REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
02/28/2023	Presentation	Dec, 2020 - Sep, 2022

4. TITLE AND SUBTITLE

ILIR Equipment Elemental and Thermal Conductivity Property Characterization for Super-Alloy Inconel C-276 after Prolonged Caustic High-Temperature Operation

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Ground Vehicle Power and Mobility (GVPM)
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Warren, MI 48397

8. PERFORMING ORGANIZATION REPORT NUMBER

OPSEC 7304

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

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10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT

DISTRIBUTION A. Approved for public release; distribution unlimited. OPSEC #7304

13. SUPPLEMENTARY NOTES

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF: Available for Public Release			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	Same as Report (SAR)	25	Theodore Burye
UNCLS	UNCLAS	UNCLAS			**19b. TELEPHONE NUMBER** *(include area code)* 586-282-3047

U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – GROUND VEHICLE SYSTEMS CENTER

ILIR Equipment Elemental and Thermal Conductivity Property Characterization for Super-Alloy Inconel C-276 after Prolonged Caustic High-Temperature Operation

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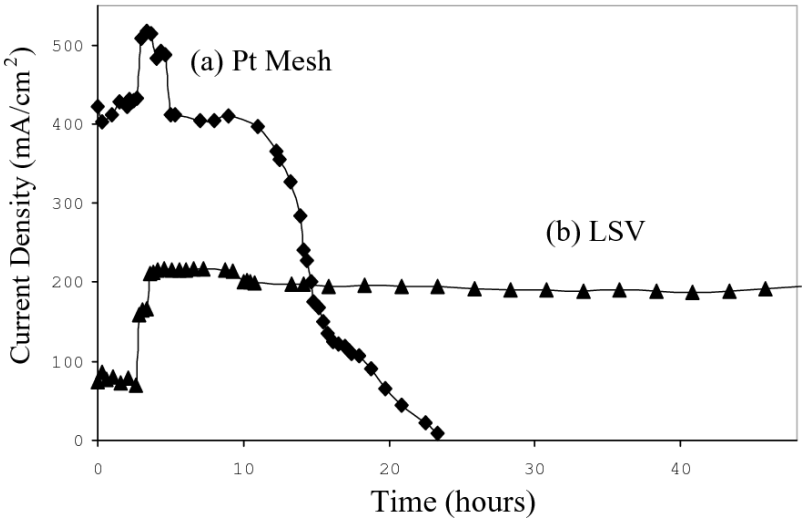
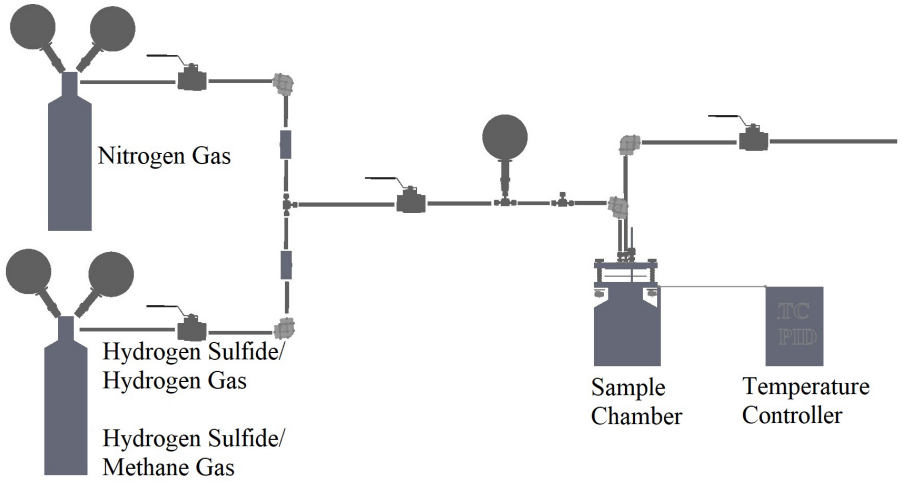
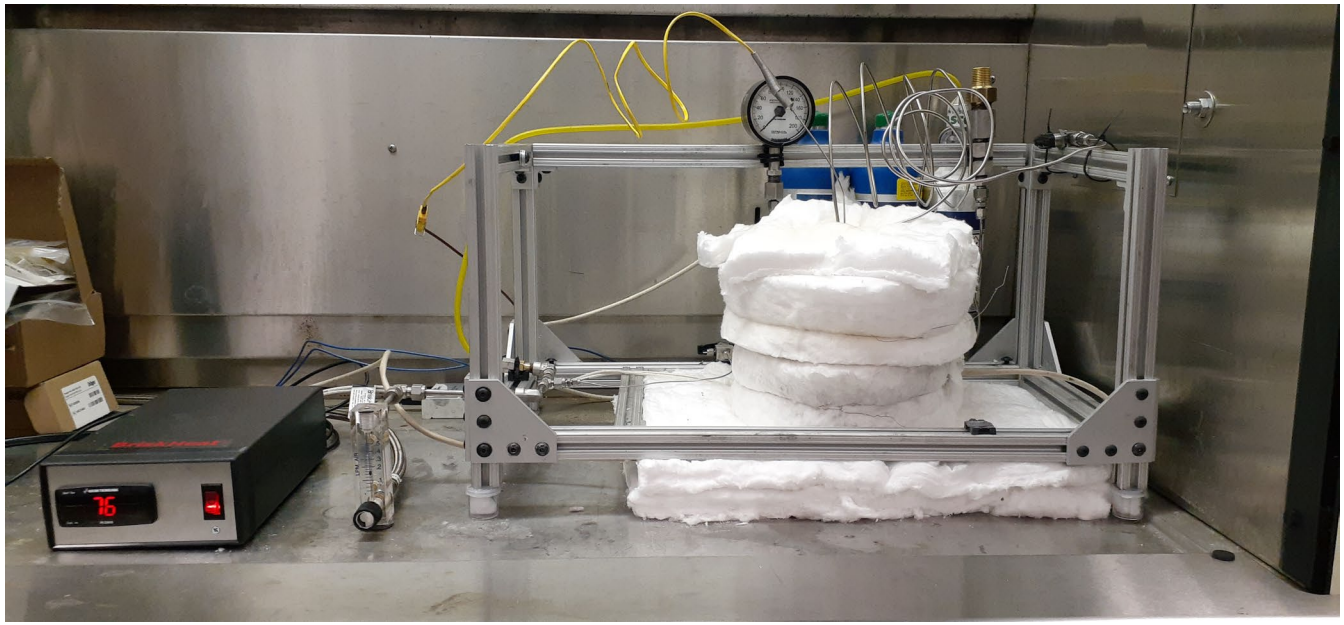
28 FEB 23

Motivation – ILIR Equipment and Testing Operation

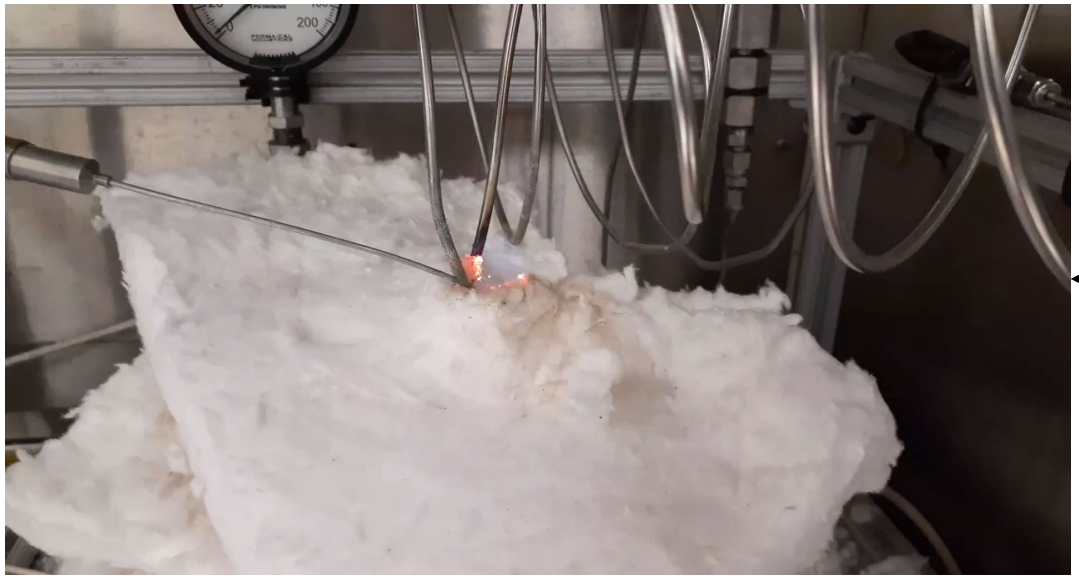






Motivation – Equipment Operation Malfunction and Long-Term Operation Safety Evaluation

