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Microstructure Characteristic of Aluminium 6061 Semi-Solid Feedstock Billet Produced with Direct Thermal Method

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ABSTRACT

Semisolid metal processing is a promising technique to overcome the imperfections of the components produced by the traditional casting process. As a semi-solid metal processing technology, a direct thermal method is capable of producing a feedstock billet with a spherical microstructure which is ideal for thixoforming. In this experiment, molten aluminium 6061 was solidified in a copper mould at temperatures between 660 °C and 680 °C. The melt was retained in the copper mould for 20 seconds, 40 seconds and 60 seconds. The molten alloy in the mould was solidified in room-temperature water after the required holding time. The microstructure development of the feedstock billets was evaluated after the feedstock billets were removed from the mould. The results show that sample (S660-20), which had a pouring temperature of 660 °C and a holding time of 20 s, has the smallest grain size with an average of 2507.87 μm^2 . It was discovered that the ferret's diameter, aspect ratio and circularity were found to be 69.4 μm , 1.34 and 0.75 respectively. Clearly, the microstructure of S660-20 was more refined and spherical. The rapid cooling of the molten metal in the copper mould resulted in a more spherical grain structure. According to the findings, the microstructure was solely dependent on the heat convection between the molten alloy and the copper mould. The rapid cooling resulted in the production of finer and more spherical microstructure that was ideal for thixoforming operations.

1. Introduction

The metal casting industry plays a crucial role in the manufacturing field in producing a wide range of components with different dimensions [1]. Improving the overall material quality such as mechanical properties and microstructure formation is the main key challenge in modern casting industries. The mechanical characteristics of materials are one of the most essential fundamental elements in the development of well-designed components utilised in a variety of industries. The

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quality of the material influences the selection criteria for structural engineering applications. Nevertheless, casting imperfections such as shrinking, pores and hot cracking have a considerable economic influence on the casting business [1-5]. In the conventional casting process, the formation of porosity will lead to low mechanical properties and a high possibility of product rejection. Nevertheless, semi-solid metal (SSM) processing may overcome the imperfections produced by the traditional casting method.

SSM processing is frequently employed in the automotive and electrical industries to cast light alloys such as aluminium and magnesium [6,7]. In contrast to the traditional casting method, the SSM processing technique employs semi-solid slurries due to their distinctive rheological behaviour allowing for a more regulated die filling process [8,9]. In 1970, SSM processing was found for the first time by Flemings and his coworkers at the Massachusetts Institute of Technology (MIT). The viscosity of the material was found to influence the development of the microstructure during SSM processing. The SSM processing takes place within the liquidus and solidus temperature of the alloy, where the fluidity changes significantly [10-13]. The molten metal viscosity can differ significantly within the solidification phase. The SSM processing promotes the formation of a spherical microstructure instead of the dendritic structure that results from the conventional casting process.

The microstructure formation influenced the viscosity of the semi-solid alloy, where the structure of the dendrite has low flowability as compared to the equiaxed structures with the same fraction solid [9,14]. The tendency of the structure of the dendrite to interact between themselves under the external force application impeded the flowability of the materials. In contrast, the globular structure gives better flowability due to its shape and ease to move under small external forces [15]. The tiny and spherical microstructure leads to high malleability as the particles are efficiently mobile and there are fewer collisions between them [13,16,17]. The SSM processing method can be classified into two main routes: thixo and rheo. These two techniques are commonly employed to process semi-solid slurries and give complicated results.

