Direct Thermal Method of Aluminium 7075
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Abstract. The evolution of microstructure affect from different pouring temperatures and holding times using a direct thermal method is presented in this paper. The direct thermal method is one of the thermal techniques which are used to produce semi-solid metal feedstock. In this experimental work, aluminium 7075 alloy was used. The experiments were carried out by processing a sample with a 0.7 °C/s cooling rate to evaluate the formation of the microstructure. In direct thermal method experiment, a molten 7075 was poured into a cylindrical copper mould at different pouring temperatures of 680 °C and 660 °C meanwhile the holding time of 20 s, 40 s and 60 s before quenched into room temperature water. The sample processed by the cooling rate of 0.7 °C/s produced a large microstructure. The formation of a spheroidal microstructure was obtained with the combination of a suitable pouring temperature and holding time. The pouring temperature of 665 °C and the holding time of 60 s produced a finer and uniform microstructure that is suitable for semi-solid feedstock.

Introduction

Semi-solid metal (SSM) processing occurs between liquidus and solidus temperature range in which fluidity of molten metal changes greatly. Instead of a dendritic microstructure as per conventional processing, a spheroidal microstructure is formed by using SSM processing. The main advantage of SSM processing compared with other conventional processes is a low shrinkage porosity defect [1-3]. It occurs from smooth die filling action which eliminated air entrapment. This produced a high integrity product that has a fine and uniform microstructure. SSM processing also are able to produce a near-net-shape product. It is used to manufacture a variety of products with complex shape geometries.

SSM processing involves with three important steps which consist of feedstock preparation, reheating or holding in a semi-solid condition and forming operation [4]. The feedstock billet which originally is in dendritic is transformed to a spheroidal microstructure by using an appropriate method and technique. The selection of the technique used is based on material type, weight and size. The spheroidal feedstock is then reheated or held in the semi-solid temperature range with a heating regime to ensure a homogenous temperature throughout the billets, by using a heating mechanism such as induction heating. Finally, the spheroidal feedstock is processed by using conventional process such as casting or forging termed thixocasting or thixo forging respectively, which has a heating condition in the range of semi-solid temperature. These processes use less force compared with excessive force in conventional process during the forming action which is affected by the existence of spheroidal microstructure within feedstock billet.

There are several methods used recently to produce SSM feedstock [4]: methods consisting of liquid metal routes, solid state routes and combination methods. In the liquid metal routes, raw material is melted above its liquidus temperature and processed to create a spheroidal microstructure. Mechanical stirring for instance is the technique used to form a spheroidal microstructure which involves a shaft rotation within the liquid metal [5, 6]. Solid state route on the other hand is a process which produces a spheroidal microstructure without melting the raw material. Strain induced melt activation and recrystallization and partial melting is the technique which is grouped in this category.
The spheroidal microstructure deformation is achieved by hot working the raw material either above or below the recrystallization temperature and then cold working. The combination of methods involves a combination of more than one technique to produce spheroidal feedstock. The self-inoculation method is the combination method which joins the cooling slope and the swirled enthalpy equilibrium device [9]. The higher superheating temperature is able to be used which indicates its advantage compared with other SSM techniques.

Direct Thermal Method (DTM) is a technique which involves pouring the molten alloy into a cylindrical copper mould to produce a globular microstructure [10, 11]. The poor external heat transfer characteristic of a cylindrical copper mould is a factor which allows rapid cooling to a low superheat alloy. This initial rapid cooling action increases the formation of multiple nucleation events within alloys during solidification. Globular shape in the primary solid is achieved by isothermal arrest which enables a diffusion-dominated process. Heat matching between the cooper mould and alloy which results from a very thin mould wall catalysed the instantaneous cooling condition. In order to capture microstructure features after the desirable pseudo-isothermal holding, the mould is quenched in room temperature water.

