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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

Spring Meeting

April 7th – 9th 2020

- Student: Nelson Delfino de Campos Neto (Mines)
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COLORADO SCHOOL OF MINES
IOWA STATE UNIVERSITY

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 coated samples

Metrics

Description	% Complete	Status
1. Literature review and development of an improved adhesion test that simulates the die casting process	45%	●
2. Identification of the mechanisms that controls the adhesion behavior.	30%	●
3. Identification of a working layer coating that avoids molten aluminum soldering and adhesion.	30%	●
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

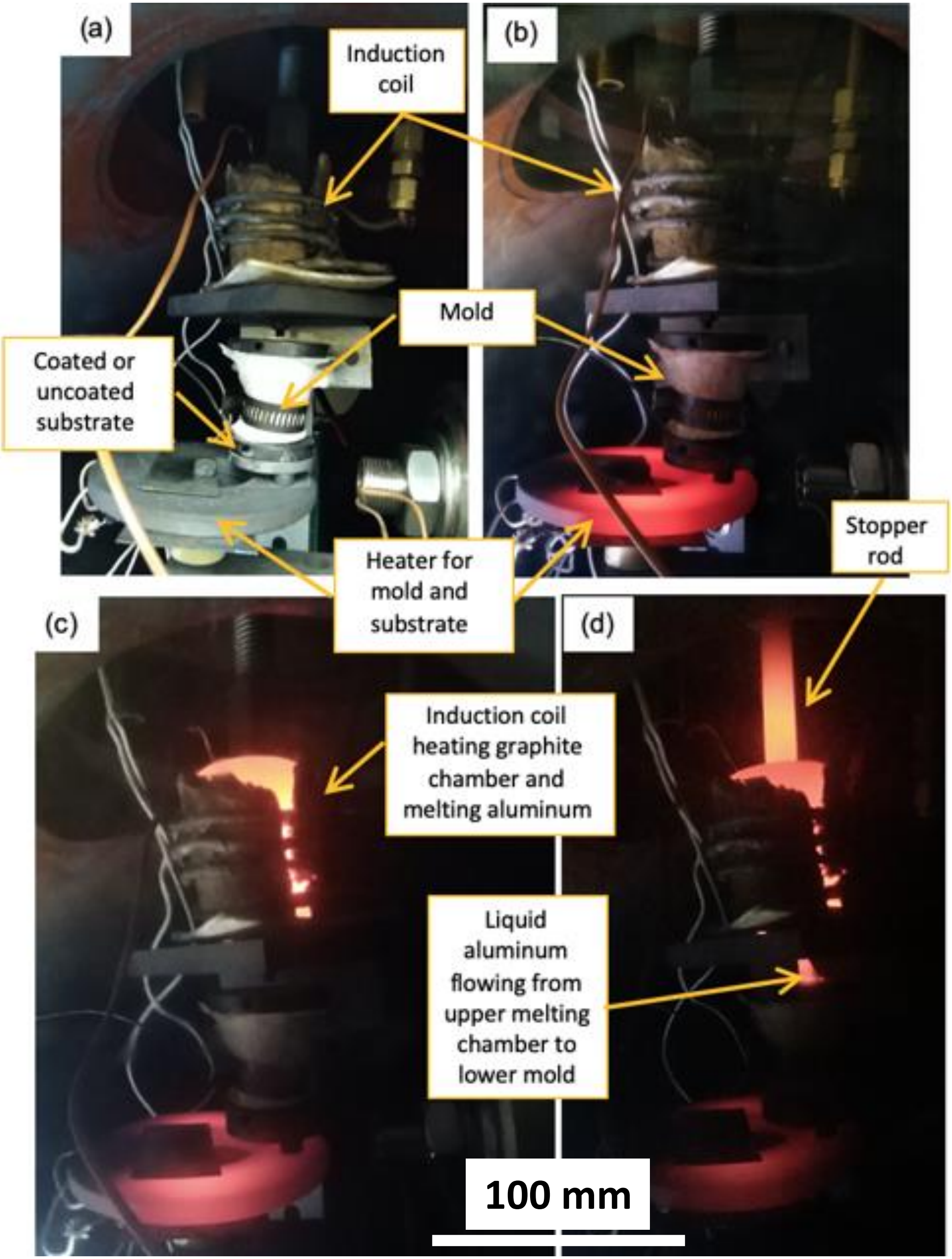
- Reducing or eliminating lubricant spray will:
  - Significantly **<u>improve the quality</u>** of the die castings
    - Reduce gas porosity and scrap
    - Allow castings to be used in higher performance applications
  - **<u>Reduce costs</u>**
    - Eliminate purchase costs for lubricants
    - Reduce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

Task 1.
Develop Improved Adhesion Test

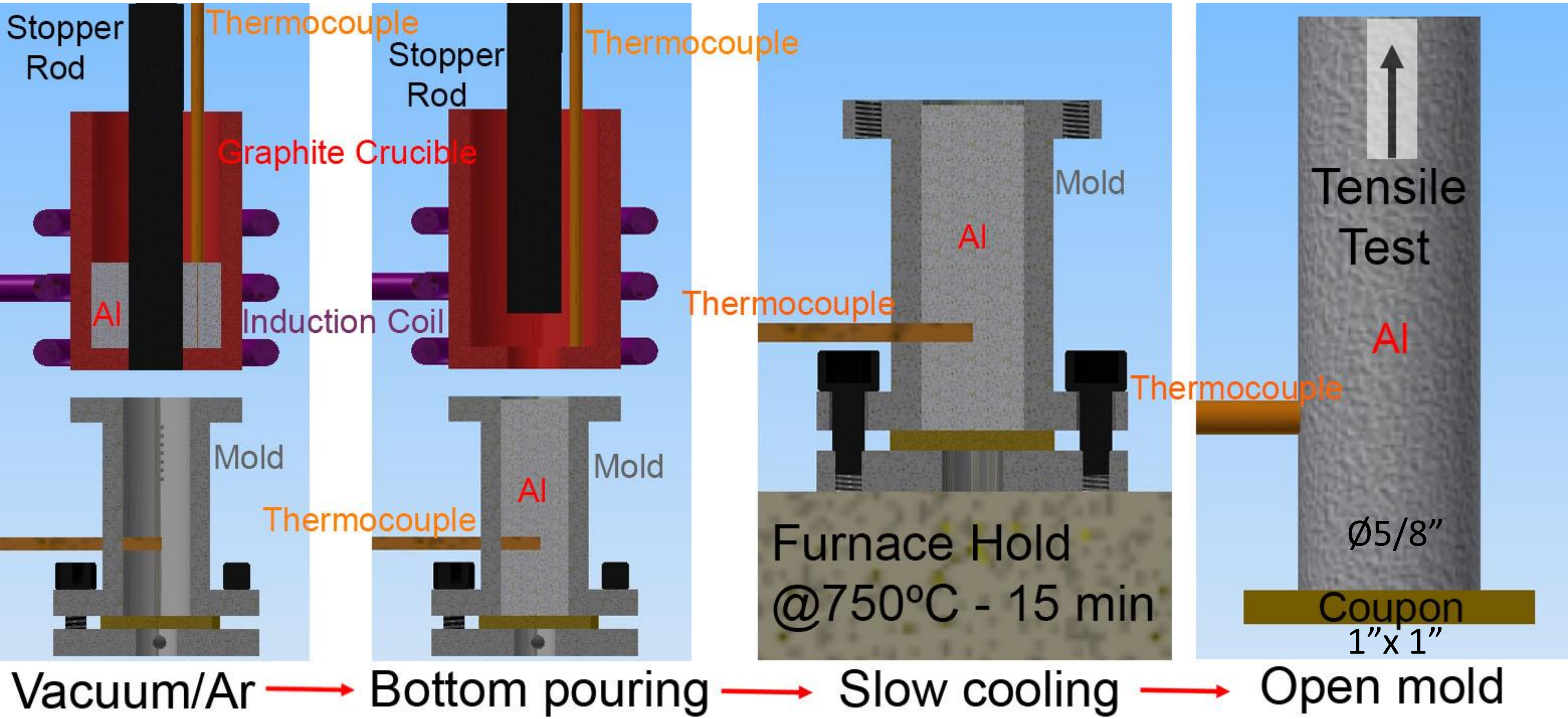
Test Performed in a Controlled Atmosphere



