37.0 ADVANCED ENGINEERED COATINGS WITH EXTENDED DIE LIFE FOR TOOLING

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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 via an 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 parts 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 has 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 the PVD coatings.

37.3 Recent Progress

37.3.1 Development of an Improved Aluminum Adhesion Test and initial tests

In the previous project performed by Wang, a laboratory test (called the Aluminum Adhesion Test, or AAT) was developed to evaluate the level of soldering and adhesion of liquid metals onto a range of coatings [37.3]. During these tests, it became clear that one of the factors that any test must ensure is good contact between the molten alloy and the substrate. Therefore, it is important to prevent the oxide that forms on the surface of the molten aluminum from becoming trapped between the liquid alloy and the substrate during pouring, thus affecting the level of adhesion. The AAT approach developed by Bo Wang used conventional pouring, and so it was difficult to assure that any oxide on the upper surface of the molten bath did not become incorporated between the molten alloy and substrate during pouring. This drawback of the previous test served as a starting point to develop an improved AAT incorporating three important parameters – (1) avoiding oxide incorporation, (2) developing better contact between the molten alloy and substrate, and (3) improved temperature control.

The concept of an improved AAT centered on the use of bottom pouring to meet these three requirements. An initial system was developed and is shown in **Figure 37.1**. The apparatus is initially at room temperature, and small pieces of aluminum alloy A380 are placed in the upper crucible, while the material coupon (coated or uncoated substrate) is placed at the bottom. The entire apparatus is placed in a room temperature resistance furnace and heated to 700 ºC. Once at the target temperature, the stopper rod is extracted to provide bottom pouring and the molten aluminum falls from the upper to the lower chamber, making contact with the substrate. The entire apparatus can then be held at 700 ºC for a controlled time period, before being removed and cooled to room temperature.

Testing of this first system identified some problems, as illustrated in **Figure 37.2** and **Figure 37.3**. The testing system uses graphite crucibles such that, during the long heating period in the resistance furnace (about 50 minutes), the surface of the crucible oxidizes and graphite powder falls from the crucible, and as shown in **Figure 37.2** collects on the upper surface of the steel coupon. This layer of graphite powder prevents intimate contact between the molten aluminum and substrate making the test less robust. This problem is exacerbated with multiple uses of the crucibles, causing the walls of the graphite crucible to become so thin that fracturing eventually occurs (see **Figure 37.3)**.

To address the problems identified with this first testing system, the modified testing procedure shown in **Figure 37.4** was developed. To reduce heating time, and therefore the amount of oxidation of the graphite crucible, an induction melting procedure is used. This also has the advantage of separating the melting process from the holding time inside the resistance furnace. The sequence of operations used with this new setup involves quickly heating and melting the Al alloy via induction (**Figure 37.4a**), which takes about 10 minutes (vs. ~50 minutes using the resistance furnace approach), bottom pouring the molten aluminum from the upper to the lower chamber (**Figure 37.4b**), separating the lower portion of the apparatus (**Figure 37.4c**), and placing the lower portion (containing the molten aluminum and steel coupon) in a resistance furnace preheated to 700 ºC and holding for the desired period of time (**Figure 37.4d**). The temperatures of both the aluminum alloy and the H13 coupon are monitored during this process. Initial tests have been performed, and many of the problems identified with the first testing system have been eliminated.

The material substrate used in these initial tests was bare H13 steel, ground with SiC to a 600 mesh surface finish, and three different holding times at 700 ºC in the resistance furnace were evaluated: 15 minutes (as used by Wang in the previous project), 1 h and 4 h. An example of the temperature profile collected during the test using a 4 h holding time is shown in **Figure 37.5**, where it can be seen that, after bottom pouring and placing the unit consisting of the material substrate and mold into the resistance furnace preheated to 700 ºC, it took around 13 minutes for the substrate to reach the 700 ºC target temperature, at which point the holding time began.

