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Center for Advanced Non-Ferrous Structural Alloys
An Industry/University Cooperative Research Center

Project 37-L: Advanced Engineered Coatings with Extended Die Life for Tooling

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Student: Nelson Delfino de Campos Neto (Mines)

Faculty: Steve Midson, Andy Korenyi-Both, Michael Kaufman (Mines)

Industrial Mentors: Paul Brancaleon (NADCA)

NSF COLORADO SCHOOL OF MINES IOWA STATE UNIVERSITY

Project 7-L: Advanced Engineered Coatings with Extended Die Life for Tooling

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- Student: Nelson Delfino de Campos Neto (Mines)
- Advisor(s): S. Midson; A. Korenyi-Both, M. Kaufman (Mines)

Project Duration
PhD: August 2018 to July 2023

- Problem: Molten aluminum tends to solder to die faces in die casting process and is used a large amount of lubricant in the attempt to reduce the adhesion but reduces the parts quality.
- Objective: Identify PVD coatings to be applied to die casting dies to avoid the molten aluminum soldering and understand the adhesion mechanisms involved.
- Benefit: Increase die casting parts quality, eliminate the use of lubricants, extend the dies life and reduce cost-per-parts.

Recent Progress

- Literature review.
- Development of an improved adhesion test.
- Initial tests using the new adhesion test.
- Initial characterization.

Metrics

Description	% Complete	Status
1. Literature review and development of an improved adhesion test that simulates the die casting process by incorporating a mechanism to pressurize/quick fill the liquid aluminum against the coating.	20%	●
2. Identification of the mechanisms that rules the adhesion behavior.	10%	●
3. Identification of a working layer coating that is free of molten aluminum adhesion by laboratory tests	5%	●
4. Development of a coating architecture (single or multi-layer) that will add sufficient durability to the die coatings to allow them to survive for as long as the die casting die itself (□100,000 shots)	0%	●
5. Guidelines for depositing the coating system on die components/die tooling and in-plant trials.	0%	●

Industrial Relevance

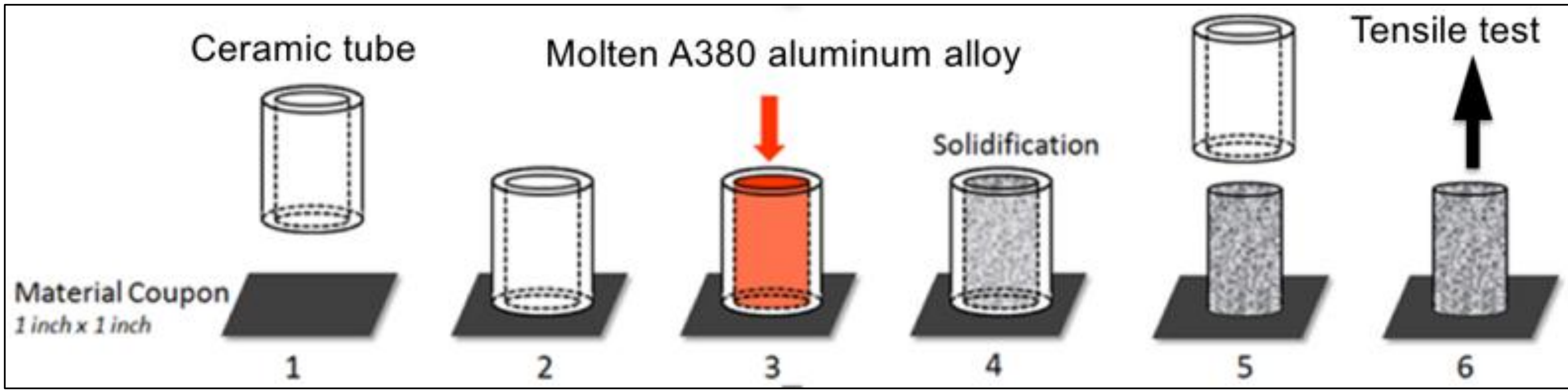
- Reducing or eliminating lubricant spray will:
  - Significantly **improve the quality** of the die castings
    - Reduce gas porosity and scrap
    - Used in higher performance applications
  - **Reduce costs**
    - Eliminate purchase costs for lubricants
    - Reduce effluent clean-up costs
    - Significantly extend die life
  - **Improve productivity**
    - Faster cycle rates

Project Tasks

1. Develop improved adhesion test
2. Define mechanism controlling wetting and adhesion of molten aluminum to coating
3. Develop a coating architecture to provide long life
4. Conduct in-plant trials
5. Create guidelines for depositing the coating on tooling

Previous Adhesion Test

- Previous project developed a laboratory test
  - To measure adhesion of aluminum to coating

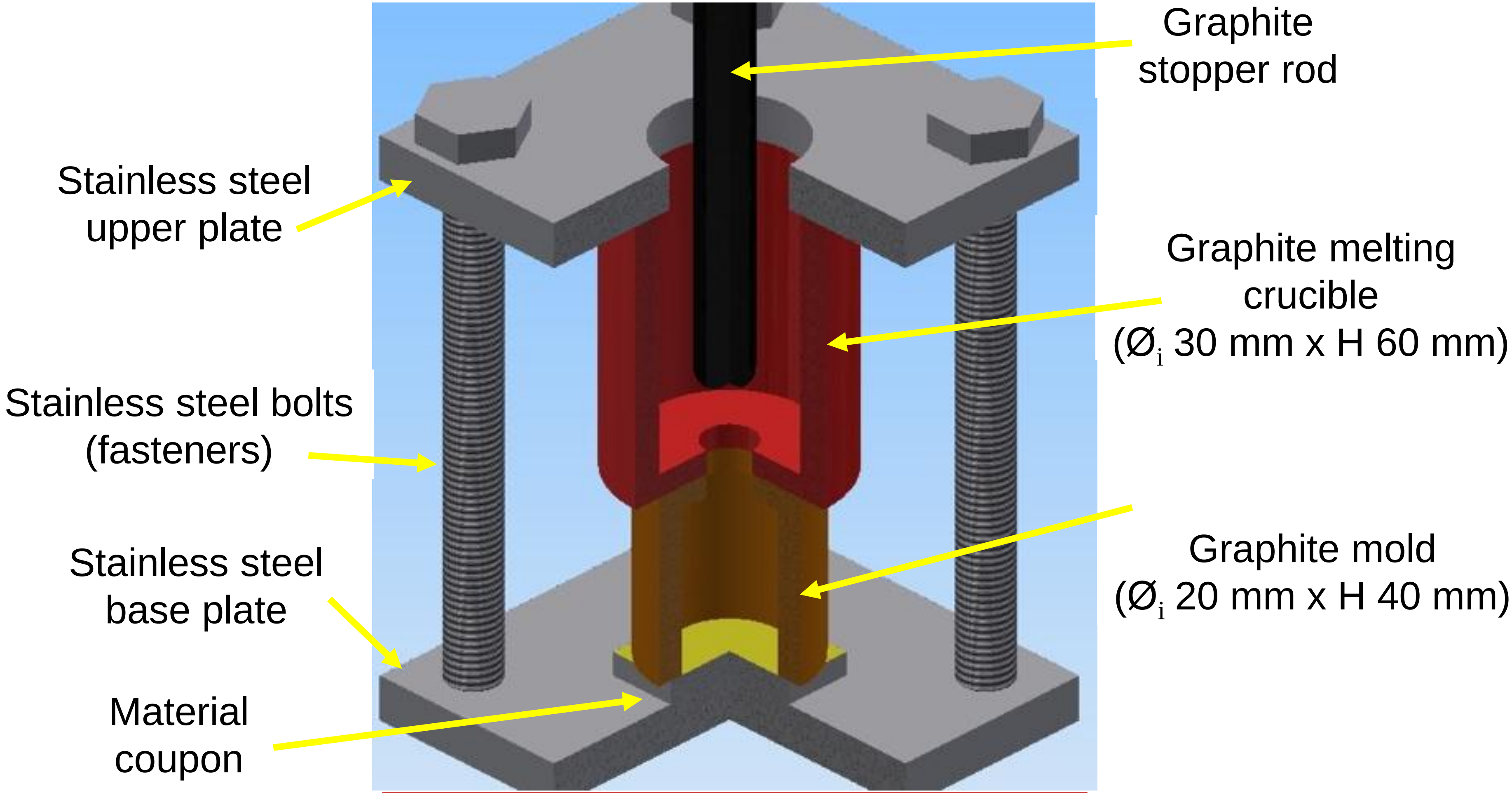


- Drawback:
  - Top pouring can carry the oxide layer from the upper surface of the molten aluminum into the crucible to contact with the material coupon

