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Characteristics of the Molten Aluminium Penetration on the Al_2O_3 Oxide Crust during In-Situ Melting

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ABSTRACT

Pouring free casting was employed for producing investment cast pure aluminum (Al) granules (99.4%) using in-situ melting for eliminating porosity of the cast Al alloys that degrade its mechanical properties. Al granules were charged in the mould and shaken by hand before heated in the furnace at the temperature of 850°C for 30min. Metallography sample was prepared and the characteristics of the Al penetration was analyzed using Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS) and X-ray Dispersive (XRD) analysis to investigate the oxide phases. The granules incompletely in-situ melted to form castings due to the granules oxidized during heating developing thin aluminium oxide (Al_2O_3) crust. The oxide crust kept the liquid Al inside from merging with other melted granules to become a larger melted Al pool. However, the liquid Al successfully penetrated the oxide crust tried to merge with granules in the mould and the penetration length of 19µm causing strong agglomeration between the heated granules that able to replicate the mold geometry. The Al protrusion simultaneously oxidized as it in contact with air hinder further motion of the molten Al and the Al_2O_3 crust created a barrier between the granules during merging of the melted granules. Therefore, suppressing the oxidation of Al granules during heating hypothetically increase the ability of the in-situ melting technique as an alternative of pouring free casting process for reducing porosity in the investment cast Al alloys.

Key words: Aluminum granules, In-situ melting, Oxidation of aluminum

1. INTRODUCTION

All industries currently moving towards lightweight materials for their industries such as electrical, manufacturing, automotive as well as civil construction [1]. Aluminium (Al) is one of the best choices for manufacturing industry. However, porosity is one of the contributing factors that reduce the mechanical properties of the Al alloys castings. Complex combination of several factors affecting the formation of porosity which can be categorized into four (4M) factors: man (foundry men pouring technique), machine (mold), method (pouring action and pouring parameters) and the materials (castings alloys). This study focuses on the materials and method factors that contribute to the porosity formation which degrade the mechanical properties of the castings. Al alloys absorb hydrogen gas in the Al melt during melting process and develop gas porosity during solidification [2]. In addition, oxidation at the surface of the Al melt during melting, transferring and pouring activities creates a thin oxide film contained in the melt. This surface oxide film later became folded due to turbulence called as the bifilm is the aluminum oxide (Al_2O_3), became nucleation sites for the porosity [2],[3]. It is because the nature of the metal that attracted to oxygen and absorb hydrogen at its liquid state, increasing the porosity in the castings. The oxide is unlikely in the castings although metal oxides have benefits in other application [4].

The casting method such as the investment casting parameters: ceramic mold preheat temperature and pouring temperature containing high hydrogen content in the melt increases the porosity resulted to the impairment of the mechanical properties [5]-[7]. Controlling the process parameters resulted with slight improvement in the porosity percentage but the consequences that arose by pouring action during casting process make it less efficient.

In fact, the nature of pouring action in the conventional casting method increases the chances of porosity formation in the castings even though the melt has been treated prior to pouring using several approaches such as inert gas degassing [8],[9], ultrasonic vibration integration to inert gas degassing [8]-[10], salts fluxing [13],[14], and removing the oxide inclusions using electromagnetic [15].

Therefore, turbulence free filling approaches have been employed in casting process to reduce the consequences of pouring action. Tilt casting [16], bottom filling [17], low pressure bottom filling [18],[17] and also in-situ microwave casting) or in-situ melting [20] have been studied. In-situ microwave casting approach produced Al castings with porosity percentage less than 2% indicating the opportunity of the in-situ alternative to be further explored. However, the setup and experiment design are complicated. This research explored the in-situ melting approach developed by [20] which is simpler and cosier for investment casting of Al as an alternative to reduce porosity via pouring free casting approach. This paper evaluates the in-situ melting approach in investment casting of pure Al granules (99.4%) and the characteristics of the molten Al that successfully penetrated the Al_2O_3 oxide crust is presented.

2. METHODOLOGY

A cylindrical mould sized 34mm diameter with 65mm height was prepared according to ordinary procedure for developing investment casting mould. Slurry made of ceramic material was mixed using Zircon ($ZrO_2.SiO_2$) flour sized 75 micron and 30% concentration colloidal silica (SiO_2). The viscosity of the slurry was controlled between 20 and 22s measured using Zahn cup no. 5. Pattern made of wax was then dipped into the slurry and dried one hour at the temperature of 24°C to develop the face coat. The pattern was dipped again into the slurry and immediately stuccoed with fine grain (0.3-0.7mm) alumino silicate ($Al_2O_3.SiO_2$) sand to develop the back-up coat. After dried, the pattern was coated with slurry again and stuccoed with coarser (0.7-1.0mm) alumino silicate ($Al_2O_3.SiO_2$) sand. The pattern was then dipped into the slurry and dried 24hr to create the final coating. The ceramic mould was heated one hour at the temperature 200^{o}C for dewaxing and fired at the temperature 800^{o}C for another one hour.

