# Casting Solutions for Readiness Lube-Free Die Casting

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#### Abstract

During high-pressure aluminum die casting, a liquid lubricant is sprayed on the die surface prior to each shot to help release the casting, cool the die, and prevent soldering from occurring. However, spraying a large amount of liquid lubricant each shot not only reduces the quality of the castings, adds time to the casting cycle, and increases the cost of producing castings, it also generates effluent that has environmental ramifications. The overall objective of this work was to develop permanent hard coatings on the surfaces of die casting dies that result in no aluminum adhesion in the absence of using liquid-based organic lubricants. This new concept is called lube-free die casting. To achieve this goal, a simple laboratory method called the aluminum adhesion test (AAT) was developed to provide a quick quantitative evaluation of the adhesion/soldering strength of the cast alloy to various substrates and/or coatings. A number of hard coatings have been evaluated using the AAT. Several coatings were identified to have zero or negligible adhesion strength between the solidified alloy and the coating. To assess the efficacy of the candidate coatings screened by the AAT in preventing such alloy adhesion under actual die-casting conditions, both a coated die insert and a fully-coated die cavity have been tested in a series of four plant trials.

The results of laboratory testing and initial plant trials suggested that an AlCrN coating applied to the die surface has the potential for dramatically reducing die lubricant use. To date, more than 19,000 castings have been produced using a die coated with AlCrN, with die spraying reduced significantly (83-to-92% less) compared with the amount of spray used for an uncoated version of the same die. In addition to reducing the amount of lubricant used, the cycle time was reduced by about 12%, as the time required to spray the die was eliminated. In addition, T6 heat treatments trials have indicated that the reduction of spray dramatically decreased the amount of entrapped gases (porosity) in the castings, and although the reduction in entrapped gasses was not quantitatively measured in this study, porosity reduction due to reduced spraying is estimated to be greater than 50%. Finally, as aluminum die casting dies typically last longer than 100,000 shots, the plant trials have not progressed sufficiently to evaluate the impact of reduced die spraying on heat checking and die life. However, the metric in this project was to increase die life by 15%, and based on laboratory testing performed at Case Western Reserve University to evaluate the impact of die spray on die life, the 83-to-92% reduction in spray achieved in the plant trials should provide a die life extension of significantly more than 15%.

The results from the laboratory studies and the plant trials have been presented at the last two NADCA Congresses, and both congress sessions were well attended by industry personnel and generated a lot of interest from attendees. In addition, the results from this research have been incorporated into a new NADCA booklet and webinar series entitled *Applications of Surface Engineering for Die Casting Dies*.

# 1. Introduction

High-pressure die casting (HPDC) is a near-net shape casting process, where liquid metal (typically an aluminum alloy) is injected into re-usable steel dies using high injection speeds and high pressures. Die casting is often the lowest-cost of all of the casting processes.

In high-pressure die casting, lubricants are sprayed onto the casting die surface prior to each shot, to prevent the liquid aluminum from soldering and adhering to the die [1-3]. Empirical data from various sources suggest that aluminum castings will stick to an uncoated steel die after only a few shots if lubricants are not used. The use of die lubricants, however, results in a number of undesirable outcomes during die casting, such as:

- Lubricants can reduce the quality of die castings, as the organic components of the lubricants can vaporize when heated by the liquid metal filling the cavity, resulting in the production of large amounts of gasses that can become trapped in the casting, increasing the level of residual porosity.
- Utilizing lubricants increases the cost of the die casting process, as the purchase price of lubricants can be high (up to 2.5% of the overall casting cost).
- The life of the die is compromised by the thermal fluctuations (cooling) caused by use of die lubricants. Laboratory data suggest that die life could be extended significantly by the elimination of lubricant spray.
- The use of die lubricants creates expensive housekeeping issues, as effluents in the air created from overspray have to be repeatedly cleaned from the die casting machine and surroundings.

The overall objective of this project was to develop permanent thin-film coatings for die casting dies to which the aluminum die casting alloys do not stick during solidification, with the long-term objective of circumventing the need to use liquid-based organic lubricants prior to each shot. This concept is called lube free die casting.

The coatings examined in this study were produced by PVD (Physical Vapor Deposition). PVD is an atomistic film deposition process, where the material to be deposited is physically vaporized from a solid source in the form of atoms or molecules. For die casting applications, metallic vaporized atoms combine with nitrogen atoms introduced into the deposition chamber, before being deposited in a controlled manner onto the substrate (the die casting die). Coatings produced by the PVD process are typically thin (around  $1-5 \mu m$ ), very hard, and exhibit excellent wear resistance.

PVD-type hard coatings have been used to protect core pins and inserts in aluminum high pressure die castings for decades [1, 4-8]. An ideal coating for die casting applications must exhibit excellent adhesion to the substrate (the casting die), good mechanical and tribological properties, high temperature oxidation resistance, and chemical inertness to, or be non-wetted by, liquid aluminum [2.9-12].

