U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

ASI Advanced Sensors and Instrumentation

Thermometry

CT-24IN070205

3:00PM – 3:20PM EST

Wednesday, Nov 6th, 2024

Advanced Sensors and Instrumentation (ASI) Annual Program Webinar

November 4, 6-7, 2024

PI: Richard Skifton, PhD; Co-PI: Brian Jaques, PhD

Idaho National Laboratory/Boise State University

Project Overview

Nuclear Thermocouple Technology:

- Complete characterization of **advanced insulation materials for use in advanced nuclear sensors for high temperature applications** – This task focuses on the development of high temperature (> 2000 ℃) insultation materials for nuclear instrumentation (e.g., thermocouple, ultrasound thermometry, SPND, etc.). The main need is the manufacturability and/or advance manufacturing capabilities in such non-oxide materials as aluminum nitride (AlN), silicon carbide (SiC), and others, for example. The high temperature materials allow for sensors to reach higher temperatures themselves without adding drift or impurities to the signal. The main tests will be performed on thermocouples, but the advanced insulations apply to other sensors, as well.
- In FY24 R&D activities were carried out in the following technical areas:
  - M3CT-24IN0702051-(Carryover) Journal article with HTIR-TC testing results using the different heat treatment methods
  - M3CT-24IN0702052-Characterization of insulation materials for use in advanced nuclear sensors for high temperature applications

Personnel:

- PI: Richard Skifton, PhD, Idaho National Laboratory
- CO-PI: Brian Jaques, PhD, Boise State University

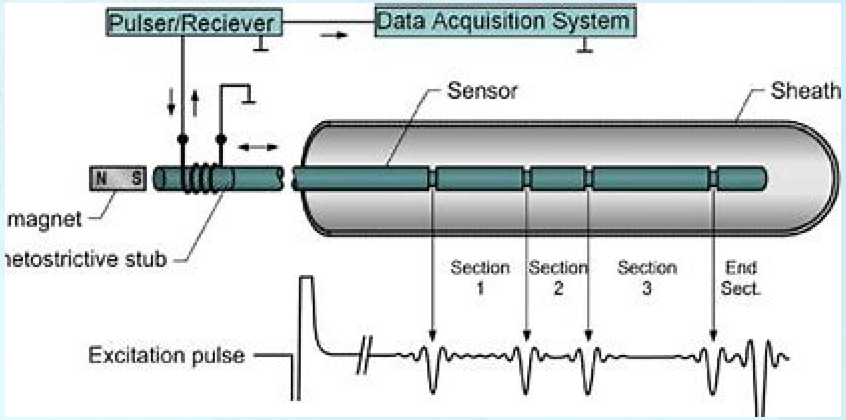
Project Overview

Schedule:

		2024											
Milestone / Activity	**STI**	**Oct**	**Nov**	**Dec**	**Jan**	**Feb**	**Mar**	**Apr**	**May**	**Jun**	**Jul**	**Aug**	**Sep**
M3CT-24IN0702051-(Carryover) Journal article with HTIR-TC testing results using the different heat treatment methods	Yes	M3											
M3CT-24IN0702052-Characterization of insulation materials for use in advanced nuclear sensors for high temperature applications	Yes												M3

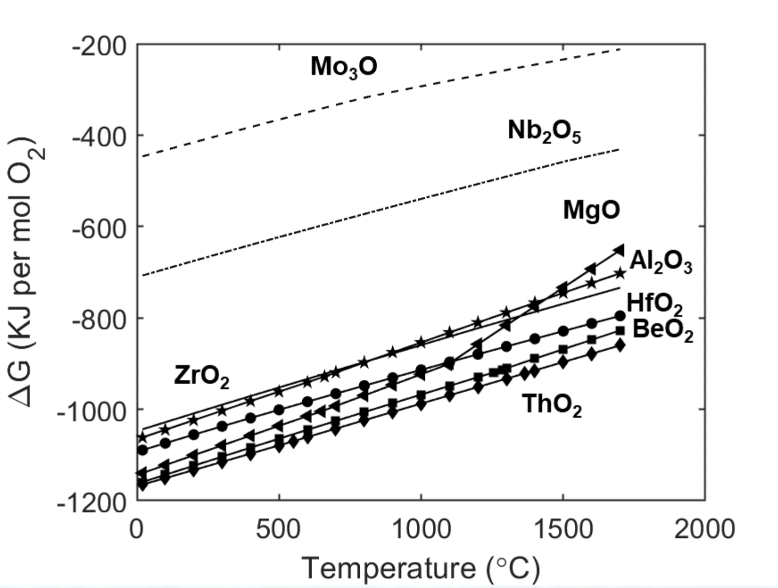
Technology Impact

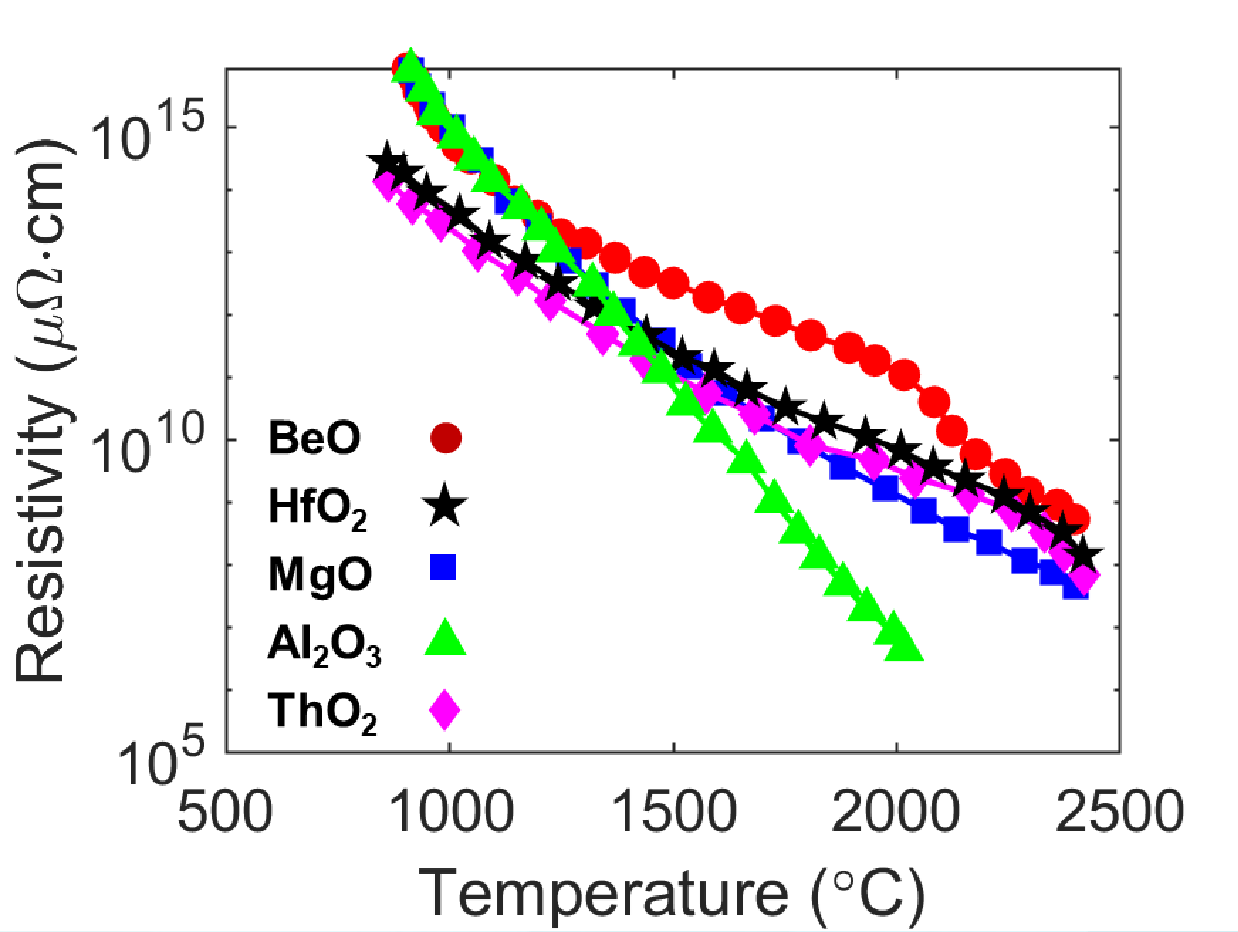
- Next generation reactors are pushing temperatures higher than ever (> 1600 ℃)
- This merits the increase in material capabilities, namely, ceramic insulators that hold most high temperature sensors in their designed position
- This work applies to thermocouples, SPNDs, ultrasound thermometry, and others