Equipment Operation Malfunction - Methane Flame



Initial Incident

- Methane flame resulted from oxidation corrosion around fittings in lid (discussed in another presentation), which were made of 316SS.
  - Inconel could not be found for fitting material
  - Safety concern

Evaluation of Key Safety/Operational Issues for Future use of Inconel C-276

1. Potential Inconel C-276 corrosion leaks.
   - Leaks/cracks form in body and lid? Need gas to exit in a controlled manner from intended location.
2. Heat tape appeared to be failing prematurely.
   - Did Inconel C-276 thermal conductivity change?
3. Potential Inconel hardness decreased.
   - Currently system is not under pressure. Overall pressure rating changed?

Test Procedure – Material Properties Characterized

Lid Initially

Lid Post Test

Body Initially

Body Post Test

Material properties evaluated/interested in?

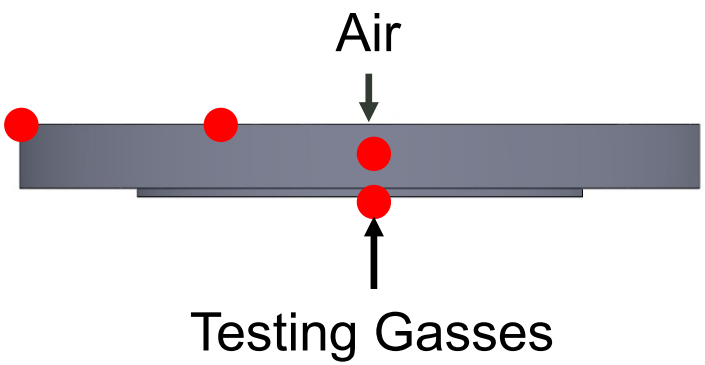
1. Corrosion resistance/gas penetration – SEM/EDS elemental characterization
2. Thermal conductivity – Thermal conduction characterization
3. Material hardness – Rockwell hardness characterization

Material property changes could impact?

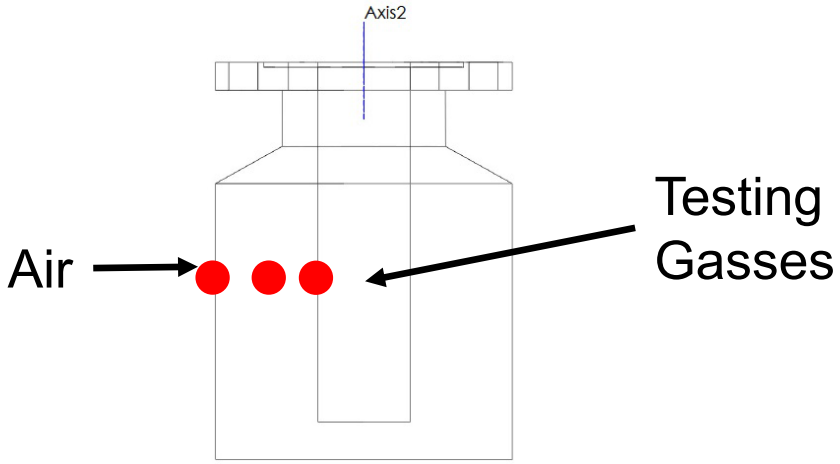
- Material structural properties
- Metal embrittlement resulting in chipping/cracking and failure of overall vessel integrity
- Heat transfer through metal surface dissipating heat from heat tape
  - Premature heat tape failure

Test Procedure – Material Property Characterization Locations

Lid



Body



Characterization locations

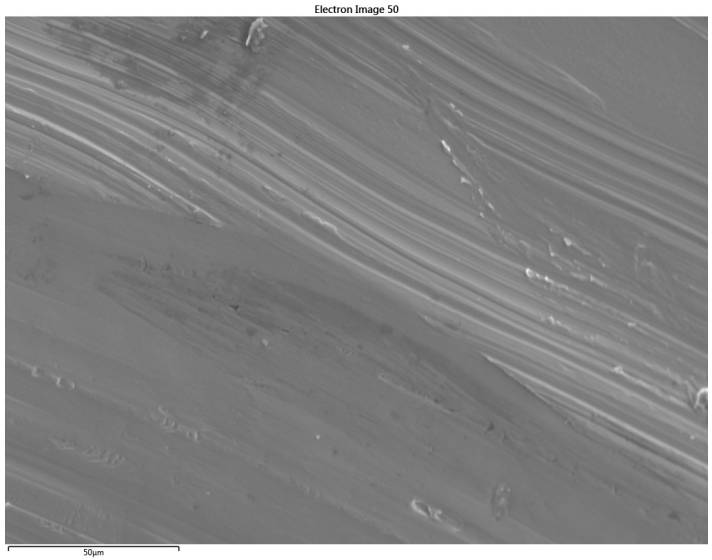
1. Lid and body
    1. External surface exposure to air
    2. Internal surface exposure to testing gasses
    3. Interior metal between external and interior surfaces

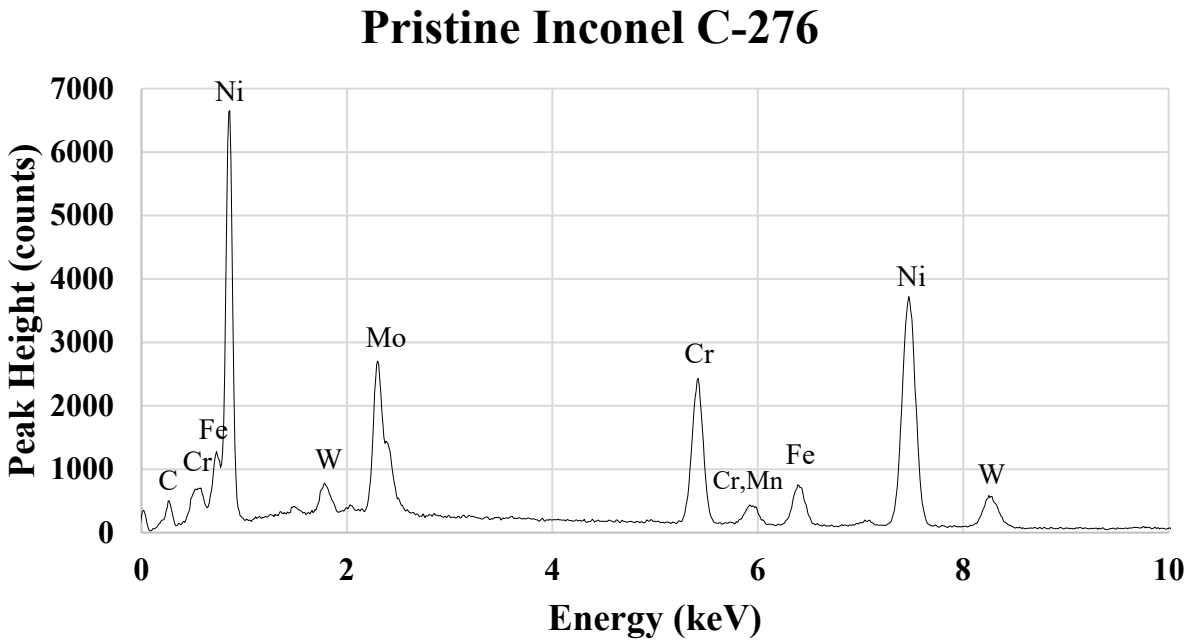
Location test parameters

1. Microscopy images – Lid & Body (external surface / interior / interior surface)
2. Elemental composition – Lid & Body (external surface / interior / interior surface)
3. Thermal conductivity – Lid (external surface)
    1. Body attempted to be characterized but curved surface prevented reliable results
4. Hardness – Body (external surface / interior)