To identify optimum coating compositions, sessile drop tests have historically been widely accepted by researchers as a means to study the wetting behavior of molten metal and alloys on solid substrates [3, 12-15]. The sessile drop test consists of placing a small sample of a solid alloy, e.g. A380 aluminum alloy, onto a test substrate and then increasing the system temperature until the alloy melts and reaches its

equilibrium geometry on the solid substrate. The contact angle between the molten drop and the substrate is measured and then correlated to adhesion through the Young-Dupré equation [3, 14, 15]. However, early testing performed as part of this study showed that that the A380 alloy did not ideally spread on the substrate surface due to a solid oxide shell that existed on the aluminum sample's surface. This precluded making any meaningful contact angle measurements and, as a result, detailed sessile drop studies were not pursued as part of this project.

A relatively simple dipping test [11, 12, 19] has also been commonly used in die casting research to study aluminum adhesion and soldering behavior. For the dipping test, a bare or hard coated core pin is inserted into a container of molten aluminum alloy and then withdrawn and allowed to cool in air. In this study, dipping tests were also evaluated using coated and uncoated H13 core pins. Dipping tests conducted under atmospheric conditions are obviously simple and direct, but are not quantitative in nature and the results are often difficult to interpret because of the physical adhesion that occurs due to aluminum shrinkage around the pin. Therefore, such tests were also not pursued in this study.

Since neither of these test methods were capable of effectively evaluating the wetting/adhesion of aluminum die casting alloys to the types of hard coatings developed for die casting applications, a simple and more direct approach was developed as part of this study, which was named the aluminum adhesion test (ATT) [22]. A variety of surface treatments were investigated and the aluminum adhesion to both uncoated and coated flat H13 steel coupons was quantified using this approach. The ultimate goal was to better understand the interactions between various substrates and the molten aluminum alloys, so that a more reliable method for predicting performance in actual die casting operations could be developed. A coating, identified from using the AAT, was chosen for trials in a die casting plant.

#### 2. Experimental Procedure

A schematic drawing of the aluminum adhesion test (AAT) is shown in Figure 1. The test used a coupon of H13 steel approximately one inch by one inch by <sup>1</sup>/<sub>4</sub>-inch thick. The upper surfaces of the coupons were polished to a 1  $\mu$ m finish, and tested either un-coated or after being PVD coated on the polished face. The test involved heating a piece of A380 aluminum alloy and a boron nitride tube in a furnace preheated to 700°C. An H13 steel test coupon (coated or uncoated) was then transferred into the hot zone of the furnace. The preheated tube was placed on the upper surface of the coupon, and the molten alloy then poured into the tube, and the coupon, tube and molten alloy were soaked in the preheated furnace at 700°C for 15 minutes. After turning the furnace off, the door was opened, and the combination allowed to slowly cool to room temperature. More details about this test have been published in reference [22].



Figure 1: Schematic drawing of the aluminum adhesion test

After the sample was cooled to room temperature, the tube was removed and a hole drilled in the top of the cast aluminum cylinder. The coupon and cast cylinder were installed into a specially designed rig, and a tensile testing machine was used to obtain a quantitative measurement of the load necessary to break the bond between the cast aluminum and the substrate.

Both bare H13 steel as well as PVD coated steel coupons with a range of hard ceramic coatings were evaluated using this aluminum adhesion test. Various coatings were obtained from a range of commercial companies (identified as Suppliers 1-6). In addition, several coatings were produced using the laboratory-scale PVD coater at the Colorado School of Mines (CSM). For most of the coatings, only a single aluminum adhesion test was performed. But for several coatings of interest, multiple tests were performed. For example, for the AlCrN coating from Supplier 1, the aluminum adhesion test was repeated at least 10 times.

The coatings were also evaluated using a range of characterization techniques. The coating hardness was measured using an MTS nanoindenter. In addition, the coating thicknesses were measured from scanning electron microscope (SEM) images of cross-sections through the coatings. The coating compositions (metallic components) were also measured using Energy Dispersive Spectroscopy (EDS) on the same cross sections. Finally, both SEM and TEM methods were used to examine the interfaces between the solidified aluminum alloy and the substrate.

The best coating was chosen for a series of plant trials. The plant trials were performed at the Mercury Castings facility in Fond du Lac, WI.

#### 3. Results and Discussion

# 3.1 Laboratory Testing

The characteristics of the various PVD coatings evaluated in this study, including hardness, Young's modulus, and coating thickness are summarized in Table 1. Also listed in Table 1 are the measurements of the "breaking strength" between the as-solidified aluminum and the various substrates using the aluminum adhesion test (ATT) method described above. The results show that the breaking strength varied from 0 MPa (no sticking) to 2.54 MPa (sticking). Note that this value is significantly lower than the ultimate tensile strength of the A380 die casting alloy (reported to be about 46 ksi [18].

The results in Table 1 indicate that three different coatings (AlCrN, AlTiN and CrWN) from three separate suppliers exhibited zero breaking strength after the aluminum adhesion test. For example, a steel coupon coated with AlCrN from Supplier 1 is shown both before and after the AAT in Figure 2a and 2b. As can be seen, the cylinder did not stick to the coupon, nor was there evidence on the coupon's surface of the contact with the molten A380 alloy. For comparison, a steel coupon coated with AlCrN from Supplier 5 is shown in Figure 2c, and in this case the cast aluminum did stick to the coupon.





