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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Industrial Mentors: Paul Brancaleon (NADCA), Rob Mayer (Queen City Forging Co.)

Project 37-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 during the die casting process. Lubricants are applied to the die to reduce soldering and adhesion, but the lubricant reduces part quality.
- Objective: Identify PVD coatings to be applied to die casting dies to prevent the soldering. Understand the mechanisms involved with adhesion.
- Benefit: Increase die casting part quality, eliminate the use of lubricants, extend die life and reduce cost-per-part.

Recent Progress
- Literature review
- Development of an improved adhesion test
- Performed initial trials using the new adhesion test
- Selected and obtained a number of PVD coated substrates
- Initial characterization of cast samples

Metrics

Description	% Complete	Status
1. Literature review and development of an improved adhesion test that simulates the die casting process	30%	●
2. Identification of the mechanisms that controls the adhesion behavior.	15%	●
3. Identification of a working layer coating that avoids molten aluminum soldering and adhesion.	15%	●
4. Development of a coating architecture that will add sufficient durability to the die coatings to allow them to survive as long as the die casting die itself (100,000 shots)	0%	●
5. In-plant trials. Guidelines for depositing the coating system on die components/tooling.	0%	●

Industrial Relevance

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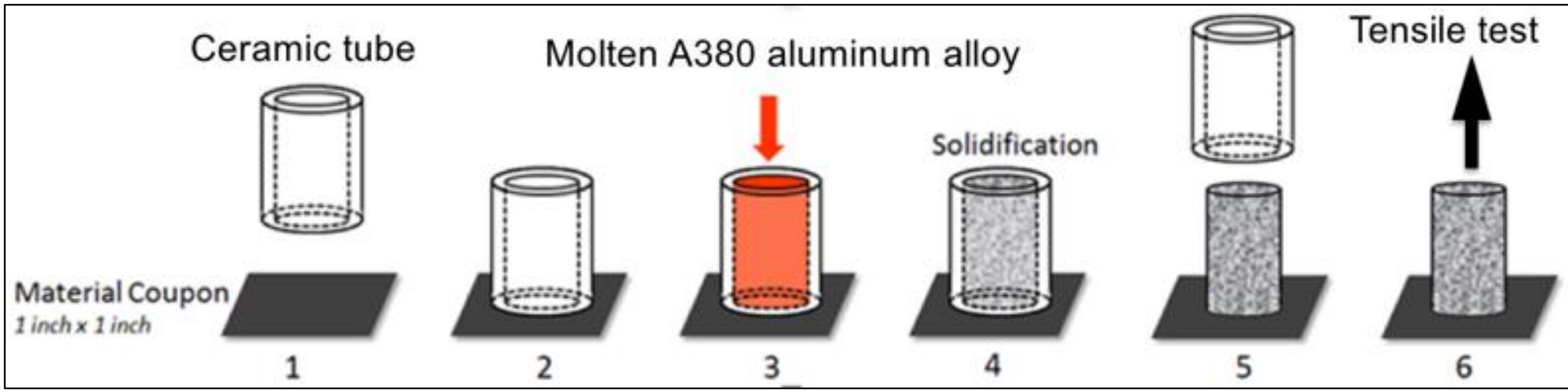
- Reducing or eliminating lubricant spray will:
  - Significantly **<u>improve the quality</u>** of the die castings
    - Reduce gas porosity and scrap
    - Allow castings to be used in higher performance applications
  - **<u>Reduce costs</u>**
    - Eliminate purchase costs for lubricants
    - Reduce effluent clean-up costs
    - Significantly extend die life
  - **<u>Improve productivity</u>**
    - 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