Traditional Thermocouple Insulators

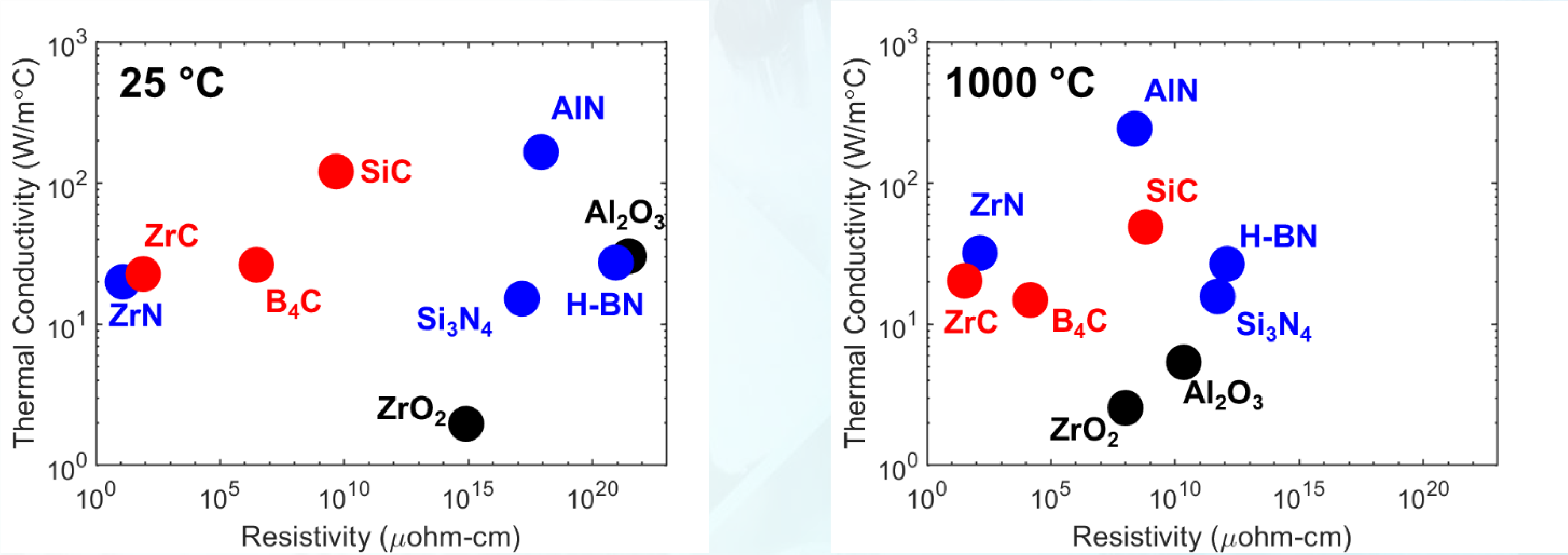
Ceramic Insulator	Interaction with Niobium Onset Temperature (°C)	Interaction with Molybdenum Onset Temperature (°C)	Melting point (°C)
HfO_2	1300	Not observed	2758
Al_2O_3	1450	Not observed	2072
BeO	1600	Not observed	2578
MgO	1800	Not observed	2852
ThO_2	1800	Not observed	3390
ZrO_2	-	-	2715





- **The electrical resistivity of alumina limits operation of the HTIR-TCs above 1600 °C.**

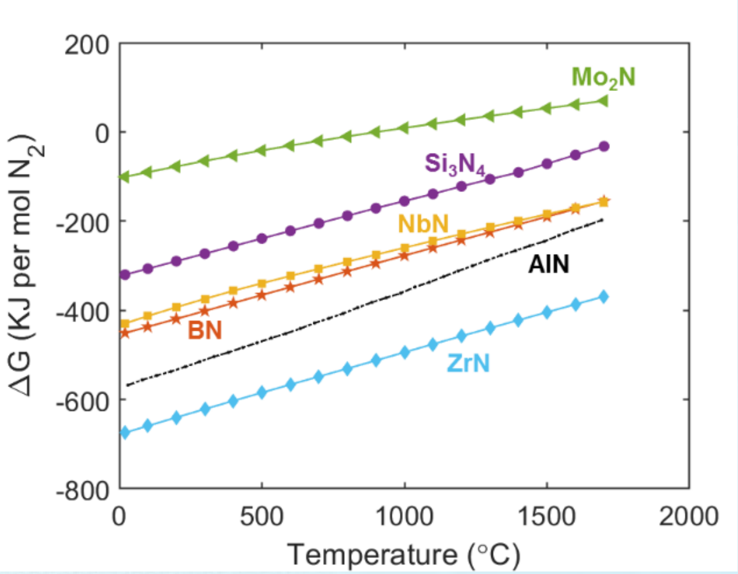
Alternative High Temperature Ceramic Insulators

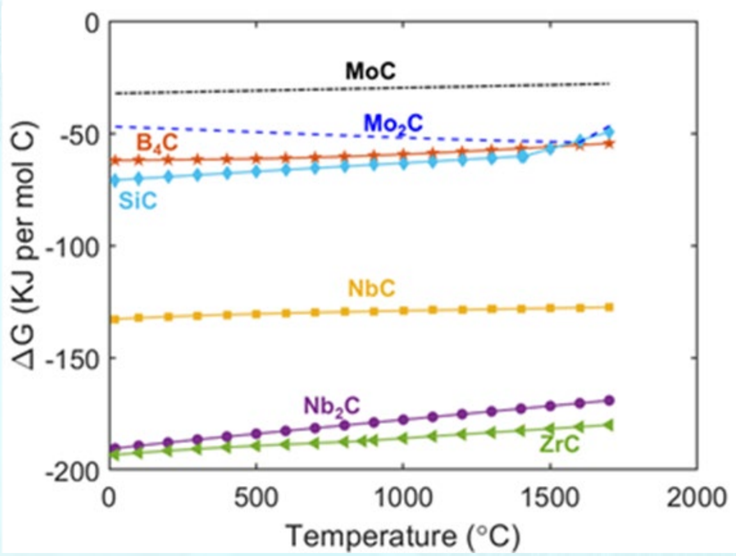


- ZrC, ZrN, and B_4C were not considered further due to their low electrical resistivity in comparison to alumina.

Alternative Thermocouple Insulators

Ceramic Insulator	Interaction with Niobium Onset Temperature (°C)	Interaction with Molybdenum Onset Temperature (°C)	Melting point (°C)
Nitride			
Si_3N_4	Not observed	1000	1900
AlN	1400	Not observed	2200
H-BN	1700	1700	2973
Carbides			
SiC	1300	1500	2730
B_4C	Not observed	1100	2350

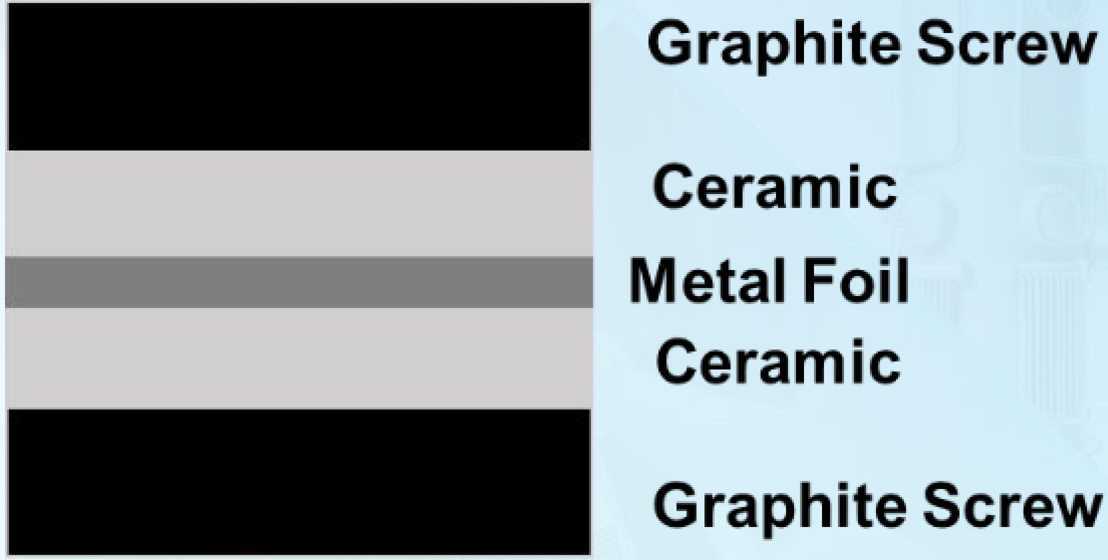




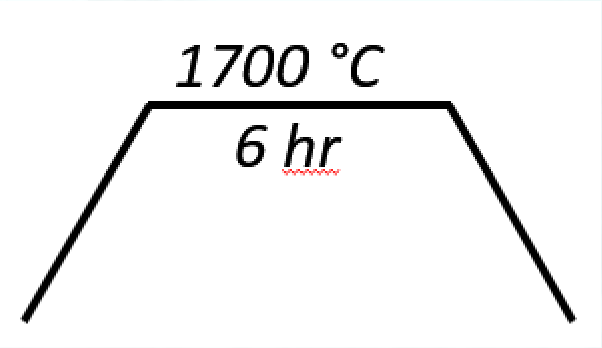
Diffusion Couple Set up

A)