As listed in Table 1, AlCrN coatings were obtained from two separate companies (Supplier 1 and Supplier 5). The coatings from Supplier 1 always exhibited non-sticking behavior, whereas those from Supplier 5 did not. When the AlCrN coating from Supplier 5 was tested in the as-deposited condition, it exhibited a breaking strength of 0.04 MPa. However, when the AlCrN coating from Supplier 5 was tested after mechanical polishing, its breaking strength increased to ~1.30 MPa. As can also be seen in Table 1, CrN, a common coating used in the die casting industry for cores and small inserts, also exhibited sticking behavior in the aluminum adhesion tests performed in this study.

Mechanical Properties Busching									
No.	Material	Producer	Method	[GPa]		Composition	Thickness	Breaking	
				Hardness	Young's Modulus	[at.%]	[µm]	[MPa]	
1	AlCrN	Supplier 1	CAE	27.5 (±1.8)	358 (±8)	$Al_{0.67}Cr_{0.33}N_x$	1.7	0	
2	AlTiN	Supplier 1	CAE	21.5 (±1.7)	306 (±27)	$Al_{0.68}Ti_{0.32}N_x$	1.5	0	
3	CrWN	Supplier 2	CAE	20.1 (±1.9)	298 (±13)	$Cr_{0.97}W_{0.03}N_x$	6.7	0	
4	AlTiN	Supplier 3	CAE	28.0 (±1.9)	344 (±14)	Al <sub>0.62</sub> Ti <sub>0.38</sub> N <sub>x</sub>	3.3	0*	
15	ZrN	Supplier 1	CAE					0*	
5	TiAlN	Supplier 3	CAE	29.7 (±2.6)	384 (±25)	Ti <sub>0.56</sub> Al <sub>0.44</sub> N <sub>x</sub>	1.8	0.01	
6	AlCrN	Supplier 5	Nitriding + CAE	30.7 (±1.5)	391 (±21)	Cr <sub>0.54</sub> Al <sub>0.46</sub> N <sub>x</sub>	5	0.04	
7	CrN	Supplier 4	CAE + Post fine-polishing	20.0 (±1.7)	300 (±17)	CrN <sub>x</sub>	4.4	0.07	
8	H13	_	_	3.1 (±0.2)	225 (±7)	_	_	0.12	
9	Cr	CSM	MS	10.5 (±0.6)	297 (±14)	Cr	2.2	0.12	
10	CrWN	Supplier 4	CAE + Post fine-polishing	23.3 (±1.6)	299 (±17)	$Cr_{0.95}W_{0.05}N_x$	4.2	0.26	
11	CrN	Supplier 5	Filtered CAE	22.1 (±2.3)	305 (±25)	CrN <sub>x</sub>	5	0.78	
12	TiN	CSM	MS	24.4 (±2.2)	325 (±13)	TiN <sub>x</sub>	0.6	0.84	
13	AlCrN	Supplier 5	CAE + Post fine-polishing	30.6 (±1.8)	391 (±25)	$Cr_{0.54}Al_{0.46}N_x$	5.1	1.30	
14	TiB <sub>2</sub>	Supplier 6	MS	11.9 (±1.5)	373 (±31)	TiB <sub>x</sub>	1.5	2.54	

**Table 1:** Summary of the coatings used and their properties\*This sample actually stuck but broke upon disassembly

To minimize adhesion between the solidified aluminum and the substrate, it was postulated that there would be minimal reaction between the molten aluminum alloy and an AlN PVD coating, as the molten aluminum alloy is unlikely to react with an AlN coating. Therefore, AlN PVD coatings were produced as part of this study. However, the AlN coatings exhibited extremely poor adhesion to the H13 steel substrate, and therefore AAT tests were not possible with the AlN coatings.

# 3.2 Microstructural Characterization of Samples Produced using AAT

One interesting feature of the data shown in Table 1 was that bare H13 (with no coating) did not exhibit the highest breaking strength. In an effort to better understand the breaking strength data in greater detail, electron microscopy was used to examine the interfacial structures between several of the coupons and the solidified aluminum alloy cylinders.

#### 3.2 1 Bare H13

SEM micrographs of the interface between a bare H13 steel coupon and a solidified aluminum A380 cylinder were taken (Figure 3) and indicate that the interface is straight and well defined. The bright phases are Al-Fe-Si intermetallic compounds. Further, the interfacial region can be divided into four zones as follows. Zone 1 exhibits a dense "solid layer" structure (Figure 3b). Above Zone 1 are three regions (Zones 2, 3 and 4), which have different morphologies. Zone 2 consists of columnar grains of an Al-Fe-Si intermetallic separated by the A380 alloy. Zone 3 contains a number of small dispersed Al-Fe-Si particles, while Zone 4 displays a smaller number of larger dispersed Al-Fe-Si particles (Figure 3a). These phases are similar in nature and composition to those reported for soldering studies of actual die castings and dipping tests 11, 23-27]. The similarities between the microstructures observed in this study (Figure 3) and for the soldering of actual die castings suggest that the aluminum adhesion test is indeed a good simulation of the commercial die casting process.



