37.0 ADVANCED ENGINEERED COATINGS WITH EXTENDED DIE LIFE FOR TOOLING

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This project started in Fall 2018, is a five-year effort, and is supported by the Defense Logistics Agency and CAPES in Brazil. 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 look at the use of 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, help reduce 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 this 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 was 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 us to develop a better understanding of the factors controlling wetting of liquid metals on bare H13 steel and on the PVD coatings.

During the first two years of the project, the main focus was on developing an improved aluminum adhesion test, based on the test described in previous reports [37.3-37.5]. Molten aluminum tests (MAT) were performed on bare H13 and five different AlCrN coatings using the new approach. Due to different behavior exhibited by the coatings in the MAT, coating characterization techniques were applied to better understand the composition, structure and performance of these coatings as described in a previous report [37.6]. Two other laboratory tests were used to test the resistance of a number of coatings to molten aluminum attack: (i) modified ejection test (MET), and (ii) rotating immersion tests (RIT), as previously reported [37.7]. For this report, the successful results from a controlled laboratory lube-free die casting trial performed using a Buhler 250-ton die casting machine at The Ohio State University (OSU) will be presented in detail.

37.3 Recent Progress

37.3.1 Controlled Laboratory Lube-free Die Casting Trial - Methodology

The lube-free laboratory die casting experiments were performed using a Buhler 250-ton die casting machine at the Center for Design and Manufacturing Excellence (CDME) at The Ohio State University (OSU). Two die casting dies were designed using the principle that, to avoid soldering and run continuously in the lube free condition, they should be simple in shape, so complex shapes including cores and changes in section thickness were avoided. To evaluate the impact of thermal mass of the casting upon soldering, one of the castings was relatively thin, while the other relatively thick. The first die (die #1) produces thin plate castings approximately 100 x 165 x 4 mm in size, with a total casting weight of 0.35 kg. The second die (die #2) produces much thicker plate castings, approximately 100 x 165 x 19 mm in size, with a total casting weight of 0.90 kg. The die inserts were fabricated using heat treated H13 with a final hardness of 46-48 HRC. The die inserts, the ejector side shot block and ejector pins for the two dies tested at OSU were PVD covered with an AlCrN coating using a duplex process that consisted of initially nitriding the heat treated H13 steel via ion implantation, followed by the deposition of a Cr adhesion layer, and then an AlCrN top layer. The two dies tested at OSU are shown in **Figure 37.1**.

For each of the die casting trials at OSU, around 130 kg of A380 aluminum alloy was melted, and nitrogen gas degassing was performed on the molten bath for 15 minutes to ensure metal cleanliness. Standard samples for composition analysis were produced for each casting trial, and measured using a desktop SpectroMaxx spark Optical Emission Spectrometer (OES). A two-axis servo-driven Rimrock ladle was used to ensure consistent casting weight during the die casting trials. For mapping the effect of process conditions on the ability to produce lube-free castings, two parameters were varied during the trials, (i) two aluminum alloy melt temperatures were examined (680 and 720°C) and (ii) the oil used to pre-heat the dies was varied between 200-305°C. **Table 37.1** shows the six combinations of conditions examined.

Prior to the trials, shot profiles were calculated based on NADCA recommendations designed to provide gate speeds of around 1,400 in/sec. Unfortunately, problems with the Buhler machine prevented programing of the shot profile, and a shot profile stored in the machine's memory from a previous run had to be used. This generated an extremely high gate speed of 2,665 in/sec, which may have encouraged soldering in the die cavity by exposing the die surface to an extremely aggressive filling condition. **Figure 37.2** shows the desired shot profile, along with the profile actually used. Each casting trial started with the application of a BN spray to the surface of the die, and then transitioned to a decreasing application of a manual spray of die lube (Chem-Trend SL-90078 diluted 1:30 in water), until the lubricant was totally removed from the process and lube-free conditions were run for a series of castings. Following ejection of each casting, a FLIR thermal camera was used to track the die surface temperature of the ejector side of the dies. To estimate the maximum temperature experienced for both dies at each of the first five conditions examined, HPDC simulations were performed using ProCast software, and these predictions were compared with actual temperatures measured by the thermal camera.

