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

Fall Meeting

October 13^{th} – 15^{th} 2020

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

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- Student: Nelson Delfino de Campos Neto (Mines)
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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 coated samples

Metrics

Description	% Complete	Status
1. Literature review and development of an improved adhesion test that simulates the die casting process	55%	●
2. Identification of the mechanisms that controls the adhesion behavior.	50%	●
3. Identification of a working layer coating that avoids molten aluminum soldering and adhesion.	50%	●
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

- Reducing or eliminating lubricant spray will:
  - Significantly **improve the quality** of the die castings
    - Reduce gas porosity and scrap
    - Allow castings to be 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 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 coatings on tooling

Testing Procedure

- Over the past 12 months or so
  - Have examined a number of different test procedures, to test soldering between molten aluminum and PVD coatings
    - Melting method (electric resistance versus induction)
    - Casting mold material (graphite versus metallic)
    - Pouring versus injecting liquid aluminum into casting mold
    - Preheating casting mold (RT versus 500^{o}C)
- Have settled on the following practices

Testing Procedures

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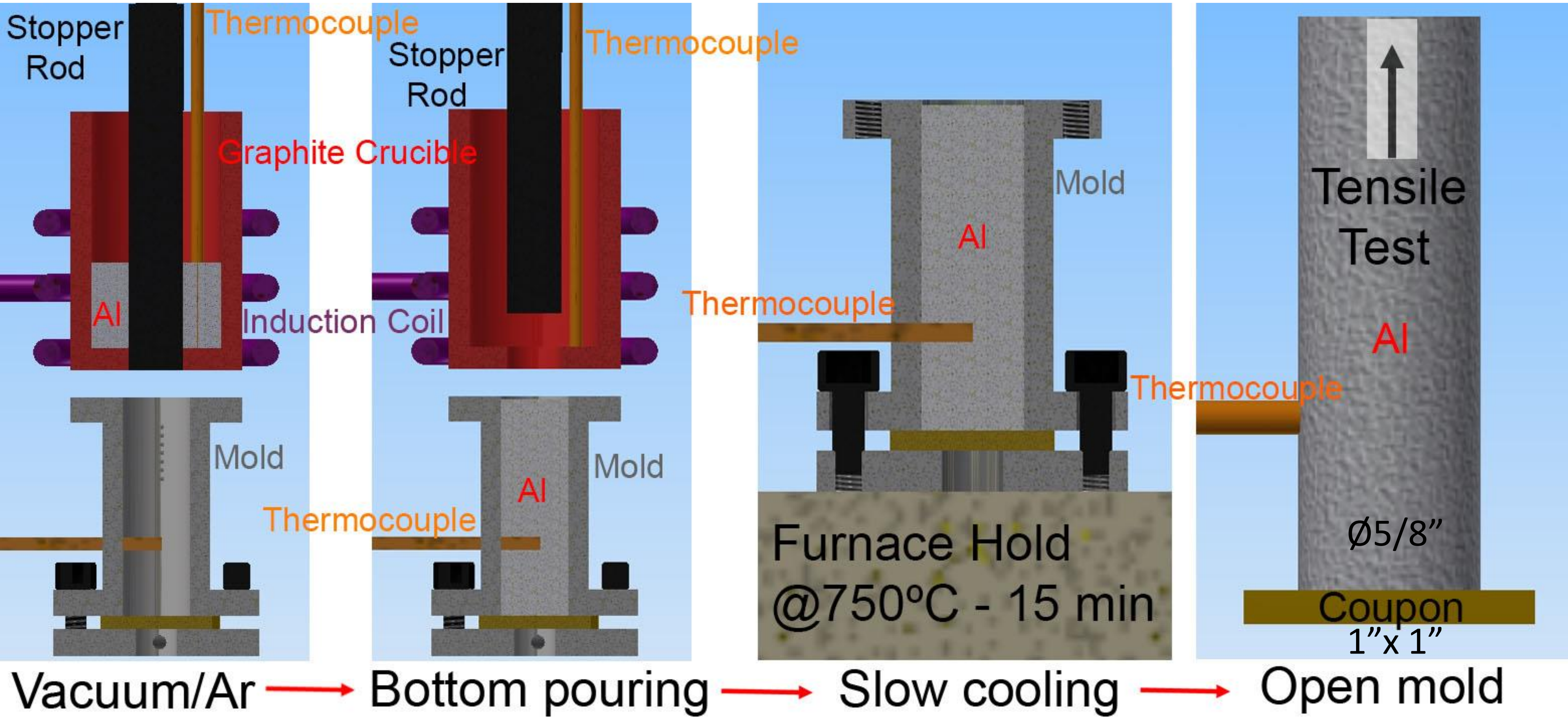
- Melt using induction power
  - Melting is very quick
  - Oxidation of aluminum is minimized
- Melt under controlled atmosphere
  - Further minimizes oxidation of aluminum
- Bottom pouring of liquid aluminum from melting crucible
  - Any oxide that does form is left behind in crucible
- Use of metallic mold
- Transfer casting mold and test coupon to pre-heated furnace
  - Allows time for reaction between molten aluminum and coupon/coating

