BORIDE BASED ELECTRODE MATERIALS WITH ENHANCED STABILITY UNDER EXTREME CONDITIONS FOR MHD DIRECT POWER EXTRACTION

University of Idaho
Indrajit Charit (Principal Investigator)
icharit@uidaho.edu

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Other Team Members: Krishnan S. Raja (Co-PI)
Current Students: Steven Sitler (Grad), James Zillinger (Undergrad)

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PROJECT GOALS

- Develop Electrode Material
  - Transition metal boride based electrode
- Required Electrode Properties
  - High electrical conductivity
  - Good oxidation resistance - borides need improvement at high temperatures
  - Durability in corrosive environments
- Increase Oxidation Resistance at High Temperatures
  - Electrochemical anodization
  - Additives

Milestones

- Y2Q1: Mechanochemical Synthesis
- Y2Q2: Spark Plasma Sintering
- Y2Q3: Anodization Process
- Y2Q4: High Temperature Oxidation
- Y2Q4: M.S. Student Graduation
- Y3Q4: Ph.D. Student Graduation

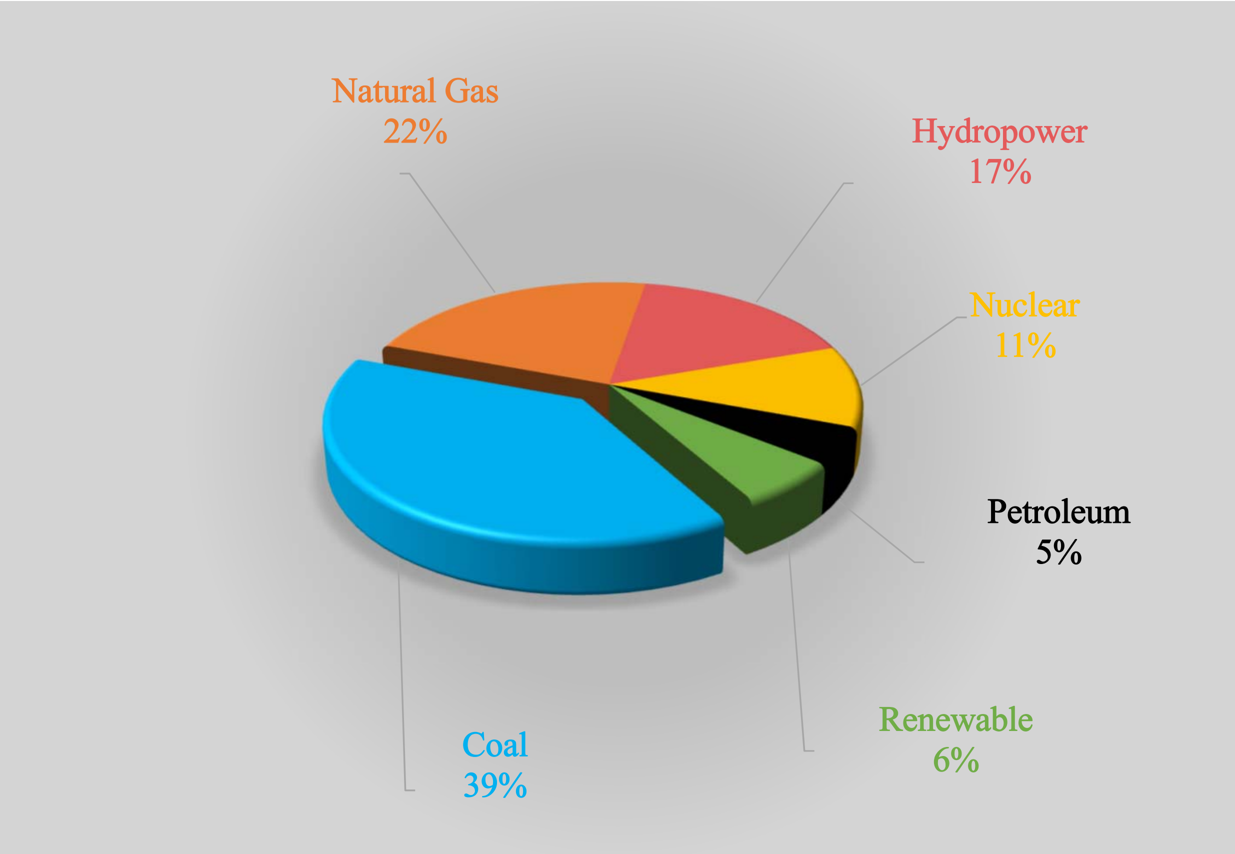
OUTLINE

- INTRODUCTION
- RESULTS & DISCUSSION
- ACCOMPLISHMENTS
- CONCLUSIONS
- FUTURE WORK

INTRODUCTION

GLOBAL ENERG

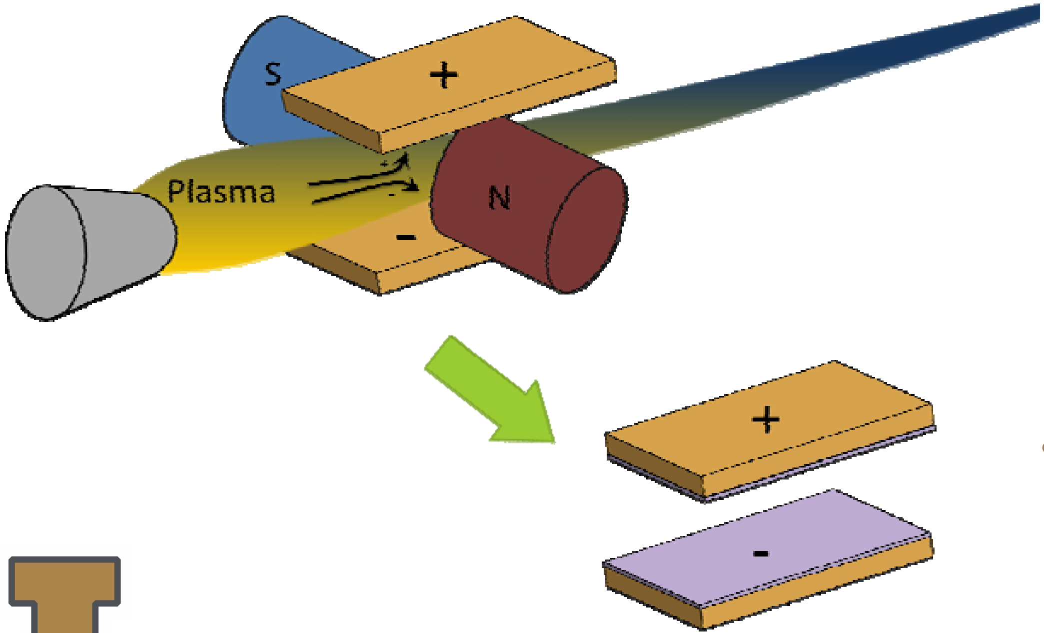
- Thirty nine percent of global
  is produced by coal power.
- The MHD system is designe
  in making coal power cleane
- By adding the MHD system
  existing coal plants, the ene
  output can be doubled.
- Effectively cut down greenh
  emissions.



Data from: http://www.tsp-data-portal.org/Breakdown-of-Electricity-Generation-by-Energy-Source#tspQvChart

MAGNETOHYDRODYNAMICS:

DIRECT POWER EXTRACTION



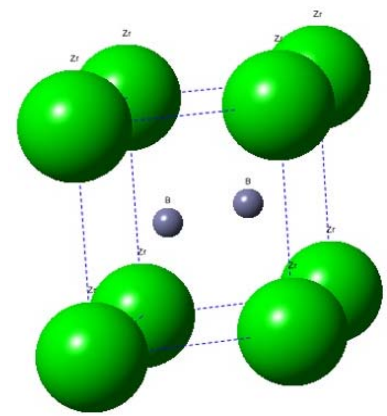
- Extraction Chamber Conditions:
  - Flame temperature: > 3000 K (2727 ^{o}C)
  - Oxidizing atmosphere with possible atomic oxygen present in the plasma
  - Potassium salt added for conductivity creating conditions for hot corrosion.
  - Temperature at electrode surface: > 1700 K (1427 ^{o}C)
- Required Electrode Coating Properties:
  - High electrical conductivity
  - Good oxidation resistance
  - Durability in corrosive environments

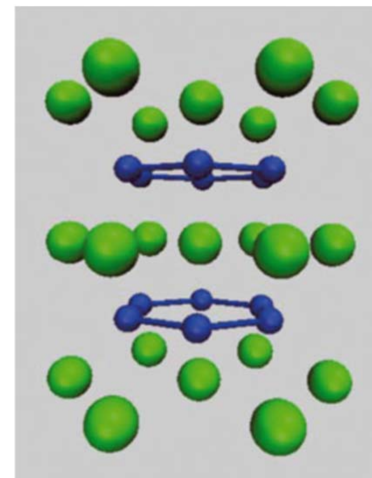
WHY DIBORIDES?

- HfB_2-ZrB_2 solid solution (strengths)
  - Superior electrical conductivity
  - Extremely high melting point
  - Good thermal shock resistance
  - Good oxidation resistance up to 1200 ^{o}C
- HfB_2-ZrB_2 solid solution (weakness)
  - Oxidation resistance at temperatures higher than 1200 ^{o}C needs improvement.

WHY MIX THEM?

- Both ZrB_2 and HfB_2 hexagonal crystal structure with a symmetry group *P6/mmm* (top)
- Similar lattice parameters
- Layered atomic arrangement of the diboride structures: illustration (bottom)





Lattice parameters of ZrB_2 and HfB_2

Material	Crystal Structure	a (nm)	c (nm)	Density (g/cm^3)	Melting Point (°C)
ZrB_2	Hexagonal (D_{6h}), *P6/mmm*	0.317	0.353	6.08	~3246
HfB_2	Hexagonal (D_{6h}), *P6/mmm*	0.314	0.348	10.5	~3250

John W. Lawson, Charles W. Bauschlicher Jr., and Murray S. Daw, Ab Initio Computations of Electronic, Mechanical, and Thermal Properties of ZrB_2 and HfB_2, J. Am. Ceram. Soc., 94 [10] 3494–3499 (2011)

WHY ADDITIVES?

LaB_6 was researched in a ZrB_2 system by Zhang *et al.*

- Significant improvement at ultra-high temperatures due to excellent oxide adhesion.
- Desired to stay away from gasification leading to porous ZrO_2 layer.

Gd_2O_3 was tested in a ZrB_2 system by Jayaseelan *et al.*

- Found that at 1600 °C, Gd_2O_3 gave best oxidation resistance improvement
- Desired to stay away from liquid silica protection layer

The effects of Ta addition to a ZrB_2 system were tested by Opila *et al.*

- Short term exposure only
- Helped at 1627 °C, but not at higher temperatures
- Higher temperatures proved Ta addition to be harmful, not helpful

1. Jayaseelan, D. D., Zapata-Solvas, E., Brown, P. & Lee, W. E. In situ Formation of Oxidation Resistant Refractory Coatings on SiC-Reinforced ZrB2 Ultra High Temperature Ceramics. J. Am. Ceram. Soc. 95, 1247–1254 (2012).
2. Zhang, X., Hu, P., Han, J., Xu, L. & Meng, S. The addition of lanthanum hexaboride to zirconium diboride for improved oxidation resistance. Scr. Mater. 57, 1036–1039 (2007).
3. Opila, E., Levine, S. & Lorincz, J. Oxidation of ZrB2- and HfB2-based ultra-high temperature ceramics: Effect of Ta additions. J. Mater. Sci. 39, 5969–5977 (2004).

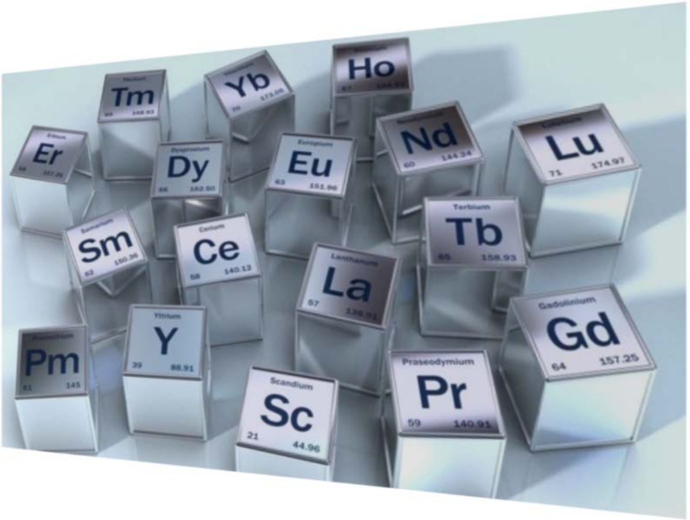
WHAT IS MISSING?