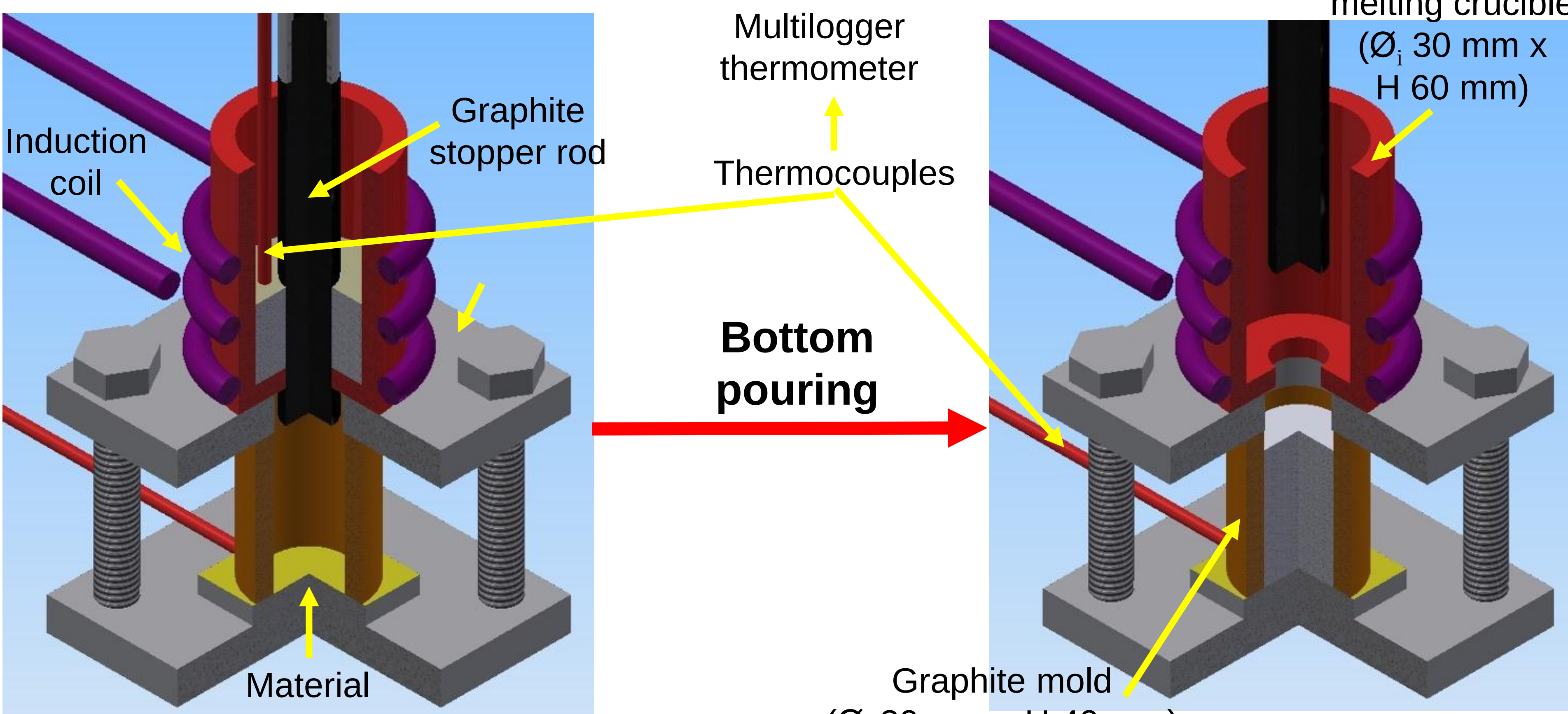


- Drawback:
  - Top pouring can carry oxide layer from the upper surface of molten aluminum into crucible to reduce contact between aluminum and material coupon

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

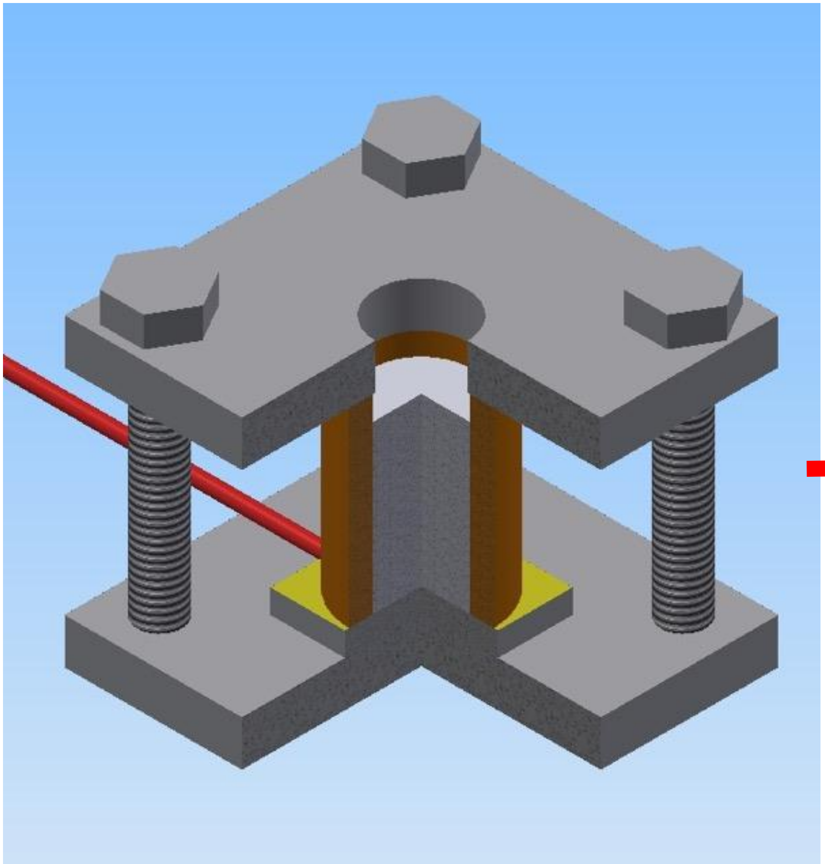
Task 1: Improved Adhesion Test

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



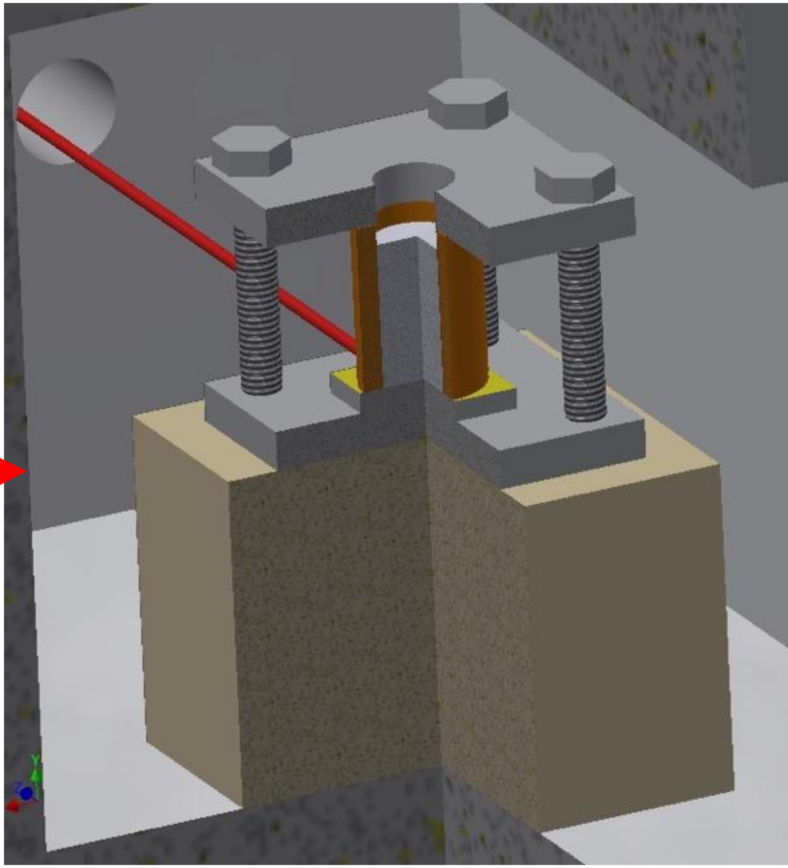
Modified Test Apparatus

- After aluminum has been poured into lower chamber
- Lower section is placed into resistance furnace pre-heated to 700ºC