37.3.2 Controlled Laboratory Lube-free Die Casting Trial - Results

The composition measurements of the A380 aluminum alloy used in the trials are listed in **Table 37.2**. For the testing performed on day 1, die #1 was installed on the die casting machine, the molten A380 temperature was kept at 680 ± 20°C, the hot oil temperature was 200 ± 5°C, and the test sequence showed in **Table 37.3** was followed for lubricant application. By the end of the trial, 40 castings had been produced under lube-free conditions (i.e., without the application of any die lubricant, although the plunger lubricant PL-208 from ChemTrend was painted onto the shot tip prior to each shot). The same die (die #1) was used for testing performed on day 2, where the objective was to examine the effect of different processing conditions on the ability to produce die castings under lube free conditions. The testing followed the conditions listed in **Table 37.4**, where the molten metal temperature and the oil temperature used to pre-heat the die were varied. As shown in **Table 37.4**, it was possible to produce 117 castings in the lube-free conditions, for all conditions examined. Combining the two days of testing, a total of 157 lube-free castings were produced using die #1. During the testing using die #1, occasionally the aluminum casting would stick to the ejector pins during ejection, as shown in **Figure 37.3 (a)**. Little to no adhesion strength was observed, since a lateral touch was enough to remove the casting. It is possible that the flat ends of the ejector pins are areas with less PVD coating, due to the line of sight nature of the PVD deposition process. **Figure 37.3 (b)** shows the ends of the ejector pins, where aluminum soldering can be observed around their periphery. Intermittent build-up of aluminum was also observed on the cavity faces close to the gate (identified by the red circles in **Figure 37.3 (c)**). This build-up would intermittently appear and then disappear after a few castings. Although this phenomenon occurred throughout the entire trial, there was no evidence of the build-up developing into soldering in these areas of the coated inserts. It is possible that this build-up may be a consequence of the extremely high gate speed used to produce these castings (as shown in **Figure 37.2**).

Die #2 was installed in the machine and tested on day 3 of the trials. The plan for this trial was to test the conditions listed in **Table 37.1**, but due to a problem with the die casting machine, the trial had to be interrupted at condition C3 (molten A380 temperature of 720°C and die oil temperature of 230°C). **Table 37.5** shows the conditions and number of castings produced in day 3 for each condition examined. This trial demonstrated that was possible to run lube-free with die #2, and that 56 castings lube free castings were produced under different conditions. Similar to that observed with die #1, sometimes the castings would stick to the tips or sides of the ejector pins with die #2, but no significant load was required to extract the castings. A different condition was noted half-way through the trial with die #2, where significantly flashing started to occur. During normal conditions, the injection pressure was around 125 bar, but when flashing was observed, a pressure spike (to above 700 bar) was observed during injection, and it is likely that this pressure spike was the cause of the flashing (see **Figure 37.4**).

The condition of the die inserts by the end of the three days of lube-free casting trials are shown in **Figure 37.5**. Figure **37.5 (a)** shows the ejector side of die #2, where aluminum marks in a vortex shape can be seen towards the center of the lower and upper portions of the cavity, and blue oxidation marks on the center cored section. **Figure 37.5 (b)** shows the fixed side of the die, with aluminum marks in a similar position towards the top of the die, and the blue oxidation marking downstream from the three gates. The red arrows in **Figure 37.5 (c)** and **Figure 37.5 (d)** show that relatively thick aluminum solder built up on the lower part of the PVD coated ejector-side shot block, and on the un-coated cover shot block. Most likely these are regions of the die that became extremely hot during die casting. Even though soldering did develop at these positions, it was not enough to stop the coated dies from running in a lube-free condition during the trials. A total of 157 lube-free castings were produce using die #1 and 56 lube-free castings using die #2.

