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

Summer 2020 Videoconference

June 29 – July 1, July 8 – 10 2020

- Student: Nelson Delfino de Campos Neto (Mines)
- Faculty: Stephen Midson, Andras Korenyi-Both, Michael Kaufman (Mines)
- Industrial Mentors: Paul Brancaleon (NADCA), Rob Mayer (Queen City Forging Co.)

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

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- 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	50%	●
2. Identification of the mechanisms that controls the adhesion behavior.	35%	●
3. Identification of a working layer coating that avoids molten aluminum soldering and adhesion.	35%	●
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

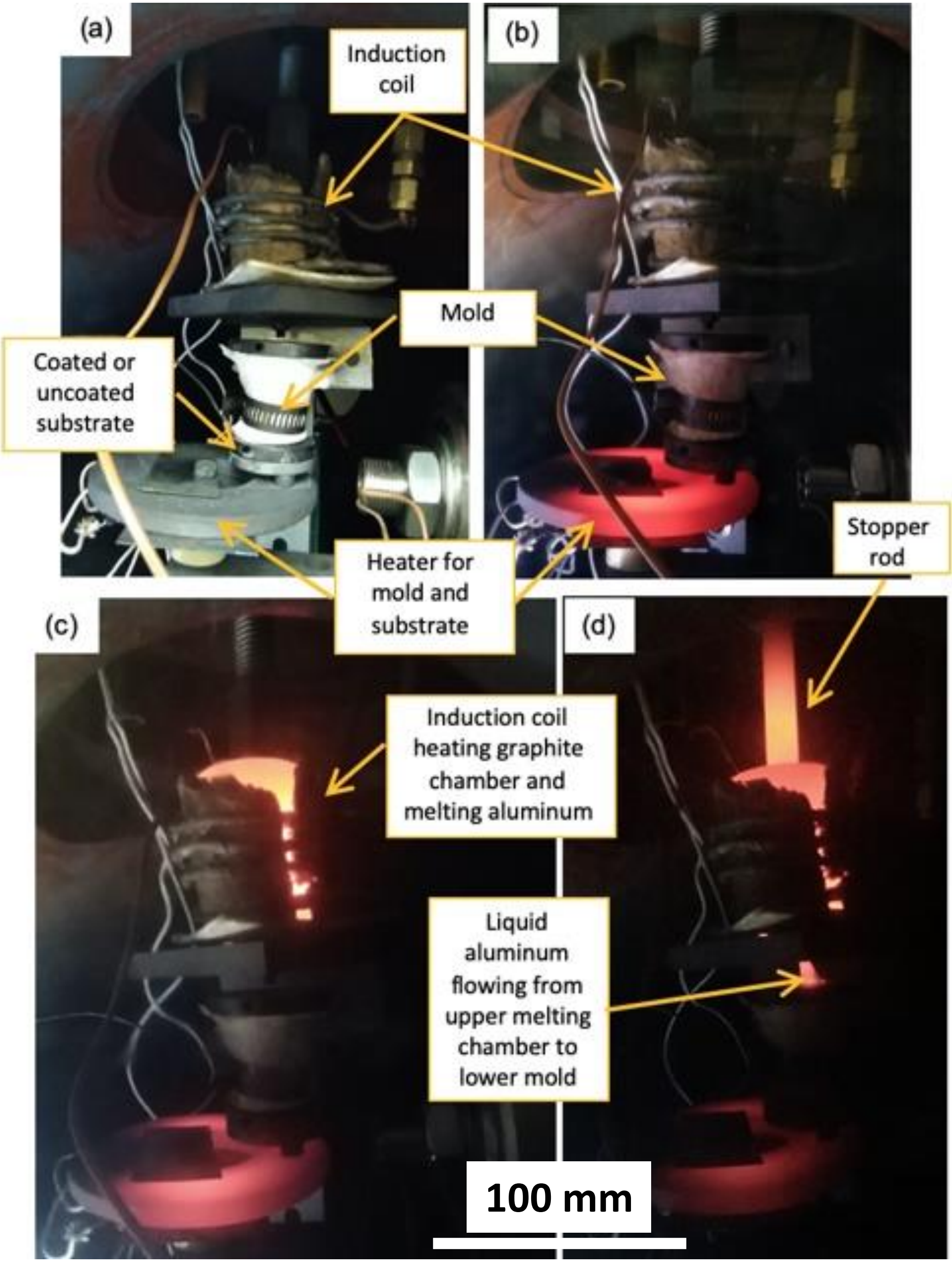
- Reducing or eliminating lubricant spray will:
  - Significantly **improve the quality** of the die castings
    - Reduce gas porosity and scrap
    - Allow castings to be used in higher performance applications
  - **Reduce costs**
    - Eliminate purchase costs for lubricants
    - Reduce effluent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