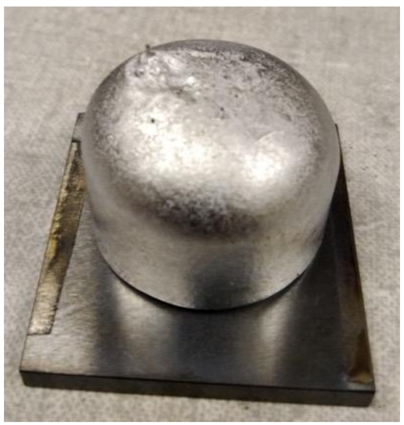
Resistance furnace at 700 ºC

Holding time

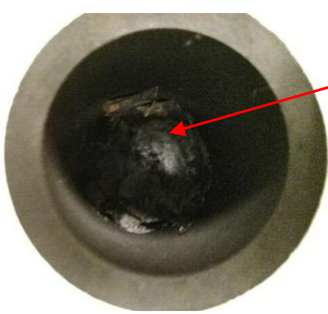


1st Test Using Induction Melting

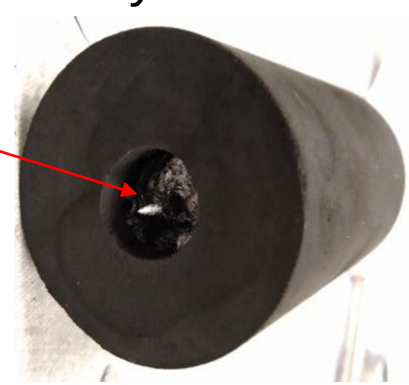
- Melted aluminum in induction coil
- Pulled stopper rod
- Molten aluminum fell onto room temperature substrate and solidified



No carbon layer



Al oxides stayed in the melting crucible

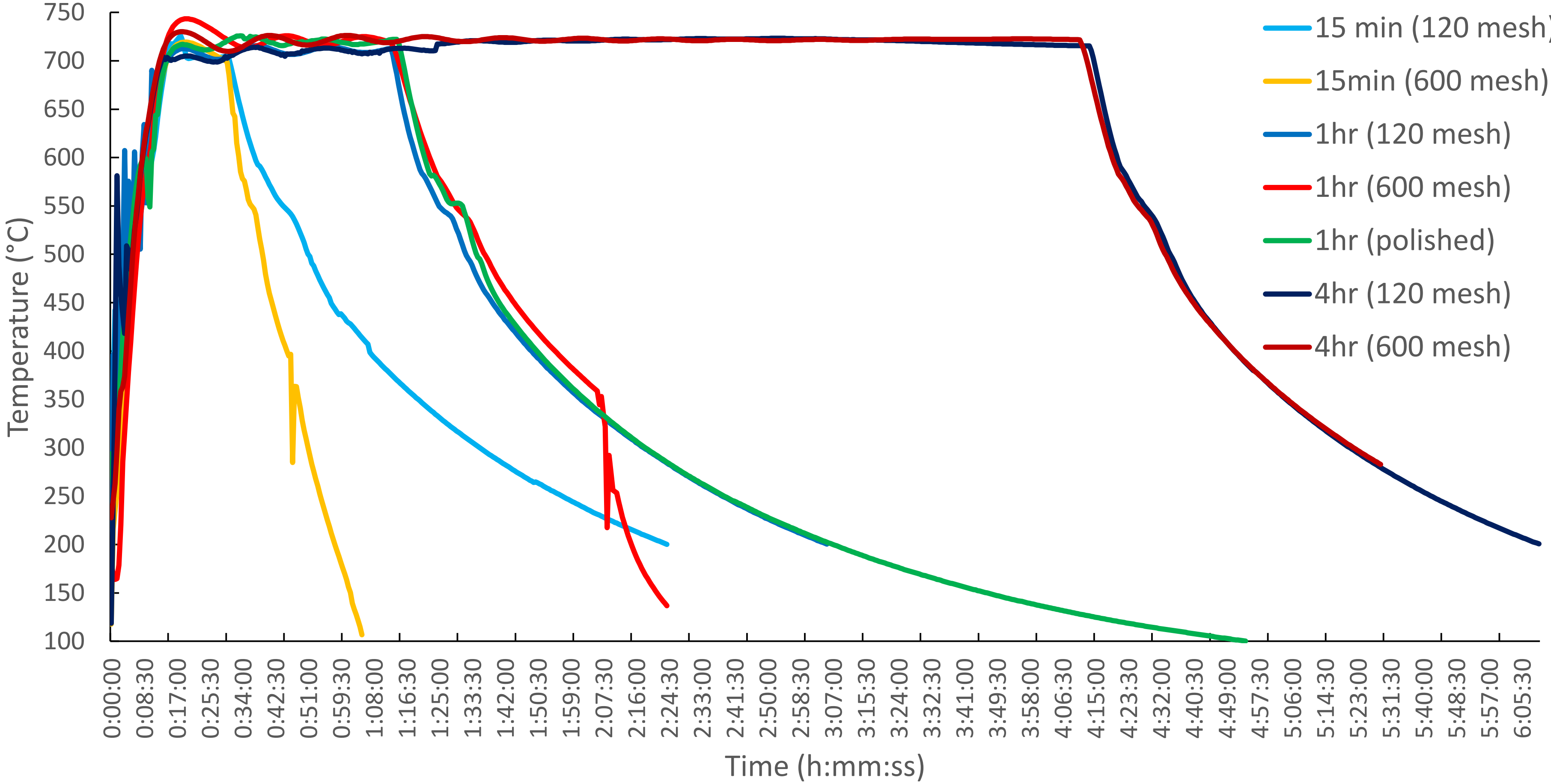


Planned Initial Experiments

- Temperature and time
  - 700 °C; 3 different holding times
    - 15mins, 1 hr, 4 hrs
- Material
  - Uncoated H13 steel substrate
    - Ground to 120 mesh SiC finish
    - Ground to 600 mesh SiC finish
    - Polished to 1μm

Temperature Profiles

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



Bare H13 (ground 120 mesh)

- Soldering occurred between molten aluminum & H13 steel substrate
  - For all times, aluminum adhered to substrate

