

FUNDAMENTAL STUDIES OF THE DURABILITY OF MATERIALS FOR INTERCONNECTS IN SOLID OXIDE FUEL CELLS

AGREEMENT NO. DE-FC26-02NT4178

(Start Date September 30, 2002)

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SECA Core Technology Workshop

February 19, 2003



University of Pittsburgh

CMU

Carnegie Mellon University

PROJECT STRUCTURE

NETL

Dr. Lane Wilson	Technical Monitor
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University of Pittsburgh

Prof. Frederick. S. Pettit	Co-principal Investigator
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Prof. Gerald. H. Meier	Co-principal Investigator
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Ms. Julie Hammer	Graduate Student
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Ms. Kelly Coyne	Senior Project Student
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Mr. Earle Hewitt	Technician
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Mr. Scot Laney	NETL Partnership Fellow
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Dr. Chris Johnson	NETL Fellowship Mentor
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Carnegie Mellon University

Prof. Jack L. Beuth	Co-principal Investigator
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Ms. Nandhini Dhanaraj	Graduate Student
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PROGRAM FOCUS

TASK I: Mechanism-Based Evaluation Procedures (Chromia-Forming Alloys)

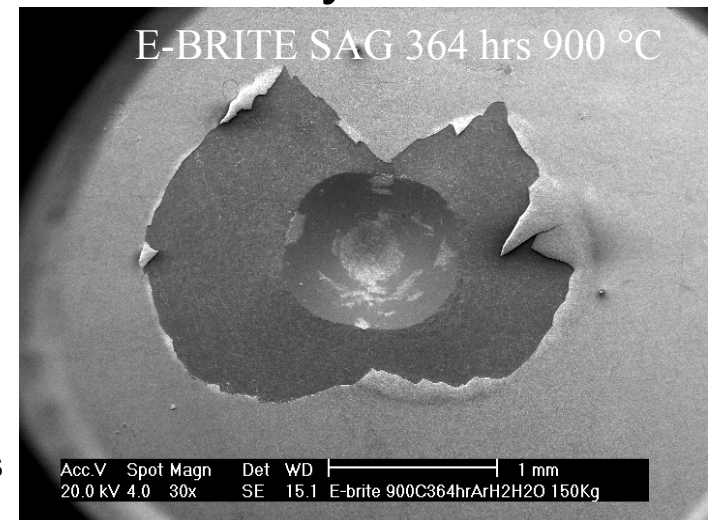
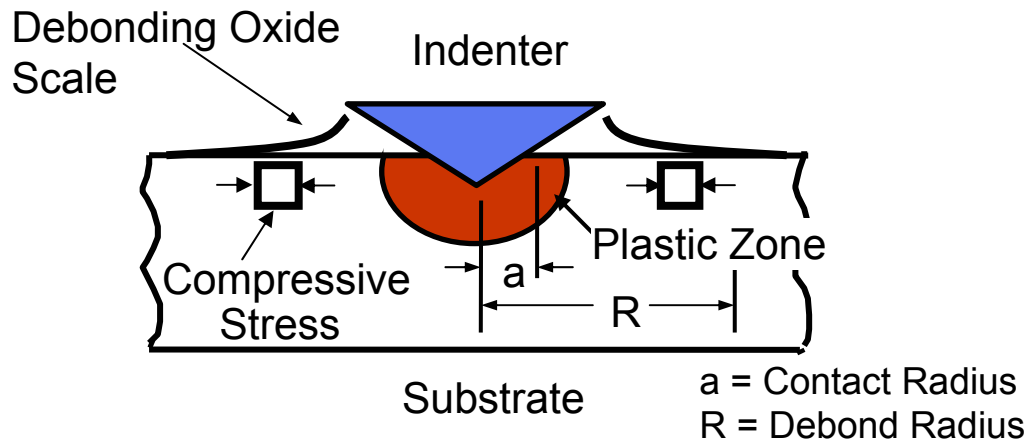
- Characterization of Exposed Fuel Cell Interfaces
- Growth Rates of Chromia Scales on Cr and Ferritic Alloys
- Adhesion of Chromia Scales
- Oxide Evaporation
- Testing of Interconnect Materials in Hydrocarbon Fuel

*Note: An important theme which cuts across Tasks I and II is the establishment of **accelerated testing protocols**.*

PROGRAM FOCUS; TASK II

FUNDAMENTAL ASPECTS OF THERMOMECHANICAL BEHAVIOR

- XRD Stress Measurements (Chromia Films)
- Indentation Testing of Interface Adhesion
- Indentation Test Fracture Mechanics Analysis



*Note: An important theme which cuts across Tasks I and II is the establishment of **accelerated testing protocols**.*

PROGRAM FOCUS

TASK III: Alternative Material Choices

This Task will involve theoretical analysis of possible alternative metallic interconnect schemes including:

- Ni and dispersion-strengthened Ni
- Low CTE Alloys Based on Fe-Ni (Invar)
- Bi-layer Alloys

TASK I: PRELIMINARY RESULTS

Oxidation of Ferritic Alloys

Alloys

- E-BRITE (26 Cr-1 Mo)
- AL 453 (22 Cr + Ce/La)
- Crofer

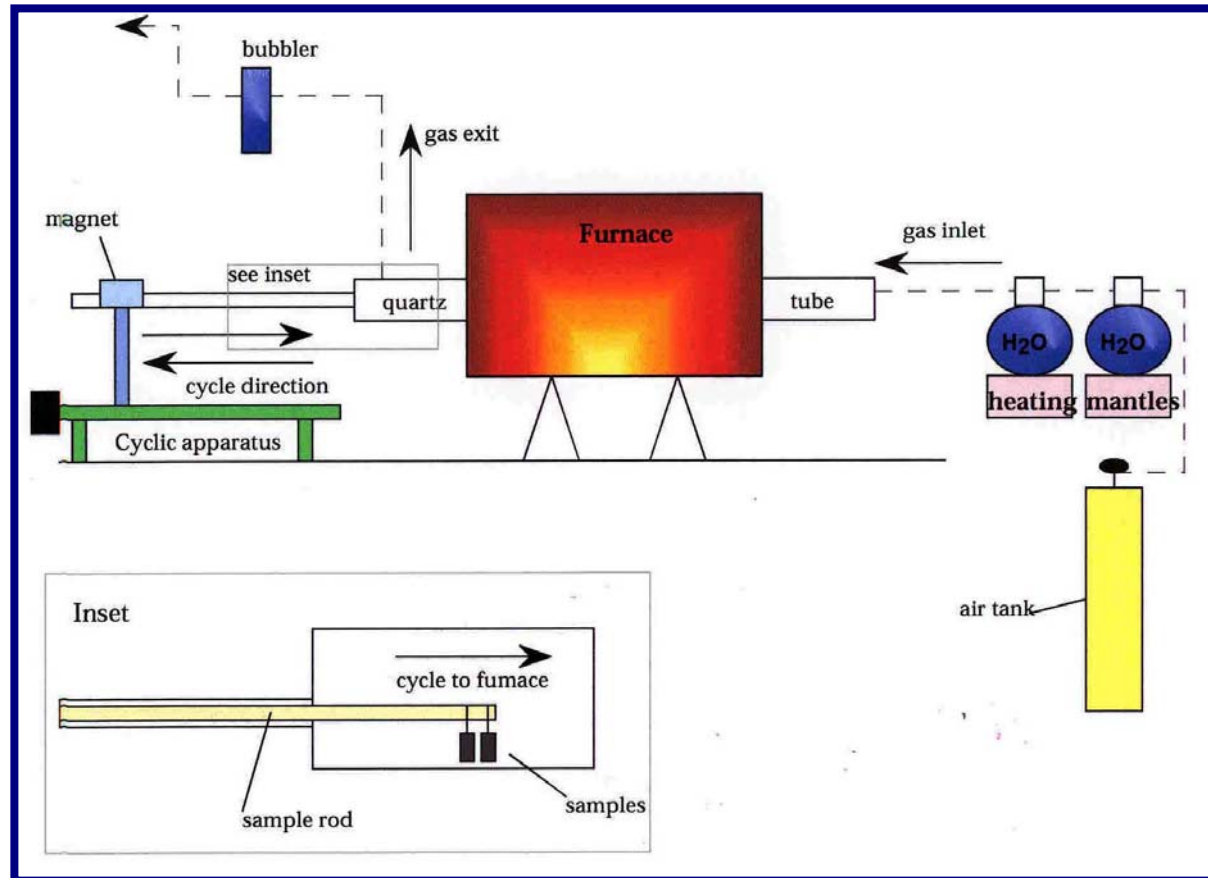
Exposure Conditions

- $T = 900^{\circ}\text{C}$
- One-Hour Cycles
- Atmospheres
 - Dry Air
 - Air + 0.1 atm H_2O
 - Ar/ H_2 / H_2O
($p_{\text{O}_2} = 10^{-17}$ atm)