The surface temperature for the ejector side of the die was monitored between shots by a thermal camera for all casting conditions in the three days of the lube-free trials. **Figure 37.6 (a)** shows the temperature distribution for all conditions for die #1, and in **Figure 37.6 (b)** for die #2. The casting number relating to each picture is shown in the Figures. The data from the thermal camera shows that the temperature increased and became more uniformly distributed as the test progressed. As expected, due to the higher thermal mass of the casting produced in die #2, the maximum temperature experienced was higher by ~60°C.

ProCast simulations were performed for both dies for the first five conditions tested (conditions C1 to C5 listed in **Table 37.1**). **Figure 37.7** shows the results of the simulation, showing the maximum die temperature for each condition. By comparing **Figure 37.6** and **Figure 37.7**, it can be seen that the simulated results match well with the experimental observations. Also, the positions of highest temperature for all conditions shown in **Figure 37.7** corresponds to the regions were soldering was observed on the shot block (shown in **Figure 37.5 (c)** and **Figure 37.5 (d)**).

Also, as was shown in **Figure 37.6 (b)** for the center of the lower and upper part of the cavity for die #2, the simulation confirmed the thermal camera results, as those positions showed the presence of some aluminum vortex of a similar shape as the highest temperatures observed in the simulation. By combining these results, the positions where soldering or aluminum marks were present in the die are similar to those where maximum temperatures were observed. This is in agreement with Han [37.8-37.9], who suggested that a critical temperature has to be achieved for soldering to occur. The difference here is that build-up was also observed, but did not progress to soldering, suggesting that Han's theory might not only be related to soldering, but also as an indicator of build-up.

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 in-plant die casting trials on a number of selected coatings.

- 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 Acknowledgements

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37.6 References

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37.7 Figures and Tables

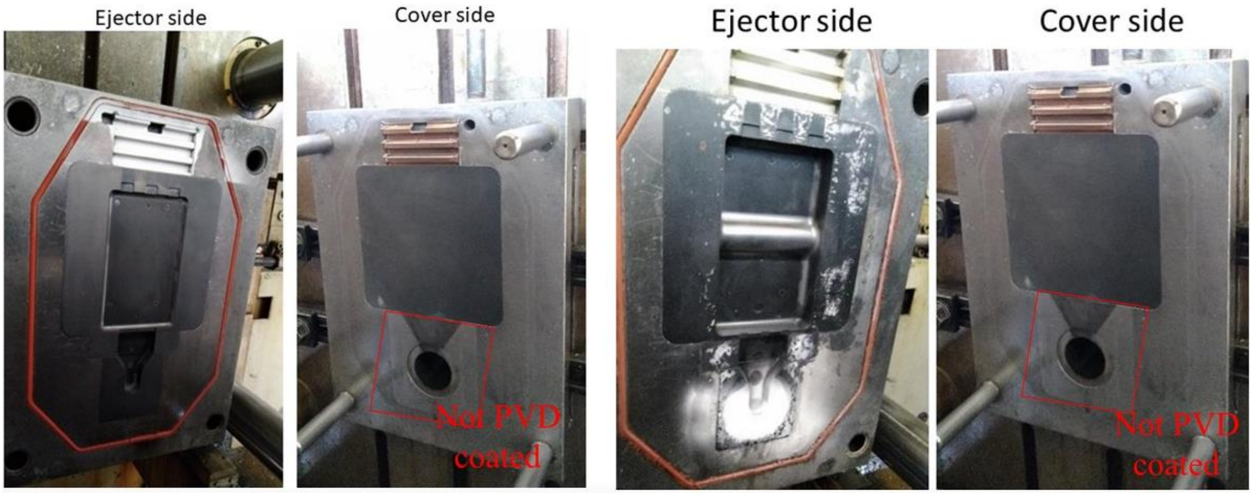

Figure 37.1: The two dies for OSU die casting trial (left) for die #1 and (right) die #2. The cover side was the same for both dies.

