

Lube Free Die Casting

Final Report

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Abstract

Die lubricants are used extensively in die casting and are expected to provide good part release, anti-solder and lubricity of the die and ejector pins. In many cases water based die lubricants are also applied to cool the die surface. While fulfilling these favorable roles, die lubricants have some undesirable consequences. Vapors from moisture left on the surface of the die after spraying can be trapped in the casting and cause excessive porosity. Decomposition of organic ingredients in the die lubricant can also cause porosity. Application of the die lubricant extends the cycle time. Unless properly addressed, die lubricant mist and residuals can pose environmental issues in the plant.

For these reasons, a lube-free die casting process is an attractive goal. To accomplish it, the functions of the die lubricant would need to be fulfilled by a substitute permanent or semi-permanent coating applied on the dies. The focus of this study was evaluation of boron nitride as a potential semi-permanent die casting coating. While expensive, boron nitride is recognized as an excellent release agent. As an inorganic substance, it is also thermally stable up to relatively high temperature. It does not react with molten aluminum, thus providing excellent protection from soldering.

The study confirmed the superior performance of boron nitride in preventing soldering, by conducting extensive exposure of boron nitride coated steel in molten aluminum. A novel test was developed to quantify the release stress during ejection of aluminum castings from the die. This test demonstrated low release stress during ejection when concentrated boron nitride coatings were applied. The release stresses increased when the concentration on the boron nitride was lowered, but were still significantly lower than in commercial die lubricants. Intermittent application of a boron nitride coating at longer intervals provided reduced yet satisfactory release. Preliminary evaluation in production at Mercury Marine provided encouraging results.

1. Introduction

Currently, die lubricant is utilized in die casting on a shot-by-shot basis and acts as a parting material for casting release after solidification. In addition, many plants rely on the application of lubricant to assist in cooling the die cavity surface. Applying the lubricant adds time to the casting cycle, contributes to the cost of producing castings, and produces effluent which has associated waste removal costs. Furthermore, the spray-on lubricant is known to be a source of porosity, so eliminating die lubricant will result in reducing gas porosity.

2. Objectives

This project proposed developing permanent or semi-permanent die coatings that are non-wetting/non-sticking in order to reduce cycle time and process cost, as well as improve quality. CWRU evaluated die surface coatings for high pressure and squeeze pressure applications.

3. Experimental Set-up and Procedures

3.1 Wettability and Soldering

To evaluate the wettability and soldering propensity of coatings, a rotating pin set-up was developed and is illustrated in Figure 3.1. It was comprised of a melting furnace with a silicon carbide crucible, in which molten aluminum alloy was held at a pre-set temperature. The coated pins were held by a sample holder, attached at one end to a bench drill.



Figure 3.1.1: Rotating pin set-up



Figure 3.1.2: Specimen holder

3.2 Release Force

An important function of die casting coatings is to facilitate the release of the casting from the die. If releasing the casting requires excessive force, the part may distort and become unusable. To measure the release load, a "Pull-Out Test" was developed. A schematic of the experimental set-up is illustrated in Figure 3.2.1.



Figure 3.2.1: Schematic of the pull-out experimental set-up

The set-up was comprised of a crucible (1) held in a fixture, inside a tensile tester. The rod (2) can be positioned at the desired level inside the crucible. In the pull-out experiment, the rod is initially lowered with the upper ram into the crucible and the end positioned 0.1" from the bottom of the crucible. Molten low-iron A356.1 alloy at 1,350°F +/-5°F is then poured into the crucible and allowed to cool down to 750 °F as measured by a thermocouple, at which time the tensile tester is turned on and the rod is pulled out from the solidified aluminum while the load is recorded. A 10,000 lbs load cell was employed for high-release load experiments, since the 500 pound load cell would not measure the higher loads required to release some of the samples. A larger frame was used to accommodate this load cell. Pictures of the experimental setup are shown in Figure 3.2.2.







Figure 3.2.2: Experimental set-up for pull-out test

Substrate

A large batch of ground steel rods were purchased to ensure uniform surface condition among substrates.



