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Investigation of pouring temperature and holding time for semisolid metal feedstock production

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Abstract. Semisolid metal (SSM) processing, as a kind of new technology that exploits forming of alloys between solidus and liquidus temperatures, has attracted great attention from investigators for its thixotropic behaviour as well as having advantages in reducing porosity, macrosegregation, and forming forces during shaping process. Various techniques are employed to produce feedstock with fine globular microstructures, and direct thermal method is one of them. In this paper, the effect from different pouring temperatures and holding times using a direct thermal method on microstructure and hardness of aluminium alloy 6061 is presented. Molten aluminium alloy 6061 was poured into a cylindrical copper mould and cooled down to the semisolid temperature before being quenched in water at room temperature. The effect of different pouring temperatures of 660 °C, 680 °C, 700 °C, and holding time of 20 s, and 60 s on the microstructure of aluminium alloy 6061 were investigated. From the micrographs, it was found that the most globular structures were achieved at processing parameters of 660 °C pouring temperature and 60 s holding time. The highest density and hardness of the samples were found at the same processing parameters. It can be concluded that the most spheroidal microstructure, the highest density, and the hardness were recorded at lower pouring temperature and longer holding time.

1. Introduction

Semisolid metal (SSM) processing is an attractive and advanced technology to produce lower cost and higher quality of engineering parts [1]. It occurs between liquidus and solidus temperature range, where within this range, fluidity of the molten metal changes significantly. SSM processing enables the production of a spheroidal microstructure instead of a dendritic microstructure as normally achieved by conventional processing technique. The thixotropic effect of the alloys produced by this method allows them to flow when shear force is given, but thicken if shear force is released [2]. SSM processing has the ability to produce near-net-shape products with high mechanical properties [2–5]. Therefore, this process is often desired for producing products with complex shape geometries. Furthermore, it has the advantages of prolonged die life due to less thermal shock, and low shrinkage porosity defect as compared to other conventional processes [3,6]. Gas entrapment is eliminated due to laminar cavity fill during SSM process. As a result, a high integrity product which has a uniform and fine microstructure is produced. Due to the potential advantages of this novel technology, research has been accelerating along with the adoption of the technology by industry since the 1990s.

SSM processing mainly comprises of three important stages; (a) preparation of globular feedstock billet, (b) reheating to the semisolid condition, and (c) forming process [7,8]. The key towards SSM

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processing is by having a non-dendritic or spheroidal feedstock billet. In recent years, many different processing routes have been developed to produce spheroidal microstructures, such as liquid metal routes, solid state routes and combination methods [9,10]. Among all these methods, the direct thermal method (DTM) is a simple route that can develop non-dendritic or spheroidal microstructure with reduced equipment and processing costs [8,11–13]. In this method, the liquid alloy is poured into a cylindrical copper mould to produce spheroidal microstructures. High thermal conductivity of copper mould is the reason that enables rapid cooling to a low superheat liquid alloy. This action creates numerous nucleation sites within the molten alloys during solidification. Heat matching between the mould and the molten alloy results in a pseudo isothermal hold within the solidification range [12]. As a result, spheroidal microstructure is formed during the holding time. Then, quenching in room temperature water is performed to freeze the formation of spheroidal microstructure.

Aluminium alloys are widely used in mechanical construction and automotive industries. This stems from the fact that aluminium is a lighter structural metal in use as its density is 2.7 g/cm³ as compared to 4.51 g/cm³ for titanium alloys and 7.2 g/cm³ for steels [14,15]. Most of aluminium alloys products are made from conventional casting process. According to Kenney et. al. [16], the energy needed to heat aluminium alloys for semisolid processing is 35% less than that required to heat the same aluminium alloys for casting process. Plenty of studies utilizing SSM processing were conducted on aluminium silicon alloy [1,12,17–22]. It is due to the alloy has better flow ability, but fared poorly in mechanical properties as compared to wrought aluminium alloys. However, recent trend has seen that several research studies are moving towards wrought aluminium alloys [8,23–26] by taking advantage of the higher mechanical properties. Ahmad et. al. [8] had conducted a study on wrought aluminium alloy 7075 to investigate the relationship between pouring temperature and holding time of DTM by using aluminium 7075 in order to produce a spheroidal microstructure for the SSM feedstock. They found that fraction solid was strongly dependent on temperature whilst combination of pouring temperature and holding time of 665 °C and 60 s, respectively, produced the best spheroidal primary phase microstructures. Furthermore, higher pouring temperature led to production of higher amount of secondary phase, which in turn result in greater fluidity of the semisolid material. In addition, structures with greater amount of secondary phase were found to have the lowest strength and hardness properties.