Fraction solid is one of the important parameters in SSM forming [12]. It determines the flow ability of the material and influences the formation of the microstructure and defects. Lashkari and Ghomashchi [12] suggest the SSM processing of the feedstock to obtain a low viscosity and a high fraction solid content. The viscosity helps the material to flow inside the mould cavity meanwhile high fraction solid prevent the possible defect to deform. In SSM forming (thixocasting), the fraction solid was typically occurs in the range of 0.30 to 0.50 [8] and in some other cases in the range of 0.60 to 0.70 [13, 14].

Aluminium 7075 is a material which is used to produce high stresses components. Mechanical properties of 7075 are higher than other aluminium alloy systems [15]. It is also known as ‘difficult to cast’ alloy due to its narrow solidification range and tend to form a hot tearing defect. There were several works which use a 7075 for SSM processing routes [8, 16]. The 7075 was thixoextruded into a die cavity by using the recrystallization and partial melting method. The selection of a proper thixoforming temperature range which results an increment in mechanical properties and reduces defect deformation. In addition, the material flows inside the die cavity was also influenced by the speed of ram used. A finer shape and more spherical microstructure throughout sample with higher tensile properties for thixoforming feedstock also occur inside the material which was produced by using the recrystallization and partial melting process [7, 17]. However, a brittle appearance was found in the thixoformed sample. In order to overcome this problem, a post-heat treatment process was applied.

In the literature it was found that the DTM was able to produce a spherical microstructure for the SSM feedstock [18, 19]. The combination of a suitable pouring temperature and a holding time which proven to produce a finer microstructure feature. The effects of different material used for the mould and the wall thickness were also separately investigated [20]. However, the previous research concentrations of DTM were more towards the mould and less attention were given to the raw materials used. The previous studies concentrated merely on the aluminium silicon alloy which has advantage in the flow ability but lower in the mechanical properties compared with the aluminium 7075. The aim of this study was 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. In addition, the effects of solidification rate and fraction solid resulting from the different pouring temperature and holding time used were particularly determined.

**Experimental Procedure**

Commercial aluminium 7075 was used for this experimental work with the chemical compositions were determined by using Oxford Instrument Arc Spectrometer and together from the literature is presented in Table 1.
Table 1: The chemical composition of the aluminium 7075

<table>
<thead>
<tr>
<th>Source</th>
<th>(wt%) Al</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
<th>Mg</th>
<th>Mn</th>
<th>Si</th>
<th>Ti</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experiment</td>
<td>88.5</td>
<td>0.20</td>
<td>2.02</td>
<td>0.24</td>
<td>2.38</td>
<td>0.12</td>
<td>0.14</td>
<td>0.09</td>
<td>6.04</td>
</tr>
<tr>
<td>ASM [21]</td>
<td>87.1-91.4</td>
<td>0.18-0.28</td>
<td>1.2-2.0</td>
<td>&lt;0.5</td>
<td>2.1-2.9</td>
<td>&lt;0.3</td>
<td>&lt;0.4</td>
<td>&lt;0.2</td>
<td>5.1-6.1</td>
</tr>
</tbody>
</table>

Normal solidification experiment was performed to evaluate the 7075 microstructure formation which was solidified at a lower cooling rate compare with a higher cooling rate in copper mould. The cooling rate of the molten 7075 inside the copper mould for DTM was found approximately at 1 °C/s [19]. Graphite crucible of 100 mm diameter and 100 mm height was used to place a 1 kg 7075 billet. The crucible was then heated to a temperature 750 °C by using a box furnace. The crucible with molten metal was then placed in a forced air flows to increase the cooling rate. The cooling rate was recorded by using a K-type thermocouple equipped with a data acquisition (DAQ).