Figure 3.2.3: Steel rods used as substrate for coating pull-out test

3.3 Coating Adhesion

This test was conducted with the Immersion Thermal Fatigue Tester that simulated the conditions encountered by die materials and coatings during die casting of aluminum alloys. The sample was usually processed to the dimensions shown in Figure 3.3.1. It was a $2 \times 2 \times 7$ inch rectangular parallelepiped specimen with a 1.5 inch diameter hole

in the center for internal water cooling. The four comers had a constant 0.010 inch radius that intensified the predominately uniaxial stress at this location. The test produced considerable constraint and high thermal fluctuations during immersion and removal from the aluminum 380 alloy bath. The experimental set-up is illustrated in Figure 3.3.2 (a) and (b). The molten bath was maintained at 1350°F and the specimen was immersed for 12 seconds and then removed from the bath for 24 seconds to produce the thermal cycle. The outer surface of the specimen was normally sprayed with a commercial water-base lubricant just before it entered the molten aluminum bath. Water flowed through the central hole at a constant rate of four gallons per minute. The standard procedure was to operate the equipment for 5,000 immersion cycles, measure the cracking pattern and follow this method for 10,000 and 15,000 total cycles. A three inches long center section along the corners, equidistant from each end, was used to measure the cracks at 100X. The crack length was categorized and recorded in 50 micron intervals. The cracking pattern was reported as the average maximum crack length and the summation of the squares of the crack length for each of the four comers. The more severe the crack pattern, the lower the thermal fatigue resistance of the tested material. The results of this test have correlated closely with the behavior of dies in industry.

The thermal fatigue immersion test has also been used for evaluation of permanent mold coating and die casting lubricants. In this case, the test was modified to closely represent the targeted process by eliminating the die lubricant spray and by modifying the cycle times.



Figure 3.3.1: Thermal fatigue specimen



Figure 3.3.2: Thermal fatigue set-up (a) Picture (b) Schematic

4. Results and Discussion

Boron nitride (BN) is a high-performance release agent. As long as the thermal control of the process is addressed with internal water cooling, BN may very well be a good dry die release agent. Evaluation of various Momentive boron nitride formulations at Case, utilizing the immersion rotating pin, the Immersion Thermal Fatigue Tester, and the pull-out experiments were conducted as part of this task. Momentive is among the leading producers of boron nitride in the world, and is headquartered in Cleveland, Ohio.

4.1 Release Testing

Five specimens were initially evaluated with the pull-out test, one uncoated and four coated with graphite and various boron nitride coatings. The tested specimens are shown in Figure 4.1.1. The cooling curve for the Specimen #1 is shown in Figure 4.1.2.



Figure 4.1.1: Tested specimens showing the coated rods and aluminum slugs



Figure 4.1.2: Typical cooling curve of molten aluminum during pull-out test

According to the cooling curve, it took about 480 sec (8 minutes) before the aluminum temperature dropped to 700°F and the specimen was pulled out. Out of this time, the aluminum was molten for about 120 sec (2 minutes). These times vary depending on the volume of aluminum poured. A longer contact time of the specimen with molten metal than in die casting may be desirable to amplify the effect of the die lubricant release efficiency. The volume of molten aluminum poured was held constant in subsequent benchmarking experiments.

The uncoated specimen maxed out the 500 lbs range of the load cell without pulling out of the aluminum. The graphite coated specimen pulled out at a minimal load of less than 50 lbs. The other specimens pulled out at intermediate loads. A couple of specimens pulled out initially, but seized again. For this experiment, only the load required for the initial pull out is of interest. The load vs. time plots for the uncoated and graphite coated specimens are shown in Figures 4.1.3 a,b.





Based on these initial results, the test seemed to provide good sensitivity to measure release loads and differentiate among die lubricants. The conditions of the test, including volume of the molten aluminum poured would need to be held constant, as they determine contact time of the specimen with molten aluminum.

Next, five specimens were tested, one coated with undiluted graphite and four coated with boron nitride coatings at 1:0, 1:1, 1:2 and 1:4 dilution ratios. The specimens are shown before and after testing in Figures 4.1.4 and 4.1.5 respectively.