Based on the literature, aluminium silicon alloys were the most popular choices of material for SSM processing, as mentioned previously. Limited information about material behaviour, especially on wrought aluminium 6061 as a raw material in DTM was the motivation for this experimental works. The aim of this present work was to investigate the relationship between pouring temperature and holding time of DTM by using aluminium alloy 6061 to produce a spheroidal microstructure for the SSM feedstock.

2. Experimental procedure

Commercial aluminium alloy 6061 was used for this experimental work with the chemical compositions determined by using Optical Emission Spectrometer and is presented in table 1.

Element (wt. %)	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr	Ni	Ti
As-received sample	97.400	1.00	0.290	0.030	0.530	0.570	0.009	0.011	0.019	0.020

Table 1. Chemical composition of aluminium alloy 6061.

2.1. Direct thermal method experiment

1 kg of aluminium alloy 6061 ingots was placed in a graphite crucible and heated to a temperature of 715 °C by using the resistance heated box furnace. The melt was poured into a cylindrical copper mould with 1 mm wall thickness, 25 mm in diameter, and 90 mm in height once the desired temperature of the melt was obtained. The temperature of the molten metal was measured by using a

K-type thermocouple that was attached to a data logger. The K-type thermocouple was used because it has good sensitivity and can measure the temperature up to 1350 °C. The different pouring temperatures were set at 660 °C, 680 °C, and 700 °C. Then, the molten metal was held for 20 s, 40 s, or 60 s respectively after pouring it into the mould. It was later quenched into room temperature water as soon as the holding time for each copper mould was achieved. Another billet with pouring temperature of 700 °C was allowed to solidify without quenching in order to achieve microstructure for the billet with the normal solidification condition. Schematic diagram for DTM apparatus is presented in figure 1 as follows:



Figure 1. Schematic diagram for the DTM used in the experimental work with (a) experimental set-up and (b) dimension for the copper mould.

2.2. Density test

Feedstock billets from the DTM process were subjected to a density test. This test was performed to determine the porosity levels for each feedstock billets. The feedstock billets were first weighted using a mass balance. Next, the feedstock billets were submerged into water. The water readings before and after the feedstock billets being submerged into water were recorded. The density of feedstock billets was then calculated by Archimedes' principle as presented in equation (1) as follows:

$$\rho_{\rm f} = \frac{W_{\rm f} \times \rho_{\rm w}}{W_{\rm w}} \tag{1}$$

where $\rho_{\rm f}$ is the density of feedstock billet in g/cm³, $W_{\rm f}$ is the weight of the feedstock billet measure by a mass balance in g, and $W_{\rm w}$ is the weight of feedstock billets fully submerged in water with the reading in ml. Whilst, $\rho_{\rm w}$ is the density of the water with the value of 1 g/cm³.

2.3. Metallography sample preparation

Samples from the DTM process were cut in a rectangular shape and approximately 2 mm in thickness by using a sectioning machine. Then, all samples were mounted in Bakelite to prepare them for grinding and polishing stages. The samples were then ground using 240, 600, 800, and 1200 grit paper, followed by polishing using 6, 3, and finally 1-micron diamond paste. Next, the samples were etched by using Keller's reagent. Optical microscope was then used to view the microstructure and a specific software was used to capture the microstructure images.

2.4. Vickers hardness test

Room temperature hardness was performed with a Vickers hardness instrument. Samples were mounted in Bakelite and subsequently lightly polished to ensure a flat surface, whilst minimizing loss of material. At least 3 measurements were made for each sample and ensuring different segments were analysed. Vickers hardness values were calculated by taking the average of the two diagonal lengths for the indent.

3. Results and discussion

3.1 Density test

Average densities and porosity levels of the samples were determined by Archimedes' principle. Based on results in table 2, there are samples that have large standard deviations than others. This shows that the results between the specimens are not consistent. This might be caused by impurities which contaminate the aluminium alloy 6061 during the DTM experiment. It might be due to graphite, which was placed along the wall of the mould for easier removal of the samples. Other than that, another explanation which contribute to the differences of results might be due to porosity that happen within the samples. Porosity normally can occur when the material undergoes shrinkage during solidification process. A cavity of air within the specimen will cause the billet to have lower weight to volume ratio which will result in the specimen having less mass while maintaining volume and ultimately results in having lower density than expected. One of the densities obtained from this experimental works was slightly higher than the density found in literature for aluminium alloy 6061 which normally at 2.70 g/cm³ [27]. The porosity level in the samples was determined by comparing the density obtained from the experimental work and the literature. The average density of the sample which obtained slightly higher than 2.70 g/cm³ indicated lower porosity and sample with lower average density showed higher porosity content. The results in table 2 is presented in a form of graph as shown in figure 2.