Characterization of Inconel C-276 Elemental Composition

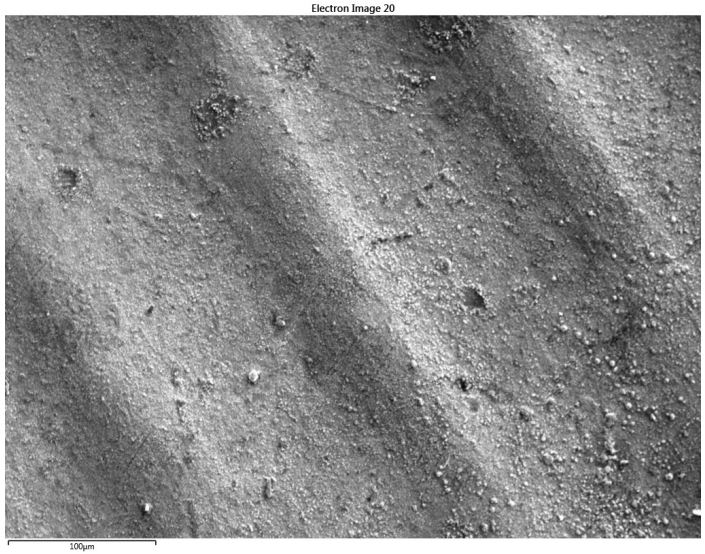
Experimental Results – Baseline

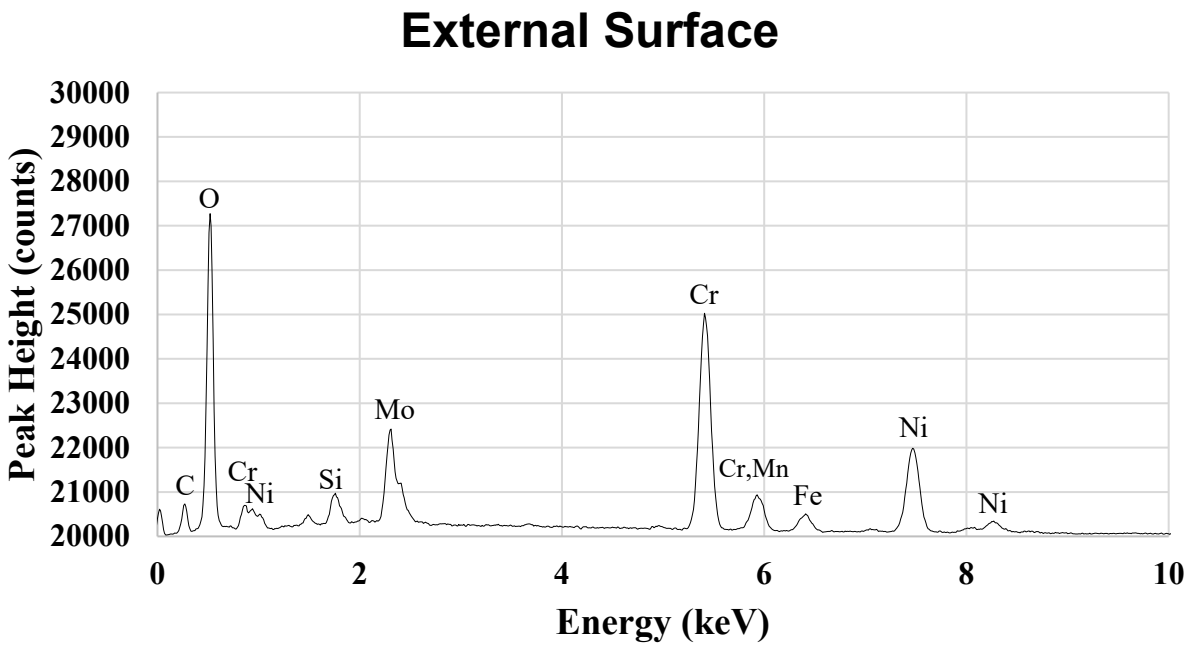




Element Type	Baseline Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	16.32	14.50-16.50
Manganese	0.41	0.00-1.00
Nickel	58.03	50.99-69.50
Molybdenum	14.63	15.00-17.00
Iron	6.34	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	0.00	0.00-0.35
Tungsten	3.72	3.00-4.50

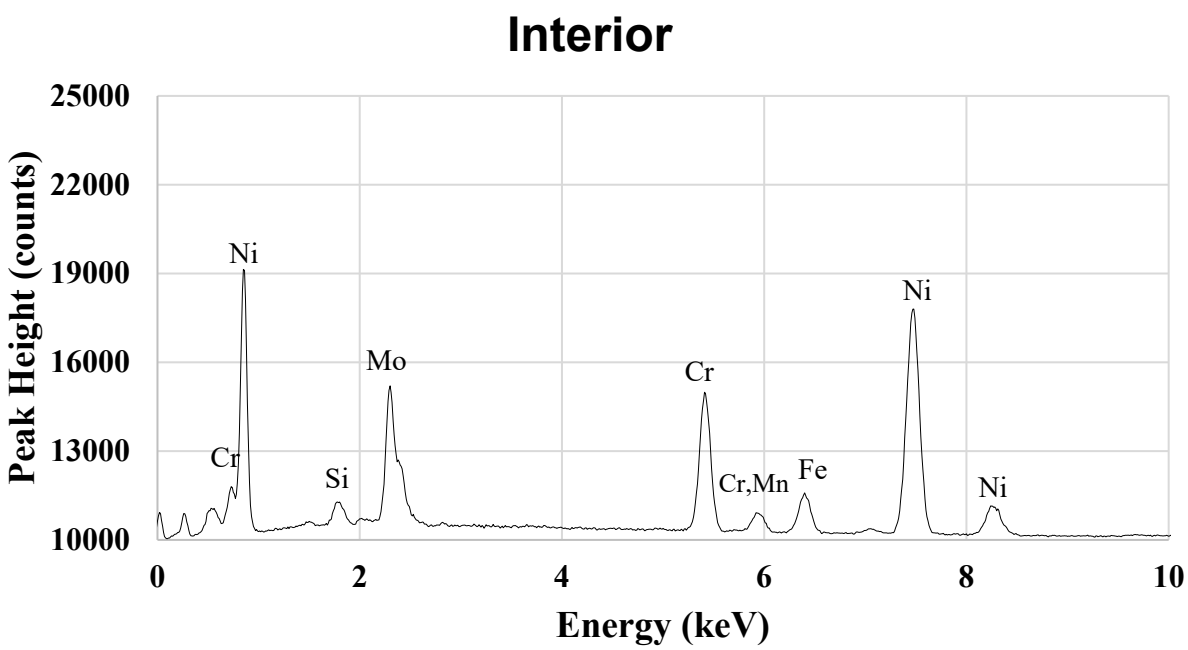
Experimental Results – Lid External Surface