The thixo-route has the ability to produce nearly reticulated components that have high mechanical properties and have been reported to have lower internal porosity [17]. The two-step of thixo routes comprises the transitional solidification stage where the processed semi-solid feedstock billets are sized into the desired specification and reheated to a solid-liquid temperature range fraction solid before being cast into components. During the forming process, the thixo route effectively implements the semi-solid behaviour and reduces macro segregation and porosity [18]. The thixo routes offered advantages where the high mechanical properties were produced compared to the conventional casting process. In contrast, the rheo routes entail the production of SSM slurries that consist of globular microstructure, which is then solidified into semi-solid form. The SSM slurries in rheo routes were then shaped in the forming form without an intermediate solidification process. Moreover, the rheo routes have attracted many researchers' attention due to their low-cost operation as the waste metal can be recycled in-house [19]. The semi-solid state of non-dendritic solid particles was created from an alloy in its fully liquid condition. The slurry was chilled to obtain the solid fraction and then the components were cast. Components shaped from SSM slurries contribute to the overall effectiveness of energy management and manufacturing.

Aluminium alloys play a significant role in most materials used in mechanical design and automotive sectors. Due to its flexibility and versatility, the traditional casting process is currently being used to process the wrought aluminium alloy to produce components [20]. The quality of materials is crucial for the production of parts and components in the manufacturing industry, such as in the production of automotive components [21]. One of the SSM processing techniques is the Direct Thermal Method (DTM), which allows the production of spherical microstructures by manipulating the processing parameters throughout the solidification process. The low superheat

alloy was retained in the cylindrical copper mould with high thermal conductivity to achieve globular microstructure by using the DTM [21-23]. Ahmad *et al.*, [11] discovered a correlation between processing factors such as casting temperature and holding duration during the DTM process for aluminium 7075. The result noted that with the processing parameters of 660 °C and 60 s, it was apparent that the most globular primary phase morphology within the SSM feedstock billets. In SSM processing, the spherical grain structure increases the viscosity of the metal, resulting in near net-shaped components with excellent tensile properties. In order to produce a fine and spherical grain structure, it is crucial to regulate the processing conditions during solidification. These current studies seek to determine how different processing parameters affect the microstructure aluminium alloy 6061 produced by DTM.

2. Methodology

The raw material of alloy 6061 was cleaned with SiC sandpaper before conducting the composition test. The grinding process started with coarse sandpaper up to fine sandpaper in the range of P120 to P1200. The purpose of grinding is to guarantee that the surface of wrought alloy is flat and to remove surface imperfections. The Foundry-Master Uv spectrometer was examined to determine the composition of wrought aluminium alloy. The average composition of wrought aluminium alloy 6061 is shown in Table 1.

Table 1

Chemical composition of wrought alloy 6061

Composition	Aluminium	Magnesium	Silicon	Copper	Iron	Chromium	Zinc	Manganese	Others
Weight (%)	97.7	0.922	0.725	0.265	0.178	0.079	0.011	0.011	0.11

2.1 Direct Thermal Method (DTM) Experiment Procedure

The graphite crucible was filled with the raw aluminium alloy 6061, which was then heated to 900 °C. The crucible was removed from the box furnace when the alloy was completely melted and the temperature of the molten metal was recorded. The temperature of the melt was determined using K-type thermocouple that connected to a Graphtec midi logger GL2200 before being poured into the mould. After the required pouring temperature was reached, the molten metal was poured into the copper mould. The pouring temperature was set from 660 °C to 680 °C with a holding time of 20 s, 40 s and 60 s. The copper mould was then released into the water tank to complete the solidification process. Figure 1 shows the apparatus setup during the DTM process and Table 2 represents the parameters used in this experimental work.