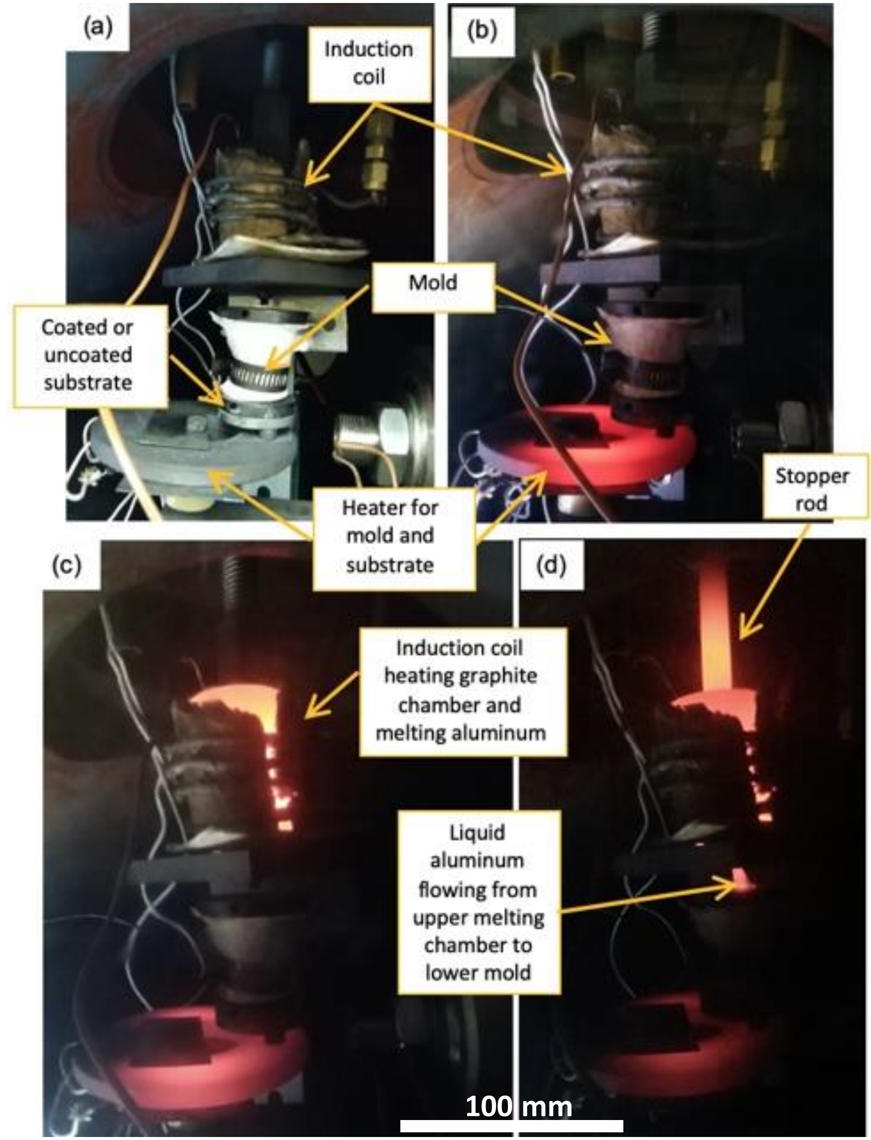
Schematic of the Test



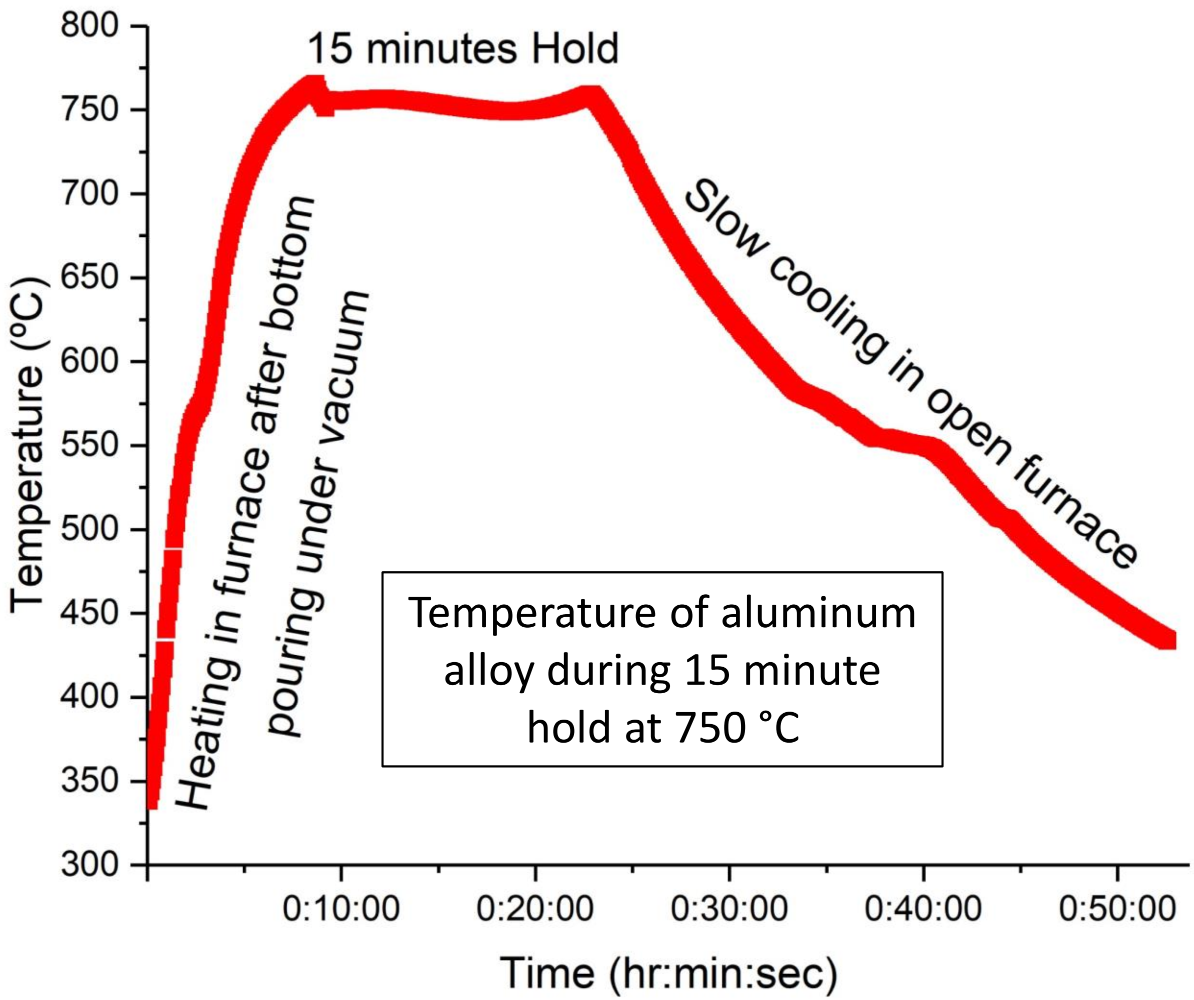
- Cleaner molten aluminum (prevents oxide) in contact with material coupon

Test in Controlled Atmosphere

- Fill the mold by bottom pouring in controlled atmosphere
- Transfer to pre-heated furnace at 850 °C
  - Holding Temp: ~750 °C
  - 15 minutes
- Does aluminum solder to substrate?
- => Tensile test to rank coatings



Temperature Profile



Materials Tested

- **Tested five AlCrN coatings from two suppliers**

Supplier	Coating
1	AlCrN
1	AlCrN + PDPT*
1	AlCrCN
2	AlCrN
2	AlCrN + ion nitriding
--	None (bare H13)

***Post deposition polishing treatment**

Molten Aluminum Test (MAT)