Task 1.
Develop Improved Adhesion Test

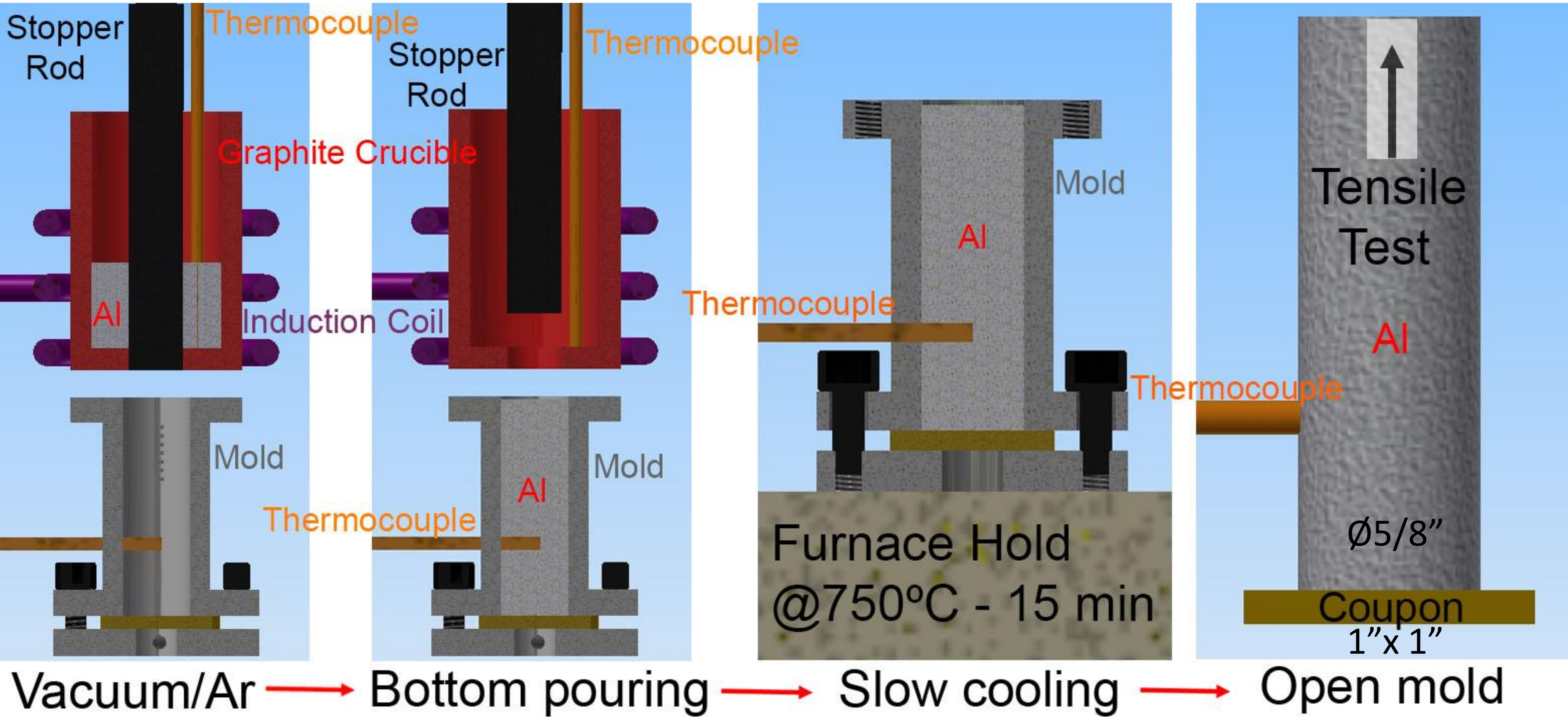
Test Performed in a Controlled Atmosphere



Test in Controlled Atmosphere

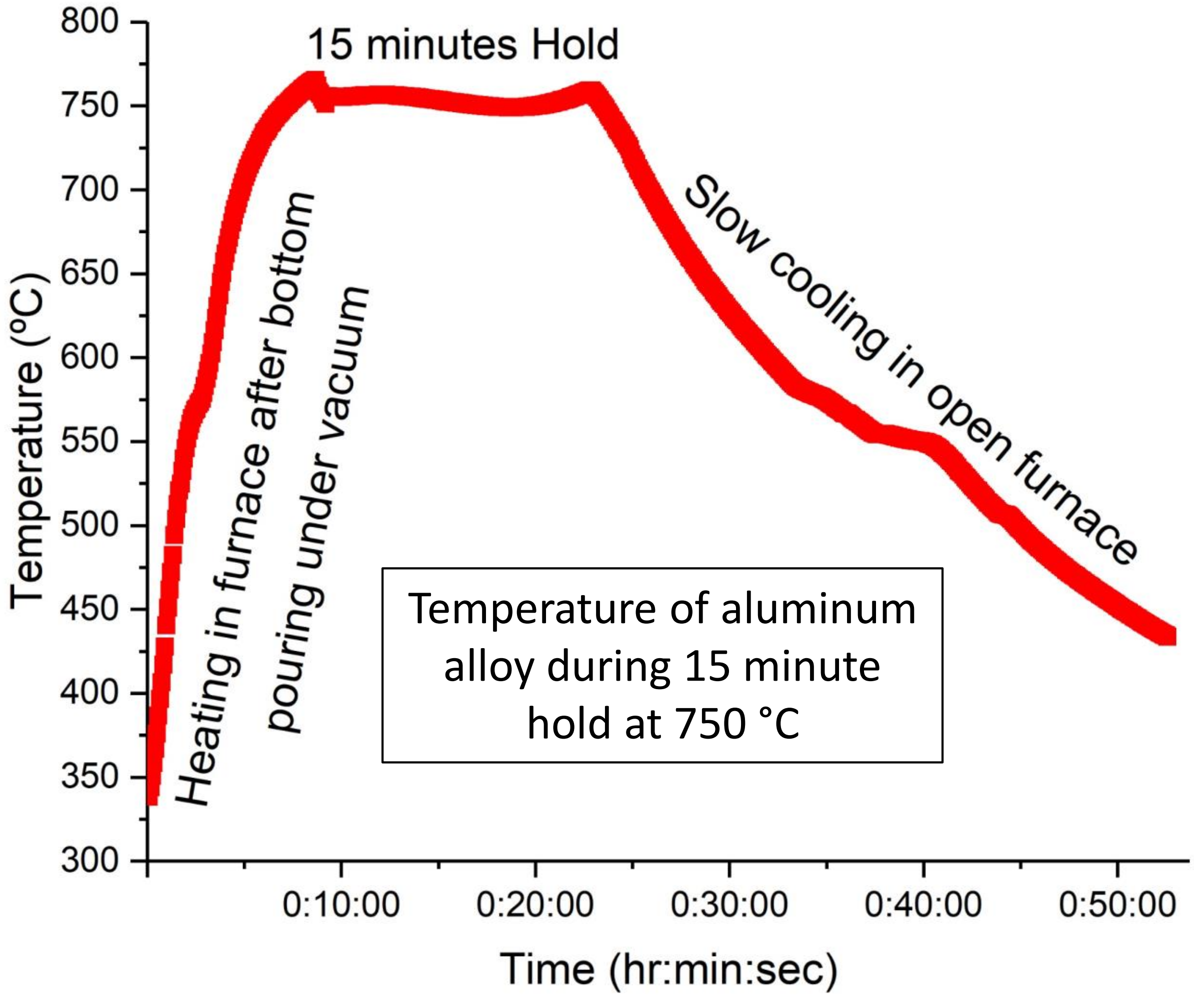
- After the test in C.A.:
- Transfer to pre-heated furnace at 850 °C
  - Holding Temp: ~750 °C
  - 15 minutes
- Does aluminum solder to substrate?
- => Tensile test to rank coatings