- Research on additives done only on single borides, not 1:1 solid solutions of HfB_2:ZrB_2
- Research done mainly for hypersonic applications
  - Not looking for electrical conductivity
  - Short term only vs. long term in the present case
- Almost all studies done incorporate 10-30 vol% SiC
  - Bulk property changes would be detrimental in the MHD system
- No research on anodization to reduce oxidation for this material

INCREASE OXIDATION RESISTANCE

1. Electrochemical anodization
   a) Single-step anodization
   b) Two-step anodization

2. Additives
   a) Rare earth additives
   b) Metal additives to get metal-rich borides



http://www.crrpe.com/UpLoadFile/logo/yh201503020926334384.jpg

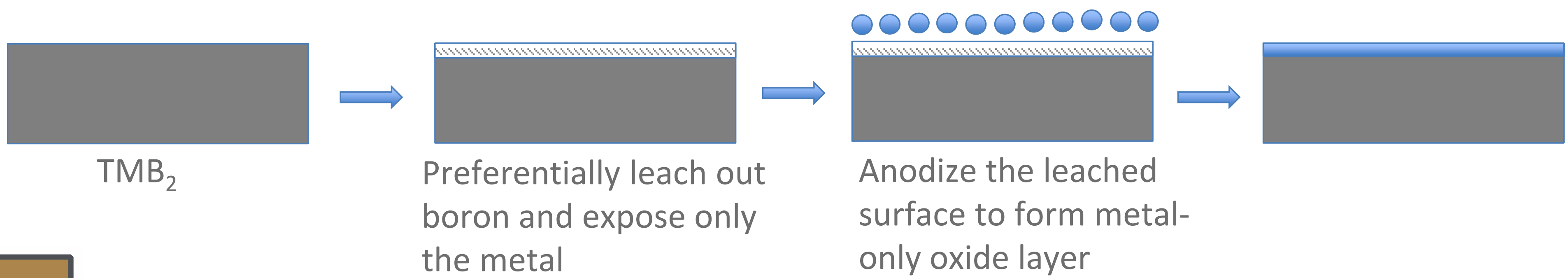
ANODIZATION

1. Single step

   Improve the oxidation resistance by creating a thin impervious oxide layer.

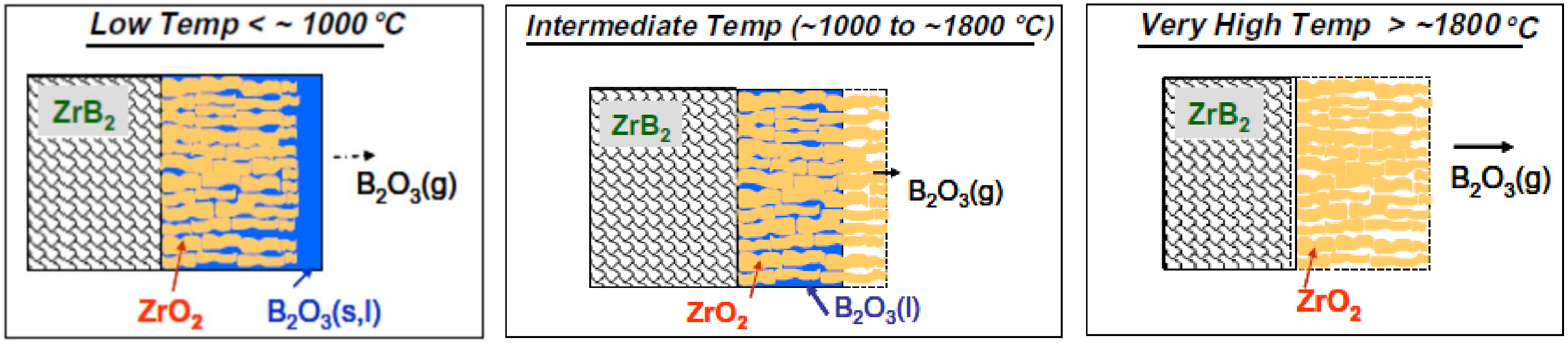
2. Two-step

   Create metal-only impervious oxide layer



ADDITIVES

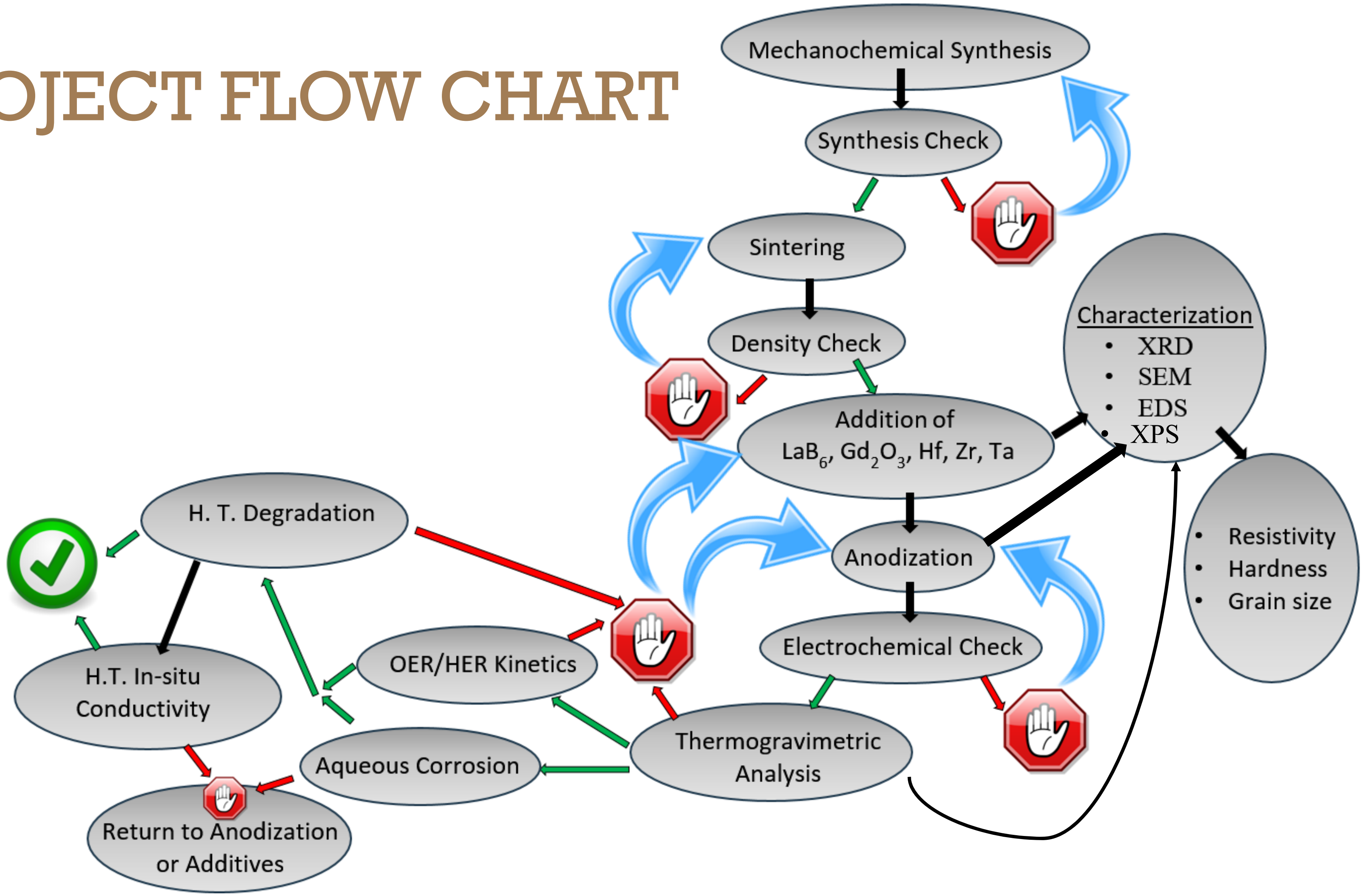
1. Rare earth additives: Gd_2O_3 and LaB_6
   a) Improve the oxidation resistance by forming cubic pyrochlore phases
   b) Also through pinning dislocations and limiting diffusion.
2. Metal additives: Hf, Zr, and Ta
   a) Added to bring Boron to Metal ratio down to 1.86.



$$B_2O_3\ (l) \rightarrow B_2O_3\ (g)\ \ (> 1100\ ^{o}C)$$

T.A. Parthasarathy et al., Acta Materialia, 55 (2007) 5999.

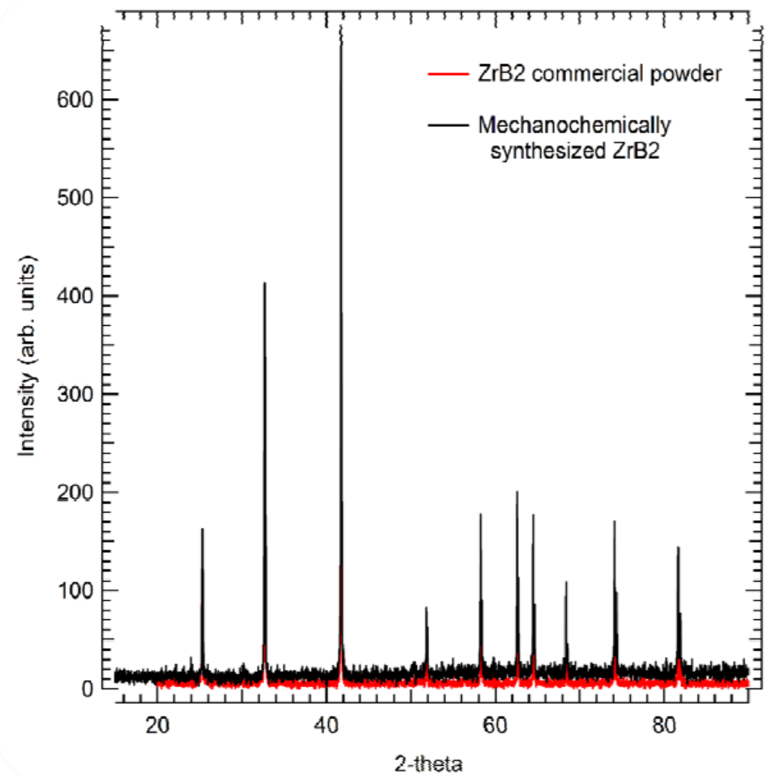
PROJECT FLOW CHART



RESULTS & DISCUSSION

ELEMENTAL vs. COMMERCIAL

- Commercially available HfB_2 and ZrB_2
- Elemental mechanochemical synthesis
  - $Zr + 2B \rightarrow ZrB_2$ $\Delta G_{(rxn,\ 298K)} = -318$ kJ/mol
  - $Hf + 2B \rightarrow HfB_2$ $\Delta G_{(rxn,\ 298K)} = -325$ kJ/mol
- XRD results show no difference.

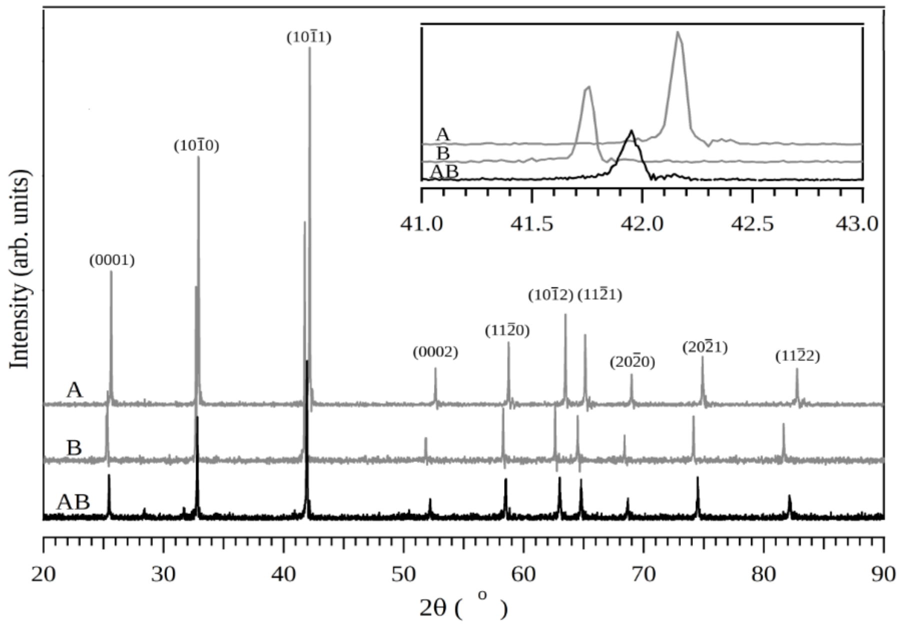


MECHANOCHEMICAL SYNTHESIS

- Achieved through ball milling
  - SPEX 8000M Mixer/mill
- Steel milling media caused contamination
- Parameters used:
  - 6.5 mm diameter, 3 mol% yttria stabilized zirconia grinding media and vial were used
  - 1:10 powder to ball ratio
  - 3 h milling time

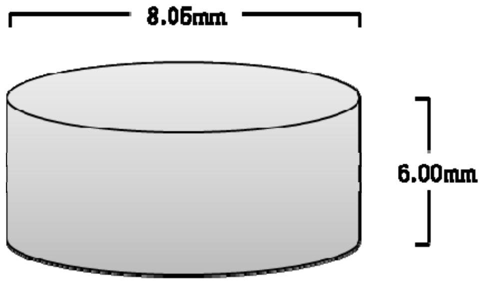
SYNTHESIS VERIFICATION

- X-Ray Diffraction was used on ZrB_2, HfB_2, and 1:1 HfB_2:ZrB_2
- If complete solid solutionizing occurred:
  - Only single peaks for 1:1 HfB_2:ZrB_2 (no split peaks)
  - Peaks for 1:1 HfB_2:ZrB_2 should fall between HfB_2 and ZrB_2
- Inset shows an example of what each peak set looked like proving complete solid solutionizing.