(700 °C are being initiated)

TASK I: PRELIMINARY RESULTS

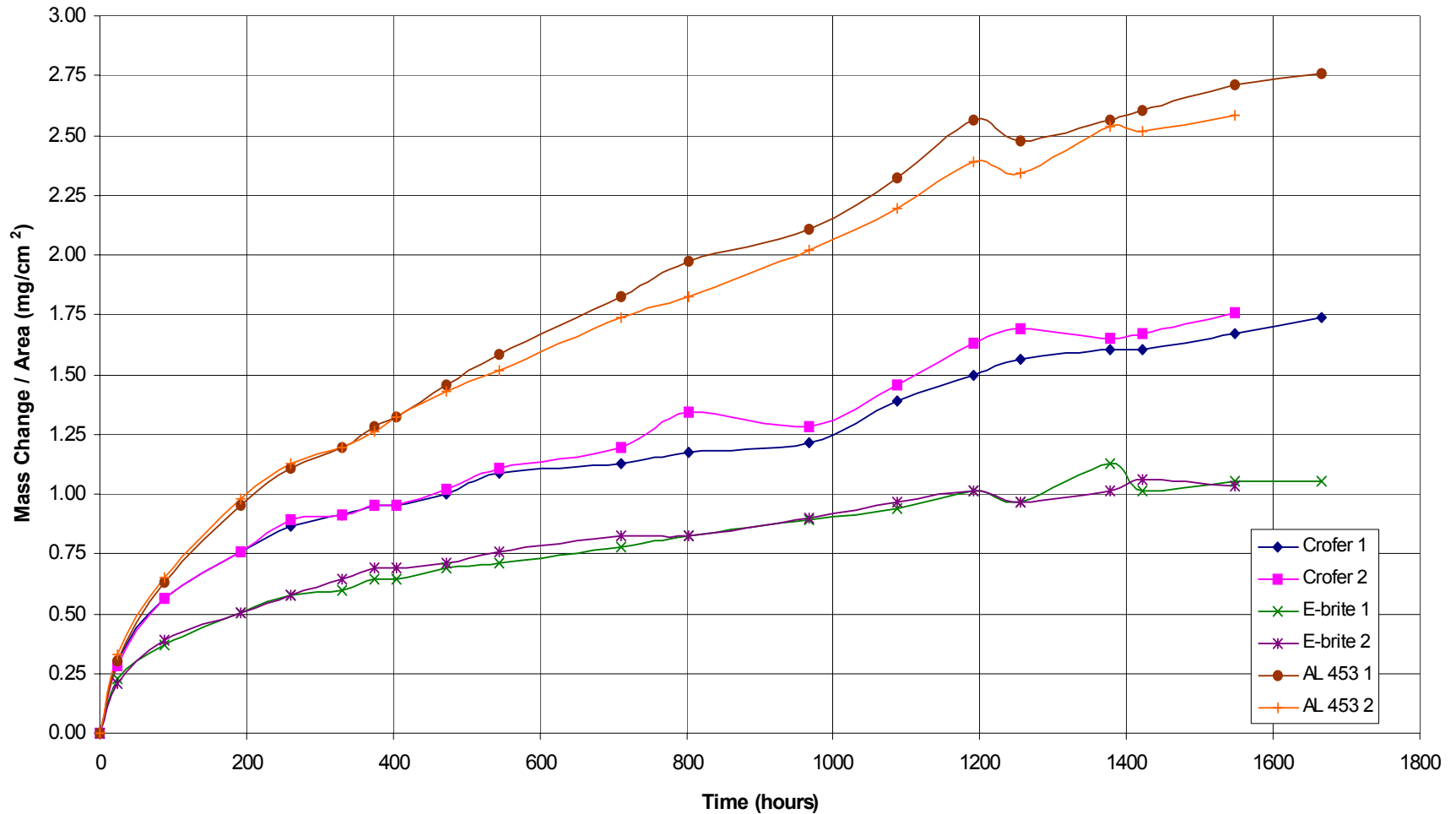
Diagram of Apparatus



TASK I: PRELIMINARY RESULTS

Dry Air Exposures – 900°C

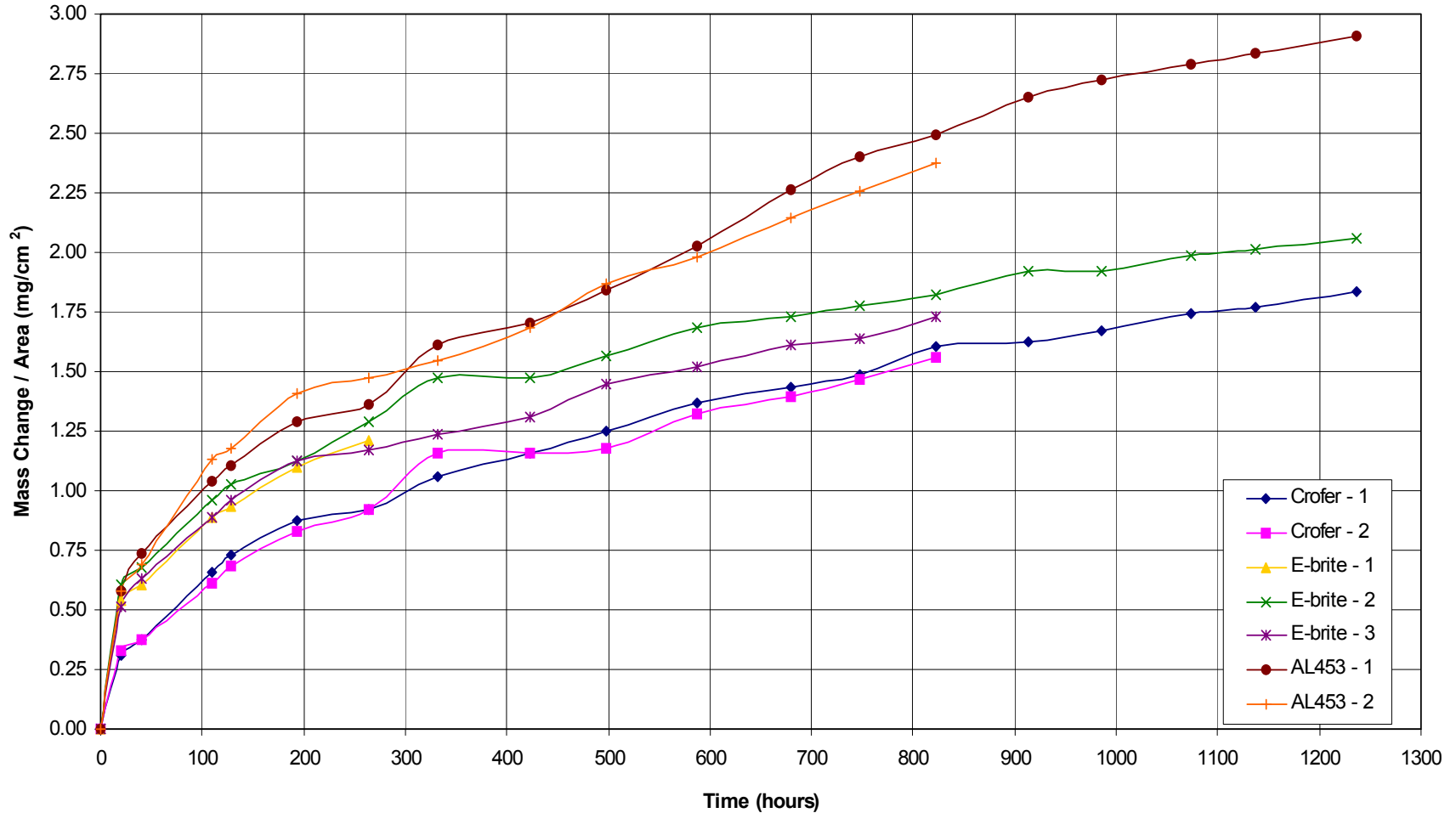
Time vs. Mass Change / Area for Crofer, E-brite, and AL453 Samples (900°C, Dry Air)