B)

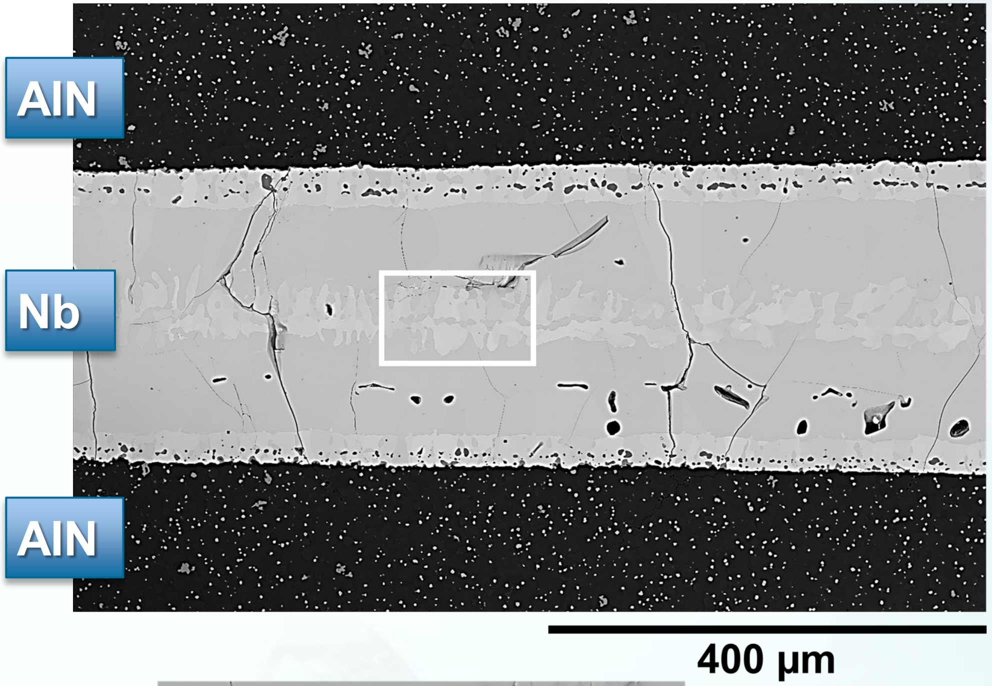


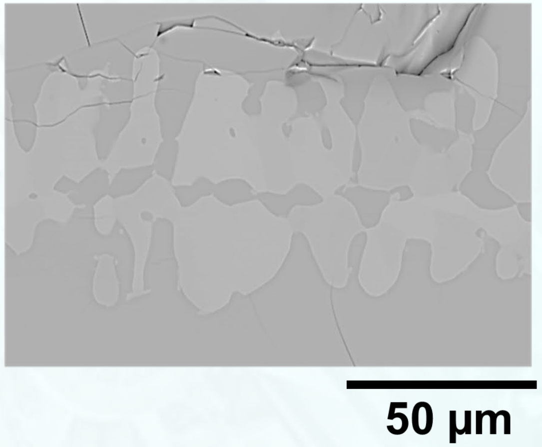
C)

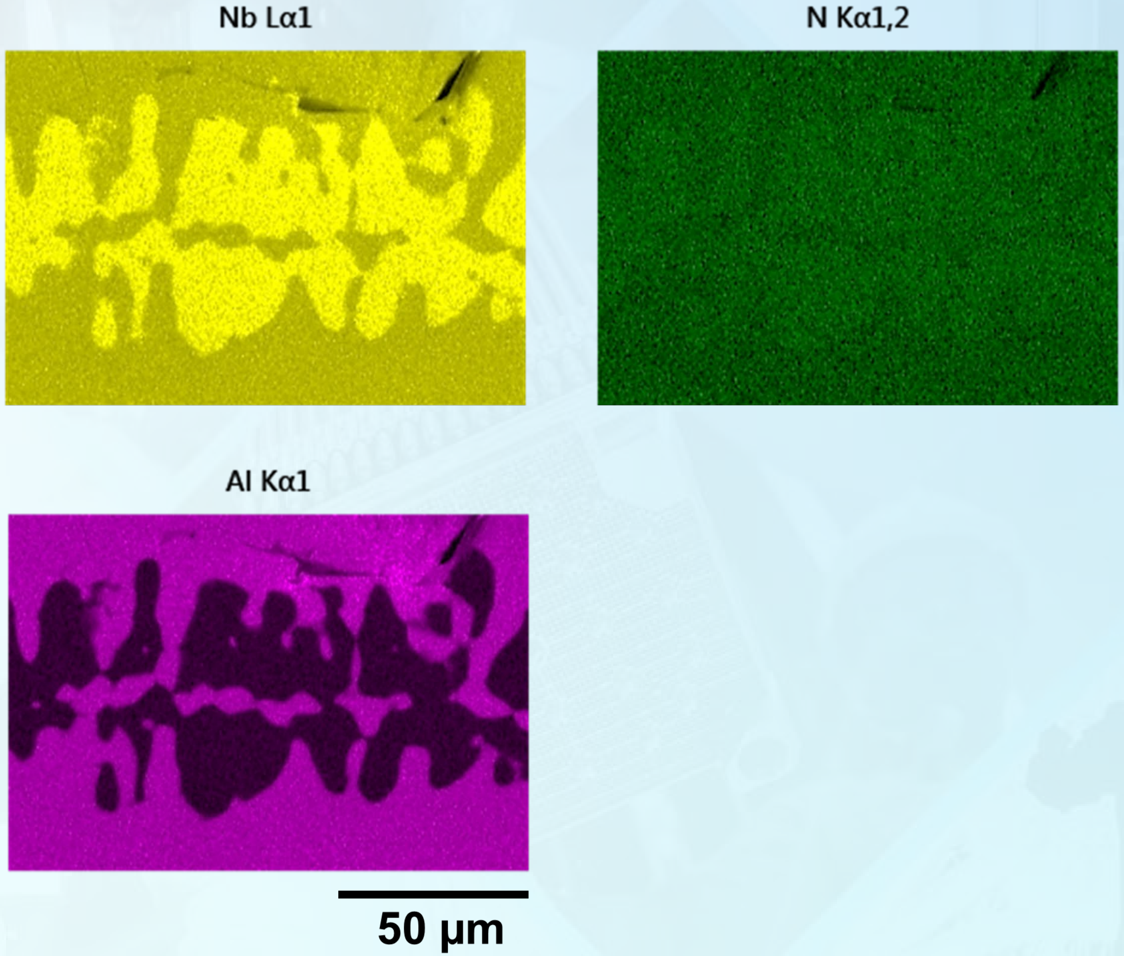


- Samples were cross-section after heat treatment and prepared for SEM characterization.