15 min

1 hour

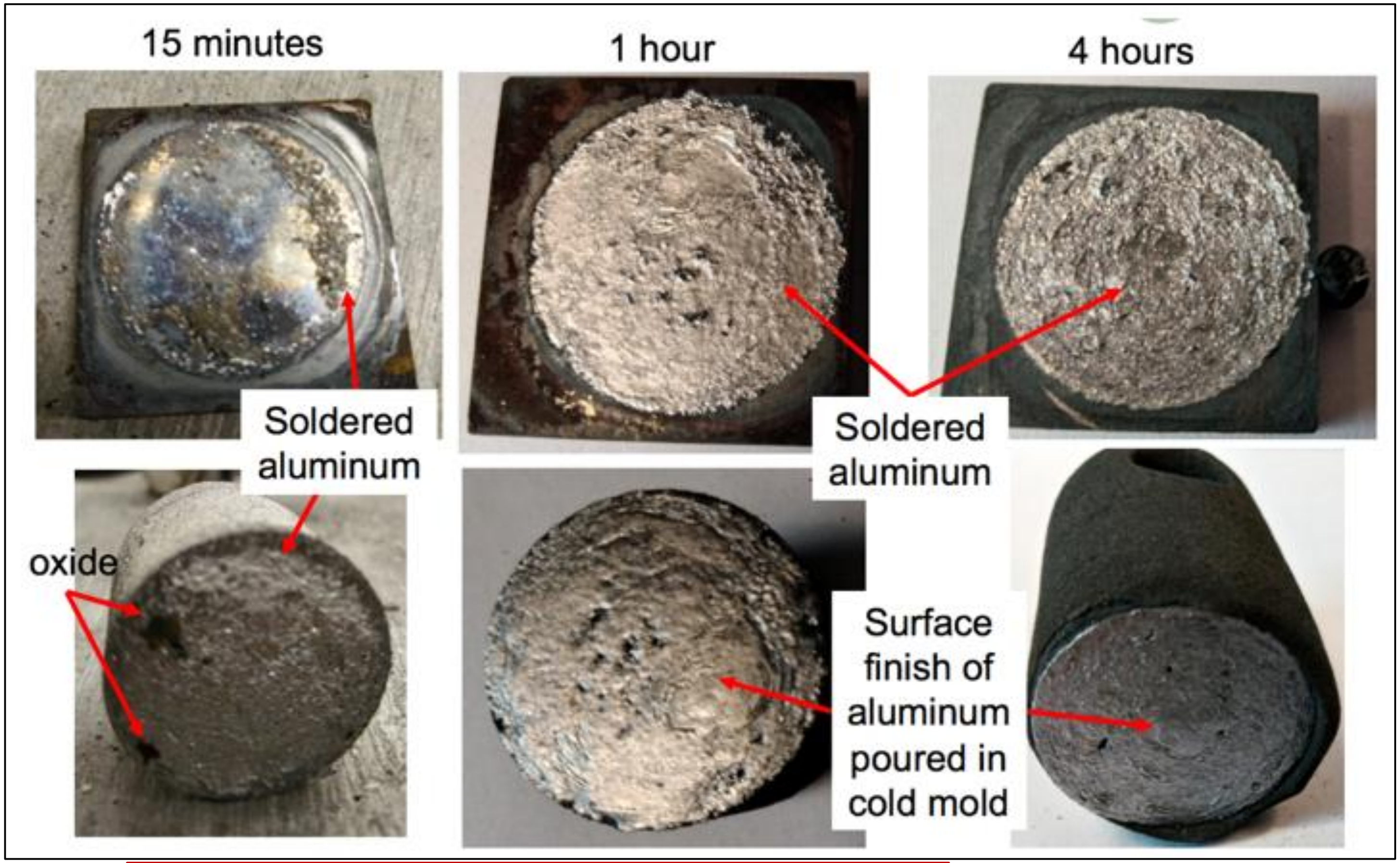
4 hours

Bare H13 (ground 600 mesh)

- Soldering 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 ground substrates

Solidified Al cylinders



Polished H13 (1μm diamond)

- 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

15 minutes | 1 hour | 4 hours

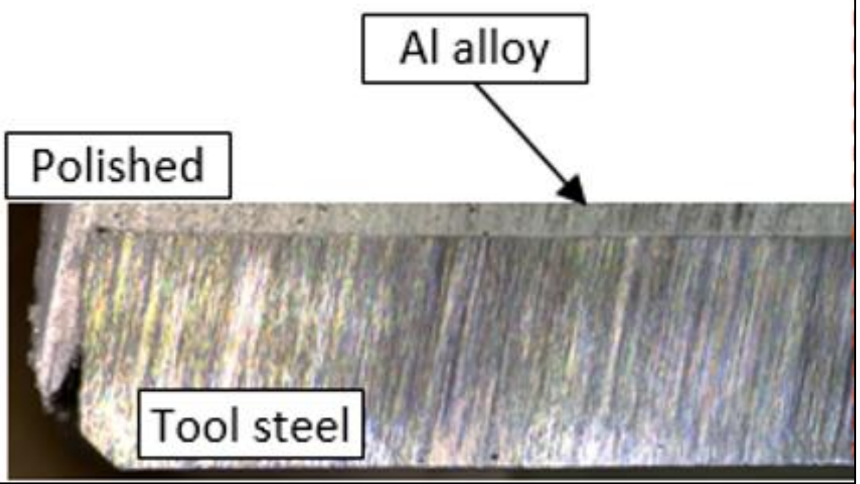
H13 Steel polished substrates

Solidified Al cylinders

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 current results
  - We saw less adhesion/reaction after 15 minutes for the rougher (600 mesh)