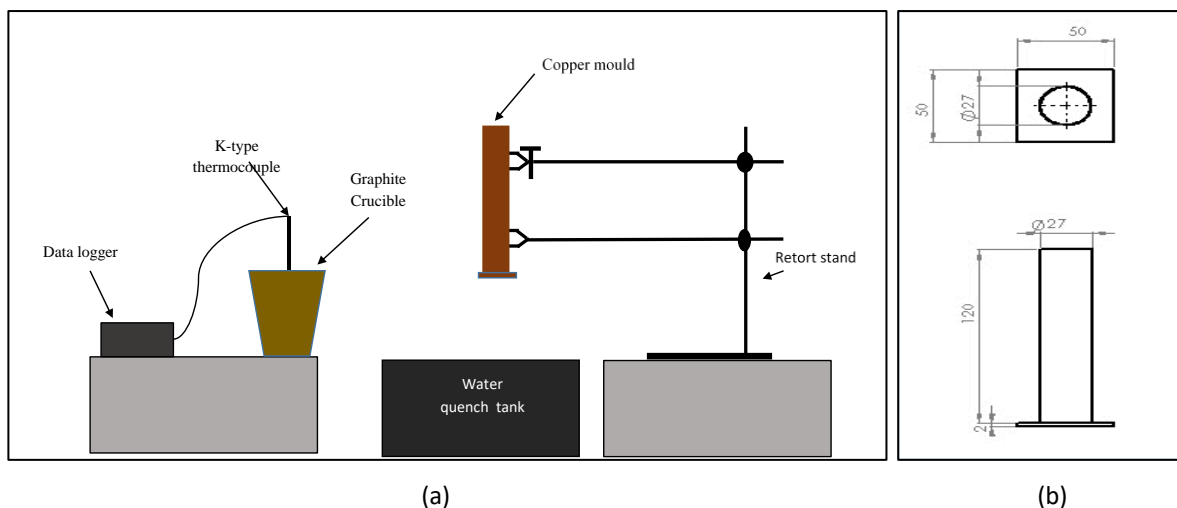


Fig. 1. Experimental setup (a) DTM (b) Copper mould dimension

Table 2

Processing parameters during DTM process

Sample	Pouring temperature (°C)	Holding time (s)
(S660-20)	660	20
(S660-40)	660	40
(S660-60)	660	60
(S680-20)	680	20
(S680-40)	680	40
(S680-60)	680	60

2.2 Metallographic Sample Preparation

The billets of solidified SSM feedstock were retrieved from the copper mould. To remove the contaminants, the samples were cut with a precision cutting machine at a distance of approximately 5 mm from the bottom of the DTM samples. The microstructure analysis samples were cut 20 mm and were then halved using precision diamond cutting with a 230-rpm cutting speed. The samples were mounted with Bakelite resin using 60 bars of pressure, 2 minutes of heating time and 3 minutes of cooling time. The sample was then manually ground with 120, 320 and 600 grit silicon carbide to minimise cutting damage.

The samples were then polished to eliminate the scratches left after grinding. Different cloths were used for 6 µm, 3 µm and 1 µm diamond suspension at a speed of 250 rpm. Finally, the samples were polished with a 0.05 µm colloidal silica dispersion. Etching was the final step in the preparation of the metallographic sample. The etching process was carried out using Keller reagent. The etchant was dabbed into cotton wool soaked with Keller's etchant reagent. Prior to being rinsed with distilled water, it was held for 15 seconds. The formation of microstructures was recorded with an optical microscope equipped with Motic software and the microstructures were analysed with Image J software. Image J analysis calculated the average grain size, aspect ratio, circularity and diameter of each microstructure formation. The circularity and aspect ratio can be calculated using Eq. (1). and Eq. (2).

$$\text{Circularity} = (4\pi \times \text{area}) / (\text{perimeter}^2) \quad (1)$$

$$\text{Aspect Ratio} = \text{major axis} / \text{minor axis} \quad (2)$$

2.3 Scanning Electron Microscopy (SEM) & Energy-Dispersive X-Ray Spectroscopy (EDXS) Analysis

The SEM and EDXS analyses were carried out to characterize the grain structure using Jeol JSM-7800F. Compositional data for the primary and secondary phase regions were analysed using the EDXS detector. The solid grain structure depicted the primary phases, whereas the liquid structure adjacent to the solid grain represented the secondary phases. The working distance was set at 10 mm to obtain accurate results and five measurements were made for each sample. In each measurement, the regions of the primary and secondary phases are covered by five spectral lines. The average composition for each primary and secondary phase region was analysed and the composition of each line spectrum was recorded.

