2017 NETL

Project Review Meeting

March 20-23, 2017

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

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SBIR Phase IIA Project: DOE 12-14C
Contract #: DE-SC-0008269

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Acknowledgement

This material herein 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
  - In-House Development & Testing
  - External Testing
- Discussion/Questions

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

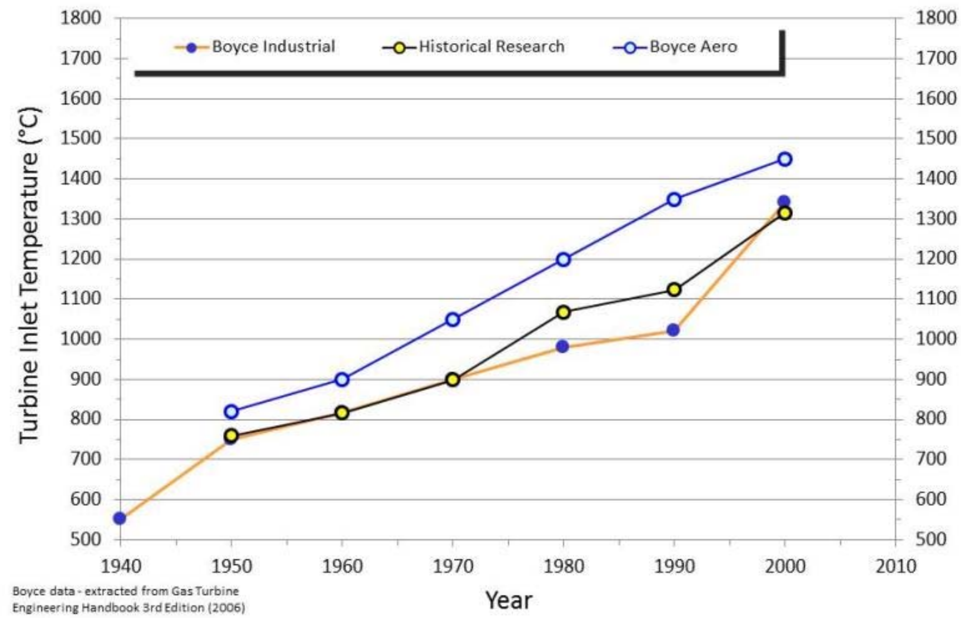
- Water Quality
- Gas Composition
- Biomedical
- Hyperspectral Imaging

Energy & Aerospace

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

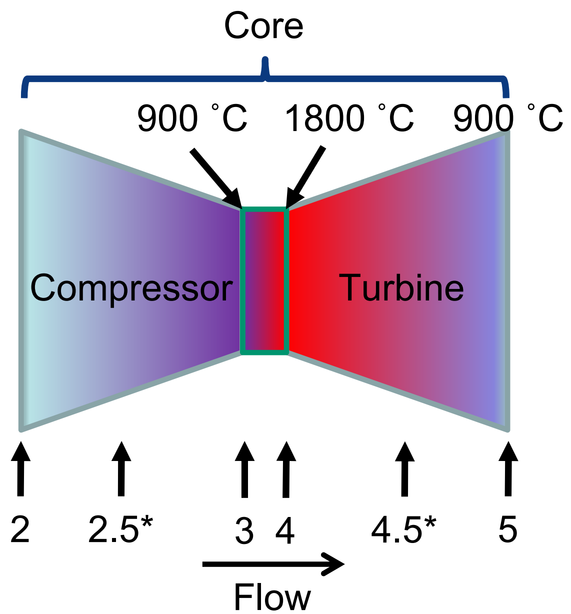
Motivation: Turbine Inlet Trends

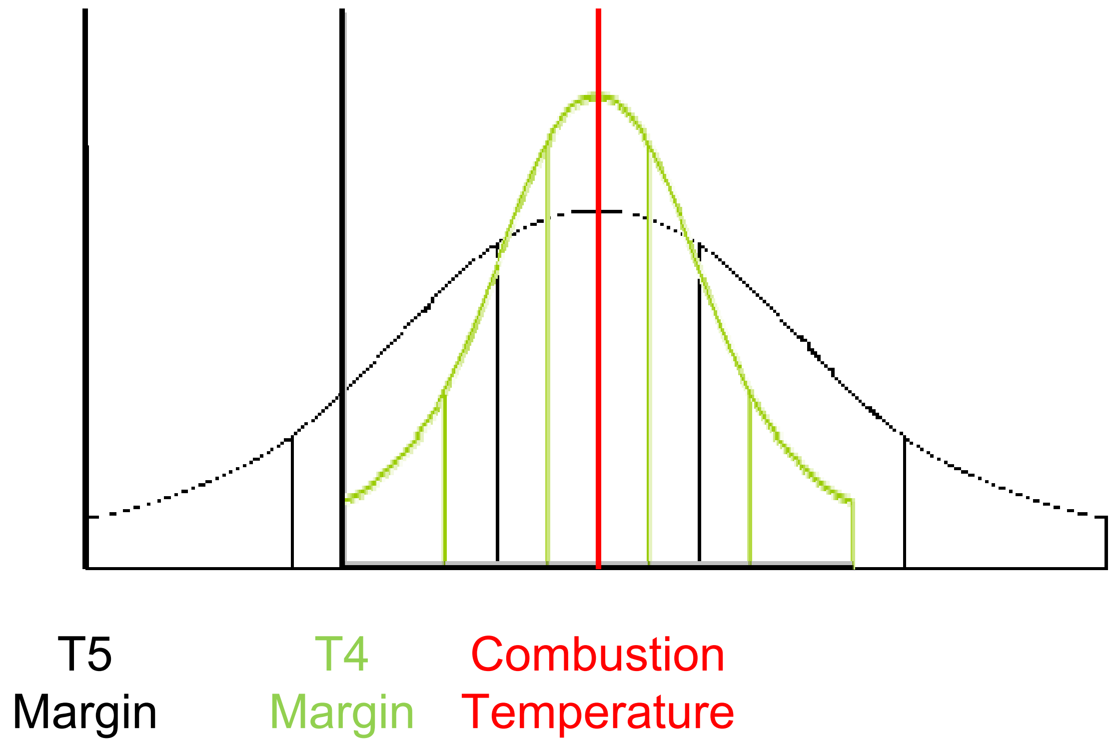
- Higher turbine efficiencies achievable at higher combustion temperatures
  - ≤1800 °C depending on fuel
- Existing temperature sensors (TCs) for combustor stage monitoring are expensive and short-lived
  - Only practical in turbine design phase
  - 50 – 100 h mean life, $5,000 per TC



Hunt RJ, "The History of the Industrial Gas Turbine." IDGTE, Morpeth, United Kingdom (2011).

Motivation: Current Opportunities – T4





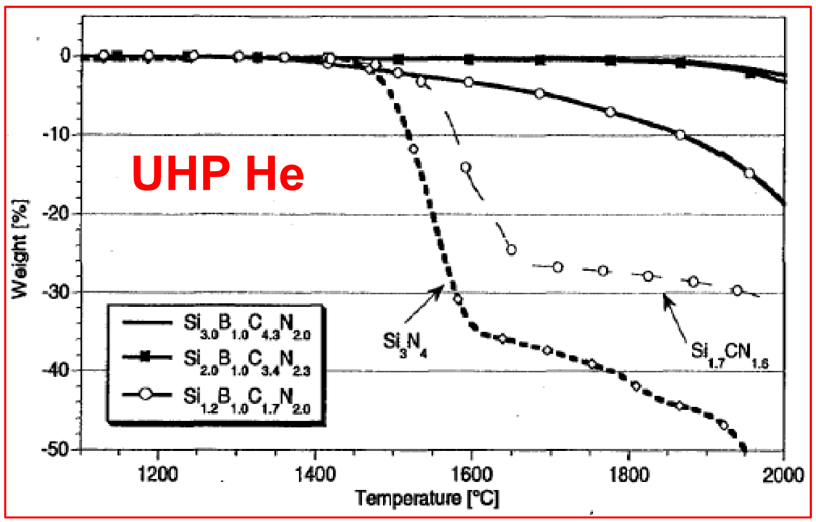
- Cheaper TCs used at turbine exhaust to infer combustor temperature – limits accuracy and reduces efficiency
- Additional efficiency gains possible with dynamic pressure measurement