Chemical Stability – Niobium with Aluminum Nitride at 1700 ℃

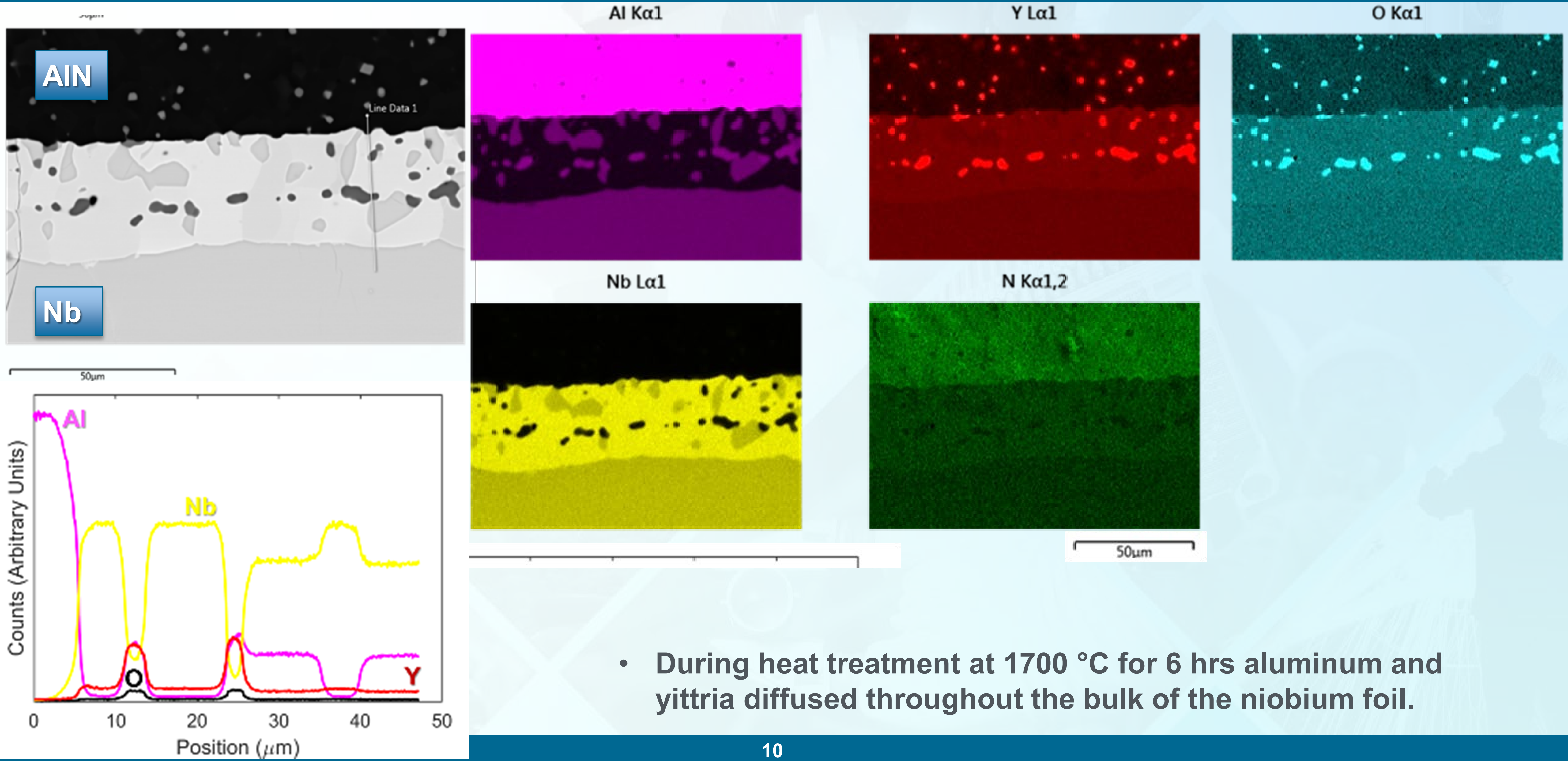






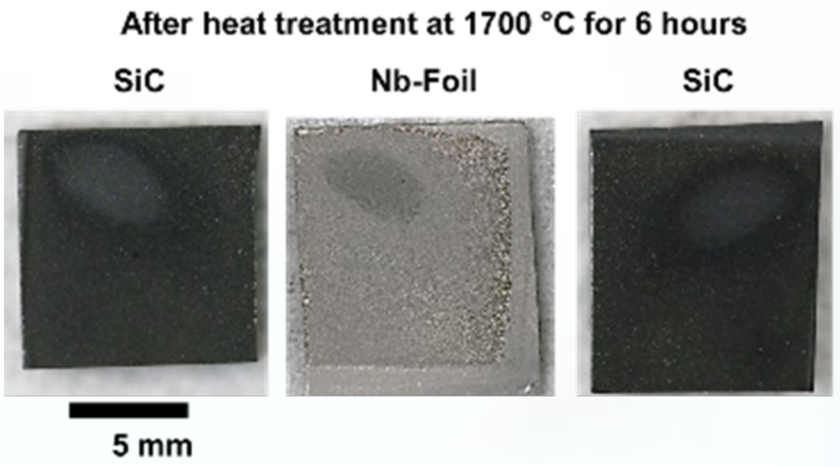
- **During heat treatment at 1700 °C for 6 hrs aluminum diffused throughout the bulk of the niobium foil.**

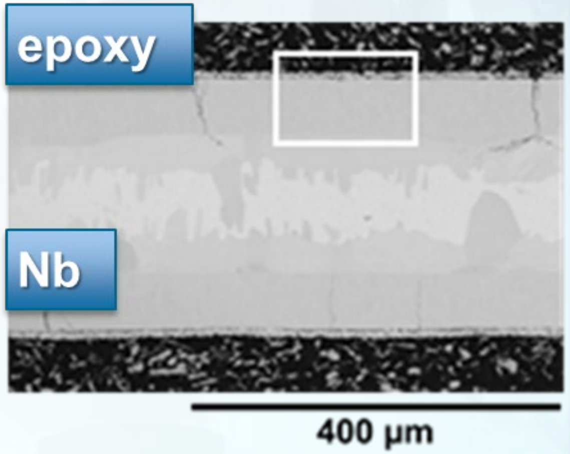
Chemical Stability – Niobium with Aluminum Nitride at 1700 °C