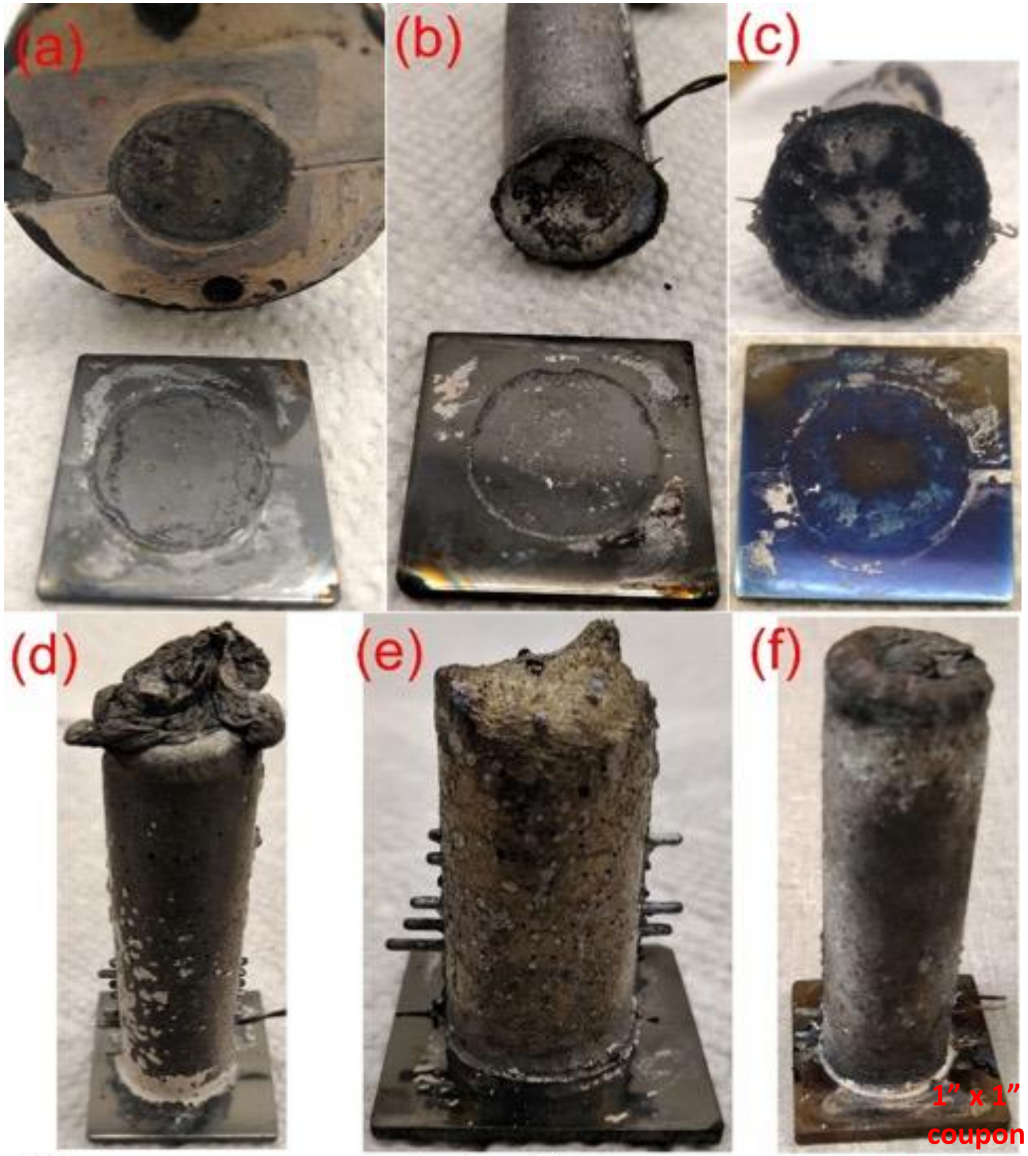
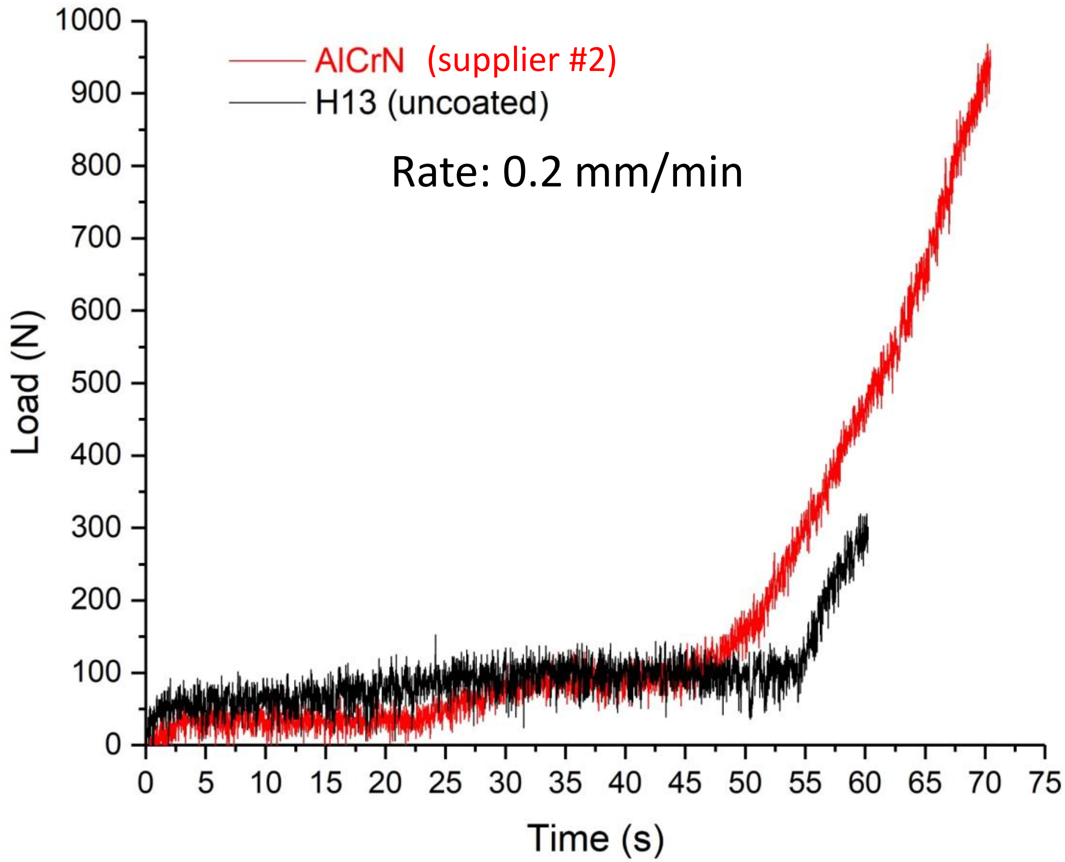
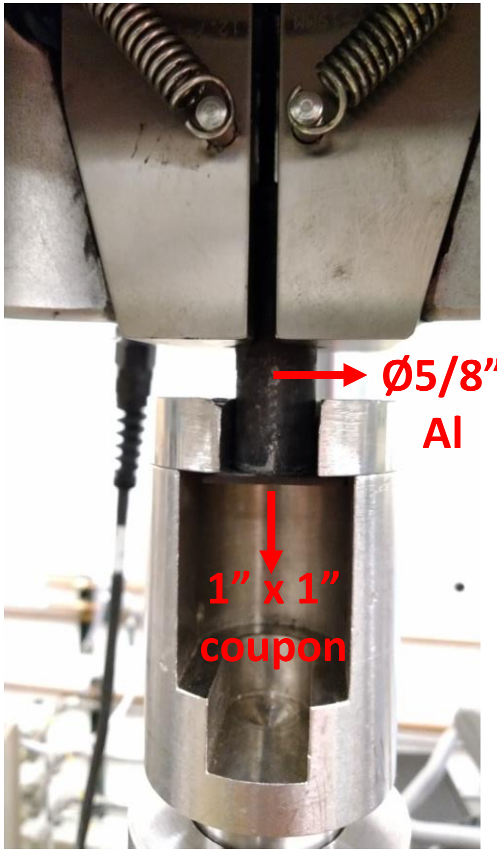