Ra = 0.07μm

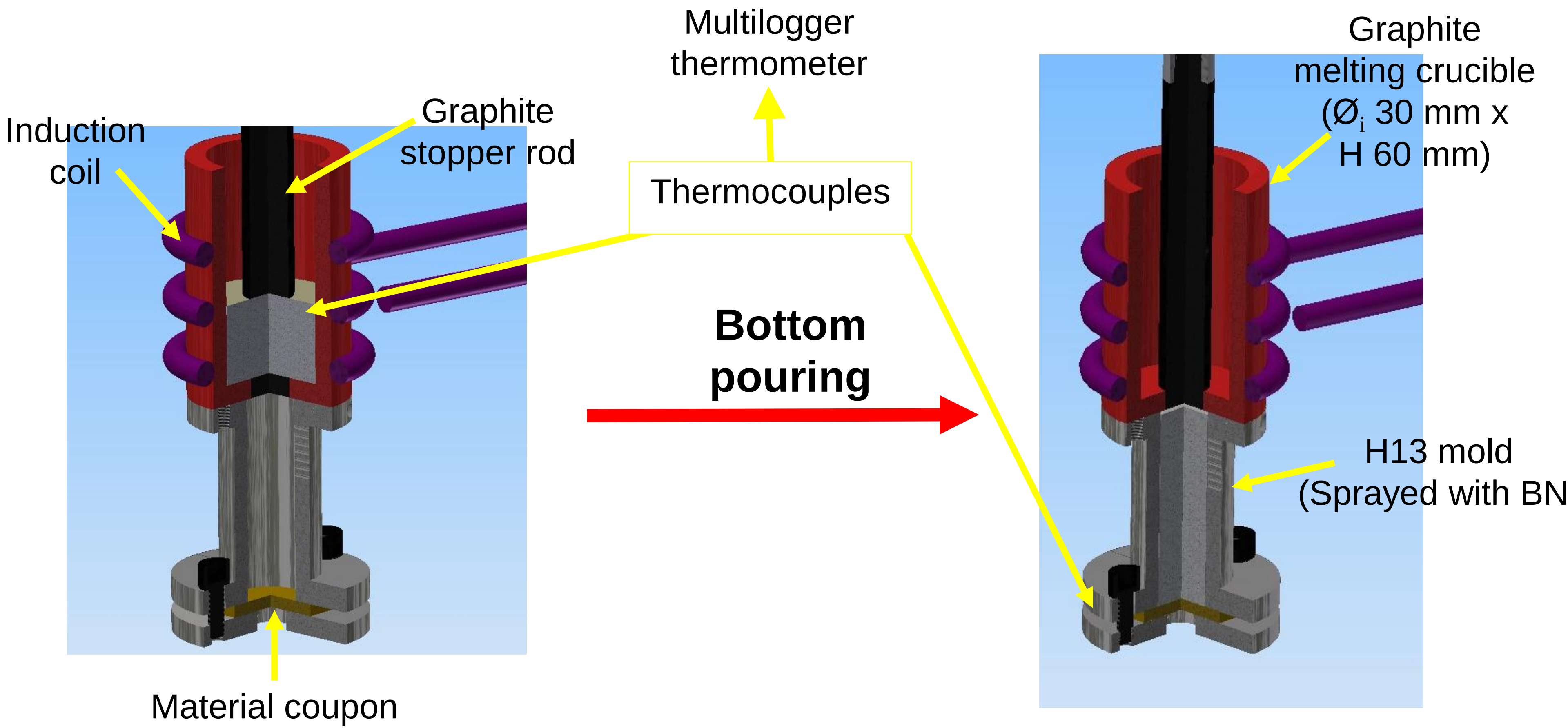


Ra = 0.4μm

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

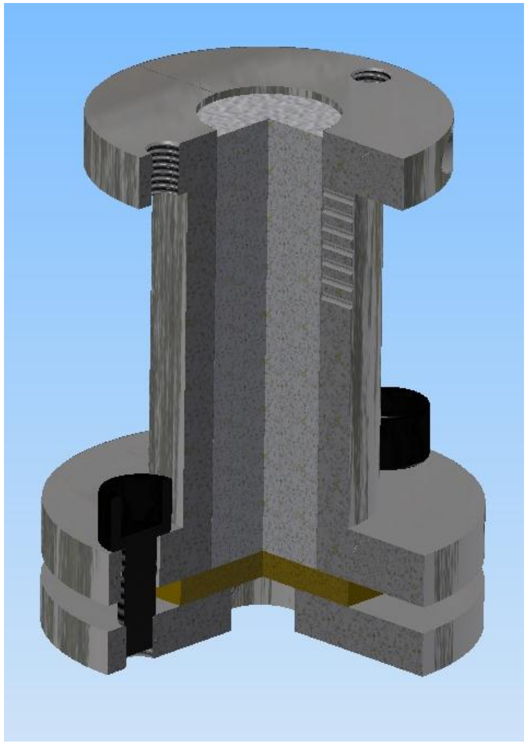
Further Improvements to Adhesion Test

- Eliminate the graphite mold
  - Use of a H13 mold (sprayed with BN)



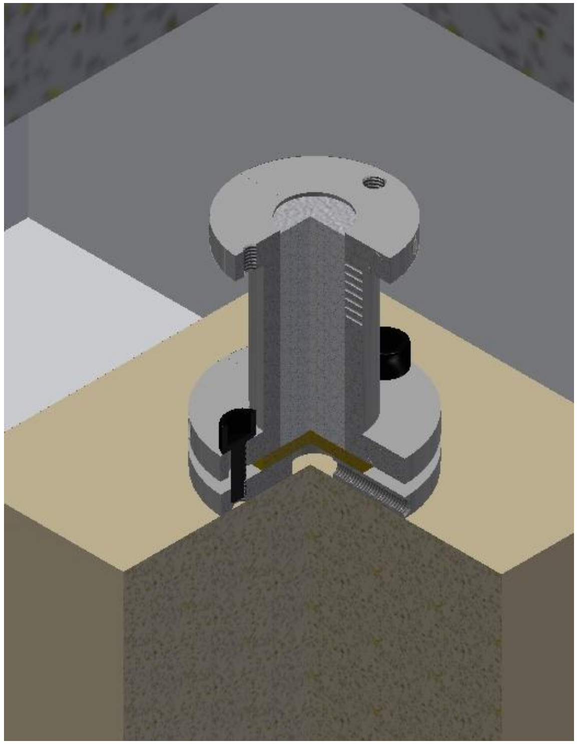
Modification of Test Apparatus

- Aluminum bottom poured into H13 mold
- Placed into resistance furnace pre-heated to 700ºC
- Different holding times at 700 ºC
  - Avoids disintegration of graphite mold



