37.0 ADVANCED ENGINEERED COATINGS WITH EXTENDED DIE LIFE FOR TOOLING

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This project started in Fall 2018 and is supported by the Defense Logistics Agency. The research performed during this project will serve as the basis for an 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 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, and the extension in die life, resulting in lower per-part costs. This also leads to a significant improvement of the quality of die castings, allowing them to be used in higher performance applications. This 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

A prior project was performed at the Colorado School of Mines (Mines) by Wang [37.1], where a variety of PVD coatings were evaluated, with the best coating identified in that project being AlCrN. This 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%. The goal of this new 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 the previous report [37.3]. The key features of this improved test include i) use of bottom pouring to avoid incorporation of the floating oxide layer between the material coupon and the cast aluminum, ii) use of induction heating to quickly heat and melt the aluminum alloy, iii) control and recording of the temperature of the material coupon during the test and iv) ability to rapidly place 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. Another focus was on identifying suitable simulation software to make predictions regarding interdiffusion between the materials and the molten aluminum. The DICTRA module of Thermo-Calc, based on the CALPHAD method, appears to be a promising tool.

37.3 Recent Progress

37.3.1 Aluminum adhesion test using the apparatus developed in this project

In the previous report [37.3], a modified apparatus for performing the aluminum adhesion test was presented. In summary, this modified test focused on the use of bottom pouring and induction heating to melt the aluminum alloy, and a schematic representation of this setup is shown in **Figure 37.1**. The sequence of operations used with this new setup involves quickly heating and melting the Al alloy via induction (**Figure 37.1 a**), which takes about 10 minutes, bottom pouring the molten aluminum from the upper to the lower chamber (**Figure 37.1 b**), separating the lower portion of the apparatus (**Figure 37.1 c**), and placing the lower portion (containing the molten aluminum and steel coupon) into a resistance furnace preheated to 700 ºC and holding for the desired period of time (**Figure 37.1 d**). The temperatures of both the aluminum alloy and the H13 coupon are monitored during this process.

The material substrate used in the initial tests was un-coated H13 steel utilizing different surface finishes: ground with SiC to 120 mesh, to 600 mesh and polished to a 1 μm finish. Three different holding times at 700 ºC in the resistance furnace were evaluated: 15 minutes (as used by Wang in the previous project), 1 hour and 4 hours. The temperature profiles collected during the tests are shown in **Figure 37.2**, where it can be seen that this new test has good repeatability and robustness.

The results of the test for the different surface finishes are shown in **Figure 37.3**, **Figure 37.4** and **Figure 37.5** for the 120 mesh, 600 mesh and 1 μm polished, respectively. For the 120 mesh **Figure 37.3**, the solidified aluminum adhered to the substrate for all holding times. For the 600 mesh surface finish (**Figure 37.4)**, the holding time of 15 minutes exhibited less soldering than for holding times of 1 h and 4 h, where a significant reaction appears to have occurred between the molten aluminum and the steel substrate for the longer holding times. For the 1 μm polished surfaces (shown in **Figure 37.5)**, the results are similar to the holding times of 1 h and 4 h for the 600 mesh sample shown in **Figure 37.4 b** and **Figure 37.4 c**, where the entire cross section of the steel substrate was covered by the solidified molten aluminum. One possible explanation for having higher adhesion for the 120 mesh samples is due to the increased area of the rougher surface, increasing the contact area between the aluminum and the steel. For the finer surface finishes, one possible reason why the aluminum was not soldered to the steel after cooling to room temperature is related to the difference in the coefficient of thermal expansion (CTE) between the H13 steel, the aluminum A380 and the brittle intermetallic phases that likely formed in the reaction zone between the steel and aluminum. This difference in CTE can produce cracking and separation during cooling from 700 ºC to room temperature, which was observed by Wang in the earlier study [37.1]. It is planned to use metallographic techniques to characterize the phases formed on the contact surface of the tested steel substrates.

37.3.2 Additional modifications to the test mold

As seen in **Figure 37.3**, the graphite mold used to hold the cast aluminum at 700 ºC tends to burn and decompose when held for extended period at such high temperatures, potentially contaminating the aluminum melt. To avoid this problem, the graphite was replaced with a permanent H13 mold design that could be used in this modified test apparatus. The testing approach used with the new mold will follow the same sequence as before. The H13 mold is a two-part design that will be sprayed with a BN coating prior to each test to prevent the aluminum alloy from reacting with the steel. Another advantage of the new H13 mold is the possibility of testing not only flat material coupons (**Figure 37.6**), but also rods (**Figure 37.7**), without any major design changes. Since this concept uses a permanent metallic mold, another possibility that arises is the pre-heating of the mold and material coupon to the desired temperature before being placed below the induction heated crucible to receive the molten aluminum by bottom pouring. Also, by the reduction of total mass of the apparatus, a faster heating rate is expected when the assembly is placed inside the pre-heated resistance furnace, allowing the tested material to reach 700 ºC in a shorter time (less than 13 minutes). This modified test apparatus incorporating the two-part H13 mold will be tested in the following months.