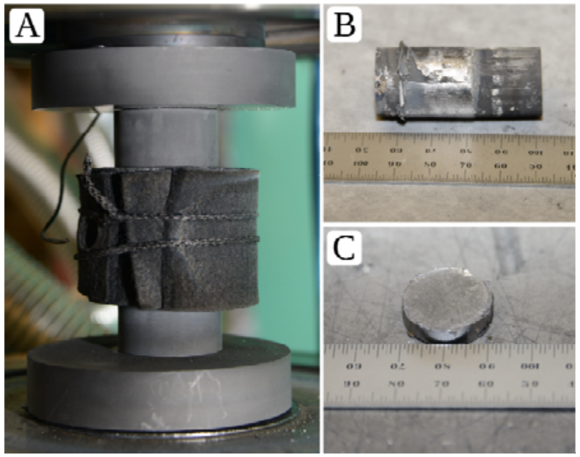


TWO TYPES OF SINTERING

- Conventional Sintering
  - Compacted pellets at ~1.0 kN
  - Sintered in argon at 1700 ºC



- Spark Plasma Sintering:
  - 5 kN force
  - 10^{-3} torr vacuum
  - 1700 ºC



SINTERED PELLET RESULTS

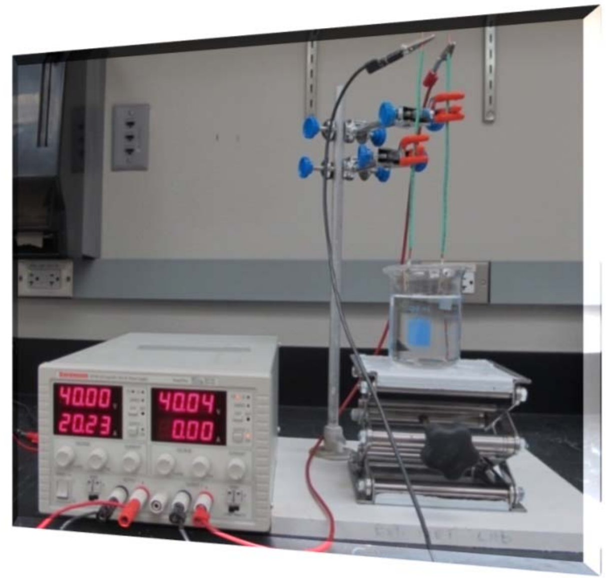
Sample Identifier	Composition	SPS Temp. (K)	Hold Time (s)	Relative Density (%)	Electrical Resistivity (μΩ-cm)
A	HfB_2	1972	180	91.4	9.2
B	ZrB_2	1972	600	87.2	6.79
AB	1:1 HfB_2 - ZrB_2	1972	600	76.2	5.4
A4B	1:4 HfB_2 - ZrB_2	1972	600	82.9	6.0
4AB	4:1 HfB_2 - ZrB_2	1972	600	76.5	9.3
ABT	1:1 HfB_2 - ZrB_2 + Ta (B/Me = 1.86)	1972	180	94.6	17.3
ABZ	1:1 HfB_2 - ZrB_2 + Zr (B/Me = 1.86)	1972	180	94.8	8.4
ABH	1:1 HfB_2 - ZrB_2 + Hf (B/Me = 1.86)	1972	180	96.2	11.6
ABL	1:1 HfB_2 - ZrB_2 + 1.8 mol % LaB_6	1972	300	97.2	12.2
ABG	1:1 HfB_2 - ZrB_2 + 1.8 mol % Gd_2O_3	1972	300	89.5	12.4

ANODIZATION OF BORIDES

- Want to create an impervious oxide layer to increase high temperature oxidation resistance.
- Maintaining electrical conductivity during this step is important.

ANODIZATION

- Initial anodization done on hafnium diboride sample to create impervious oxide layer.
    - Ethylene Glycol
        - 3 – 10% H_2O
        - 0.01 – 0.14 M NH_4F
    - Potassium Hydroxide
        - 0.1 M KOH
    - Potential: 3 – 60 V
    - Time: 5 – 120 minutes
    - Counter electrode: Pt foil



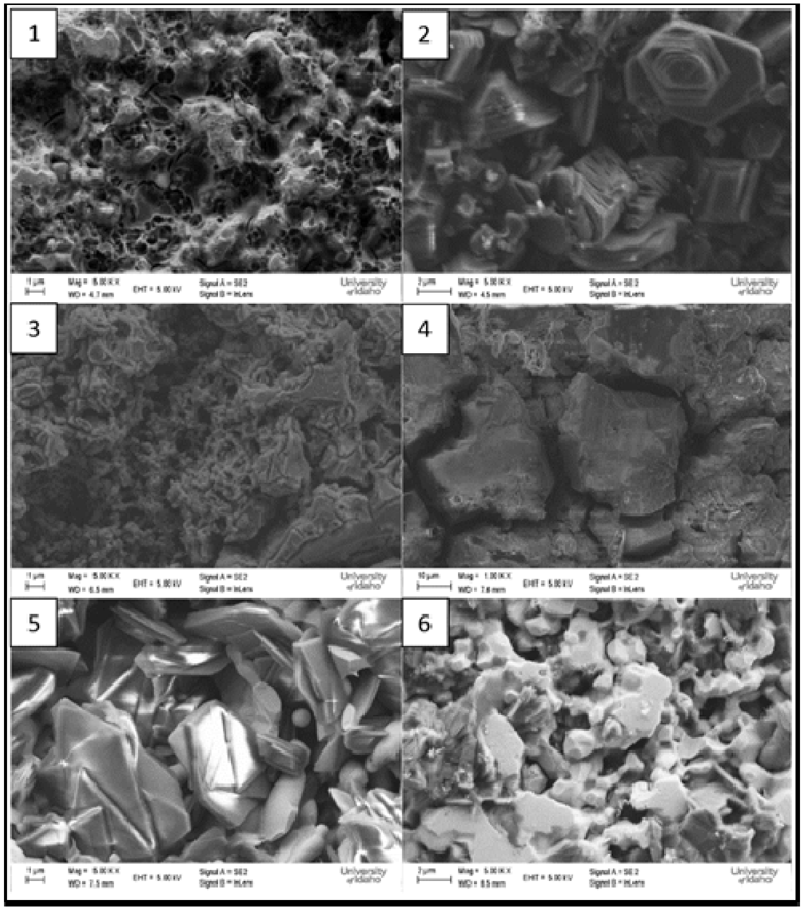
$$(TM) \rightarrow (TM)^{4+} + 4e^-$$

$$(TM)^{4+} + 4OH^- \rightarrow (TM)O_2 + 2H_2O$$

$$(TM)B_2 + 8OH^- \rightarrow (TM)O_2 + 2H_3BO_3(aq) + 2H^+ + 6e^-$$

HfB2 ANODIZATION TESTS

Hafnium Diboride					
Ethylene Glycol			0.1 M Potassium Hydroxide		
Sample	Voltage	Time	Sample	Voltage	Time
AnA1	20 V	30 min	AnA7	5 V	30 min
AnA2	30 V	30 min	AnA8	3 V	30 min
AnA3	20 V	60 min	AnA9	20 V	90 min
AnA4	20 V	120 min	AnA10	10 V	60 min
AnA5	5 V	120 min	AnA0	0 V	0 min
AnA6	10 V	60 min			


1
2
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4
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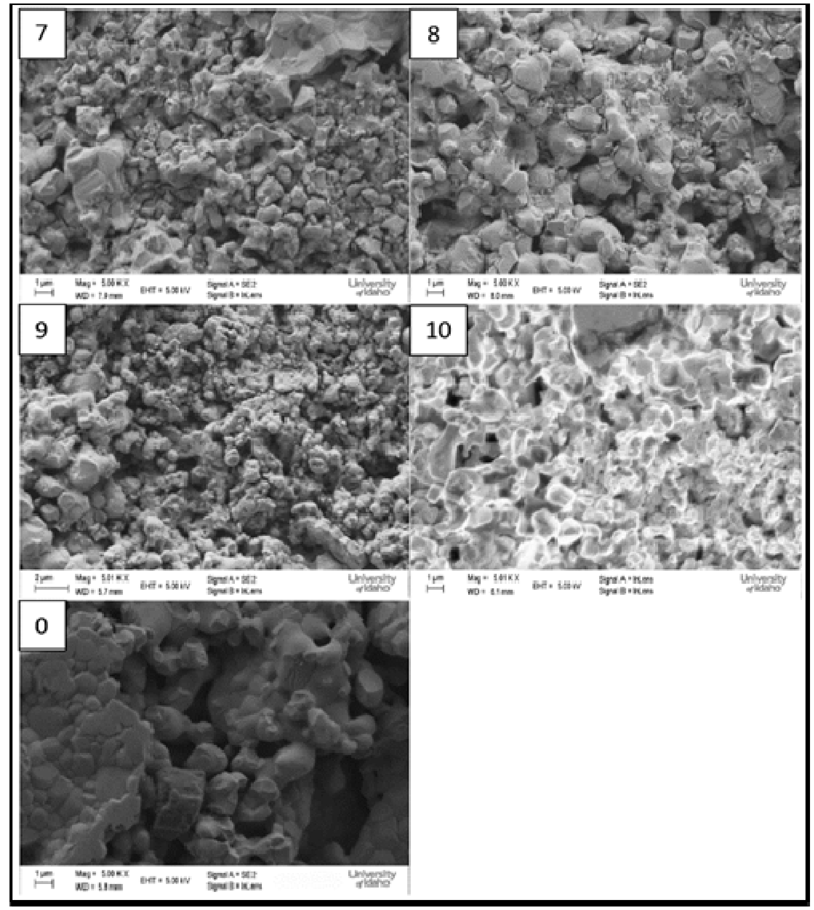
6


7
8
9
10
0

PRELIMINARY OPTIMIZATION

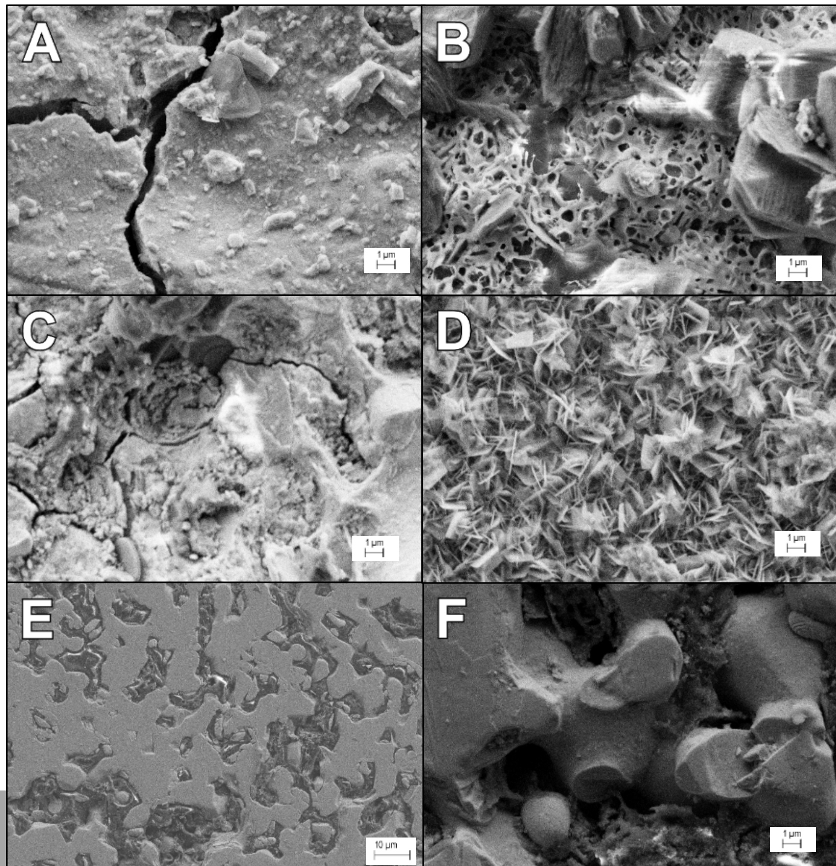
- Testing based on ABL sample
  - Highest density and oxidation resistance performance
  - Contains both Hf and Zr vs. just Hf
- Two-step anodization
  - Designed to leach boron from surface
  - Creating solid metal oxide vs. porous metal oxide

Electrolyte	Potential (V)	Time (m)	Electrolyte	Potential (V)	Time (m)
KOH	20	30	High fluoride	5	60
KOH	40	30	High fluoride	6	60
KOH	60	30	High fluoride	7	60
KOH	40	120	High fluoride	8	60
KOH	60	120	High fluoride	9	60
KOH	60	1440	High fluoride	10	30
High fluoride & phosphate	30	30	High fluoride	10	120
High fluoride & phosphate	60	60	High fluoride	11	30
High fluoride	20	30	High fluoride	12	30
High fluoride	30	30	High fluoride	13	30
High fluoride	40	30	High fluoride	14	30
High fluoride	60	30	High fluoride	15	30

PRELIMINARY 2-STEP TESTS

1st Electrolyte	Potential (V)	Time (m)	2nd Electrolyte	Potential (V)	Time (m)
Phosphoric & fluoride	0	5	Low fluoride & phosphate	40	30
Phosphoric & fluoride	0	5	Low fluoride & phosphate	60	30
High fluoride & phosphate	20	30	Low fluoride & phosphate	20	30
High fluoride & phosphate	20	30	Low fluoride & phosphate	30	30
High fluoride & phosphate	60	60	Low fluoride & phosphate	60	60

PRELIMINARY ANODIZATION TESTS



(A) High fluoride electrolyte at 60 V for 30 minutes

(B) High fluoride & phosphorus at 20 V for 30 minutes

(C) High fluoride at 20 V for 30 minutes then low fluoride at 30 V for 30 minutes

(D) High fluoride & phosphate at 30 V for 30 minutes.