Fig.	Supplier	Coating
a	1	AlCrN
b	1	AlCrN + PDPT
c	1	AlCrCN
d	2	AlCrN
e	2	AlCrN + ion nitriding
f	--	None (bare H13)

Tensile Test





- Similar results have been reported in previous work [1]:
  - It was suggested that cracking between the solidified aluminum and the bare H13 steel occurring during cooling was the reason for the lower load required to separate the adhered aluminum from the bare H13 steel substrate

AlCrN Coatings

- We are seeing a range of behavior in the Molten Aluminum Test from the different varieties of AlCrN coatings
  - Differences in behavior between coatings are often also observed in commercial die casting operations
- Performed a characterization of the coatings
  - To better understand their differences
- Examined five coatings from two suppliers

Testing Performed

Property/Performance	Test
Adhesion to molten Al	MAT
Coating thickness	Calo test, SEM, TEM
Surface roughness	Profilometry
Adhesion to substrate	Scratch test, VDI3198
Wear rate, coefficient of friction	Pin-on-disk
Chemical analysis	X-ray photoelectron spectroscopy (XPS)
Coating microstructure	SEM/TEM

Coating Thickness

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Supplier	Coating	Thickness (μm)		
		SEM	Calo	TEM
1	AlCrN	1.2	1.0	--
1	AlCrN + PDPT	2.1	2.0	2.4
1	AlCrCN	1.3	1.4	--
2	AlCrN	6.6	8.5	--
2	AlCrN + ion nitriding	8.4	8.9	--

- Coatings from supplier 2 are significantly thicker than coatings from supplier 1

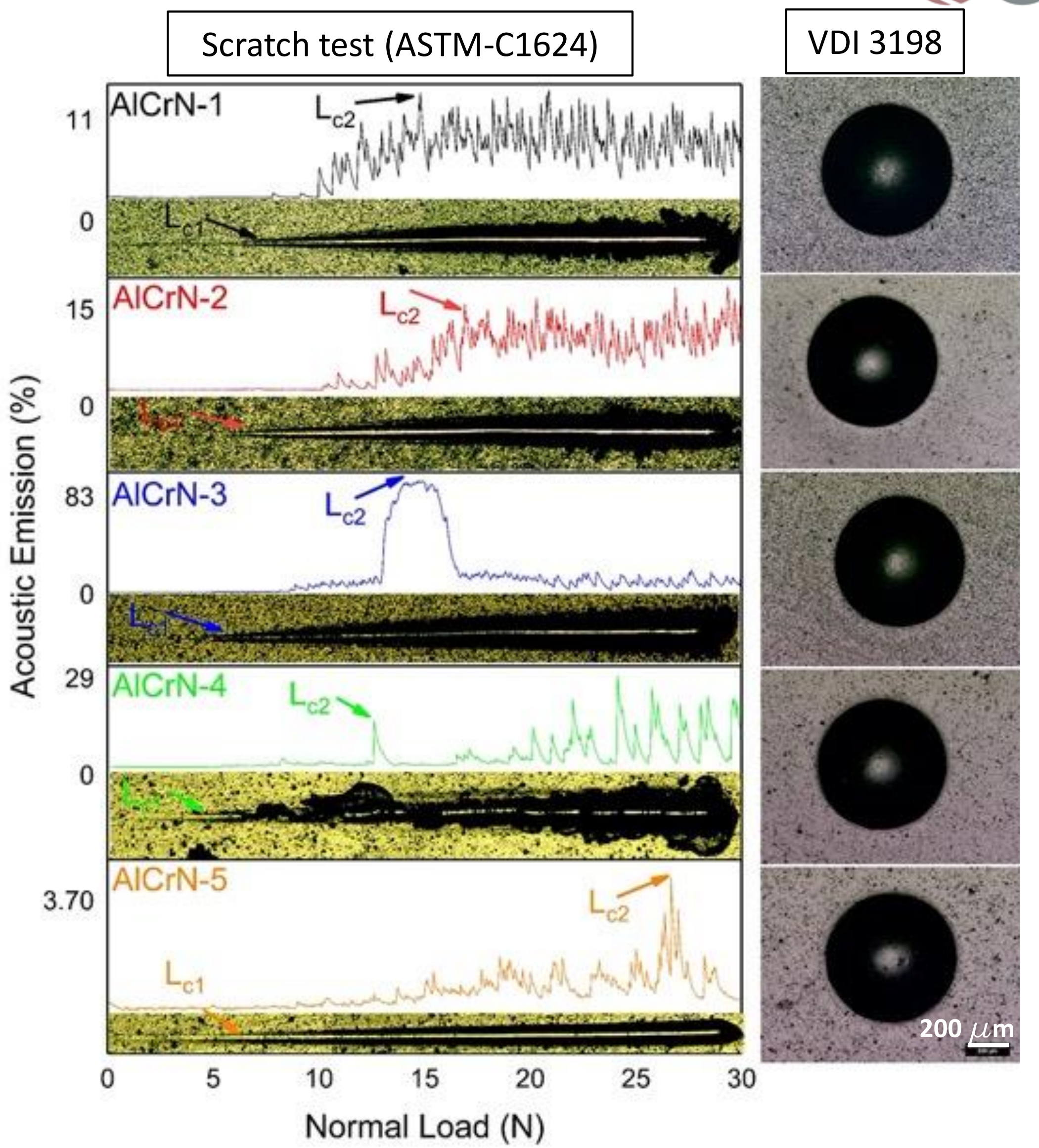
Chemical Composition

- Measured by XPS

Supplier	Coating	Composition (at. %)				
		Al	Cr	N	C	Ti
1	AlCrN	42.5	11.1	46.5	-	-
1	AlCrN + PDPT	40.4	12.2	47.5	-	-
1	AlCrCN	5.7	1.6	3.7	80.2	9.0
2	AlCrN	33.9	19.0	47.0	-	-
2	AlCrN + ion nitriding	**29.3**	**21.0**	**49.8**	**-**	**-**