The surfaces of the H13 samples after testing are shown in **Figure 37.6**. None of the holding times resulted in the solidified aluminum being stuck to the substrate, although it is clear that for holding times of 1 h and 4 h, significant reaction appears to have occurred between the molten aluminum and the steel substrate. One possible reason why the aluminum was not soldered to the steel is related to the difference in the magnitude of the coefficients of thermal expansion between H13 steel, aluminum A380 and the brittle intermetallic phases that likely formed in the reaction zone resulting in cracking and separation during cooling from 700 ºC to room temperature. A similar result was observed by Wang [37.1]. Metallographic techniques will be used to examine the phases formed on the upper surface of the steel substrate samples shown in **Figure 37.6.**

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 induction melting system (second testing procedure). Tests will be performed using H13 substrates with polished surfaces, to evaluate the influence of surface finish on soldering behavior.
- Start experimental work using the induction melting system with PVD coated substrates.

- Start the characterization of the soldered surfaces to understand solder and adhesion, inter-diffusion, phase formation, and defects.

37.5 References

[31.1] 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.

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

[31.3] B Wang, G. R. Bourne, A. L. Korenyi-Both, A. K. Monroe, S. P. Midson, M. J. Kaufman, Method to evaluate the adhesion behavior of aluminum-based alloys on various materials and coatings for lube-free die casting, Journal of Materials Processing Technology, 237, 2016, 386–393.

37.6 Figures and Tables

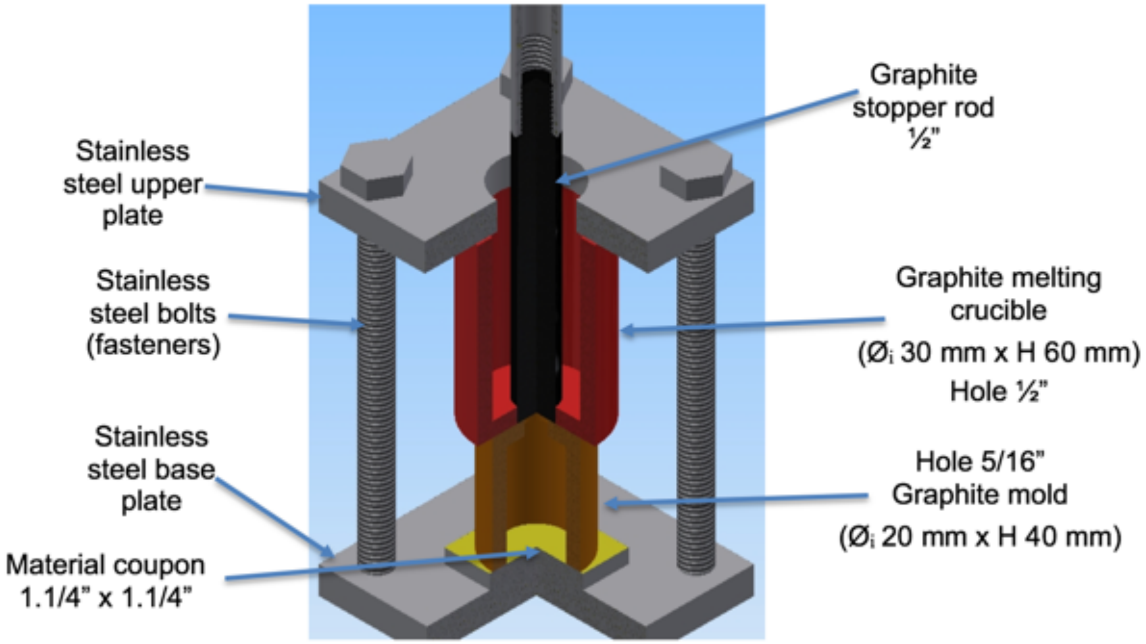

Figure 37.1: First iteration of the improved Aluminum Adhesion Test, where the entire system is placed inside a resistance furnace.