*Figure 3:* SEM micrographs of the interface between the solidified aluminum and the bare H13 coupon

As noted earlier, the bare H13 did not exhibit the highest breaking strength of all the materials evaluated (see Table 1). This is attributed to cracks observed at the aluminum-steel interface (Figure 4), which would likely reduce the stress required to separate the aluminum from the H13 coupon.



**Figure 4:** Low magnification SEM image of the interface between the solidified aluminum and the bare H13 steel, showing the presence of interfacial cracks (highlighted by arrow) following the aluminum adhesion test

# 3.2.2 CrN from Supplier 5

As noted above, CrN is a PVD coating commonly used on core pins and small die inserts in the die casting industry. However, in this study CrN from supplier 5 was found to adhere quite strongly to the solidified aluminum cylinders, and so a metallographic examination of the interface between the CrN and the solidified aluminum alloy was performed. As shown in Figure 5, there appeared to be no gap between the alloy and the coating, suggesting good contact between the A380 and the CrN coating.



*Figure 5:* SEM image of a cross-section through the interface between A380 alloy and CrN coating (from Supplier 5)

TEM was used to examine this interface in greater detail and, as seen in Figure 6, small second phase reaction products were observed at the alloy/CrN interface (Fig. 6a). Through the use of micro-diffraction in the TEM, these particles were determined to be spinel (MgAl<sub>2</sub>O<sub>4</sub>, space group Fd-3m) (Figure 6b).



**Figure 6:** Bright field TEM image (a) of the interface between the CrN coating (Supplier 5) and a silicon particle in the A380 alloy. The diffraction pattern (b) was taken from one of the small particles at the interface and was indexed as a <112> zone from spinel

In addition to the small spinel particles, there appeared to be a thin black interfacial layer between the CrN coating and A380 alloy. To determine additional information about this interfacial layer, an elemental line-scan analysis was performed across this layer (Figure 7). The line scan showed an increase in magnesium and oxygen concentration at the interfacial position, suggesting that a continuous MgO layer about 25 nm thick existed at the interface between the CrN layer and the solidified A380 aluminum alloy.



**Figure 7:** Elemental line-scan taken in the TEM at the interface between the solidified aluminum alloy and the CrN coating from Supplier 5

# 3.2.3 AlCrN Coating from Supplier 5

As seen in Table 1, the polished AlCrN coating obtained from Supplier 5 also exhibited strong adhesion to the A380 alloy with a breaking strength of 1.3 MPa. Therefore, the interface between the AlCrN coating and the solidified alloy was also examined to determine if reaction layers were present that could be causing the adhesion (Figure 8). Again, there appears to be no gap between the alloy and the coating (similar to CrN coating described above), suggesting good contact between the molten A380 alloys and the AlCrN coating.



*Figure 8:* SEM images of the cross-section through the interface between the alloy and the AlCrN coating (from Supplier 5)

TEM images of the AlCrN coating/alloy interface at two different magnifications are shown in Figure 9 and, as can be seen, appear to be free of any sort of reaction layer. An elemental line scan was performed across this interface (Figure 10) and is consistent with the absence of a reaction layer.



*Figure 9:* Bright field TEM images of the interface between the AICrN coating (Supplier 5) and adhered A380 alloy



Figure 10: Elemental line scan taken in the TEM across the Al-AlCrN interface shown in Figure 9

# 3.2.4 Impact of Surface Roughness of Adhesion

Research performed during this project also examined the mechanism controlling the adhesion of the solidifying aluminum alloy to the various substrates. Several potential mechanisms were identified, including the roughness of the substrate, the chemical composition of the substrate, and the presence of an insert oxide on the substrate.

In an attempt to determine which of these mechanisms might be impacting adhesion, a series of tests were performed utilizing the AlCrN coating from Supplier 1, which has always been non-sticking when using the Aluminum Adhesion Test (AAT). Half of each of the AlCrN coated surfaces were covered with a thin metallic layer of either chromium (Cr) or titanium (Ti). This was achieved by masking half of the coated substrate, and PVD coating a thin (nano-meters) metallic layer on the un-masked side. By applying only a thin metallic layer, the surface topography of the AlCrN surface layer should be unchanged.

The Aluminum Adhesion Test was performed using standard procedures, and it was found that the aluminum A380 alloy stuck to both substrates. After removal of the aluminum, examination of the substrates showed that the aluminum had stuck only to the side with the metallic coating (Figure 11), and there appeared to be no adhesion to the bare AlCrN coating. This suggests that the chemical composition of the coating is very important in controlling adhesion of the aluminum to the substrate.



**Figure 11a.** Photograph of the AlCrN coated substrate, with a thin metallic layer of titanium on the righthand side. The photograph shows the sample after the Aluminum Adhesion Test, indicating that the aluminum alloy stuck to the titanium-coated side of the sample, but not to the bare AlCrN coating



**Figure 11ba.** Photograph of the AlCrN coated substrate, with a thin metallic layer of chromium on the right-hand side. The photograph shows the sample after the Aluminum Adhesion Test,

# indicating that the aluminum alloy also stuck to the chromium-coated side of the sample, but not to the bare AlCrN coating

# 3.3 Plant Trials

This section of the report will describe the results of plant trial performed at Mercury Castings. As the AlCrN coating from supplier 1 always performed well in the laboratory aluminum adhesion test, it was chosen for further evaluation on actual die casting dies.