Test in Controlled Atmosphere

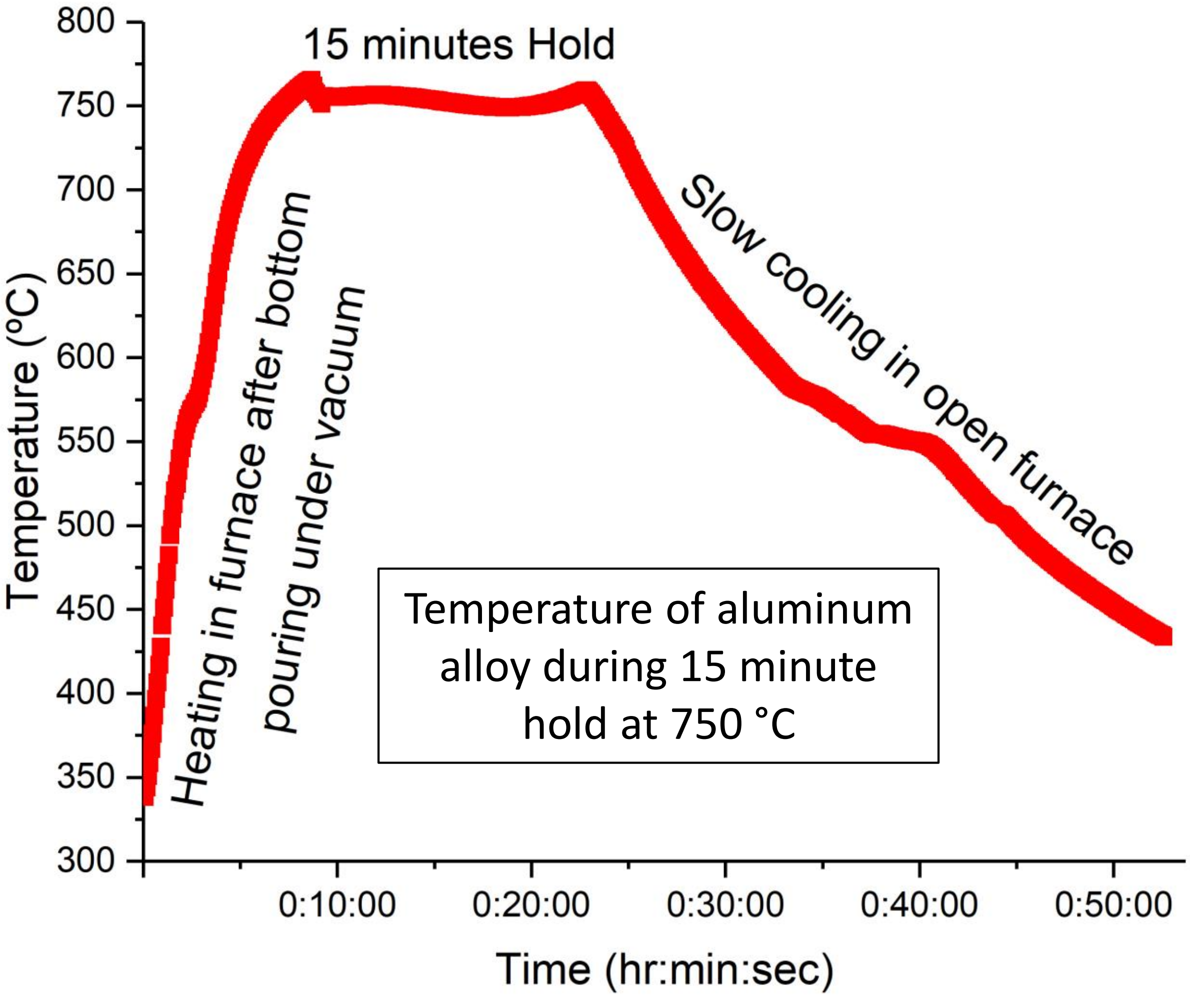
- After the test in C.A.:
- Transfer to pre-heated furnace at 850 °C
  - Holding Temp: ~750 °C
  - 15 minutes
- Does aluminum solder to substrate?
- => Tensile test to rank coatings

Schematic of the Test



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

Temperature Profile



Temperature of aluminum alloy during 15 minute hold at 750 °C

Test 1 and 2: Bare H13 Steel

- Polished H13 steel and Al 380 alloy
- Transferred to pre-heated furnace at 850 °C
  - Holding temp: ~750 °C
  - 15 minutes
- Aluminum soldered to bare substrate