TASK I: PRELIMINARY RESULTS

Simulated Anode Gas (Ar-4%H₂, H₂O) Exposures – 900°C

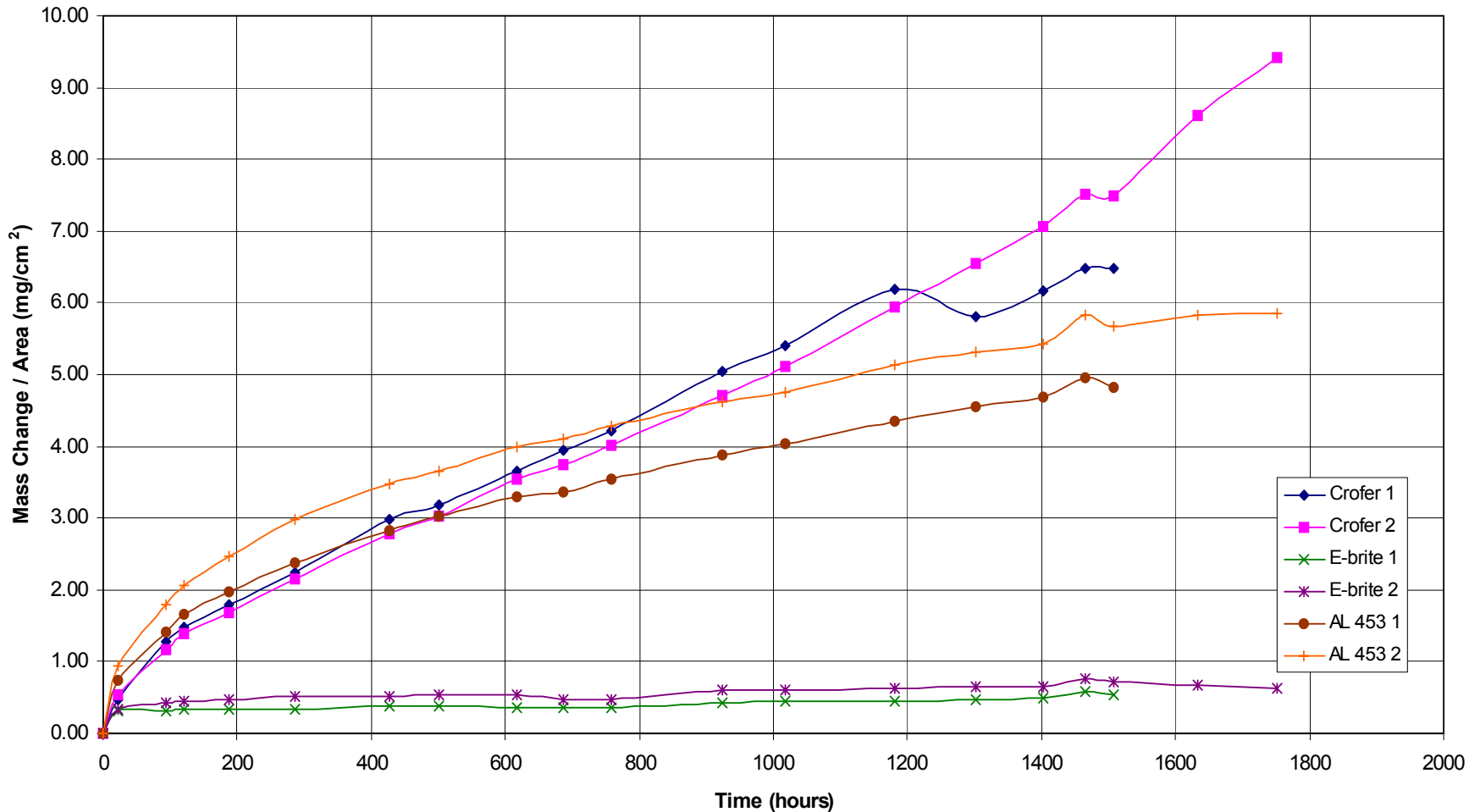
Time vs. Mass Change / Area for Crofer, E-brite, and AL453 Samples (900°C, Ar/H₂/H₂O)



TASK I: PRELIMINARY RESULTS

Wet Air (0.1 atm H₂O) Exposures - 900°C

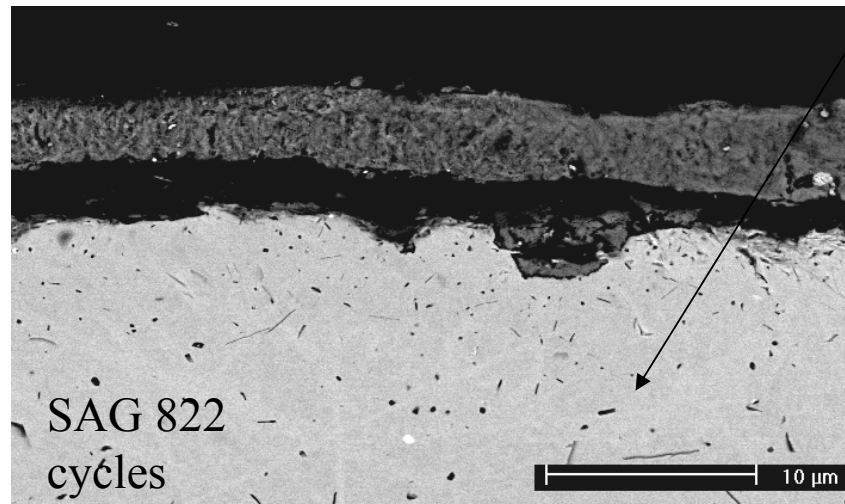
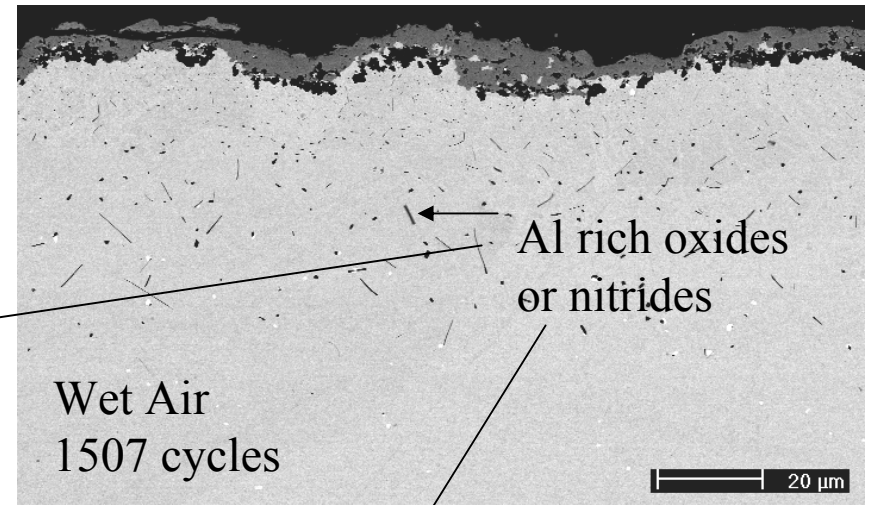
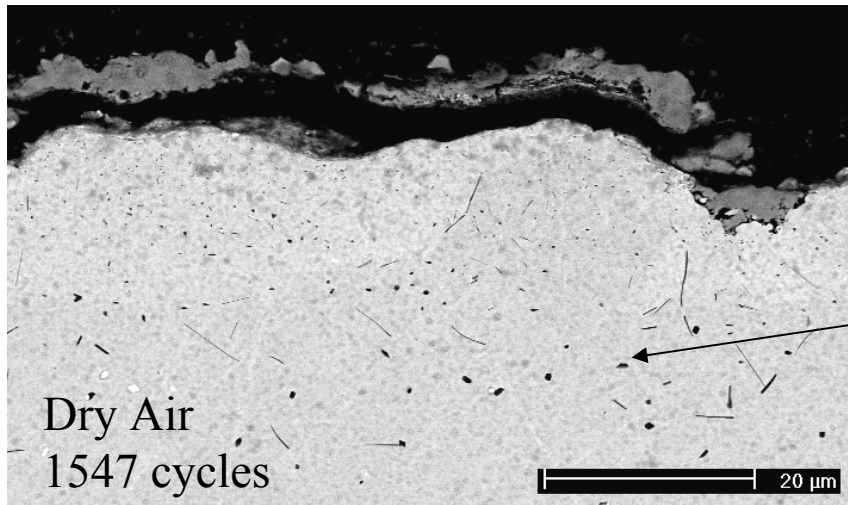
Time vs. Mass Change / Area for Crofer, E-brite, and AL453 Samples (900°C, 1/10 atm H₂O)