Ultra-High Temperature (UHT) Ceramics

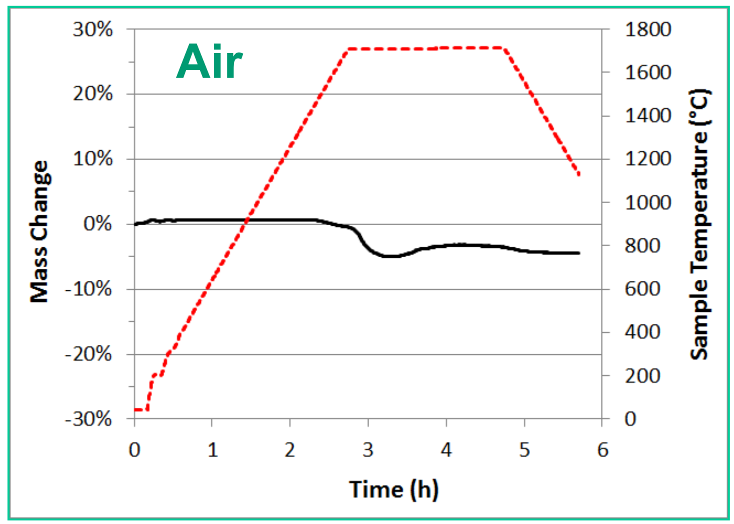
- SiCN materials show excellent HT thermo-mechanical properties.
- Sporian's proprietary polymer-derived ceramic (**PDC**) SiCN formulations dependably function as sensor materials **<1400 °C**
- Si**B**CN shows stability up to **1800 °C**

Thermogravimetric Analysis of SiBCN

Literature – Conventional Ceramic Process



Sporian – PDC Processing



Sporian PDC SiBCN – TGA: 2 h at 1700 °C in air ---> ~ 5 % loss

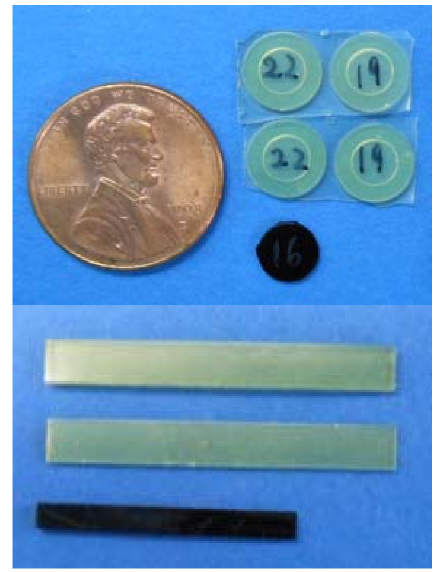
- Sensor-ready material
- Comparable with best-case literature results

Synthesis, Evaluation of SiBCN

- Synthesized B-doped PDC precursors with high stability and workability
- Patterned, molded, photo-curable (UV) polymer resins – COTS initiators
- Achieved dense SiBCN ceramic materials and defect-free parts on benchtop scale

PDC precursor

SiBCN sensor/coupons



Challenges with SiBCN PDC:

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

Sporian SiBCN PDC Materials: Phase IIA

- B-doped PDC composition focus
  - optimized precursor process
  - ‘green’ part fabrication and handling
  - fully-dense part and device processing
- Increased thermal stability under application-relevant conditions (tested in-house)
- Evaluated mechanical and chemical properties at increased temperatures
- Incorporate PDC best results into packages for 1800 °C temperature and 1600 °C pressure sensor suites

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Prior & Related PDC Work <1400 °C

Pressure Sensor Performance Benchmarks

Specification	PIWG* Target (2017)	Sporian Capability
Pressure Range (**psi**)	25-750	15-1000
Operation Temperature (**°F**)	0-1400	75-2500
Natural Frequency	>100 kHz	TBD
Internally Compensated Temp. Range (**°F**)	0-1400	75-2500
Probe Length (**in**)	<1.25	1 – 10
Diameter (**in**)	0.062 - 0.19	0.2
Sensitivity/Combined Uncertainties @ STP	≤ 1% FS	≤ 1% FS
Sensor Input (**VDC**)	5-10	12

***Propulsion Instrumentation Working Group**

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Current Effort Progress Update: Phase IIA

- Extend Sporian's existing ceramic sensor materials to UHT: 1600 – 1800 °C
- Build on PII tasks for sensor packaging and 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

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OEM Collaboration/Coordination

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

Rolls-Royce

SwRI®

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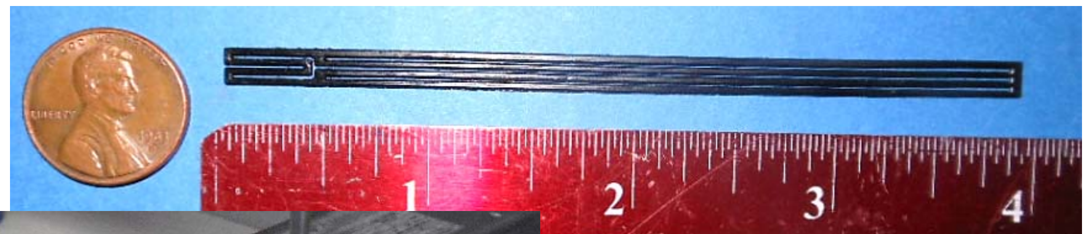
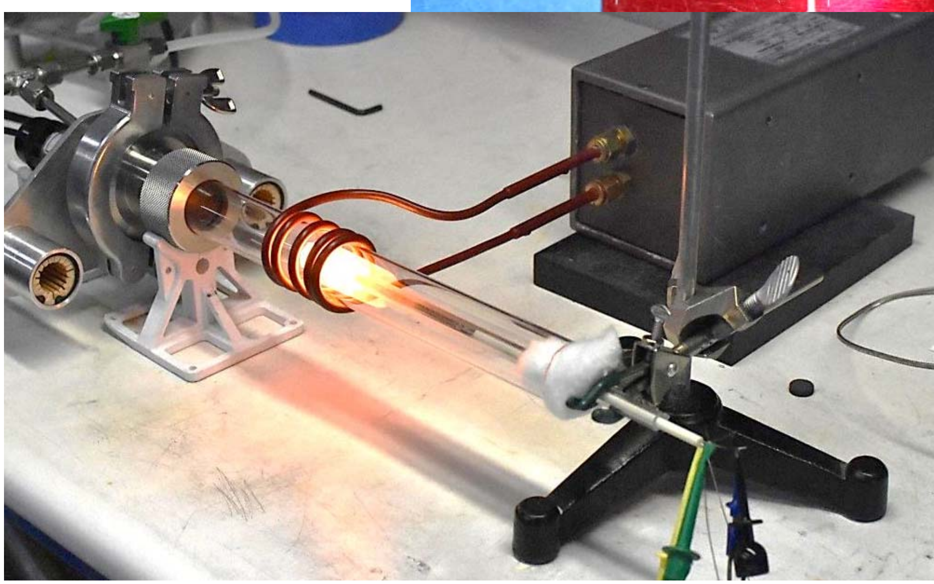
In-House Development & Testing

- SiBCN Sensors
- Alternative UHT Sensors: $MoSi_2$

SiBCN UHT Sensor Elements

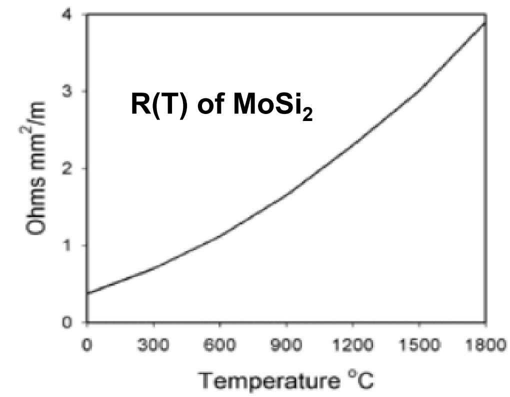
- Formulation and curing processes tuned to produce desired geometry
- Post-curing processing adjusted based on TGA experiments done on cured polymers
- Long-format sensor elements produced with high repeatability
  - **Advantage**: low-temp interconnects can be used