37.3.3 PVD coating selection

A number of PVD coatings had been selected to be studied and the list is shown in **Table 37.1**. Some of the coatings, AlCrN and CrN were tested by Wang in the previous project [37.1] and will be tested in the current study for baseline comparison. The selection criteria at this step was based on commercial availability and good reports found in literature for those coatings in aluminum die casting or in related industries. The previous project essentially focused on nitride coatings, but in the current study, carbides, carbonitrides and complex C-containing and Si-containing coatings will also be evaluated. For two coatings (AlCrN and TiCN) the effect of surface finish will be examined, while all other coatings will be evaluated in their as-deposited condition. The coatings have been deposited on polished down to 1 μm flat 1” x 1” material coupon and 3/8” rods. It is anticipated that this broader spectrum of coating composition and complexity will provide a better guide to finding the best working layer for lube-free aluminum die casting applications.

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 test apparatus. Tests will be performed on the list of PVD coated samples.
- Start a characterization of the PVD coated samples using a range of techniques.
- Start the 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. Midosn, M. J. Kaufman. ADVANCED ENGINEERED COATINGS WITH EXTENDED DIE LIFE FOR TOOLING. CANFSA Report, Project 37, March 25, 2019.

37.6 Figures and Tables

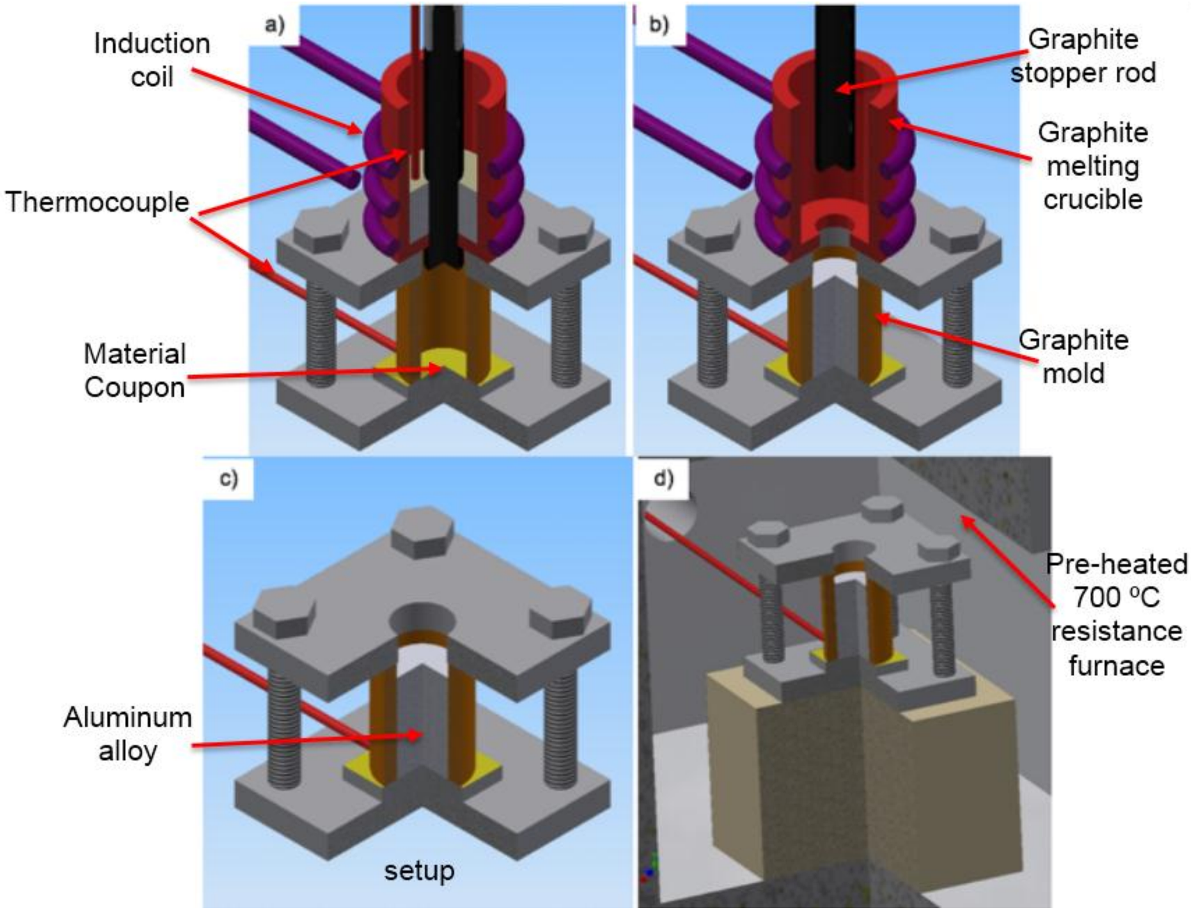

Figure 37.1: Improved test apparatus: a) induction melting of aluminum alloy, b) bottom pouring by lifting the stopper rod, c) removing the lower portion of the apparatus from the induction melter, and d) placing the apparatus inside a resistance furnace preheated to 700 ºC.

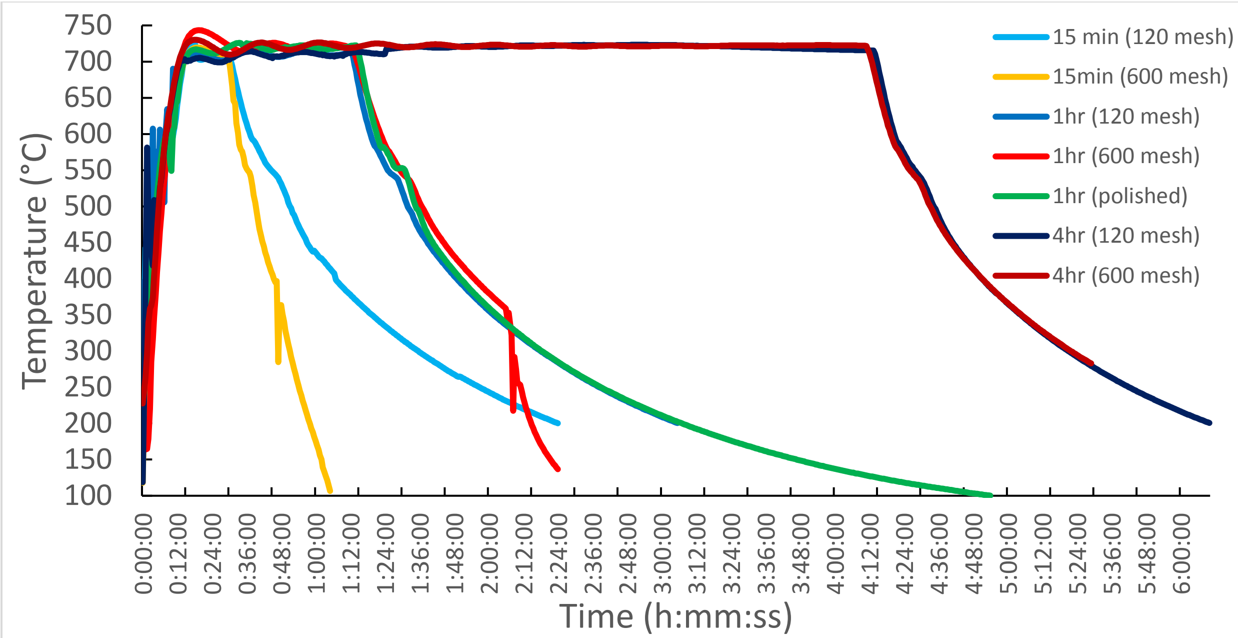

Figure 37.2: Temperature profiles recorded during the tests.