Another 1 kg aluminium 7075 ingot which was placed in the graphite crucible was heated to a temperature 700 °C by using the resistance heated Carbolite 1600 box furnace. Once the desired temperature of the melt was obtained, it was poured into a cylindrical copper mould with 1 mm wall thickness, 25 mm in diameter, and 100 mm in height. The different pouring temperatures were set at 665 °C and 685 °C. After pouring molten metal into the mould, it was held for 20 s, 40 s or 60 s at semi-solid temperatures, before quenched into room temperature water. Schematic diagram for DTM apparatus is presented in Fig.1 as follows:

![Figure 1: Schematic for DTM used in the experimental work.](image)

The semi-solid temperature range of 7075 alloy is quoted with the different solidus and liquidus temperatures depending on the source of reference. In one previous work this range was reported as 472.8 °C to 636.7 °C [22]. Fig. 2 shows the fraction solid vs temperature curve of 7075 at 0.7 °C/s cooling rate. The cooling rate was calculated from the portion of the cooling curve between 10 °C and 20 °C above the liquidus temperature. This fraction solid curve was selected because it was close to DTM cooling rate. It was then used to estimate the amount of solid inside the material before quenching. Temperatures for 7075 which was in the range of 0.5 to 0.7 fraction solid were between 618 °C to 630°C respectively (see Fig. 2).

Samples which were obtained from a starting material and a solidified alloy from a normal solidification experiment were divided at three different locations. The normal solidification specimens were sliced 50 mm from the top, at the centre, and both walls of the crucible. The DTM specimens were cut at 10 mm, 20 mm and 95 mm from the top of the mould respectively by using the lathe machine. The samples were mounted in Bakelite and were ground using 240, 600, and 1200 grit paper, polished using 9 micron and given a final polish to 0.5 micron by using diamond paste. The samples were then etched by using Keller’s etch. SEM Reichert MeF2 universal camera optical microscope was then used to view the microstructure and Buhler Omnimet Enterprise software was used to capture the microstructure images.
Results and Discussion

Starting Material. The microstructure of a starting material is presented in Fig. 3. Grain structures of 7075 were elongated and unrecrystallised by the contrast within the elongated grains.

Figure 3: As-received microstructure for aluminium 7075 viewed using optical microscope with 40x magnification.

Normal Solidification. The microstructure of aluminium 7075 which solidified inside a graphite crucible is presented in Fig. 4. The molten alloy solidified at a cooling rate of 0.7 °C/s (before the liquidus temperature). The formed microstructures were large, dendritic and less spheroidal compared with the microstructure produced by using the DTM. This result reveals the cooling rate which was used for processing a material gives a significant effect to microstructure formation.
Different Pouring Temperature. The microstructure for pouring temperature of 685 °C and 665 °C are presented in Fig. 5. It was apparent from these figures the microstructure for pouring temperature of 685 °C was less spheroidal compared with the microstructure for pouring temperature of 665 °C. The microstructure in Fig. 5 (a), Fig. 5 (b) and Fig. 5 (c) shows there were several spheroidal microstructures occurred but it was dominant in Fig. 5 (d), Fig. 5 (e) and Fig. 5 (f). This result shows that the formation of more spheroidal microstructure was at a lower pouring temperature. The microstructure was more spherical and in a uniform shape.

Different Holding Time. Microstructure for specimens with the different pouring temperatures and holding times are presented in Fig. 5. The role of holding time in the DTM was to make sure the adequate temperature was achieved before quenching. As the temperature in the copper mould drops at 1°C/s, the quenching temperatures were estimated accordingly. The quenching temperatures for pouring temperature of 685 °C with the holding time of 60 s, 40 s and 20 s were at 625 °C, 645 °C and 665 °C respectively. Meanwhile, the quenching temperatures for 665 °C with the holding time of 60 s, 40 s and 20 s were at 605 °C, 625 °C and 645 °C.