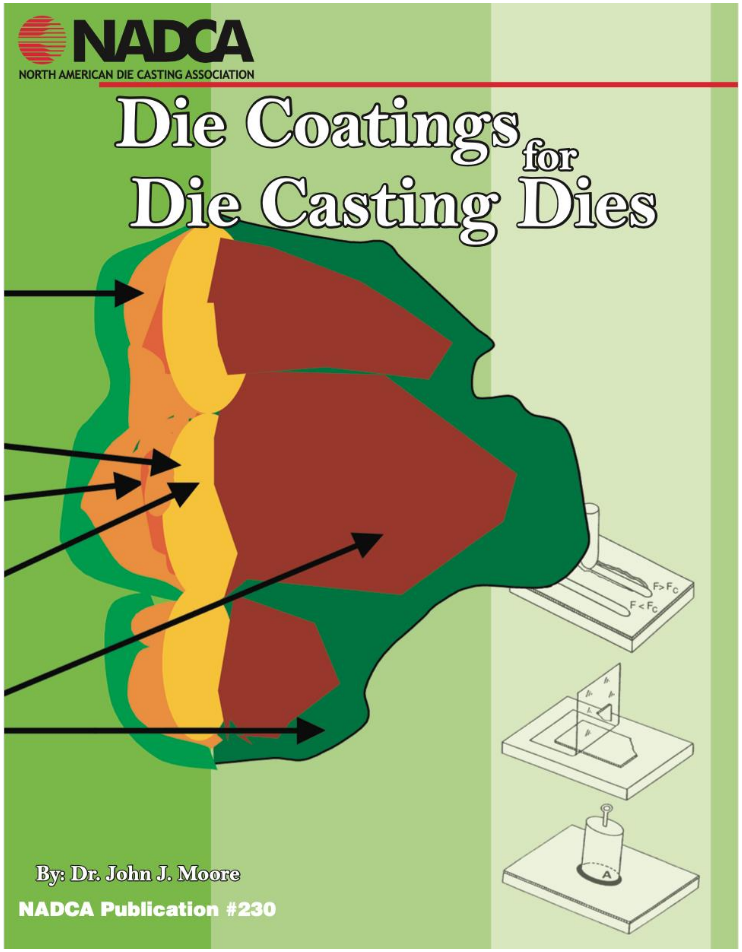
Scratch Test & VDI 3198

Sup.	Coating
1	AlCrN
1	AlCrN + PDPT
1	AlCrCN
2	AlCrN
2	AlCrN + ion nitr.



- Scratch test and VDI 3198 measure the quality of the coatings
- Tests shows that all five coatings have good cohesion
  - Quality of coating is good
- And good adhesion
  - Quality of bonding to substrate is good

Wear and Oxidation Resistance

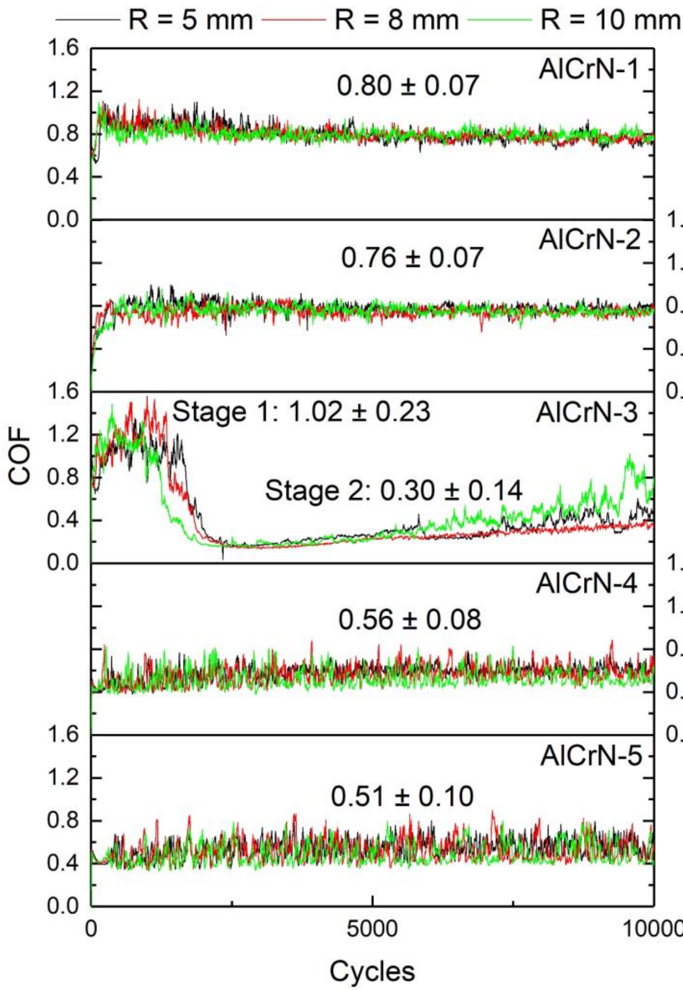


- The NADCA book on PVD coatings described some important properties that a coating should have
  - Wear resistance
  - Oxidation resistance
- Current AlCrN coatings
  - Measured wear resistance using pin-on-disk
  - Measured air oxidation resistance using TGA equipment

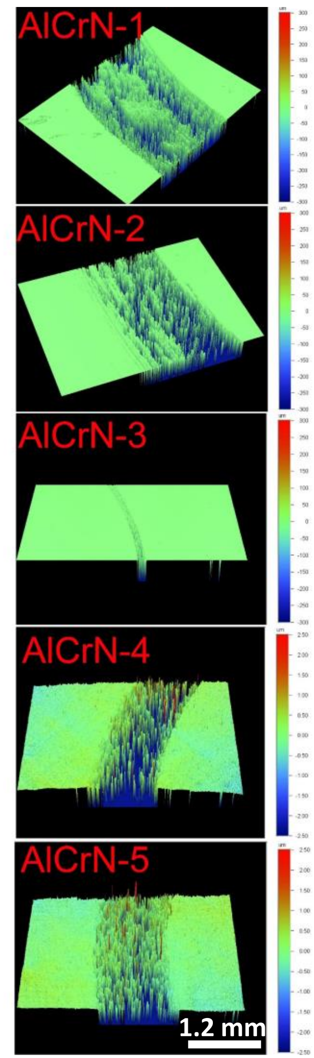
Aluminum Pin-on-Disk Wear Test

Sup.	Coating
1	AlCrN
1	AlCrN + PDPT
1	AlCrCN
2	AlCrN
2	AlCrN + ion nitr.