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

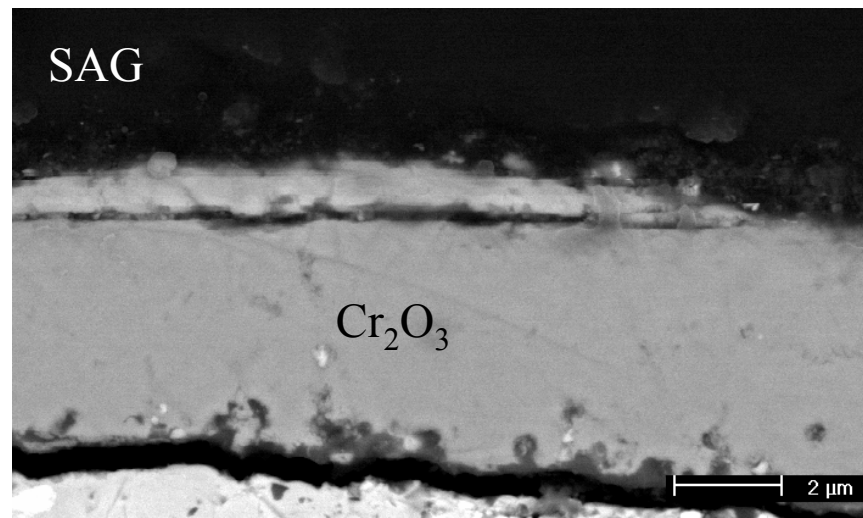
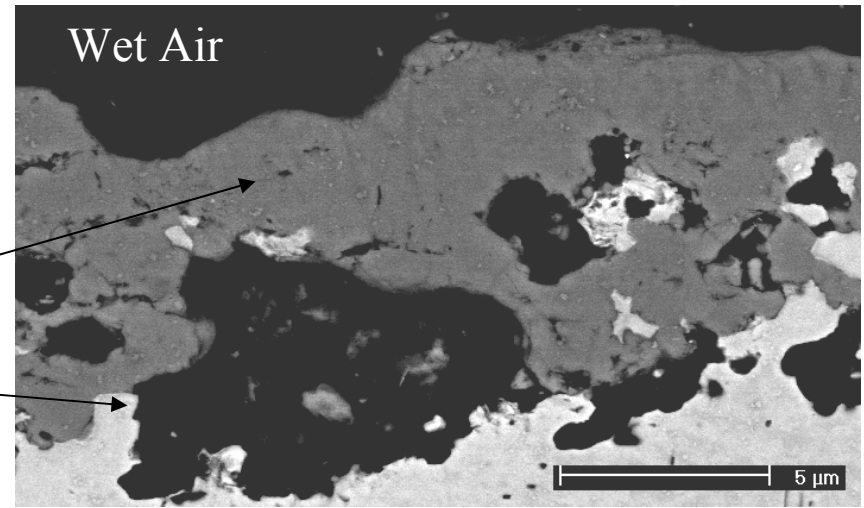
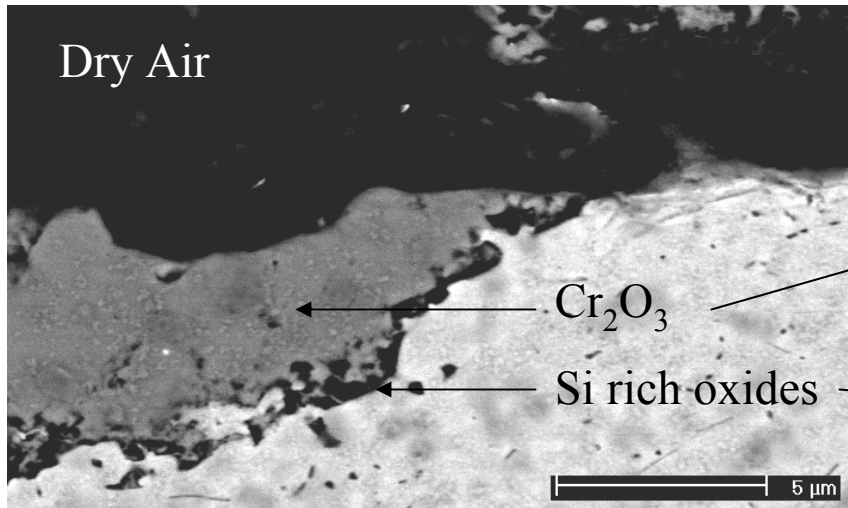
E-brite 900°C



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

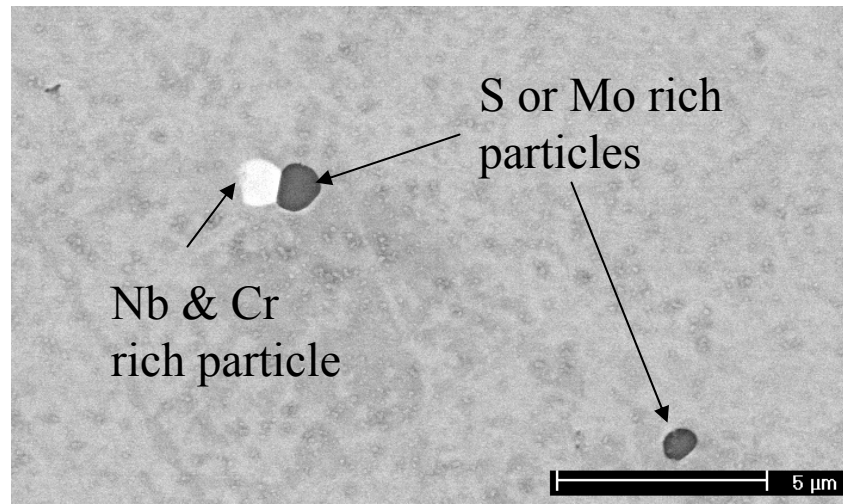
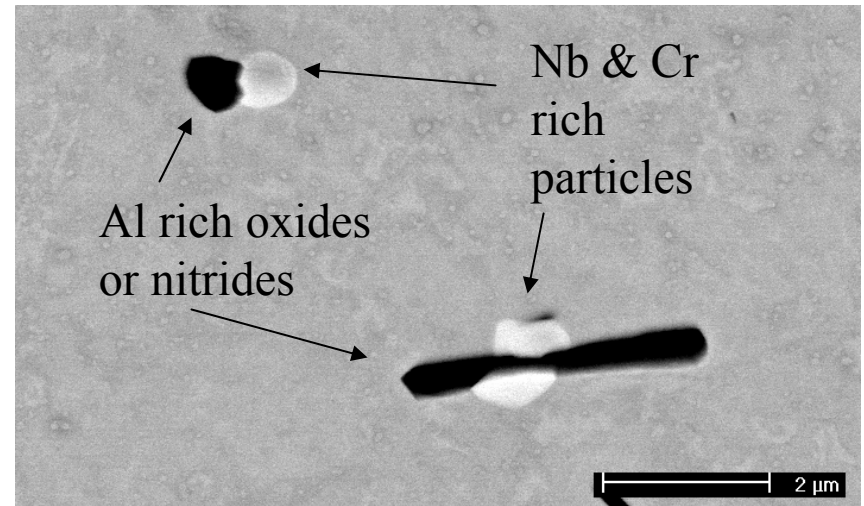
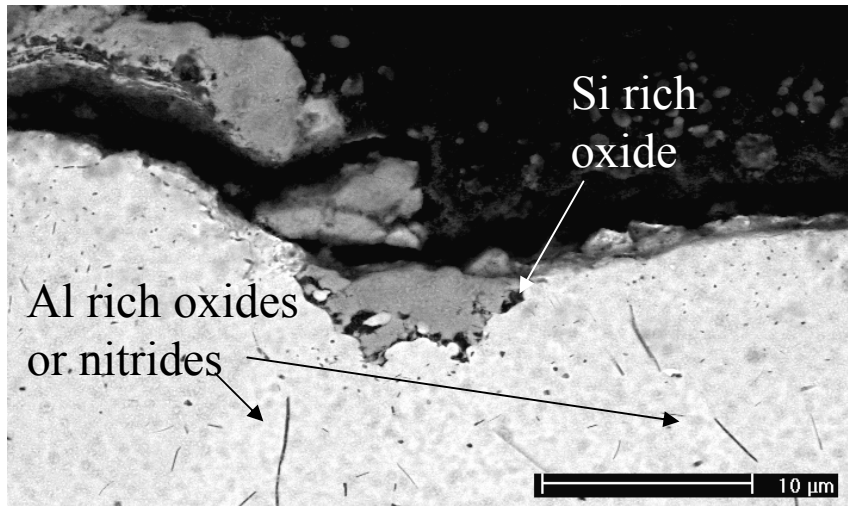
E-brite 900°C