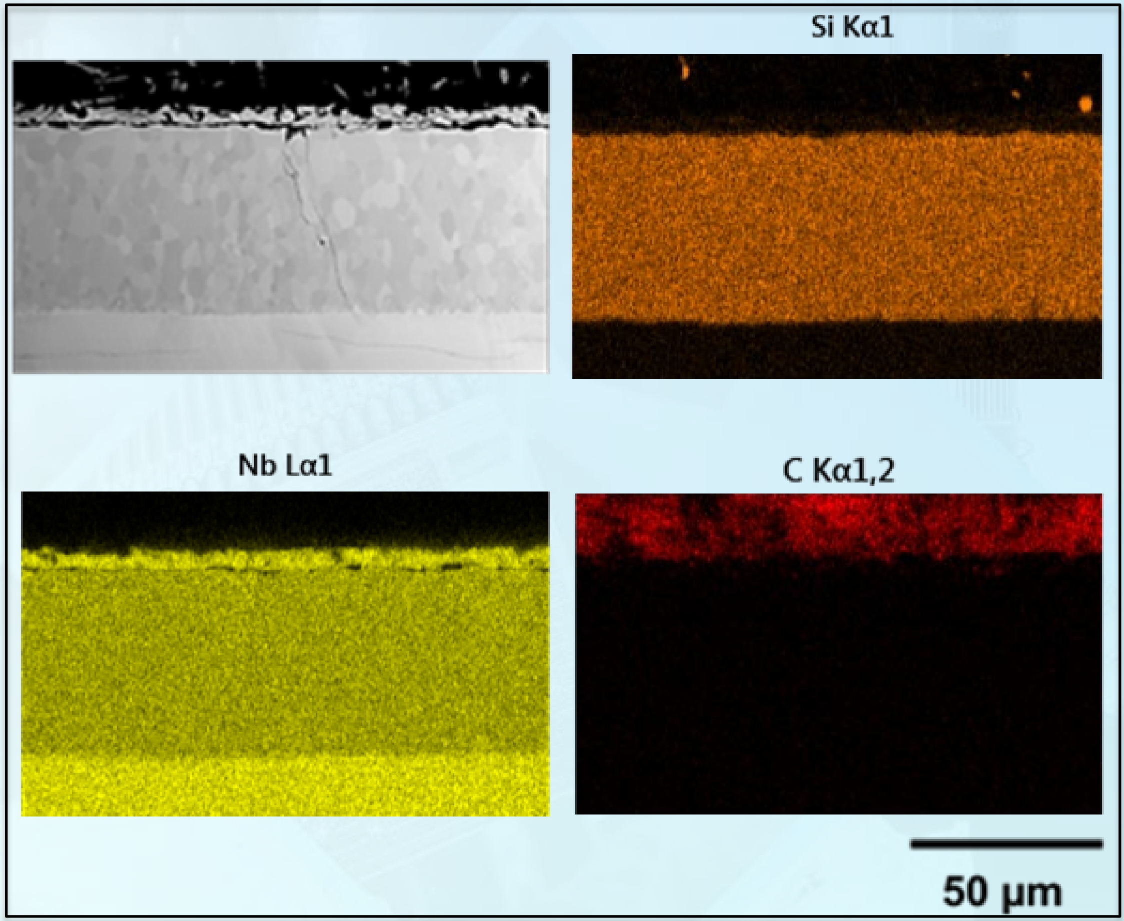


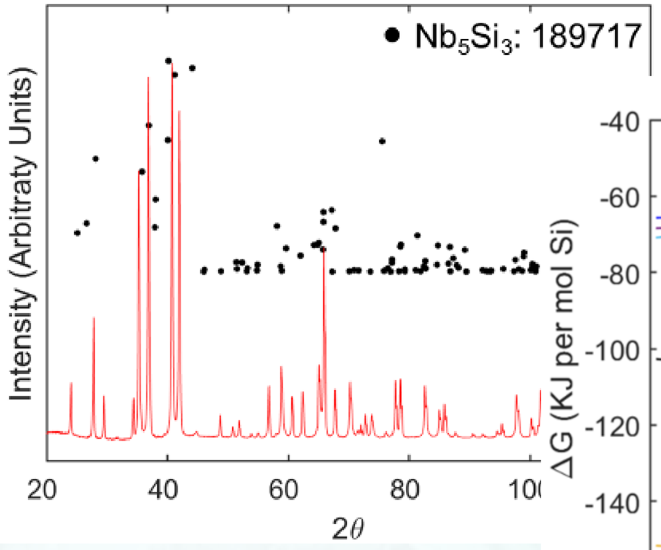
- **During heat treatment at 1700 °C for 6 hrs aluminum and yttria diffused throughout the bulk of the niobium foil.**

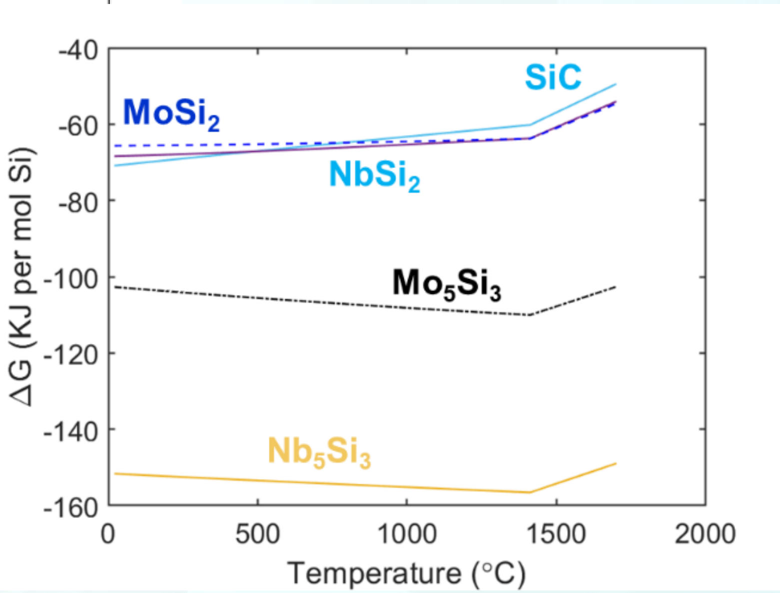
Chemical Stability – Niobium with Silicon Carbide at 1700 ℃









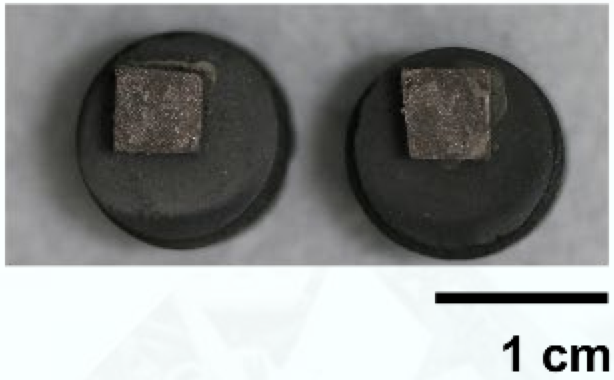


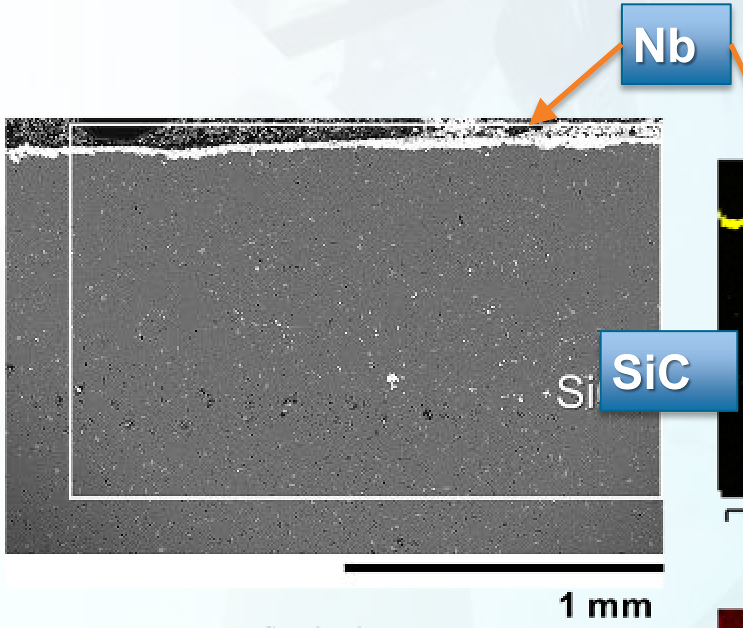
- During heat treatment at 1700 °C for 6 hrs the SiC dissociated and the silicon formed a silicide diffusion region within the niobium foil.

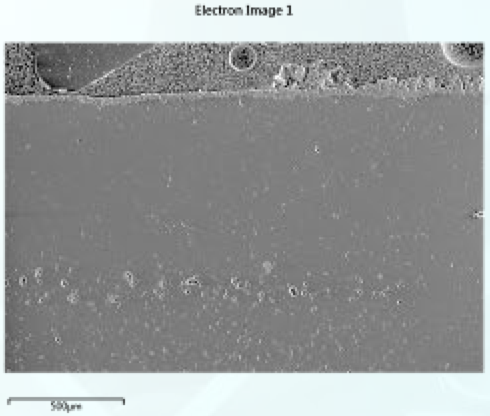
Chemical Stability – Niobium with Silicon Carbide at 2000 °C

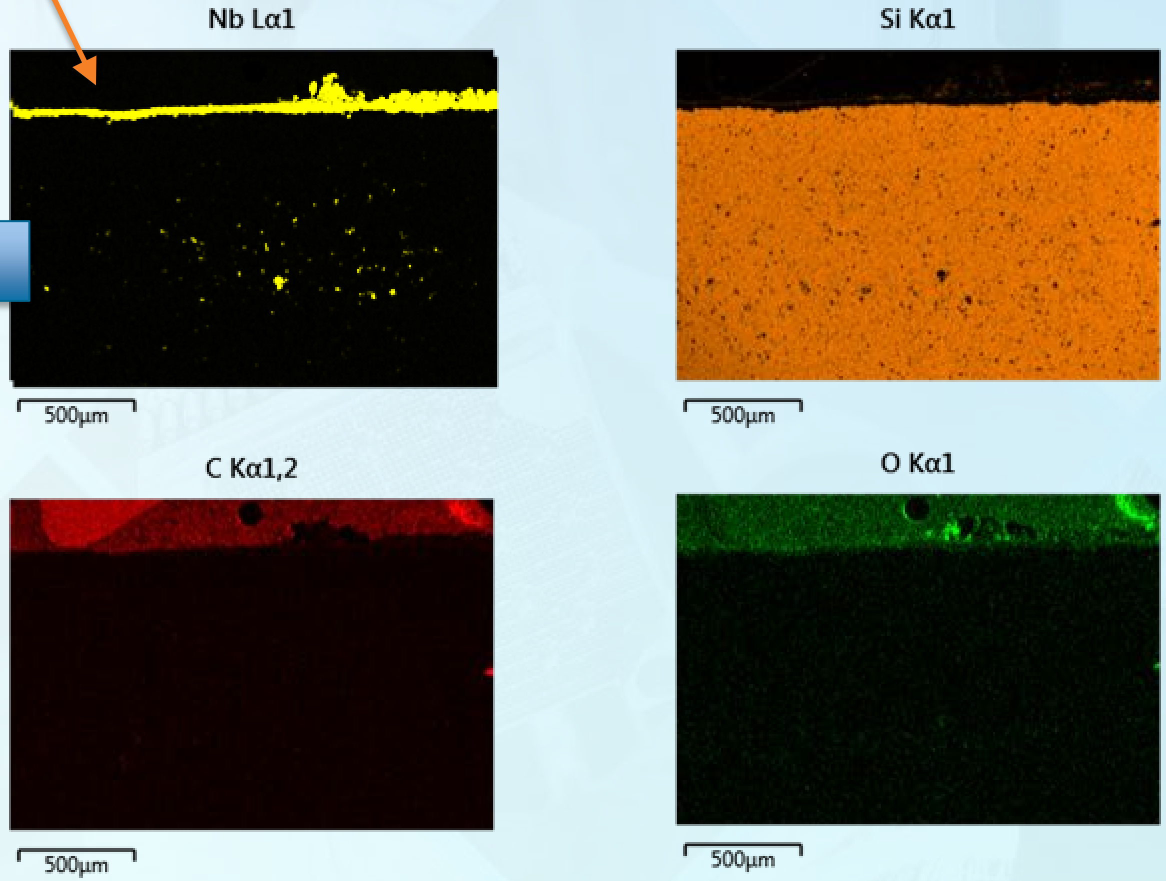
After heat treatment at 1700 °C for 6 hours