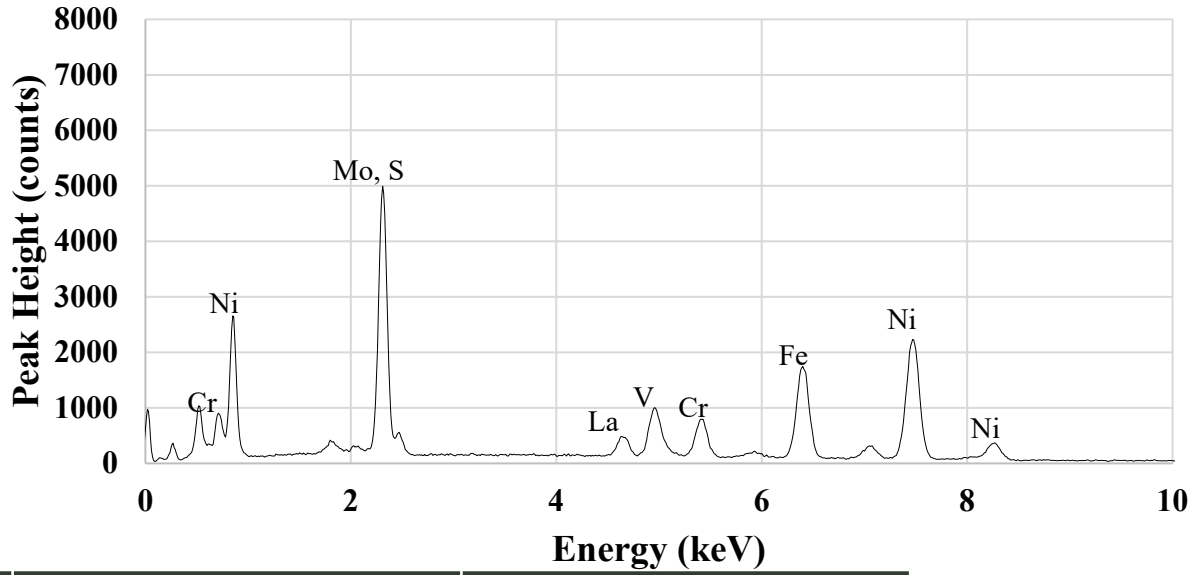
Element Type	External Surface Sample Weight %	Literature Weight %
Silicon	**1.96**	0.00-0.08
Chromium	**40.90**	14.50-16.50
Manganese	**2.08**	0.00-1.00
Nickel	**33.41**	50.99-69.50
Molybdenum	17.07	15.00-17.00
Iron	4.64	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	0.00	0.00-0.35
Tungsten	**0.00**	3.00-4.50

Experimental Results – Lid Interior



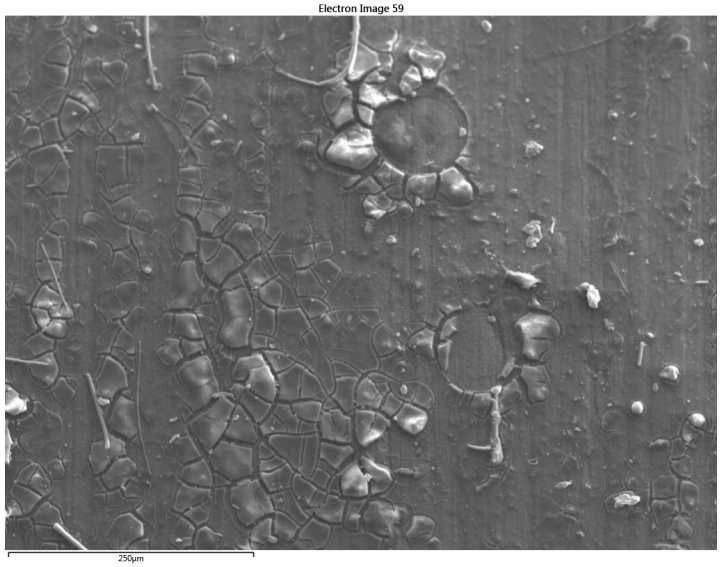
Element Type	Interior Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	16.16	14.50-16.50
Manganese	0.44	0.00-1.00
Nickel	55.80	50.99-69.50
Molybdenum	16.35	15.00-17.00
Iron	6.23	4.00-7.00
Cobalt	0.10	0.00-2.50
Vanadium	0.18	0.00-0.35
Tungsten	4.03	3.00-4.50

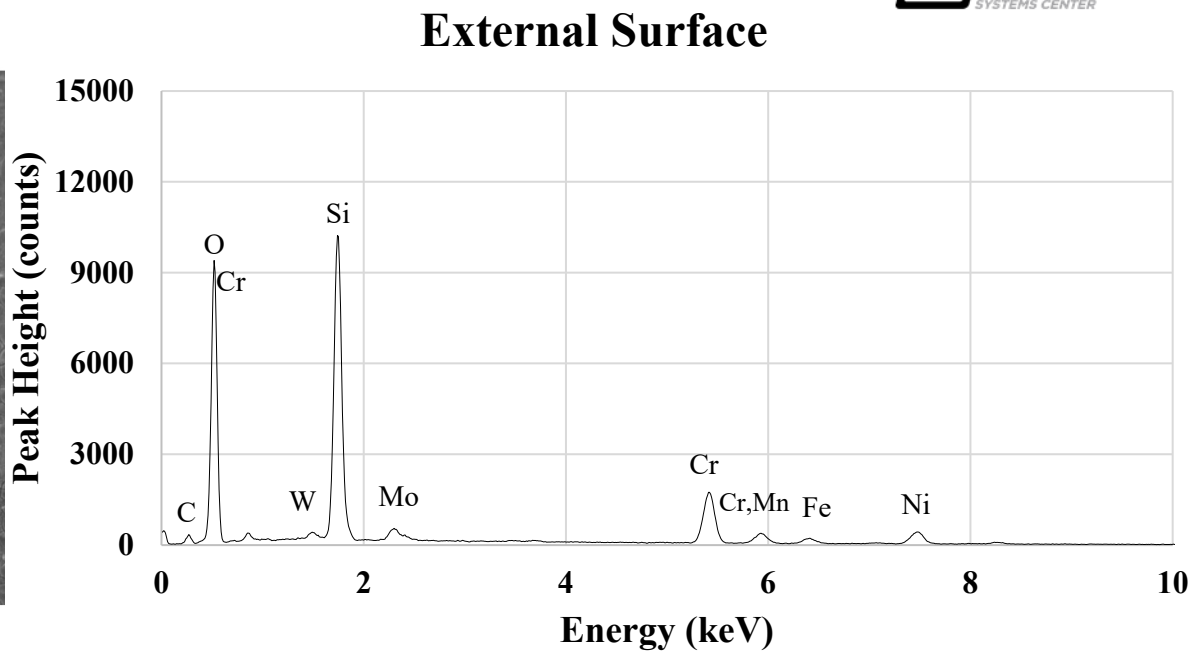
Experimental Results – Lid Interior Surface



Element Type	Interior Surface Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	**4.42**	14.50-16.50
Manganese	0.46	0.00-1.00
Nickel	**40.56**	50.99-69.50
Molybdenum	**0.67**	15.00-17.00
Iron	**20.34**	4.00-7.00
Cobalt	0.16	0.00-2.50
Vanadium	6.33	0.00-0.35
Tungsten	3.86	3.00-4.50
Lanthanum	6.57	0.00
Strontium	1.27	0.00
Sulfur	**17.30**	0.00

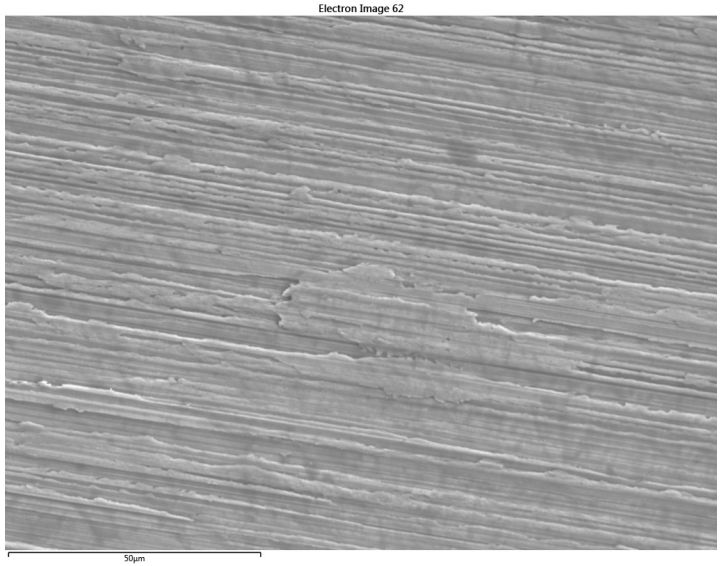
Experimental Results – Body External Surface