Pouring temperature (°C) / Holding time (s)	Average density (g/cm ³)	Porosity level
660 / 20	2.70 ± 0.09	High
680 / 20	2.68 ± 0.05	Moderate
700 / 20	2.63 ± 0.03	Low
660 / 60	2.74 ± 0.04	Highest
680 / 60	2.49 ± 0.11	Lowest
700 / 60	2.69 ± 0.01	Moderate
700 / NQ ^a	$2.61{\pm}0.03$	Low

Table 2. Average density and porosity level of aluminium alloy 6061 samples at different pouring temperature and holding time determined by Archimedes' principle.

^a No quenching

According to figure 2, the specimen with the highest density which is 2.74 g/cm³, is achieved by the combination of pouring temperature and holding time of 660 °C and 60 s, respectively. This finding suggests that the specimen has the lowest porosity. Combination of pouring temperature 680 °C with holding time 60 s produces the lowest density of sample that is 2.49 g/cm³ which

indicated that it has the highest porosity content. The sudden drop of density might be due to porosity which occur during solidification. The standard casting technique sample (700 °C of pouring temperature and without quenching) has a density of 2.61 g/cm³, which is lower than the density of sample which is subjected to 660 °C pouring temperature with 60 s holding time. This finding might suggest that the mechanical properties of the thixoformed billet is better than the one which is formed by the standard casting technique, as increased in density would lead to increase of strength and hardness.



Figure 2. Density of the samples at different pouring temperature and holding time.

3.2 Characterization analysis

The microstructure for pouring temperature of 660 °C, 680 °C, and 700 °C are presented in figure 3. It was observed that the microstructure for pouring temperature of 660 °C was more spheroidal compare with the microstructure for pouring temperature of 680 °C and 700 °C. This finding shows that lower pouring temperature leads to the formation of more spheroidal microstructure and uniform in shape. According to Ahmad et. al. [8], lower pouring temperature retarded the formation of microstructure and was considered the most significant factor in DTM. This in turn, determined the successful of the process. Furthermore, cooling rate is also affected by the pouring temperature. Higher cooling rate is achieved when lower pouring temperature is used, as less superheat needs to be extracted from the sample. On the other hand, higher pouring temperature can cause reduction in cooling rate. The samples need longer time to extract the heat from the above to below liquidus temperature. In this study, it was obvious that the low pouring temperature of 680 °C and 700 °C, as the low pouring temperature helps to increase the cooling rate. This result is in agreement with the findings by Ahmad et. al. [8].



Figure 3. Microstructures for different pouring temperatures and holding times with (a) pouring temperature 660 °C and holding time 20 s, (b) pouring temperature 660 °C and holding time 60 s, (c) pouring temperature 680 °C and holding time 20 s, (d) pouring temperature 680 °C and holding time 60 s, (e) pouring temperature 700 °C and holding time 20 s, and (f) pouring temperature 700 °C and holding time 60 s.

3.3 Vickers hardness test

Figure 4 shows the graph of samples hardness at different pouring temperature and holding time. From this data, the highest hardness is 62.1 HV which experienced by the combination of 660 °C pouring temperature with 60 s holding time. Whilst sample subjected to 700 °C pouring temperature with 60 s holding time has the lowest hardness of 48.4 HV. Based on the results, it can be seen that sample with the highest hardness corresponds to the sample with the highest density in section 3.2, where both of these attributes occur at 660 °C pouring temperature with 60 s holding time. This finding is in agreement with the previous work by Ahmad [28], where higher density led to increase in strength and hardness.



Figure 4. Hardness of the samples at different pouring temperature and holding time.

4. Conclusions

The effect of pouring temperature and holding time to the evolution of the microstructure, density, and hardness of aluminium alloy 6061 were successfully investigated. The results obtained from the experimental works have shown that the combination of lower pouring temperature and longer holding time produced a globular microstructure. Lower pouring temperature has led to higher cooling rate, which in turn results in more spheroidal microstructure. The same processing parameters have been observed to produce the highest density and hardness within the material. It can be concluded that the combination of pouring temperature at 660 °C and the holding time of 60 s, produced a spherical microstructure feature, highest density, and hardness for the 6061 feedstock billets.

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