Source: B. Wang, Ph.D. thesis, Colorado School of Mines, 2016

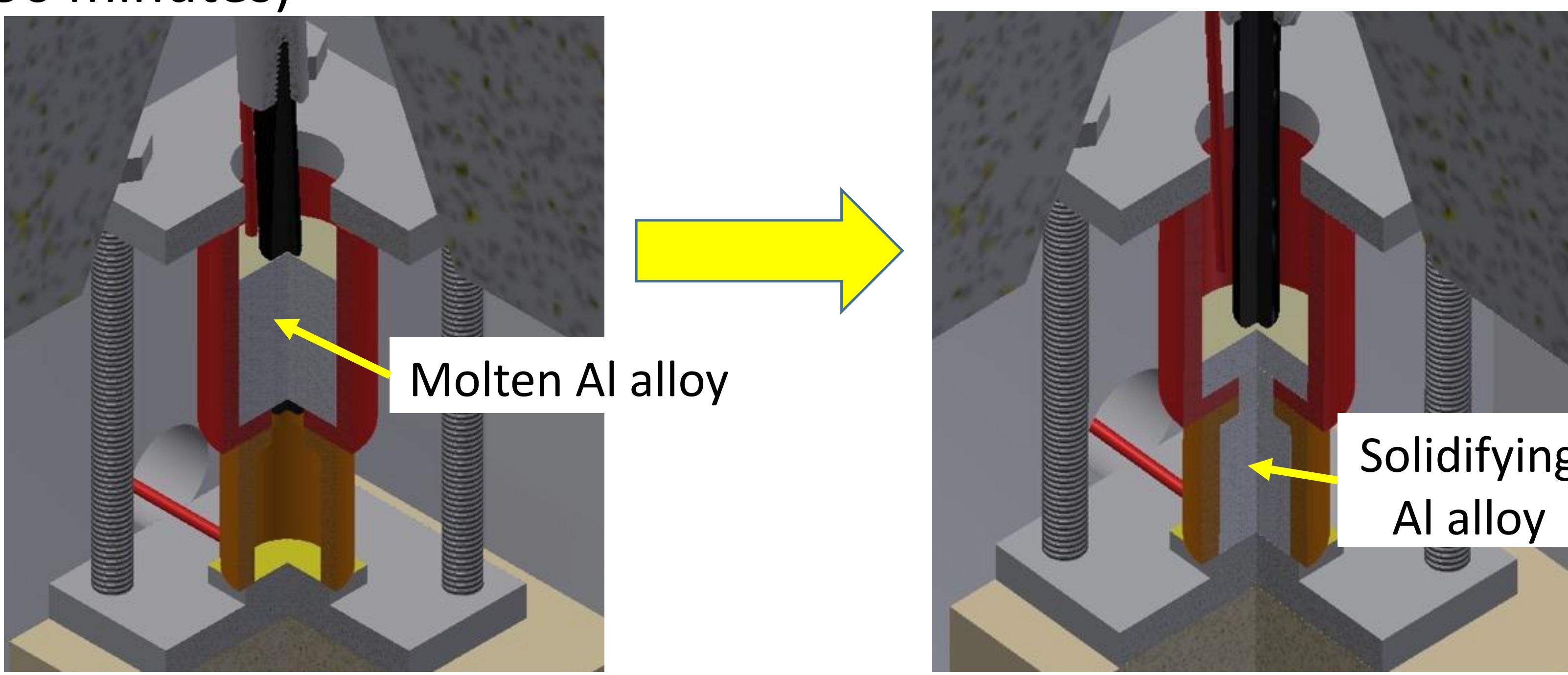
1st Proposed Solution

- Development of a setup to allow bottom pouring and avoid the top oxide layer from the molten alloy be in contact with material coupon



Bottom Pouring at Resistance Furnace

- Entire unit is placed into room temperature resistance furnace
- Aluminum pieces are placed into upper chamber
- Unit is heated to 700°C (~50 minutes)
- Stopper rod is removed
- Aluminum falls into lower chamber and contacts substrate test material
  - Surface oxide remains behind in upper chamber



1st Proposed Adhesion Test

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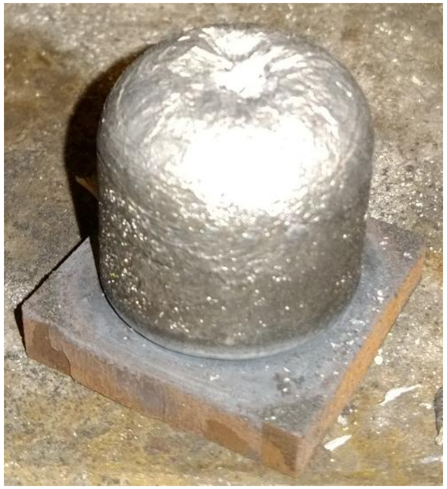
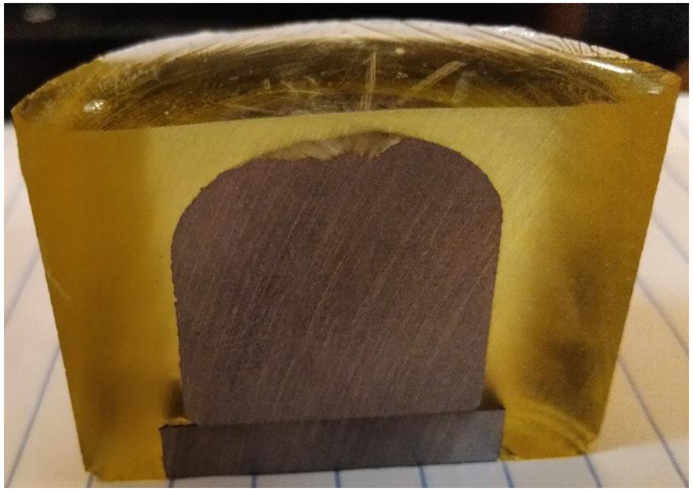
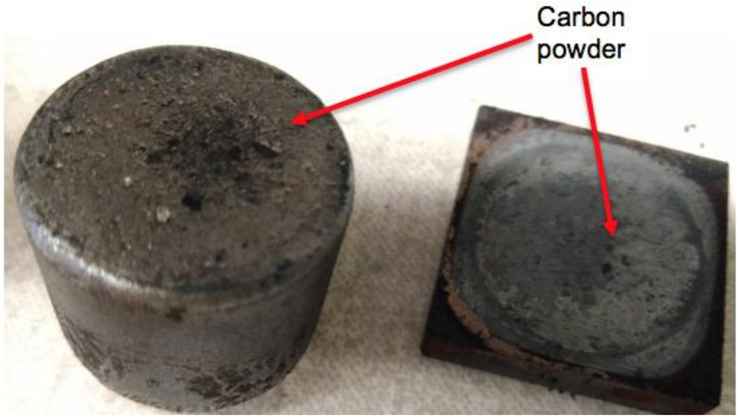
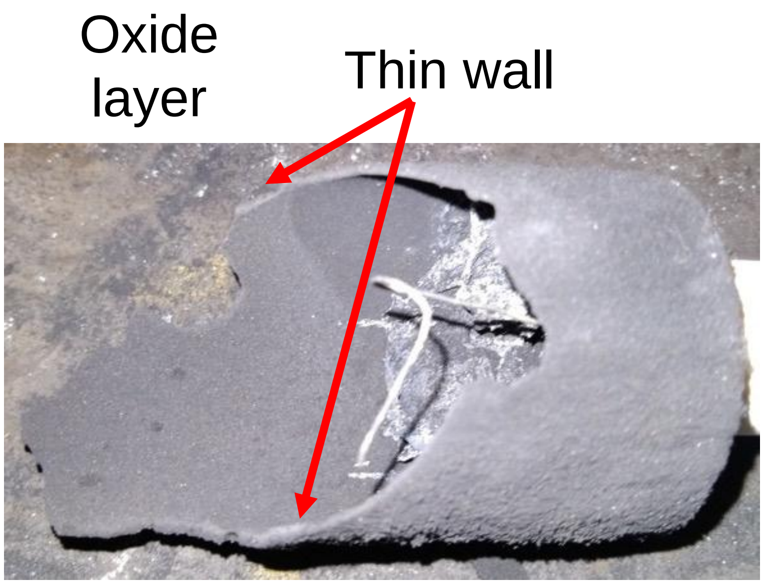
- Molten aluminum is held in contact with substrate for controlled period of time
- Temperature is recorded during entire experiment
- Entire unit is slowly cooled to room temperature
- Setup is disassembled
- Sample can be tensile tested





Drawbacks of this approach

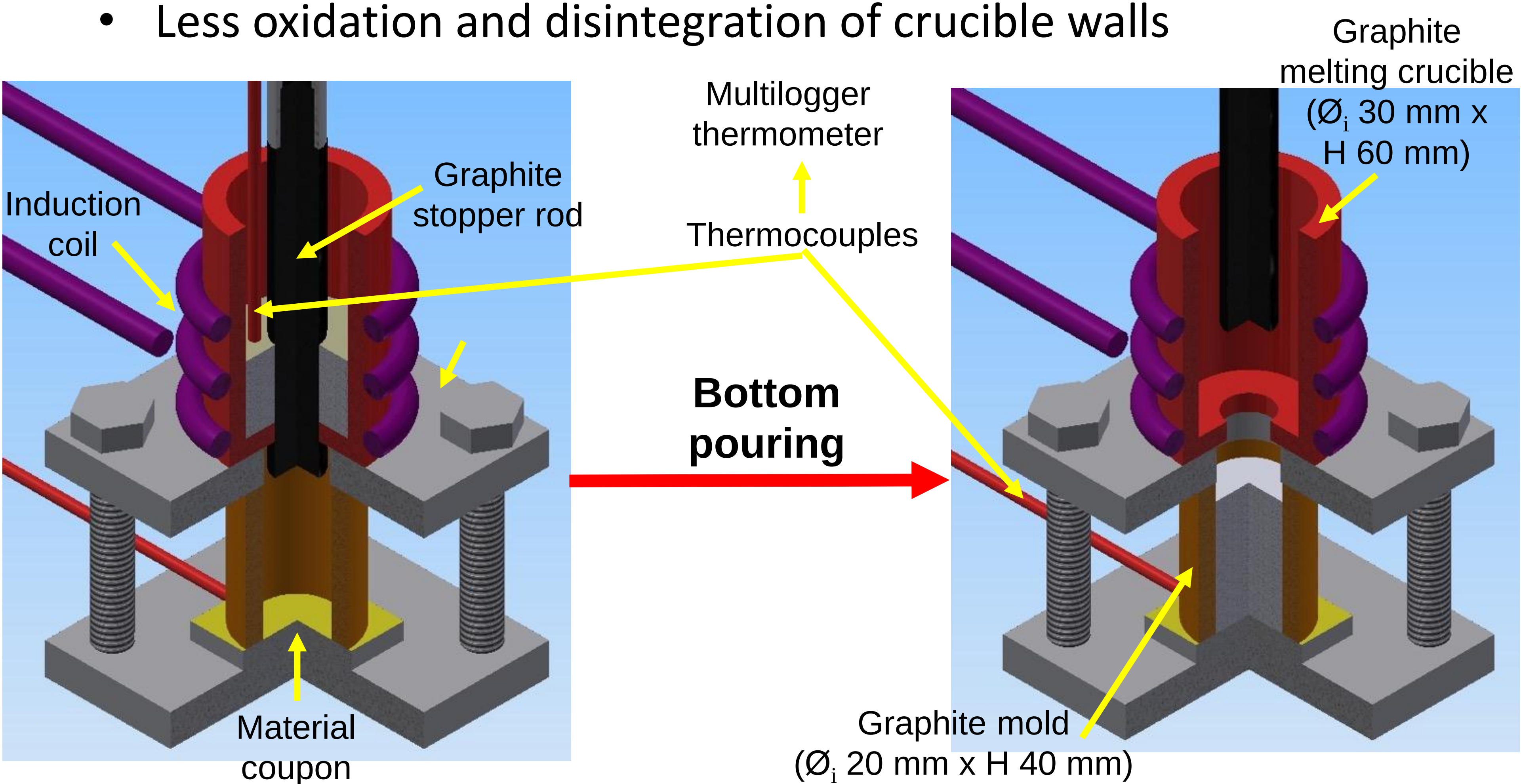




- Carbon powder was observed between solidified aluminum and steel substrate
- Most likely originated from walls of graphite crucibles
- Fell onto upper surface of steel substrate during heating
- Prevented contact between molten aluminum and substrate