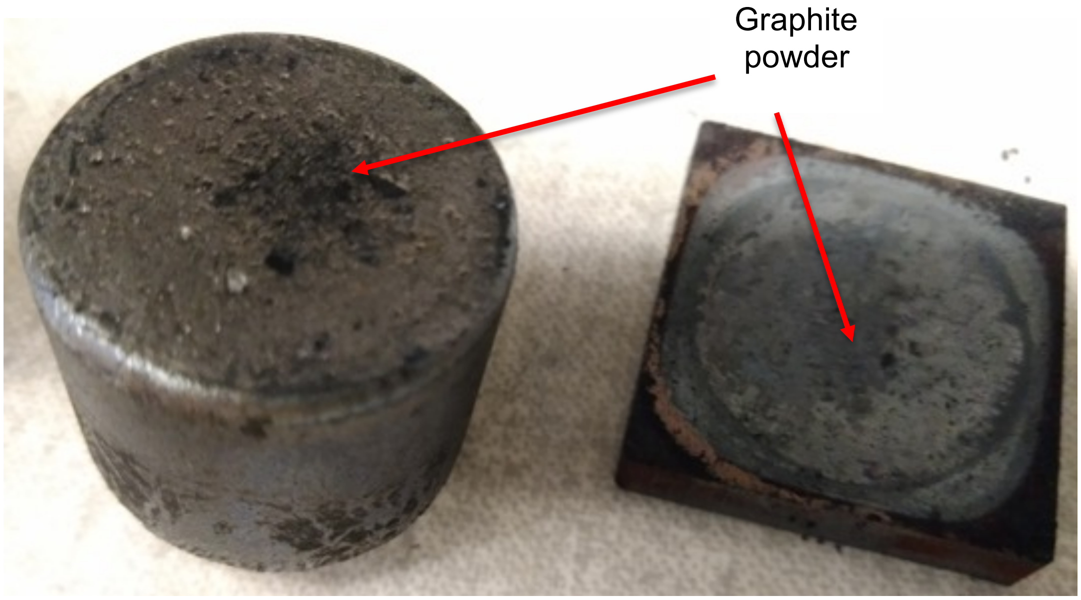

Figure 37.2: Black graphite powder layer on top of steel substrate. This layer prevented good contact with the molten aluminum alloy in the first iteration of the improved AAT.

Figure 37.3: Photo of the graphite crucible after heating in air for several hours at 700 ºC in a resistance furnace using the first iteration of the improved AAT.