# 3.3.1 Plant Trial Number 1

In the first plant trial, only a single cavity insert was coated and tested. The objective of the trial was to test the durability of the coating in a commercial die casting environment, and to make sure that the coating did not introduce any problems to the die casting operation. This trial was performed in December 2015.

The casting tested was a 350 HP Verado® gear case (see Figure 12). This casting is produced using two loose die inserts that are ejected from the die casting die with the aluminum casting, which are then extracted from the casting on the bench adjacent to the die casting machine. As shown in Figure 12, two loose inserts are used to produce each casting, but only one was coated for this trial (the left-hand insert shown in red in Figure 12). Mercury typically uses about four separate sets of inserts, which are used in rotation in the casting cycle.



# *Figure 12:* Models of the 350 HP Verado® gear case showing the location of the AlCrN coated loose die insert

Figure 13a-d shows four views of the insert after coating with AlCrN, but before casting. For the plant trial, the first five shots were produced with lubricant sprayed onto the coated insert, but for the next 20 shots no lubricant was applied to the coated insert, and no evidence of sticking or soldering was observed. On the following day, another 20 shots were produced without lubricating the coated insert, and again no evidence of soldering or sticking was observed. The Mercury plant-floor personnel suggested that they would expect sticking or soldering to occur after about three shots for an uncoated and un-lubricated insert.

Photographs of the insert after the trial are shown in Figure 13e-h, and show little evidence of soldered aluminum.



**Figure 13:** Photographs of the AlCrN coated loose die insert a-d: Before casting e-h: After the casting trial

# 3.3.2 Plant Trial Number 2

Following the success of the first plant trial, a casting was chosen for a second plant trial that involved coating of the entire die. The casting chosen for this second plant trial was the balance shaft housing shown in Figure 14. The casting weighs 1<sup>3</sup>/<sub>4</sub> lbs and is produced in a single cavity die using a 700-ton die casting machine. Prior to the trial, Mercury Castings had experience producing this casting in an uncoated die, where 90,000 shots had been produced. Experience with the old die indicated that 12 seconds spraying was necessary to avoid sticking and soldering of the castings to the uncoated die.

All surfaces of the balance shaft housing die that contact the liquid aluminum were covered with the AlCrN coating. As shown in Figure 15, this involved coating the shot block, cavity insert and chill vent on the moving side of the die, and the cavity insert and chill block (not shown) on the fixed side of the die.



*Figure 14:* Model of the balance shaft housing used in plant trials numbers 2-4

This trial was performed in May 2016 and involved an initial Production Parts Approval Process (PPAP) run, which required the production of several hundred castings. On the first day of the trial, 70 castings were produced using a two second spray time, and no evidence of sticking was observed. This is an 83% reduction in spray compared with that used with the older die. The Mercury personnel then reduced the spray time to one second (a 92% reduction in spray), and again little-to-no sticking was observed. The Mercury personnel then attempted to produce a casting with no spray, and the first casting stuck and bent on ejection (Figure 16a). Once the stuck casting was extracted, it was found that one of the core pins had been bent (Figure 16b), and so the die was disassembled and all the core pins replaced with new, coated pins. The die was then re-assembled and re-installed on the machine.



*Figure 15:* Model of the balance shaft housing die



*Figure 16:* Photographs of the stuck casting and bent pin in the AlCrN coated die

The portion of the casting colored red in Figure 17 shows the position of the casting that stuck to the die for the casting produced without lubricant in the AlCrN-coated die. Clearly this is the thickest portion of the casting, but it is not clear at present whether this was the reason for the observed sticking. For castings produced in uncoated dies, it is to be expected that sticking will occur in the hottest regions of the die. For example, Viswanathan and Han [28] have suggested that the die has to be above a critical die temperature ( $T_c$ ) for soldering to occur, and identified that soldering occurs due to the molten aluminum reacting (alloying) with the die steel to produce Al-Fe-based intermetallic phases. However, when PVD coatings are applied to a die casting die, the molten aluminum no longer contacts the die steel, so the mechanism responsible for the type of sticking observed in Figure 16 is not clear. Additional research is needed to identify the mechanisms responsible for sticking of the molten aluminum to PVD coatings.



**Figure 17:** The portion of the casting colored red shows the location of sticking for the casting produced without lubricant in the AlCrN-coated die

On the second day of this trial, another 96 castings were produced using the one-second spray. Again, little-to-no evidence of sticking was observed. Analysis of the spray pattern used with the one-second spray showed that only the gate area of the casting was actually being sprayed (see Figure 18), with the upper portion of the cavity essentially operating in an un-lubricated (lube-free) condition.