After heat treatment at 2000 C for 6 hours





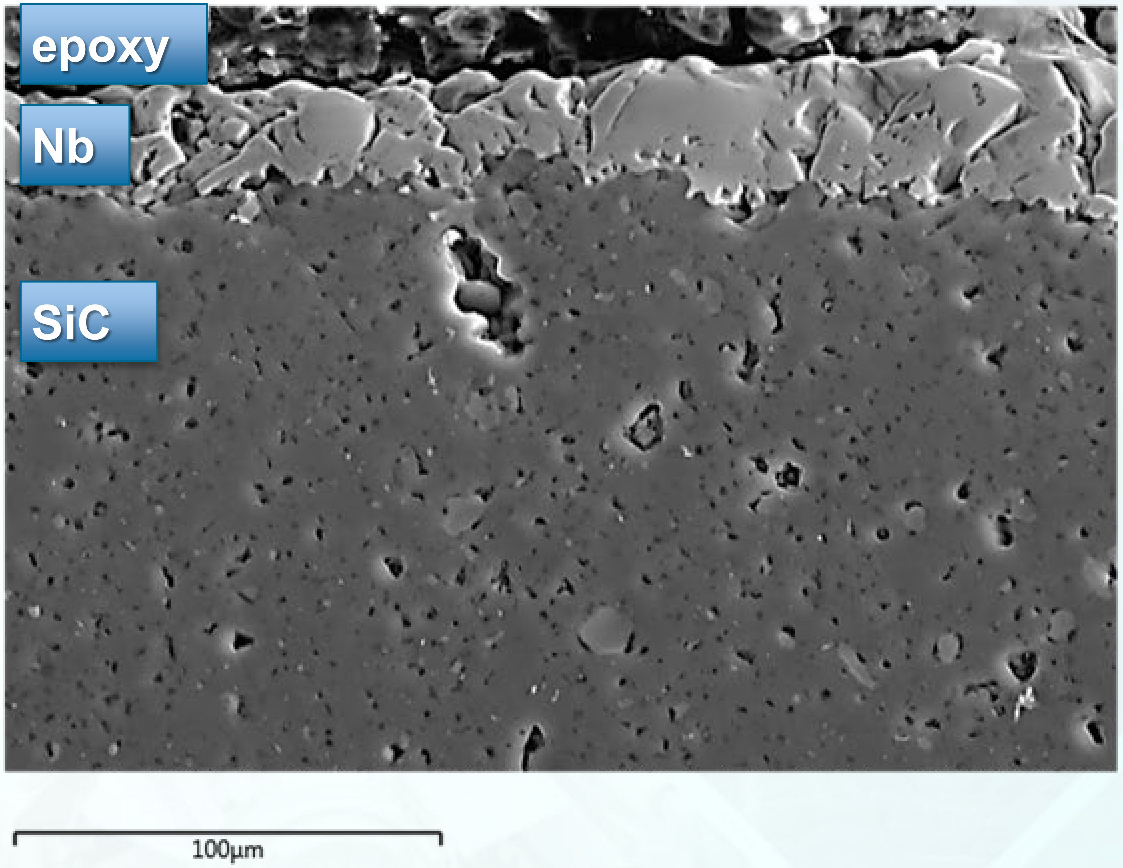




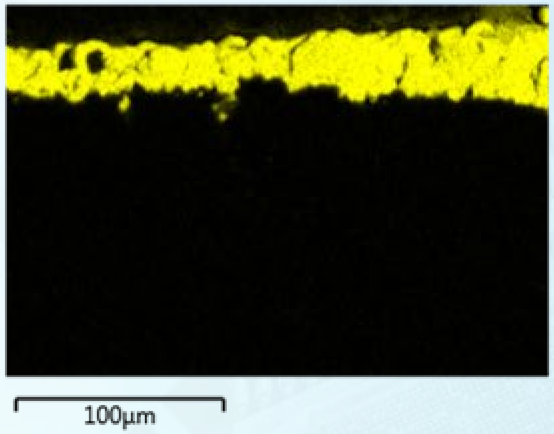
- During heat treatment at 2000 °C for 6 hrs the niobium appeared to form a eutectic phase with the SiC.