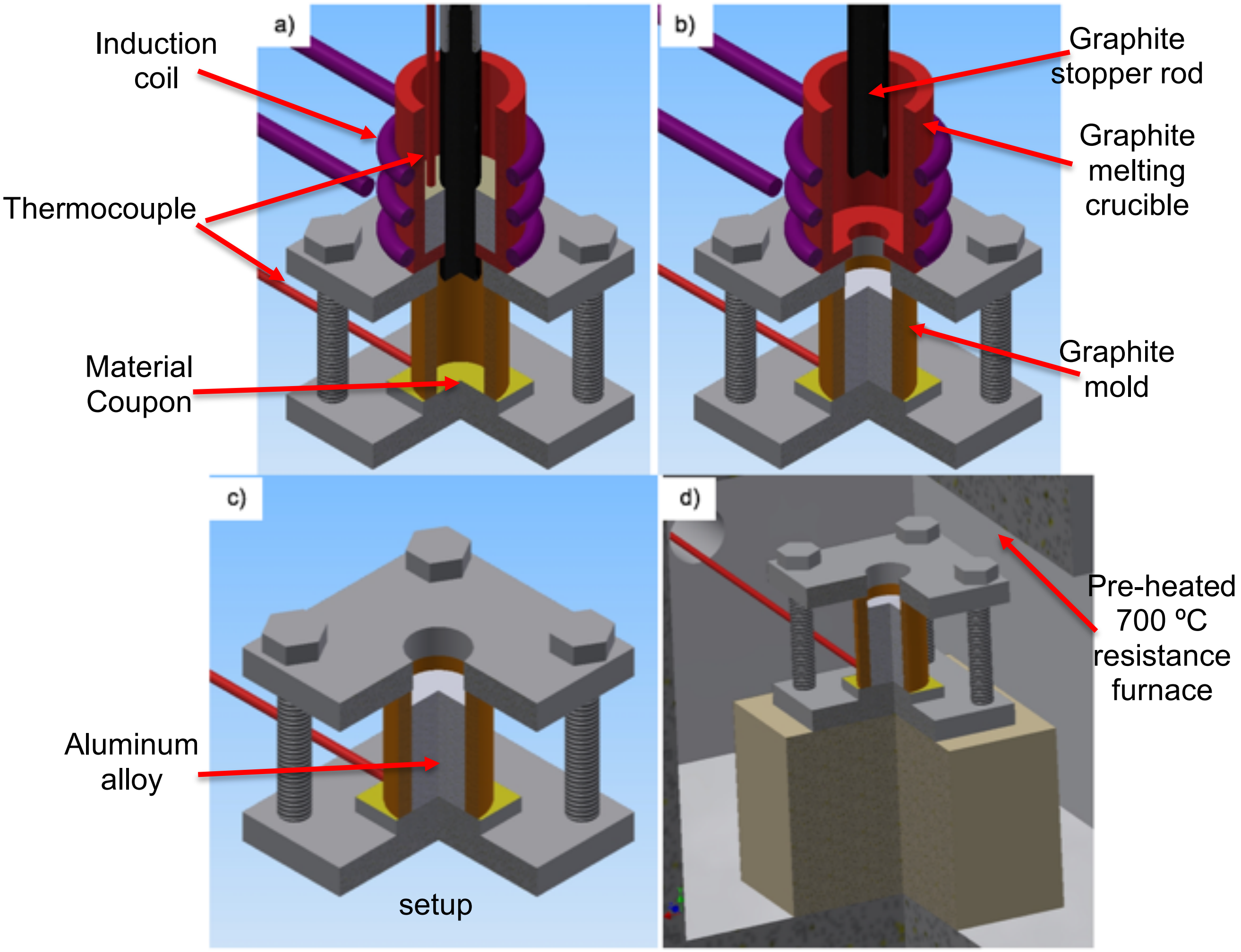

Figure 37.4: Second iteration of the improved ATT test apparatus: (a) induction melting of aluminum alloy, (b) bottom pouring by lifting the stopper rod, (c) removing the lower portion of the apparatus from the induction melter, and (d) placing the apparatus inside a resistance furnace preheated to 700 ºC.