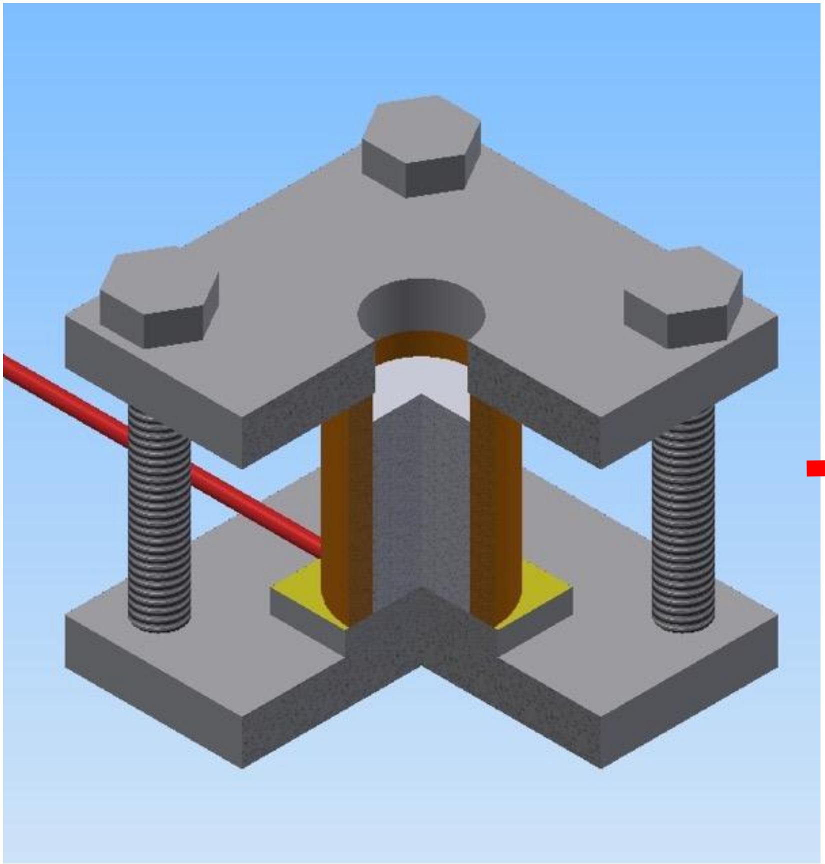
Task 1: Improved Adhesion Test

- Use induction melting
- Rapid heating to melt aluminum (~10 minutes)
  - Less oxidation and disintegration of crucible walls



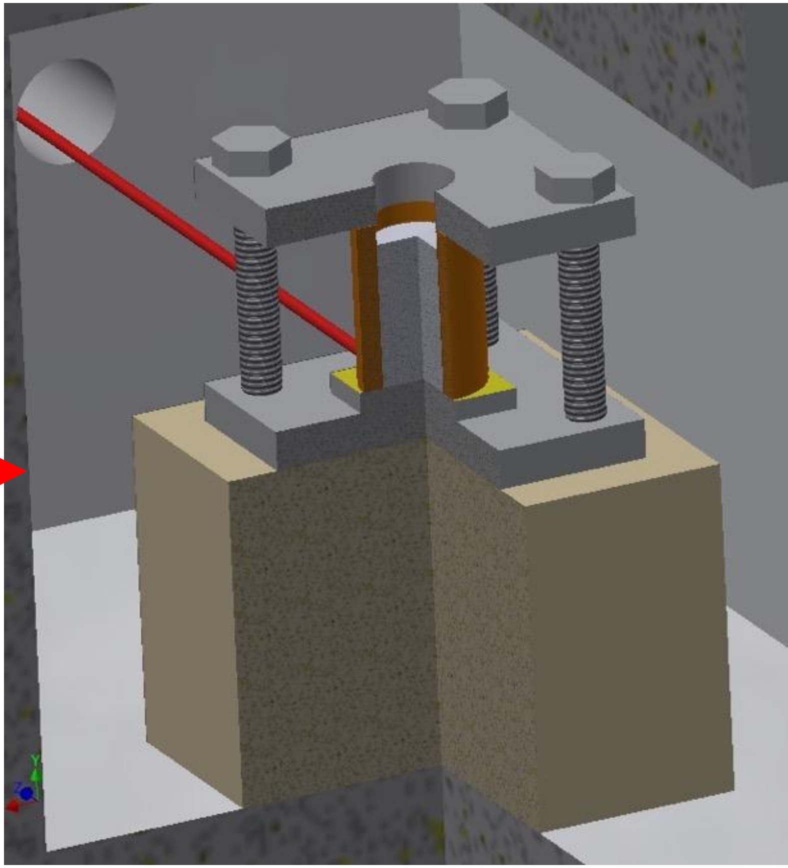
Modified Test Apparatus

- After aluminum has been poured into lower chamber
- Lower section is placed into resistance furnace pre-heated to 700^{o}C



Resistance furnace at 700 ºC

Holding time



1st Test Using Induction Melting

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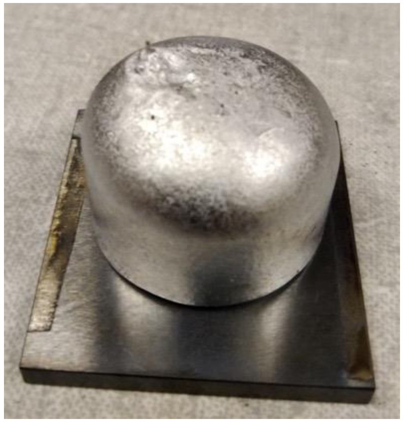
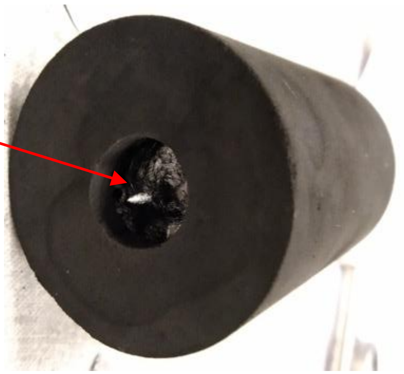
- Melted aluminum in induction coil
- Pulled stopper rod
- Molten aluminum fell onto room temperature substrate and solidified



No carbon layer

Al oxides stayed at the melting crucible



Planned Initial Experiments

- Planned initial experiments
  - 700 °C; 3 different holding time
    - 15mins, 1 hr, 4 hrs
  - Uncoated H13 steel substrate
    - Ground to 600 mesh SiC finish
    - Polished to 1μm
  - PVD coatings
    - ZrN

Temperature Profiles

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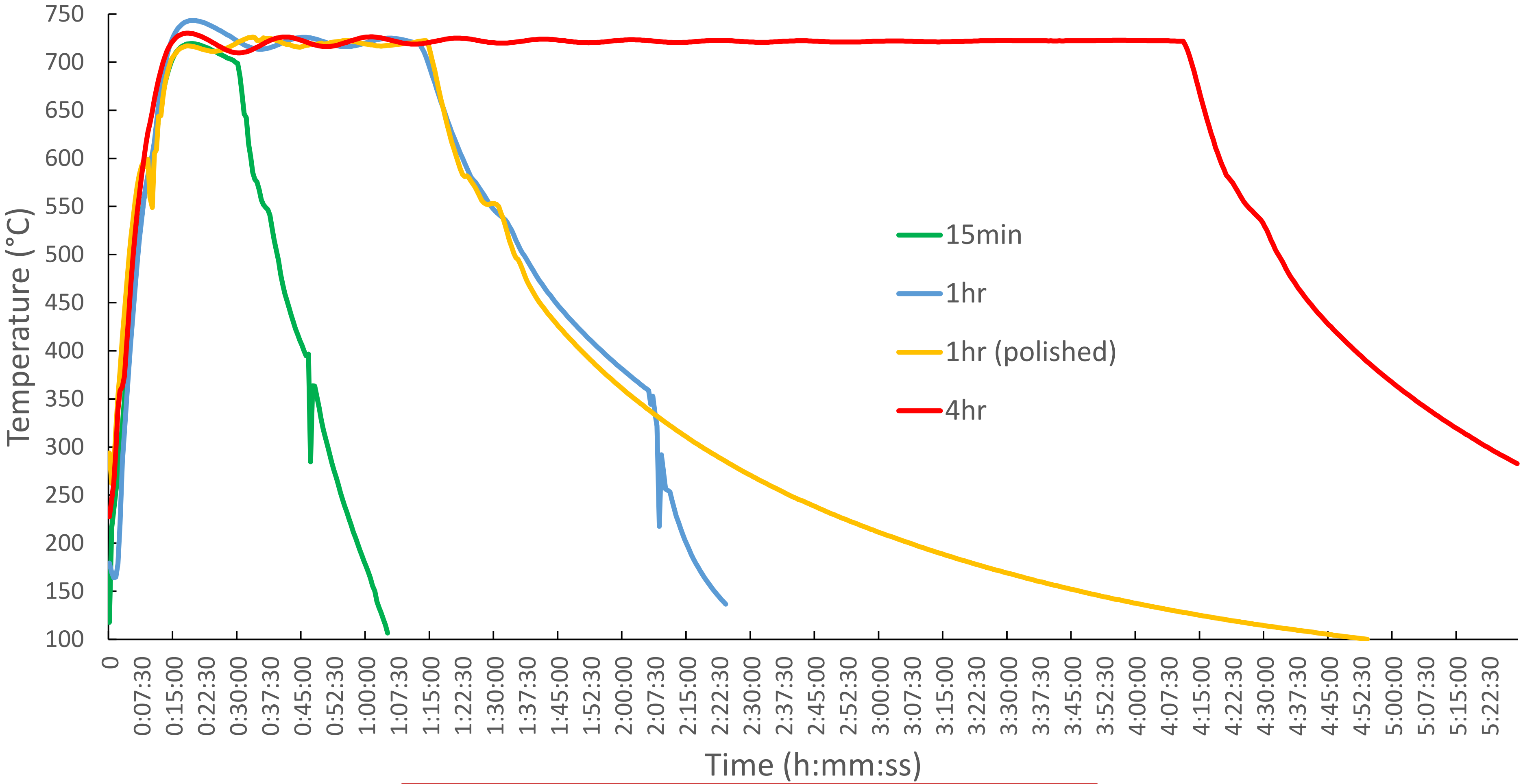
- Same average behavior in temperature profile for H13 steel substrate after bottom pouring and placed at 700 ºC pre-heated resistance furnace
  - ~13 minutes to reach 700 ºC