Schematic of the Test



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

Temperature Profile



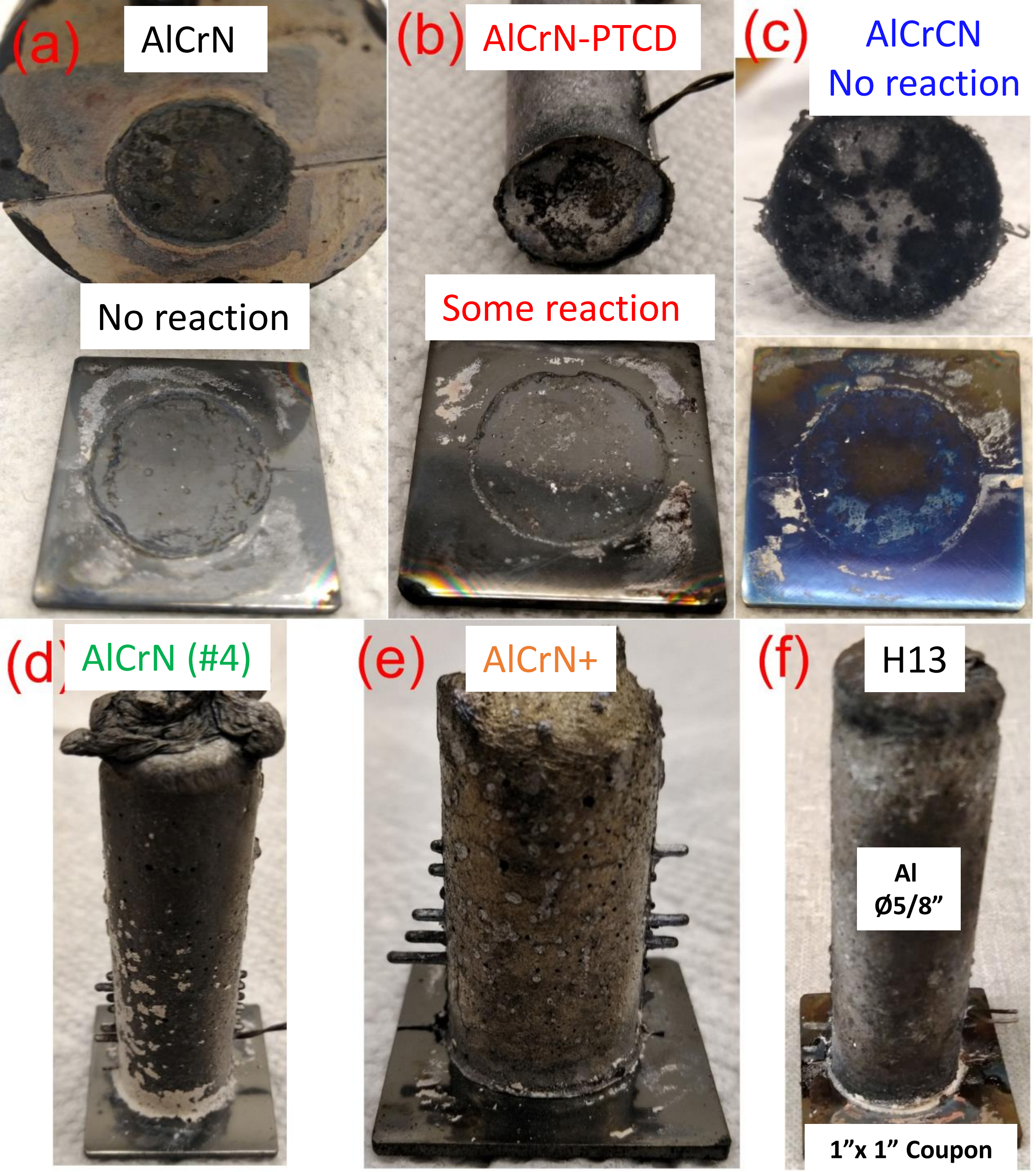
Tasks 2 & 3.
Identify Best Coatings

Materials Tested

- Tested AlCrN coatings from two suppliers

Supplier	Coating
2	AlCrN
2	AlCrN-PTCD*
2	AlCrCN
4	AlCrN
4	AlCrN + ion nitriding
--	None (bare H13)