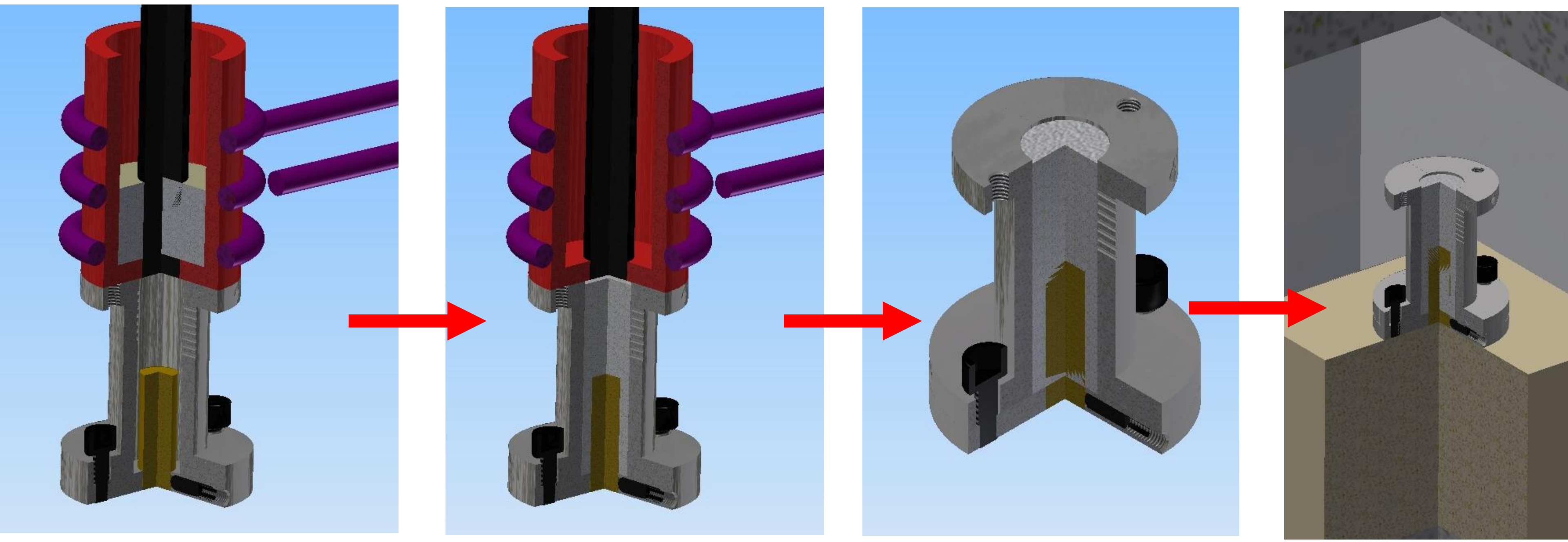
Resistance furnace at 700 ºC

Holding time



Different sample geometry

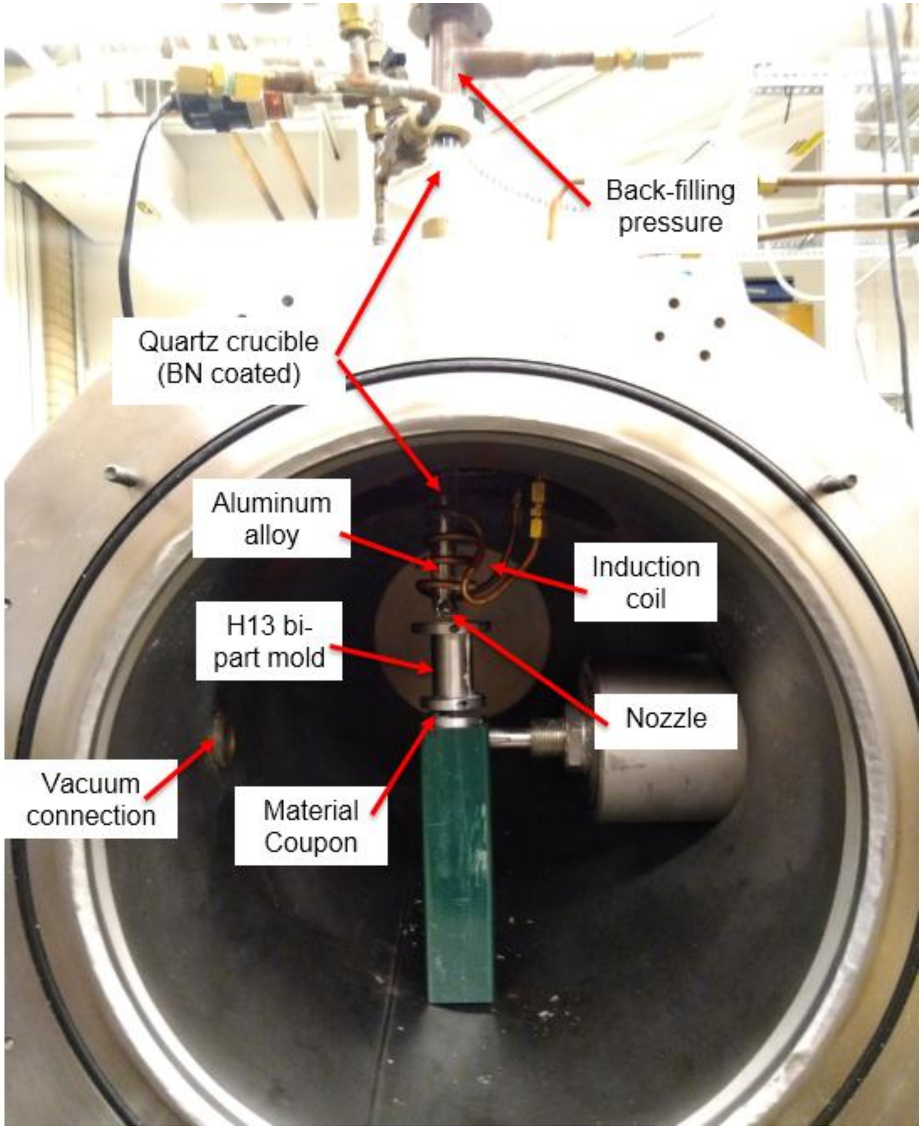
- Can use same procedure for testing Ø3/8” rods



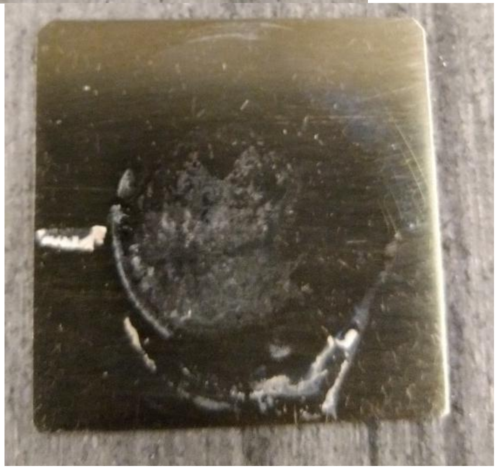
- Different sample geometry possible
  - 1”x1” flat plate
  - Ø3/8” rod