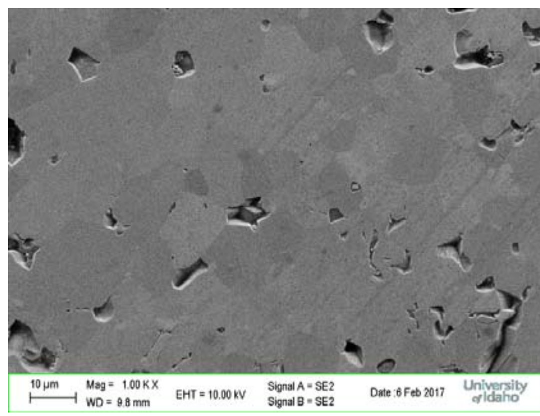
(E) Potassium hydroxide at 60 V for 30 minutes

(F) Phosphoric & fluoride at 0 V for 5 minutes then low fluoride at 60 V for 30 minutes.

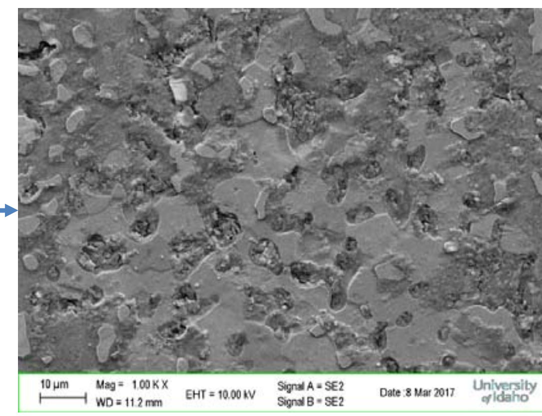
FINAL OPTIMIZATION

- Utilized sample AB (1:1 HfB_2 - ZrB_2)
- Varied water content of ethylene glycol based solutions
  - 3-10%
- Best anodization results
  - Ethylene glycol
  - 0.14 M NH_4F
  - $6\%_{wt}$ H_2O
  - 12.5 V for 60 minutes

Freshly Polished



Anodized



RT AQUEOUS CORROSION

- Wanted to rank the compositions for testing in hot corrosion environments.
- Resistance to room temperature, potential controlled, aqueous corrosion can be translated into high temperature corrosion resistance in the presence of relevant species (chlorides, sulfates, etc.).
- Recently submitted a journal article to *Electrochimica Acta*.

AQUEOUS CORROSION

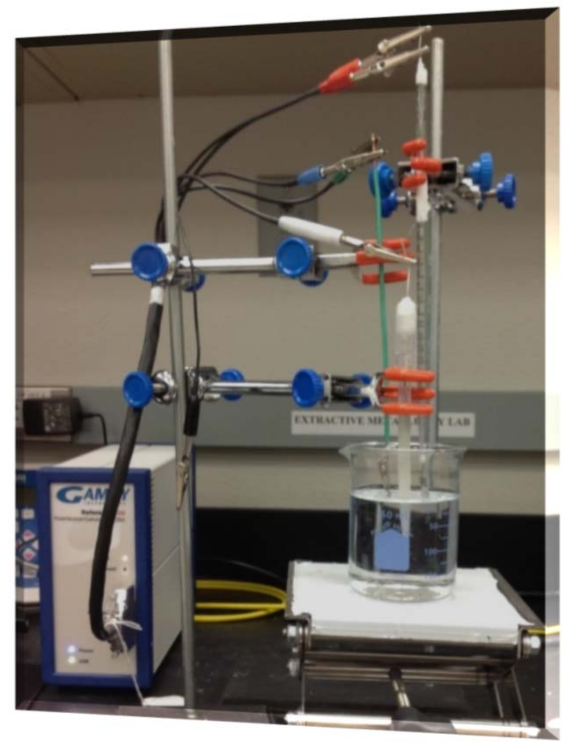
Potentiodynamic polarization with three-electrode configuration.

- 0.1 M H_2SO_4
- 0.1 M NaCl
- 0.1 M NaOH
- 0.1 M NaOH + 0.1 M NaCl

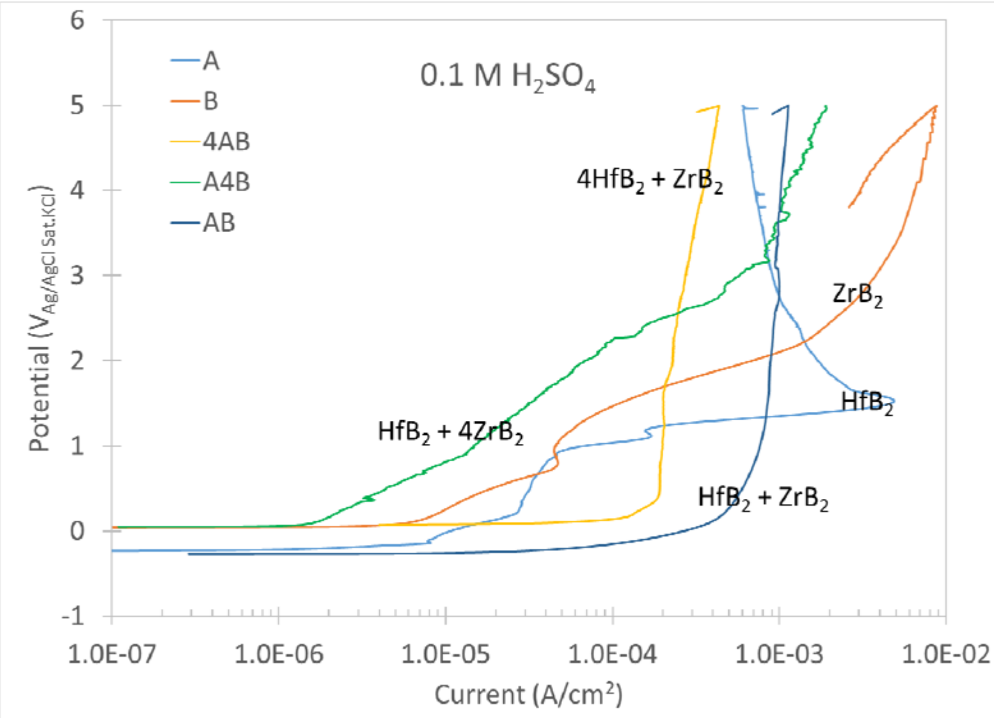
Reference electrode: Ag/AgCl in saturated KCl

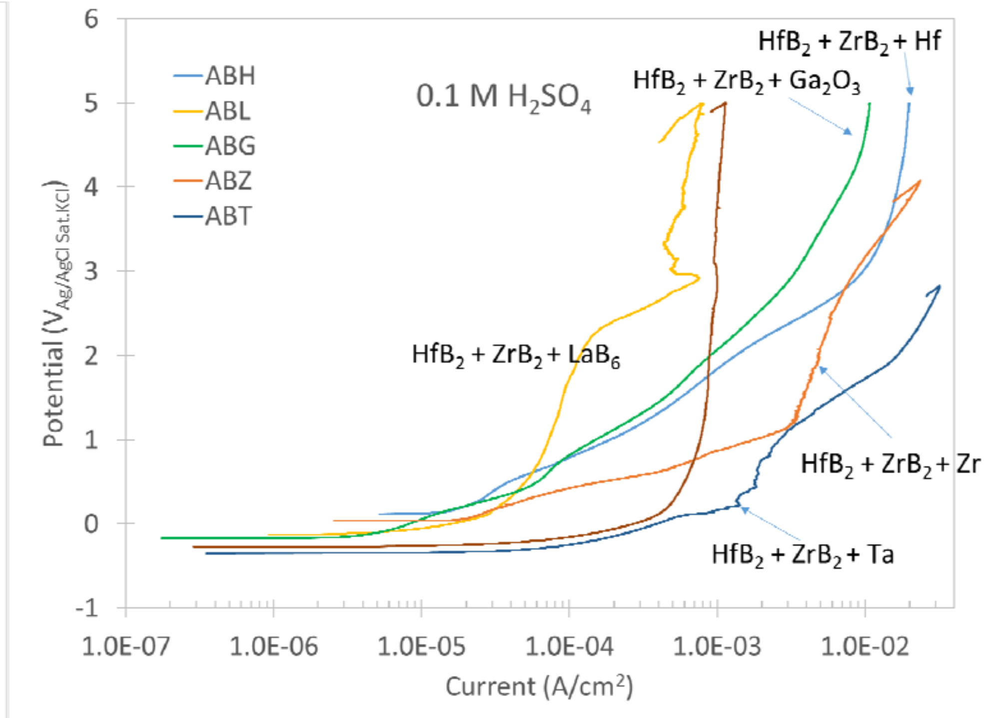
Counter electrode: Platinum foil

- Sample ABL stood out as consistently doing well.