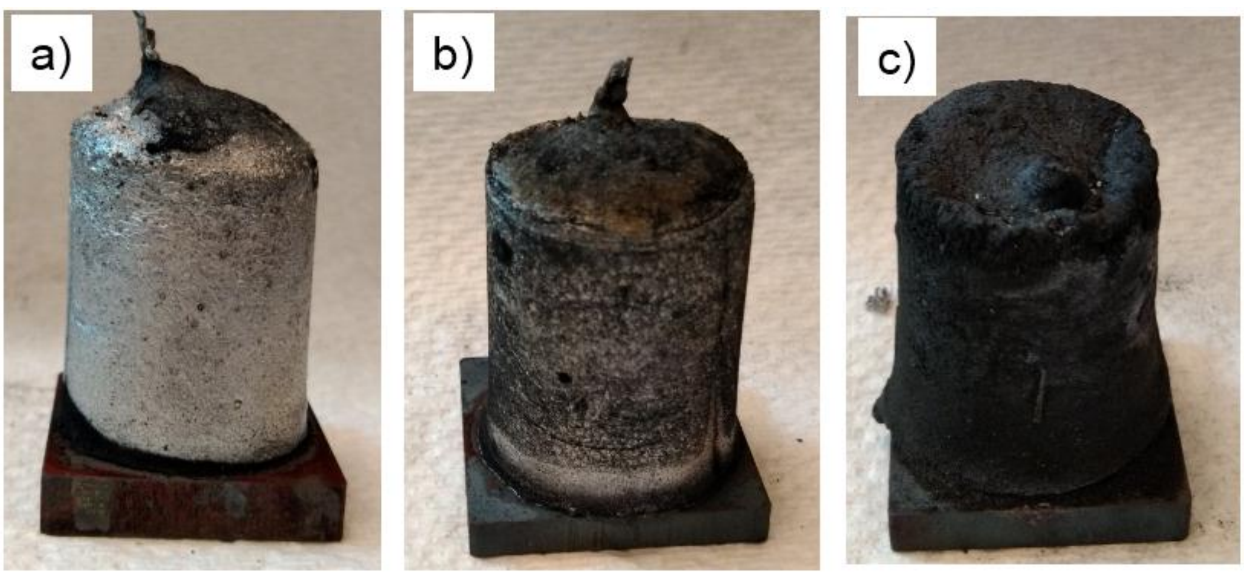

Figure 37.3: Al castings on H13 steel substrates ground to 120 mesh after holding times at 700 ºC of a) 15 minutes, b) 1 h, and c) 4 h.

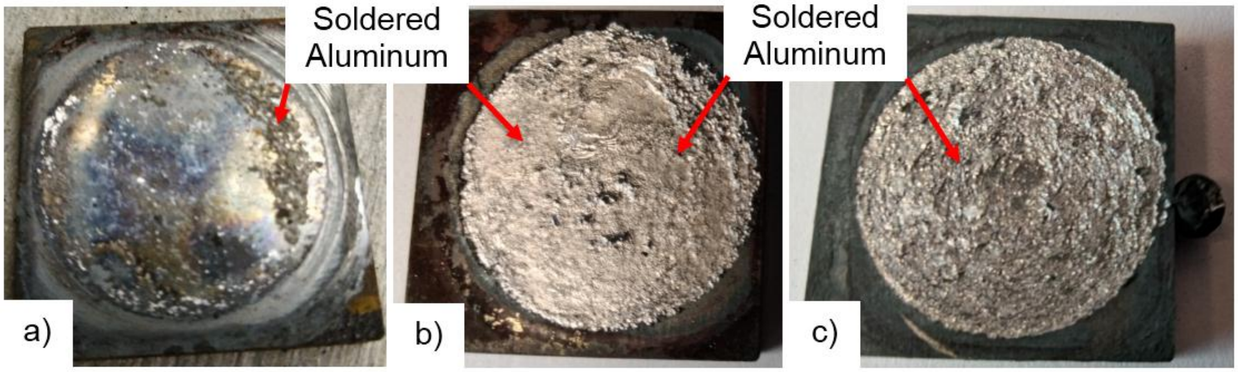

Figure 37.4: Surface condition of the H13 steel substrates (after Al casting removal) ground to 600 mesh after holding times at 700 ºC of a) 15 minutes, b) 1 h, and c) 4 h.