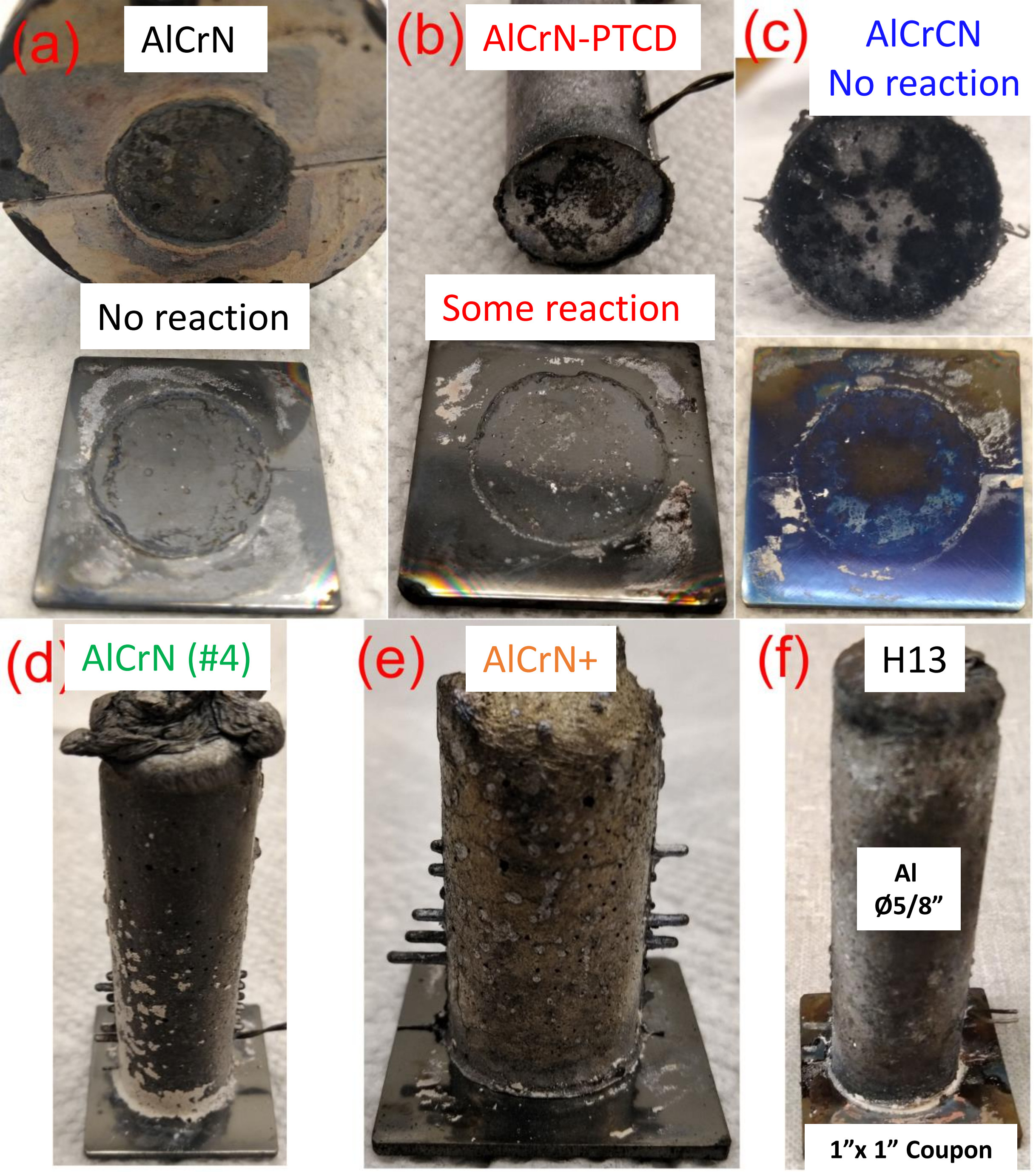
Tasks 2 & 3.
Identify Best Coatings

Commercial PVD Die Coatings

Substrate	Coating	Supplier	Tested During Lube Free project
H13	AlCrCN	Supplier #2	NO
	AlCrN	Supplier #2	YES
	AlCrN-PTCD	Supplier #2	NO
	AlCrN	Supplier #4	YES
	AlCrN+	Supplier #4	YES
	CrC	Supplier #1	NO
	CrN	Supplier #1	YES
	MoN	Supplier #1	NO
	TaN	Supplier #1	NO
	TiAlSiN	Supplier #2	NO
	TiCN	Supplier #2	NO
	TiCN-PTCD	Supplier #2	NO
	TiN	Supplier #1	NO
	VC	Supplier #1	NO
	WC+C	Supplier #3	NO
	ZrN	Supplier #2	NO

- 1”x1” flat plates and 3/8” diameter rods
- Different types of coatings: Nitrides, carbides, carbo-nitrides

PTCD: Post Treatment Class D



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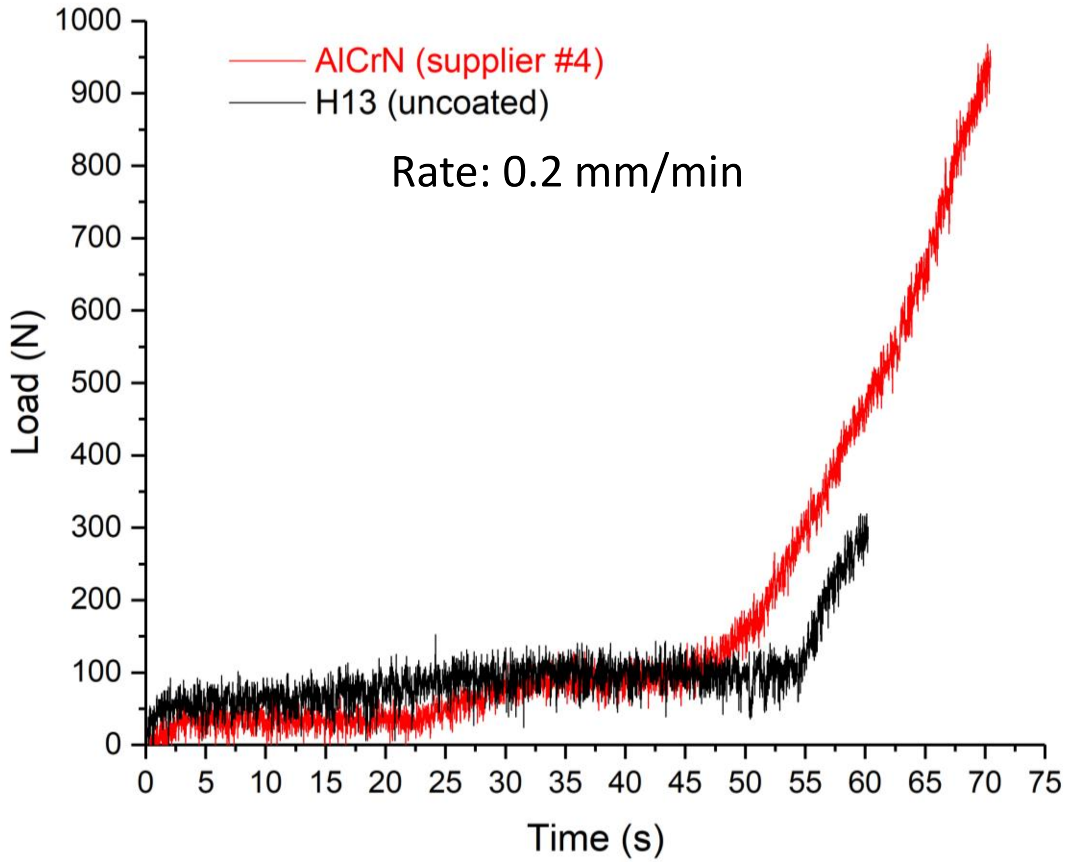
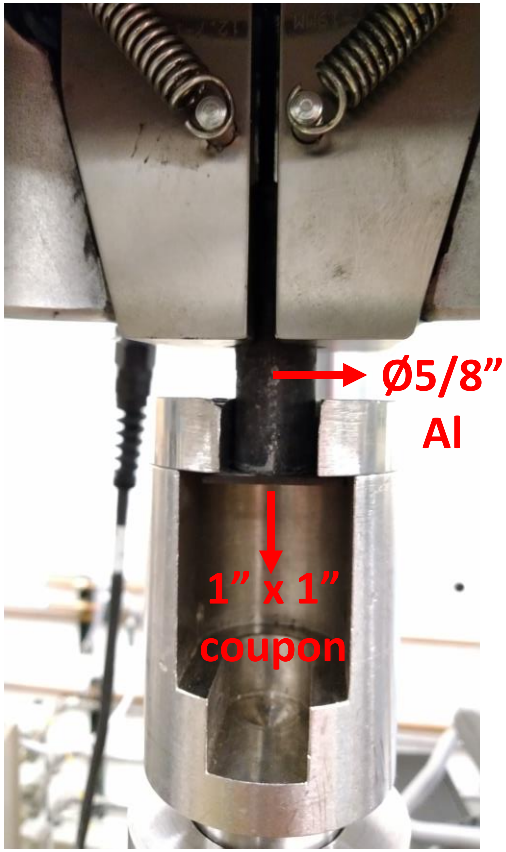
Aluminum Adhesion Test on AlCrN coatings

- No pre-heating of mold/coupon
- Transferred to pre-heated furnace at 850 °C
  - Holding temp: ~750 °C
  - 15 minutes

