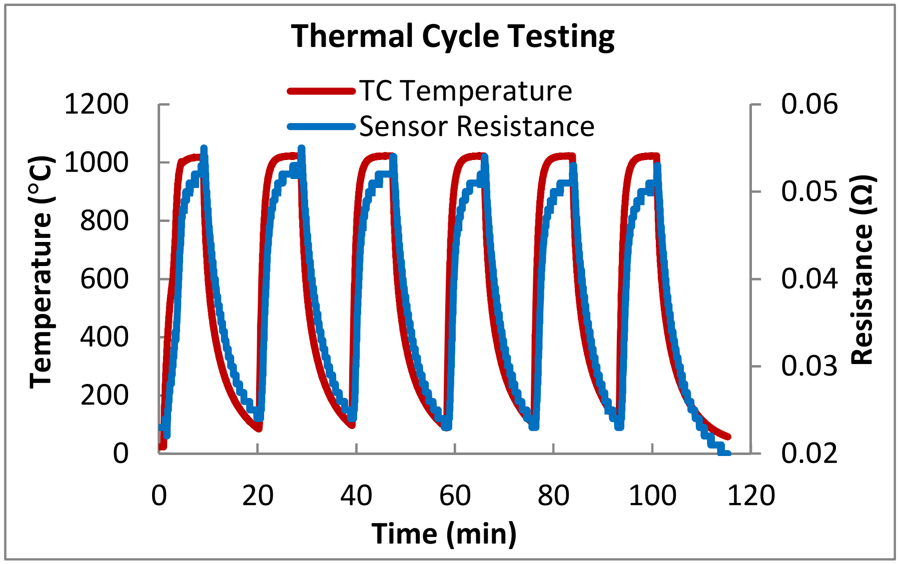
'Smart' Signal Conditioning Electronics

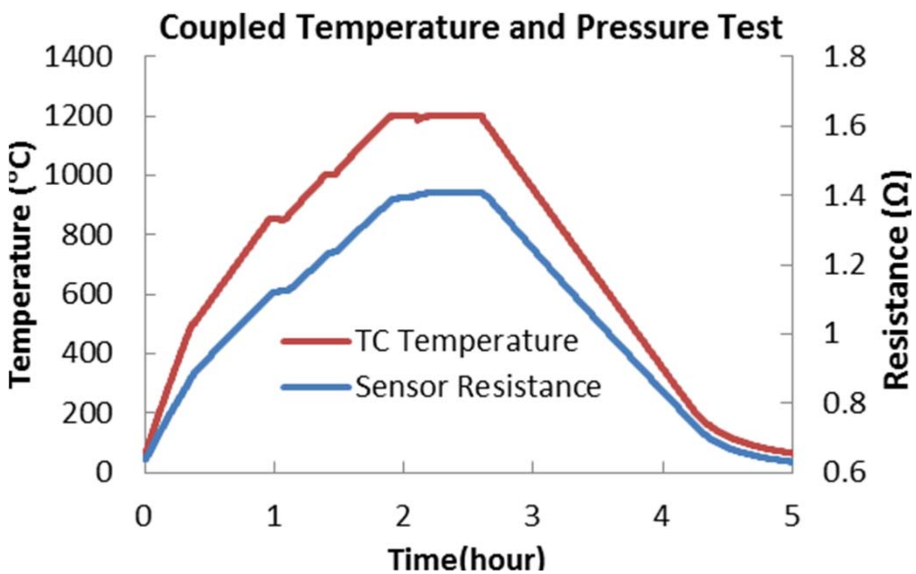
Features:

- Sapphire-sheathed ultra-high temperature sensor packaging.
- Probe is suitable for high-pressures, high-temperatures and small particles.
- Length 8.5", OD 0.375", Fitting: 1" MNPT, Length of HT MI cable: 3.5"
- "Smart" Signal conditioning electronics is capable of driving the sensor over its entire operational range and measure the response.