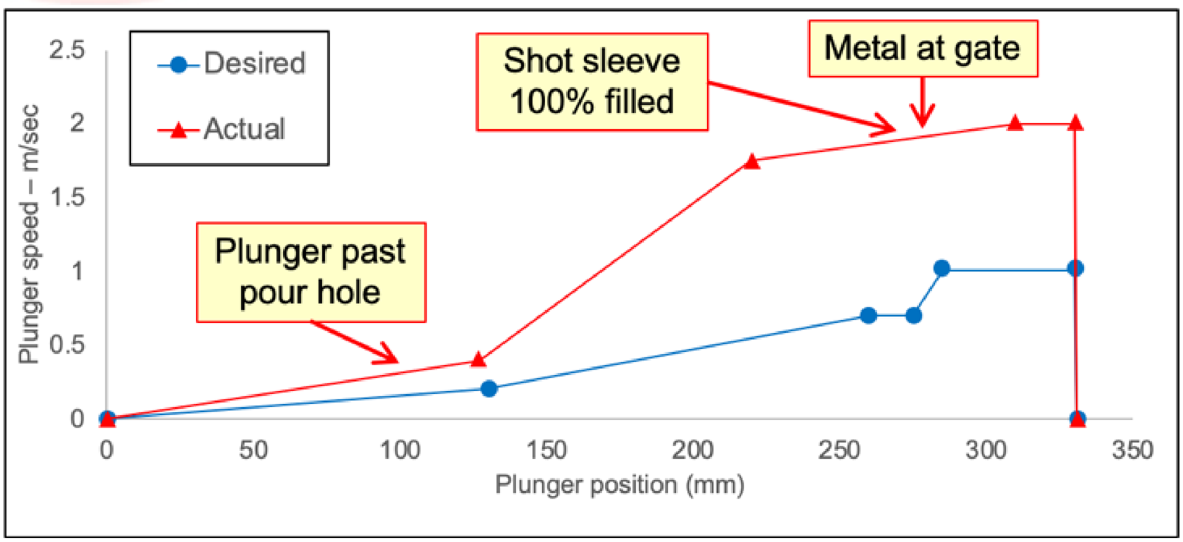

Figure 37.2: Differences in shot profiles from the desired to the profile actually used during the test.

Figure 37.3: Lube-free casting trial using die #1 where (a) ejector side showing the casting stuck to the tip of the ejector pins, (b) side of tip of the core pins with aluminum adhered, and (c) intermittent aluminum build-up at the coated die surface appearing at casting #20 and disappearing at casting #21.