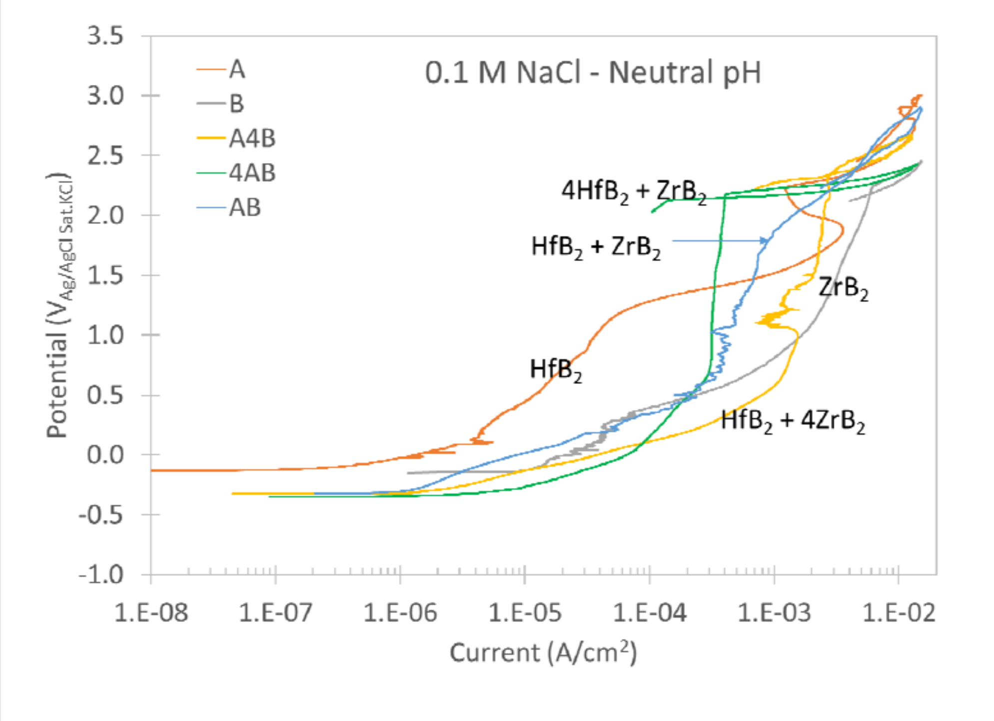
AQUEOUS CORROSION - 0.1 M H_2SO_4

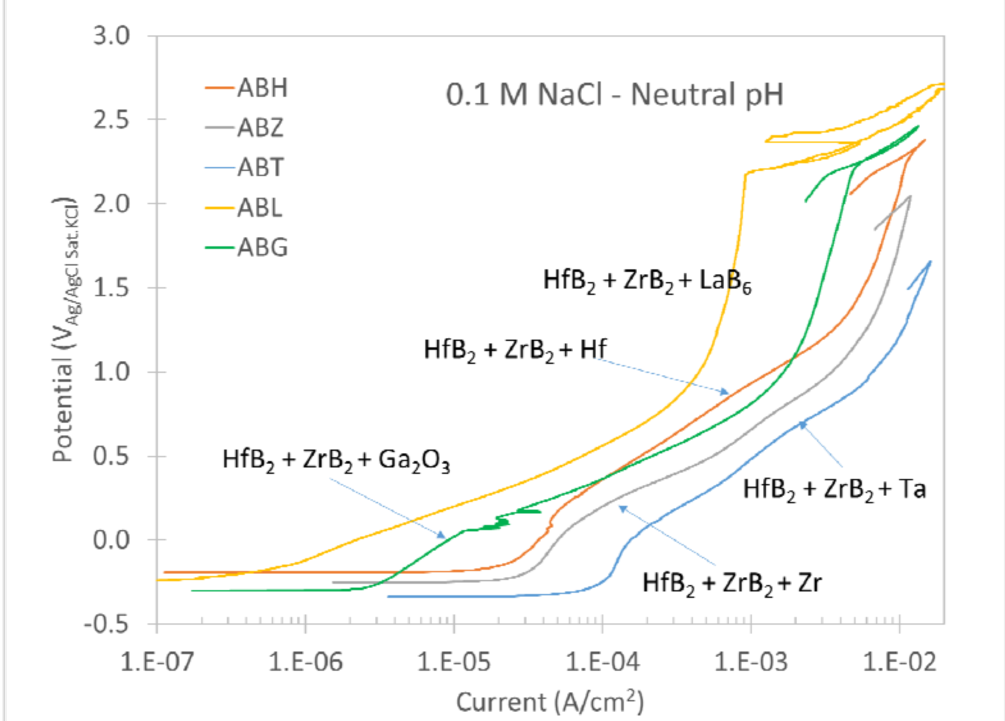




$(TM)B_2 + 5H_2O \rightarrow (TM)O_2 + B_2O_3 + 10H^+ + 10e^-$ $(TM)O_2 + H_2O \rightarrow H(TM)O_3^- + H^+$ $La \rightarrow La^{3+} + 3e^-$

AQUEOUS CORROSION - 0.1 M SODIUM CHLORIDE





$B + 5OH^- \rightarrow HBO_3^{2-} + 2H_2O + 3e^-$

$B + 3H_2O \rightarrow H_3BO_3 + 3H^+ + 3e^-$

$(TM)B_2 + 5H_2O \rightarrow (TM)O_2 + B_2O_3 + 10H^+ + 10e^-$

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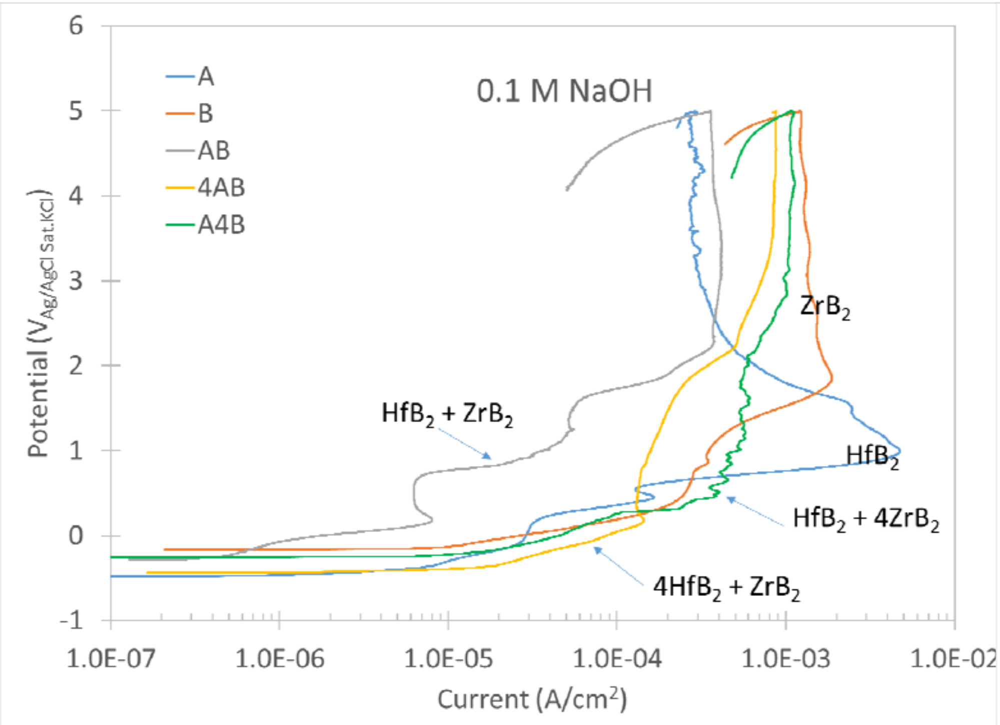
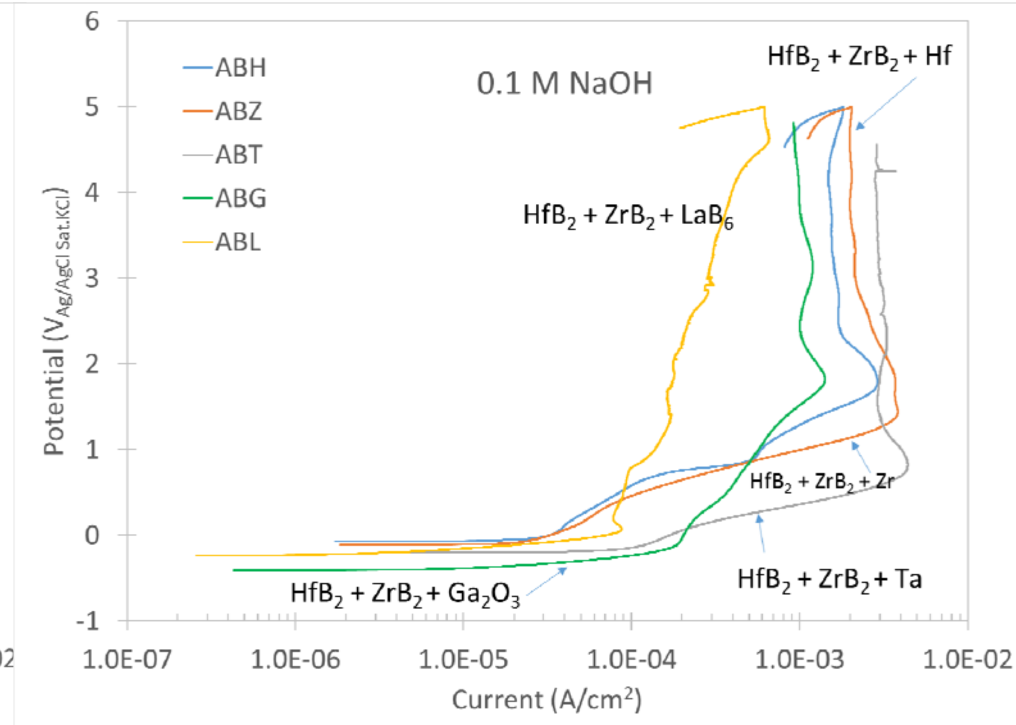
AQUEOUS CORROSION – 0.1 M SODIUM HYDROXIDE



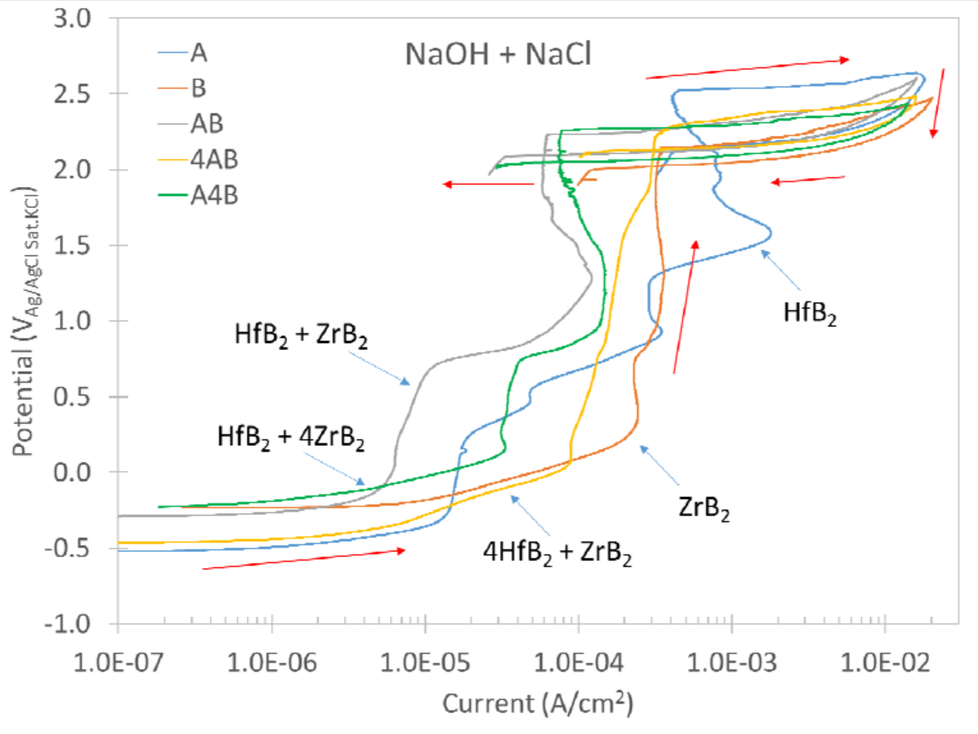


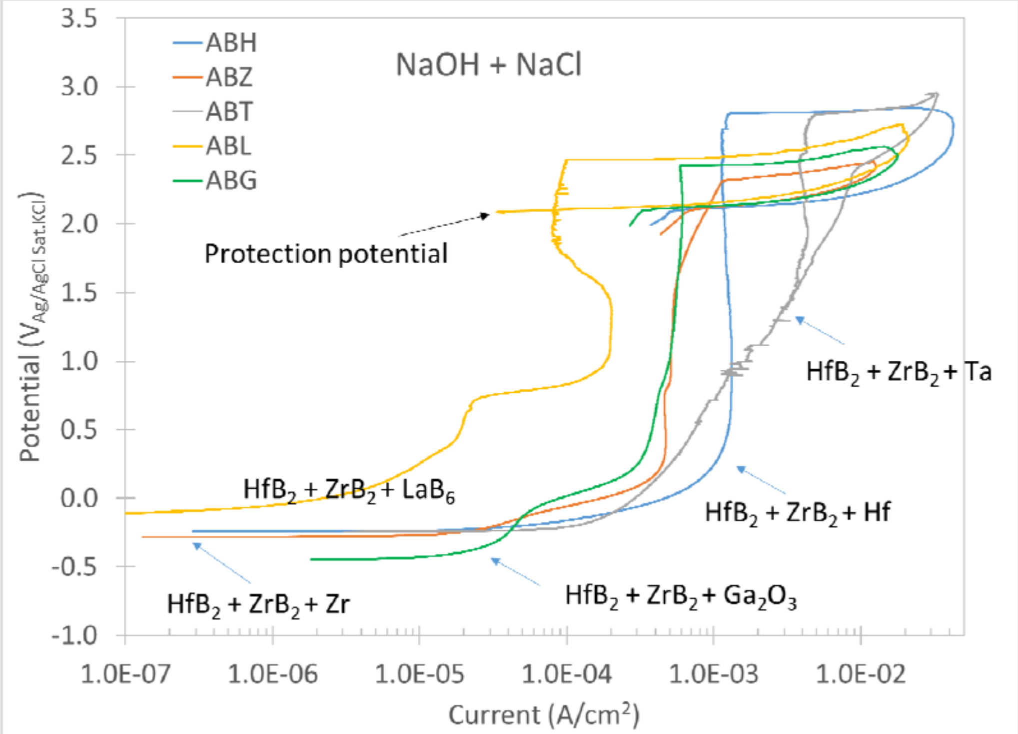
$$(TM)B_2 + 8OH^- \rightarrow (TM)O_2 + 2H_3BO_{3(aq)} + 2H^+ + 6e^-$$

$$La^{3+} + 3OH^- \rightarrow La(OH)_3$$

$$2La(OH)_3 \rightarrow La_2O_3 \bullet 3H_2O$$

AQUEOUS CORROSION – CHLORIDE IN BASIC SOLUTION





HER KINETICS STUDY

- To help rank the compositions for testing in hot corrosion environments and to understand the hydrogen evolution kinetics to get a better picture of the broader impact this material may have.
- Two journal articles have been published on this topic
- Broken into two categories:
  - Samples, A, B, AB, 4AB, and A4B
  - Samples AB, ABH, ABZ, ABT, ABL, and ABG
- Done in 2 different electrolytes, 1 M H_2SO_4 and 1 M NaOH, using electrochemical characterization (EIS, PS, PD, CV)