*Figure 18:* Spray pattern for castings produced in the PVD coated die utilizing one-second spraying

To evaluate the impact of reduced die lubrication on internal quality, castings produced in the AlCrN-coated die were T6 heat treated at Mercury as follows:

1000°F/4 hours + water quench + aging

Due to entrapped air and lubrication, die castings given such a long solution heat treatment would be expected to exhibit significant surface blistering. However, as shown in Figure 19, only minor blistering was observed after the heat treatment. Therefore, this suggests that reducing the amount of die lubrication has decreased the amount of entrapped gases, improving the internal quality of the castings.

No attempt was made in this project to provide quantitative measurements of the impact of the lower lubricant on the actual porosity content of the die cast balance shaft housings, but data published by Naizer and Mobley [30] suggests that it may be significant. Their data is reproduced in Table 2, showing that castings made without die lubricant reduced the porosity content of their die castings by more than half.

# <u>**Table 2:**</u> Porosity content measurements performed by Naizer and Mobley [30], showing the impact of die spray on the porosity content of die castings

Condition	Average Porosity Content		
No die lube/cooling off	0.48% to 0.83%		
3 seconds die lube/cooling on	1.33% to 2.14%		



*Figure 19:* Photographs of a balance shaft housing showing only minimal surface blistering after T6 heat treatment

The metric in this project was to reduce gas porosity by 50%, and although this was not measured directly as part of this study, the data from Naizer and Mobley [30] suggests that this metric was met.

# 3.3.3 Plant Trial Number 3

Following the initial PPAP run, the coated die was approved for commercial production. Mercury ran this production in the first half of 2017, and ran 11,652 shots on the coated die over a  $2\frac{1}{2}$  week period. Mercury reported that the die ran well with few problems, and that they started off spraying for one second, but by the end of the run had increased spray time to between  $1\frac{1}{2}$ -to-2 seconds. However, this is still significantly less than the 12 seconds of spraying used for the uncoated die.

By drastically reducing spraying of the die, Mercury has been able to reduce cycle time by about 12% for the coated die, allowing a significantly faster production rate. This cycle time improvement has been analyzed by Al Miller from The Ohio State University [29], and the results shown in Figure 20 compare the cycle times for the 11,652 shots produced in the coated die versus 27,733 shots produced in the older, uncoated die. The results show the approximate 12% improvement in the median rates described above, but possibly more important is the 18% improvement in the third quartile cycle times (i.e., castings produced at slower cycle rates). This significant improvement in third quartile data suggests that running

the PVD coated die is providing a more consistent cycle rate, due to a reduced need to stop and clean the die.



# Figure 20: Data on cycle time improvement

Mercury examined the condition of coating after this initial production run, and a photograph of the die surface is shown in Figure 21. Following the trial, the coating appeared to still be in place. Following cleaning and maintenance, Mercury put the coated die back on the shelf and in preparation for the next production run.

As shown in Figure 22, Mercury did report seeing minor heat checking at sharp radii in the die after the 11,652 shots. However, Mercury believes this is a <u>lower</u> level of heat checking than they would have expected to see when compared with the conventional 12-second spraying, and therefore they expect that the die will last longer until it needs to be replaced (excessive heat checking is normally the reason why die casting dies need replacing).



*Figure 21* Photograph of the surface of the AlCrN coated die after the production of 11,652 shots



*Figure 22:* The highlighted locations in the die show the onset of minor heat checking

The plant trials performed to-date have not been sufficiently long to evaluate the impact of reduced die spraying on heat checking and die life (aluminum die casting dies typically last longer than 100,000 shots). However, laboratory studies performed by Zhu et al. [31] suggest that die life extensions may be significant

with the lower level of die spraying. Zhu et al. utilized laboratory testing equipment (called a dunk tester) located at Case Western Reserve University (CWRU) that has been used in North America for more than 40 years to provide an accelerated test to generate data on the impact of steel composition, heat treatment and processing conditions on die life. A schematic drawing of the dunk tester is shown in Figure 23a. The test involves the repeated dunking a precisely-machined steel sample into a bath of molten aluminum for a predefined period, removing the sample and spraying with a lubricant. One such cycle take less than 30 seconds, and the cycle is repeated up to 15,000 times, and the degree of thermally induced cracking (heat checking) at the corners of the sample is measured. Figure 23b shows the results of testing performed at CWRU to examine the impact of die lubricant spray time on the degree of heat checking, and the data shows that for a sample without spraying, no heat checking was observed even after 15,000 cycles in the dunk tester. For samples that were sprayed between dunks, the degree of cracking (heat checking) was observed to increase with spray time. This laboratory testing performed by Zhu et al. [31] suggest that reducing spraying in actual die casting plants will also significantly reduce heat checking of coated dies.

The metric in this project was to increase die life by 15% by eliminating thermal shock from the die sprayer. As noted above, the plants trials have not yet completed sufficient cycles to test this matric, but initial data from Mercury Castings together with the laboratory testing from Case Western Reserve University suggest that die life should be extended by at least 15%.





- a) Schematic drawing of the test equipment
- b) Results from the test showing the impact of spray time on cracking (heat checking)

3.3.4 Plant Trial Number 4

Mercury has continued to run the AlCrN-coated die, which as of mid-September, 2017 had accumulated more than 19,000 shots. Die spraying continues to remain low, cycle time remains faster, and Mercury is experiencing cost savings due to reduced downtime and minimal spray usage. At this point, the die appears to be holding up very well in terms of coating durability and overall heat-checking resistance.