3. Results

3.1 DTM Experiment

Based on the literature, the liquidus and solidus temperatures of wrought aluminium alloy 6061 are 652 °C and 582 °C, respectively. The microstructure formation was observed and analysed to describe the influence of different combinations of pouring temperature and holding time on the SSM feedstock billet. Figure 2 and Figure 3 show the microstructure for the pouring temperature of 660 °C and 680 °C with a holding time of 20 s, 40 s and 60 s. The microstructure formation study, shown in Figure 2 and Figure 3, demonstrated significant variation in microstructure development amongst all of the samples.

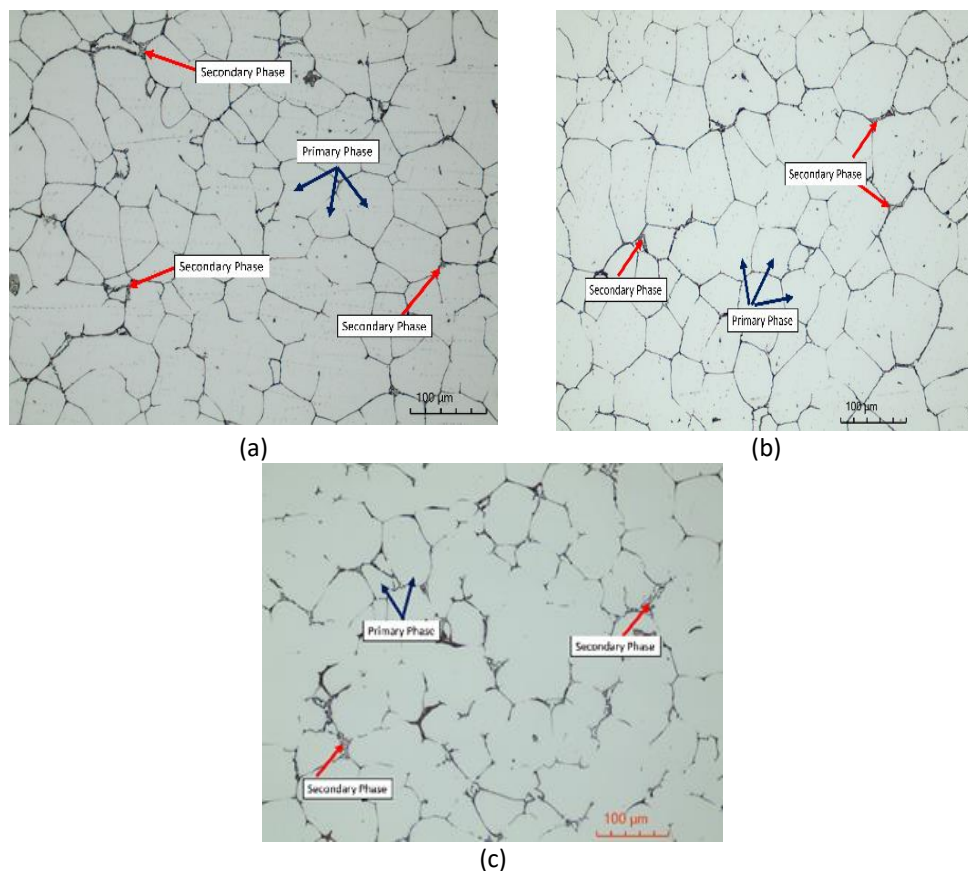


