37.0 ADVANCED ENGINEERED COATINGS WITH EXTENDED DIE LIFE FOR TOOLING

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

This project started in Fall 2018, is a five-year effort, and is supported by the Defense Logistics Agency. The research performed during this project will serve as the basis for a Ph.D. thesis for Nelson Delfino de Campos Neto.

37.1 Project Overview and Industrial Relevance

Die coatings produced by physical vapor deposition (PVD) started being used in the die casting industry in the 1990s, but at that time the coatings were relatively simple in nature and tended to be used only to minimize soldering of molten aluminum to core pins. Since then, die casters have developed more complex multi-layer coating architectures, and have also started to use the coatings for "lube-free" applications. However, the factors that prevent the die cast aluminum alloys from sticking to the coatings are still not fully understood, precluding optimal coating compositions from being identified. In addition, die coating architectures need to be identified that will allow the coatings to last as long as the dies (~100,000 shots).

The PVD coatings help prevent aluminum die castings from soldering to the die surfaces, allowing the amount of lubricants that are applied to the die to be reduced or even eliminated. Minimizing the use of lubricants will reduce production costs arising from the purchase of the lubricants, the clean-up of effluents, reducing cycle time, and provide an extension in die life, resulting in lower per-part costs. Reduced lubricant also leads to a significant improvement of the quality of die castings, allowing them to be used in higher performance applications. Cost optimization is important for part manufacturers, as die casting is normally the lowest cost approach for producing complex-shaped components from aluminum alloys.

37.2 Previous Work – Literature Review and Background

A prior project was performed at the Colorado School of Mines (Mines) by Wang [37.1], where a variety of PVD coatings were evaluated, finding that AlCrN had the best performance of those tested. The AlCrN coating was applied to a commercial die and a plant trial was conducted at Mercury Castings, where they were able to reduce the use of conventional organic lubricants by ~85% and significantly reduce cycle time. The goal of the current follow-on project is to build on the research performed by Wang, and achieve the complete elimination of conventional lubricants for the die casting process.

The initial phase in the current project involved performing a literature review of several related fields, including brazing, the dissolution of materials by liquid metals, and the wetting and reaction between ceramics and liquid metals. The goal is to identify the types of ceramic coatings that can minimize wetting and soldering during the die casting process. To date, the literature review has primarily focused on understanding wetting behavior. A relevant publication is a recent review paper by Eustathopoulos [37.2], who reviewed the factors controlling the wetting of ceramics by liquid metals, and the concepts reported by Eustathopoulos have been correlated with the results presented by Wang [37.1]. This has enabled the current authors to develop a better understanding of the factors controlling wetting of liquid metals on bare H13 steel and on PVD coatings.

During the first year of the project, the main focus was on developing an improved aluminum adhesion test, based on the test described in previous reports [37.3-37.4]. The key features of this improved test include i) using bottom pouring to avoid incorporation of the floating oxide layer between the material coupon and the cast aluminum, ii) using induction heating to quickly heat and melt the aluminum alloy, iii) controlling and recording of the temperature of the material coupon during the test, iv) performing melting inside a controlled environment chamber (vacuum + back filling with inert gas), v) pre-heating the casting die and material coupon prior to bottom pouring of the molten aluminum, and vi) rapidly placing the cast aluminum and material coupon into a pre-heated furnace for extended holding times at the desired temperature, to allow for diffusion at the interface between the material coupon and the molten aluminum alloy.

37.3 Recent Progress

37.3.1 Aluminum adhesion test in controlled atmosphere

Further improvements have been made to the test setup described in previous reports [37.3-37.4]. As shown in **Figure 37.1**, a pancake electric resistance heater has been added to the apparatus to allow the coated or uncoated material coupon to be heated under an inert Argon atmosphere to a temperature more representative of the commercial die casting process. The heater is capable to heating the material coupon close to 500 ºC. This test apparatus is placed into a chamber that allows atmosphere control. A vacuum can be pulled to remove most of the air from the chamber down to ~150 micron, and then the chamber can be back-filled with an inert gas (argon) to prevent excessive oxidation of the material coupon as it is heated to 500 ºC. The modified apparatus is shown in Figure 37.1. The pancake electric resistance heater can be clearly seen in Figure 37.1a and Figure 37.1b. Otherwise the test procedure is as described previously, where the aluminum is placed in a graphite upper melting chamber (Figure 37.1a) and induction coils are used to heat the graphite chamber and melt the aluminum (Figure 37.1b). A thermocouple is placed in the upper melting chamber to monitor the temperature of the aluminum, and once the molten aluminum reaches 750 ºC, the graphite stopper rod is raised allowing the molten aluminum to flow from the upper chamber to the lower mold and contact the pre-heated material coupon (Figure 37.1d). The material coupon can be in the shape of a flat plate or a rod; in the latter case, the molten aluminum covers a large portion of the rod.