In summary, initial empirical data from the Mercury plant trial is suggesting that reducing spraying has resulted in a faster cycle rate, a reduction in the level of heat checking, lower entrapped gas, and that using the PVD coating to reduce lubrication has the potential for significantly improved die life, thereby reducing production costs.

# 3.4 Transition to Industry

Engineers at Mercury Castings have been extremely excited about the results from the plant trials performed at their facility. Alex Monroe, Engineering Project Manager at Mercury Castings reported that they "have been most impressed with the unexpected reductions in cycle time as well as the reduced downtime. It will be exciting to see if these early results of reduced heat checking persist. This technology is on the right track to make large energy, environmental, production, and cost gains as it gets rolled out on a larger scale".

The results from the laboratory studies and the plant trials have been presented at the last two NADCA Congresses, and both congress sessions were well attended by industry personnel and generated a lot of interest from attendees. It's always a little difficult to know whether organizations are interested in new technology, as they often want to keep their interest confidential, but certainly large die casting companies such as Nemak and Chrysler appear to be interested in the lube free coating technology. In the opinion of one of the current authors (Midson), if the excellent results from the plant trial at Mercury Castings continue, it is very likely that the lube free coating technology will be applied widely within the die casting industry.

# In addition, the results from this research have been incorporated into a new NADCA booklet and webinar series entitled *Applications of Surface Engineering for Die Casting Dies*.

The lube-free technology should also be applicable to squeeze casting and semi-solid casting. Squeeze and semi-solid casting are modified die casting processes used to produce castings with improved mechanical properties, and therefore are used in applications that require higher integrity castings. Both squeeze and semi-solid casting typically have lower residual porosity levels than conventional die casting, and they achieve this by utilizing die filling processes that are significantly less turbulent, and by using larger gates that allow the better feeding of shrinkage porosity. As squeeze and semi-solid casting are targeting higher quality (lower porosity) castings, the reduced porosity from the lube-free approach (implied by the data in Table 2) will be very important for these modified die casting processes.

#### 4. Summary and Conclusions

- A laboratory test (called the aluminum adhesion test) has been developed to measure the magnitude of adhesion of aluminum alloy A380 solidified against both uncoated and coated H13 substrates. The objective of this test was to simulate the soldering process during high pressure die casting, to identify permanent PVD-type coatings that can be used to minimize or eliminate lubricant spraying during die casting. The test provides a quantitative estimate of the level of adhesion.
- 2. H13 steel coupons with six different coatings were obtained from six commercial suppliers. In addition, two coated coupons were produced using the laboratory PVD coater at CSM.
- 3. Several of the coatings (AlCrN from Supplier 1, AlTiN from Supplier 1 and Supplier 3, and CrWN from Supplier 2) exhibited a zero breaking strength, indicating that during the aluminum adhesion test, the solidified aluminum did not stick to the coating.
- 4. The metallurgical structure at the interface between the solidified aluminum and coupons was examined for three samples that did stick bare H13, CrN (from Supplier 5), and AlCrN (from Supplier 5). For the bare H13, several Al-Fe intermetallics formed between the aluminum and the steel, very similar in nature to those observed for soldering in actual die casting operations. For the CrN coating, a thin layer of magnesium oxide was observed between the solified aluminum and the CrN coating. For the AlCrN coating from Supplier 5, the slidified aluminum was observed to make excellent contact with the coating, but no reaction products were observed to form between the aluminum and the coating.
- 5. Four plant trials were performed using the AlCrN coating from Supplier 1.
- 6. The first plant trial involved coating a single insert with the AlCrN coating. After lubricating the first five shots, forty castings were produced without spraying any lubricant onto the insert. and there was no evidence of the aluminum sticking or soldering.
- 7. A second plant trial was performed where the entire cavity was coated with AlCrN. The spray time for the lubricant was successfully reduced from 12 seconds to one second. However, an attempt to produce the casting without any lubricant was not successful.
- 8. A third plant trial was performed using the AlCrN-coated balance shaft housing die, and an additional 11,652 shots were produced. The spraying was maintained between one and two seconds, a reduction of 83-92% of the spray used for the uncoated die. In addition to reducing spray, it was possible to reduce the cycle time by about 12%, as the time required to spray the die was eliminated.
- 9. After a fourth plant trial with the coated die, total shots exceeded 19,000. The dies appear to be holding up very well in terms of coating durability and overall heat checking resistance.
- 10. T6 heat treatments trials have indicated that the reduction of spray significantly decreased the amount of entrapped gases (porosity) in the castings. Although the amount of gas entrapped in the castings was not quantitatively measured, porosity reduction in the castings due to reduced spraying is estimated to be greater than 50%.
- 11. Plant trials have not progressed sufficiently to evaluate the impact of reduced die spraying on heat checking and die life. However, the metric in this project was to increase die life by 15%, and based on laboratory testing performed at Case Western Reserve University to evaluate the impact of die spray on die life, the 83-to-92% reduction in spray achieved in the plant trials should provide a significant extension in die life.