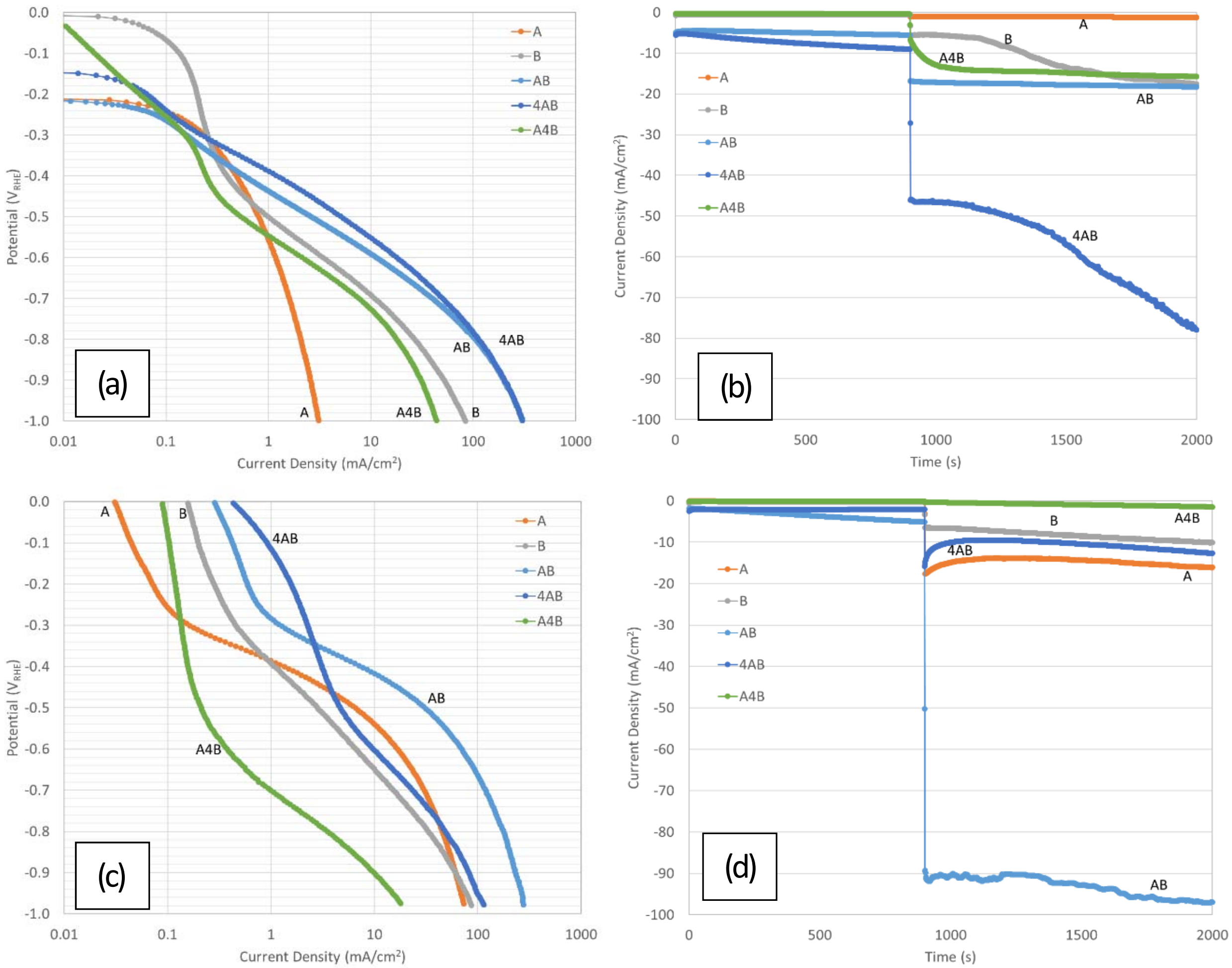


Fig 2. Electrochemical hydrogen evolution characteristics of HfB_2-$ZrfB_2$ solid solutions. (a) I-V plots in 1 M H_2SO_4, (b) I-t plots in 1 M H_2SO_4 (c) I-V plots in 1 M NaOH, and (d) I-t plots in 1 M NaOH.

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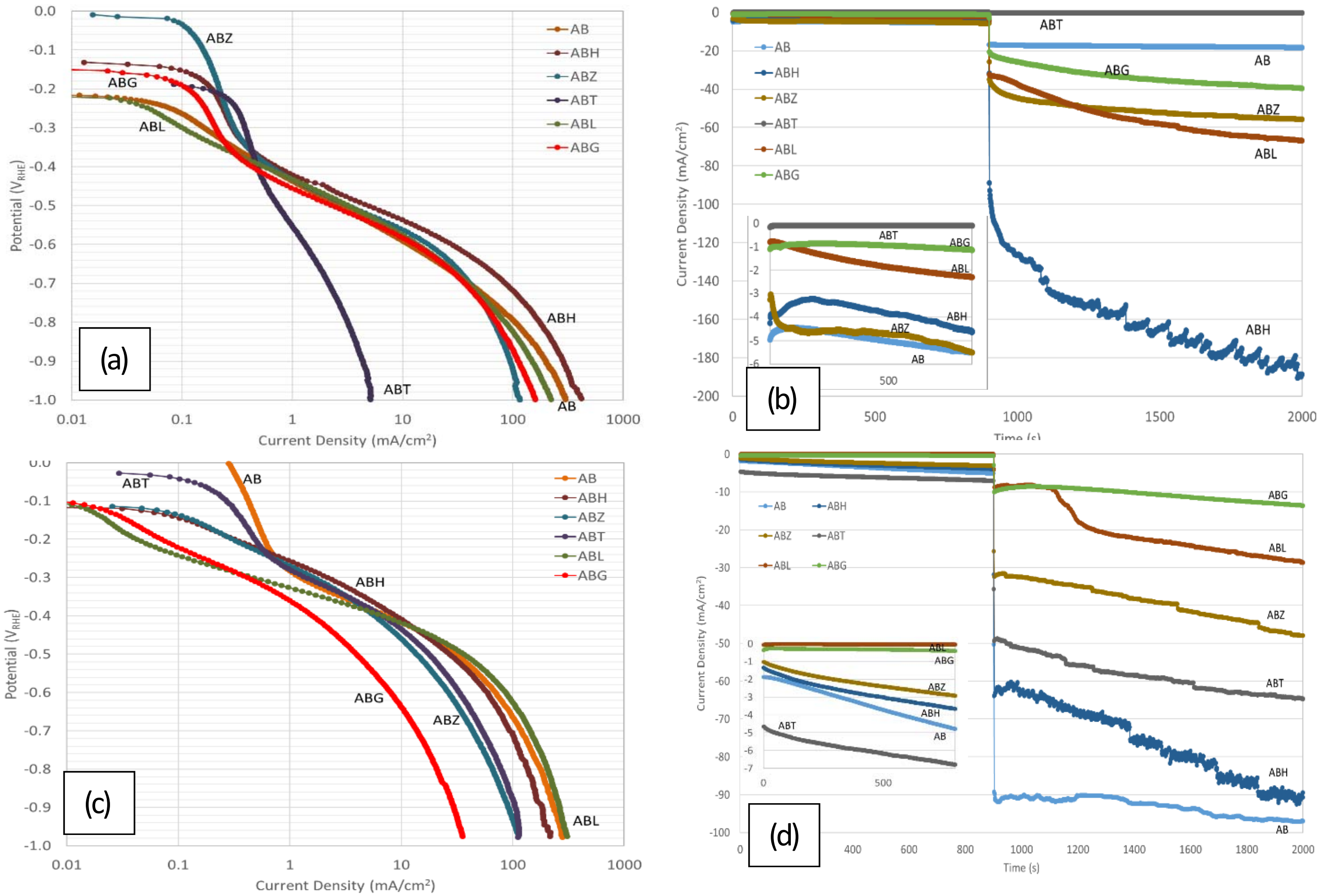

Electrochemical hydrogen evolution characteristics of HfB_2-$ZrfB_2$ solid solutions with additives, identifiers are shown in table 1. (a) I-V plots in 1 M H_2SO_4, (b) I-t plots in 1 M H_2SO_4 at -0.4 and -0.6 V_{RHE} (c) I-V plots in 1 M NaOH, and (d) I-t plots in 1 M NaOH at -0.2 and -0.5 V_{RHE}.

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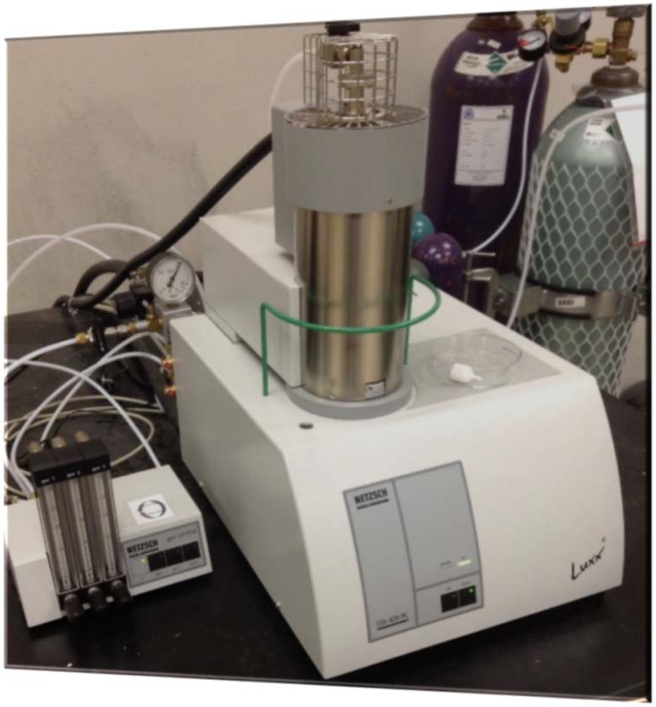
HT OXIDATION

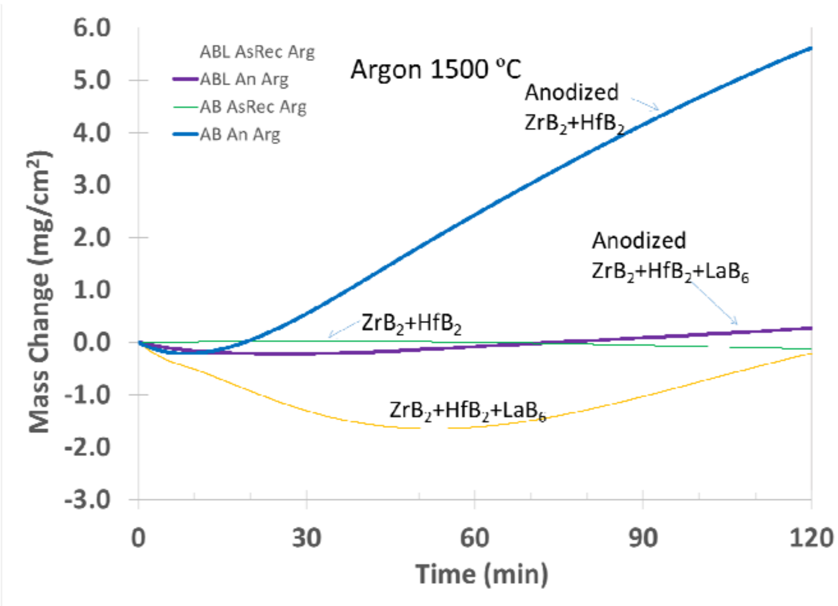
- We have tested samples AB and ABL for oxidation resistance at high temperature under three different oxygen partial pressures.
- We monitored the mass change as a function of time which gave important details on the oxidation kinetics.
- The total gain in mass showed that the additives and anodization process aided in oxidation mitigation