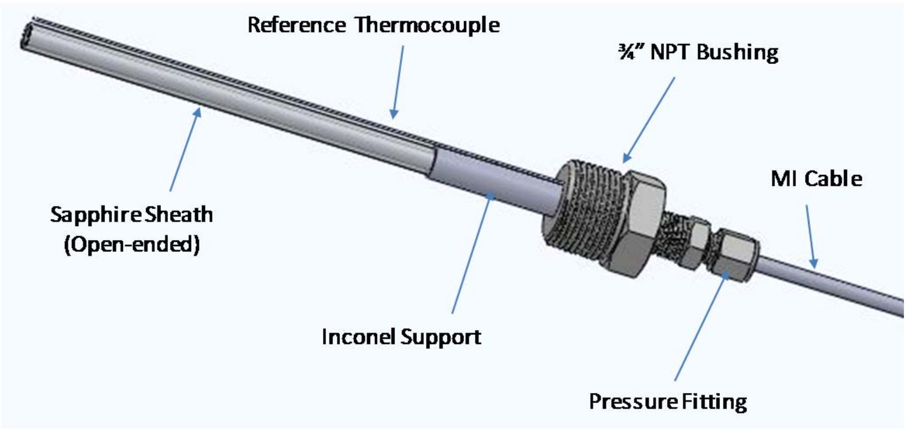
Example Sensor Data

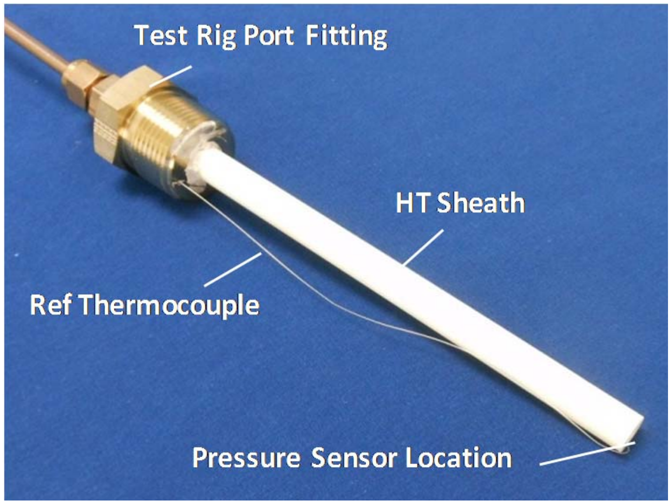


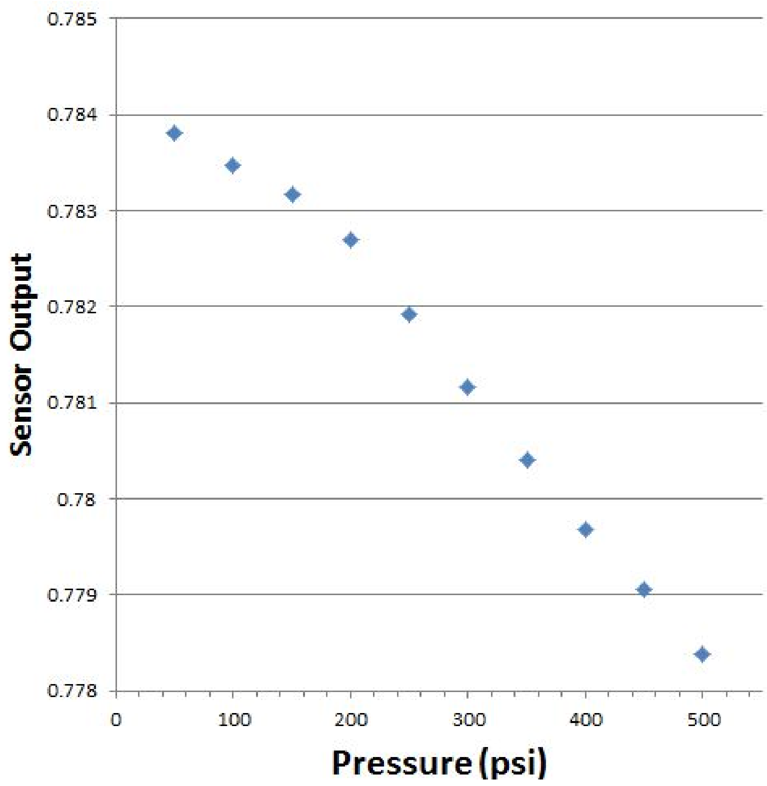




Current Prototype 1600ºC P/T Sensor
(Designed for NETL Rig Testing)