Aluminium (Al) granules (99.4% pure) with dimension 4 mm diameter and 5mm length were used as shown in Figure 1. The granules were heated in a high-temperature muffle furnace model KSL 1800X for 30 min at 850^{o}C and cooled in the furnace to 200^{o}C.The heated Al granules were characterised using scanning electron microscope (SEM) model S-3400N and field emission scanning electron microscope (FESEM) model Zeiss Supra 35VP, with energy dispersive X-ray spectroscopy (EDS) to analyse the oxide crust.

Figure 1: Ceramic mould filled with Al granules before heating.

3. RESULT AND DISCUSSION

3.1 Morphology of the Al_2O_3 oxide crust

Figure 2 (a) shows SEM image of the surface of the heated Al granules which oxidized during heating in the furnace at 850^{o}C for the duration 30 min. All granules unable to completely in-situ melted and formed castings. Al_2O_3 oxide skin developed and growth on the granules' surfaces encapsulating the granules' body is called as the oxide crust. This phenomenon preventing the melted entity to burst out of the oxide crust (oxide encapsulation) and merge with other adjacent granules. The oxides formed and growth on the surface of the Al granules were agglomerated. FESEM image in Figure 2 (b) shows the cross-sectional images of the oxide crust. The average measured thickness of the oxide crust is 8.515μm having uneven structure. It is due to the oxide layer experiencing localized thickening during heating of the granules forming ridges morphology on the surface.

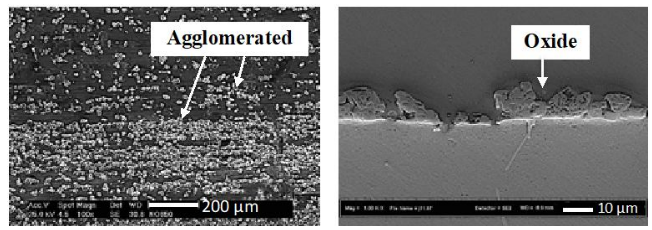
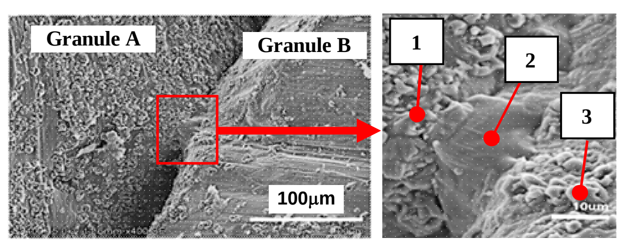


Figure 2: (a) Al_2O_3 oxides crust morphology on the Al granules and (b) Oxide crust thickness of the Al granules after heat-ed at 850^{o}C for 30 min.

3.2 Al penetration during heating at 850^{o}C for 30 min

Penetration of the liquid Al was found between granules on the heated sample as shown in Figure 3 (a). The Al protrusion out of the oxide crust indicates the in-situ melting of Al granules was present although unable to produce castings(Figure 3b). The Al granules surfaces oxidize as early as the heating temperature reach 400^{o}C and creating an oxide crust keeping the liquid Al from burst out for merging with other granules that experiencing similar phenomenon.



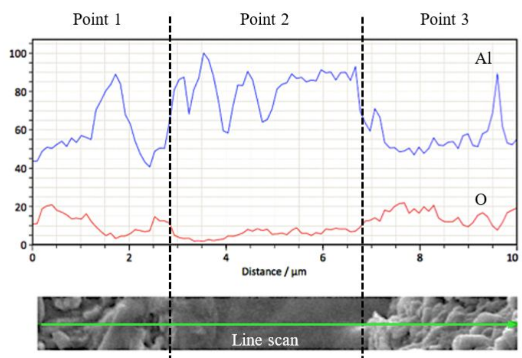
(a) (b)

Figure 3: (a) Al penetration on the Al_2O_3 oxide crust, (b) protruded Al

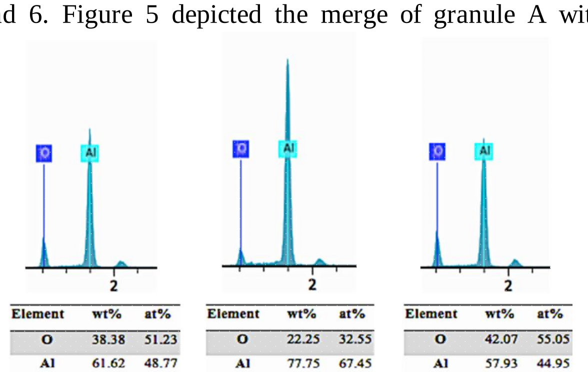
The Al protrusion as in Figure 3 (b) between the two heated granules was 19μm significantly shows that the molten Al successfully burst the oxide crust trying to merge with other molten Al from other granules. However, as it in contact with air in the furnace environment it oxidised too thus prevented it from flowing further. The tiny penetration of Al on the granule's surfaces with multiple similar occurrences, able to form the agglomeration and replicating the mould geometry.