Figure 4.1.4: Tested specimens showing the specimens before testing



Figure 4.1.5: Tested specimens showing the specimens after testing

Shown below are the pull-out load vs. displacement curves for the five specimens tested. The significant feature is the first peak load, when the pin is displaced. This is essentially the shear load required to pull the coated pin out of the solidified aluminum. In many cases, the pin "seizes" again rather than sliding out of the aluminum. This however is irrelevant. The plot in Figure 4.1.6 summarizes the pull out loads for these experiments.

Note the peak load for the undiluted graphite coating was only about 95 lbs. The undiluted ZYP boron nitride coating pulled out at about 180 lbs. The 1:1 diluted ZYP BN coating took about 270 lbs load to pull out. Further dilution of the ZYP BN coating to 1:2 and 1:4 increased the load to about 380 lbs.

The displacement readings do not have any meaning in this test. They just show how far the cross-head moved before the specimen hit the stop and allowed the load to be applied on the specimen.







12: ZYP BN 1:4



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The results of the pull-out test described above are sensitive to the dilution ratio. In addition to the coating and dilution ration, the loads necessary to pull out the coated pins also depend on the diameter and the surface roughness of the pin. The pull out load range may need to be extended beyond 500 lbs to accommodate die casting commercial coatings and higher dilution ratios.

The surface condition of the specimens can affect the results of the release load. To determine the effect of surface condition, steel pins were treated with the nitrocarburizing Dynablue process. The load range required to release Dynablue-treated 3/8" pins from a solidified 380 alloy (about 1" deep) was tested. The load range required to release Dynablue-treated 3/8" + BN coated pins from a solidified 380 alloy (about 1" deep) was also evaluated.

Four specimens were tested: (a) as-treated with Dynablue (b) as-treated with Dynablue + BN 1:2 die lubricant (c) as-treated with Dynablue Gibbs 1 die lubricant (d) as-treated with Dynablue + Gibbs 2 die lubricant. The specimens are shown before and after testing in Figures 4.1.7 and 4.1.8 respectively.



Figure 4.1.7: Tested specimens showing the specimens before testing



Figure 4.1.8: Tested specimens showing the specimens after testing

Shown below are the pull-out load vs. displacement curves for the specimens tested. The significant feature is the first peak load, when the pin is displaced. This is essentially the shear load required to pull the coated pin out of the solidified aluminum. In many cases, the pin "seizes" again rather than sliding out of the aluminum. The displacement readings do not have any particular meaning in this test. They just show how far the cross-head moved

The 1:2 diluted BN released at about 100 lbs. The as-treated with Dynablue + Gibbs concentrated die lube #2 released at 500 lbs. The as-treated with Dynablue only and the as-treated with Dynablue + Gibbs concentrated die lube #1 did not release up to 500 lbs. This being the maximum capacity of the bench top tester, the samples had to be transferred to a larger capacity 10,000 lbs Instron machine. Both pulled out at about 2,200 lbs.





Figure 4.1.9: Pull-out force of Dynablue and BN coated specimens

The Momentive BN coating formulations shown in Table 4.1 were evaluated next.

	Substrate condition	Dilution Ratio (BN Paint: Water/Lube) by weight	Coatings to be tested	Samples
1	Used Pins	50:50	Momentive BN coatings (FPC, LPC, GPC) vs. Zyp, graphite, Gibbs Lube	
1			Momentive FPC	4
2			Momentive LPC	4
3			Momentive GPC	4
4			ZYP	4
5			Graphite	4
6			Gibbs Lube	4
2	Used Pins	05:95	Momentive BN coatings (FPC, LPC, GPC) vs. Zyp, graphite, Gibbs Lube	
7			Momentive FPC	4
8			Momentive LPC	4
9			Momentive GPC	4
10			ZYP	4
11			Graphite	4
12			Gibbs Lube	4
2 7 8 9 10 11 12	Used Pins	05:95	Momentive BN coatings (FPC, LPC, GPC) vs. Zyp, graphite, Gibbs Lube Momentive FPC Momentive LPC Momentive GPC ZYP Graphite Gibbs Lube	4 4 4 4 4 4

Table 4.1: Momentive coatings evaluated by the pull-out test

The coatings were applied by dipping of the pre-heated specimens in a solution, reheating, and dipping for a second time. Pictures of the specimens before and after testing are shown in Figure 4.1.10 followed by the load vs. distance for each. The peak of this curve indicates the release load. A table and bar chart of the release loads for the tested coatings is shown in Figure 4.1.11