Coefficient of friction versus distance

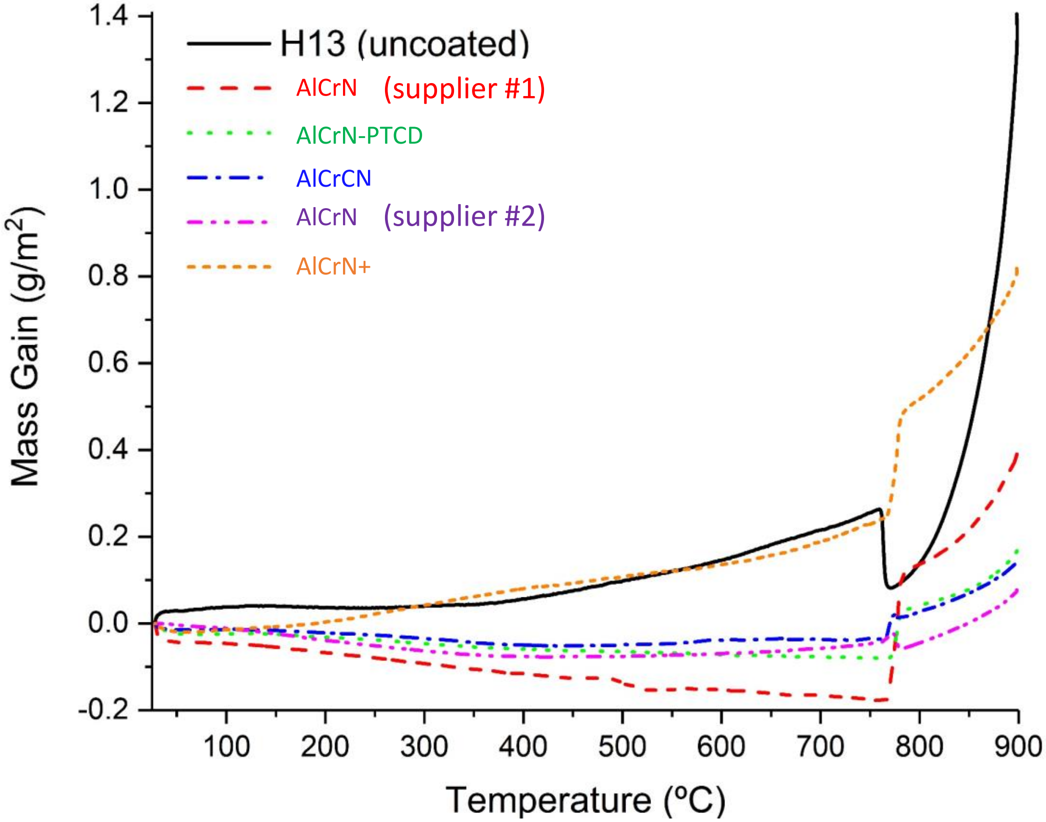


Wear track in coating



- Wear track for AlCrCN coating is significantly smaller and has better wear resistance
- Supplier #1 AlCrN and AlCrN-PDPT coatings have the same COF ~ 0.80
- AlCrCN coating have 2 different stages:
  - 1st high COF ~1.02
  - 2nd low COF ~0.30
- Supplier #2 AlCrN and AlCrN + ion nitriding coatings have the same COF ~ 0.55

Air Oxidation Resistance



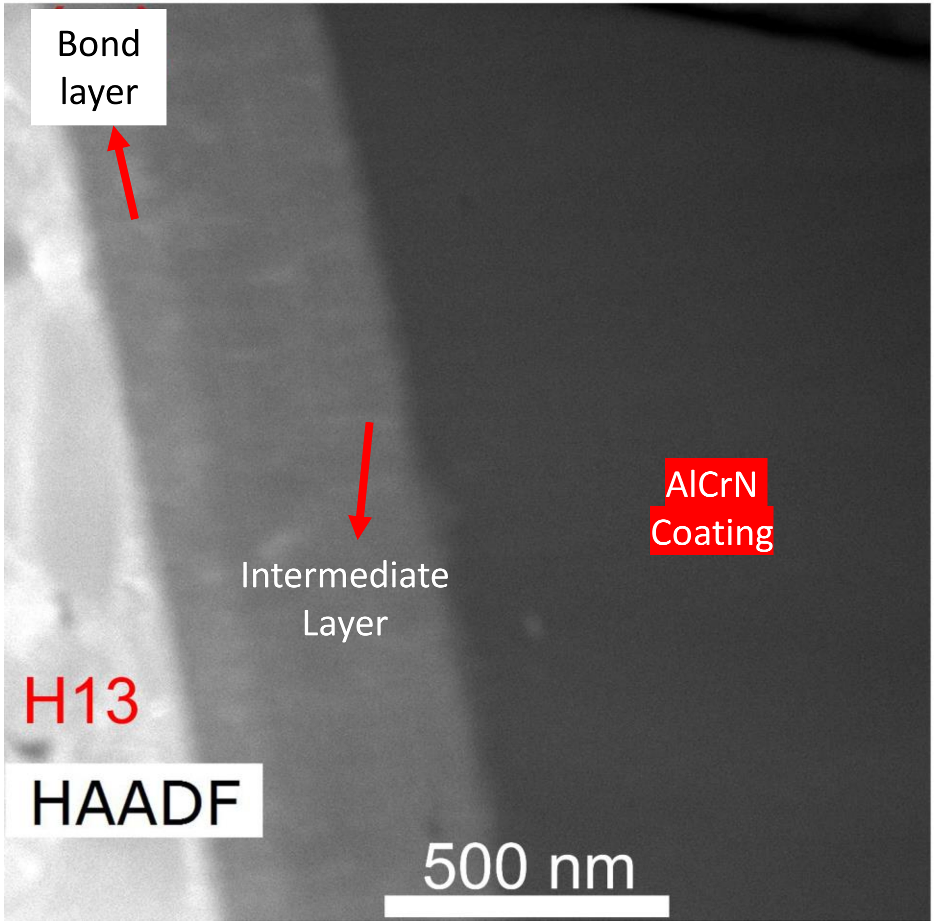
- All coatings have a higher air oxidation resistance than uncoated H13
- AlCrN-PDPT, AlCrCN and AlCrN (#2) have higher oxidation resistance