Another possibility with this new modified aluminum adhesion test (AAT) is to transfer the mold with molten aluminum in contact with the material coupon to a pre-heated furnace to hold it for a pre-determined amount of time to enhance reaction at the interface between the molten alloy and the material coupon. **Figure 37.2** shows a schematic of this variation of the test, which involves i) induction melting of alloy A380 in the upper melting crucible under an inert atmosphere, ii) bottom pouring of the alloy from the upper melting crucible to the lower mold and contacting the material coupon, iii) transferring the alloy, mold and material coupon to a pre-heated furnace and holding for 15 minutes at 750 ºC followed by slow cooling to room temperature, iv) disassembling the mold and performing a tensile test if the coupon is adhered to the aluminum alloy.

37.3.2 Results of the tests on coated coupons using the new AAT

Figure 37.3 shows the temperature profile for the molten aluminum in the casting mold, showing that the aluminum rapidly cools after it is poured into the casting mold, (down to about 300 ºC), but then quickly heats back to 750 ºC when it is transferred into the pre-heated furnace. The apparatus is held at 750 ºC for 15 minutes, and then is slowly cooled back to room temperature.

Five different AlCrN coatings have been obtained from two commercial suppliers, and the results of molten aluminum testing in the modified AAT equipment are shown in **Figure 37.4** for the five AlCrN coatings and bare H13. Note that the testing was performed using the second configuration described above, without pre-heating the material coupon and mold. **Figure 37.4a** shows the result for the first AlCrN coating where the solidified aluminum did not adhere to the coated coupon, and there is no evidence on the coated surface of a reaction between the molten aluminum and the coating. The second coating is referred to as AlCrN-PTCD (AlCrN deposition followed by a post treatment class D) and as shown in **Figure 37.4b**, it behaved differently, as there is evidence of a limited reaction between the aluminum alloy and the coating. The third coating contains carbon, is called AlCrCN, and again, a slightly different behavior was observed. As shown in **Figure 37.4c** the AlCrCN coating did not react with the molten aluminum alloy, but the heating/cooling has led to a color change of the coating that may be a sign of surface oxidation. **Figure 37.4d** AlCrN and **Figure 37.4e** AlCrN+ are coatings from a second supplier, where in both cases the solidified aluminum stuck and adhered to the coated coupons. The same behavior was observed in **Figure 37.4f** for the test performed using a polished uncoated H13 steel substrate. **Figure 37.5** shows the result of the tensile tests for the uncoated H13 and the AlCrN coating from supplier 2, where the coated sample required about three-times more load than the uncoated sample to separate both surfaces. This is similar to results reported by Bo Wang in his previous study [37.1], where Wang attributed the lower load measured for the bare H13 steel coupon to cracking occurring between the solidified aluminum and the H13 substrate during cooling, due to differences in the coefficient of thermal expansion of the two materials.

37.4 Plans for Next Reporting Period

- Continue to evaluate the published literature to characterize wetting, PVD coatings, chemical interactions between liquid metals and ceramics, and brazing.
- Continue the experimental work using the new test apparatus. Tests will be performed on the list of PVD coated samples provided in Table 37.1.
- Characterization of the PVD coated samples using a range of techniques: electron microscopy, tribology, and surface analysis.
- Characterization of the soldered surfaces to understand solder and adhesion, interdiffusion, phase formation, and defects.

37.5 References

[37.1] 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.

[37.2] N. Eustathopoulos, Wetting by Liquid Metals—Application in Materials Processing: The Contribution of the Grenoble Group, Metals 5, 2015, 350-370.