Chemical Stability – Niobium with Silicon Carbide at 2000 °C

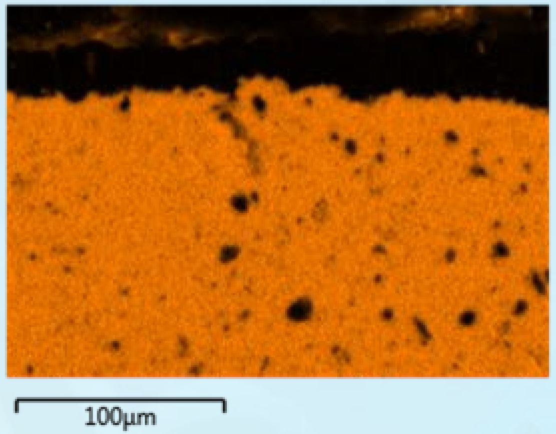
Electron Image 3



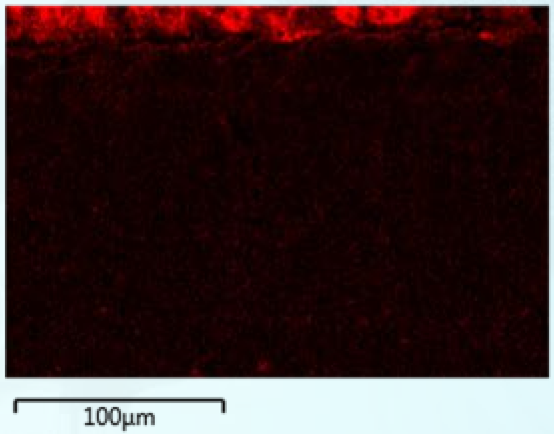
Nb Lα1



Si Kα1

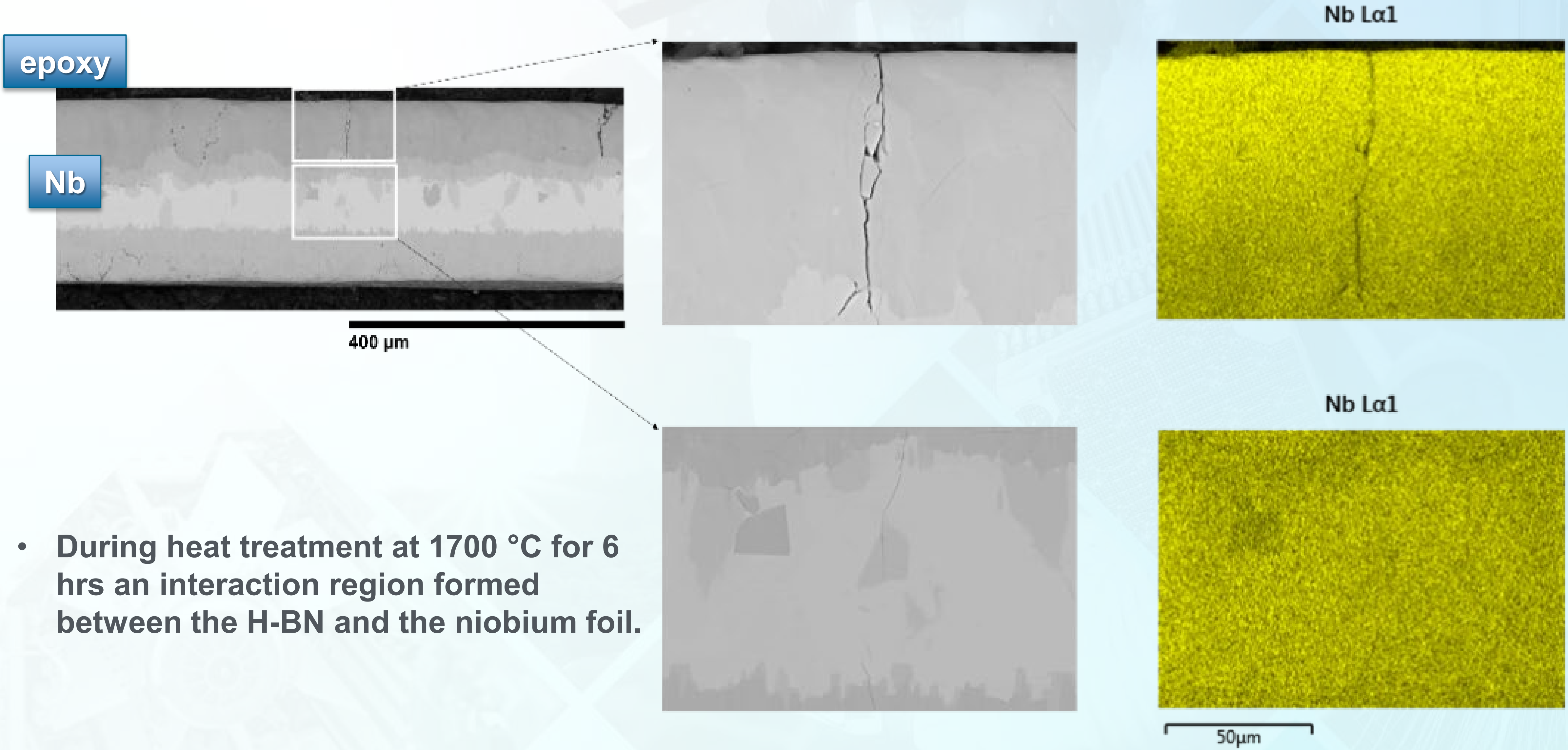


C Kα1,2



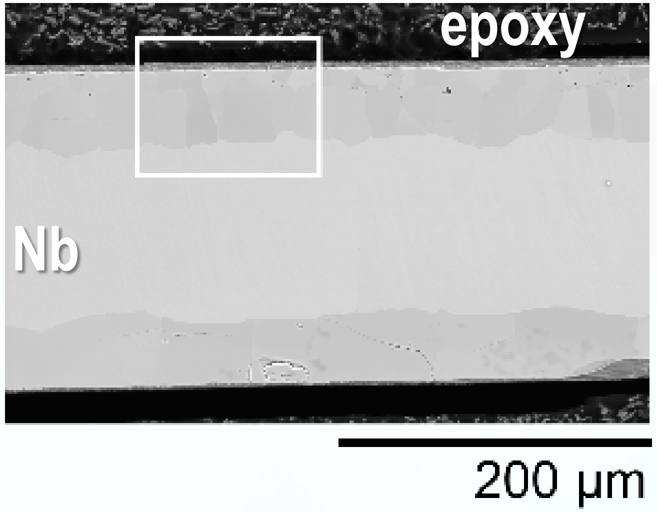
- **During heat treatment at 2000 °C for 6 hrs the niobium was wetted to the surface of the SiC but there is a lack of a diffusion region forming.**

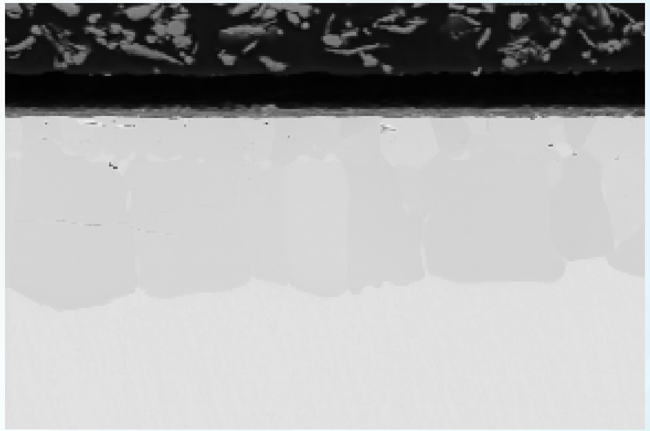
Chemical Stability – Niobium with H-BN at 1700 °C

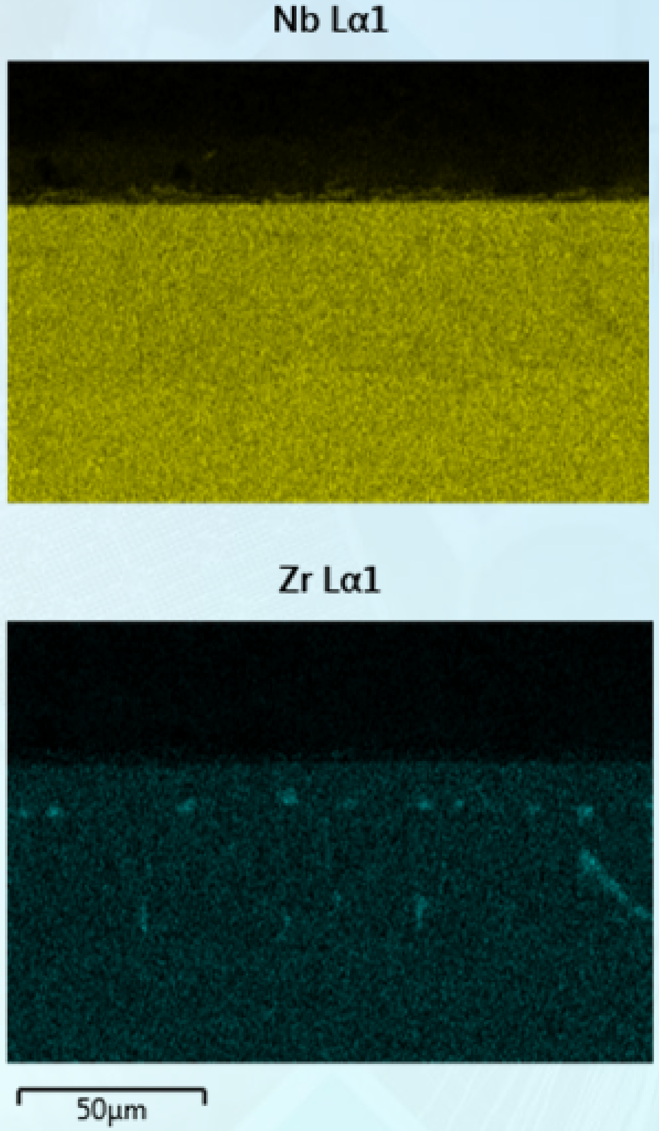


- **During heat treatment at 1700 °C for 6 hrs an interaction region formed between the H-BN and the niobium foil.**