THERMOGRAVIMETRIC ANALYSIS

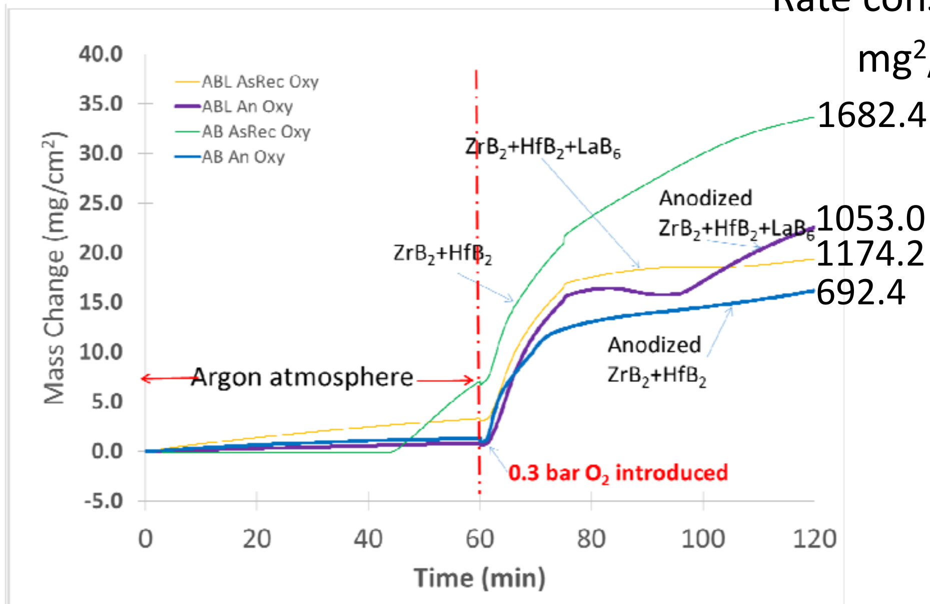
- Netzsch STA 409 PC
  - 3 ºC/min ramp rate
  - 1500 ºC for 2 hours
    - Argon with O_2 getter: pO_2 ~ 0.001 Pa
    - Argon: pO_2 = 0.1 Pa
    - Oxygen: pO_2 = 0.3 x 10^5 Pa



TGA Results: Argon (pO_2 = 0.1 Pa) and Oxygen (0.3 bar O_2)



Rate constants (k_p), $mg^2/cm^4\,h$



$$(TM)B_{2(s)} + {}^{5}/_{2}O_{2(g)} \rightarrow (TM)O_{2(s)} + B_2O_{3(l)}$$

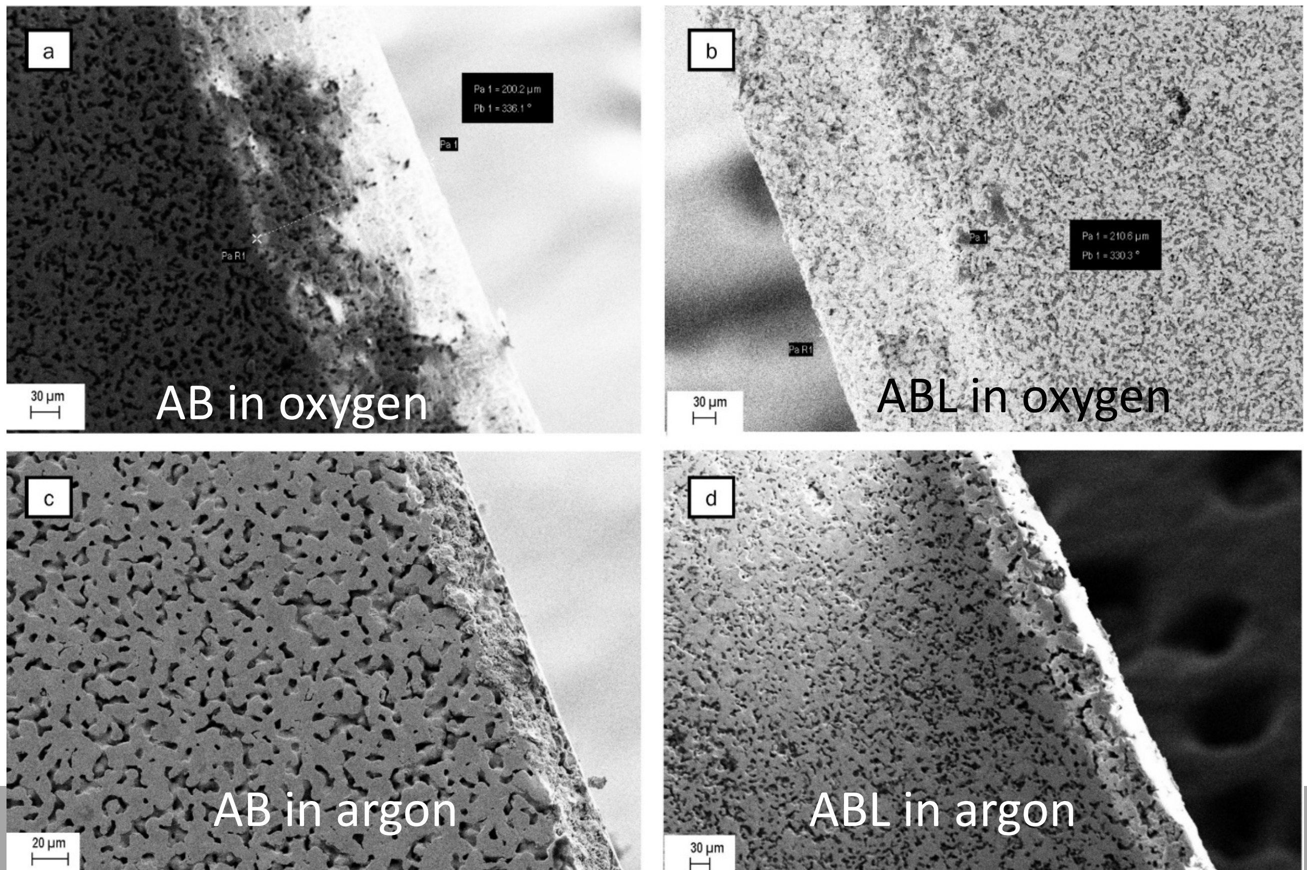
$$B_2O_{3(l)} \rightarrow B_2O_{3(g)} \quad (T>1100\ °C)$$

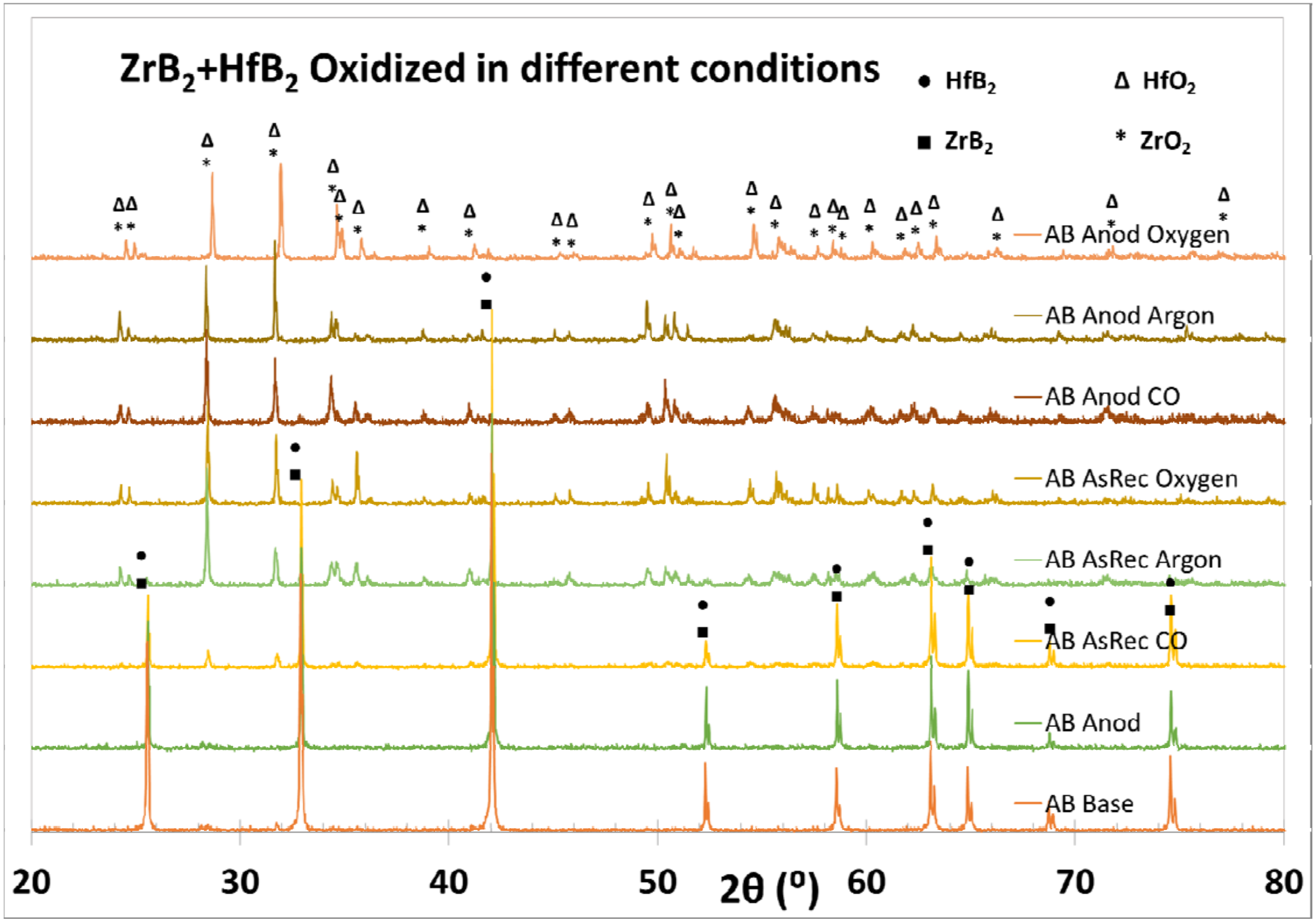
$$LaB_6(s) + {}^{21}/_{4}O_2(g) \rightarrow {}^{1}/_{2}La_2O_3(s) + 3B_2O_3(l)$$

$$La_2O_3(s) + (TM)O_2(s) \rightarrow La_2(TM)_2O_7(s)$$

S.J. Sitler, C. Hill, K.S. Raja, I. Charit, Transition Metal Diborides as Electrode Material for MHD Direct Power Extraction: High Temperature Oxidation of ZrB$_2$-HfB$_2$ Solid Solution with LaB$_6$ Addition, *Metall. Mater. Trans. E*, **6**, 11991-12002 (2016). DOI: 10.1007/s40553-016-0072-2

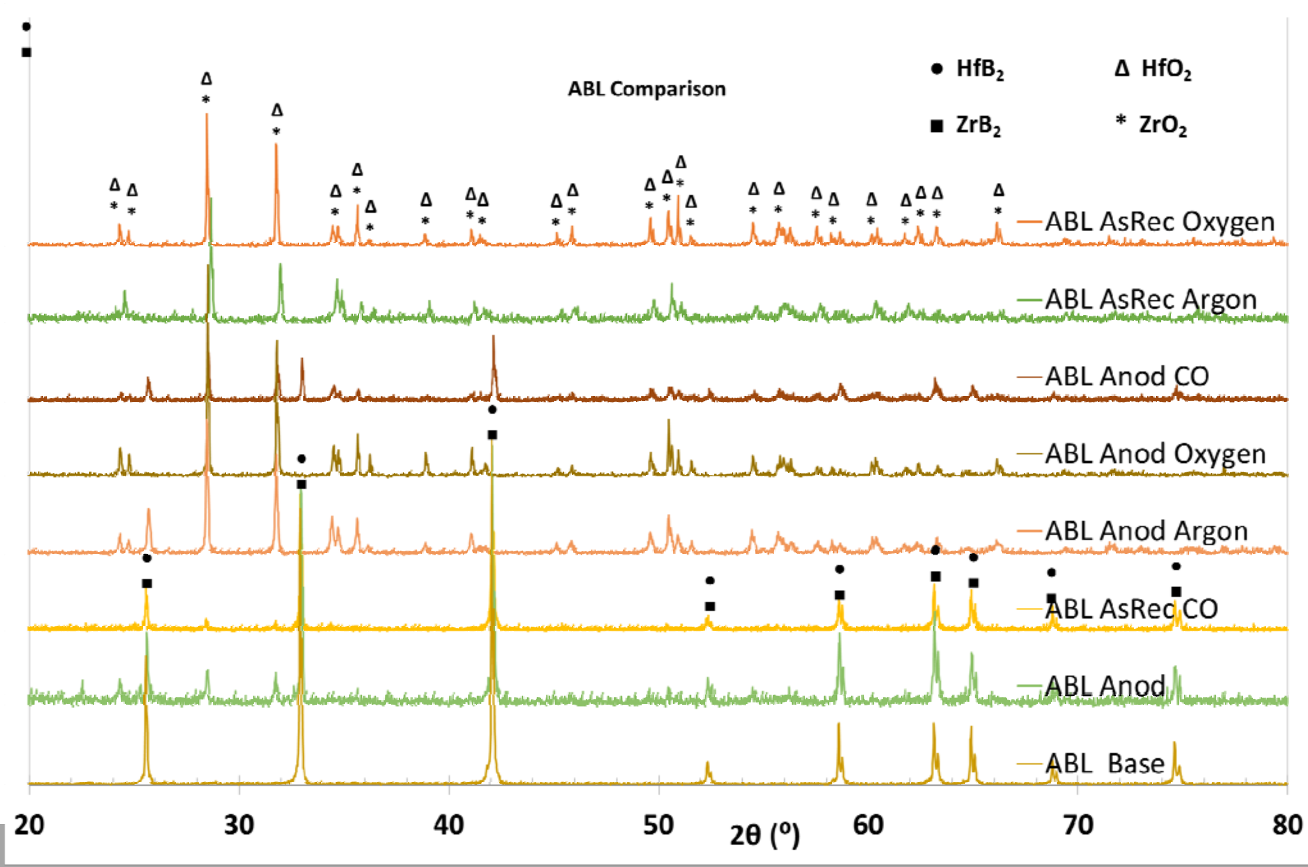
SEM OF OXIDES OF AS-SINTERED SPECIMENS





ZrB₂+HfB₂ Oxidized in different conditions
HfB₂
HfO₂
ZrB₂
ZrO₂
AB Anod Oxygen
AB Anod Argon
AB Anod CO
AB AsRec Oxygen
AB AsRec Argon
AB AsRec CO
AB Anod
AB Base
20
30
40
50
2θ (°)
60
70
80