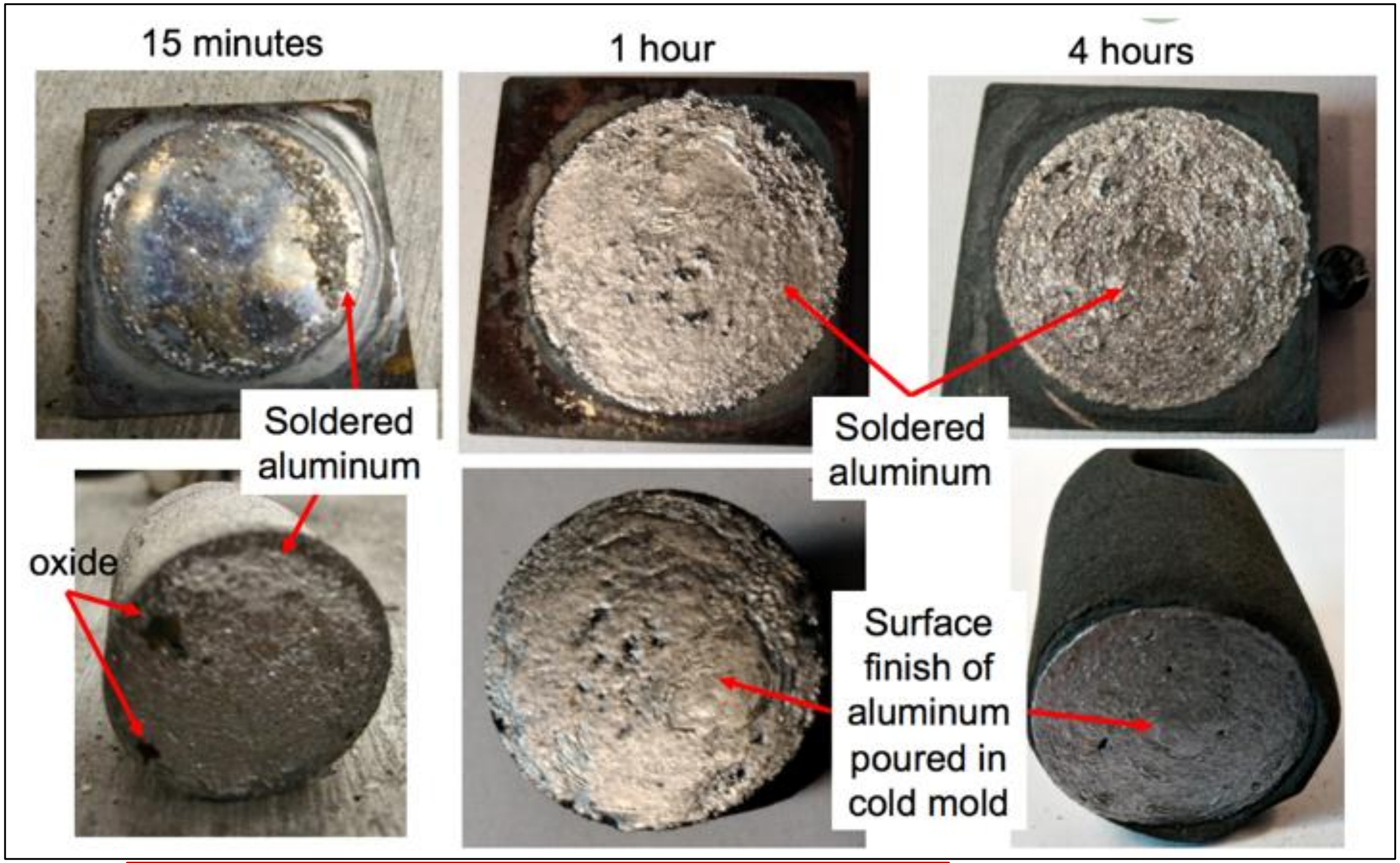


Bare H13 (ground 600 mesh)

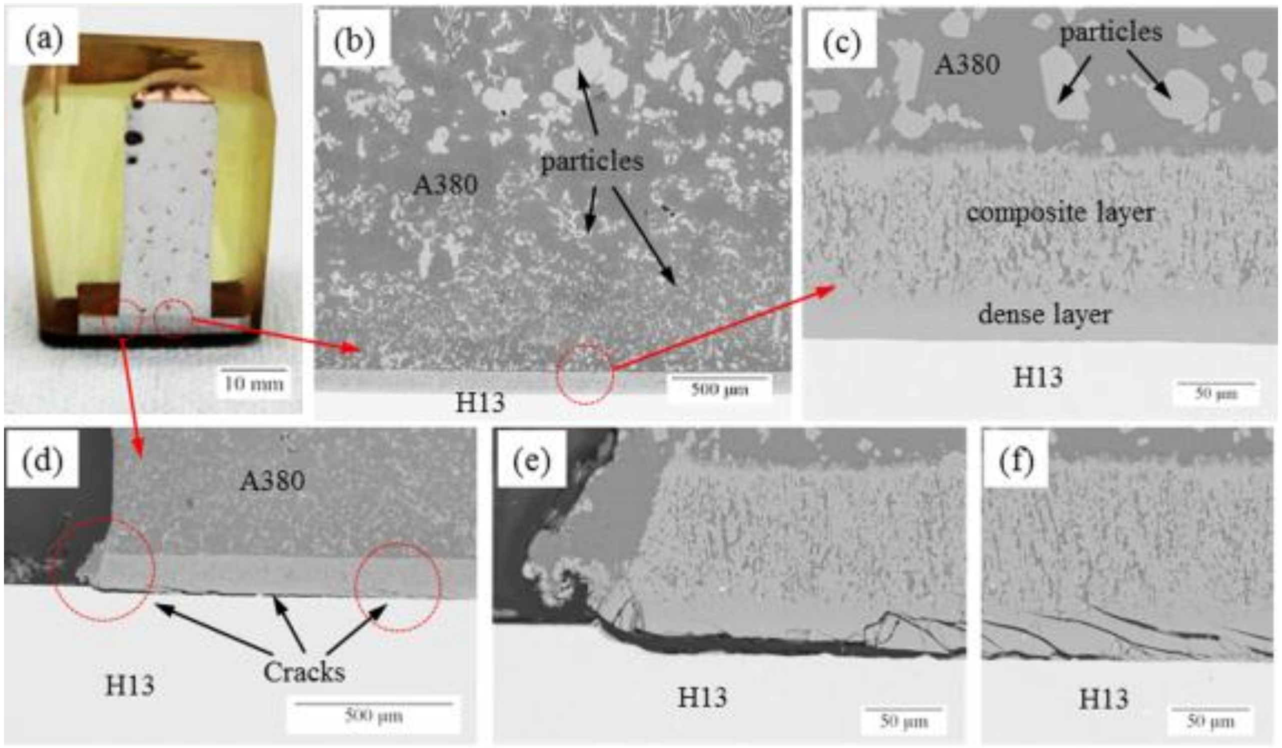
- Reaction occurred between molten aluminum & H13 steel substrate
  - Area of reaction was only small after 15 minutes
- Aluminum separated from steel during cooling to room temperature

H13 Steel substrates

Solidified Al cylinders



Al + Bare H13 Substrate: Results from Bo Wang



- Bo Wang saw a similar phenomena
  - Cracking between the solidified aluminum and bare H13 substrate