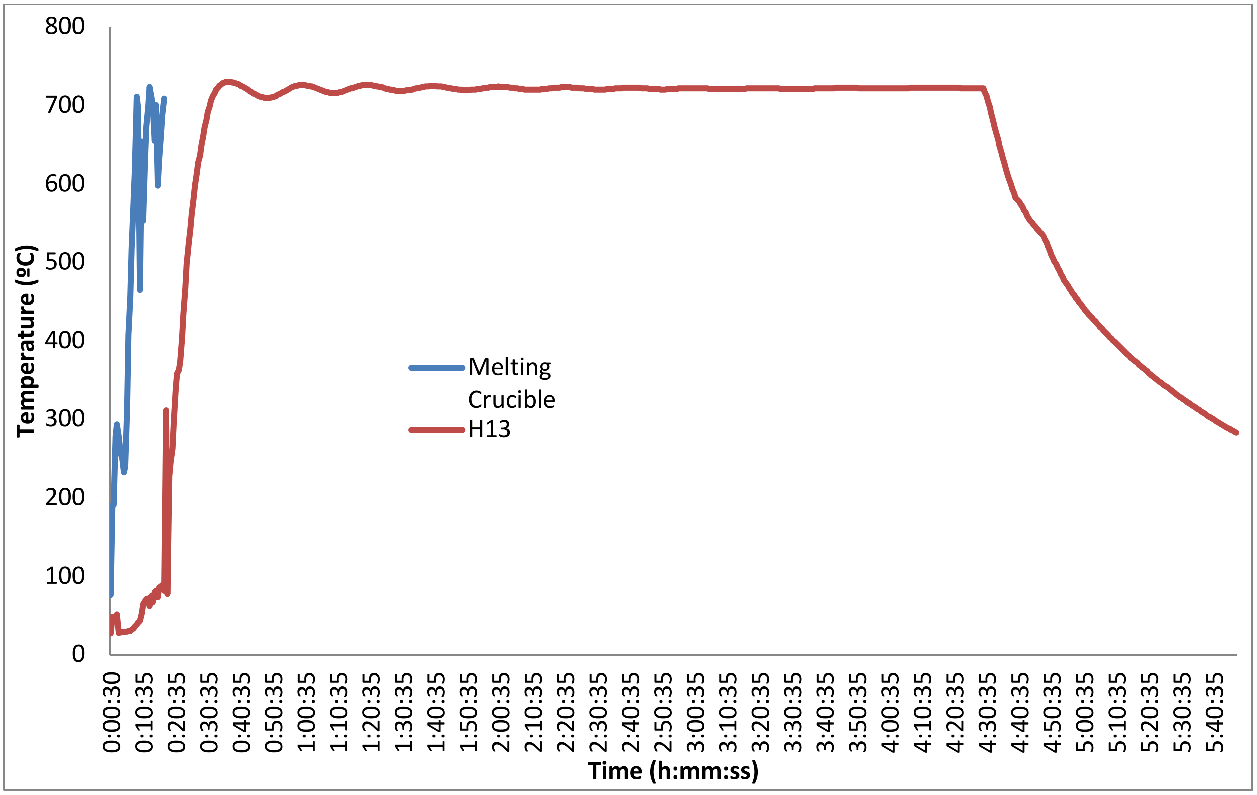

Figure 37.5: Temperature profiles for the four hours holding time experiment showing data collected from the melting crucible and the H13 material coupon.

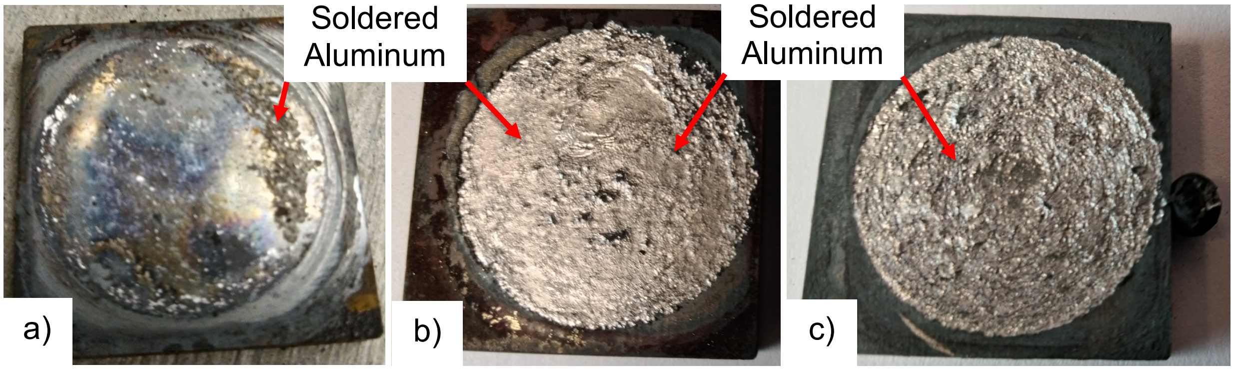

Figure 37.6: Surface condition of the bare H13 steel substrates after holding times at 700 ºC of (a) 15 minutes, (b) one hour, and (c) four hours.