3.3 SEM, FESEM and EDS analysis

EDS line scan analysis conducted on the protruded Al as shown in Figure 4 (a) showed that high intensity of oxygen at point 1 and point 3 compared to point 2. The EDS analysis by area selection revealed Al element at point 2 was relatively high 77.75 wt. % compared to point 1 and point 3 which are 61.62 wt.% and 57.93 wt.% respectively as shown in Figure 4 (b-d). Point 2 had high intensity of oxygen suggested that the oxide was formed due to oxidation of the liquid Al that penetrated out of the oxide crust. Liquid Al spontaneously oxidised once it in contact with air during heating that recorded 22.25 wt % oxygen in the EDS spectrum as indicated in Figure 4(c).



(a)



(b) (c) (d)

Figure 4: (a) EDS line scan analysis at the bridge between granule A and B. EDS analysis at (b) point 1, (c) point 2 and (d) point 3 on the bridge as shown in Figure3 (b).

FESEM and EDS was conducted on the cross section of the agglomerated granules (sample A and B) as shown in Figure 5 and 6. Figure 5 depicted the merge of granule A with granule B, however the entity oxidised and the oxides continuously grow hindering further merging of the liquid Al.

The measured thickness of the oxide crust was between 3.295 and 6.198 µm as depicted in Figure 5. The EDS line scan analysis in Figure 6 shows 21.26 wt.% oxygen element found at the bridge between granules A and B (Figure 5) was Al_2O_3.

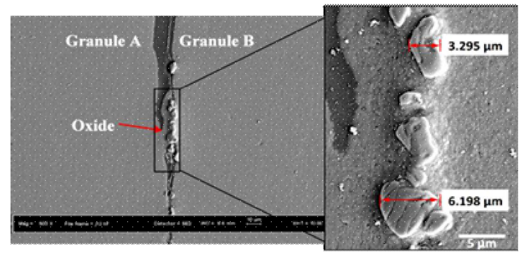


Figure 5: FESEM cross-sectional images showing the bridge between granules A and B

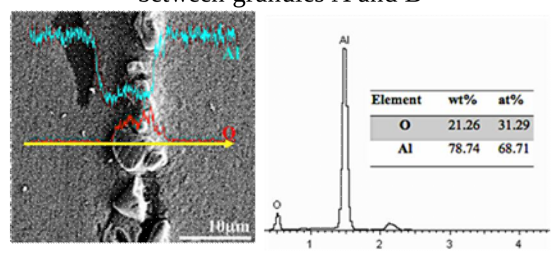


Figure 6: EDS line scan result conducted across the bridge

The mechanism for the fusion of the Al granules during heating is schematically illustrated in Figure 7. During heating, Al granules in the ceramic mould oxidised at the temperature 400°C forming a thin oxide skin on the granules' surfaces. When the temperature increased to 850°C, the Al granules was in-situ melting when the temperature reached 660°C and the liquid portion of Al expand although being kept in the oxide crust. The change of the granule's volume during expansion develops tension to the oxide crust and also the coefficient of thermal expansion of Al that was ten times greater than the Al_2O_3 as well as the expansion of the Al in the liquid phase is higher than the solid Al causing the oxide crust to crack [21], [22]. Therefore, the liquid Al seep through the cracks to merge with other granules unfortunately oxidised as depicted in Figure 7 (a). These phenomena explain the agglomeration of the granules in the ceramic mould. In addition, oxidation of the Al granules proceeds simultaneously as the phenomenon takes place increasing the thickness of the oxide crust during heating in the furnace. The granules physically deformed was due to the impact of the granules on the upper section of the mould and contraction during slow cooling in the furnace that leads to volume shrinkage as shown in Figure 7(b).

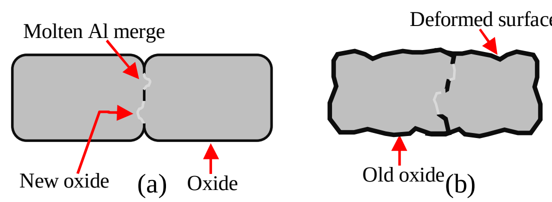


Figure 7: A schematic illustration of the agglomerated granules. (a) liquid Al merge with adjacent granules and kept inside the oxide crust and (b) deformed Al granules.

4. CONCLUSION

It can be concluded that the Al granules experienced in-situ melting during heating at the temperature 850°C in 30min. Penetration of the molten Al observed under the SEM and EDS analysis proved that the molten Al tried to merge with other granules for developing a pool of molten Al and finally became castings. Active oxidation of the granules' surface develops the Al_2O_3 oxide crust that kept the molten Al inside and the simultaneous oxidation of the molten Al protrusion hinder the complete merge of granule in the ceramic investment casting mold. Suppressing the oxidation of Al granules during heating potentially increase the ability of in-situ melting approach for investment casting of Al alloys particularly for reducing porosity in the castings as it is one of pouring free casting technique.

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