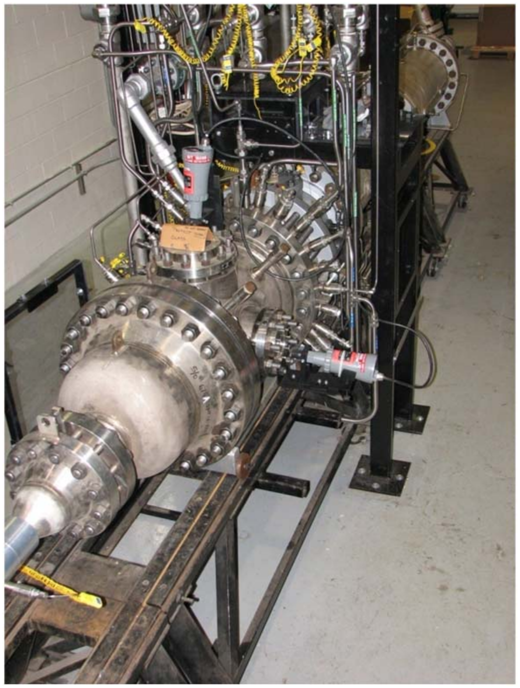


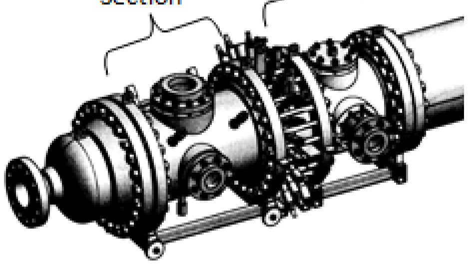




NETL Rig Testing Results (1st Round)

NETL Aerothermal Rig

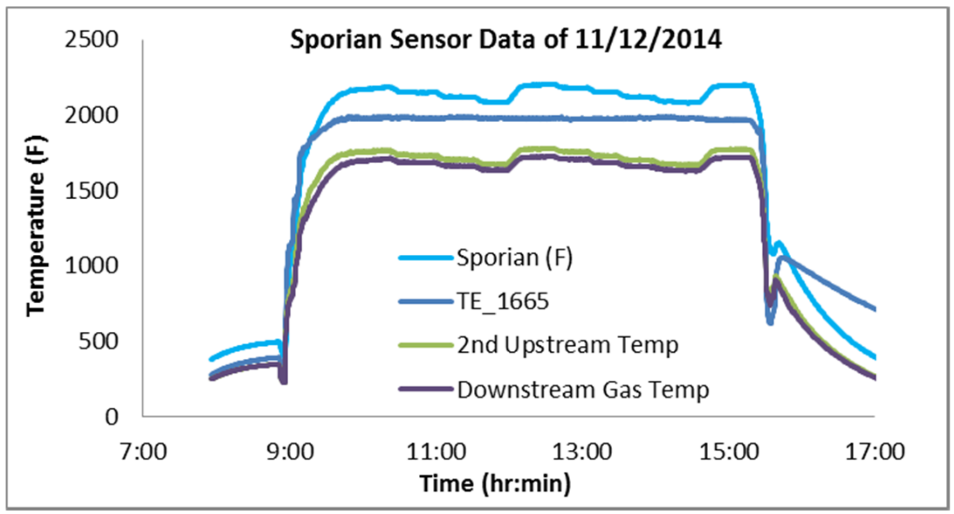




2014 Preliminary Results:

- Testing date: 10-29, 11-5, 11-12-2014
- Test cycles: Three
- Maximum T : 2000F (1100°C)
- Total duration: 30 hours
- Stable response and performance

Typical Sporian Probe Response Data



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Summary

1. Developed UV-curable/patternable precursor
2. Obtained defect-free, polymer derived, fully dense SiBCN
3. Thermal stability to 1600°C in Argon - weight loss <1.3%
4. Oxidation resistance to 1550°C in air - weight loss <2%
5. Stable mechanical strength up to 1400°C
6. UHT packaging temperature probes survived 1800C in lab and 2000F (1100°C) 30hr NETL Aerothermal Rig Test