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

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Tasks 2 & 3.
Identify Permanent Coatings

Characterization of Five Different AlCrN Coatings

Coating Thickness

Coating	Thickness (µm)		
	SEM	Calo	TEM
AlCrN (supplier #2)	1.2	1.0	
AlCrN-PTCD	2.1	2.0	2.4
AlCrCN	1.3	1.4	
AlCrN (supplier #4)	6.6	8.5	
AlCrN+	8.4	8.9	

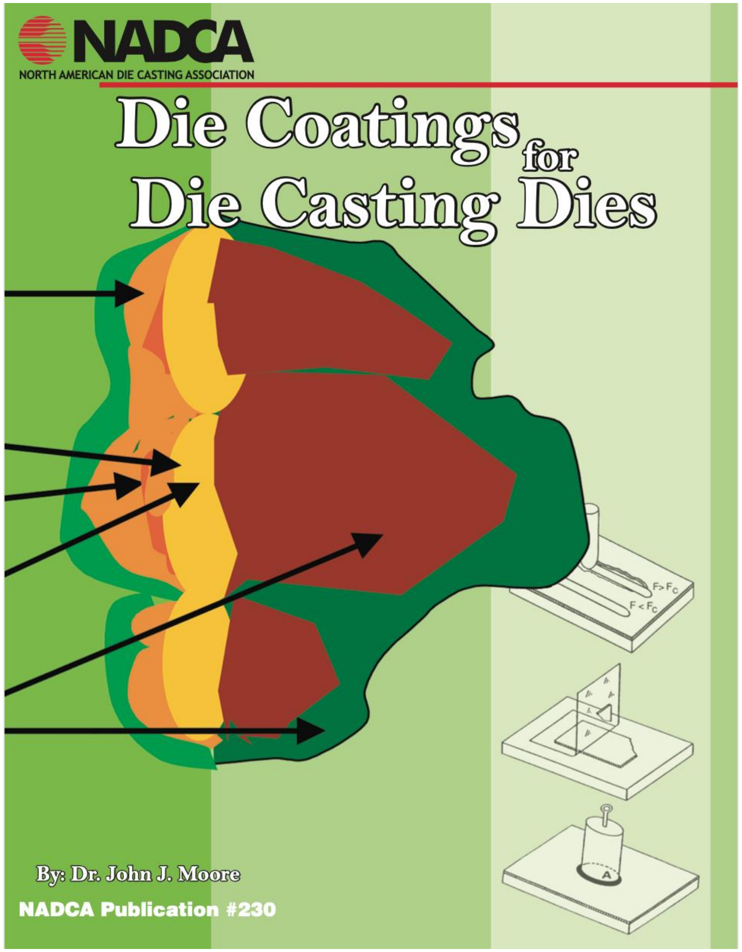
- Coatings from supplier #2 are relatively thin
- Coatings from supplier #4 are relatively thick
  - PVD coatings are typically between 1-10 µm
- All coatings have slightly different thickness

Composition Measured by XPS

Coating	Composition (at. %)				
	Al	Cr	N	C	Ti
AlCrN (supplier #2)	42.5	11.1	46.5	-	-
AlCrN-PTCD	40.4	12.2	47.5	-	-
AlCrCN	5.7	1.6	3.7	80.2	9.0
AlCrN (supplier #4)	33.9	19.0	47.0	-	-
AlCrN+	29.3	21.0	49.8	-	-

- AlCrN (sup#2) and AlCrN-PTCD have similar compositions
- AlCrCN has a different composition and contains Ti
- AlCrN (sup#4) and AlCrN+ have similar compositions

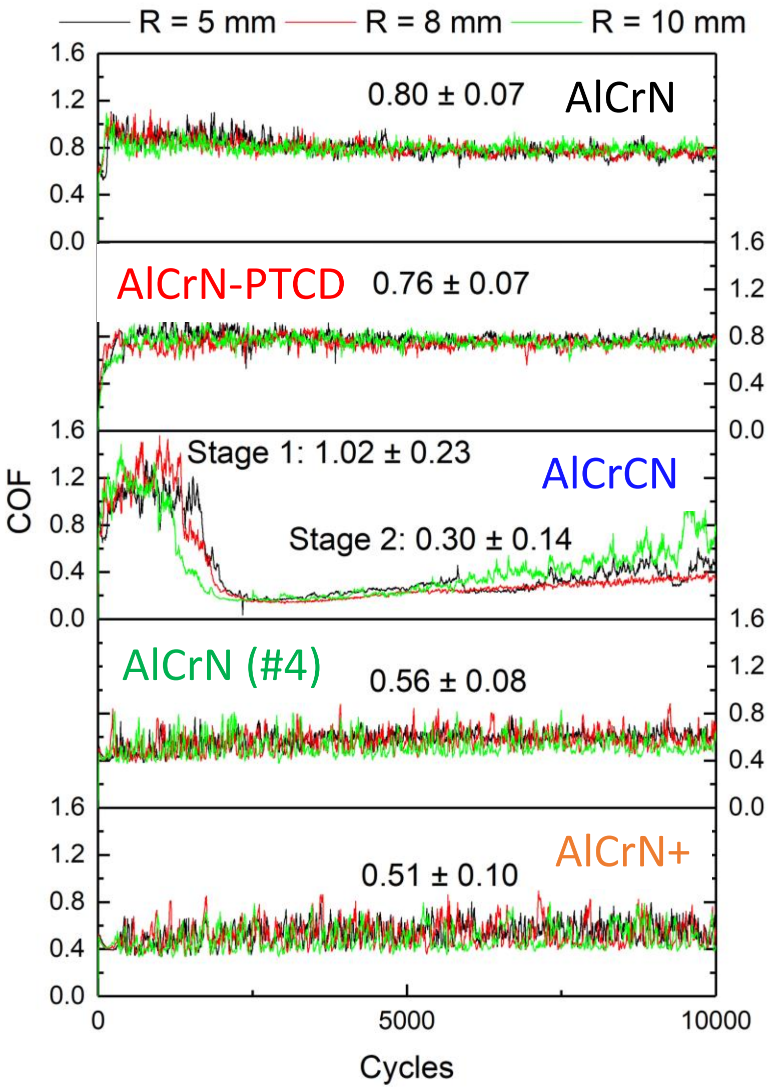
Wear and Erosion Resistance



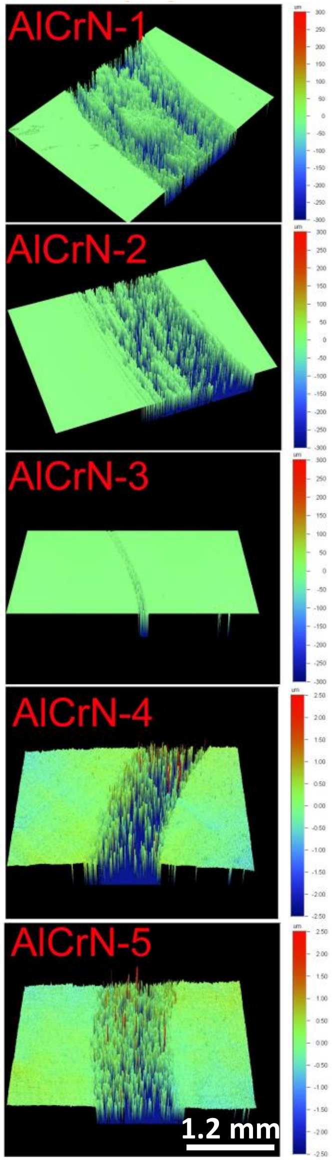
- In his NADCA book on PVD coatings [2]
  - John Moore noted that coatings can be damaged by wear and erosion
- So wear resistance of coatings is important
  - Measured wear resistance of the five AlCrN coatings using pin-on-disk