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

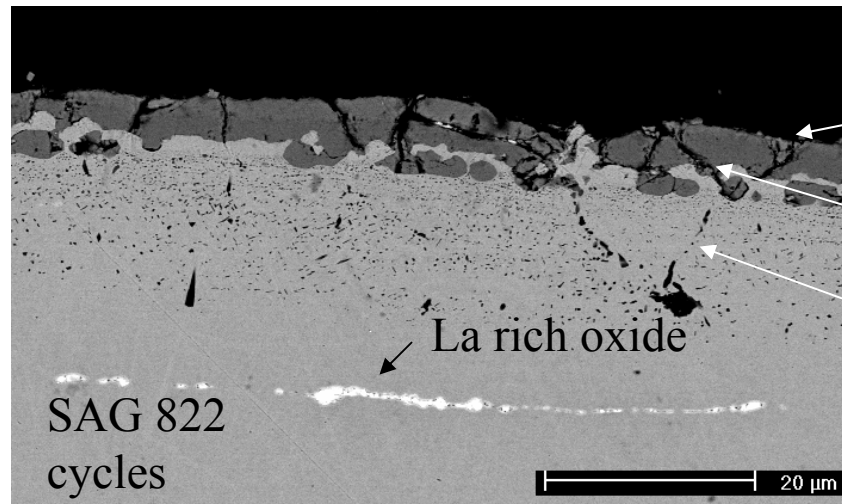
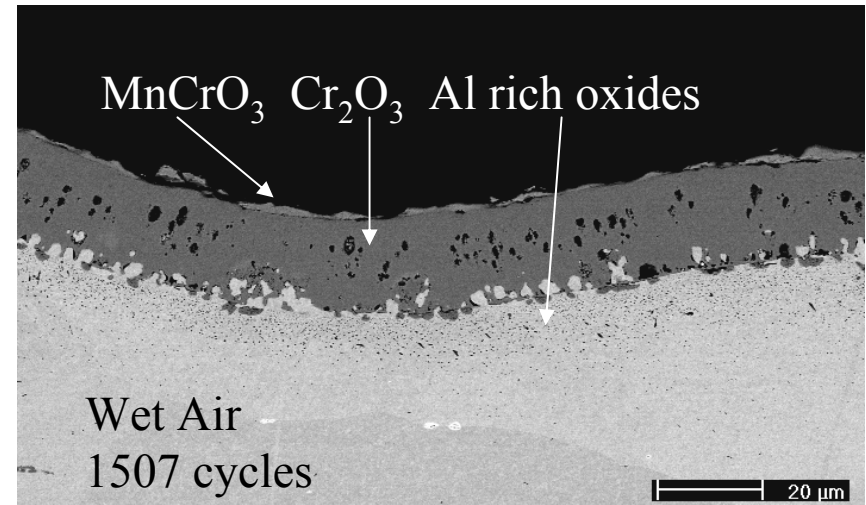
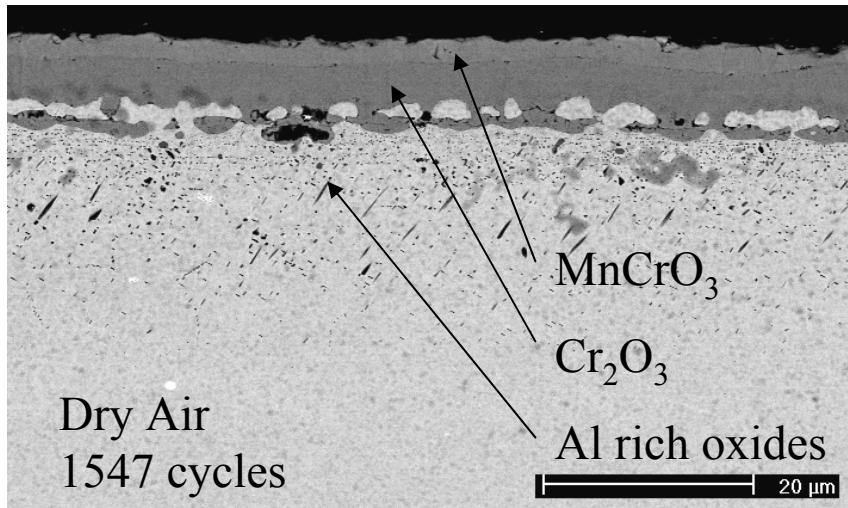
E-brite 900°C



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

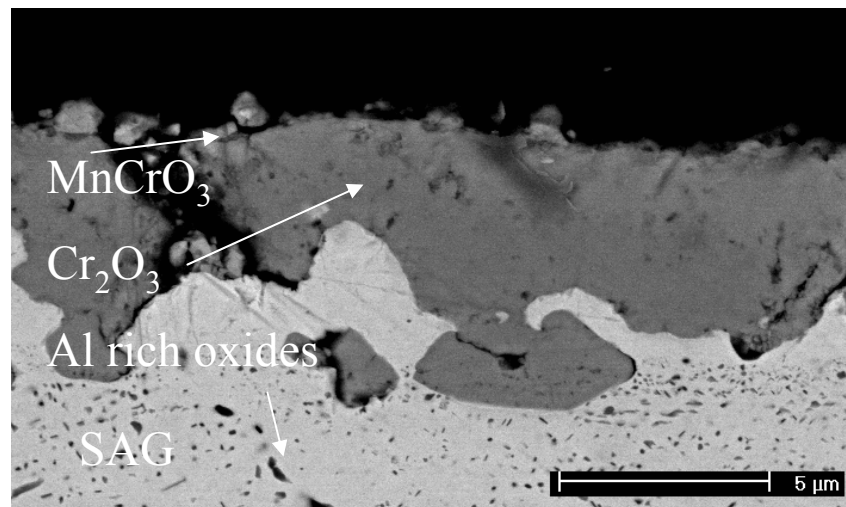
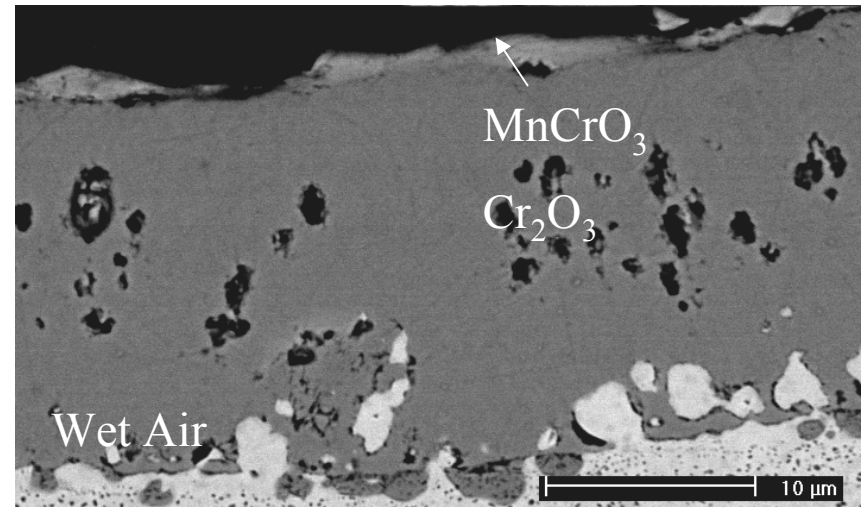
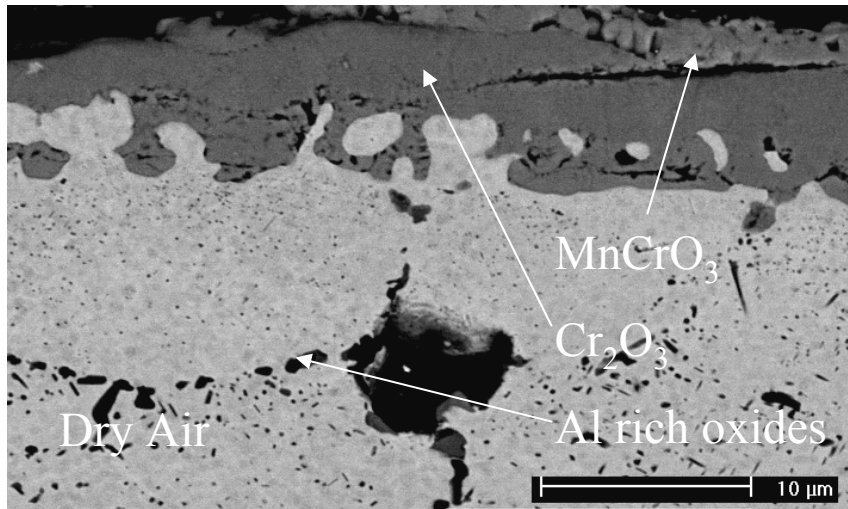
Crofer 900°C