[37.3] N. D. Campos Neto, A. Korenyi-Both, S. Midson, M. J. Kaufman. Advanced Engineered Coatings with Extended Die Life for Tooling. CANFSA Biannual Report, Project 37, March 25, 2019.

[37.4] N. D. Campos Neto, A. Korenyi-Both, S. Midson, M. J. Kaufman. Advanced Engineered Coatings with Extended Die Life for Tooling. CANFSA Biannual Report, Project 37, October 2, 2019.

37.6 Figures and Tables

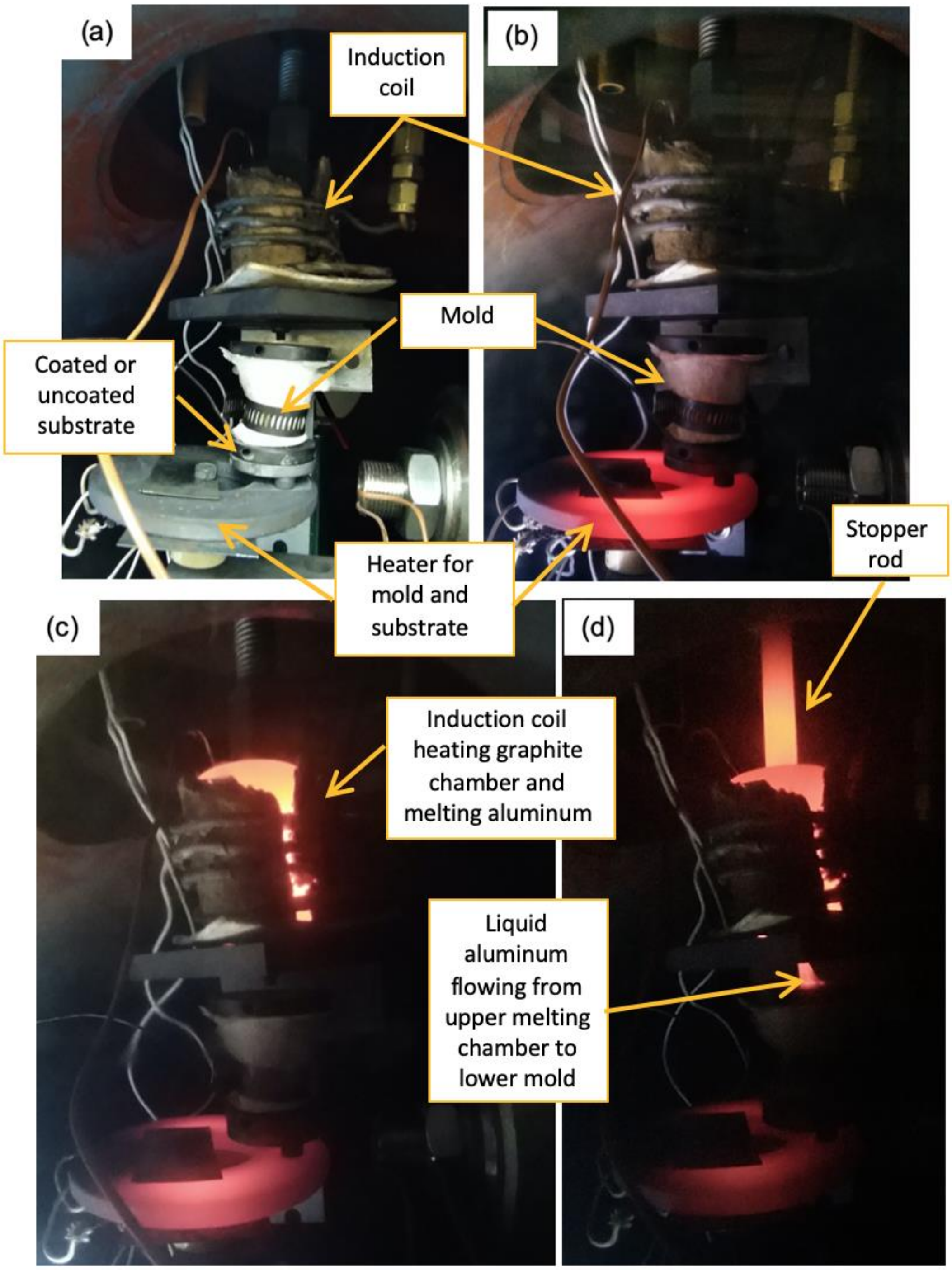

Figure 37.1: Modifications to the setup used to test uncoated and coated samples (a) the overall apparatus, (b) showing the 4 inches diameter heater used to pre-heat the substrate and die, (c) induction melting of the aluminum alloy and (d) pulling up the stopper rod to allow bottom pouring of the molten aluminum alloy to fill the die and contact the coupon.