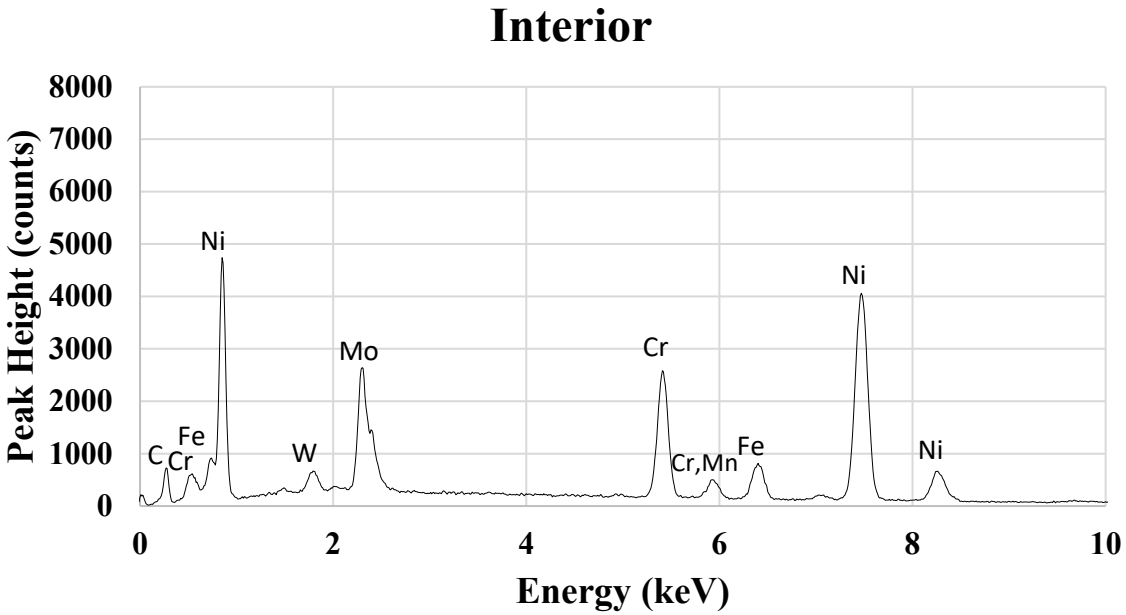




Element Type	External Surface Sample Weight %	Literature Weight %
Silicon	**46.92**	0.00-0.08
Chromium	**25.70**	14.50-16.50
Manganese	**2.13**	0.00-1.00
Nickel	**12.63**	50.99-69.50
Molybdenum	**6.24**	15.00-17.00
Iron	**3.64**	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	0.16	0.00-0.35
Tungsten	**1.04**	3.00-4.50

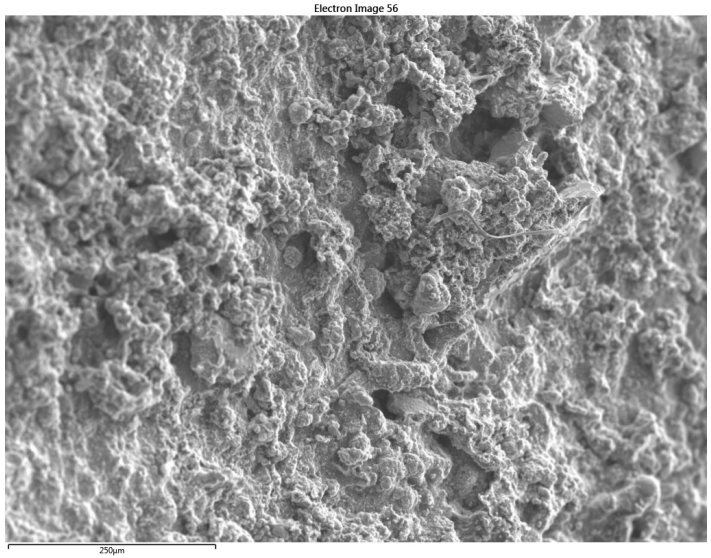
Experimental Results – Body Interior

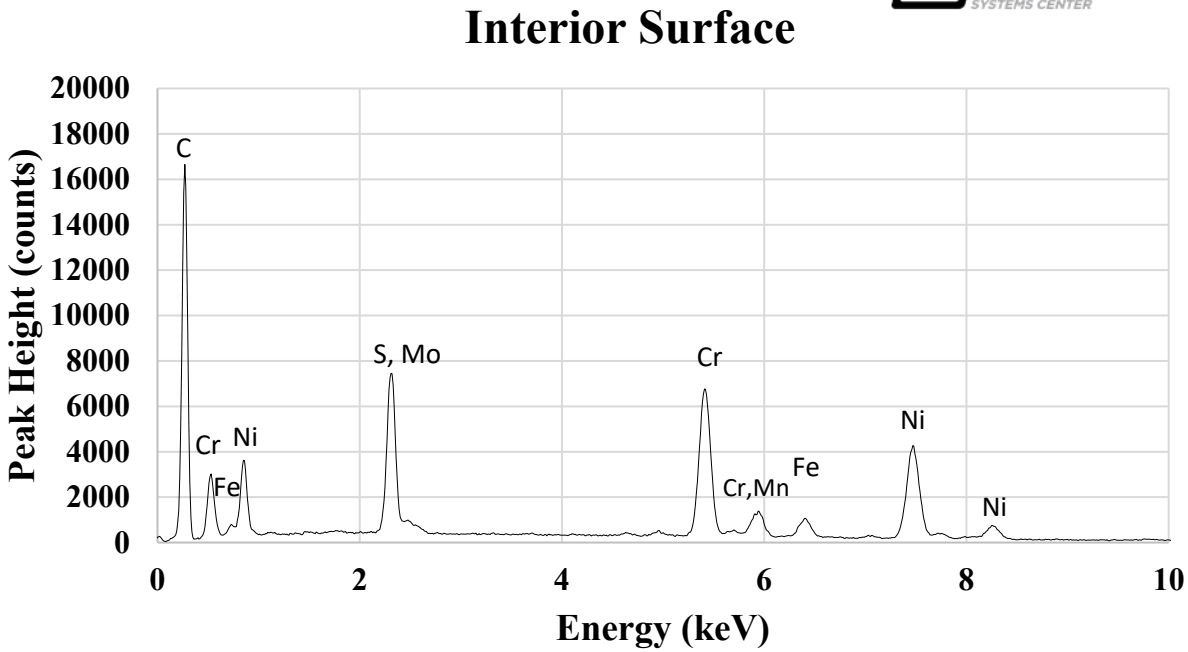




Element Type	Interior Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	16.04	14.50-16.50
Manganese	0.61	0.00-1.00
Nickel	56.60	50.99-69.50
Molybdenum	16.67	15.00-17.00
Iron	6.24	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	0.32	0.00-0.35
Tungsten	3.52	3.00-4.50

Experimental Results – Body Interior Surface



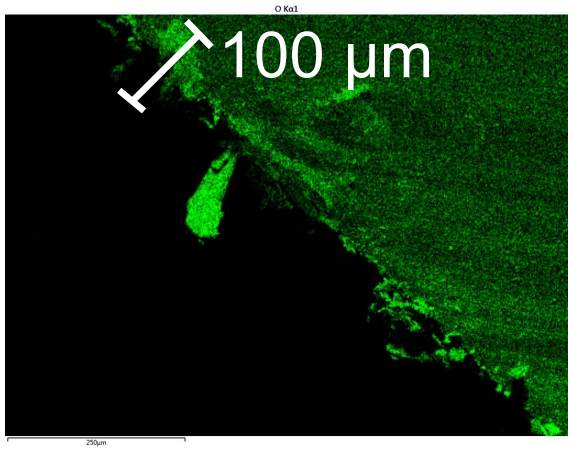


Element Type	Interior Surface Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	**32.45**	14.50-16.50
Manganese	**2.08**	0.00-1.00
Nickel	**39.96**	50.99-69.50
Molybdenum	**5.23**	15.00-17.00
Iron	4.78	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	1.00	0.00-0.35
Tungsten	**0.00**	3.00-4.50
Sulfur	**17.63**	0.00

Experimental Results – Elemental Composition Summary

Lid Interior

Element Type	Interior Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	16.16	14.50-16.50
Manganese	0.44	0.00-1.00
Nickel	55.80	50.99-69.50
Molybdenum	16.35	15.00-17.00
Iron	6.23	4.00-7.00
Cobalt	0.10	0.00-2.50
Vanadium	0.18	0.00-0.35
Tungsten	4.03	3.00-4.50