*Post deposition polishing treatment

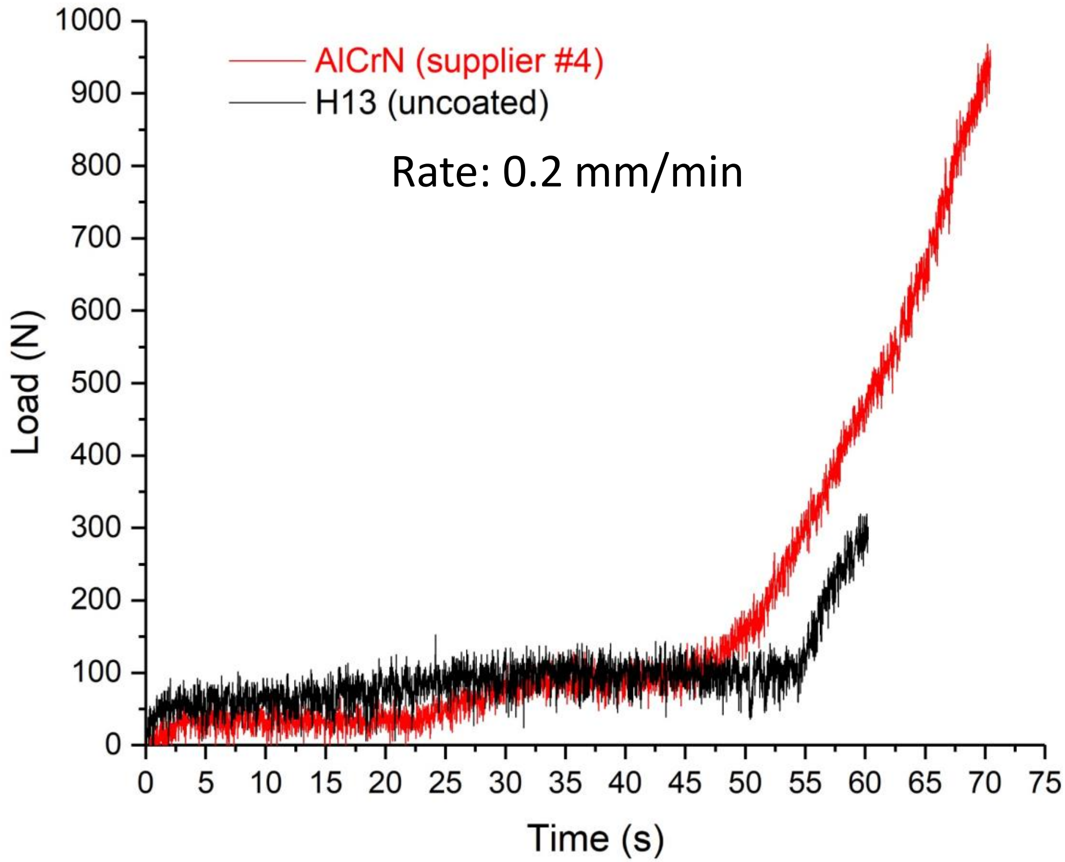


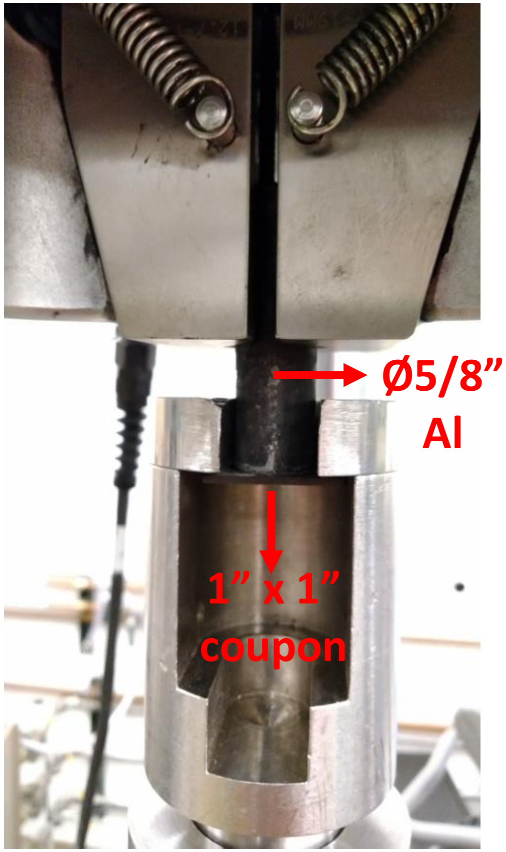
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

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

Tasks 2 & 3.
Identify Permanent Coatings

Characterization of Five Different AlCrN Coatings

Coating Thickness

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

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

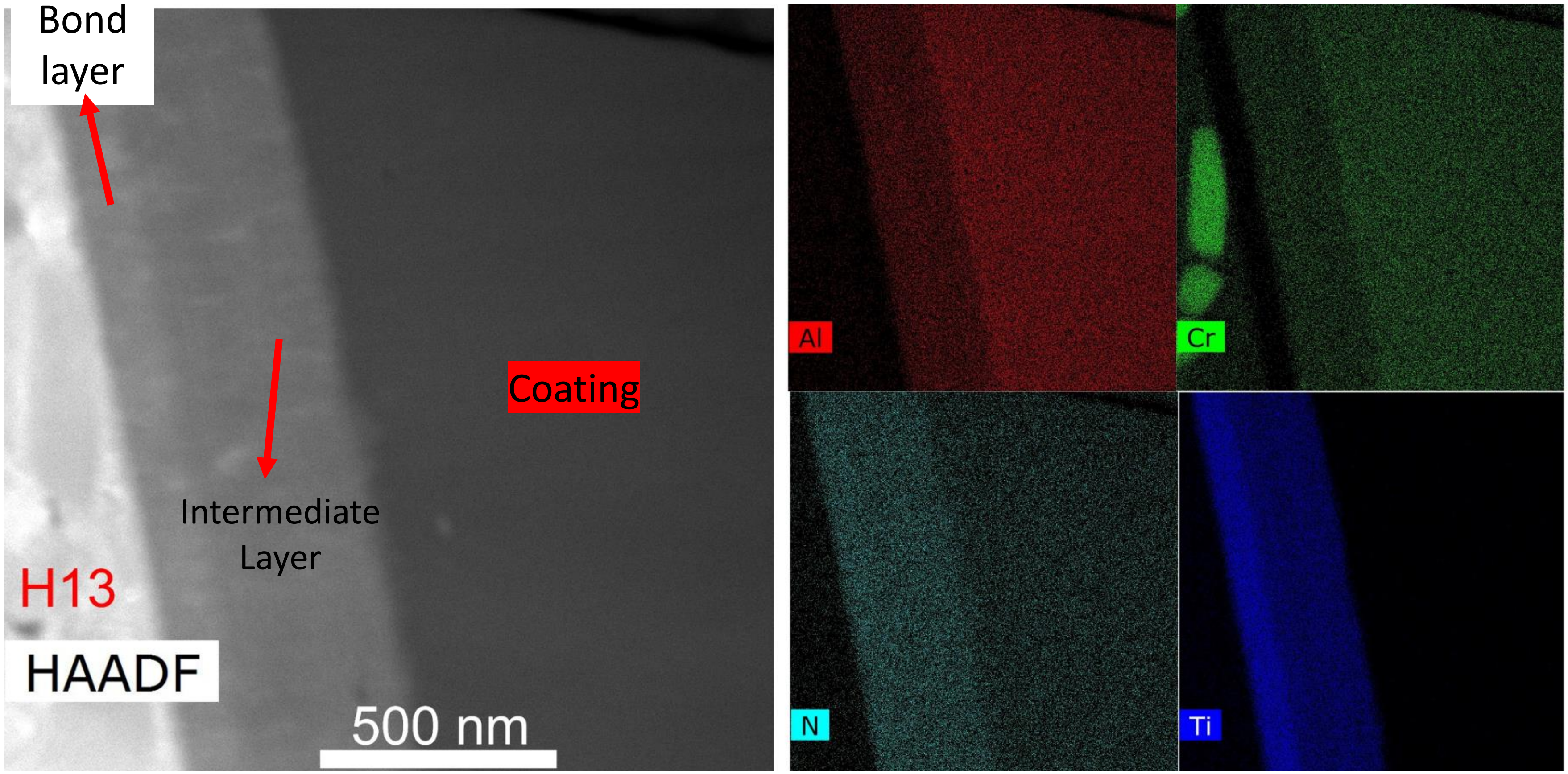
- AlCrCN has a huge different composition and contains Ti
- AlCrN (sup#2) and AlCrN-PTCD have similar compositions
- AlCrN (sup#4) and AlCrN+ have similar compositions