Fig. 2. Microstructure formation of (a) S660-20, (b) S660-40 and (c) S660-60

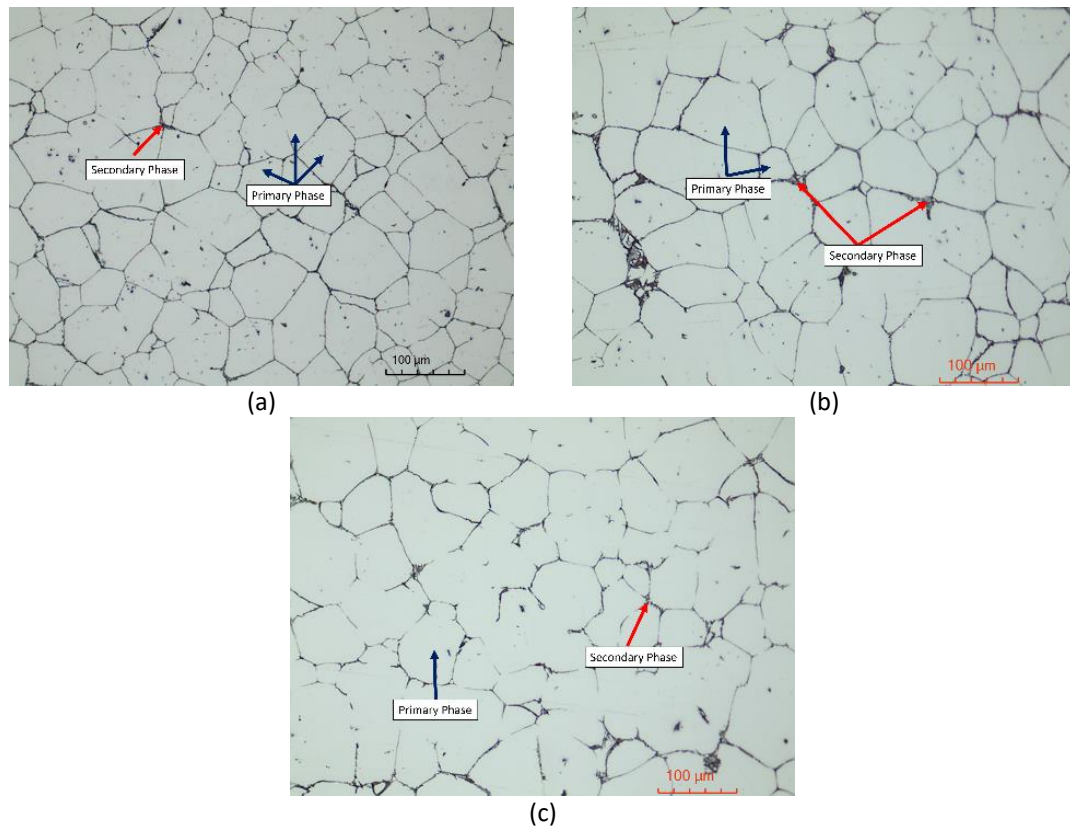


Fig. 3. Microstructure formation of (a) S680-20, (b) S680-40 and (c) S680-60

It was apparent that S660-20 produced a fine grain size structure at $2507.87 \mu\text{m}^2$. In contrast, S680-60 produced the largest grain size structure at $5254.69 \mu\text{m}^2$. Meanwhile, the circularity, aspect ratio and Feret diameter for S660-20 were 0.75, 1.34 and $69.4 \mu\text{m}$ respectively. The circularity, aspect ratio and Feret diameter for S680-60 were 0.69, 1.64 and $95.81 \mu\text{m}$ respectively. Figure 4 shows the result of the grain structure formation.

