Journal of Materials Processing Technology 237 (2016) 386–393

Contents lists available at ScienceDirect

Journal of Materials Processing Technology

journal homepage: www.elsevier.com/locate/jmatprotec

Method to evaluate the adhesion behavior of aluminum-based alloys on various materials and coatings for lube-free die casting

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ARTICLE INFO

Article history:
Received 9 March 2016
Received in revised form 1 June 2016
Accepted 21 June 2016
Available online 22 June 2016

Keywords:
Die casting
Hard coatings
H13 tool steel
Soldering
Sticking
Aluminum adhesion test

ABSTRACT

A simple and direct method called the aluminum adhesion test (AAT) has been developed to provide a quantitative assessment of the adhesion strength between an aluminum alloy and both uncoated and coated H13 die steel. Twelve different hard coatings were investigated. The H13 exhibited soldering similar to that observed by previous investigators. At least three coatings exhibited negligible adhesion and one of these, AlCrN, was further investigated because of its excellent oxidation resistance. Actual die-casting trials were conducted using H13 inserts coated with this AlCrN coating and the trial results confirmed the efficacy of using the AAT for predicting non-sticking behavior.

1. Background and introduction

During high-pressure aluminum die casting, soldering frequently occurs because of the chemical interaction of the casting alloy with the die steel, resulting in the sticking of casting alloy to the die surface as reviewed by Domkin et al. (2009). Bhat et al. (1999) pointed out that soldering not only decreases the quality of cast components, but also causes the die surface damage and the failure of core pins due to excessively high ejection force required to remove the casting. To avoid detrimental soldering, a liquid lubricant is commonly sprayed on the die surface prior to each shot. A large amount of liquid lubricant is required for each shot to prevent soldering and this not only adds time to the casting cycle and increases the cost of producing castings, but also produces effluent that has environmental ramifications. The overall objective of this work is to develop advanced coatings for die casting dies that are non-wetted by liquid aluminum, with the long-term objective of circumventing the need to use liquid-based organic lubricants each shot. This concept is called lube-free die casting.

Hard coatings have been used to protect core pins and inserts in aluminum high pressure die casting dies for decades. Chellapilla et al. (1997) concluded that an ideal coating for die casting applications must exhibit good adhesion to the substrate, good mechanical and tribological properties, high oxidation resistance, chemical inertness to liquid aluminum, and be non-wetting by liquid aluminum. Historically, there are two commonly-used approaches to assess the wetting behavior of liquid Al to different materials. The first is a dipping test as reported by Gorokhovsky et al. (2001), and it has been commonly used in die casting research to evaluate aluminum adhesion and soldering behavior. For this test, a bare or coated core pin is inserted into a crucible containing a molten aluminum alloy for a controlled period of time under atmospheric conditions, and then withdrawn and allowed to cool in air. The soldering resistance is then assessed by measuring the weight loss of the pin after dissolving any adhered aluminum using an aqueous sodium hydroxide solution. Due to the lower weight loss and a lower amount of surface pitting, Gorokhovsky et al. (2001) concluded that multilayer coated pins exhibited superior soldering resistance when compared to both single layer coated pins and uncoated pins. In addition, Joshi et al. (2003) reported that aluminum adheres to both coated and uncoated pins using this test but their results were not quantitative in nature and, therefore, it is difficult to interpret whether the sticking they observed was

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http://dx.doi.org/10.1016/j.jmatprotec.2016.06.031
0924-0136/

related to aluminum wetting behavior, or whether it was caused by mechanical adhesion resulting from the aluminum solidification and shrinking around the pins.

Later, Lin et al. (2006b) developed an "ease-of-release" test with the goal of providing more quantitative measurements of the aluminum adhesion behavior. Core pins with and without coatings were partially dipped into an aluminum alloy melt and then the aluminum was allowed to solidify. The load required to pull the core pin out of the solidified aluminum was measured in an effort to quantify the apparent bonding strength of the core pin to the aluminum matrix. However, the measured load may also not reflect the intrinsic adhesion of aluminum to the die material or coating, due to other factors such as aluminum shrinkage around the pin, and friction between the core pin and the aluminum matrix.