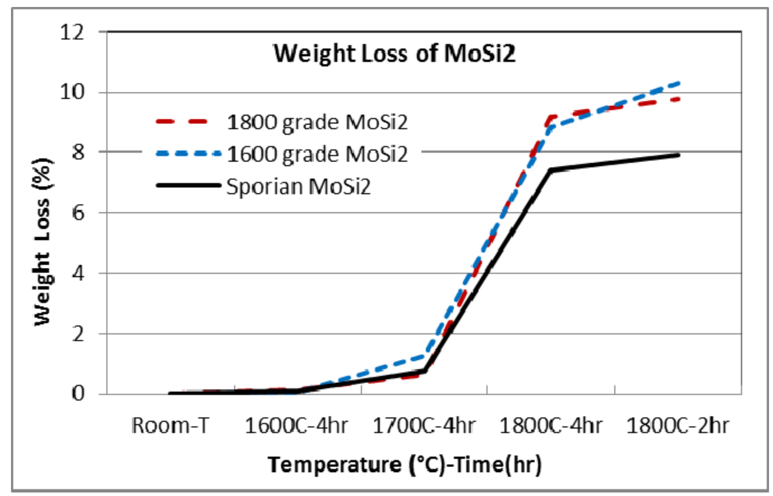
- In-house experiments using induction heater:
  - **1330 °C, sensor and interconnect stable**
- Optimizing fabrication and assembly to facilitate long-duration soak and rapid cycle testing in lab furnaces

UHT $MoSi_2$–based Sensors



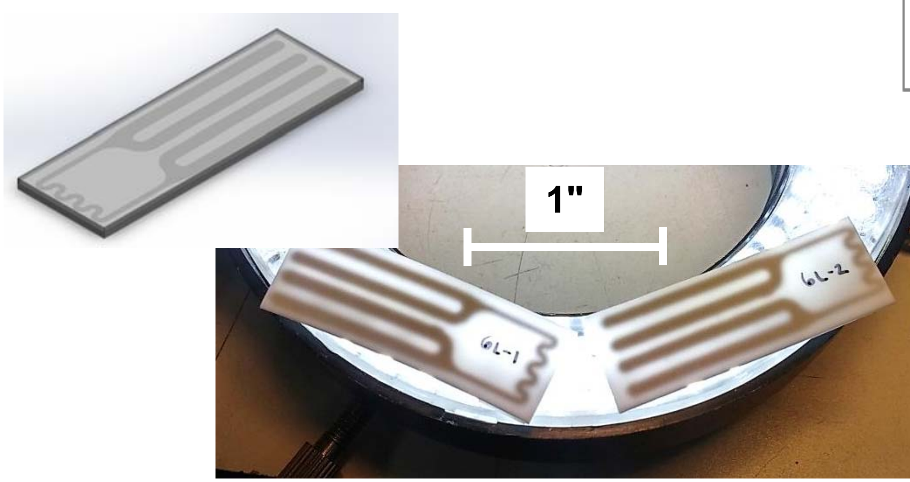
Sporian $MoSi_2$ Features:

- HTCC tapes and thick-film pastes used to create embedded sensors
- Thermal stability at 1800 °C
- Comparable to commercially available UHT grade $MoSi_2$ (heater elements)



Sporian Prototypes:

- Compatible CTE with HTCC substrates and tapes already in use



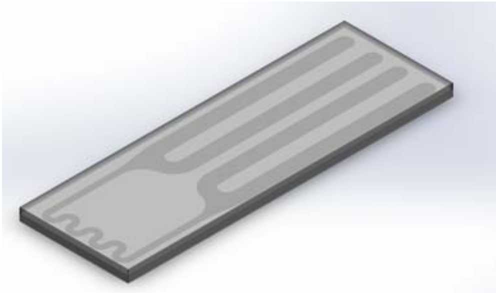
$MoSi_2$ Challenges:

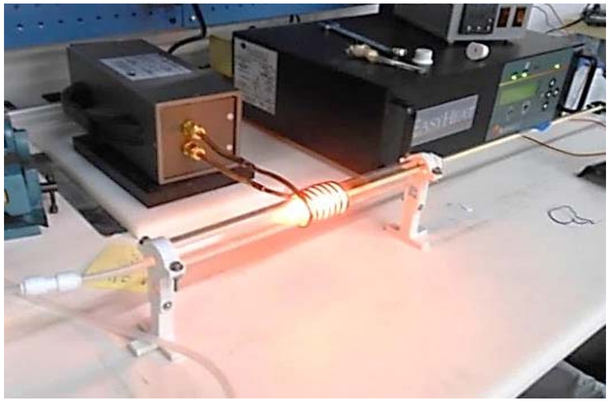
- Chemical stability in application
- Optimizing embedding layers for sensor efficiency, accuracy
- Interconnects to sensor electronics

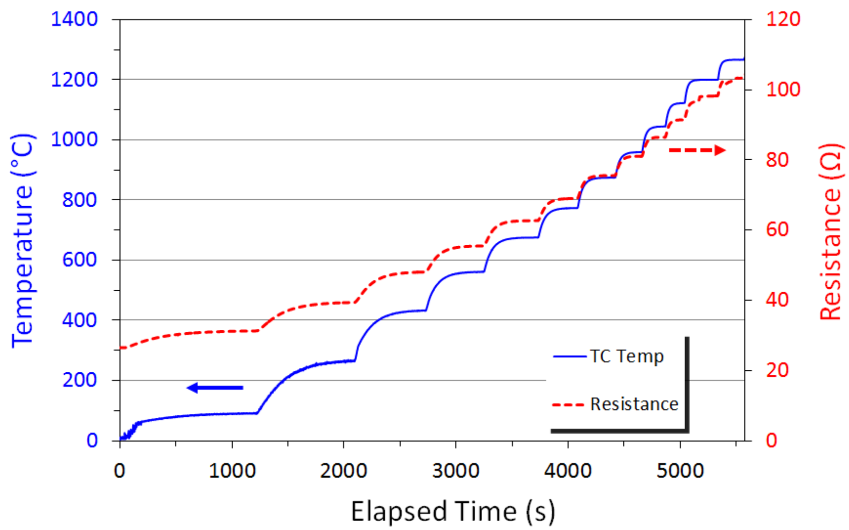
UHT $MoSi_2$–based Sensors

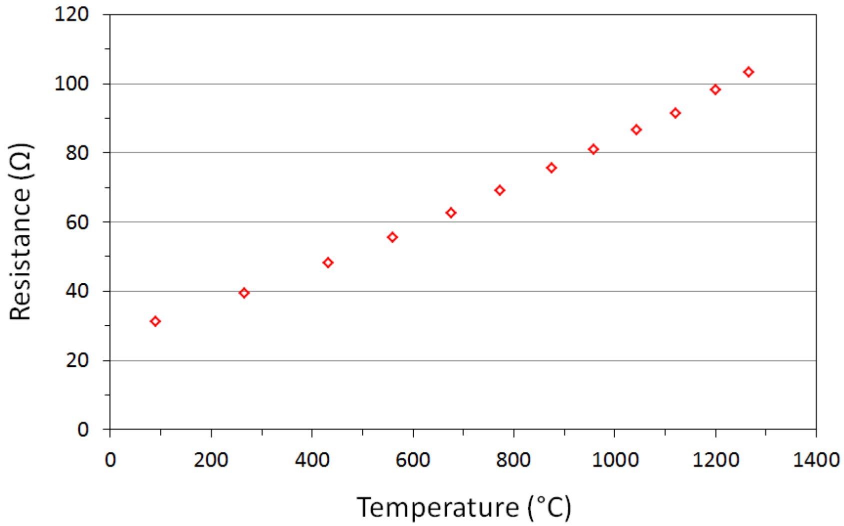
In-House Testing:

- Induction Heater
- Stepped heating
- Cycling
- Thermal Shock









Stepped Heating:

- Sensor tracks excellently with system thermocouple
- Pseudo-linear response of sensor resistance vs. temperature