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

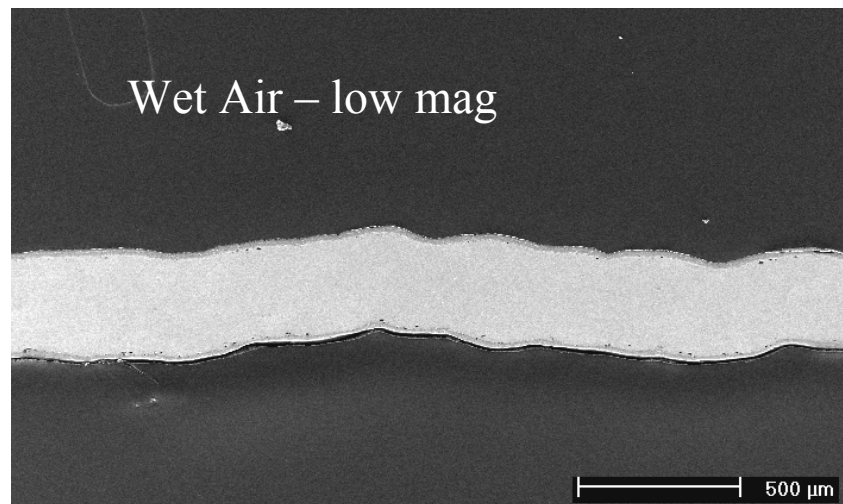
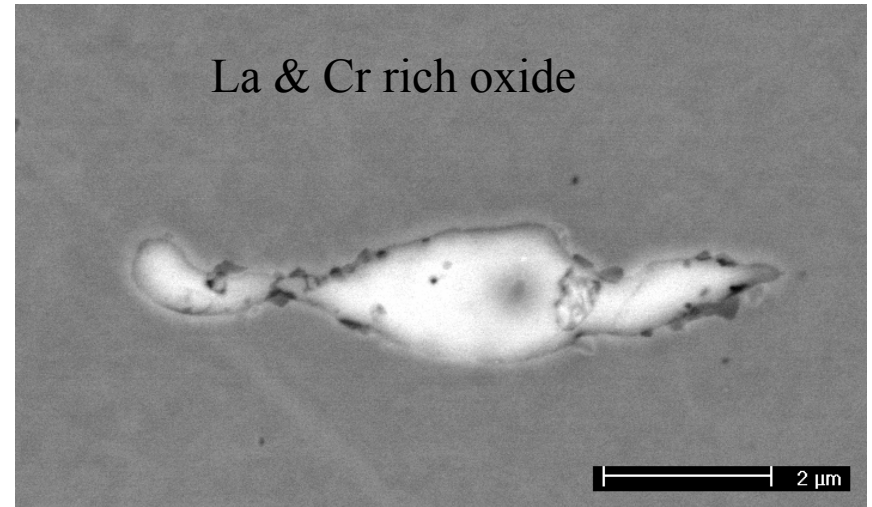
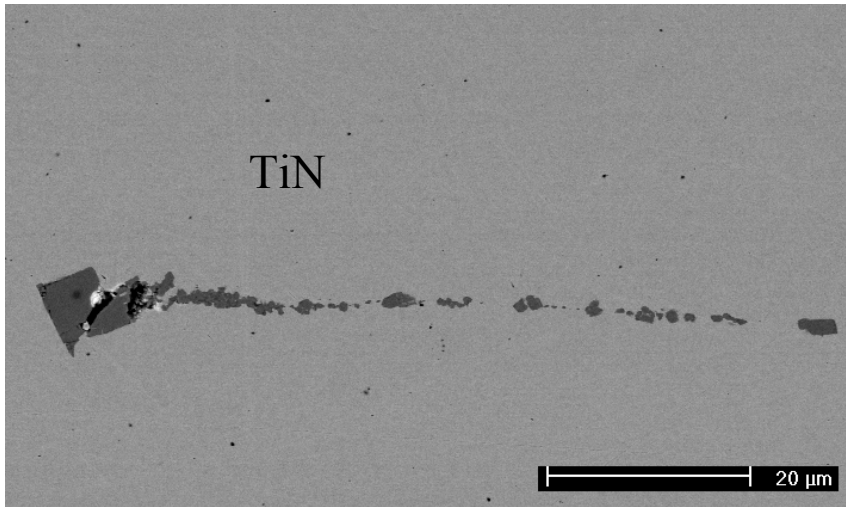
Crofer 900°C



TASK I: PRELIMINARY RESULTS

Microstructural and Phase Identification

Crofer 900°C



TASK I: FUTURE WORK

Work Planned for Next Six Months

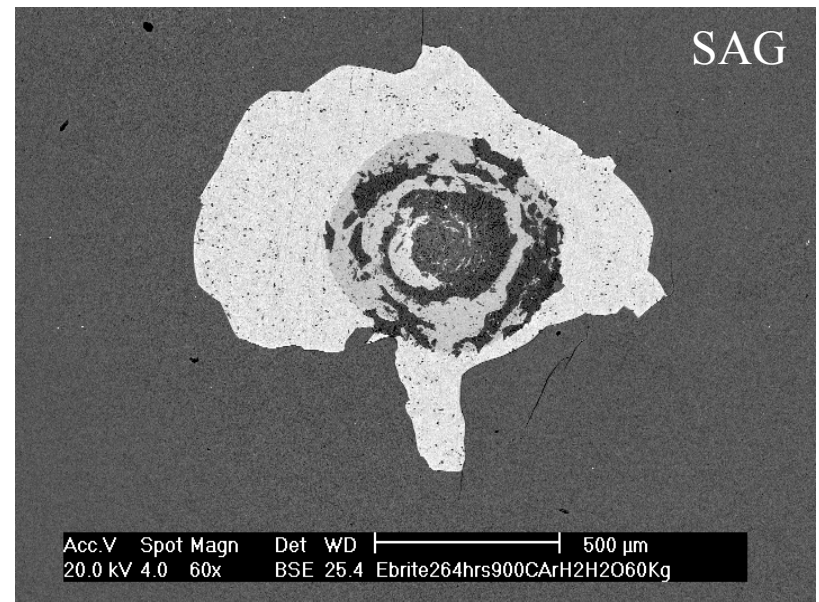
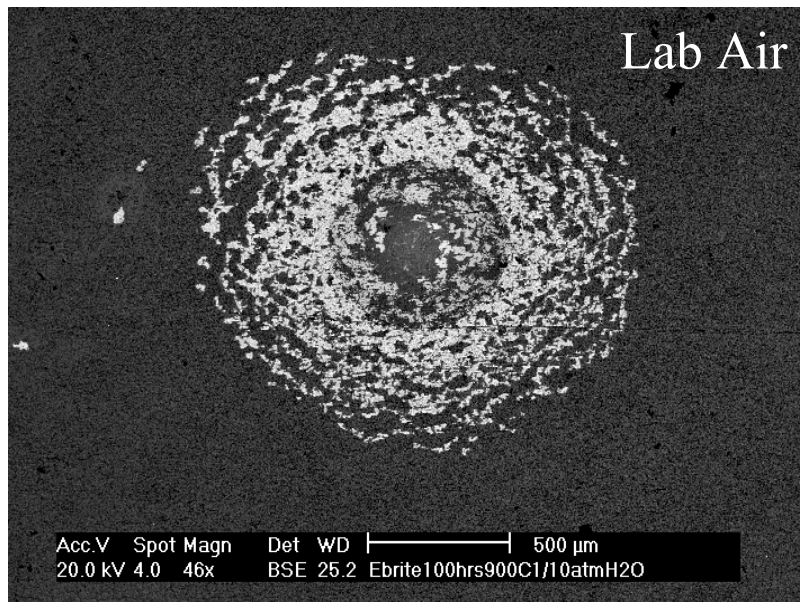
- Cyclic Oxidation at 700°C.
- Initiate Conductivity Measurements on Scales
- Study Effect of Contact with Anode and Cathode Materials
- Experiments to Decrease Chromia Growth Rate (Reactive Elements, Elimination of Grain Boundaries in Chromia)
- Study Effects of Contact with Sealant Glasses
- Investigate Effects of Simultaneous Exposure to Cathode and Anode Gases
- Study Effects of Coatings (Chromate) on Chromia Growth and Evaporation