Source: B. Wang, Ph.D. thesis, Colorado School of Mines, 2016

Wang's Aluminum Adhesion Test Results

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#	Material	Supplier	Method & Treatment	Composition	Thickness [μm]	Breaking Strength [MPa]
1	AlCrN	1	CAE	$Al_{0.67}Cr_{0.33}N_x$	1.7	0
2	AlTiN	2	CAE	$Al_{0.68}Ti_{0.32}N_x$	1.5	0
3	CrWN	2	Nitriding + CAE	$Cr_{0.97}W_{0.03}N_x$	6.7	0
4	AlTiN	3	CAE	$Al_{0.62}Ti_{0.38}N_x$	3.3	0*
12a	AlCrN	5	Nitriding + CAE	—	—	0*
5	TiAlN	3	CAE	$Ti_{0.56}Al_{0.44}N_x$	1.8	0.01
12b	AlCrN	5	CAE	—	—	0.04
6	CrN	4	CAE + Fine Polish	CrN_x	4.4	0.07
7	H13	—	—	—	—	0.12
8	Cr	CSM	MS	Cr	2.2	0.12
9	CrWN	4	CAE + Fine Polish	$Cr_{0.95}W_{0.05}N_x$	4.2	0.26
10	CrN	5	Filtered CAE	CrN_x	5.0	0.78
11	TiN	CSM	MS	TiN_x	0.6	0.84
12	AlCrN	5	CAE + Fine Polish	$Cr_{0.54}Al_{0.46}N_x$	5.1	1.30
13	TiB_2	6	MS	TiB_x	1.5	2.54

- H13 did not have the highest breaking strength
  - Possibly due to cracking at interface

Source: B. Wang, Ph.D. thesis, Colorado School of Mines, 2016

Polished H13 (1μm diamond)

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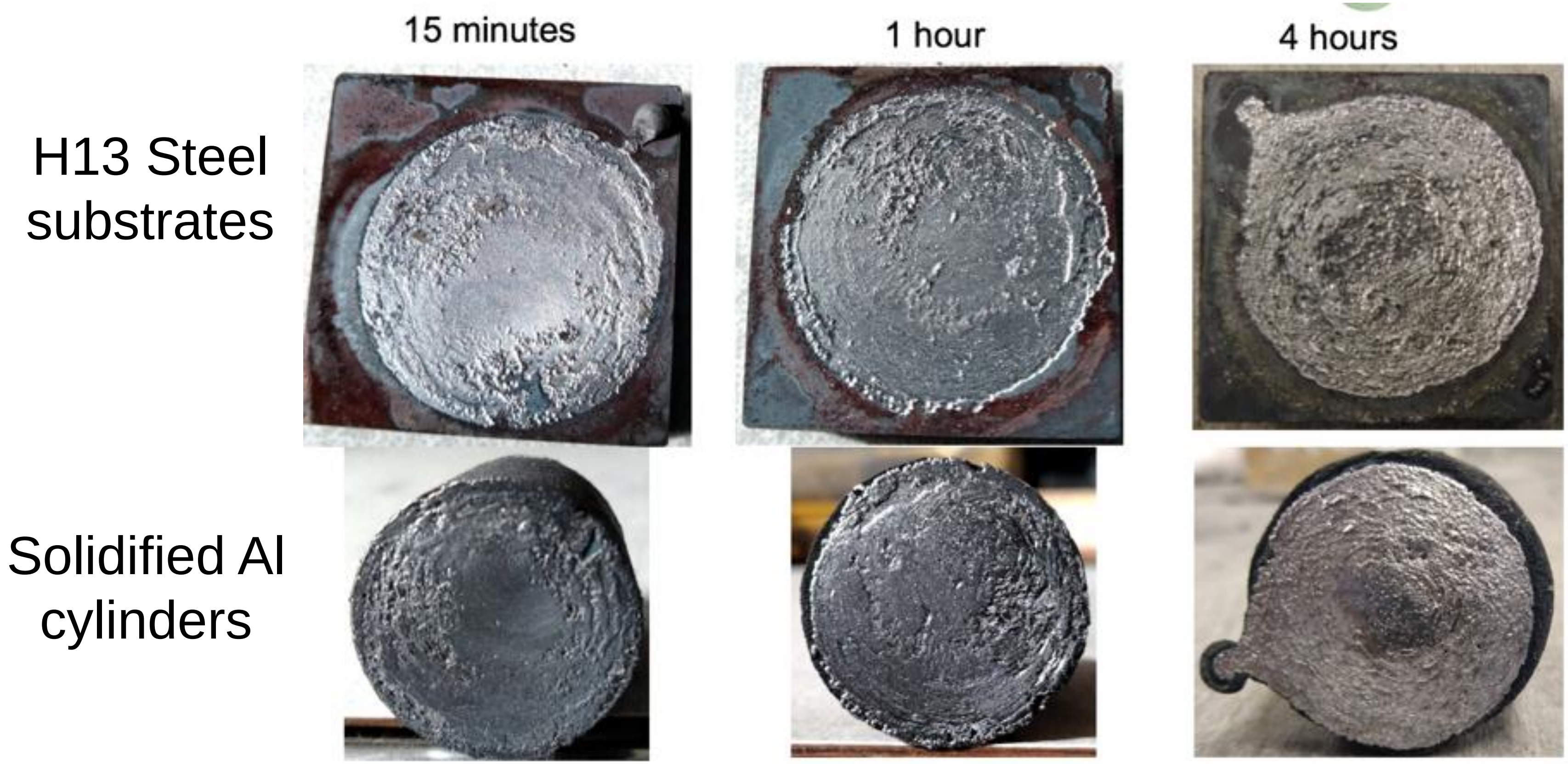
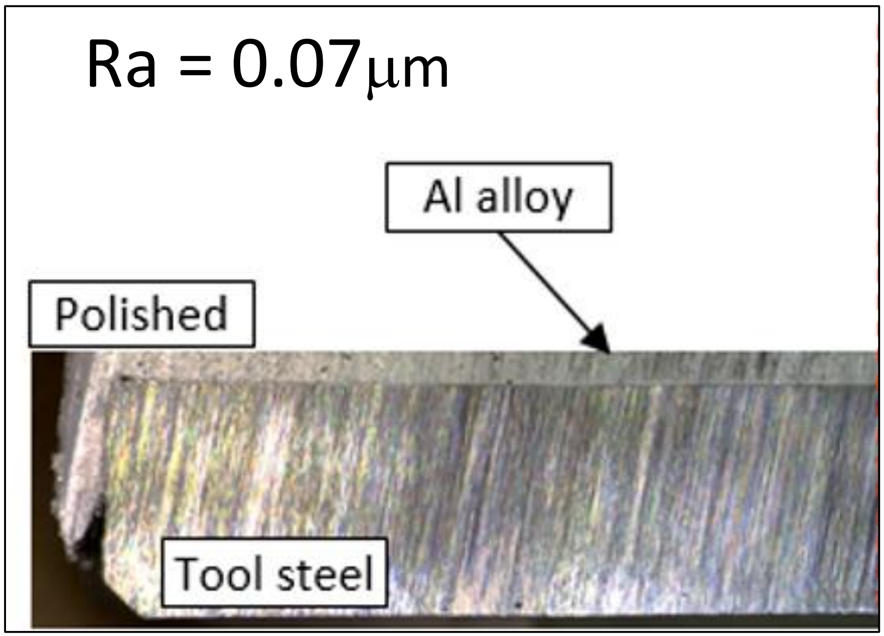
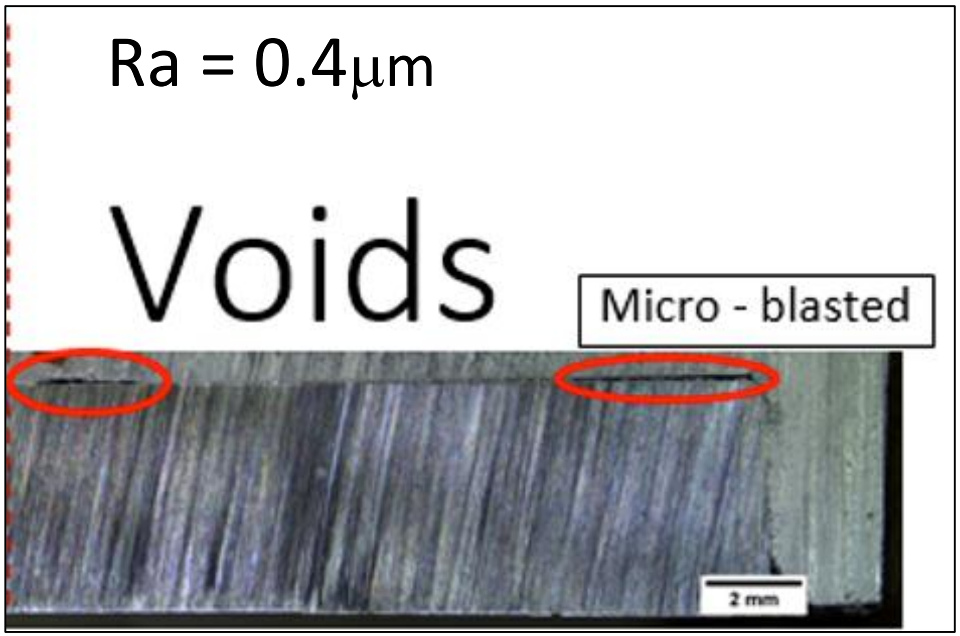
- Similar reaction occurred between molten aluminum & H13 steel substrate
  - Area of reaction was much larger for 15 minute sample
- Aluminum separated from steel during cooling to room temperature



Influence of Surface Finish: Published Data

- Testing performed by Gobber et al. in Italy
  - Examined impact of surface roughness on adhesion of molten Al to H13 steel
  - Rougher H13 substrate resulted in less contact between solidified aluminum and steel
- Similar to our results
  - We saw less adhesion/reaction after 15 minutes for the rougher sample