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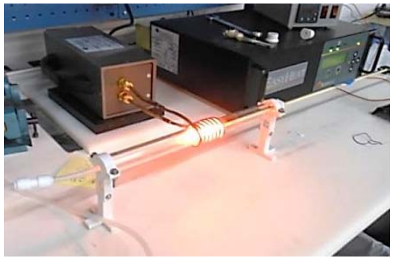
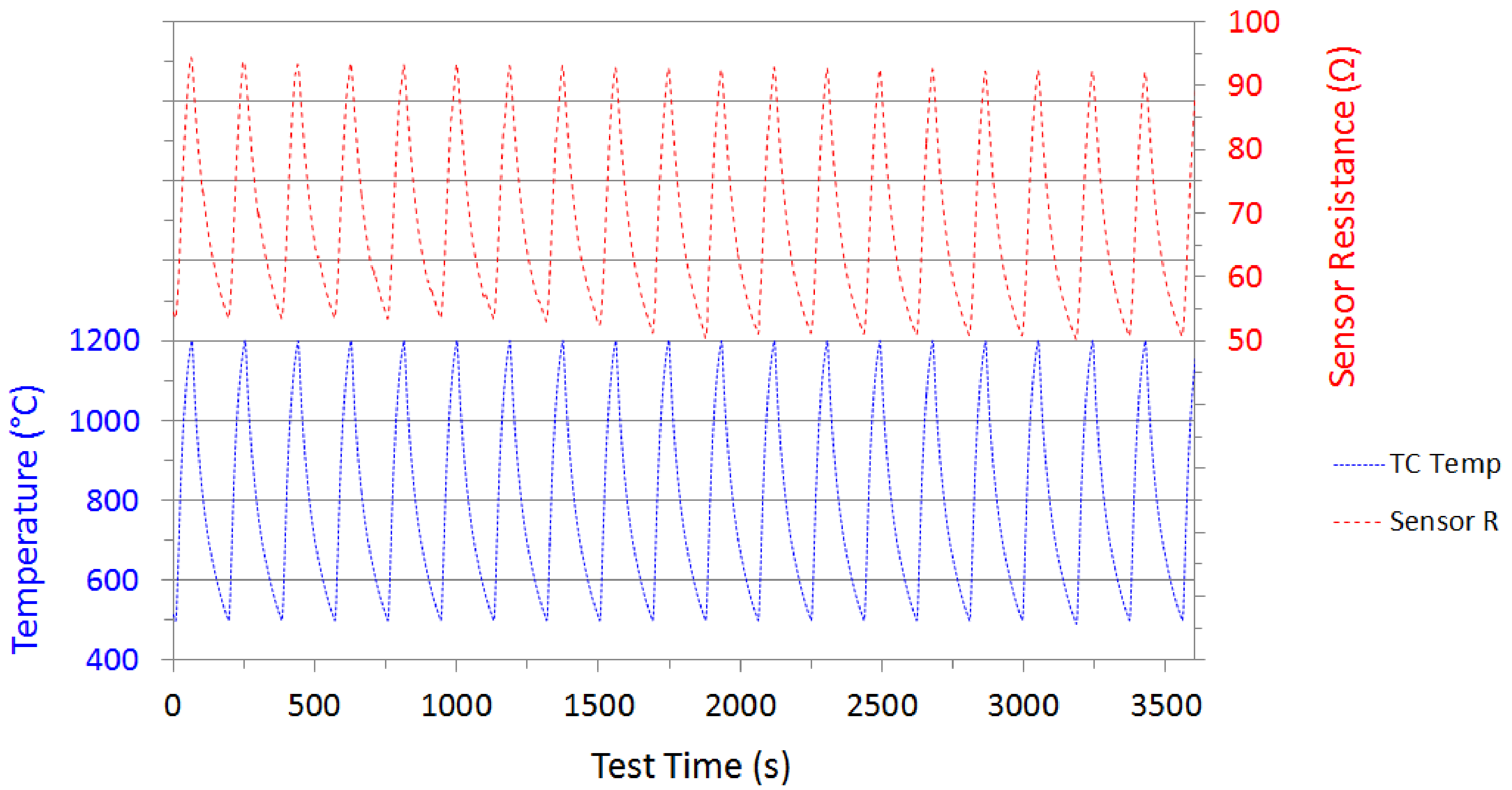
UHT $MoSi_2$–based Sensors



Thermal Cycling and Shock:

- Sensor response is reliable over 20+ cycles: 500 – 1200 °C
- Sensor and package hold up under thermal shock



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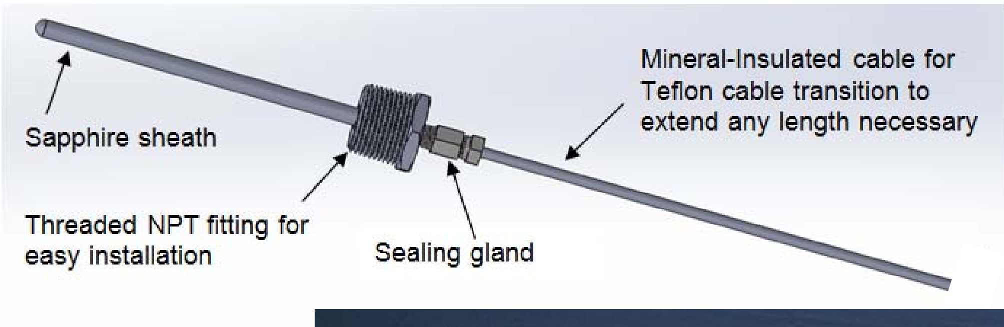
External Testing

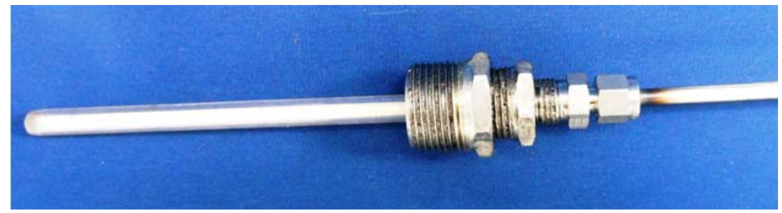
- SwRI Combustor
- Rolls-Royce Combustors (planning)

Prototype 1800 °C Temperature Sensor

Designed for NETL Aerothermal Rig and SwRI Rig Testing

Sporian Sensor Packaging Design and Probe Assembly





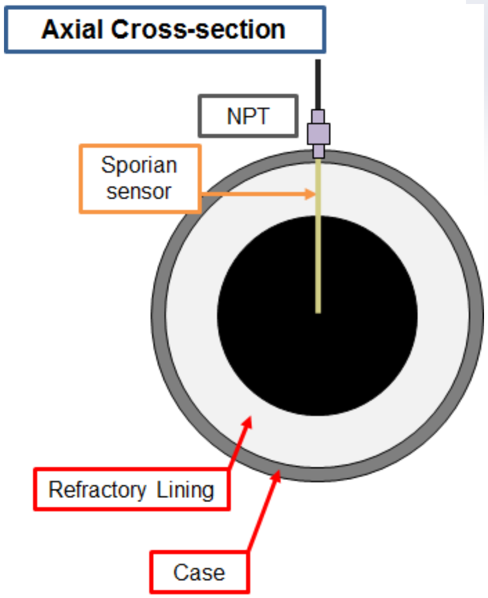
'Smart' Signal Conditioning Electronics

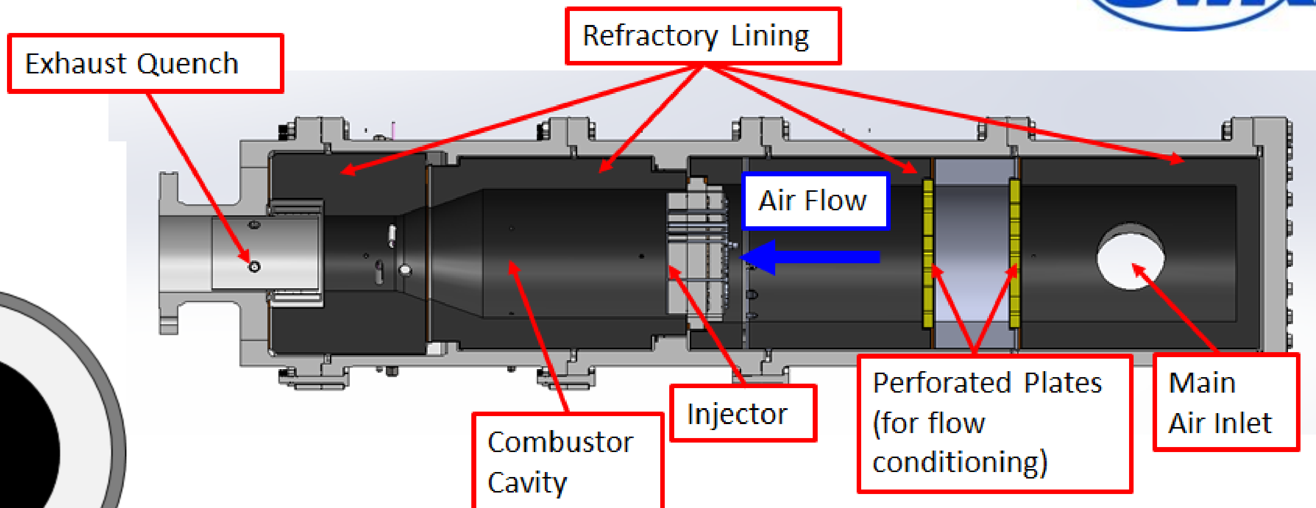
Features:

- Sapphire-sheathed UHT sensor packaging.
- Probe suitable for high pressures, high temperatures and particulate exposure.
- Sensor/package stable at **1800 °C for >1 h**: **in-house test**
- "Smart" signal conditioning electronics can drive the sensor over its entire operational range and measure the response.