Chemical Stability – Niobium with ZrO_2 at 1700 °C



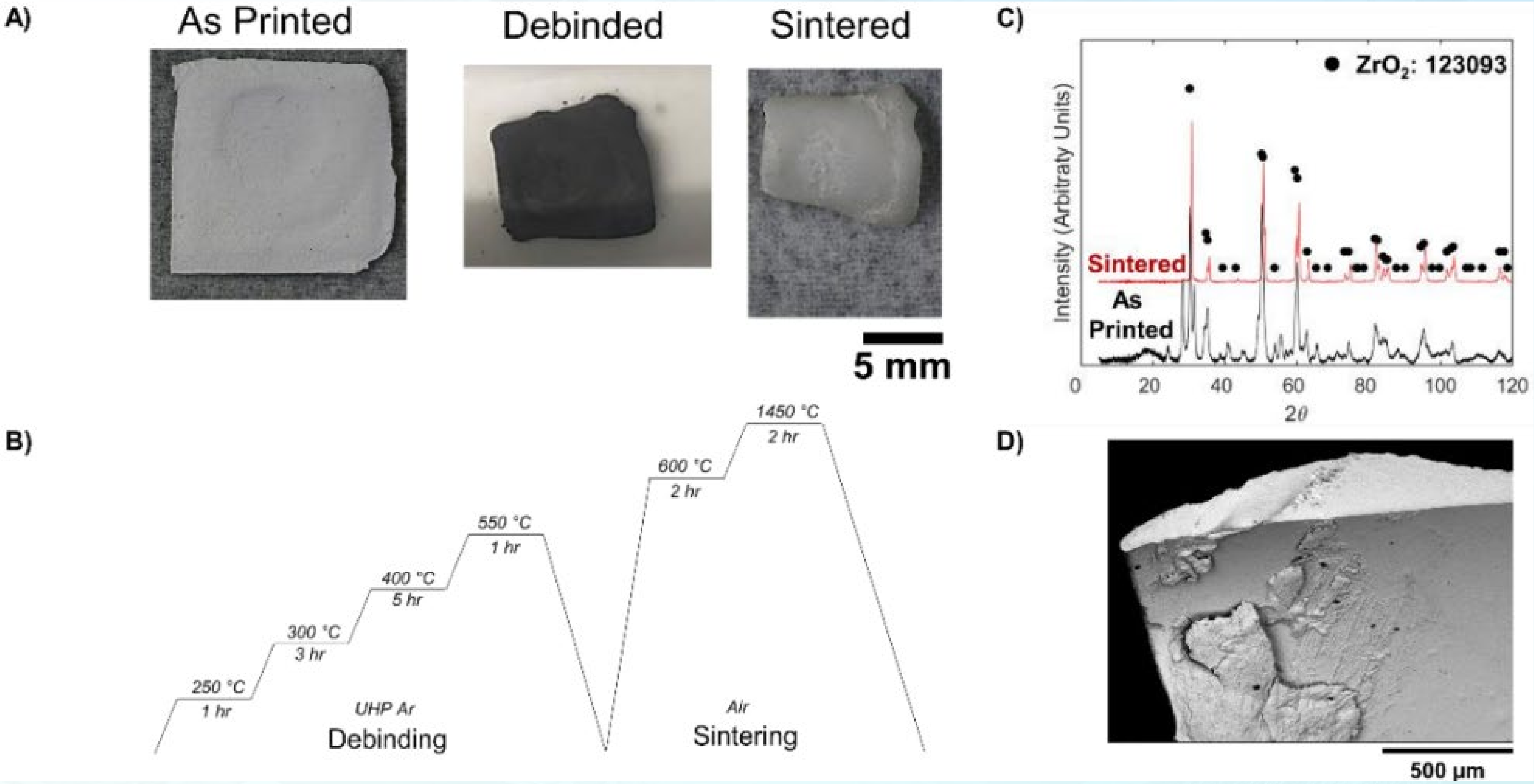




- During removal of the ZrO_2/Nb/ZrO_2 diffusion couple from the graphite fixture the ZrO_2 fractured and crumbled into pieces.
- During heat treatment at 1700 °C for 6 hrs an interaction region formed between the ZrO_2 and the niobium foil.

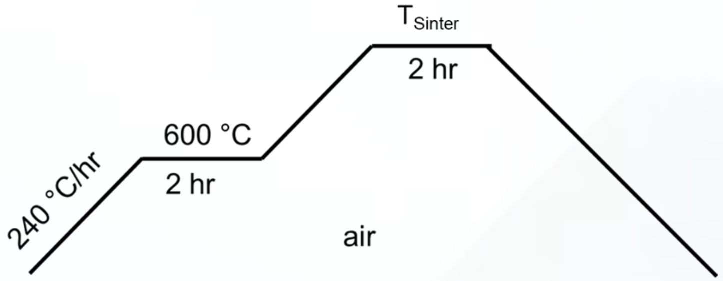
Digital Light Processing printing of ZrO_2 (Surrogate Ceramic)

Image of zirconia test print using the Kudo3D Titan 1 DLP printer

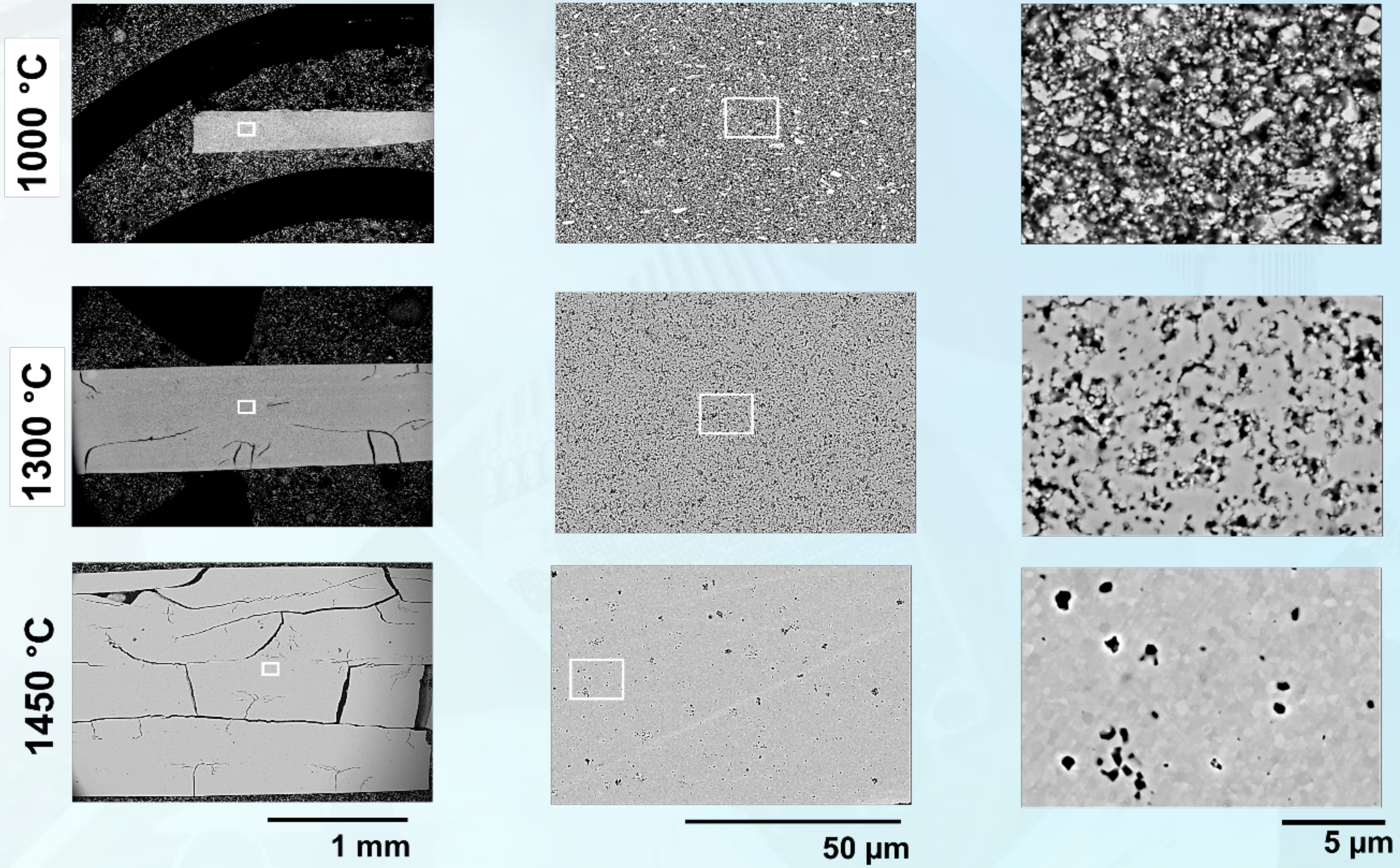


- **Sintering of the as printed ZrO_2 resulted in a high density phase pure ZrO_2.**

Optimization of Printed ZrO_2 Porosity



ZrO_2 Sample #	Sinter Temperature (°C)
1	600
2	1000
3	1300
4	1450

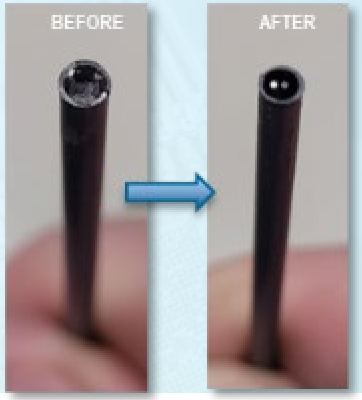


- **The sintering profile was adjusted to increase the porosity of the ZrO_2, and in doing so produce a crushable part.**

Concluding Remarks

The stability of alternative high-temperature ceramic insulators was conducted with HTIR-TC relevant materials. **The down selection of AlN, SiC, ZrO_2, and H-BN was made based upon thermodynamic predictions, electrical resistivity and thermal conductivity at elevated temperatures**, and empirically determined chemical stability with niobium and molybdenum.

- The interaction with surrogate Nb and Mo was shown
- The printing of relevant ceramics in advanced sensor geometries was trialed
- Additionally, powder packing of relevant ceramic powders was verified



- Patent application from current work:

My Completed Records

IDR #	Docket #	Title	Accepted
13485	BA-1609	Particulate insulation packing and wire alignment for sensors using acoustic nodal points	6/5/2024

- Journal submissions are forthcoming from the current work

References

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Thank You