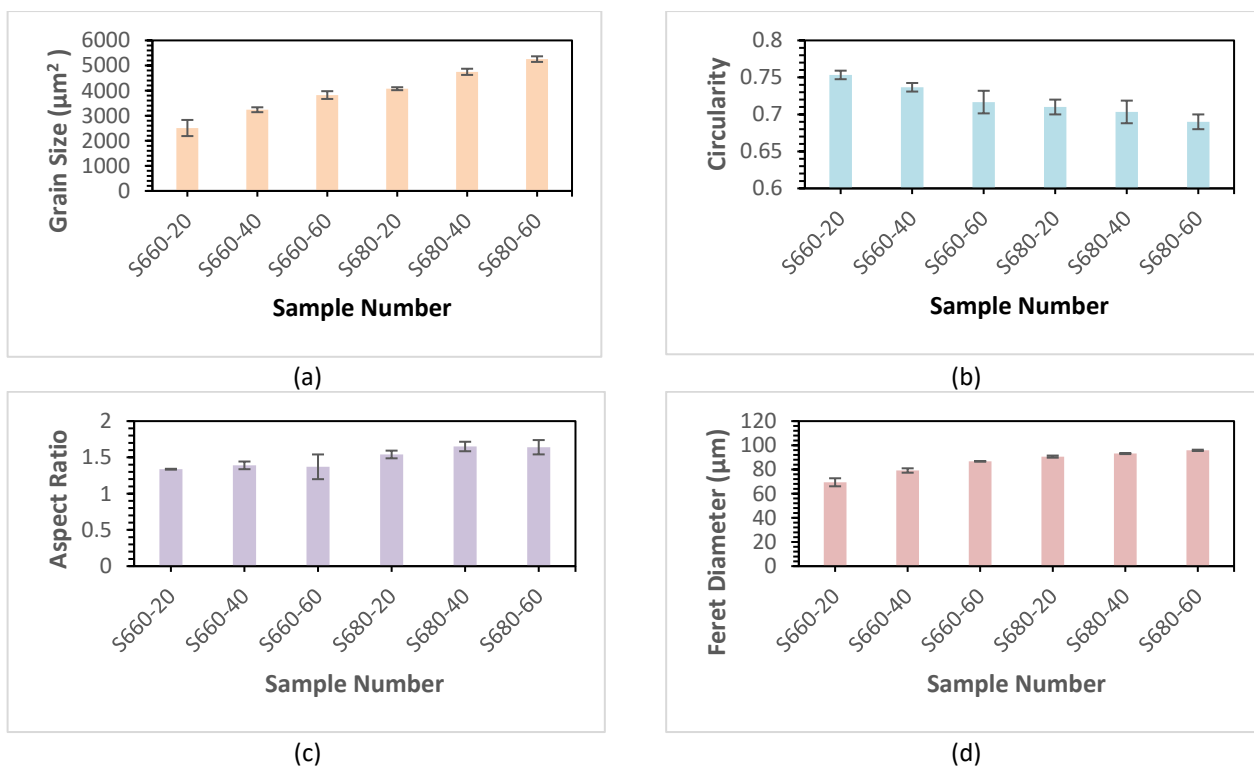


Fig. 4. Analysis of grain formation with (a) average grain size (b) average circularity (c) average aspect ratio (d) average Feret diameter

The microstructure of the quenched samples varied significantly relying on the different combinations of processing parameters. A previous study suggested that the lower pouring temperature may result in finer primary and secondary morphology, resulting in more spheroidal grain structure. The pouring temperatures of S660-20, S660-40 and S660-60 are only slightly above the liquidus temperature of the wrought aluminium alloy 6061 so that less superheat can be retrieved from the copper mould and a high cooling rate is achieved. Low pouring temperature delays grain structure formation during solidification [24-26]. Due to the ability of the cylindrical copper shape to dissipate heat, the molten aluminium rapidly cooled to a semisolid state. The rapid cooling process promotes the development of numerous nuclei during solidification. In contrast, S680-20, S680-40 and S680-60 produce larger grain size as compared to the other samples. The cylindrical copper mould extracts more superheat when the pouring temperature is higher than the liquidus. Hence, S680-20, S680-40 and S680-60 will exhibit a slow cooling effect throughout the solidification process.

However, the grain size and circularity between samples S660-20, S660-40 and S660-60 were apparent. The variance in the size distribution of the particles is due to the varying holding times during solidification. The prolonged period promoted grain formation within the alloy, resulting in a larger grain size [10,27]. In contrast, S680-20, S680-40 and S680-60 have larger grain sizes compared to other samples. The higher pouring temperature beyond the liquidus temperature resulted in more time needed for the temperature to be extracted by the mould [10]. The rapid cooling during the solidification process has increased the development of the solid primary phase without slowing down the formation of the nuclei. As a result, the grain structure expands. The high pouring temperature of the molten metal results in slow cooling rates as it takes longer to extract the heat.