SwRI PHTFF Rig Testing – Results So Far

- Pressurized High-Temperature Flow Facility (PHTFF)
- First Test Period: **January 25 – February 2, 2017**





PHTFF Features:

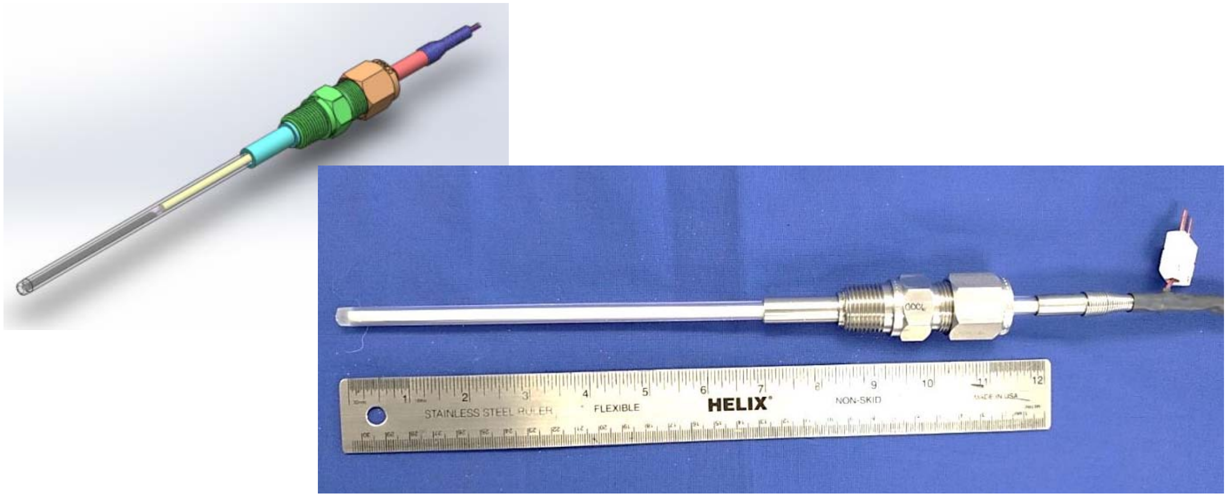
- 1.9 MW electric heater pre-heats combustor air
- Natural gas fuel
- New injector design commissioned for test

Sporian Sensor Testing:

- Run at ambient pressure due to compressor availability issues
- Test profile set up with full access to rig's hot section
- Evaluate UHT Temp Sensors: estimated ~1800 °C **near injector**

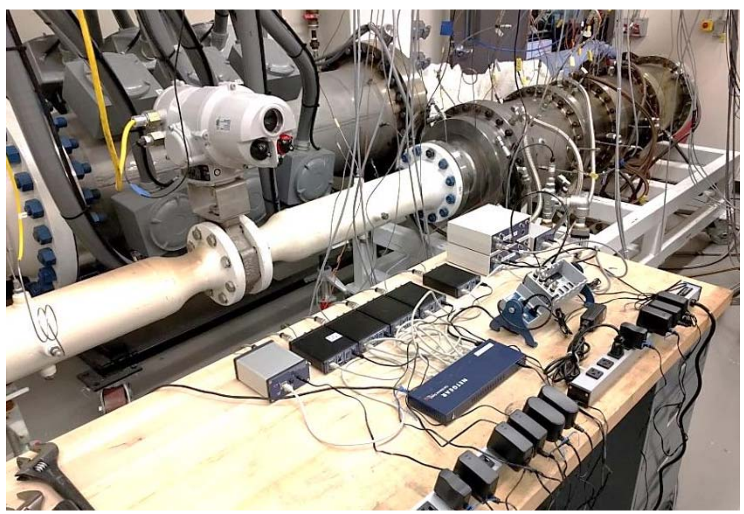
SwRI PHTFF Rig Testing – Results So Far

Sensors and Packages: 4x UHT Temp Sensors



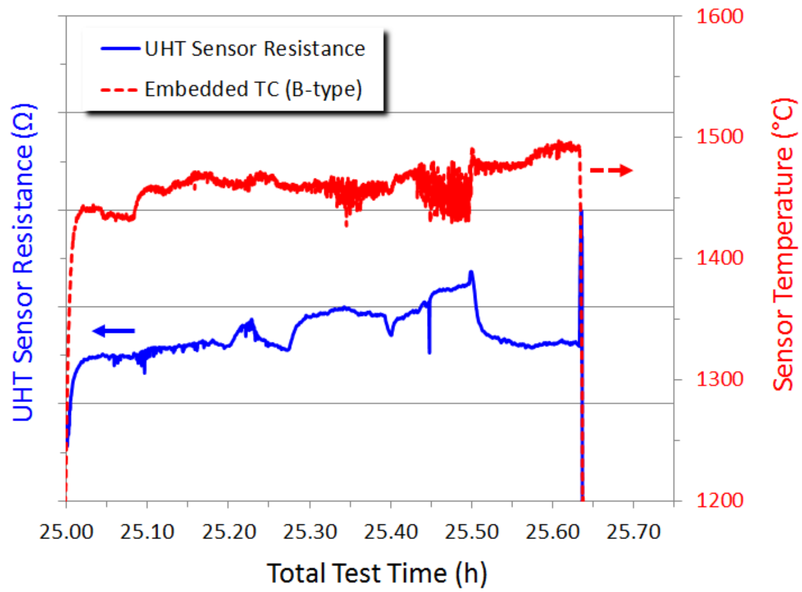
Integrated B-type TC for accurate calibration and response comparison

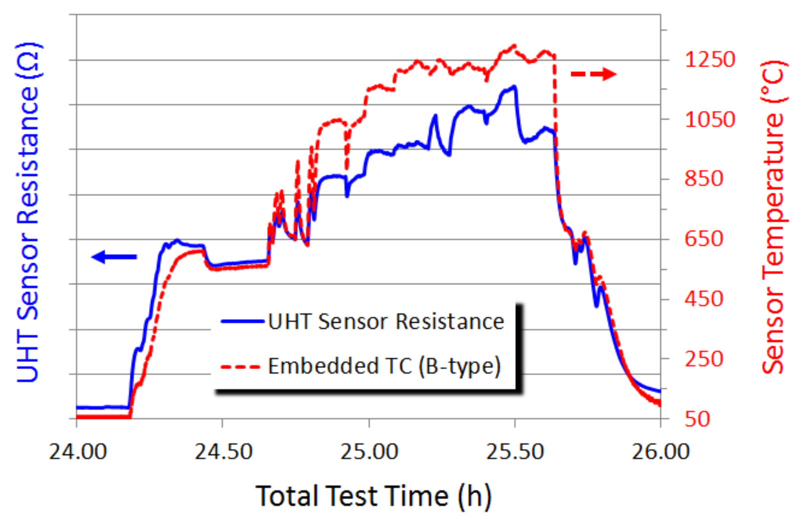
Installed at SwRI, driven by Sporian electronics and DAQ