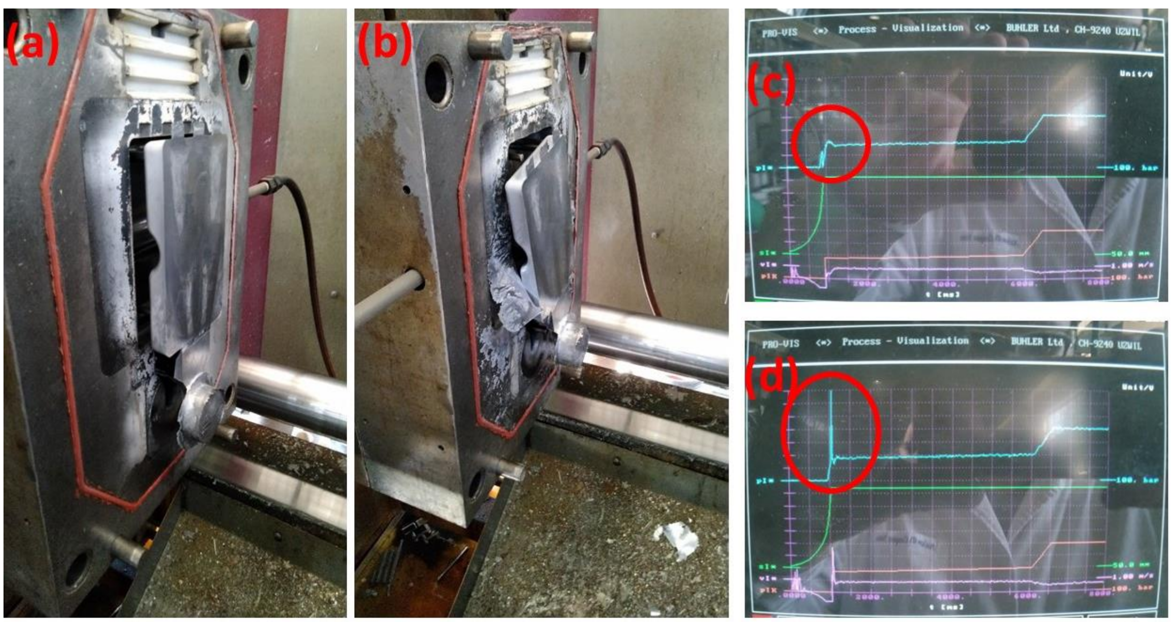

Figure 37.4: Lube-free casting trial on day 3 using die #2 where (a) ejector side showing the casting stuck to the side/tip of the ejector pins, (b) casting with aluminum flashing, (c) normal injection pressure, and (d) pressure spike during injection.

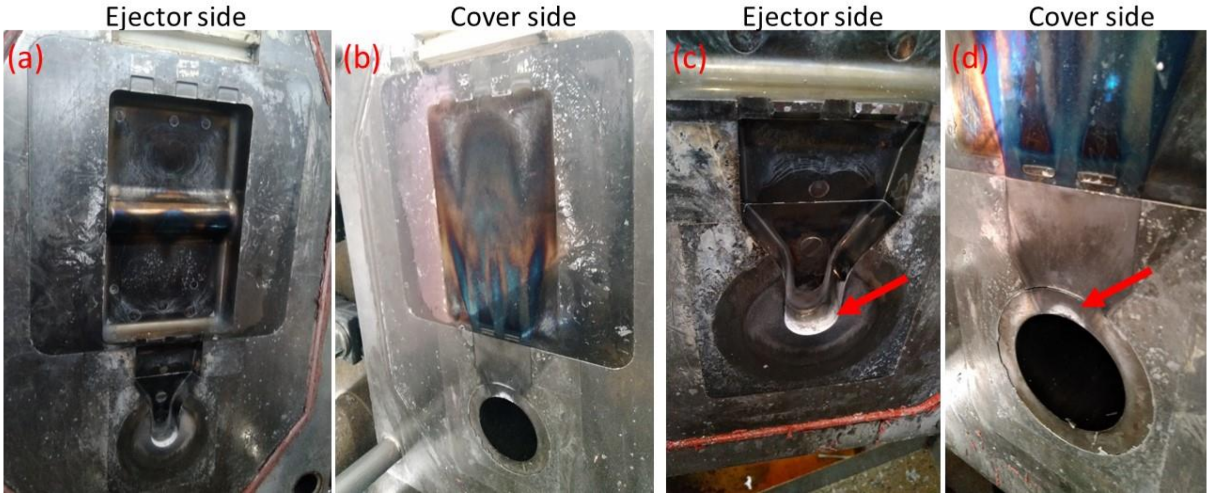

Figure 37.5: Condition of the die inserts by the end of the three days of lube-free trials, (a) ejector side showing the presence of aluminum marks, (b) aluminum marks on the top and blue oxidation marks at the gate area, (c) soldering on the coated shot block, and (d) soldering on the uncoated cover shot block.