Aluminum Pin-on-Disk Wear Test

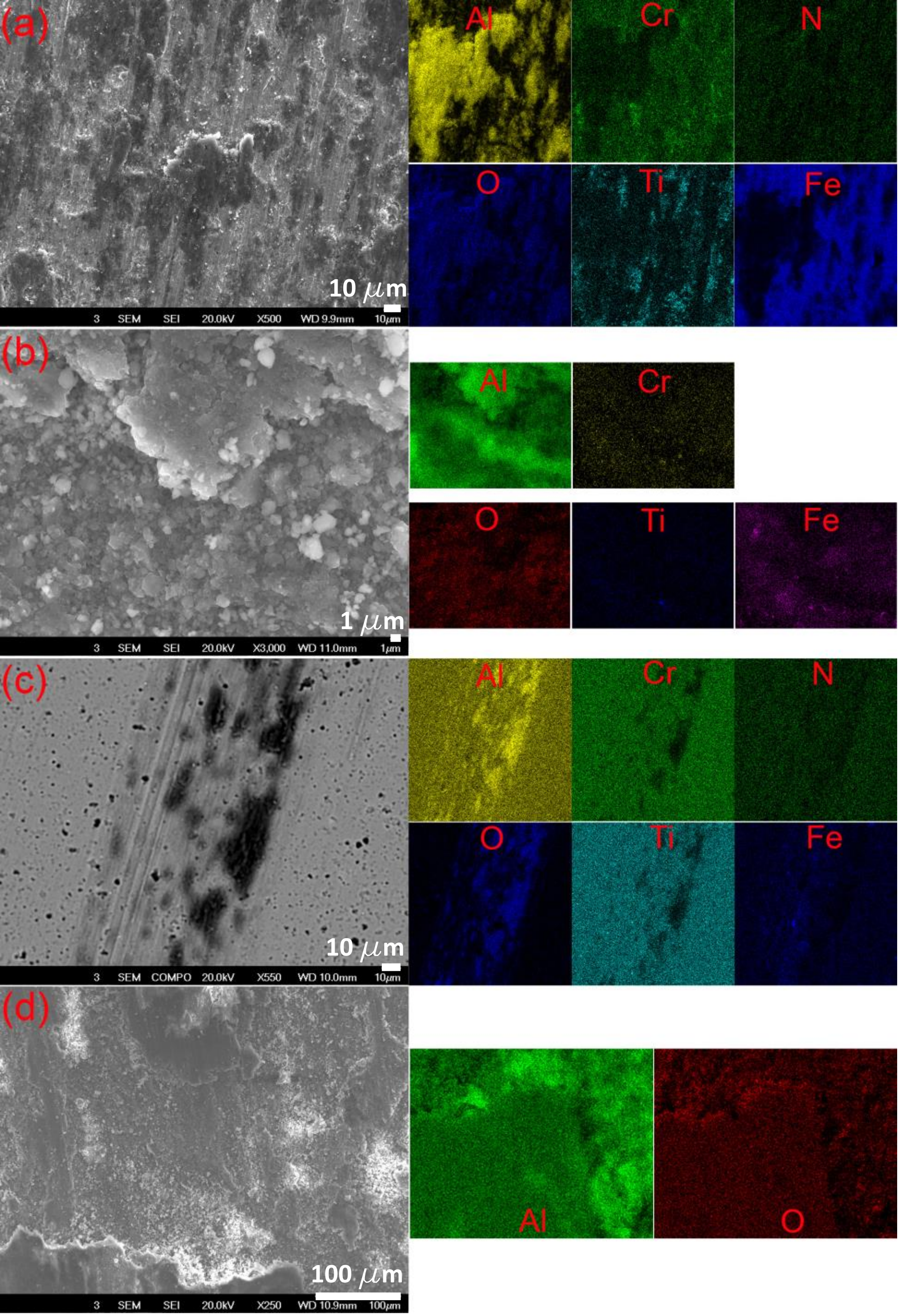
Coefficient of friction versus distance



Wear track in coating



- Wear track for AlCrCN coating is significantly smaller
  - Shows that the AlCrCN coating has better wear resistance
- Supplier #2 AlCrN and AlCrN-PTCD coatings have the same COF ~ 0.80
- AlCrCN have 2 different stages:
  - 1^{st} high COF ~1.02
  - 2^{nd} low COF ~0.30
- Supplier #4 AlCrN and AlCrN+ coatings have the same COF ~ 0.55



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Pin-on-disk Wear against Al ball

- (a) and (b) AlCrN (supplier #2):
- (a) track: combination of abrasive and adhesive wear
  - Transfer of Al from the ball
- (b) ball: combination of abrasive and adhesive wear
  - Transfer of coating to the ball
- (c) and (d) AlCrCN:
- (c) track: small abrasive and more adhesive
  - Transfer of Al from the ball
- (d) ball: combination of abrasive and adhesive wear
- No transfer of material from the coating