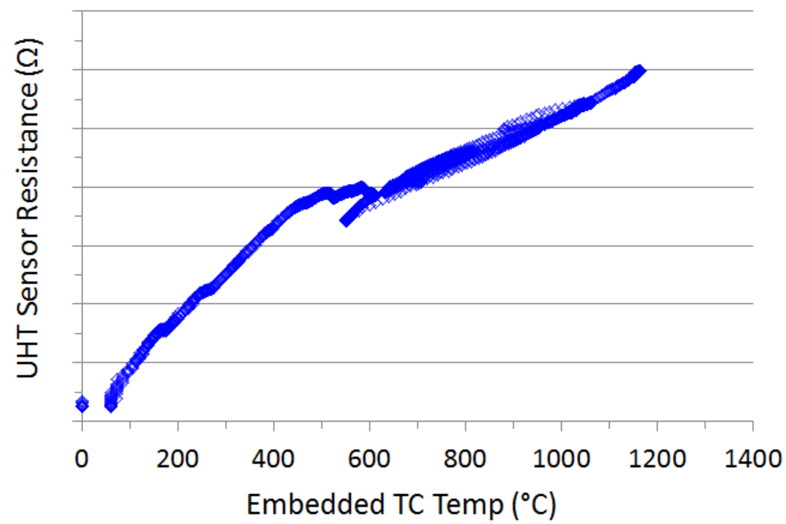
SwRI PHTFF Rig Testing – Results So Far

Test Data





- Max temp reached 1500 °C
- Sensors tracked with embedded TCs
  - Linear response across broad Temp range
- Observed temps from 1200 – 1500 °C between 4x sensors, mapping out combustor interior



SwRI PHTFF Rig Testing – Results So Far

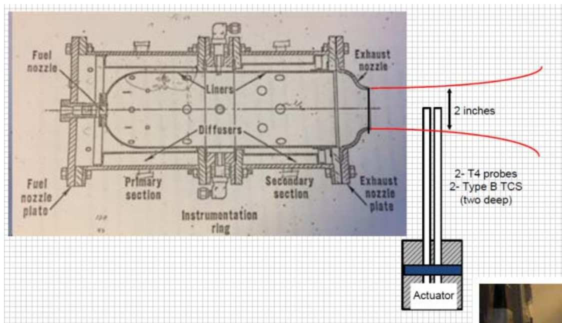
Testing Status: Pending Restart in Summer

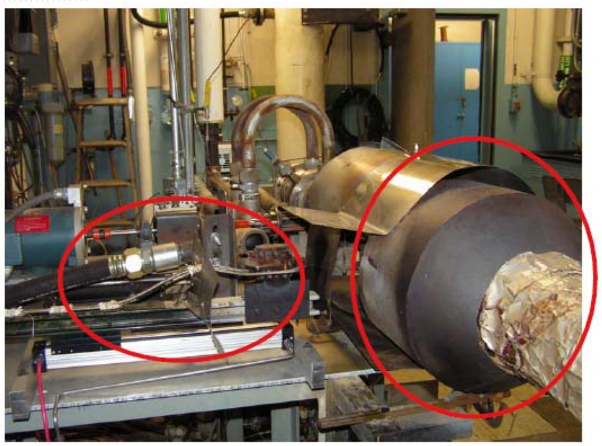
- After ~8 h of total test time, 1.9 MW heater on SwRI rig failed
- All Sporian sensors survived commissioning, testing, heater failure, and troubleshooting
- Testing to resume in May/June during re-commissioning of repaired heater
  - Anticipate 30 h of test profile – fast ramps, fast cools, and multi-hour soaks at HT
  - SwRI estimates repaired configuration will reach 1800 °C

Upcoming Sensor Testing in Phase IIA

Rolls-Royce – June, 2017

- HT cycling: 1200 °C – 1500 cycles
- UHT performance: Flametube – pressurized, up to 1650 °C
- Sporian UHT T sensors (Cycling) and UHT P/T sensor (Flametube)





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Summary

- Photo-curable PDC precursor materials and processes produced long-form sensor elements for prototyping and testing
- Conducted high-temperature combustion testing of 4x Sporian UHT temperature sensors in PHTFF rig
  - Test profile abbreviated due to rig heater failure
- SwRI External Testing will continue in May/June during commissioning of new combustor rigs (as piggyback test)
  - Temps up to 1800 °C possible
- RR External Testing in planning stage for June
  - HT Cycling rig
  - UHT Flametube rig

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Thank you for your attention!

Questions?

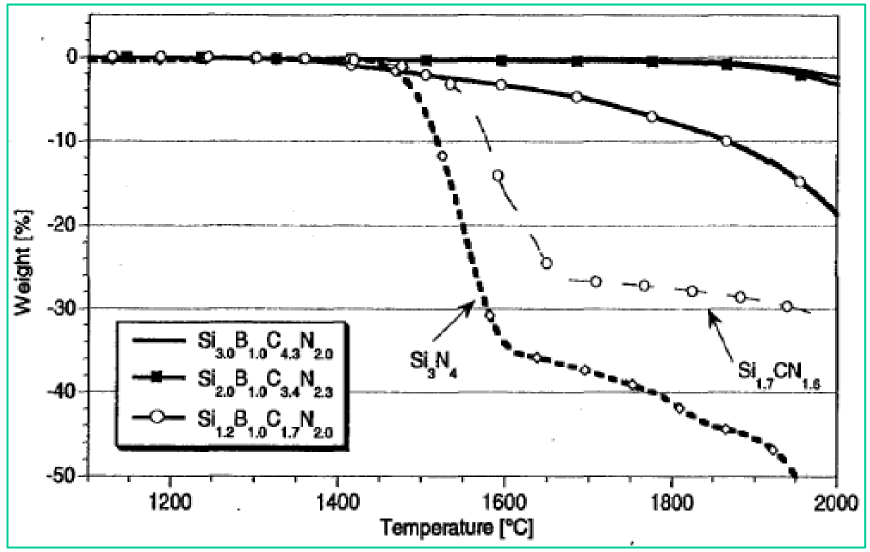
Ultra-High Temperature (UHT) Ceramics

- SiCN has shown excellent HT thermo-mechanical properties.
- Sporian's proprietary polymer-derived ceramic (**PDC**) SiCN formulations dependably function as sensor materials **<1400 °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)



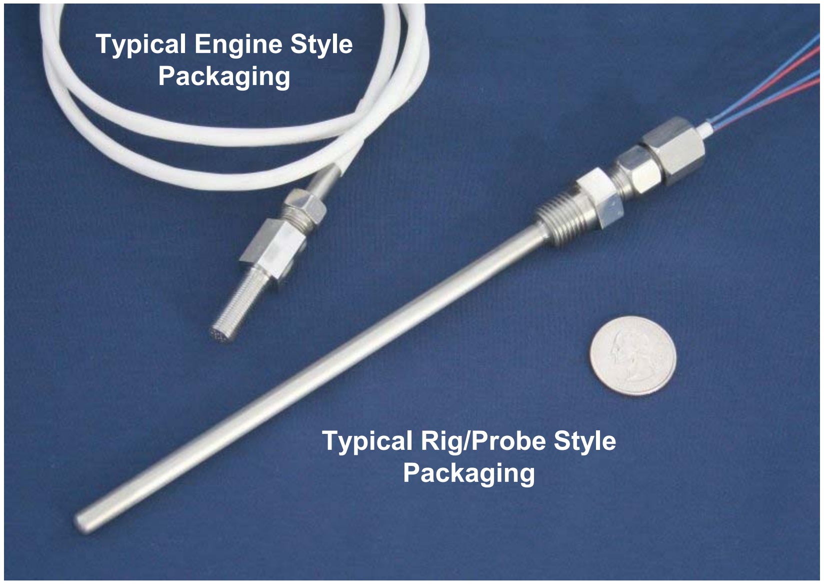
Challenges:

- 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

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 effort focusing on extending capabilities to 1800 °C**



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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 Rig	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.**