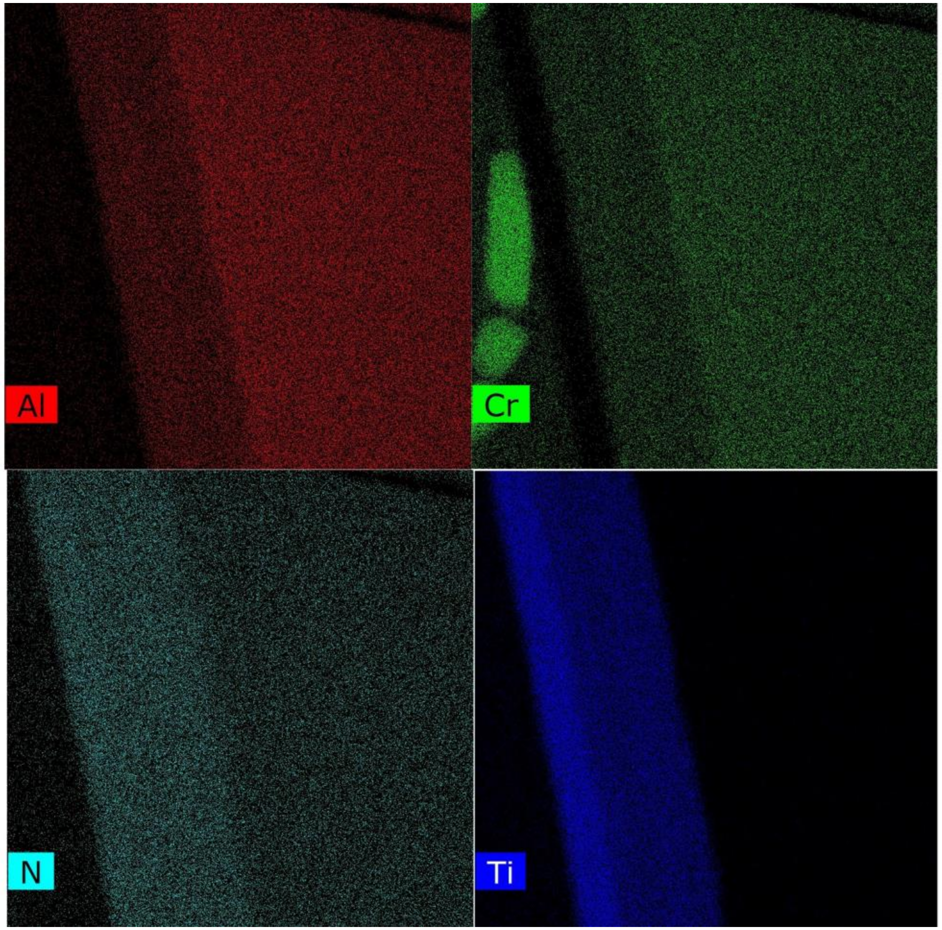
Coating Structure

- Based on analysis in the SEM and TEM

Supplier	Coating	Adhesion Layer	Coating Structure
1	AlCrN	Ti/TiN	Single layer
1	AlCrN + PDPT	Ti/TiN	Single layer
1	AlCrCN	Ti/TiN	Multi-layer
2	AlCrN	Cr/CrN	Single layer
2	AlCrN + nitriding	Cr/CrN	Single layer

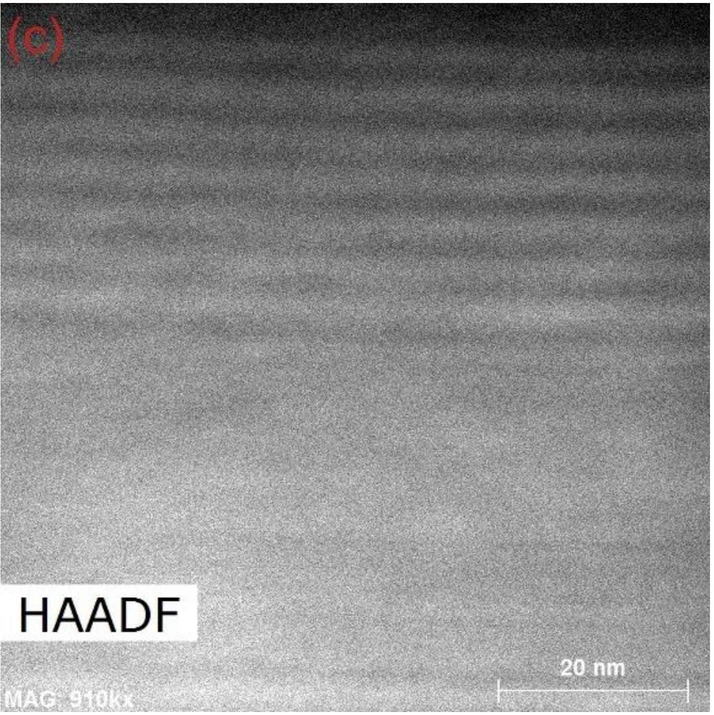
TEM - AlCrN-PDPT (Supplier #1)

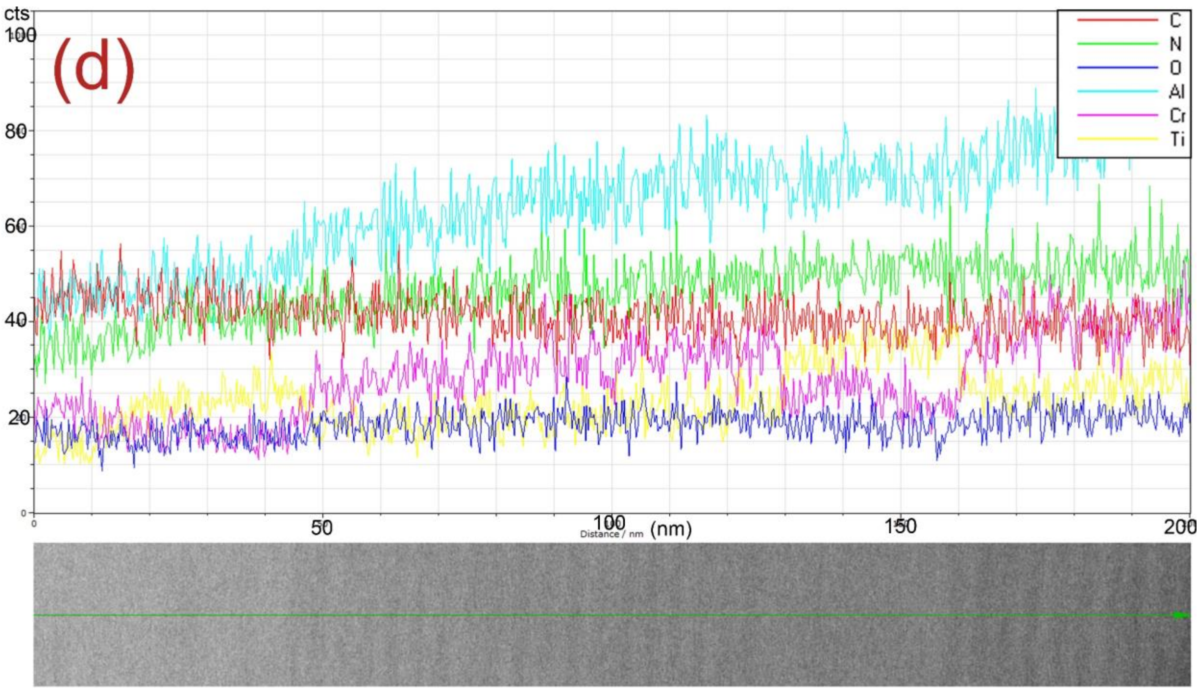




Substrate	Bond Layer	Intermediate Layer	Coating
H13 steel	Ti/TiN	TiAlCrN	AlCrN

TEM – AlCrCN (Supplier #1)