Cross-Section of Coatings Examined in SEM

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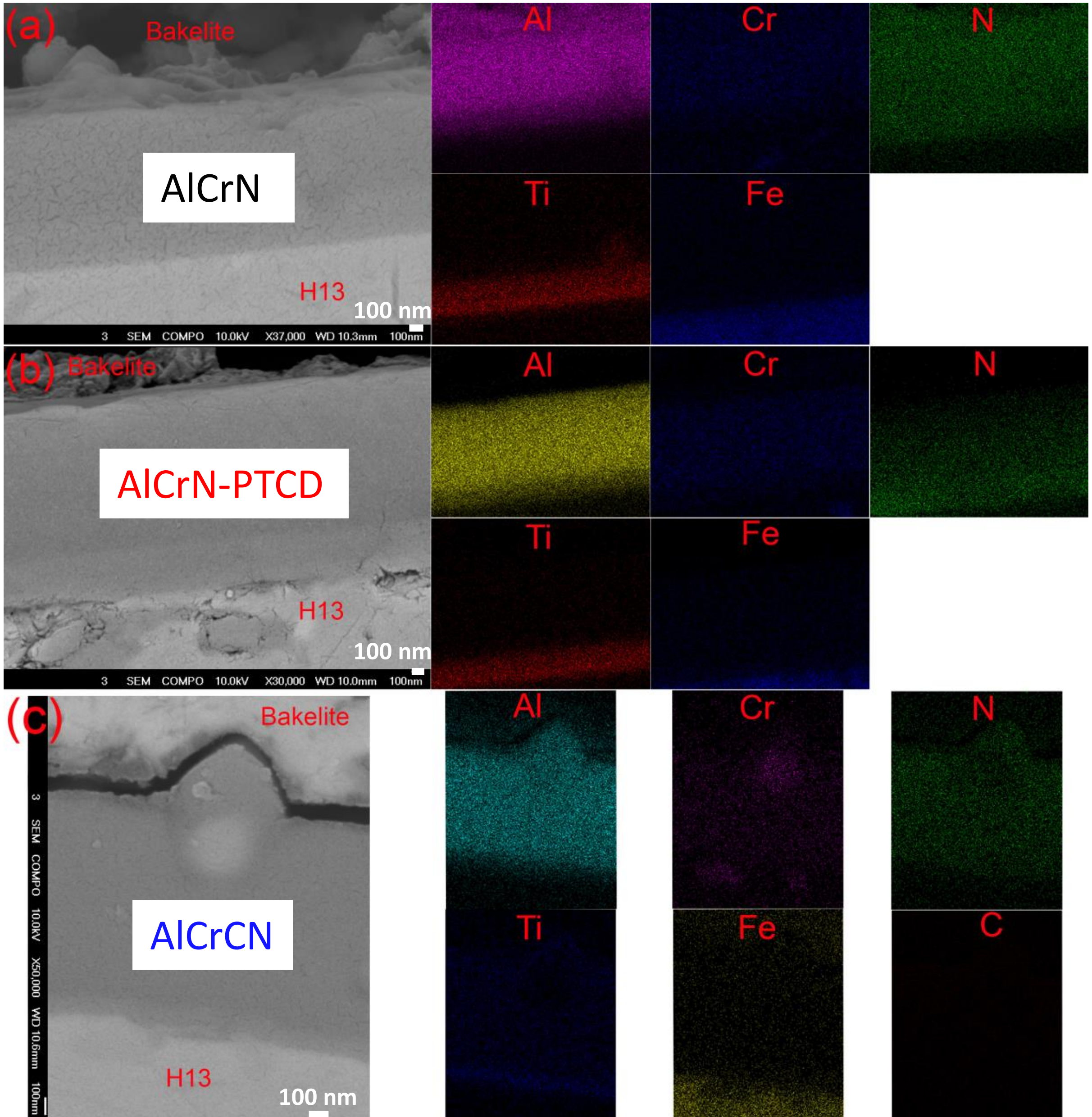
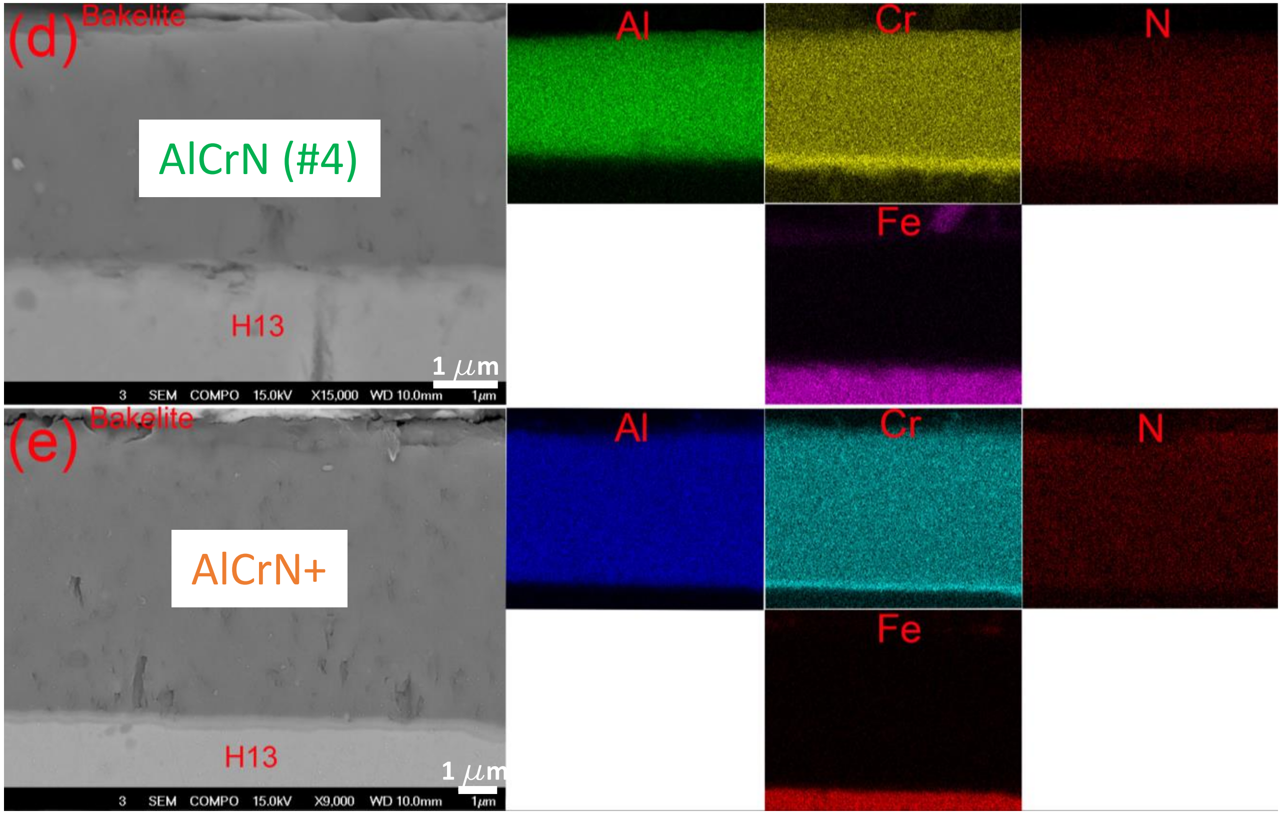


- AlCrN (supplier #2) and AlCrN-PTCD coatings are very similar:
  - H13
    => Ti/TiN bond
    => AlCrN
- AlCrCN:
  - H13
    => Ti/TiN bond
    => AlCrTiCN