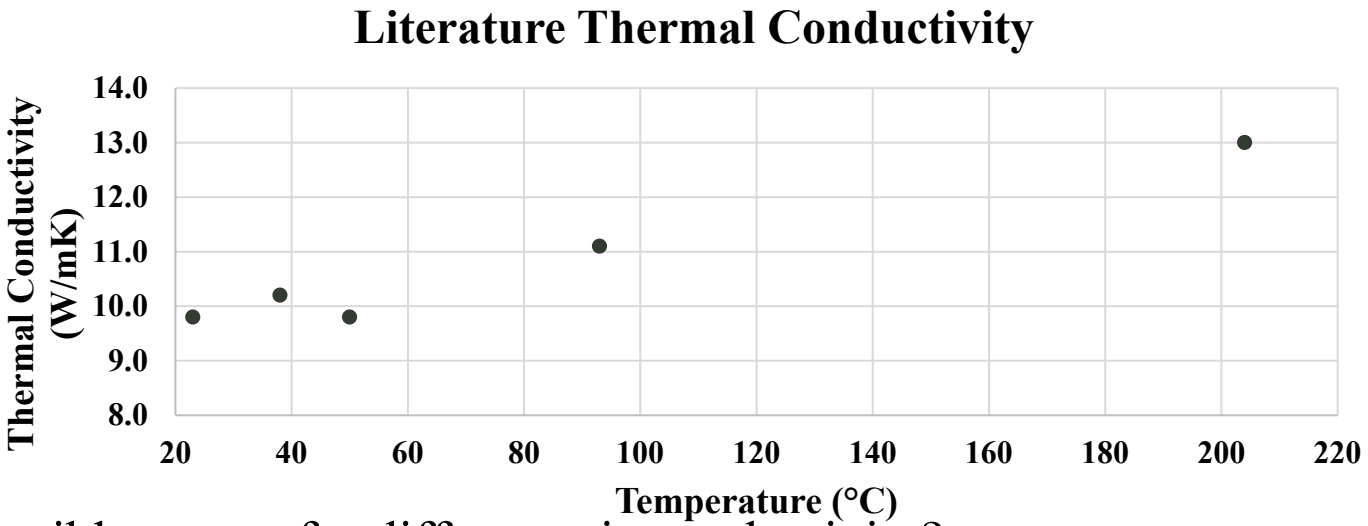
Body Interior

Element Type	Interior Sample Weight %	Literature Weight %
Silicon	0.00	0.00-0.08
Chromium	16.04	14.50-16.50
Manganese	0.61	0.00-1.00
Nickel	56.60	50.99-69.50
Molybdenum	16.67	15.00-17.00
Iron	6.24	4.00-7.00
Cobalt	0.00	0.00-2.50
Vanadium	0.32	0.00-0.35
Tungsten	3.52	3.00-4.50

- Contamination just a surface layer, low penetration.
  - 100 µm = 0.004 inch (Human hair = 70 µm ± 20 µm).
  - Wall thickness = 1.5 inches
- Embrittlement and corrosion cracking internally unlikely as internal structure elementally unchanged. Unlikely to form leaks/cracks in parts made of Inconel.
  - No internal cracks observed either

Characterization of Inconel C-276 Thermal Conductivity

Experimental Results – Literature Inconel Thermal Conductivity

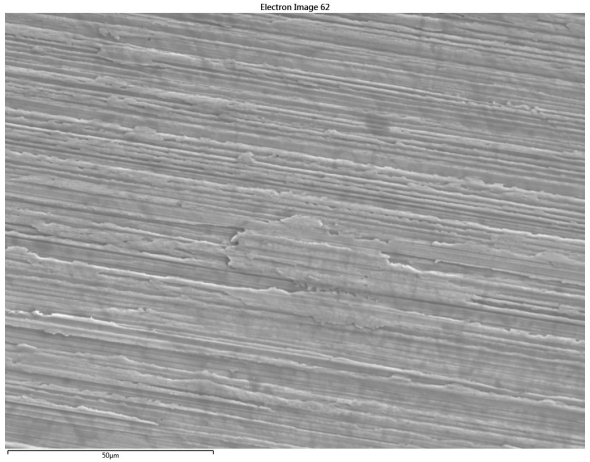


- What possible causes for difference in conductivity?
  - Bulk grain size – Larger grain size increases conductivity.
    - Heat typically increases grain size
  - Elemental differences can change conductivity - Surface oxidation
- High surface silicon & chromium levels present
    - Silicon dioxide (SiO_2) – 1.05 to 1.67 W/mK (200C to 800C)
    - Chromium oxide (Cr_2O_3) – 9.99 W/mK (25C)
- Silicon dioxide possible explanation
  - Where is Si or Cr originating from?

CRC Materials Handbook H. Dong, Scientific Reports, 4, 7037, (2014) D. Xu, Scientific Reports, 7, (2017), 10.1038/s41598-017-04371-8

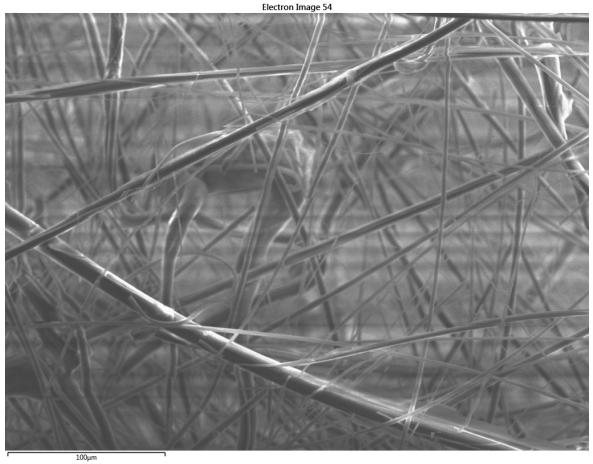
Experimental Results – Initial Sources of Silicon and Chromium