- Alternate layers in a nanolayer structure (80 nm thick)
  - Alternating layers with different chemical compositions with 2 nm thickness each
  - 35 nm thicker layer separating them

Summary & Conclusions

- An improved Molten Aluminum Test (MAT) has been developed
- Test involves the following parameters
  - Melting aluminum using induction power
  - Melting is performed under a controlled atmosphere
  - Aluminum is bottom poured from the melting crucible
  - Metallic mold is used
  - Casting mold and test coupon are transferred to pre-heated furnace
    - To allows time for reaction between molten aluminum and coupon/coating

Summary & Conclusions

- H13 and five different AlCrN coatings from two suppliers have been tested using the Molten Aluminum Test (MAT)
  - AlCrN and AlCrCN from supplier 1 exhibited no reaction
  - AlCrN-PDPT (also supplier 1) had a minor reaction at the surface
  - H13, AlCrN and AlCrN + ion nitriding from supplier 2 stuck
- Due to differences in adhesion behavior of the five AlCrN coatings
  - Characterization of the coatings has been performed
    - Coating thickness & microstructure
    - Wear rates & coefficient of friction
    - Chemical analysis & oxidation rates
  - Significant differences between coatings were identified
- Possibly a specification needs to be developed for coatings for die casting inserts
  - Similar to AMS2444A for TiN coatings

Future Work

- Literature review
  - Better understand the various PVD coatings currently used by die casters and other industries
  - Better understand the chemical interactions between liquid metals and solid materials
  - Examine and understand proposed mechanisms of solid-liquid metal interaction
- 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 substrate

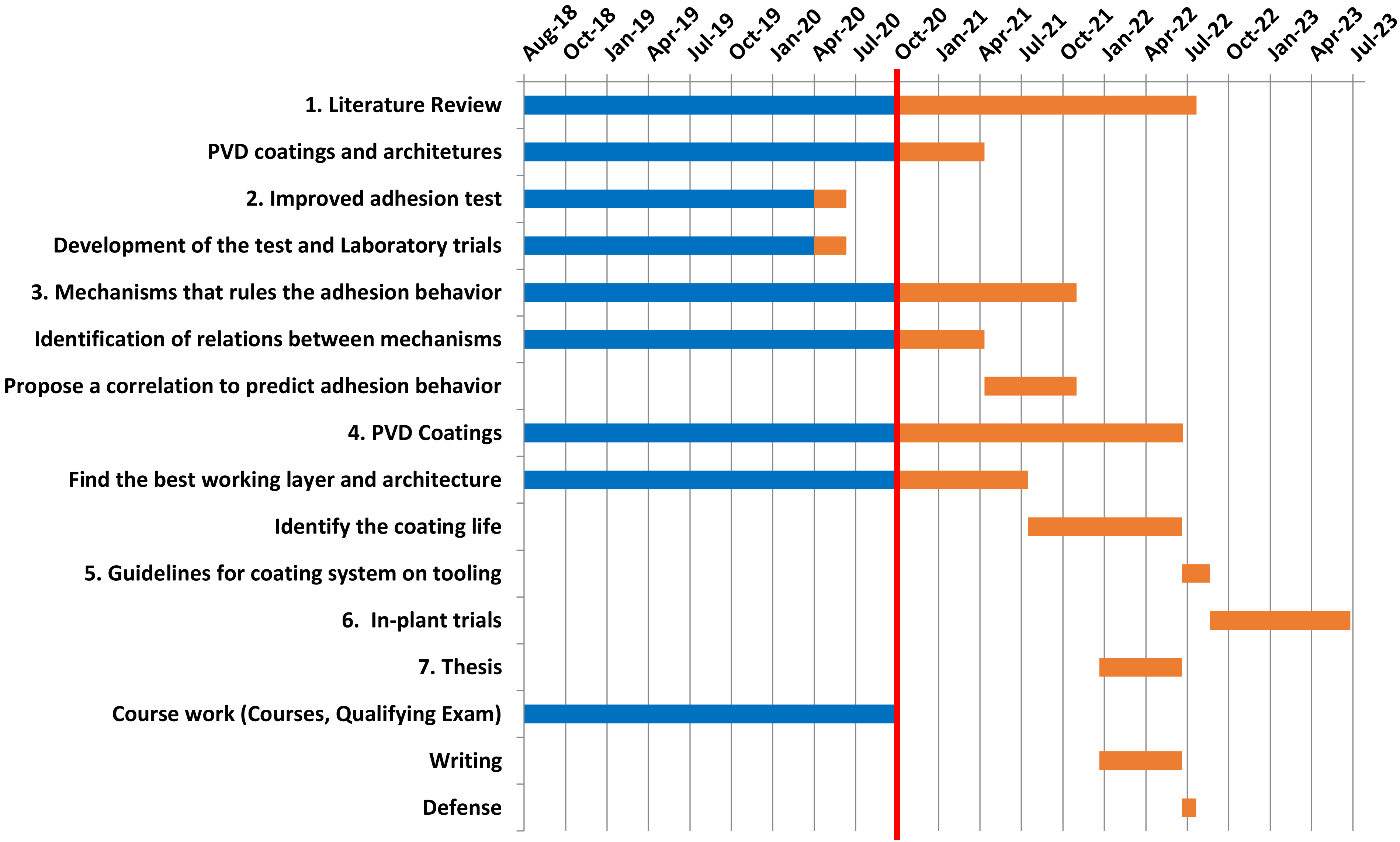
Future Work

- One approach being considered is the incorporation of transition metals into the AlCrN PVD coatings
  - Elements such as Mo, W and V form oxides at die casting temperatures
    - Around 500-600^{o}C (930^{o}F)
  - These oxides have a layered, shearable crystal structure
    - Similar to graphite and MoS_2
  - Reports from the literature suggest that these oxides can form on the surface of PVD coatings
    - Provide a lubricious surface layer
    - Further reduce friction during ejection of the casting from the die

Challenges & Opportunities

- The PVD equipment at Mines is in the process of being repaired
- Started to examine non-PVD coatings (e.g., thermal spray)
- Run industrial trials on selected coatings deposited on core pins at an automotive die casting plant
- Nelson will be 6 months at The Ohio State University during 2021
  - Run controlled tests in a laboratory size die casting machine (Buhler 250 ton)
  - Access to thin film capabilities
  - Access to higher resolution microscopy

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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References

[1] B. Wang, An Investigation of the Adhesion Behavior of Aluminum on Various PVD Coatings Applied to H13 Tool Steel to Minimize or Eliminate Lubrication During High Pressure Die Casting, PhD thesis, CSM, 2016.

[2] J.J. Moore, Die Coatings for Die Casting Dies, NADCA Publication #230, 2003.