Coating Structure

- Based on analysis in the SEM

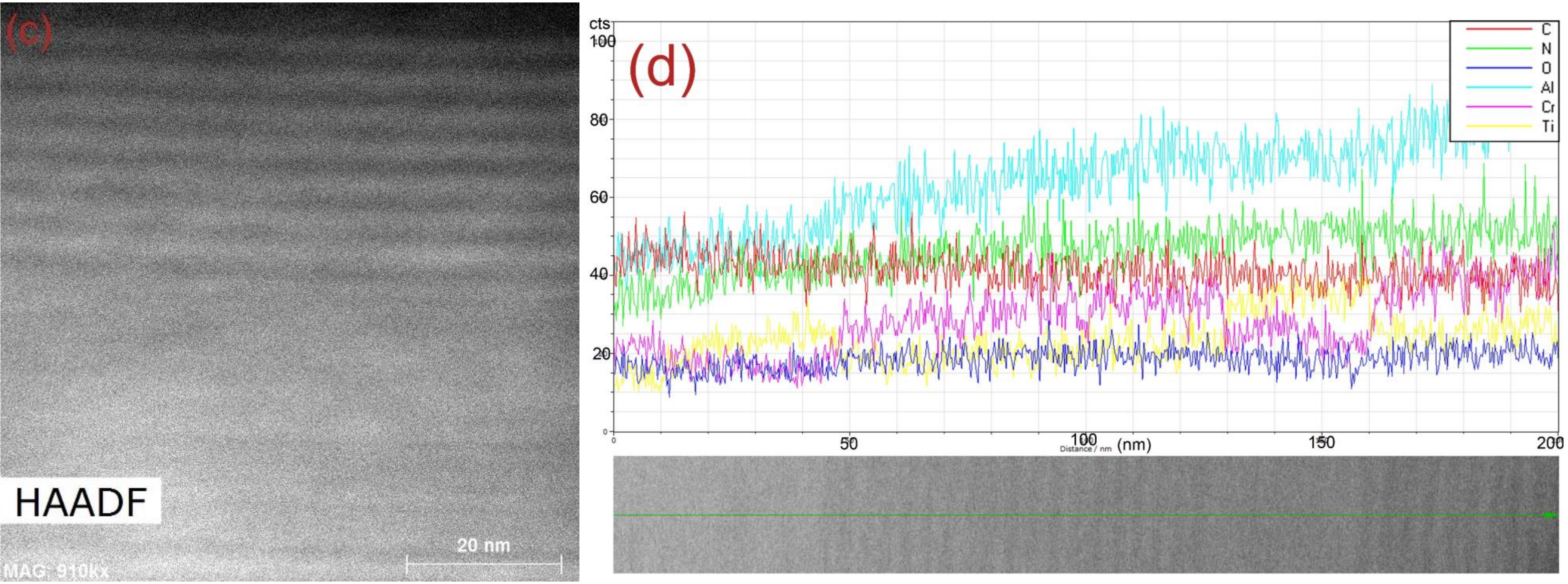
Supplier	Coating	Adhesion Layer	Coating Structure
1	AlCrN	Ti/TiN	Single layer
1	AlCrN + PTCD	Ti/TiN	Single layer
1	AlCrCN	Ti/TiN	Multi-layer
2	AlCrN	Cr/CrN	Single layer
2	AlCrN + nitriding	Cr/CrN	Single layer

TEM - AlCrN-PTCD (supplier #2)



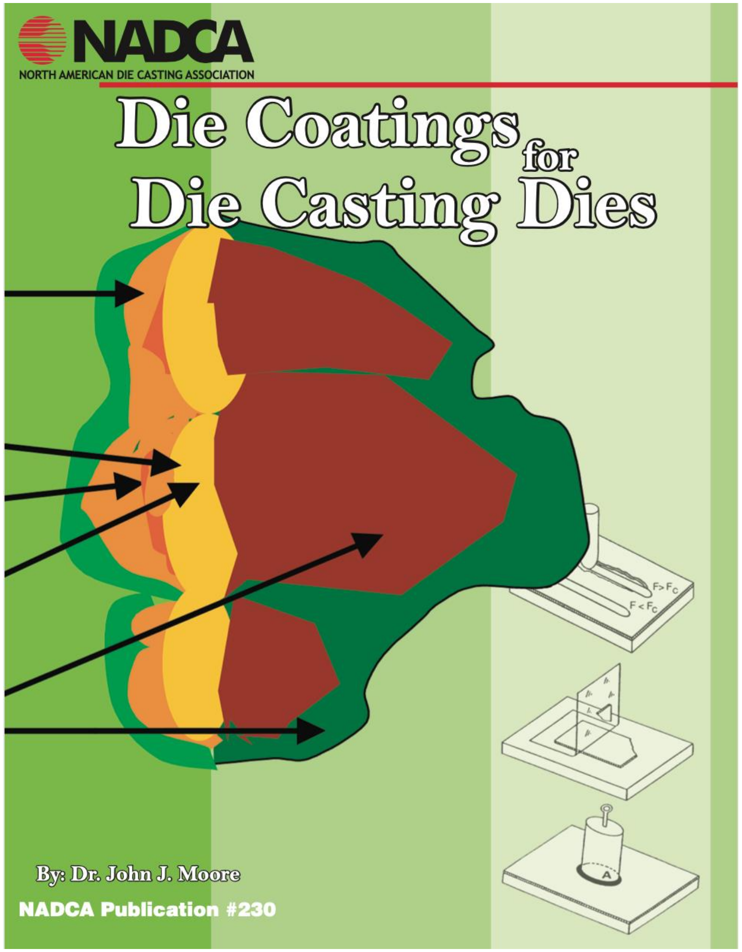
Substrate	Bond Layer	Intermediate Layer	Coating
H13 steel	Ti/TiN	TiAlCrN	AlCrN

TEM – AlCrCN (supplier #2)