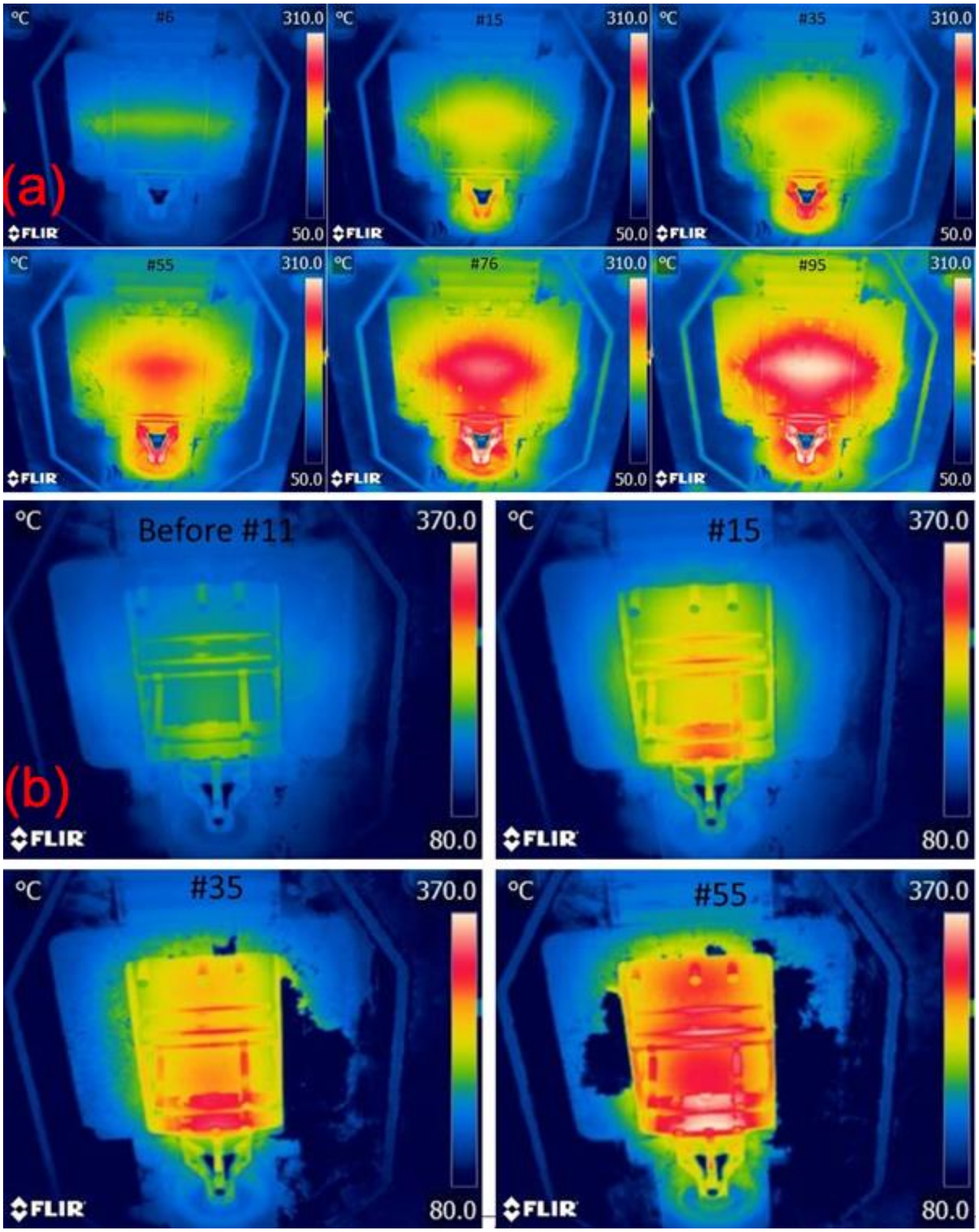

Figure 37.6: Temperature distribution for (a) die #1 and (b) die #2 during the lube-free trial for all tested conditions.