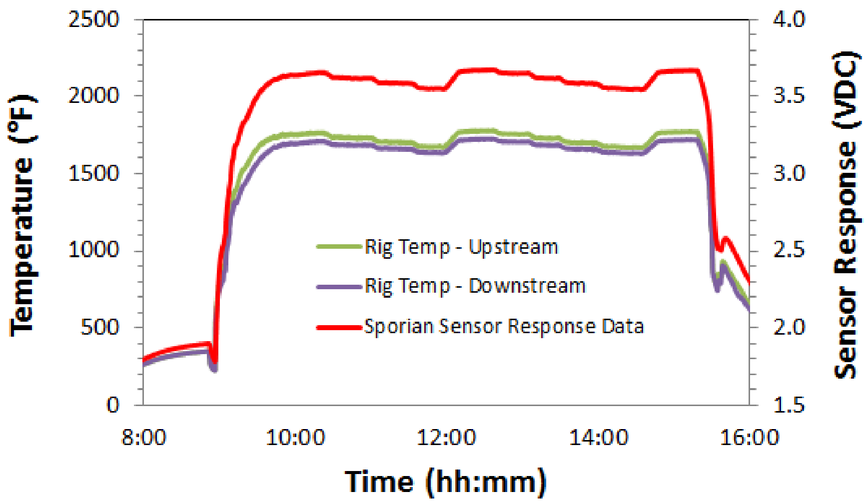
** **Proprietary**

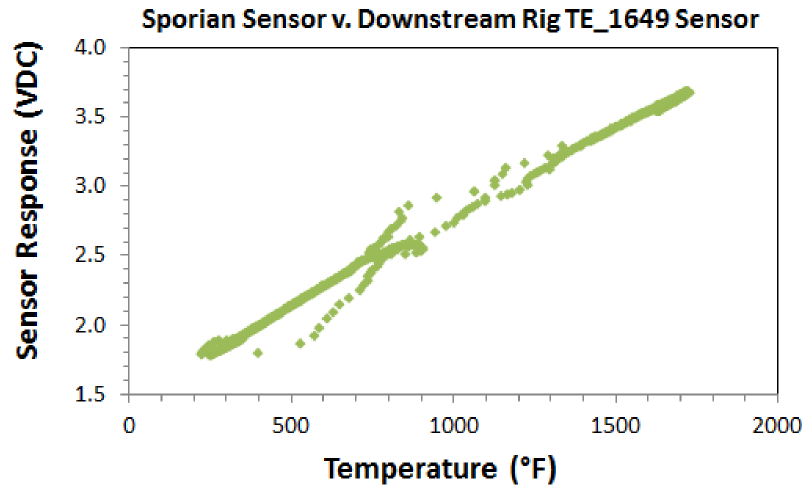
Prior & Related PDC Work <1400 °C

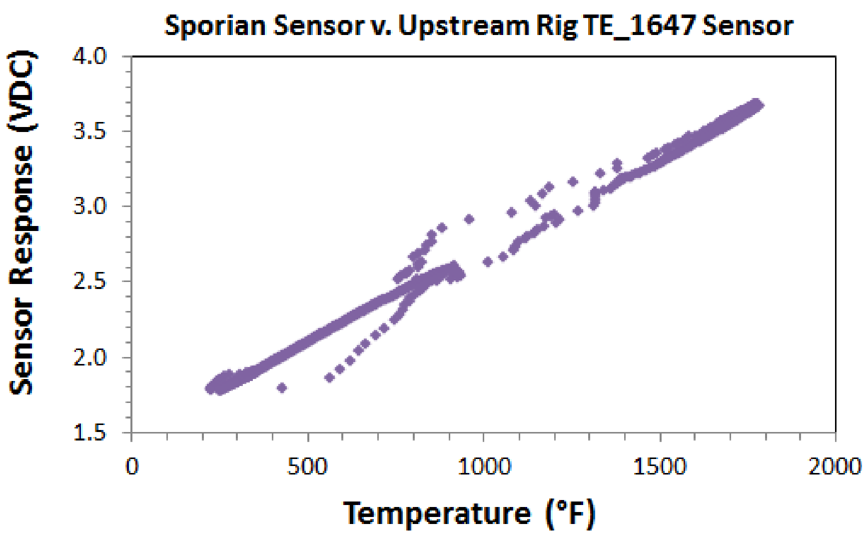
Features, Advantages, and Benefits

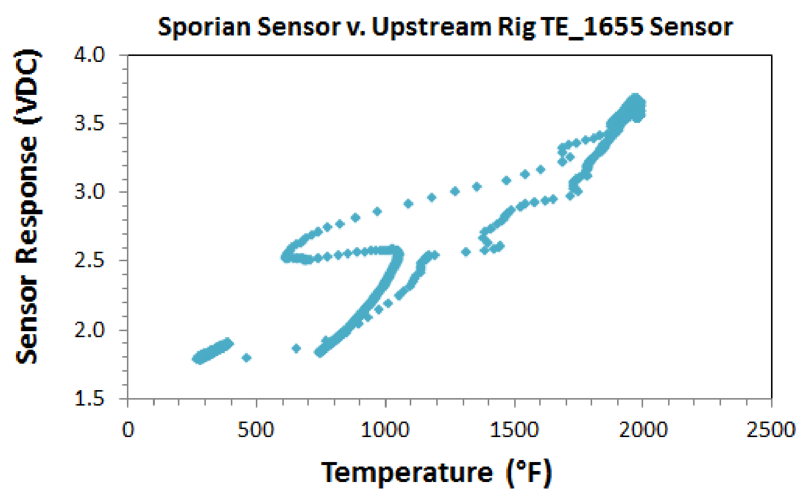
Features	Advantages	Benefits
Polymer-derived ceramic (**PDC**) materials	• Operating temperature >1000 °C • **No** liquid cooling or fiber routing req'd! • Stable at pressures ≥1000 psia • High oxidation/corrosion resistance • Thermal shock resistance	• Lower weight, smaller size • Lower cost, low-maintenance • 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 (low density) • Improved dynamic response • Reduced latency • **Avoid failure mechanism**
Electronics	• Compatible with existing controls & CBM	• Lower cost

NETL Aerothermal Rig Testing Results



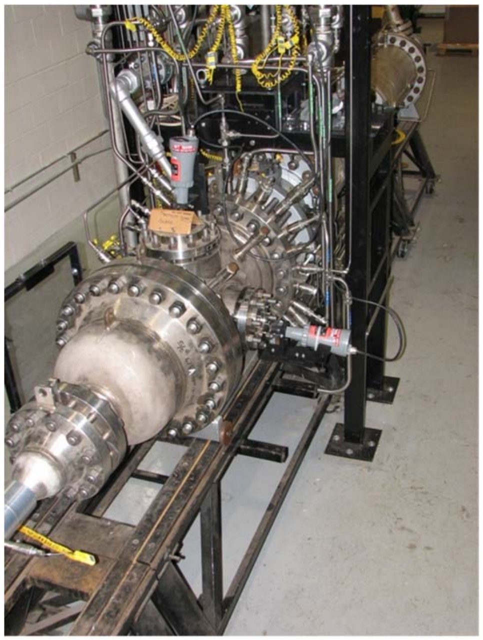


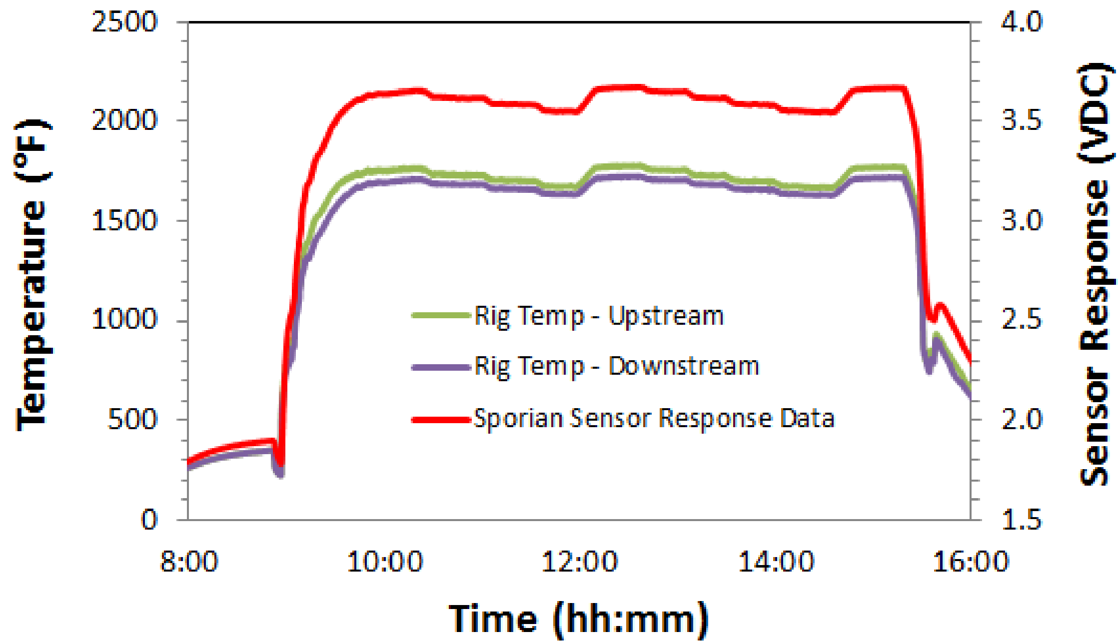




NETL Rig Testing Results

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**

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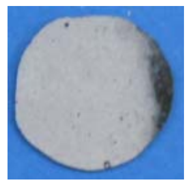
Phase II In Brief