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2: Momentive LPC 50:50



3: Momentive GPC 50:50



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4: ZYP 50:50





5: Graphite 50:50





5: Graphite 05:95





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#	Substrate condition	Dilution Ratio (BN Paint: Water/Lube) by weight	Coatings to be tested Momentive BN coatings (FPC, LPC, GPC) vs. Sam Zyp, graphite, Gibbs Lube)		Pull-out Test Results (lbs)				Average (lbs)	Std. Dev.
1	Used Pins	50:50	FPC, LPC, GPC vs. Zyp, graphite, Gib	bs Lube	Α	В	С	D		
1			Momentive FPC	4		5	18	14	12	7
2			Momentive LPC	4	46	20	30	35	33	11
3			Momentive GPC	4	30	29	24	19	26	5
4			ZYP	4		75	75	120	90	26
5			Graphite	4	35		16	18	23	10
6			Gibbs Lube	4						
2	Used Pins	05:95	FPC, LPC, GPC vs. Zyp, graphite, Gib	bs Lube						
7			Momentive FPC	4						
8			Momentive LPC	4						
9			Momentive GPC	4						
10			ZYP	4						
11			Graphite	4	105	210		350	222	123
12			Gibbs Lube	4						



Figure 4.1.11: Table and bar chart of the release loads for the tested coatings

BN and graphite samples coated with 50:50 formulations released below 500 lbs loads. The samples coated with the 95:5 graphite also released below 500 lbs. Preliminary results for the other coatings indicate a release load higher than 500 lbs.

Pictures of the next round of specimens after testing are shown below, followed by the load vs. distance for each. The peak of these curves indicates the release load. A table and bar chart of the release loads for the tested coatings is shown in Figure 4.1.12 (a)(b)(c).









6: Gibbs Lube 50:50





7: Momentive FPC 05:95





8: Momentive LPC 05:95





9: Momentive GPC 05:95





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10: ZYP 05:95





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A. Pure Lube





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Figure 4.1.12 (a), (b), (c)

#	Dilution Ratio (BN Paint: Water/Lube) by weight	Coating	Samples	Pull-	out Te (II	est Re os)	sults	Average (lbs)	Std. Dev.	Pull-	out Te	est Re N)	sults
Group1	50:50			Α	B	С	D			Α	B	С	D
6		Gibbs Lube	4	196	162			178.9	23.9	870	720		
Group2	05:95												
7		Momentive FPC	4	1800	117	225	146	162.8	55.9	8000	520	1000	650
8		Momentive LPC	4	1013	90	83.3	248	140.3	92.9	4500	400	370	1100
9		Momentive GPC	4	563	135	108	117	120.0	13.7	2500	600	480	520
10		ZYP	4	169	56.3	90	49.5	65.3	21.7	750	250	400	220
Group3		Formulated Lubes		1	2	3	4			1	2	3	4
	Pure Lube	Sample A	4	3.38	5.63	6.75	15.8	7.9	5.4	15	25	30	70
	50:50	Sample B	4	24.8	4.5	5.18	5.63	10.0	9.8	110	20	23	25
	25:75	Sample C	4	90	58.5	113	92.3	88.3	22.3	400	260	500	410
	10:90	Sample D	4	185	140	180	164	167.1	20.3	820	620	800	730
	5:95	Sample E	4	113	113	113	203	135.0	45.0	500	500	500	900



Figure 4.1.12: Release loads of evaluated coatings



Figure 4.1.13: Release load dependency on dilution of the coatings

Observations

- The release load for Momentive BN die lube formulation increases with dilution. The pure die lube requires minimal load to release; so does the 50:50 die lube. The difference between 10:90 and 5:95 is minimal.
- The release load for the other Momentive 5:95 diluted coatings is in the 140-160 lbs range, with the exception of the ZYP that is lower, about 60 lbs.
- 3. Please note samples 7A, 8A, 9A, 10A were initially tested with the 500 lbs load cell and did not release. These samples are highlighted in Figure 4 and were not included in the averages. They were re-tested with the 10,000 lbs load cell. By the time this test was conducted, the aluminum had chilled to room temperature. Much higher release loads were measured. Solidification contraction of the aluminum around the steel rod accounted for most of the higher release load. It is interesting to notice the jagged load profile near the

maximum pull-out load, indicating repeated release and seizing. This effect has not been observed when the aluminum is still hot.