- 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

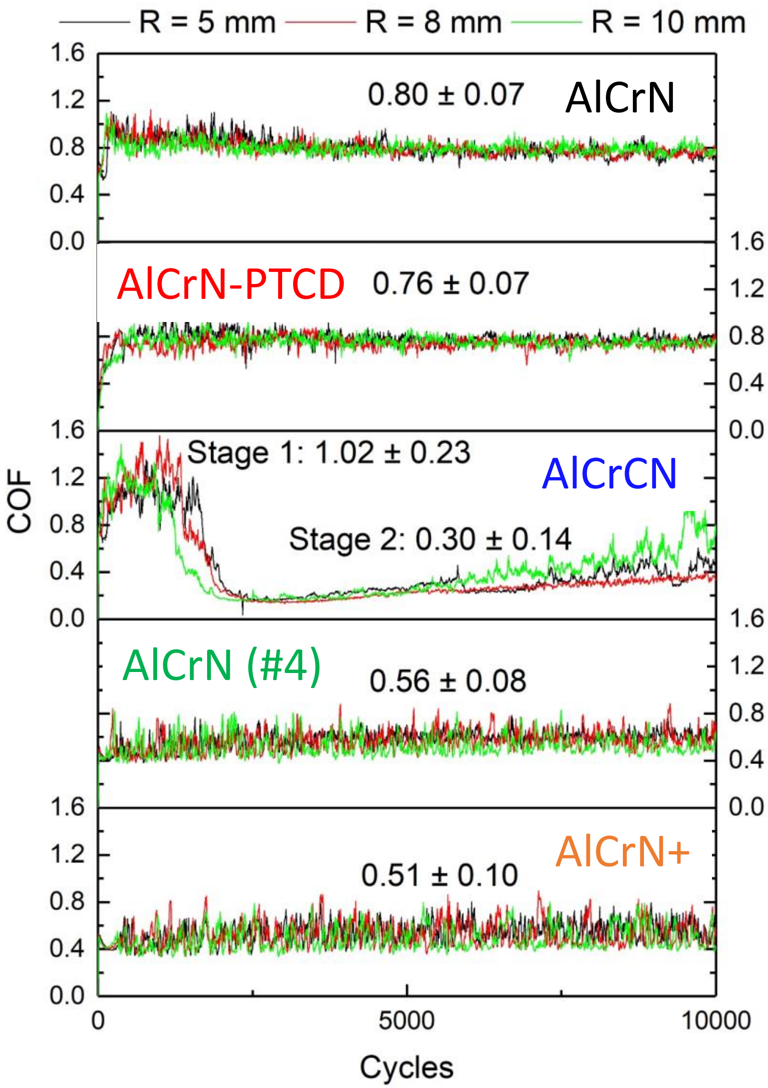
Wear and Oxidation Resistance



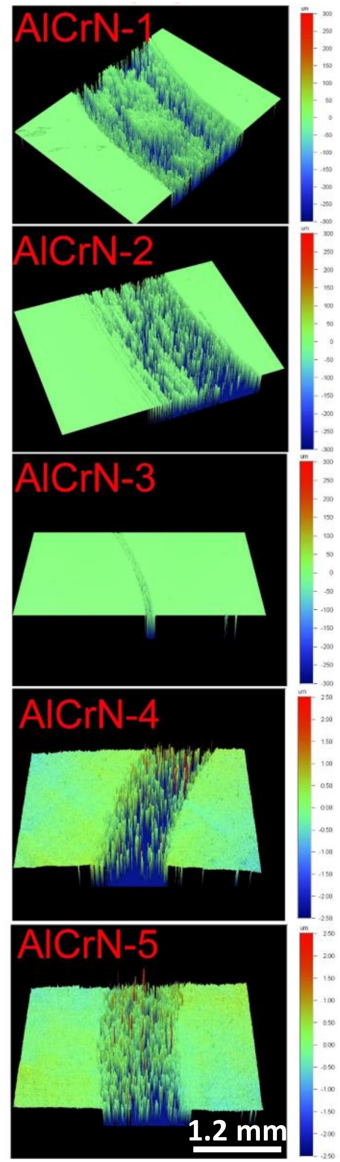
- The NADCA book on PVD coatings described some important properties that a coating should have
  - Wear resistance
  - Oxidation resistance
- Current AlCrN coatings
  - Measured wear resistance using pin-on-disk
  - Measured air oxidation resistance using TGA equipment