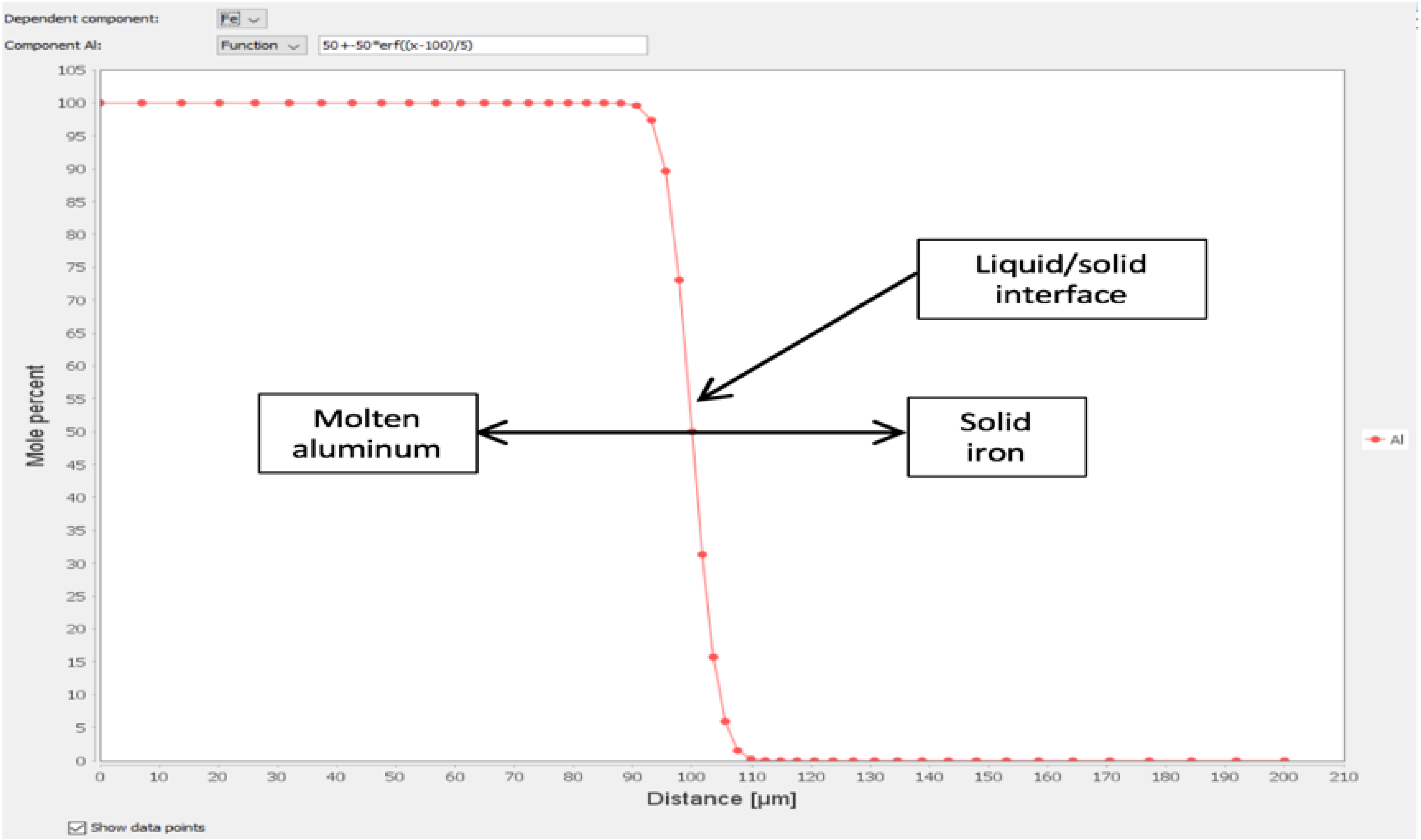
Source: F.S. Gobber, D. Ugues and M. Rosso, S2P, 2018.

Task 2: Adhesion Mechanism

- Understand the inter-diffusion behavior between molten aluminum and steel
- Simulation packages can provide a powerfull resource
- CALPHAD method
  - Using DICTRA module of Thermo-Calc
- Initially evaluated a simplified diffusion-couple model
  - Pure Al in contact with pure Fe
  - Held at 680^{o}C for different times

DICTRA Simulation

- Starting condition



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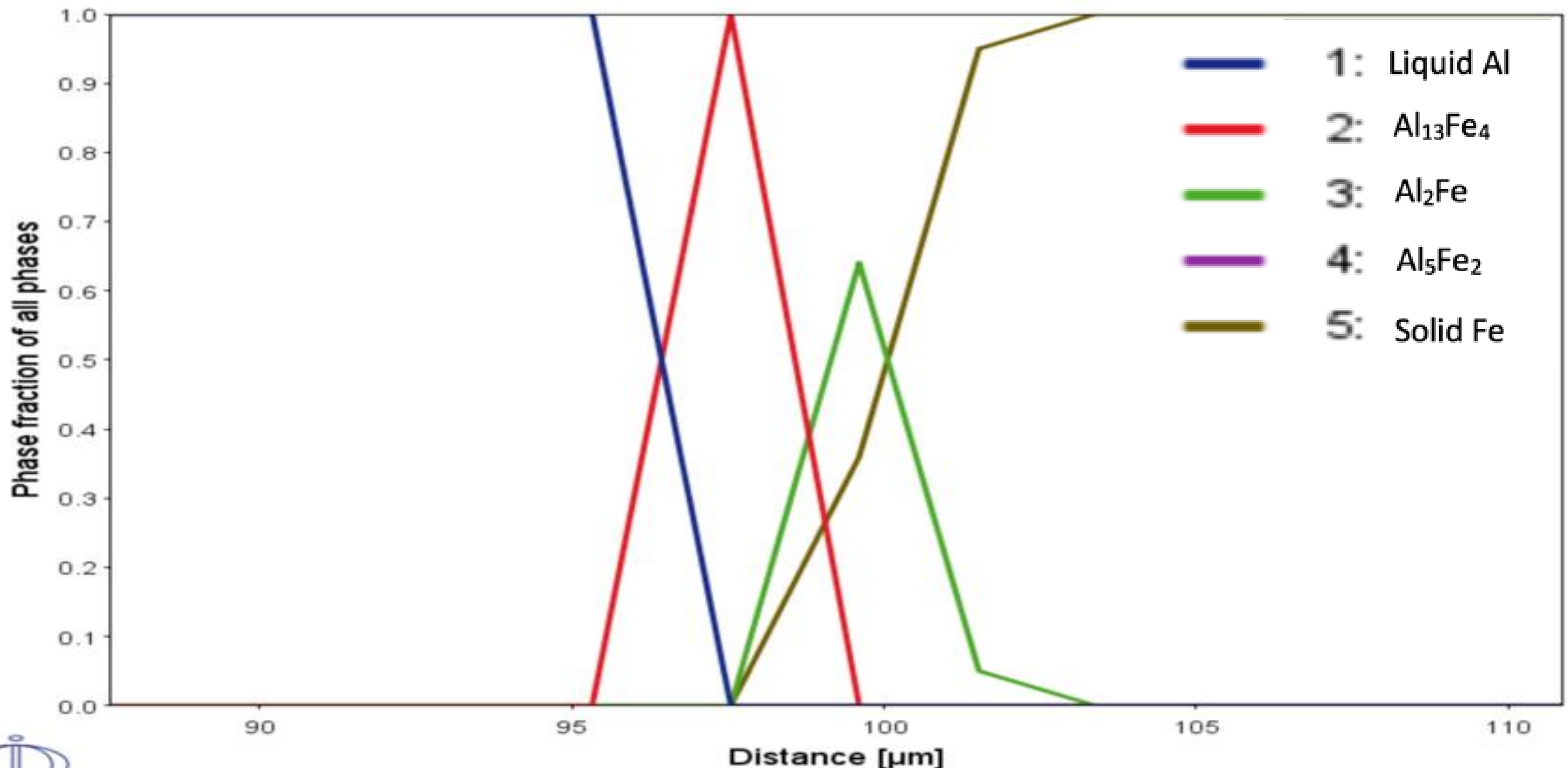
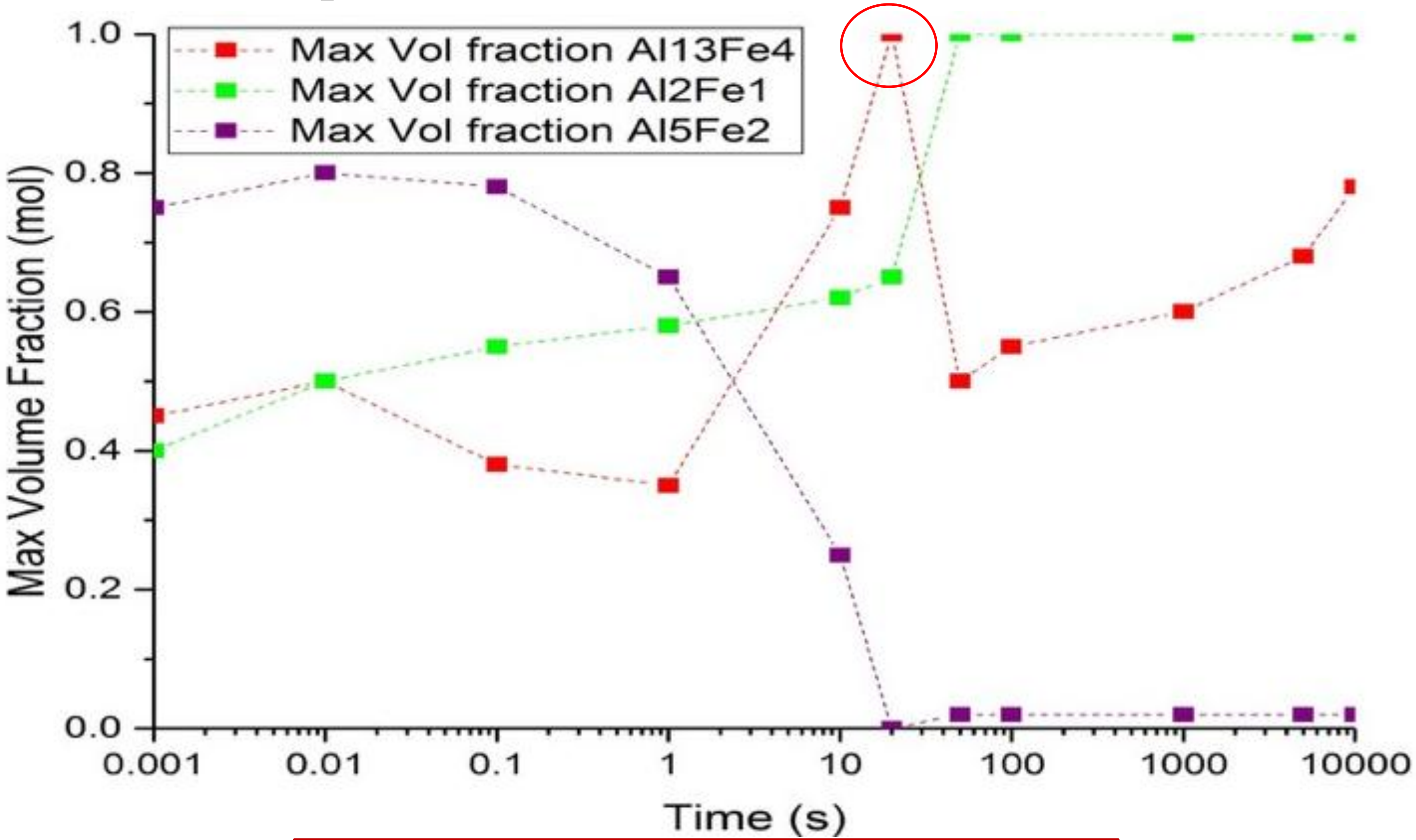
20 seconds at 680 ºC