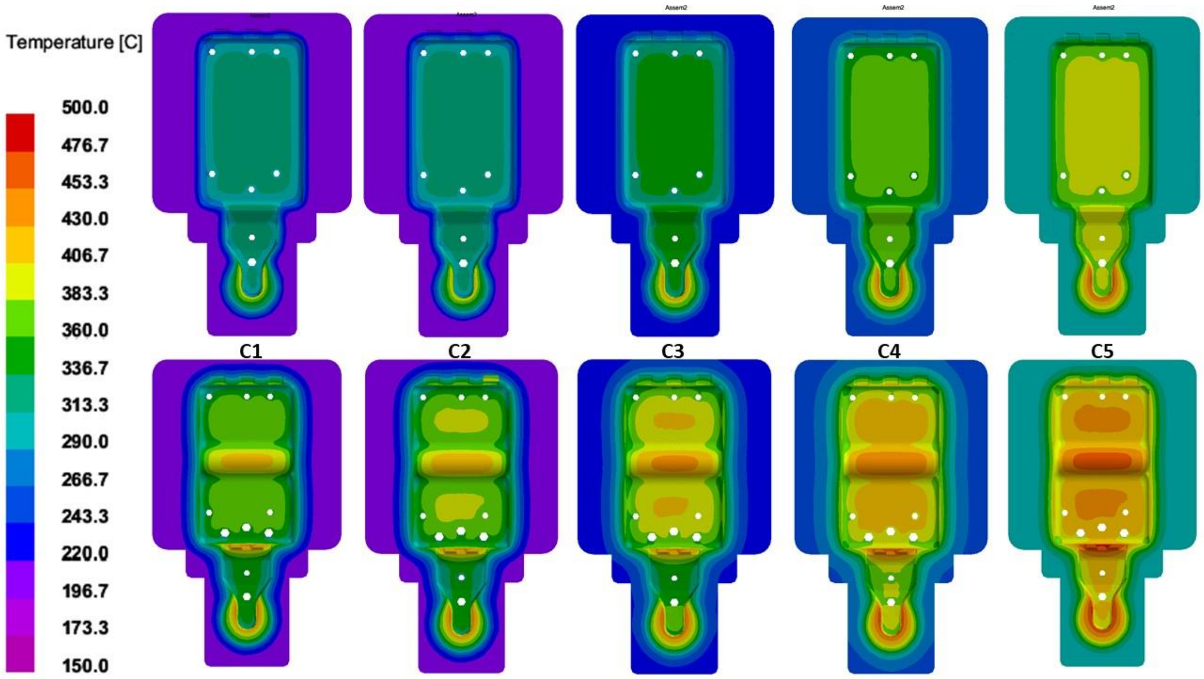

Figure 37.7: Simulated temperature distribution for die #1 and die #2 for conditions C1 to C5.

Table 37.1: Six conditions of A380 melt temperatures and heating oil temperatures examined in this study.

Condition	Temperature (°C)	
	A380	Oil
C1	680	200
C2	720	200
C3	720	230
C4	720	260
C5	720	290
C6	720	305

Table 37.2: Measured composition of the A380 alloy used at OSU trials.

A380	Al	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti
Day 1	Balance	8.2	1.0	3.1	0.20	0.20	0.03	0.05	1.0	0.06
	Balance	8.4	1.0	3.1	0.20	0.21	0.03	0.05	1.1	0.06
Day 2	Balance	8.0	1.0	3.1	0.19	0.22	0.03	0.05	1.2	0.06
	Balance	8.1	1.0	3.2	0.19	0.22	0.03	0.05	1.2	0.06
Day 3	Balance	9.1	1.0	3.1	0.19	0.18	0.03	0.05	1.2	0.06

Table 37.3: Die casting trial sequence at Day 1.

Castings		# of die lube sprayed			
#	Quantity	cavity	runner	cover	shot block
1 to 6	6	BN			
7 to 11	5	3	3	3	3
12 to 14	3	2	2	2	2
15 to 17	3	1	1	1	1
18 to 57	40	Lube-Free			

Table 37.4: Die casting trial sequence at Day 2 mapping six different combinations of A380 and oil temperatures.

Castings		Temperature (°C)		Condition
#	Quantity	A380	Oil	Lube
1 to 5	5	680	200	BN, spray
6 to 25	20	680	200	**Lube-Free**
26 to 45	20	720	200	
46 to 65	20	720	230	
66 to 85	20	720	260	
86 to 105	20	720	290	
106 to 112	7	720	305	
113 to 122	10	720	200	

Table 37.5: Die casting trial sequence at Day 3 mapping the first three combinations of conditions.

Castings		Temperature (°C)		Condition
#	Quantity	A380	Oil	Lube
1 to 5	5	680	200	BN, spray
6 to 25	20	680	200	Lube-Free
26 to 45	20	720	200	
46 to 61	16	720	230	
-	-	720	260	-
-	-	720	290	-