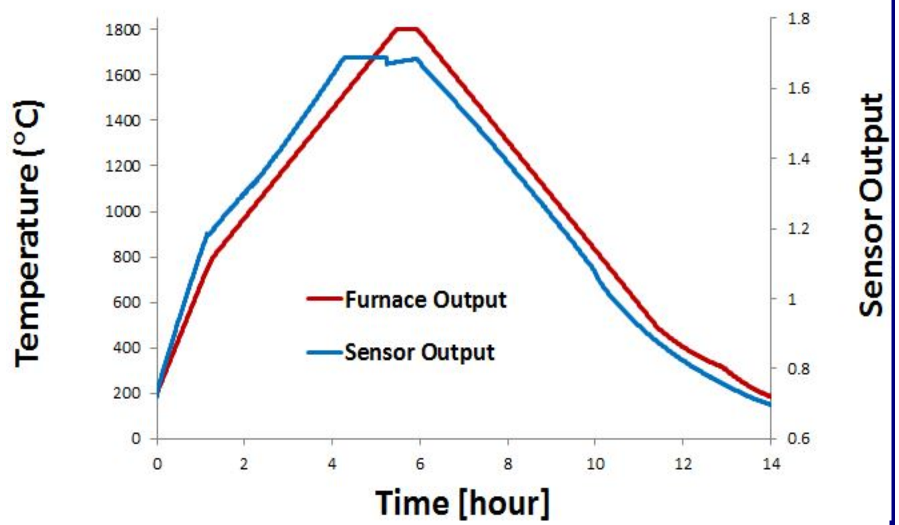
1700°C-4hr
Argon

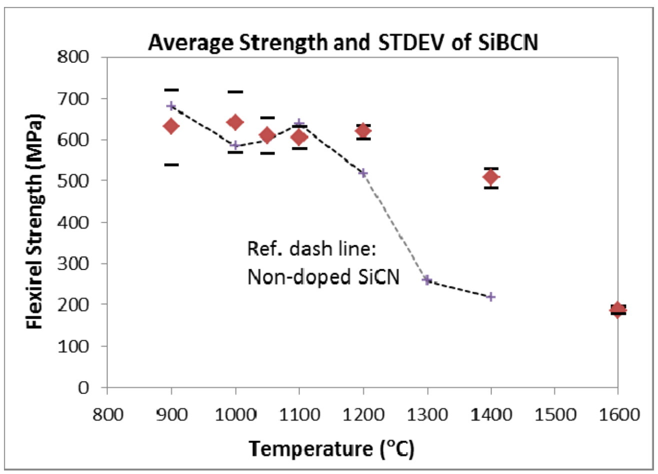
12 % loss

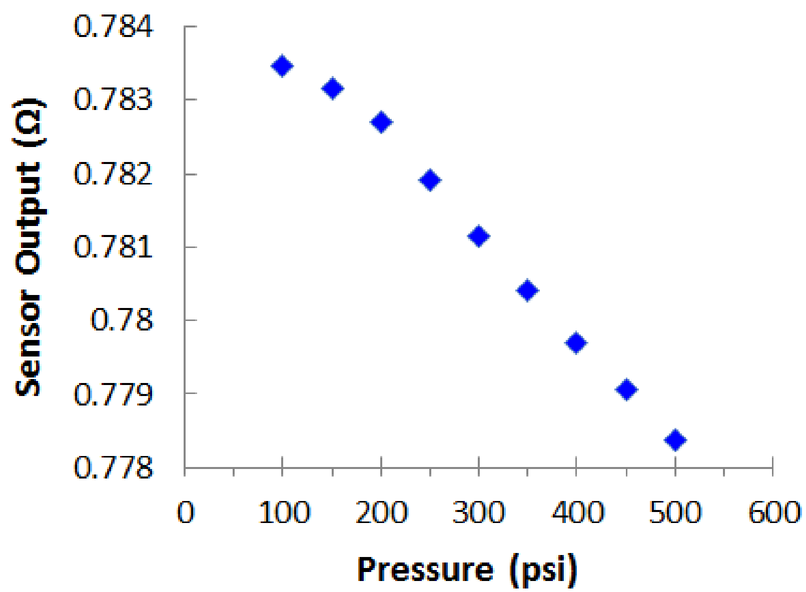
1600°C-4hr
Air



~3 % loss







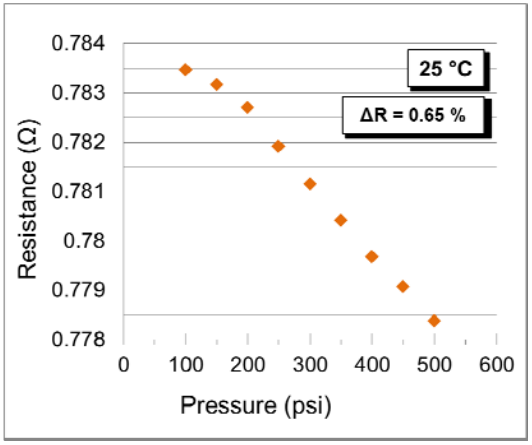
- Developed sensors and packaging capable of 1800 °C operation
- Conducted pressure response testing on 1600 °C-capable probe

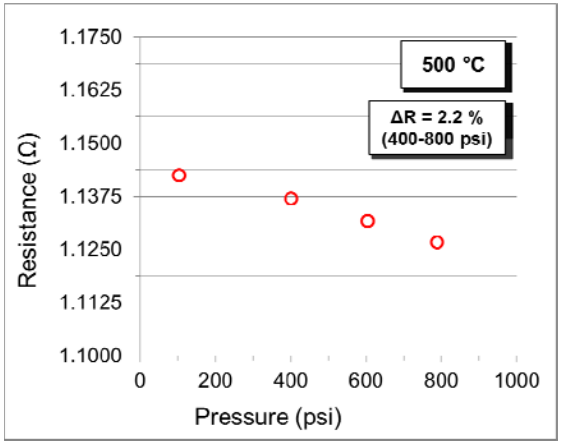
HT Testing of Sporian Prototypes

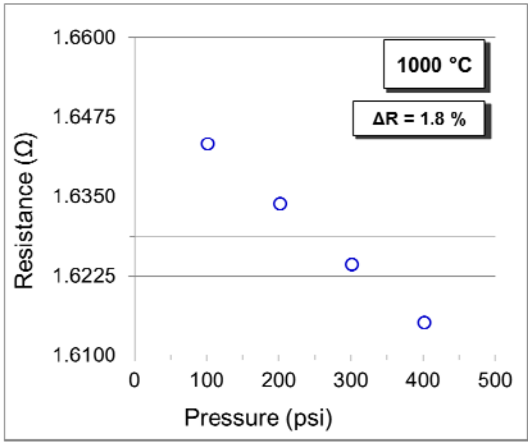
Test under Pressure at Temp:

- Exterior reference TC to track temperature
- 25, 500, and 1000 °C
- 15 – 800 psi
- Sensor response increased with increasing pressure

Sporian In-House Pressure Test







SiBCN UHT Sensor Elements

- In-house experiments using induction heater:
  - **1330 °C, sensor and interconnect held**
  - **HT drift ~15 %, partly due to induction field, needs improvement**
- Optimizing fabrication and assembly to facilitate long-duration soak and rapid cycle testing in lab furnaces