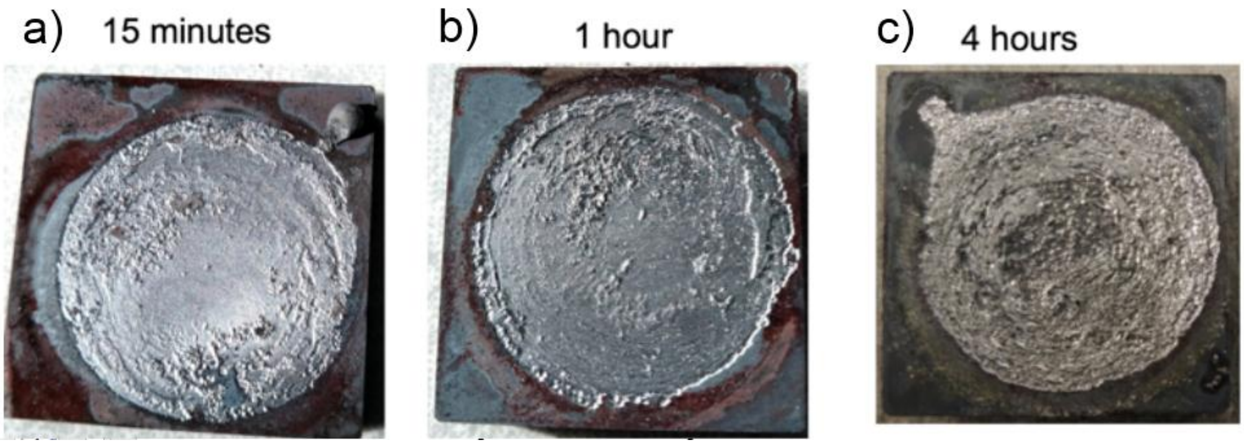

Figure 37.5: Surface condition of the H13 steel substrates (after Al casting removal) polished with 1 μm after holding times at 700 ºC of a) 15 minutes, b) 1 h, and c) 4 h.

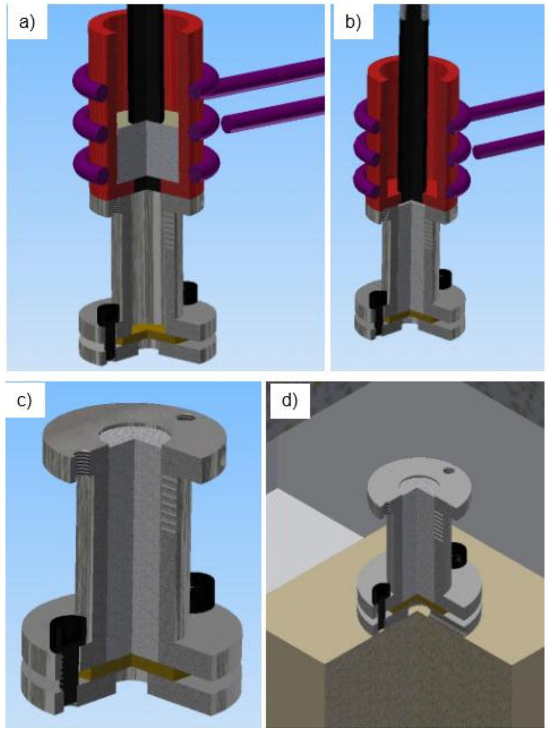

Figure 37.6: Schematic of new H13 mold set up for testing a flat material coupon: a) induction melting of Al alloy; b) bottom pouring by lifting the stopper rod; c) removing the lower portion of the apparatus and d) placing inside the resistance furnace preheated at 700 ºC.

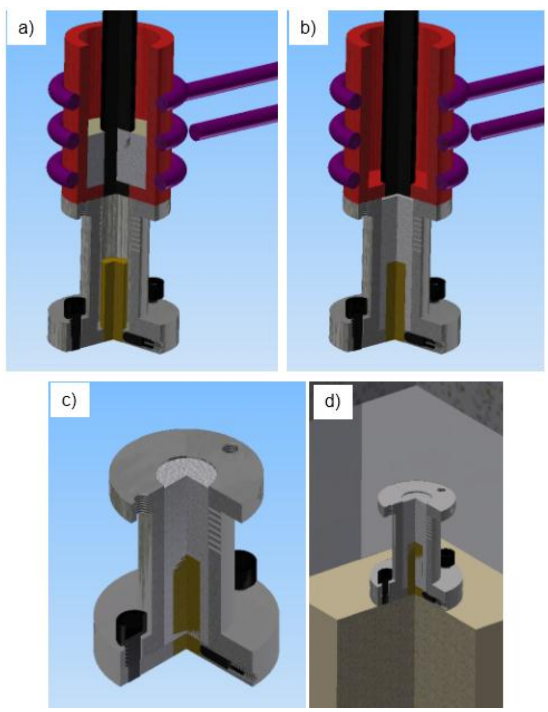

Figure 37.7: Schematic of new H13 mold testing a rod material coupon: a) induction melting of Al alloy; b) bottom pouring by lifting the stopper rod; c) removing the lower portion of the apparatus and d) placing inside the resistance furnace preheated at 700 ºC.

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

Table 37.1: List of PVD coatings that was selected to be studied in the project.