In order to evaluate the relationship between microstructure evolution and quenching temperature, the respective quenching temperatures were then referred back to Fig. 2 to estimate the value of a fraction solid. The fraction solid values for the quenching temperature of 665 °C, 625 °C and 605 °C were approximately at 0.06 and 0.08 respectively. The formation of more spherical microstructure was at the pouring temperature of 665 °C with the holding time of 60 s as in Fig. 5 (d). At this temperature, fraction solid within the sample was approximately at 0.80. Less spherical microstructure occurred at the pouring temperature of 685 °C with the holding time of 20 s as shown in Fig. 5 (c) at approximately 0 fraction solid. The microstructure of the sample with the pouring temperature of 685 °C and the holding time of 40 s (Fig. 5 (b)) was similar to microstructure in Fig. 5 (a) that equivalent to 0.60 fraction solid. This was due to a similar quenching temperature which was applied for both specimens that reflects the same fraction solid within sample. These results indicated that the value of fraction solid which occurs inside the molten metal during quenching plays the important roles in the formation of microstructure.
Figure 5: Microstructures for different pouring temperatures and holding times with (a) pouring temperature 685 °C and holding time 60 s, (b) pouring temperature 685 °C and holding time 40 s, (c) pouring temperature 685 °C and holding time 20 s, (d) pouring temperature 665 °C and holding time 60 s, (e) pouring temperature 665 °C and holding time 40 s and (f) pouring temperature 665 °C and holding time 20 s.

**Effect of Cooling Rate.** Cooling rate determine the successful of DTM to produce a spheroidal microstructure. The principle of DTM which allows the quick heat change retards the formation of microstructure. The chilling effect which occurred in the copper mould produced the smaller nuclei and affects the formation of a spheroidal microstructure. The formation of the microstructure depended on cooling rate which was used for material processing [23-25]. Lower cooling rate produced a dendritic microstructure which is less spheroidal. It was clearly shown by the
Effect of Pouring Temperature. Lower pouring temperature is the important parameter in producing a globular microstructure in DTM. The lower pouring temperature retards the formation of microstructure [19]. The literature shows the pouring temperature was the important factor in DTM which determines the successful of the process. The pouring temperature is related to the cooling rate. The lower pouring temperature leads to a higher cooling rate as less superheat has to be extracted. The higher pouring temperature leads to the slower cooling rate as more time needed by the systems to extract the heat from the above to below the liquidus temperature. The low pouring temperature which was used in this work produced a globular microstructure as the low pouring temperature helps to increase the cooling rate. The microstructure which was executed with the pouring temperature of 665 °C was more spherical compared with the pouring temperature of 685 °C.

Effect of Holding Time. The results show that fraction solid has influenced the formation of a microstructure. The formation of a globular microstructure was dominant at a higher fraction solid (see Fig. 5 (d)), which was accordance to 0.80 fraction solid. The fraction solid is one of the important parameter in SSM which determines the flow ability of the material, similar to flow ability in conventional casting process. The different fraction solids used in this experimental work showed the apparent changes in microstructure evolution. In SSM processing, the fraction solid was typically used in the range of 0.3 to 0.7 [14]. In this range, the material has the suitable microstructure features which able to produce more globular microstructure. However, 0.80 fraction solid which was used in this experiment produced a fine microstructure feature compared with other samples. The results show that, the formation of a globular microstructure for 7075 was higher at the higher volume of fraction solid. The formation of a less spheroidal microstructure in other specimens due to the specimens were quenched while it still in the liquidus condition. Thus, reduces the ability to transform to a globular microstructure.

Conclusion

This paper has described the effect of a pouring temperature and a holding time to the evolution of the microstructure. The results obtained from the experimental works have shown that the combination of adequate pouring temperature and a holding time produced a globular microstructure. In particular, the cooling rate and volume of fraction solid play the important rules in determine the formation of a spheroidal microstructure. The higher cooling rate and higher volume of fraction solid which were used for SSM feedstock processing has produced a finer, globular and a uniform microstructure. The combination of pouring temperature at 665 °C and the holding time of 60 s which equivalent to fraction solid at 0.80, produced a spherical microstructure feature for the 7075 feedstock billets.

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References