PRELIMINARY RESULTS

TASK II: THERMOMECHANICAL BEHAVIOR

INDENTATION TESTING OF EXPOSED E-BRITE

- Significant Difference Seen in Lab Air vs. Simulated Anode Gas Exposures at 900°C *at Early Times (100-264 hrs)*
- Consistent with *Long-Term* TGA Results
- Lab Air Specimens Show a Non-Uniform Toughness, with Density of Debonding Decreasing with Radial Distance
- SAG Specimens Show a Peeling of Intact Chromia Scale – Due to Thicker Scale and/or Poorer Adhesion

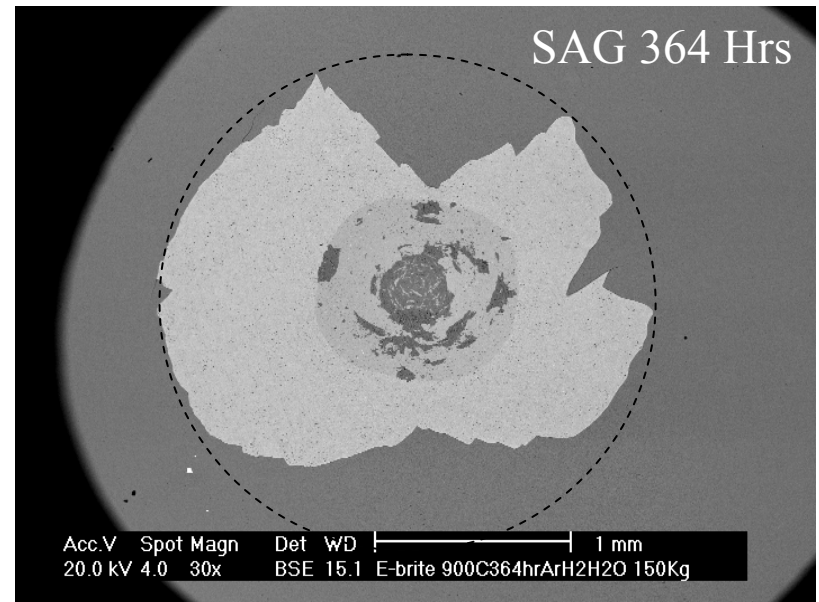
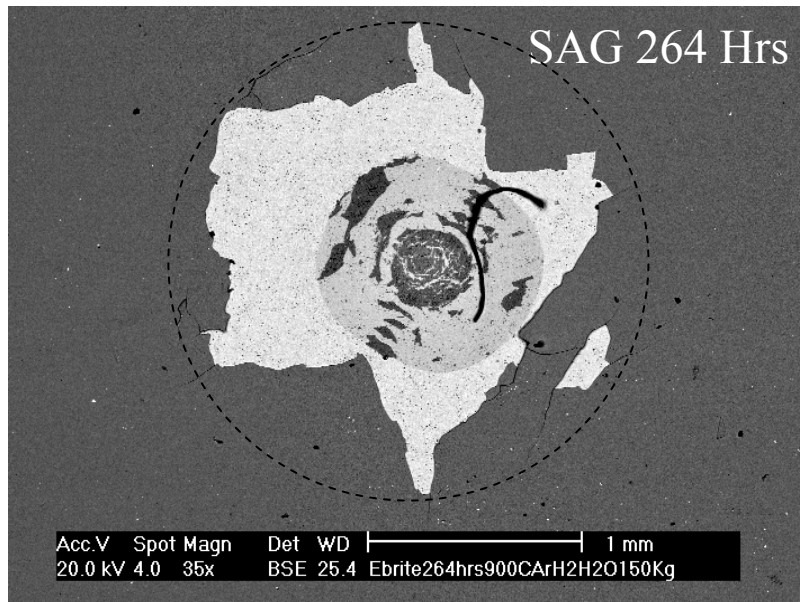


PRELIMINARY RESULTS

TASK II: THERMOMECHANICAL BEHAVIOR

SAG SPECIMEN TOUGHNESS ESTIMATES

- Debond Size Appears to Increase from 264 to 364 Hours of Exposure
- Could be Due to Increased Scale Thickness or Loss of Adhesion (to be determined).
- Indentation Model Results Coupled with Oxide Thickness and XRD Residual Stress Measurements will Yield Interfacial Toughness Values and an Understanding of Mechanisms Leading to Spallation

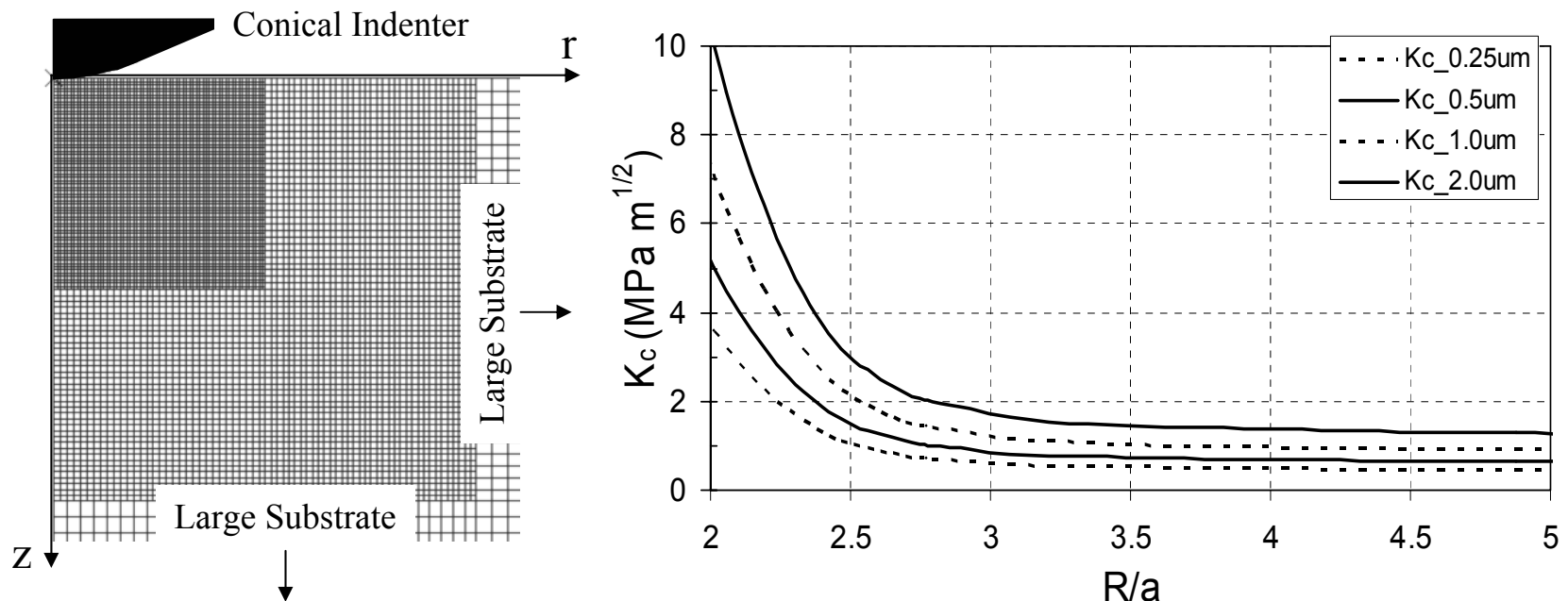


PRELIMINARY RESULTS

TASK II: THERMOMECHANICAL BEHAVIOR

SAG SPECIMEN INDENT FRACTURE MODELING

- Finite Element Model of the Indent Problem: Substrate Strains Transferred to the Chromia Scale
- Fracture Mechanics Formulas Estimate K_c or G_c vs. Normalized Debond Radius (Residual Stress of 1.3 GPa in Chromia Scale)
- $R/a = 2.5$ and $t_{\text{oxide}} = 2\mu\text{m}$ Yields: $K_c = 3 \text{ MPa m}^{1/2}$; $G_c = 35 \text{ J/m}^2$