- Shows volume fraction of three phases formed at interface between liquid aluminum and solid iron
  - $Al_{13}Fe_4$, Al_2Fe, and Al_5Fe_2



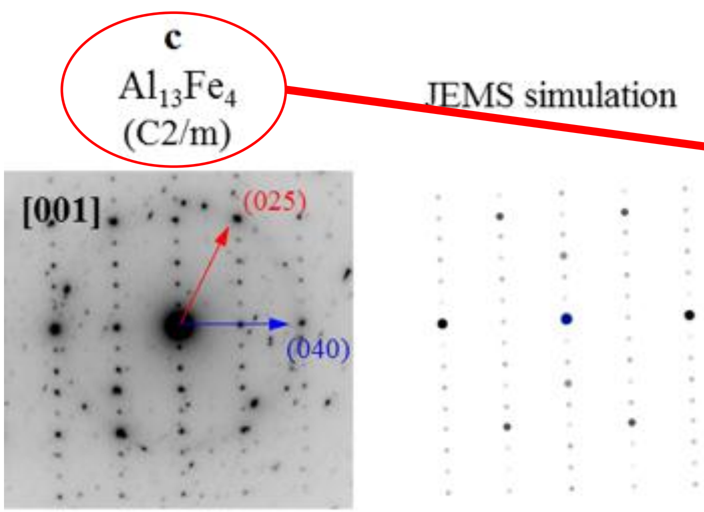
Volume Fraction

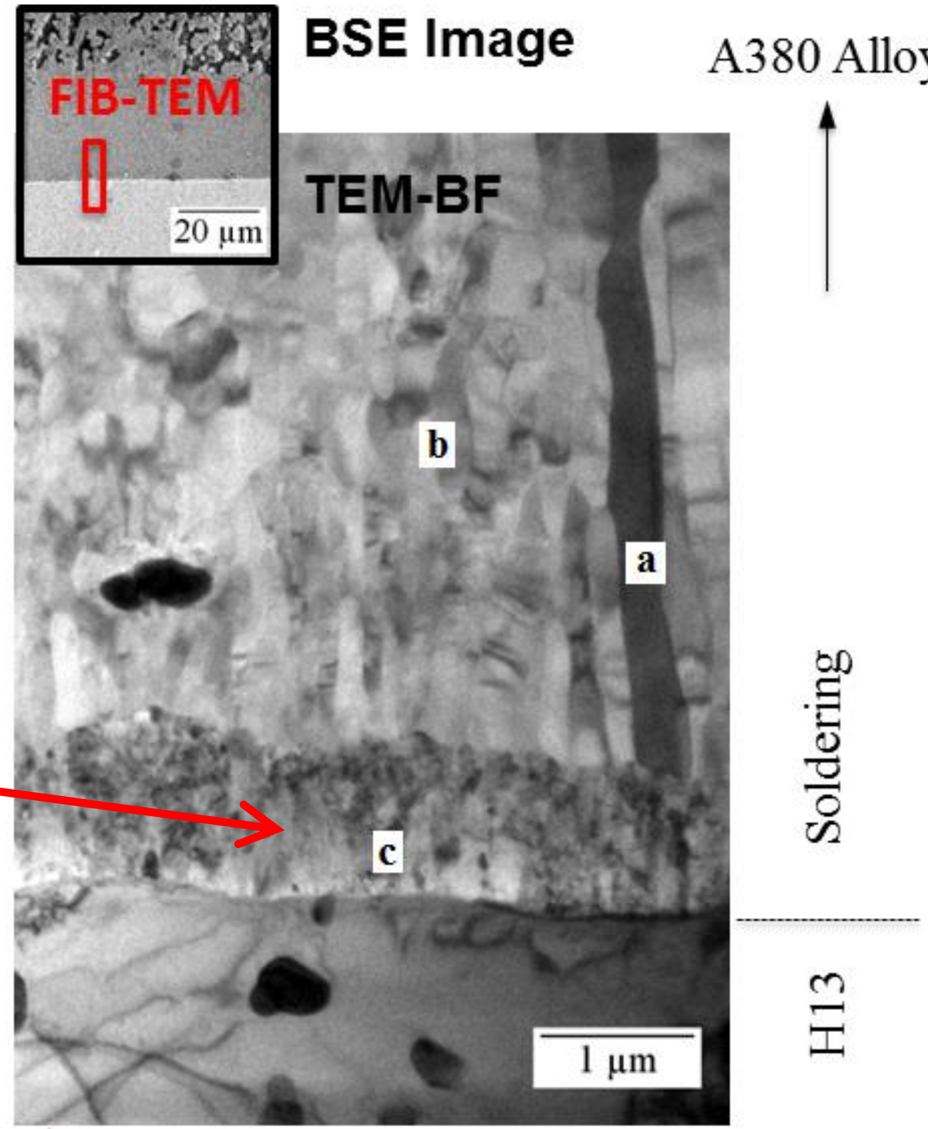
- Shows volume fraction change of each phase with time
  - Al_5Fe_2 initially forms
  - At about 20 seconds, $Al_{13}Fe_4$ had the highest volume fraction
  - Longer times, Al_2Fe had the highest volume fraction



Bo Wang's results using uncoated H13 steel

- Bo Wang detected $Al_{13}Fe_4$
- Did not detect Al_2Fe or Al_5Fe_2
- Need to increase the complexity of the DICTRA simulation





Summary & Conclusions

- Developed a modified adhesion test using induction heating: initial tests
  - 600 mesh ground H13: aluminum soldering occurred, but the interface cracked separating the solidified aluminum and the steel
  - 1 μm polished H13: same results, but more aluminum soldered with the 15 minutes test

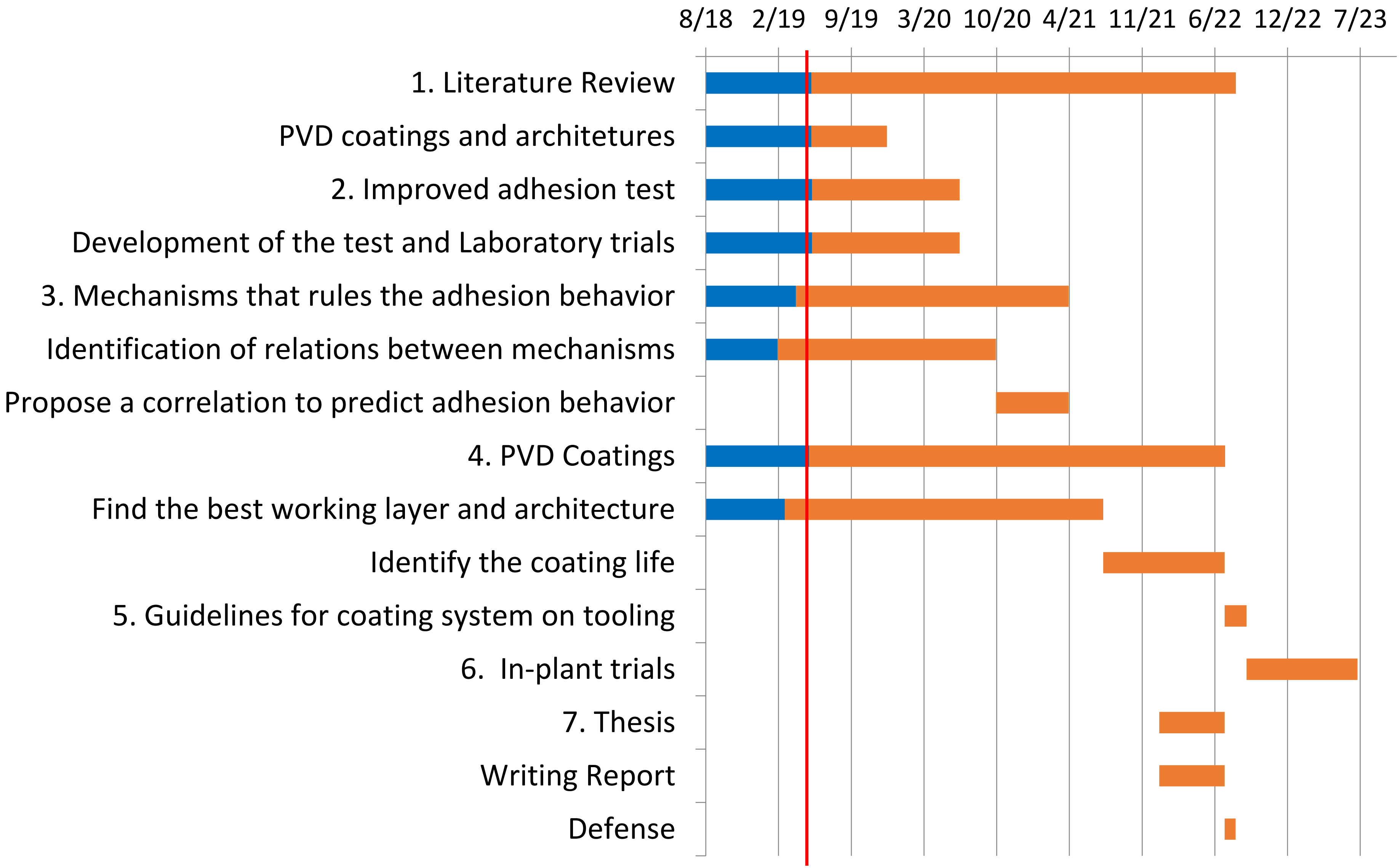
Summary & Conclusions

- Adhesion mechanism: Thermo-Calc simulations
  - Simplified model predicted one of the phases experimentally observed in the project performed by Bo Wang
    - Use DICTRA to predict diffusion profiles
  - Use the Property Model Calculation module to evaluate wetting of coatings by molten aluminum
  - Simulation can be a powerful resource to predict suitable coatings