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#### References

- [1] C. Mitterer, F. Holler, F. Ustel, D. Heim, Surface and Coatings Technology, 125 (2000) 233-239.
- [2] L. Shi, P. Shen, D. Zhang, E. Dong, Q. Jiang, J. Mater. Sci., 47 (2012) 8372-8380.
- [3] M. Naka, M. Kubo, I. Okamoto, J. Mater. Sci. Lett., 6 (1987) 965-966.
- [4] P.H. Mayrhofer, C. Mitterer, L. Hultman, H. Clemens, Progress in Materials Science, 51 (2006) 1032-1114.
- [5] H. Willmann, P.H. Mayrhofer, P.O.Å. Persson, A.E. Reiter, L. Hultman, C. Mitterer, Scripta Materialia, 54 (2006) 1847-1851.
- [6] P.D. Ownby, K.W.K. Li, D.A. Weirauch, J. Am. Ceram. Soc., 74 (1991) 1275-1281.
- [7] V. Joshi, R. Shivpuri, S. Dixit, G. Umeda, P. Cheng, North American Die Casting Association, T03-051 (2003) 319-326.
- [8] V. Joshi, A. Srivastava, R. Shivpuri, E. Rolinski, Surface and Coatings Technology, 163–164 (2003) 668-673.
- [9] S. Chellapilla, R. Shivpuri, S. Balasubramaniam, Proc. 19th Int. Die Casting Congress and Exposition, Minneapolis, MN, USA, (1997).
- [10] J. Lin, S. Carrera, A.O. Kunrath, S. Myers, B. Mishra, P. Ried, J.J. Moore, D. Zhong, Surface and Coatings Technology, 201 (2006) 2930-2941.
- [11] S. Carrera, K. Kearns, O. Salas, J. Lin, H. Swenson, I. Sterling Meyers, S. Lee, B. Mishra, G. Mustoe, P. Ried, in: Transactions of the North American Association (NADCA) congress and Exposition, 2001.
- [12] W. Jung, H. Song, S. Park, D.-Y. Kim, Met. and Mat. Trans. B, 27 (1996) 51-55.
- [13] N. Eustathopoulos, M.G. Nicholas, B. Drevet, Wettability at high temperatures, Elsevier, 1999.
- [14] N. Eustathopoulos, N. Sobczak, A. Passerone, K. Nogi, J. Mater. Sci., 40 (2005) 2271-2280.
- [15] M. Ksiazek, N. Sobczak, B. Mikulowski, W. Radziwill, I. Surowiak, Materials Science and Engineering: A, 324 (2002) 162-167.
- [16] P. Shen, H. Fujii, T. Matsumoto, K. Nogi, Scripta Materialia, 48 (2003) 779-784.
- [17] P. Shen, H. Fujii, T. Matsumoto, K. Nogi, J. Am. Ceram. Soc., 87 (2004) 2151-2159.
- [18] A.H. Volume, ASM international, (1990) 3470.
- [19] K.A. Nazari, S.G. Shabestari, J. Alloy. Compd., 478 (2009) 523-530.

- [20] V. Joshi, K. Kulkarni, R. Shivpuri, R.S. Bhattacharya, S.J. Dikshit, D. Bhat, Surface and Coatings Technology, 146–147 (2001) 338-343.
- [21] R.R. Aharonov, S. Chellapilla, B. Janoss, R. Shivpuri, A. Lakare, NADCA Transactions, Cleveland, (1999) T99-113.
- [22] B. Wang, G.R. Bourne, A.L. Korenyi-Both, A.K. Monroe, S.P. Midson, M.J. Kaufman, J. Mater. Process. Technol., (2016).
- [23] K.A. Nazari, S.G. Shabestari, J. Alloy. Compd., 478 (2009) 523-530.
- [24] Q. Han, H. Xu, P. Ried, P. Olson, International Journal of Cast Metals Research, 23 (2010) 296-302.
- [25] S. Shankar, D. Apelian, Met. and Mat. Trans. B, 33 (2002) 465-476.
- [26] J. Song, T. DenOuden, Q. Han, Met. and Mat. Trans. A, 43 (2012) 415-421.
- [27] C. Mitterer, F. Holler, F. Üstel, D. Heim, Surface and Coatings Technology, 125 (2000) 233-239.
- [28] Srinath Viswanathan and Qingyou Han, "Mechanism of Die Soldering During Aluminum Die Casting", NADCA 2002 Congress, paper number T02-034
- [29] R. Allen Miller, Private Communication, July 2017
- [30] J. Naizer and C. Mobley, "Porosity and Contained Gas Measurements as Assessments of Die Casting Process Control", Report from the Engineering Research Center for Net Shape Manufacturing, The Ohio State University, 1992
- [31] Y. Zhu, D. Schwam, X. Zhu & J.F. Wallace, "Factors that Affect the Die Casting Die Life", 111th Metalcasting Congress (2007)

# **Appendix 1: Students**

The student that worked on this project was Bo Wang. He received his Ph.D. from the Colorado School of Mines in August 2016, and is currently working for a coating company (Ionbond) in Chengdu, China.