Inconel Sample



Heat Tape

Insulation

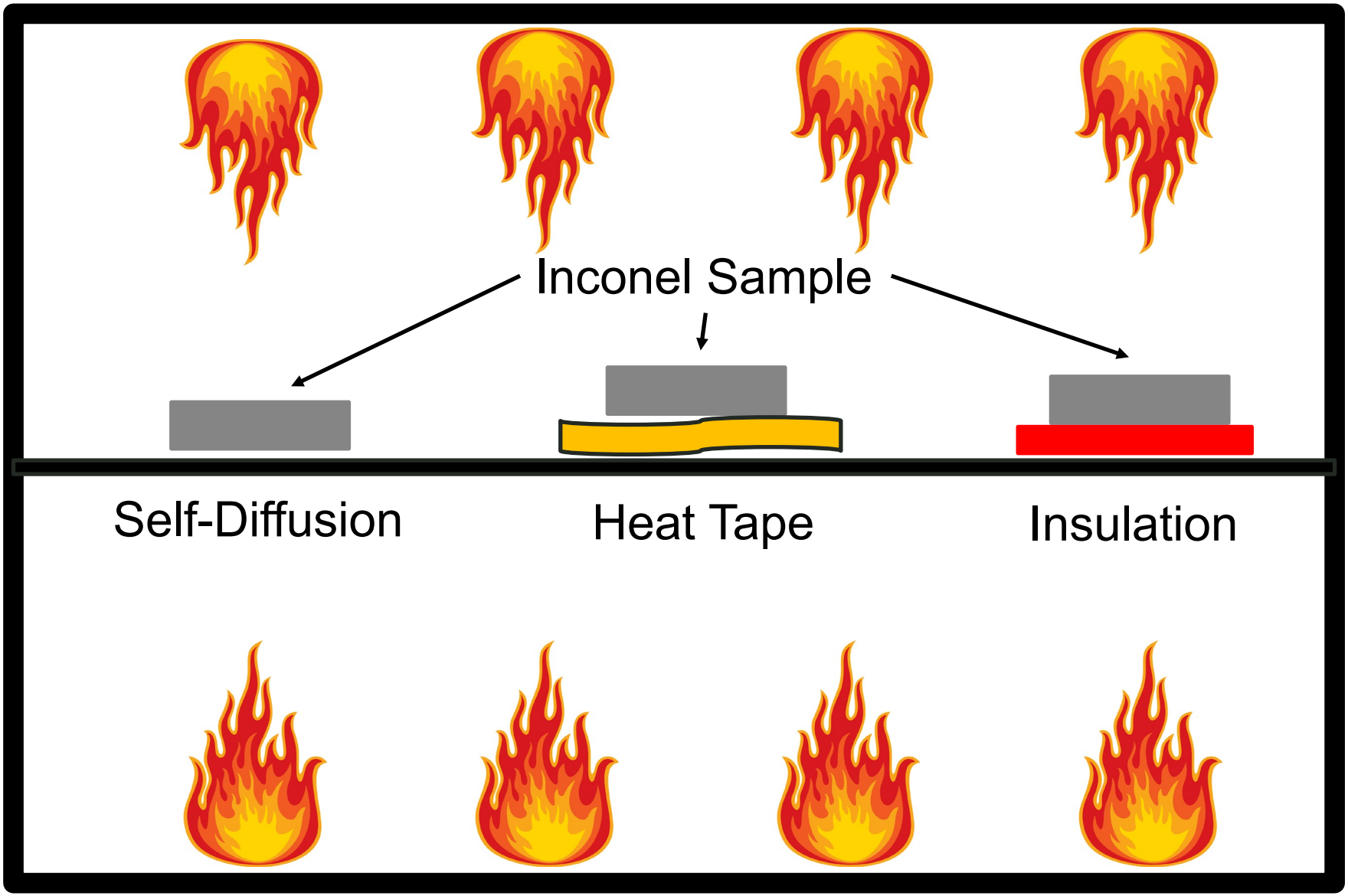


Initial Elemental Levels

Element Type	Inconel Sample (wt%)	Heat Tape (wt%)	Insulation (wt%)
Silicon	~0.00 (may be undetectable)	66.45	24.67
Chromium	~16.00	0.00	0.00

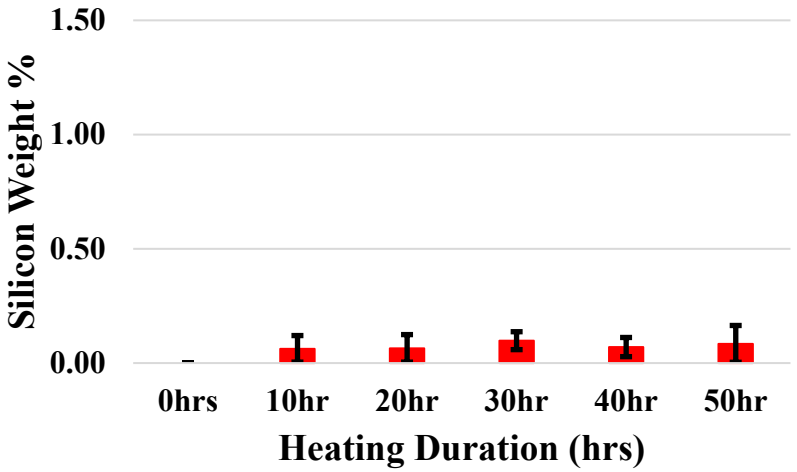
- Chromium present only in sample
  - Internal diffusion (from heat) probably moving Cr to surface
- Silicon could be from heat tape, insulation or both
  - Examine baseline Inconel samples w/ heat tape and insulation