Cross-Section of Coatings Examined in SEM

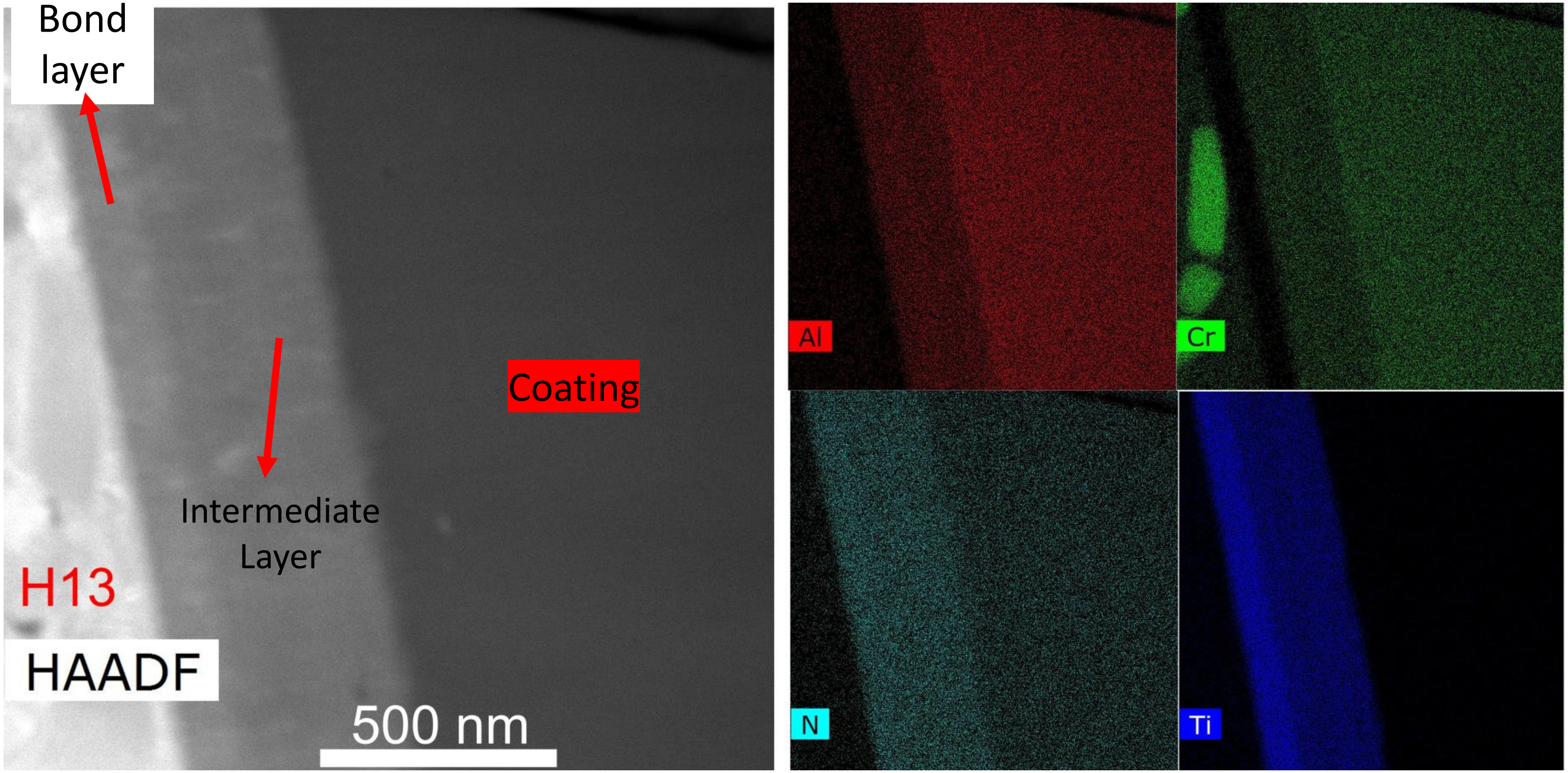


- AlCrN (supplier #4) and AlCrN+ coatings are very similar:
  - H13

    => Cr/CrN bond

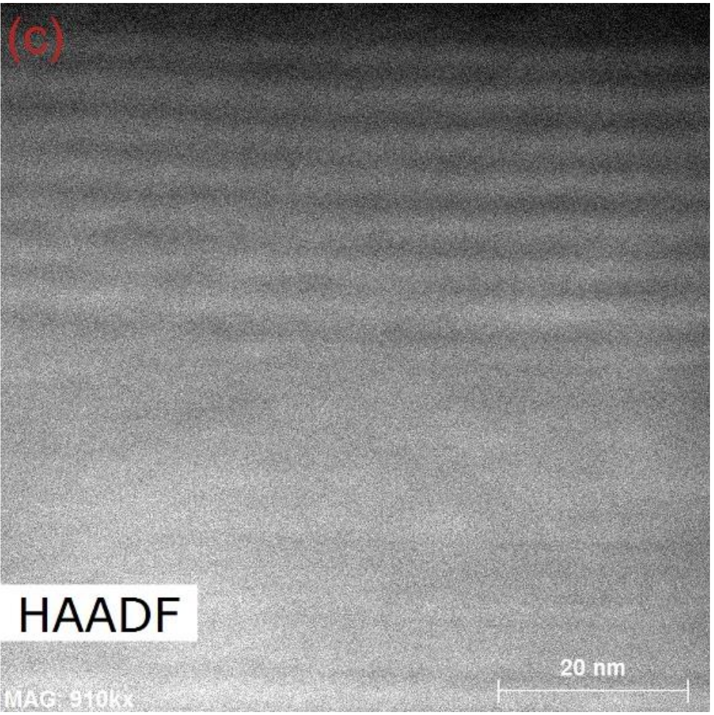
      => AlCrN

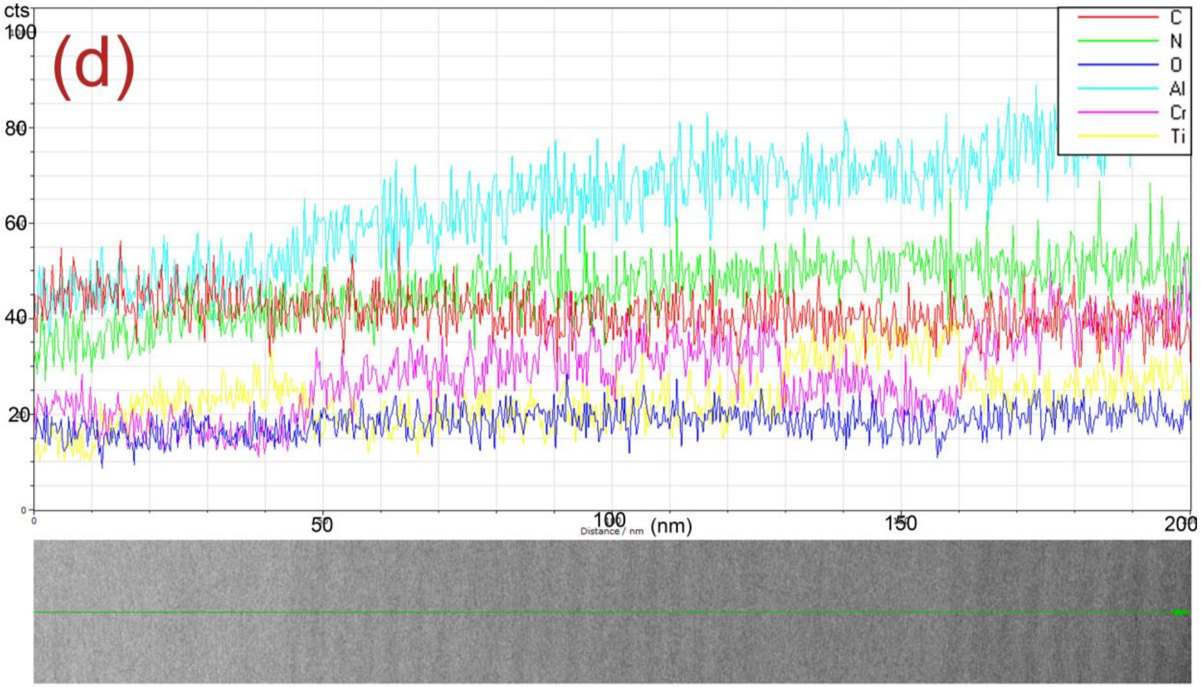
TEM - AlCrN-PTCD



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

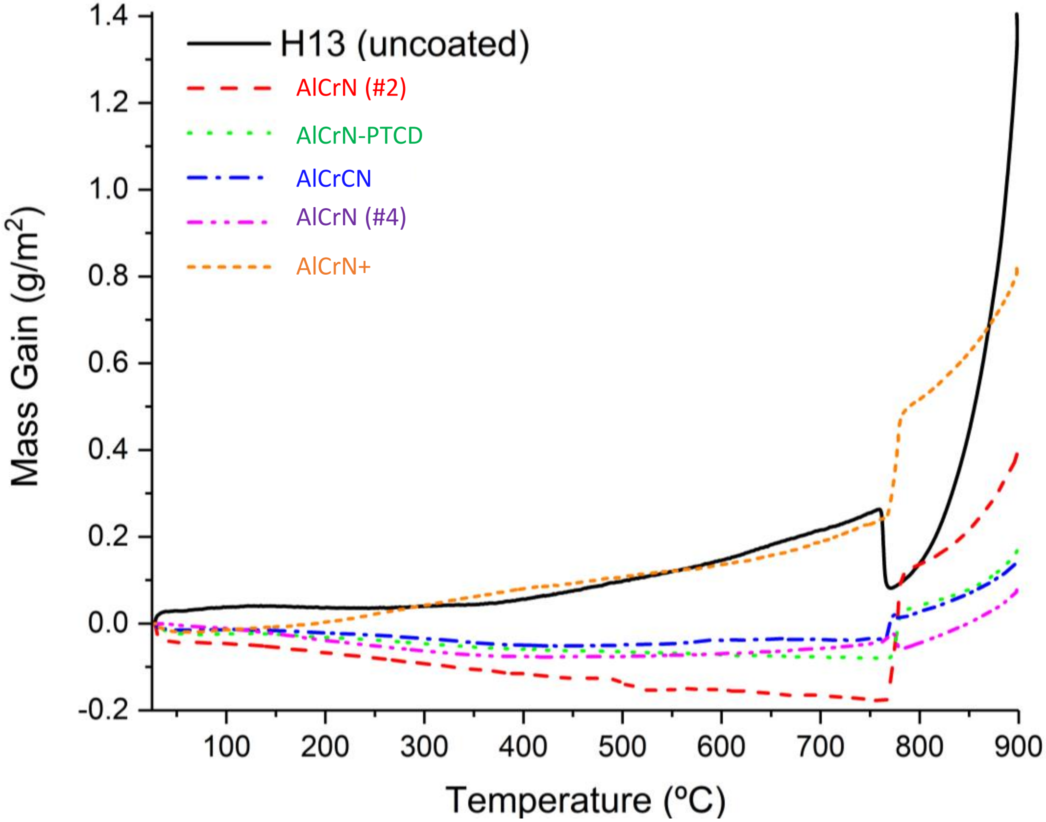
TEM - AlCrCN





- Alternate layers in a nanolayer structure (80 nm thick)
  - High and low Z layers with 2 nm thickness each
  - 35 nm thicker layer separating them

Air Oxidation Resistance



- All coatings have a higher air oxidation resistance than uncoated H13
- AlCrN-PTCD, AlCrCN and AlCrN (#4) have higher oxidation resistance

Summary & Conclusions

- An improved molten aluminum test has been developed
- H13 and five different AlCrN coatings were tested
  - AlCrN and AlCrCN no reaction (supplier #2), AlCrN-PTCD showed reaction at the surface
  - H13, AlCrN and AlCrN+ from supplier #4 stuck
  - AlCrN (supplier #4) required 3 times more load to separate than H13
    - Possible some cracks at the interface between H13 and solidified aluminum
- Characterization of five different AlCrN coatings
  - No difference in mechanical properties / roughness