Test in Controlled Atmosphere

- Continue using bottom pouring concept
- Reduce oxide formation by melting in controlled atmosphere
- Higher injection speed of molten aluminum into mold
  - Better simulation of the die casting process
- Pre-heating of substrate possible
  - Less oxidation of substrate due to inert gas



1st Test in Controlled atmosphere



PVD Coated Samples

- Range of PVD coatings have been obtained
  - 1"x1" flat plates (3 samples) and 3/8" diameter rods (2 samples)

surface finish

Coating	Finish	Supplier	Tested During Lube Free project
TiCN	as-deposited	Supplier #2	NO
	Post treatment	Supplier #2	NO
AlCrN	as-deposited	Supplier #2	YES
	Post treatment	Supplier #2	YES
WC+C	as-deposited	Supplier #3	NO
ZrN	as-deposited	Supplier #2	NO
CrN	as-deposited	Supplier #1	YES
CrC	as-deposited	Supplier #1	NO
MoN	as-deposited	Supplier #1	NO
TaN	as-deposited	Supplier #1	NO
VC	as-deposited	Supplier #1	NO
AlCrCN	as-deposited	Supplier #2	NO
TiAlSiN	as-deposited	Supplier #2	NO

- Different coating compositions and post-treatments will be evaluated.

Summary & Conclusions

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

Summary & Conclusions

- A further improvement in the test apparatus has been made to avoid disintegration of the graphite mold
  - Next tests will be performed using this setup.
- A number of PVD coatings have been deposited onto H13 flat substrates (1”x1”) and 3/8” diameter rods
  - These will be tested in the near future

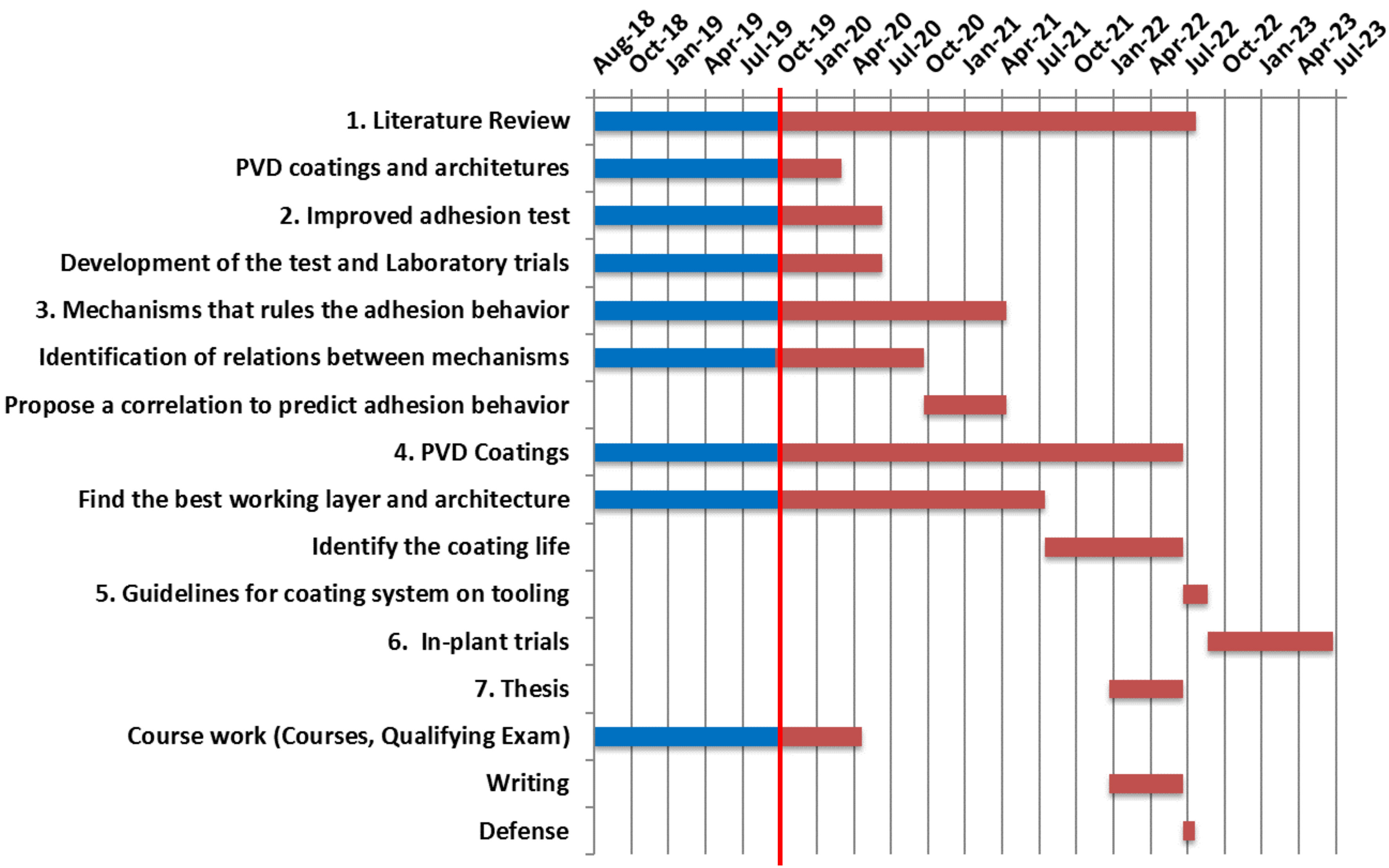
Future Work

- Literature review
  - Characterize PVD coatings currently used by die casters and other industries
  - Characterize chemical interactions between liquid metals and solid materials
  - Examine mechanisms involved with wetting of solids by liquid metals
- Experimental work
  - Characterization of PVD coatings deposited onto H13 substrates
  - Adhesion test using the PVD coated samples
- Characterization of aluminum adhesion tested samples
  - Examine the phases formed at the interface between the solidified aluminum and the tested substrates

Challenges & Opportunities

- The PVD equipment at Mines is currently inoperative due to loss of controlling software. Manual control is being attempted.
- Targeting an improved laboratory adhesion test that simulates the commercial die casting process
  - The main focus has been on trying to minimize the influence of aluminum oxide on the test
  - Controlled atmosphere, improved control of substrate temperature and performing multiple sequential tests are additional goals
- Also attempting a different approach other than PVD
  - Thermal sprayed samples will also be tested in the aluminum adhesion test.