- 4. While the 10,000lbs load cell can pull out all the samples, it may not provide as high a resolution as the 500lbs load cell. The pros and cons of using either one of the load cells need to be factored in, depending on the objectives of the test.
- **5.** The test was continued past the first peak in the load. Often, the sample seized again only to eventually release at higher load.

4.2 Adhesion Testing

Two graphite substrates were coated; the first with one coat underwent one-hundred forty immersion cycles in Aluminum 356 alloy, held at 1350 F (~732.2 C). The second with two coats underwent one-hundred immersion cycles in Aluminum 356 alloy, held at 1350 F (~732.2 C). The coated graphite specimens are shown after the immersion test in Figures 4.2(a) and 1(b).



Figure 4.2.1(a): One Coat 140 Cycles



Figure 4.2.1(b): Two Coats 100 Cycles



Figure 4.2.2: Post Dunker Aluminum Coating Adhesion test

The Momentive GPC BN coating performed well under the conditions of the dunker experiment. No damage to the coating was observed during the immersion testing. After the test, the aluminum "skin" solidified on the sample was easily peeled off the BN GPC coated samples, but partially removed the BN coating in some areas as illustrated in Figure 4.2.2. The BN coating stuck to the aluminum foil. To further quantify the adhesion of the coating to the substrate, the ASTM scratch test kit was used. This test provides a tool for scribing a grid of squares in the coating as illustrated in Figure 4.2.3.

Classification	% of Area Removed	Surface of Cross-cut Area From Which Flaking has Occured for 6 Parrallel Cuts & Adhesion range by %
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
OB	Greater than 65%	

Figure 4.2.3: ASTM Adhesion evaluation scratch test classification guidelines A standard tape is applied on top of the grid and then removed. Depending on the % area of the coating removed, the coating can be ranked and compared to other coatings.

Figure 4.2.4 shows side-by-side four Momentive boron nitride coatings after 100 cycles of thermal fatigue (dunk test) and then after the scratch test. Note coating ZYP displays least spalling of the coating. GPS is the worst performer, with LPC and FPC inbetween.



Figure 4.2.4: Momentive BN coatings after ASTM scratch test

4.3 Evaluation in Production

The performance of a Momentive boron nitride coating was evaluated in production at Mercury Marine. The evaluation comprised a sequence of aluminum 380 die casting trials with this release coating. Multiple shots (up to ten) were made without replenishing the coating with satisfactory release. More production testing would be required to determine the maximum number of shots that can be made without replenishing the coating.

J&S Chemical, a supplier of die casting release agents and collaborator in the study, has adopted the design of the Pull-out-Test for in-house evaluation of coatings.

5.0 Conclusions

Semi-permanent coatings are widely used in permanent mold casting. These coatings are generally applied by spraying once per shift and re-touched as needed by spraying or brushing. Silica and alumina based semi-permanent coatings are commercially available. These coatings have not been widely used in die casting. Die casters instead use release agents that are applied before every shot. The main reason is the desire to maintain a short cycle time in die casting. Most semi-permanent coatings used in permanent mold casting are insulating and would extend the cycle time. If applied generously, some can also affect the tight tolerances required from die cast components.

Boron nitride is more conductive than semi-permanent coatings and can be applied in rather thin layers that would not affect the dimensional tolerance of die castings. This study has evaluated boron nitride as a potential semi-permanent coating for die casting. The results indicate boron nitride has excellent release capabilities. The release loads generally increased with dilution ratio of the coating. Multiple shots were feasible without replenishing the boron nitride coating. More experiments would be required to determine the maximum number of shots feasible.

The Pull-Out Test designed and fabricated as part of this project has demonstrated good sensitivity to variations in the coating release capability. The effect of coating

dilution could be readily quantified. The release capability of coatings from different sources were compared and documented.

The surface roughness of the die is an important variable in retention of the coating and affects the release load. More experiments are needed to determine the effect of this variable.

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