Aluminum Pin-on-Disk Wear Test

Coefficient of friction versus distance



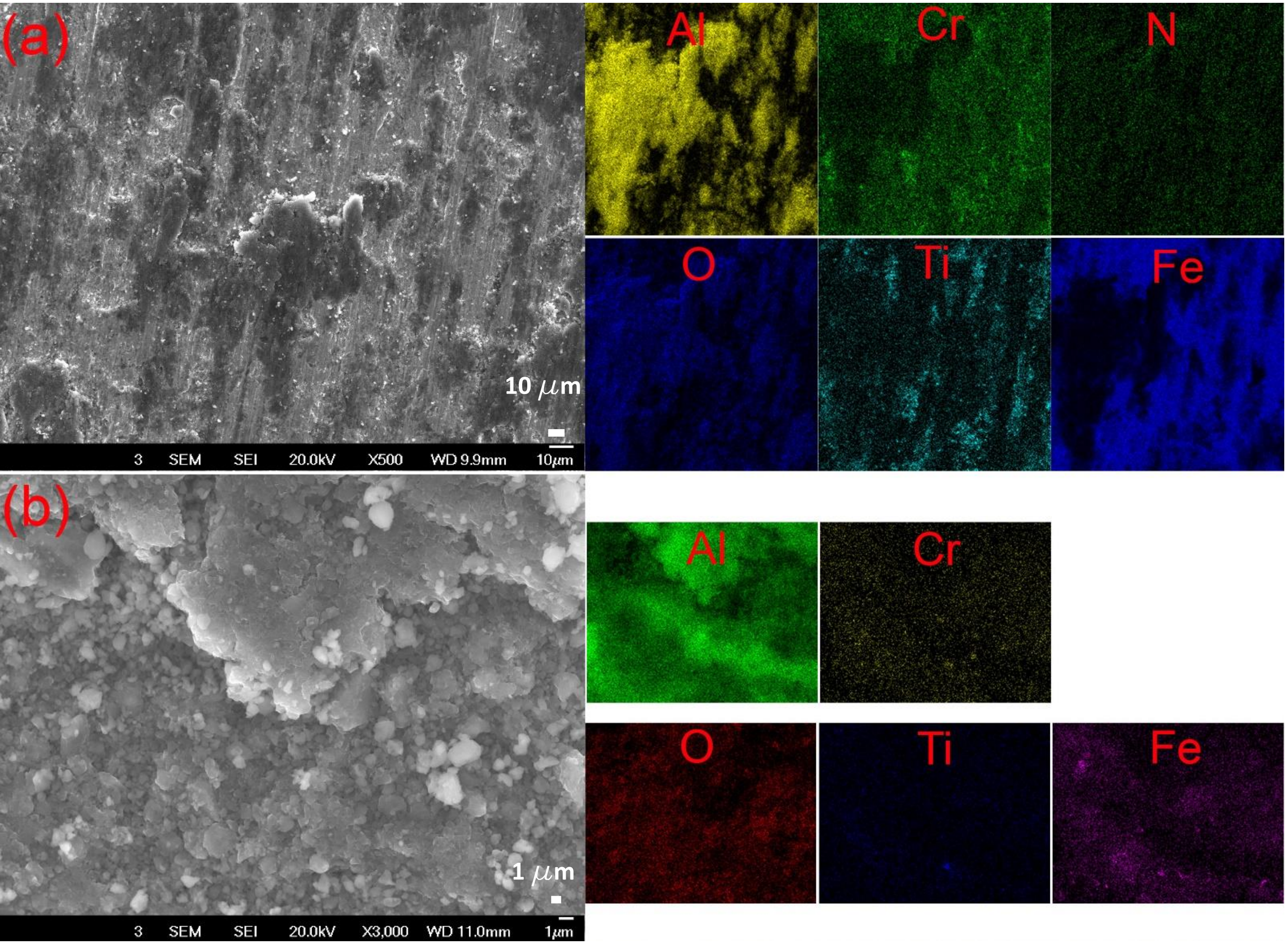
Wear track in coating



CANFSA CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

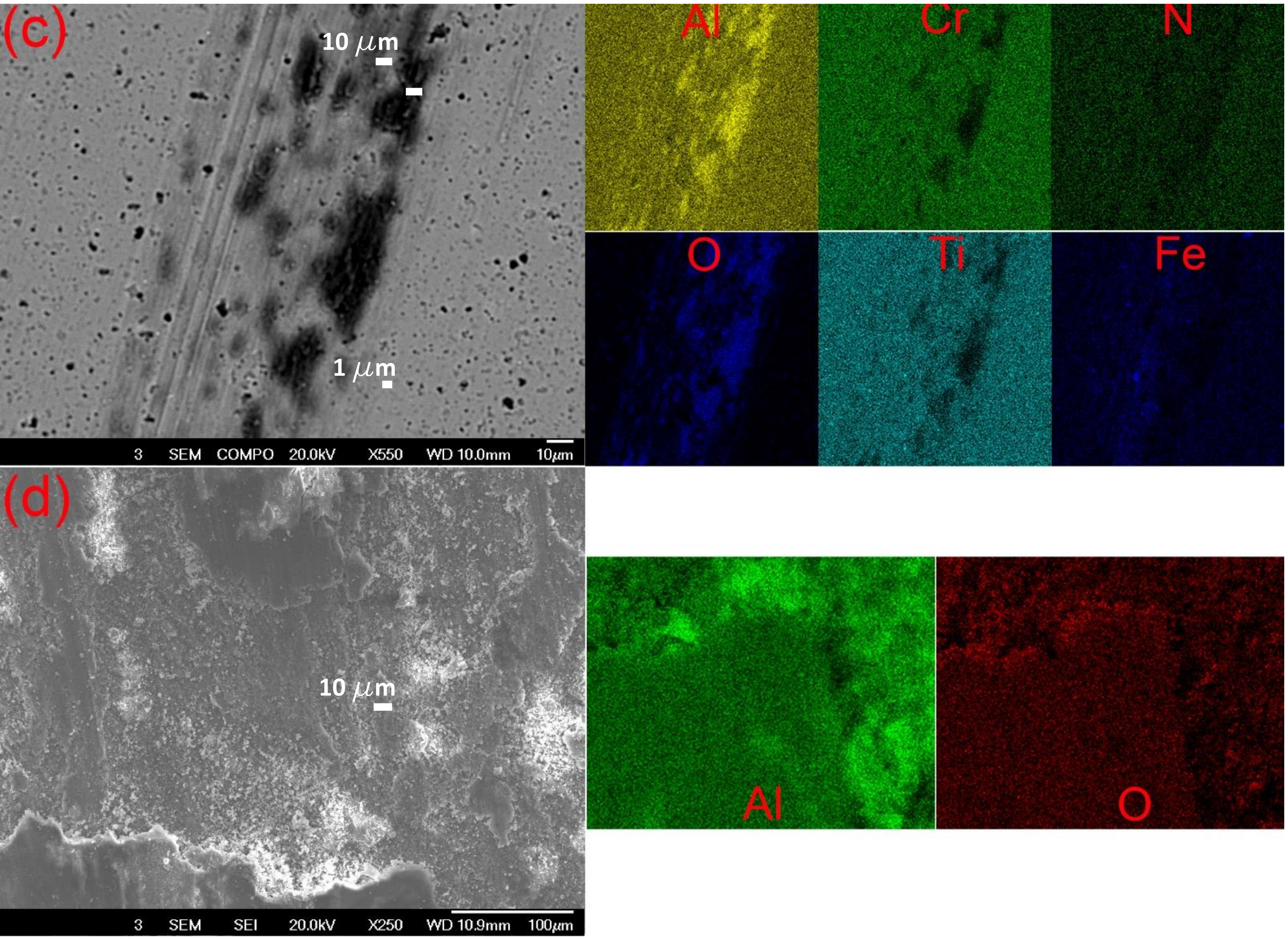
- Wear track for AlCrCN coating is significantly smaller and has better wear resistance
- Supplier #2 AlCrN and AlCrN-PTCD coatings have the same COF ~ 0.80
- AlCrCN coating have 2 different stages:
  - 1st high COF ~1.02
  - 2nd low COF ~0.30
- Supplier #4 AlCrN and AlCrN+ coatings have the same COF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