LaB_6 containing ZrB_2+HfB_2 Oxidized in different conditions

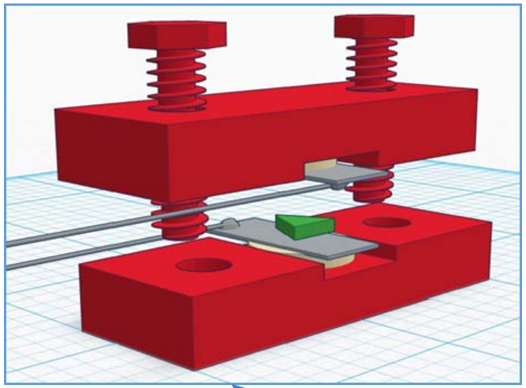


HT CORROSION:

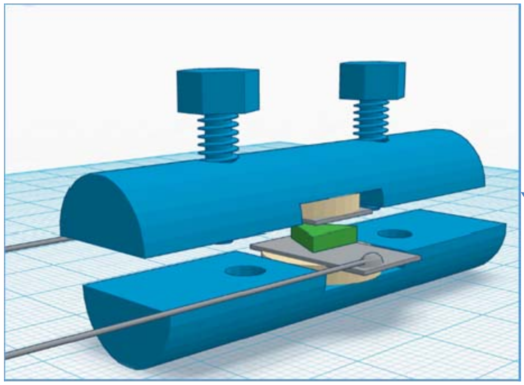
- Want to understand high temperature corrosion in the presence of potassium salts.
- Also want to measure high temperature in-situ electrical conductivity.
- Will be used to make final determination on viability of borides for electrode coating

PRELIMINARY WORK

Preliminary testing for conductivity has been done up to 1500 °C using Zircaloy-4 on AB and up to 1100 °C using the Kanthal clamp on ABL.



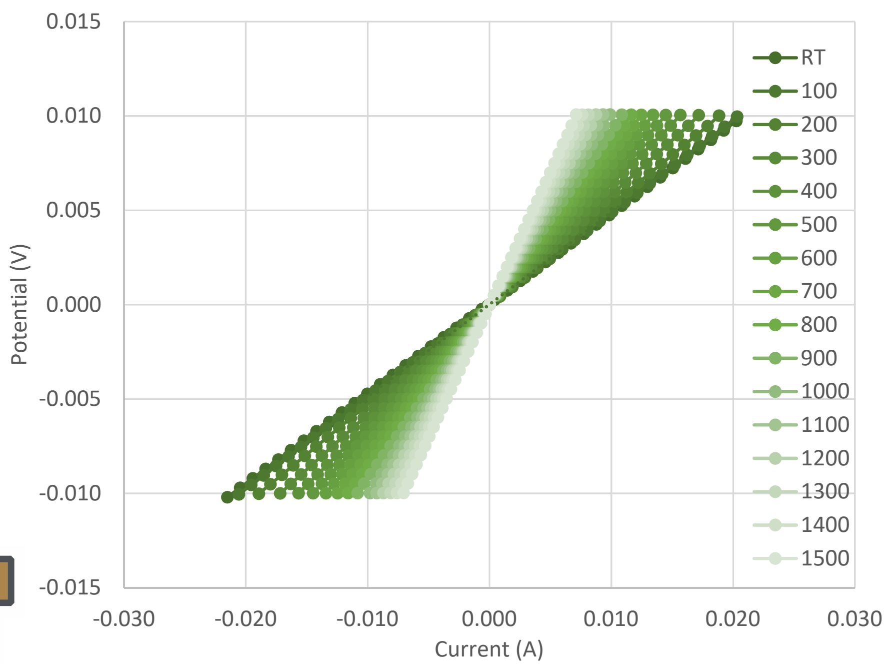
Kanthal (MP ~1500 °C)

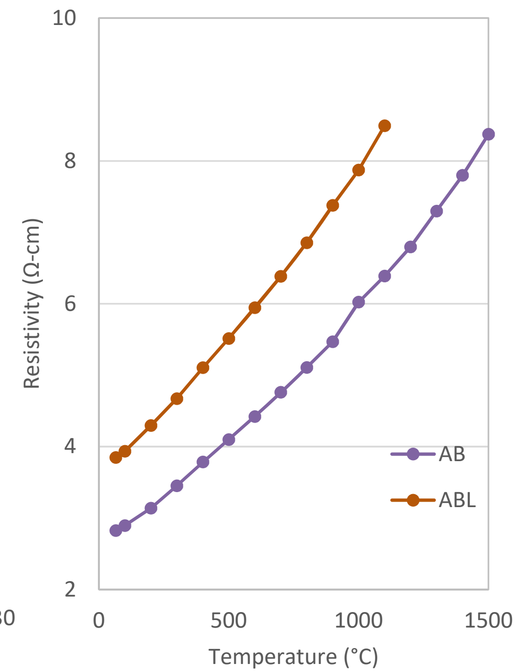


Zircaloy-4 (MP ~1850 °C)

High temperature clamp designs for testing electrical conductivity.

HT CONDUCTIVITY





Temperature (°C)	Resistivity (Ω-cm)	
	AB	ABL
65	2.82	3.85
100	2.89	3.93
200	3.14	4.30
300	3.45	4.67
400	3.79	5.10
500	4.10	5.51
600	4.42	5.95
700	4.76	6.38
800	5.11	6.85
900	5.47	7.38
1000	6.02	7.87
1100	6.39	8.49
1200	6.80	
1300	7.30	
1400	7.80	
1500	8.37	

ACCOMPLISHMENTS

MILESTONES

- Y1Q4: Publication of journal article "Transition Metal Diborides as Electrode Material for MHD Direct Power Extraction: High Temperature Oxidation of ZrB_2-HfB_2 Solid Solution with LaB_6 Addition"
- Y2Q1: Mechanochemical Synthesis: Optimized parameters for densification of boride powders
- Y2Q2: Spark Plasma Sintering: Achieved sample compaction with densities >95% theoretical full density goal.
- Y2Q3: Anodization Process: Unoptimized anodization still provided a clear increase in oxidation resistance at high temperatures.
- Y2Q4: High Temperature Oxidation: Enough tests were completed to successfully prove that additives and anodization both aid in oxidation mitigation.
- Y2Q4: M.S. Student Graduation: Completed early with a thesis titled "Processing and Characterization of HfB_2 and ZrB_2 Based Solid Solution Composites for Magnetohydrodynamic (MHD) Power Generation Applications" with a M.S. in Materials Science and Engineering
- Y3Q1: Submission and publication of two journal articles: "Metal-Rich Transition Metal Diborides as Electrocatalysts for Hydrogen Evolution Reactions in a Wide Range of pH" and "ZrB_2-HfB_2 solid solutions as electrode materials for hydrogen reaction in acidic and basic solutions"

PUBLICATIONS

Published Journal Articles

- Sitler, S. J., Hill, C. D., Raja, K. S. & Charit, I. "Transition Metal Diborides as Electrode Material for MHD Direct Power Extraction: High Temperature Oxidation of ZrB_2-HfB_2 Solid Solution with LaB_6 Addition" *Metall. Mater. Trans. E.* **3**, 90–99 (2016). DOI: 10.1007/s40553-016-0072-2
- Sitler, S. J., Raja, K. S., Charit, I. "Metal Rich Transition Metal Diborides as Electrocatalysts for Hydrogen Evolution Reactions in a Wide Range of pH" *J. Electrochem. Soc.* **163**, H1069–H1075 (2016) DOI: 10.1149/2.0201613jes
- Sitler, S. J., Raja, K. S., Charit, I. "ZrB_2-HfB_2 Solid Solutions as Electrode Materials for Hydrogen Reaction in Acidic and Basic Solutions" *Materials Letters.* **188**, 239-243 (2016) DOI: 10.1016/j.matlet.2016.10.122

Journal Articles Under Consideration

- Sitler, S. J., Charit I., Raja, K. S. "Room Temperature Corrosion Behavior of ZrB_2-HfB_2 Solid Solutions in Acidic and Basic Aqueous Environments" Electrochimica Acta. Manuscript number: EO17-1334.
- Hill, C. D., Sitler, S. J., Charit, I. & Raja, K. S. "Processing and Characterization of ZrB_2-HfB_2 Solid Solutions for Magnetohydrodynamic (MHD) Applications" 11th International Conference on Ceramic Materials and Components for Energy and Environmental Applications. Vancouver, BC, Canada.

PRESENTATIONS

- Sitler, S. J., Raja, K. S. & Charit, I. “High Temperature Oxidation Study of Hafnium & Zirconium Diborides: MHD Electrode Coatings” 2016 Materials Science & Technology Conference & Exhibition, Salt Lake City, UT. October 23-27, 2016. *Poster Presentation*
- Sitler, S. J., Raja, K. S. & Charit, I. “Electrochemical Corrosion of Various HfB_2-ZrB_2 Solid Solutions: A Predictive Study” 2016 Materials Science & Technology Conference & Exhibition, Salt Lake City, UT. October 23-27, 2016. *Oral Presentation*
- Sitler, S. J. “Energy Crisis: A HOT New Solution” 2016 3-Minute Thesis (3MT) Competition, University of Idaho, Moscow, ID, April 12, 2016. *Oral Presentation.*
- Sitler, S. J., Hill, C. D., Raja, K. S.& Charit, I., “Processing of Transition Metal Diborides for Advanced Energy Applications” under the symposium “Advanced Powder Processing for Energy Applications” 2015 Materials Science & Technology Conference & Exhibition, Columbus, OH, October 4 – 8, 2015. *Oral Presentation.*
- Hill, C. D., Sitler, S. J., Charit, I. & Raja, K. S., “Processing and Characterization of ZrB_2-HfB_2 Solid Solutions for Magnetohydrodynamic (MHD) Applications” 11th International Conference on Ceramic Materials and Components for Energy and Environmental Applications. Vancouver, BC, Canada. June 14 – 19, 2015. *Poster Presentation.*
- Charit, I., Sitler, S. J., Hill, C. D. & Raja, K. S., “MHD: ZrB_2-HfB_2 Solid Solution Electrode Material for Extreme Conditions.” NETL Crosscutting Research Review Meeting, Pittsburg, PA, April 27 – 30, *2015. Poster Presentation.*

ASSOCIATED STUDENT AWARDS

- Alumni Award for Excellence – 2016 University of Idaho
- Outstanding Student Publication Award – 2016 University of Idaho, College of Engineering
- Nomination for 2016 COGS "Outstanding Graduate Student Award"
- Nomination for 2016 GPSA "Outstanding Graduate Student Award"
- Nomination for 2016 GPSA "Outstanding Student Research & Creative Activity Award"

CONCLUSIONS

- Elemental and commercial powders of both HfB_2 and ZrB_2 are found from XRD analysis to be essentially equivalent.
- The mechanochemical synthesis of HfB_2-ZrB_2 has been confirmed by XRD to produce a solid solution.
- Electrical resistivity measurements show that a 1:1 mixture of HfB_2 and ZrB_2 has better conductivity than either of the individual borides.
- Anodization does aid in reducing oxidation.
- LaB_6 additive leads to highest increase in density and shows an increase in oxidation resistance.

WORK IN PROGRESS

ANODIZATION

- We have already started the high temperature oxidation tests as well as the high temperature electrical conductivity and corrosion tests using this newly optimized anodization in order to compare to the base 1:1 HfB_2-ZrB_2 samples.
- These tests should give us some final results about the viability of the borides for electrode coating materials in the magnetohydrodynamic system.

HT OXIDATION

- TGA experiments are underway using samples with the newly optimized anodization layer.
- The samples will be characterized before and after through electrical conductivity tests, allowing us to see if an impervious oxide was obtained from anodization.
- Cross-sectional SEM will also be utilized for post-test analysis.

HT CORROSION

- Probably the most important testing being performed during this project is the high temperature corrosion testing.
- Current plans are to perform corrosion tests by applying a constant amount of either or both KCl and K_2SO_4 salts to the surface of the boride samples before heating them up to 1700 °C under both air and CO_2 atmospheres. At least three samples are planned in both the anodized and un-anodized conditions.
- Electrical conductivity measurements

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Vandals

THANK YOU!