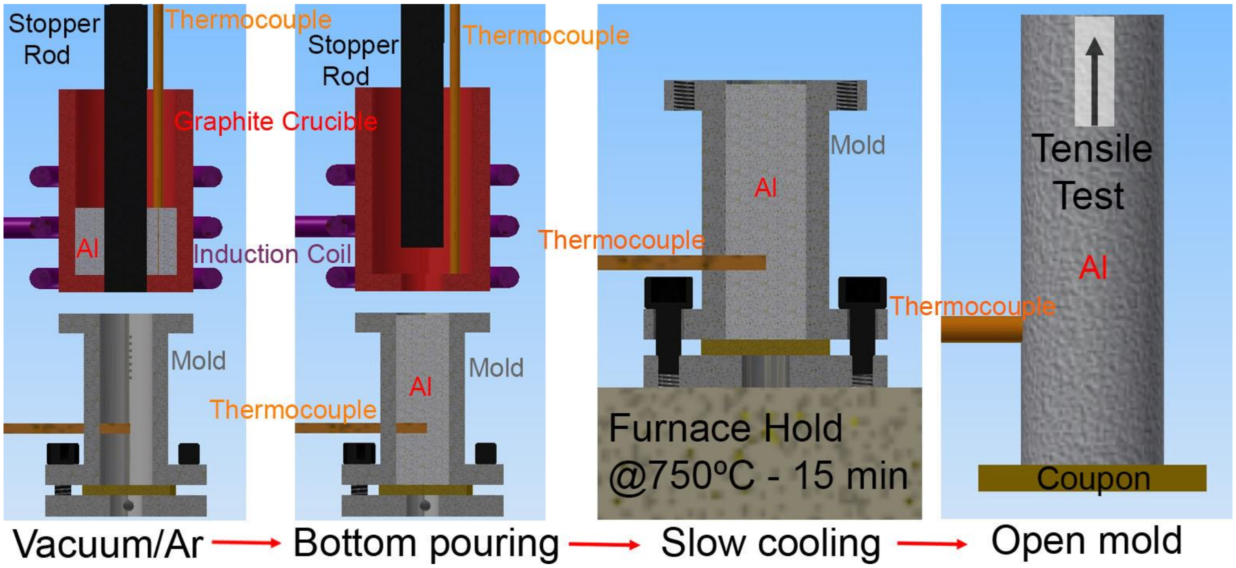

Figure 37.2: Modified aluminum adhesion test and the sequence of the test for a 1x 1 inch coupon.