Progress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1. Literature Review
PVD coatings and architetures
2. Improved adhesion test
Development of the test and Laboratory trials
3. Mechanisms that rules the adhesion behavior
Identification of relations between mechanisms
Propose a correlation to predict adhesion behavior
4. PVD Coatings
Find the best working layer and architecture
Identify the coating life
5. Guidelines for coating system on tooling
6. In-plant trials
7. Thesis
Course work (Courses, Qualifying Exam)
Writing
Defense

Acknowledgement

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Thank you!

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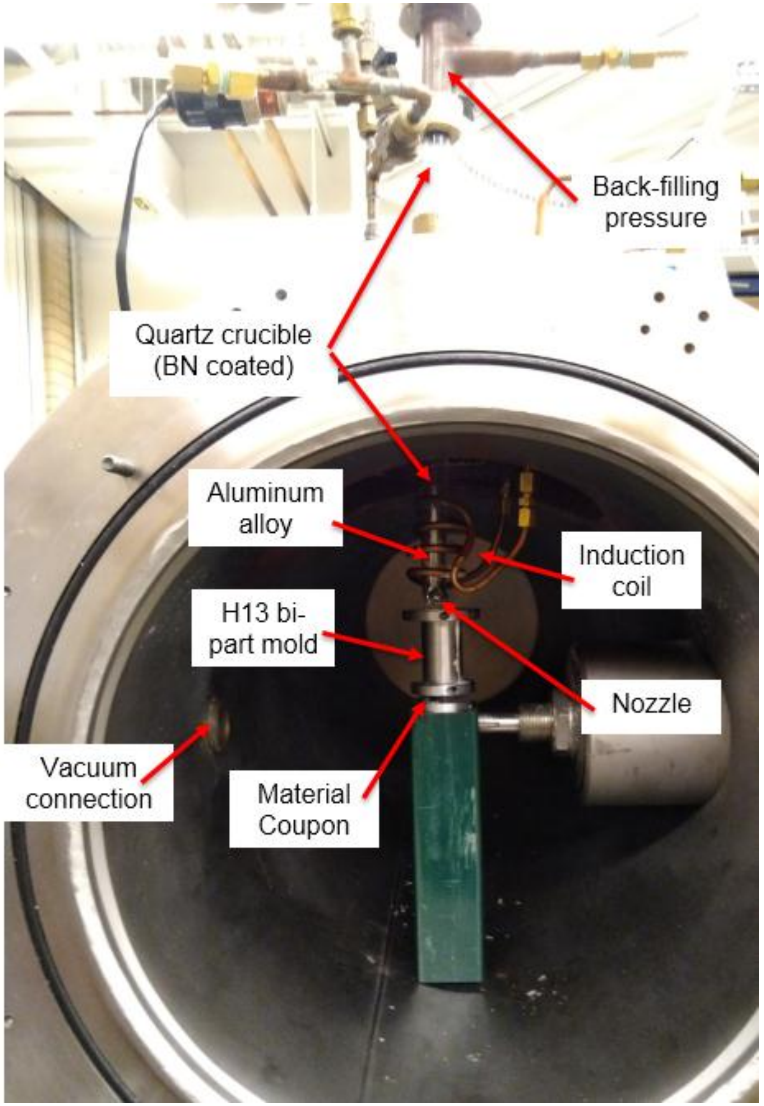

Achievement

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

Significance and Impact

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

Research Details

- 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.

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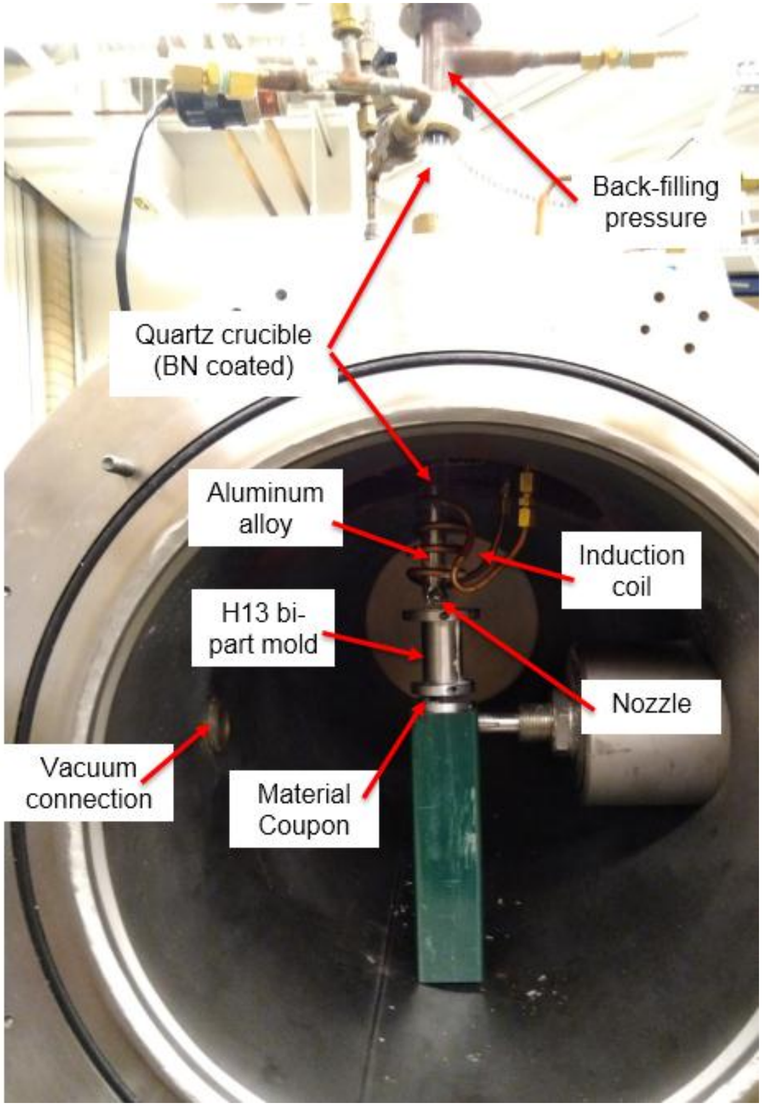

Program Goal

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

Approach

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

Benefits

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