Experimental Results – Possible Silicon Accumulation Pathways

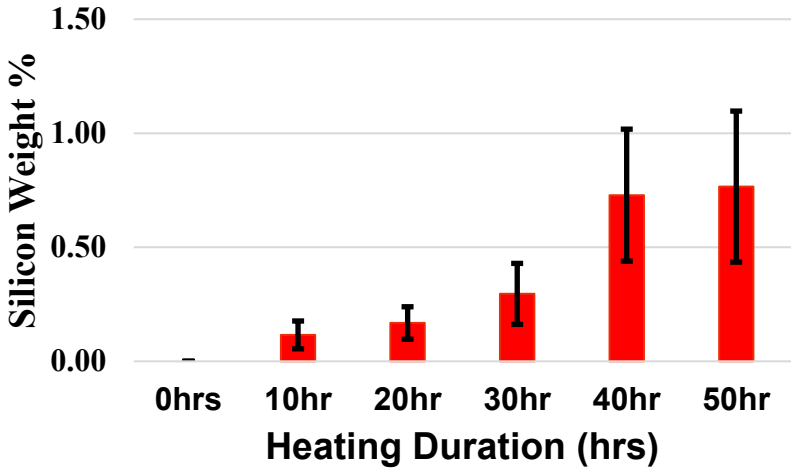


Experimental Results – Possible Silicon Accumulation Pathways Test Results

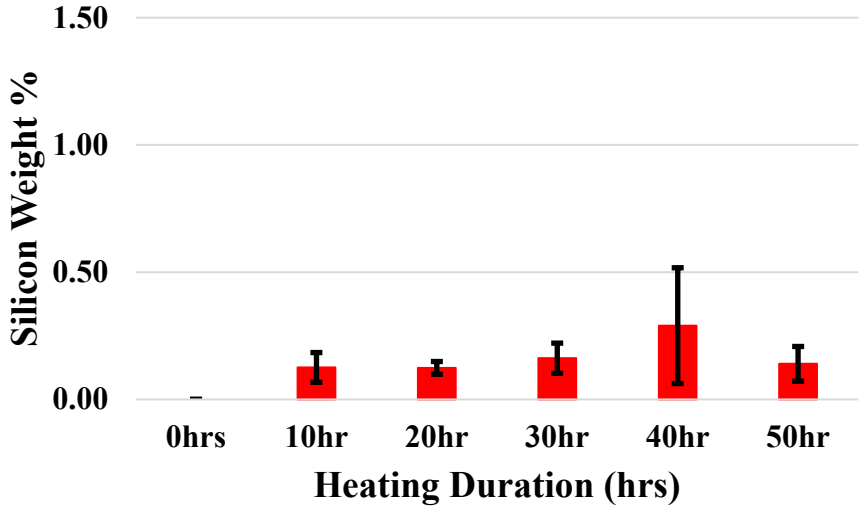
Self-Diffusion



Heat Tape



Insulation

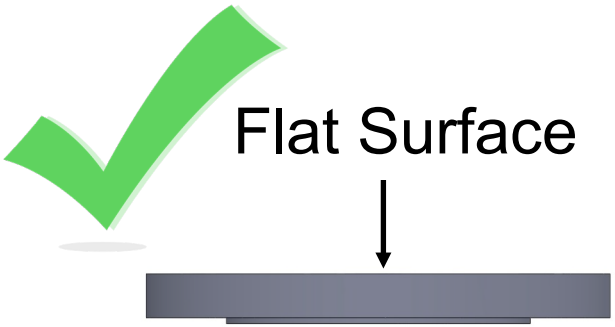


Experimental Results – Test Equipment for Thermal Conductivity

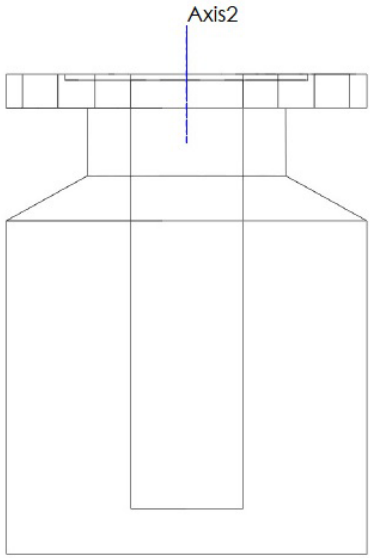
C-Therm Technologies Trident

Characterization & Failure Analysis Group

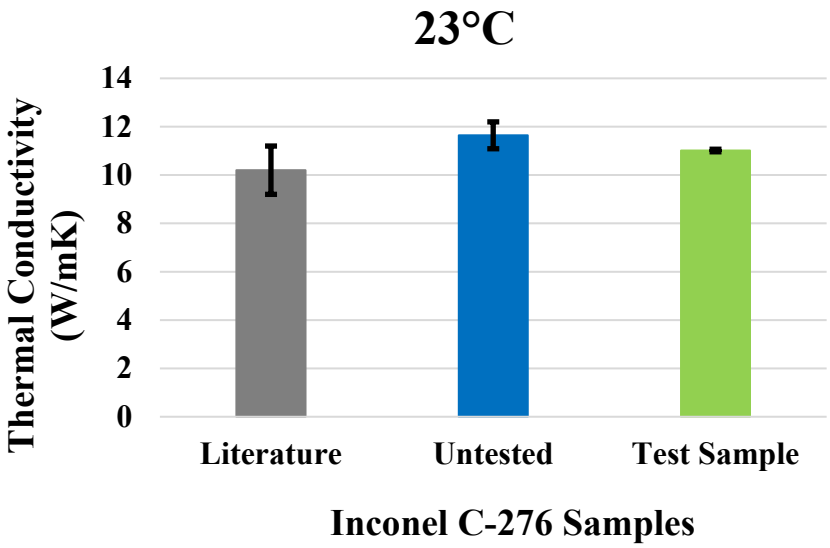
Room Temperature to 200C

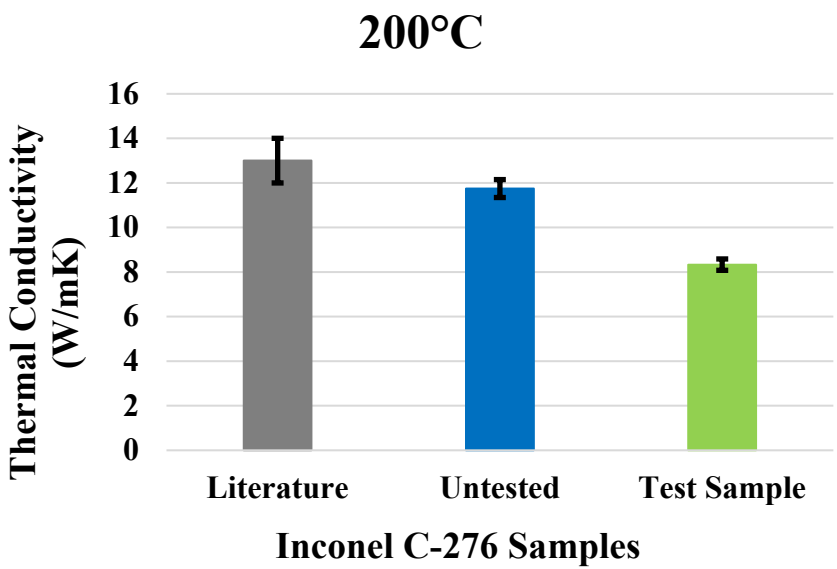


Curved Surface



Experimental Results –Thermal Conductivity Sample Experimental Results



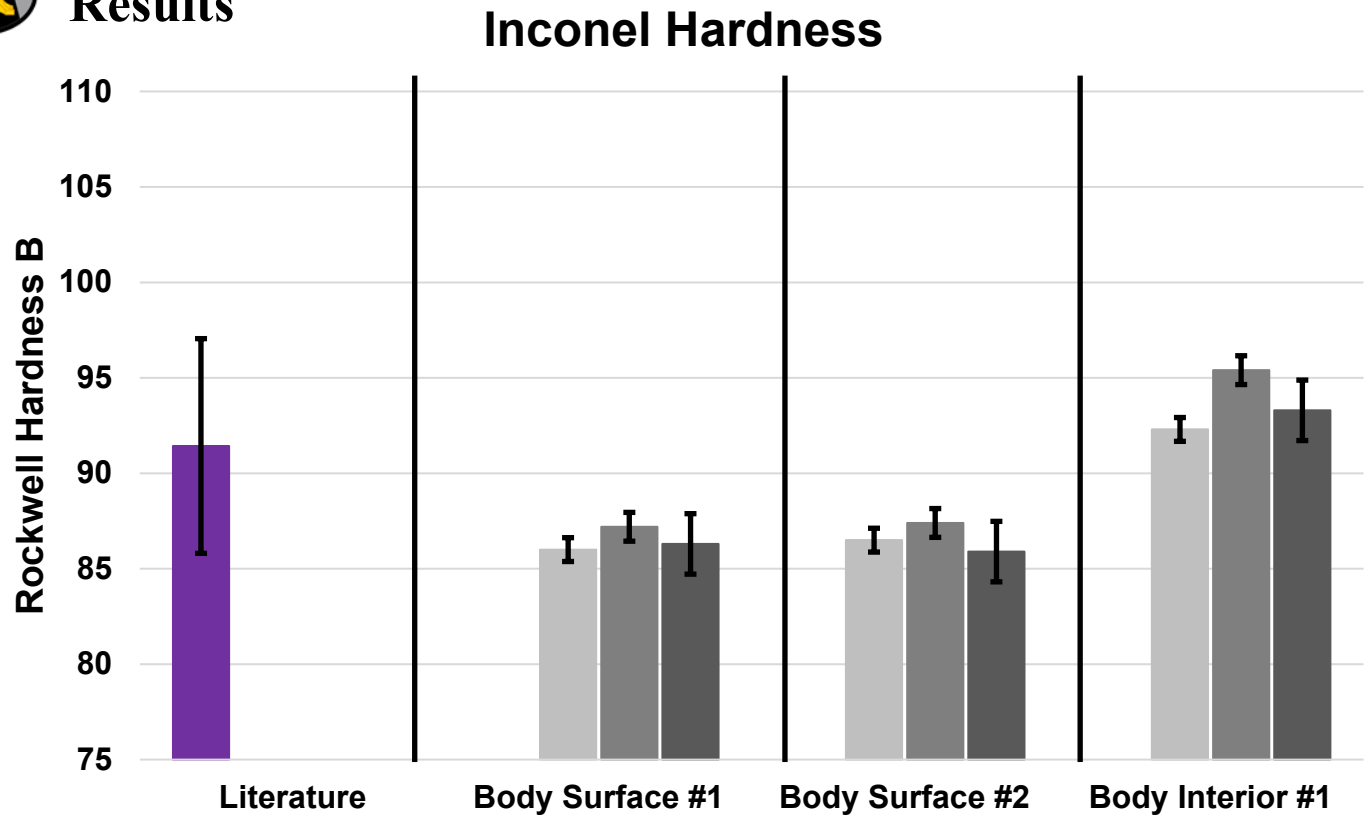


- No statistically significant difference

- Test sample ~36% lower than literature
- This was the lid, which only had 1.96wt% Si. Body had 46.9wt%.
- This was 200C. Difference between literature and sample could be much greater at 700C.
- Further decreased thermal conductivity could realistically result in hot spots and cause failure.

Characterization of Inconel C-276 Hardness

Experimental Results – Sample Hardness Experimental Results



- Three samples tested:
  - 2 exterior body samples near heat tape
  - 1 interior body sample
- Three measurements taken per sample
- Some variability, but all samples within the standard deviation from literature
- Overall, no noticeable changes to material hardness from heat or elemental changes

Conclusions

1. Inconel C-276 elemental composition was altered on the interior and exterior surfaces. The interior elemental composition remained within the sample range shown in literature.
    1. Thick oxide scale present on exterior surface and thick coating present on interior surface.
    2. Exterior and interior material hardness did not appear to be statistically different compared to literature.

2. The exterior surface contained significantly elevated levels of chromium and silicon. SiO_2 would have the greatest impact on thermal conductivity.

3. Heat tape contains a large amount of silicon and experimental results show it is possible to transfer silicon to Inconel samples.

4. Thermal conductivity measurements from lid sample are ~36% lower than literature at 200C.
    1. Difference expected to be larger at 600-700C where premature heat tape failure occurred.
    2. Sample from body could not be tested but would likely increase difference further due to ~47% Si, instead of 1.9%.

Acknowledgements

Office of the Chief Scientist, GVSC

Fuel Cell Technologies Branch, GVSC

Fuels and Lubricants Branch, GVSC

Water Treatment and Handling Branch, GVSC

Characterization and Failure Analysis Branch, GVSC