Another more quantitative approach that has been accepted by researchers as a means of studying the wetting behavior of molten metals/alloys on different solid substrates is the sessile drop test as reviewed by Eustathopoulos et al. (2005). This test consists of placing a small sample of solid 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 alloy and the substrate is measured and then correlated to adhesion through the Young-Dupré equation. This approach works well for noble, non-oxide formers, e.g. copper, but is difficult for aluminum and other metals that oxidize readily. Consequently the use of the sessile drop technique for Al and Al alloys results in wide variability in the results. For example, the wetting of sapphire by molten aluminum has been studied by many groups but published results are inconsistent. Shen et al. (2004) summarized the literature data of the contact angles generated from conventional sessile drop tests for molten aluminum on α-alumina ranged from 85° to 105° at about 700 °C. The large scatter in the data is assumed to be caused by variations in the oxide scales that exist on the aluminum surfaces. Shi et al. (2012) calculated that the equilibrium oxygen partial pressure at 800 °C has to be lower than 5×10^{-39} Pa to avoid aluminum oxidation, and such low oxygen partial pressures cannot be practically achieved even under ultra-high vacuum conditions. It is widely accepted that the native aluminum oxide layer can be frequently disrupted at temperatures between 800 and 1000 °C due to a gaseous sub-oxide formation: $4Al(l) + Al_2O_3\ (s) = 3Al_2O\ (g)$. Thus, elevated temperatures (over 800 °C) result in more reliable wetting data and fewer test errors. However, temperatures over 800 °C are not representative of actual die casting processes in which the actual aluminum melt is usually held in the 600–700 °C range prior to casting. In efforts to obtain better intrinsic wetting data at these lower casting temperatures with less oxide layer effects, a modified sessile drop technique has been developed by Shen et al. (2004). Performed under high vacuum, or in an inert gas atmosphere, a small amount of aluminum is melted inside of a ceramic tube and then pushed through the tube so that the melt from the tube end drops onto the test substrate. The contact angle measured using this method is much closer to the intrinsic value and more reliable than that generated by a conventional sessile drop test. However, few data have been reported using this method for aluminum wetting on various hard coatings. In addition, the high cost of vacuum generation, the complex tube designs and process controls, coupled with long test times for the vacuum condition have limited the usefulness of this method. Further, the results generated using this method may not be representative of actual die casting applications because die casting is usually carried out under standard atmospheric conditions.

In this paper, a simple and direct approach, the aluminum adhesion test (AAT), has been developed to provide a more quantitative assessment of the aluminum adhesion behavior on various die casting substrates, especially focusing on hard coatings. One highlight of the AAT is that it uses flat coupons instead of round core pins, and the properties of hard coatings on a flat coupon can be more easily controlled and characterized by standard thin-film test methods, such as nanoindentation, pin-on-disk testing, Rockwell C adhesion testing, scratch testing, etc. The ultimate goal was to better understand the surface interactions between molten aluminum and the various substrates, so that a more reliable laboratory method for predicting performance in actual die casting operations could be developed. In this study, a variety of surface coatings were investigated and the aluminum adhesion to both uncoated and coated flat H13 steel coupons was quantified using this test. At least three coatings showed negligible aluminum adhesion strength and an AlCrN coating from Supplier 1 appeared to be the best candidate because of its excellent oxidation resistance and non-sticking behavior. As a result, actual die-casting trials were conducted on components coated with this coating in order to verify the applicability of the AAT for selecting hard coatings with the greatest likelihood of exhibiting non-sticking behavior in a lube-free environment.

2. Experiments

2.1. Aluminum adhesion test

The methodology used with the AAT was based on two phenomena observed in our preliminary experiments. First, when the liquid aluminum alloy was gradually poured out from an alumina tube an oxidation product from the melt always stuck to the crucible inner wall and bottom, indicating that the liquid aluminum poured from the crucible left the majority of the oxide on the melt surface in the tube. Second, although new oxide scale forms immediately on the liquid during the pouring process, it is thin and easily ruptured upon contact with the coupon such that fresh liquid is expected to come into contact with the coupon surface.

The main items for the aluminum adhesion test include a furnace capable of heating above 700 °C, a ceramic tube, a melting crucible, a pair of tongs, and the H13 steel test coupon. The alumina tube purchased from CoorsTek Inc. is AD-998 grade and Φ28 mm × 40 mm. Preliminary tests revealed that boron nitride (BN) outperformed steel or graphite as a tube material given that BN is highly inert and non-sticking to liquid aluminum, is easily machined, and has good oxidation resistance in air. The BN tubes were machined from a BN rod (Φ25 mm, AX05 grade from Saint-Gobain Boron Nitride Inc.) with an outer diameter (OD) of 25 mm, an inner diameter (ID) of 13 mm and a height of 55 mm. The aluminum alloy used in this study was A380 alloy obtained from a secondary aluminum smelter. The composition of the alloy is shown in Table 1 as characterized by optical emission spectroscopy (OES) following an ASTM standard (E716-10).

The H13 test coupons, provided by Bohler-Uddeholm Corporation in the annealed condition, were machined to sizes of (25–30) × (18–25) × (3–5) mm. The surfaces of the coupons were mechanically ground using a range of SiC papers from 240 grit to 1200 grit, followed by conventional polishing to 1 μm using diamond suspensions on LECO polishing cloths. The polished coupons were rinsed in isopropyl alcohol, gently rubbed with cotton, dried by an electric fan and stored in plastic sample boxes. Prior to aluminum adhesion testing or coating, these coupons were again cleaned ultrasonically in acetone and isopropyl alcohol, and dried using a nitrogen gas flow.

A schematic of the AAT is shown in Fig. 1. It involved the following steps. First, a fresh piece of solid A380 alloy (14.5 ± 1.5 g) was placed into an alumina crucible and heated along with the BN tube from room temperature to 700 °C in about 60 min. An H13 steel test coupon (coated or uncoated) was then transferred into the hot zone of the furnace, the hot BN tube was immediately