  - AlCrN and AlCrN-PTCD (supplier #2) are very similar in composition and wear resistance but behave differently in contact with molten aluminum
  - AlCrN and AlCrN+ (supplier #4) are very similar in composition, but behaved differently than the other coatings: they exhibited intermediate wear resistance and behaved the worst in the molten aluminum tests.
  - AlCrCN (supplier #2) is a multi-nanolayer coating with Ti addition and displayed the highest wear resistance, among the highest air oxidation resistance and no apparent reaction with molten aluminum.

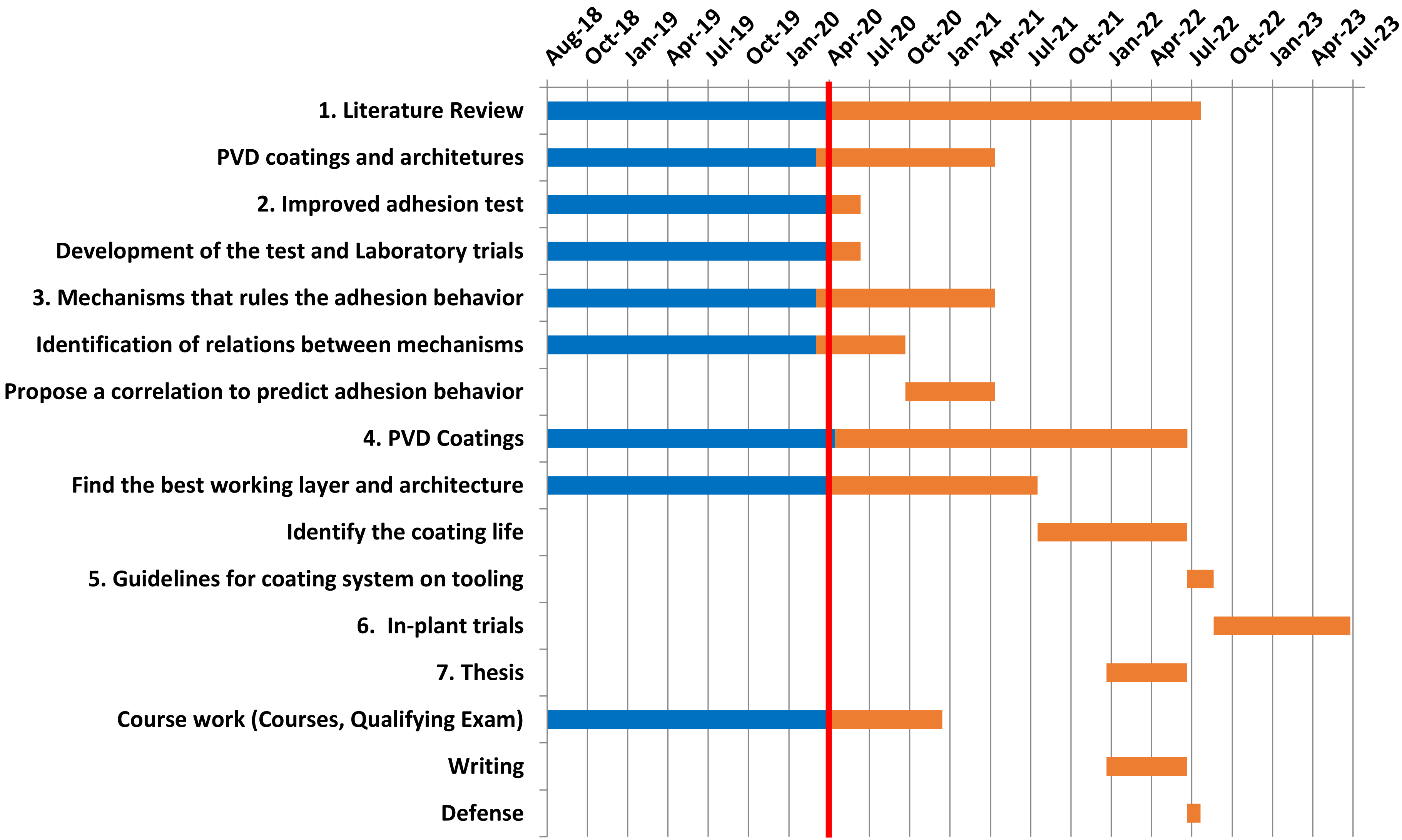
Future Work

- 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 substrates

Challenges & Opportunities

- The PVD equipment at Mines is in the process of being repaired
- 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
- Examine non-PVD coatings (e.g., thermal spray)

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

- This research is sponsored by the DLA – Troop Support, Philadelphia, PA and the Defense Logistics Agency Information Operations, J62LB, Research & Development, Ft. Belvoir, VA.

- Nelson is thankful to CAPES-Brazil for the scholarship.

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 Cotings for Die Casting Dies, NADCA Publication #230, 2003.