Future Work

- Literature review
  - PVD coatings currently used by die casters and other industries
  - Chemical interactions between liquid metals and solid materials
  - Wetting of solids by liquid metals
  - Brazing
- Experimental work
  - Uncoated H13 with rougher surface (ground 80 mesh SiC)
  - PVD coated samples: ZrN, carbides
- Characterization
  - Examine the phases formed at the interface between the solidified aluminum and H13 steel substrate

Progress (Gantt chart)



Acknowledgement

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Thank you!

Nelson Delfino de Campos Neto
ndelfino@mymail.mines.edu

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Industrial Partners: *Paul Bracaleon (NADCA)*

Project Duration: *Aug. 2018 – July 2023*

Achievement

- Identify PVD coatings to be applied to die casting dies to avoid the molten aluminum soldering and understand the adhesion mechanisms involved.

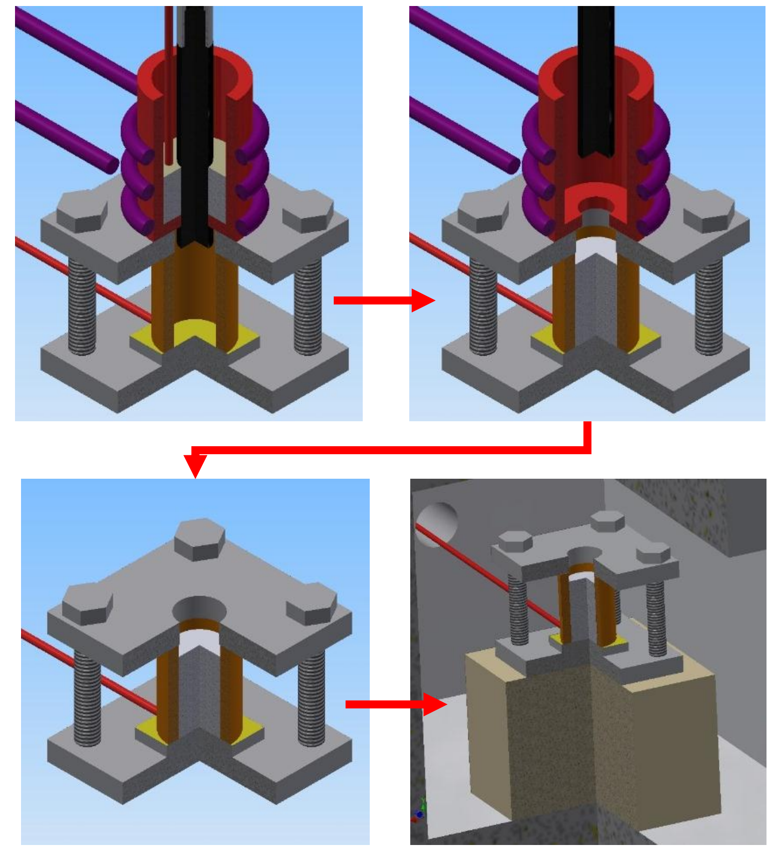
Significance and Impact

- Increase die casting parts quality, eliminate the use of lubricants, extend the dies life and reduce cost-per-parts.

Research Details

- Development of an advanced laboratory test to simulate the aluminum die cast process in order to find the best PVD coatings and understand the adhesion mechanisms involved.

Induction Melting — Bottom Pouring



Place in a furnace — Holding time at 700 ºC

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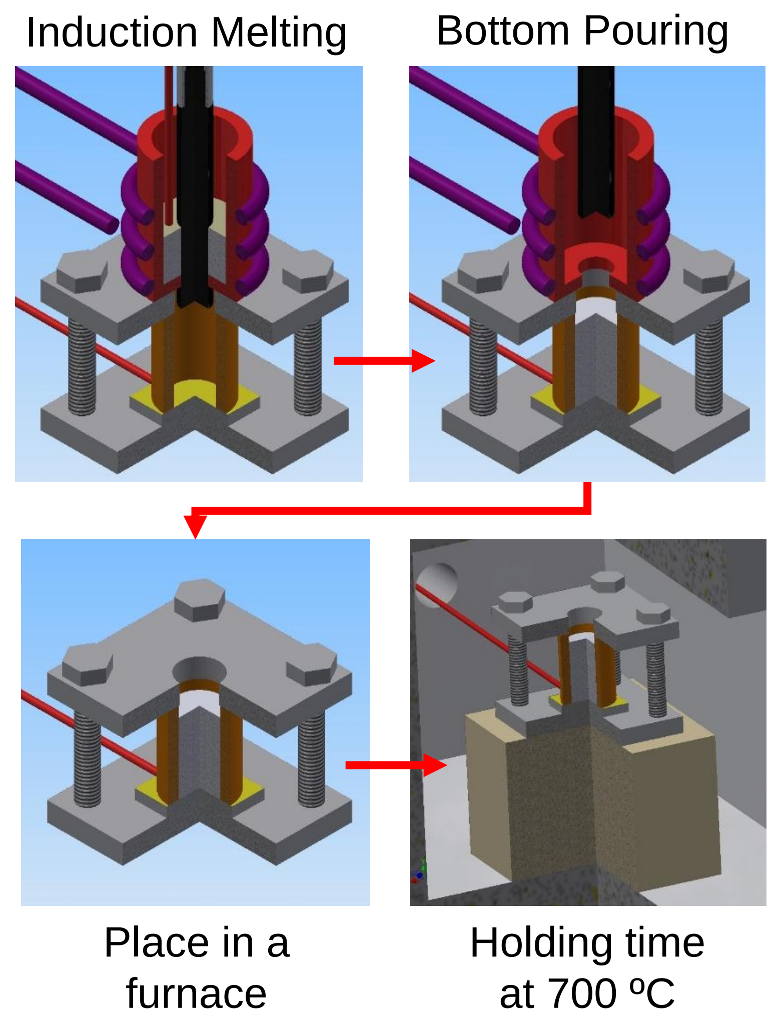

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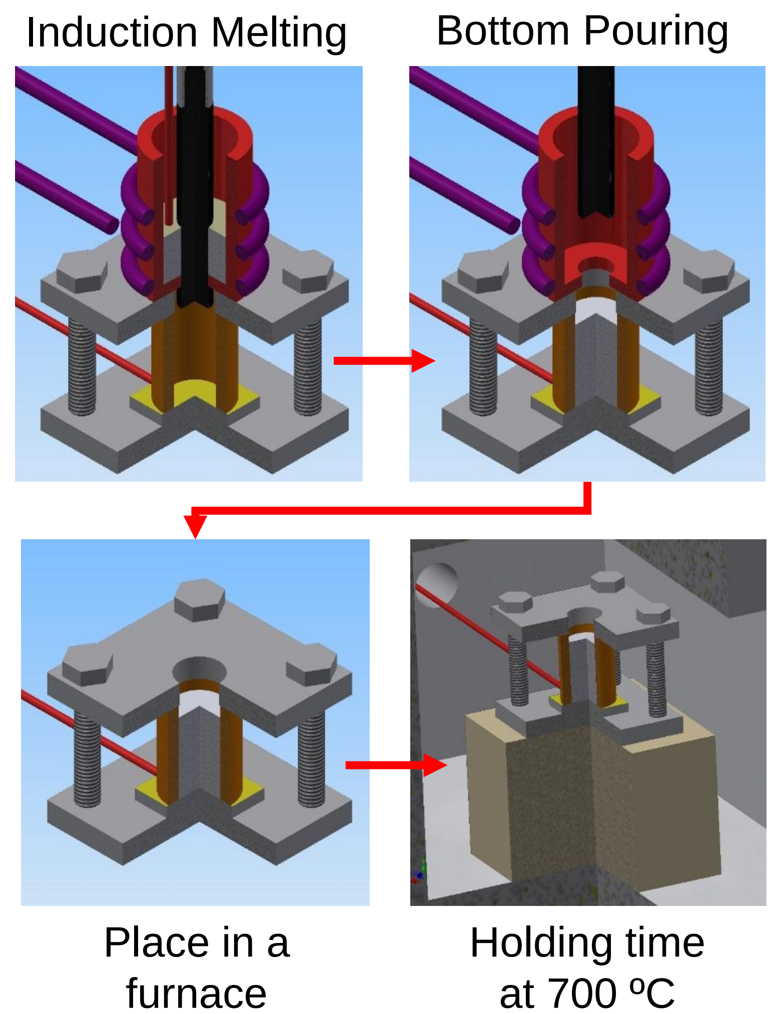

Program Goal

- Identify PVD coatings to be applied to die casting dies to avoid the molten aluminum soldering and understand the adhesion mechanisms involved.

Approach

- Develop an advanced laboratory test to simulate the aluminum die cast process in order to find the best PVD coatings and understand the adhesion mechanisms involved.

Benefits

- Increase die casting parts quality, eliminate the use of lubricants, extend the dies life and reduce cost-per-parts.