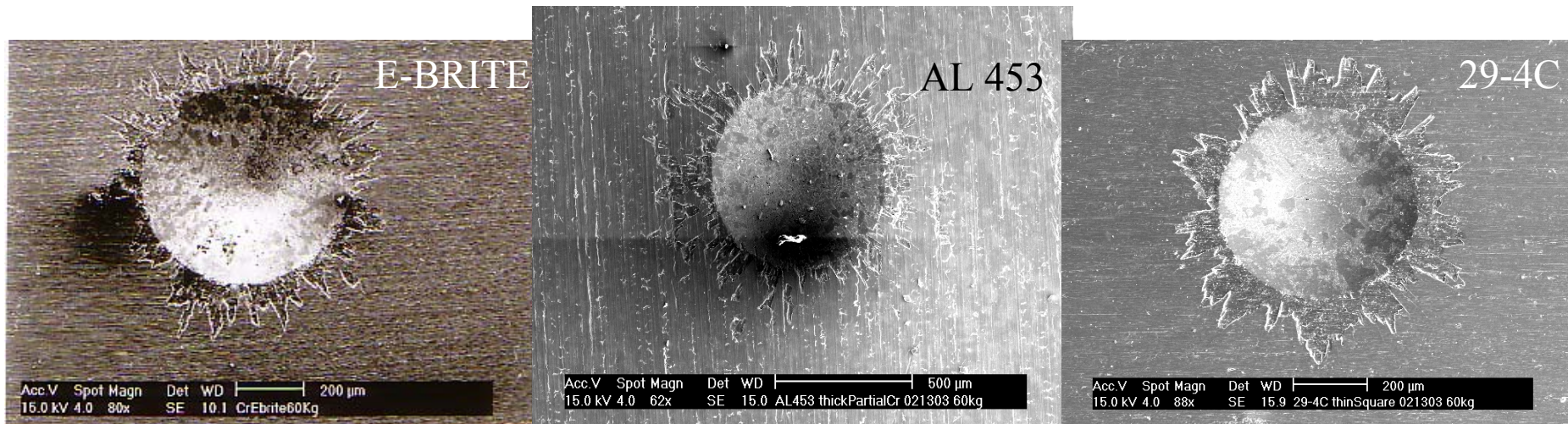


PRELIMINARY RESULTS

TASK II: THERMOMECHANICAL BEHAVIOR

AS-PROCESSED CHROMATE COATED SPECIMENS

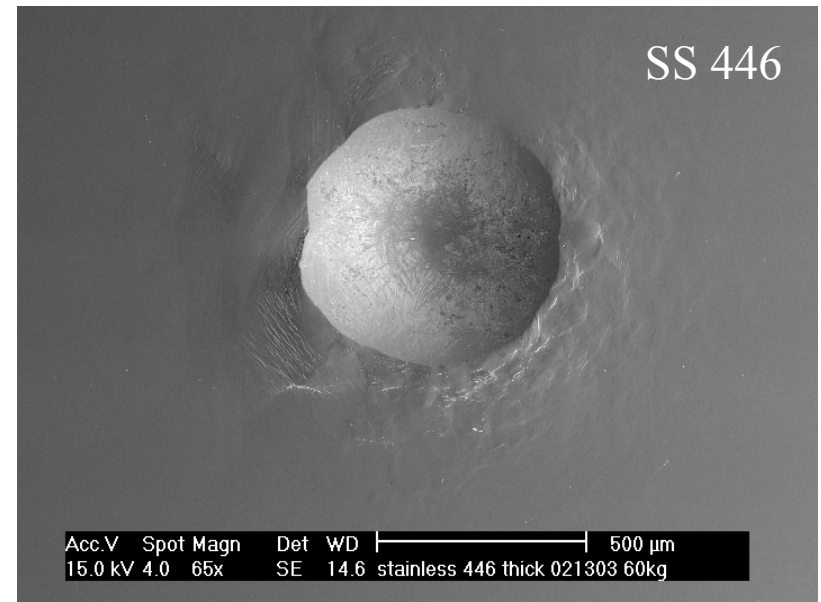
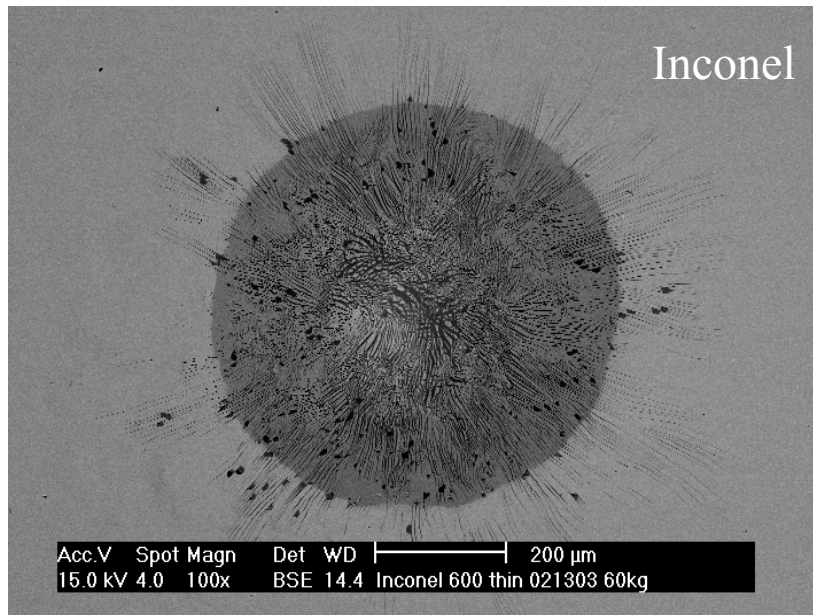
- Specimens of $\text{La}_{0.8}\text{Sr}_{0.2}\text{CrO}_3$ on E-BRITE, AL 453 and AL 29-4C , Indented Before Exposure (from PNNL)
- Coating can Add to the Stored Energy Driving Chromia Spallation
- All 3 Substrate Systems Show Indent-Induced Spallation (Confined to the Near-Indent Region)



PRELIMINARY RESULTS

TASK II: THERMOMECHANICAL BEHAVIOR AS-PROCESSED CHROMATE COATED SPECIMENS

- LaCrO_3 Coating on Inconel and SS 446
- NETL and Drexel Providing Specimens, Coating Properties
- Spallation is not Seen, but Radial Cracking Seen for Inconel
- Key: Is As-Processed Behavior Indicative of Long-Term Behavior?



TASK II

WORK PLANNED FOR NEXT SIX MONTHS

- Modeling of Indentation of E-BRITE and Other Substrate Systems: Track Toughness Loss in Systems Exhibiting Debonding, Identify Mechanisms
- Indentation Tests on E-BRITE for Longer Exposures in Lab Air and Simulated Anode Gas : Incorporate Oxide Thickness and XRD Stress Measurements
- Study of Adherence of Exposed Coated Specimens
- Study Adherence of Sealant Glasses on Interconnect Materials
- Feasibility Study of Toughness Testing of Anode-Supported Fuel Cell Structures (LaSrMnO_3 and YSZ on Porous Ni/YSZ Substrate)

TASK III: PRELIMINARY RESULTS

Alternative Material Choices

- This Task involves a theoretical evaluation of alternate metallic materials which have properties superior to the ferritic alloys.
- The most promising materials will be fabricated and tested in Phase II.

COLLABORATION

Collaboration, advice, and support from the following organizations is gratefully acknowledged:

- PNNL (P. Singh, G. Yang)
- NETL Morgantown (C. Johnson, L. Wilson, G. Richards)

We would welcome the opportunity to collaborate with other branches of the National Laboratories and Industry.

SUMMARY AND CONCLUSIONS

The aim of this project is to evaluate the chemical and thermomechanical stability of ferritic alloys in the fuel cell environment.

The understanding gained will be used to attempt to optimize the properties of the ferritic alloys.

A parallel study is evaluating the potential use of alternate metallic materials as interconnects.