Pin-on-disk using Al ball



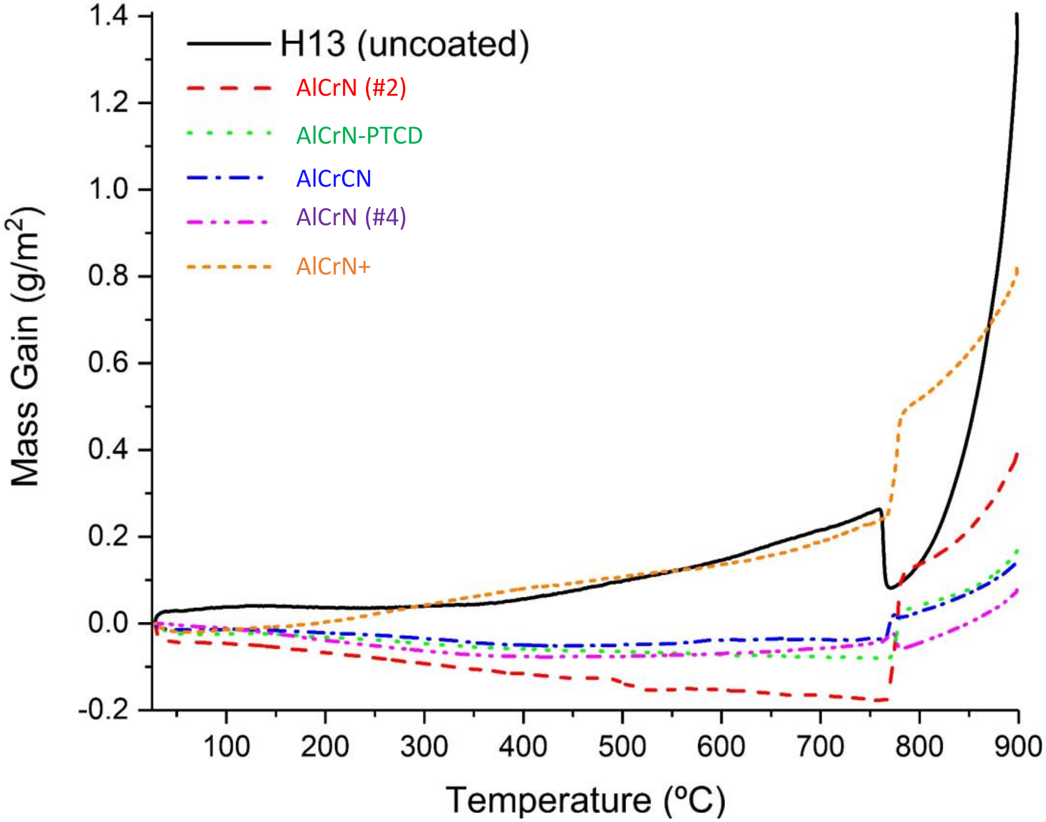
- AlCrN (supplier #2)
- (a) Track: combination of abrasive and adhesive wear
  - Transfer of Al from the ball
- (b) Ball: combination of abrasive and adhesive wear
  - Transfer of coating to the ball

Pin-on-disk using Al ball



- AlCrCN (supplier #2)
- (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

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 exhibited no reaction (supplier #2)
  - AlCrN-PTCD showed reaction at the surface (supplier #2)
  - 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

Summary & Conclusions

- Characterization has been performed for 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
    - Exhibited intermediate wear resistance and behaved the worst in the molten aluminum tests
  - AlCrCN (supplier #2) is a multi-nanolayer coating with Ti addition
    - 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

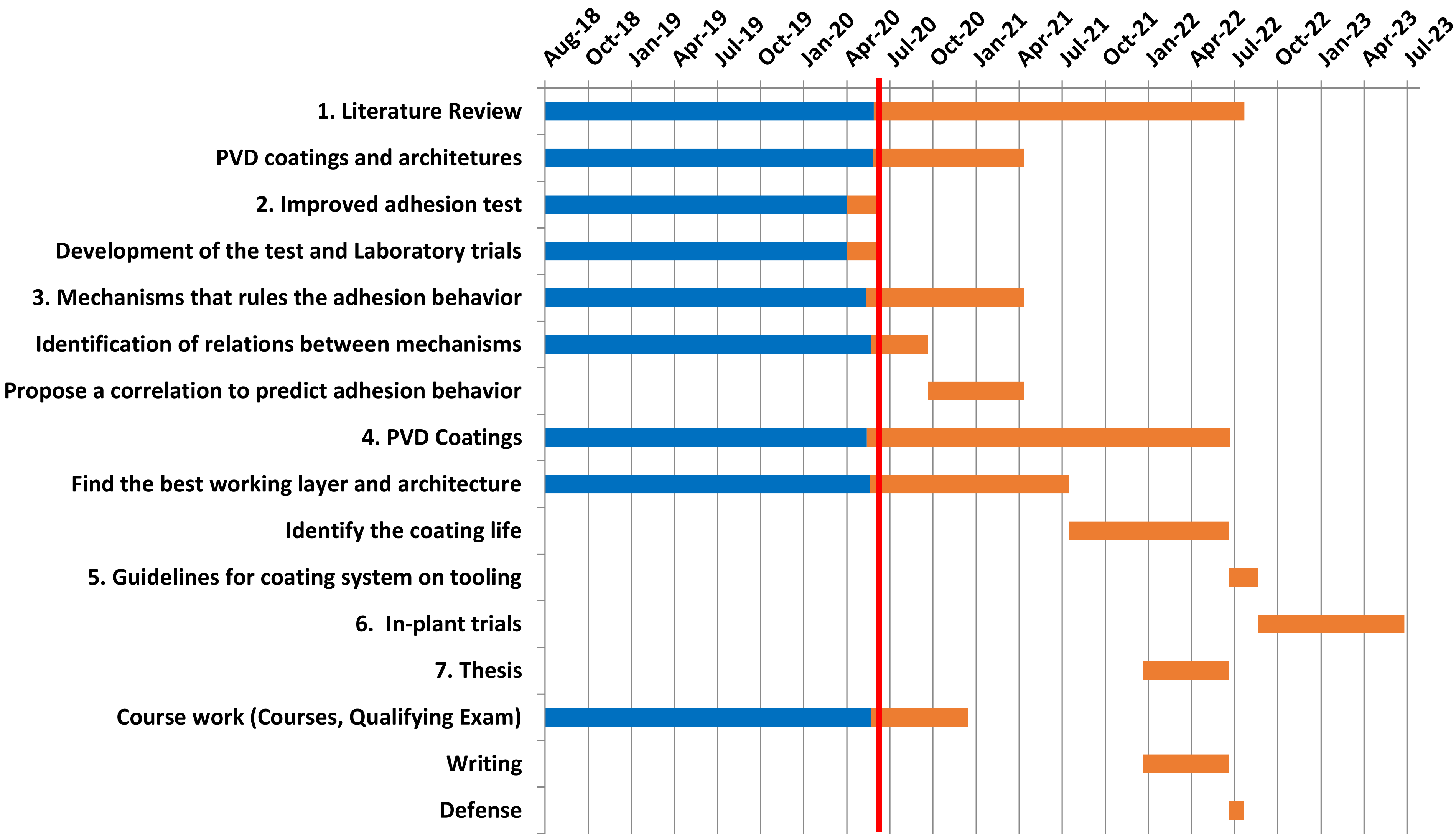
Future Work

- 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°C (930°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
- 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 oxidation on the test
  - Using controlled atmosphere, improved control of substrate temperature and performing multiple sequential tests are additional goals
- Examine non-PVD coatings (e.g., thermal spray)

Progress


Aug-18
Oct-18
Jan-19
Apr-19
Jul-19
Oct-19
Jan-20
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
Jul-21
Oct-21
Jan-22
Apr-22
Jul-22
Oct-22
Jan-23
Apr-23
Jul-23
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, J68, Research & Development, Ft. Belvoir, VA.

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

Thank you!

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

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.