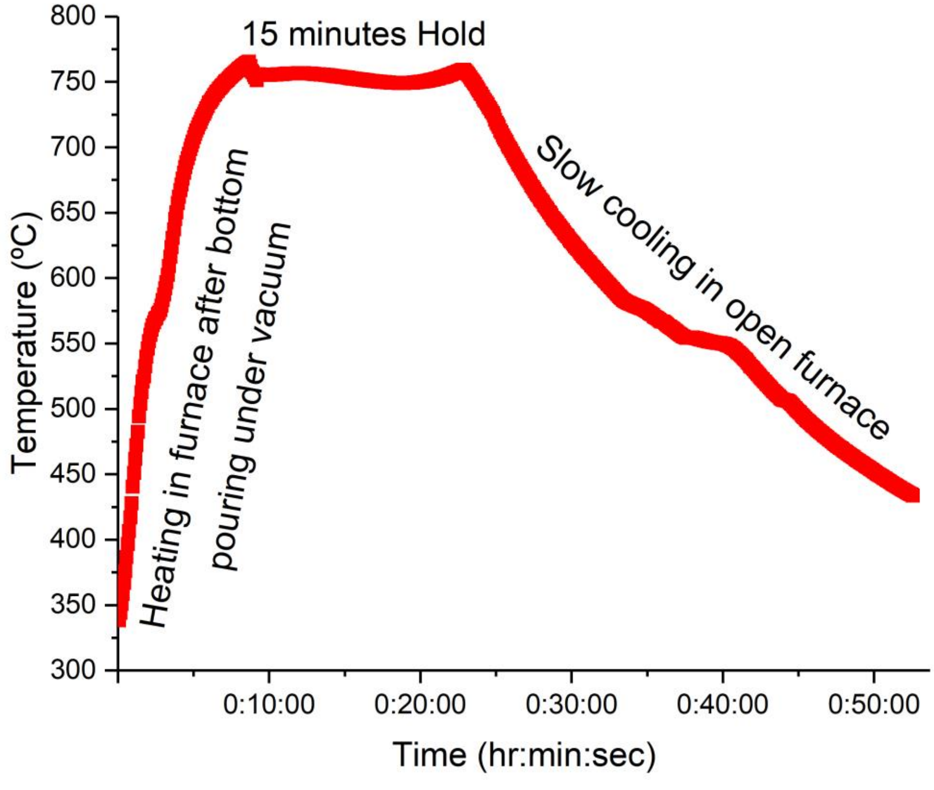

Figure 37.3: Example of the profile of temperature vs time during the test.

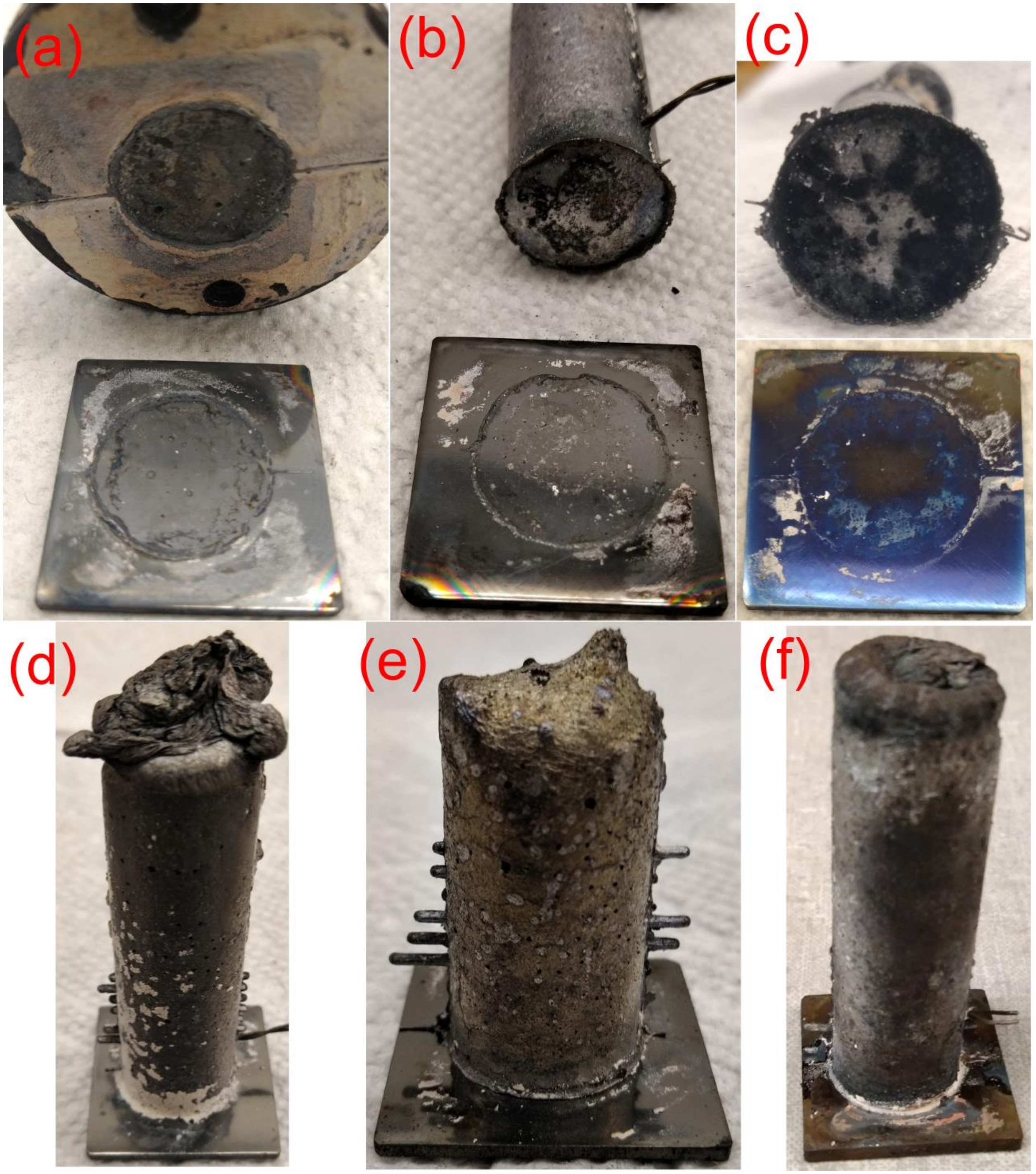

Figure 37.4: Modified AAT results (a) AlCrN coating showing no reaction at the surface, (b) AlCrN-PTCD coating with a significant amount of reacted area at the surface, (c) AlCrCN coating showing no reaction but a change to a blue color, (d) AlCrN coating from supplier 2 that stuck, (e) AlCrN+ coating from supplier 2 that stuck, and (f) test performed on a polished un-coated H13 steel substrate.

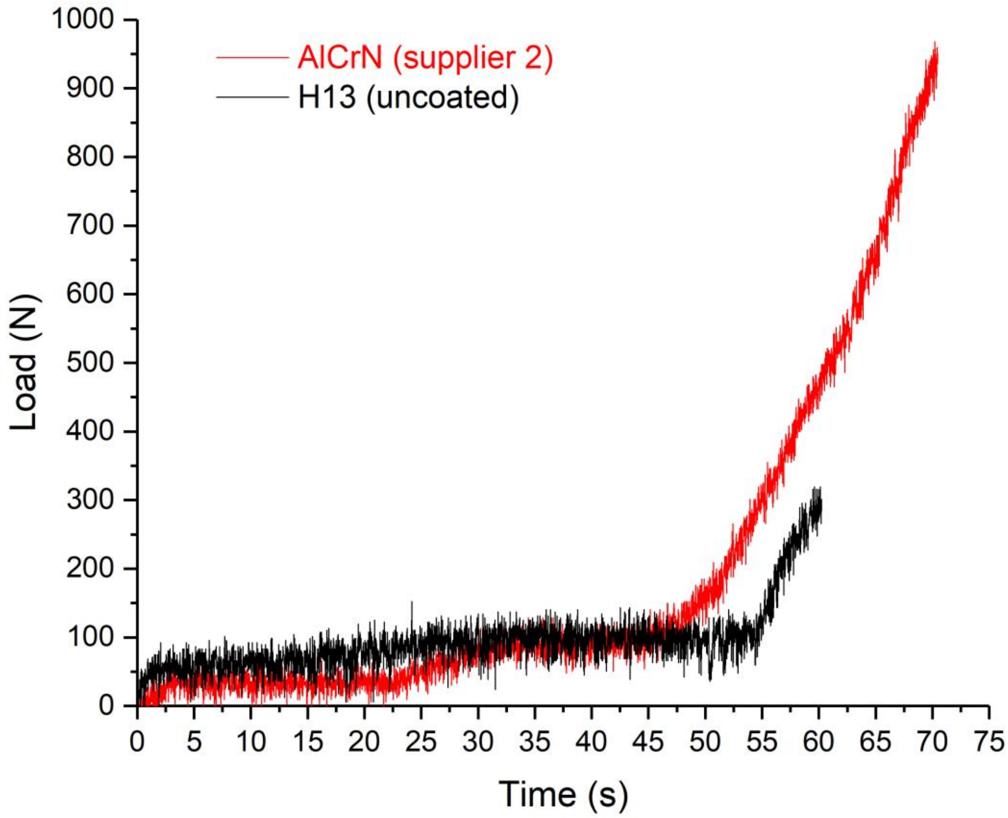

Figure 37.5: Results of the tensile test of H13 and AlCrN from supplier 2 after modified AAT.

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

Figure 37.1: List of PVD coated samples.