During the solidification process, the fraction solid within the alloy increases as it approaches the solidus temperature of the alloy. The increase in solid fraction leads to the development of larger nuclei and affects the circularity of the grain structure. In addition, the pouring temperature, which is just above the liquidus temperature of the alloy and the shorter holding time result in a fine and

spherical grain structure, leading to improved flowability throughout the forming process. The fine and spherical microstructure improves formability due to increased mobility of the particles and reduced collisions between particles inside the grain structure [13,16,17]. Thus, the fine and globular microstructure is suitable for SSM processing.

3.2 SEM & EDXS Analysis

The primary phases area and secondary phases area were subjected to the SEM and EDXS analysis to characterize the microstructure formation during the solidification. The chemical composition of S660-20 was chosen due to this parameter produced small and globular grain structure. In both the primary and secondary phases, three significant elements were identified. The three most important elements were aluminium (Al), silicon (Si) and magnesium (Mg). The selected region of both primary and secondary grain structure for EDXS analysis were depict in Figure 5 and Figure 6. The chemical composition of the DTM samples for both the primary and secondary phases is shown in Table 3 and Table 4. The SF660-20 comprises between 98.94% and 99% aluminium in the primary phase area and between 88.77% and 74.89% aluminium in the secondary phase area. In addition, the magnesium and silicon content in the primary phase range is 0.48 to 4.57 % and 0.52 to 0.83 %, respectively, while the secondary phase range contains 1.49 to 10.53 % magnesium and 10.26 to 25.0 % silicon.

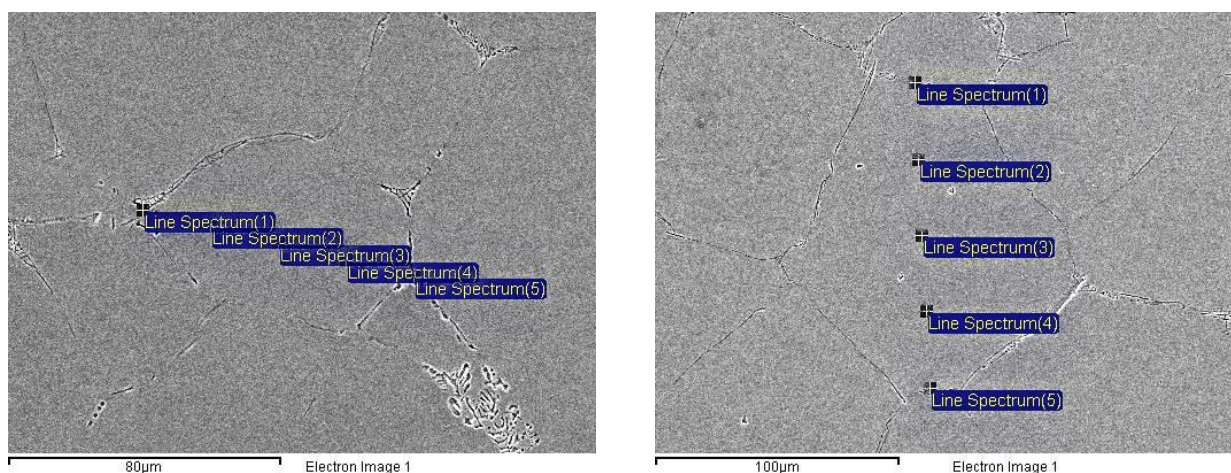


Fig. 5. The selected primary phases area

Table 3
 Composition of primary phases area

Elements	Aluminium	Magnesium	Silicon
Spectrum (wt%)	98.94-99	0.48-4.57	0.52-0.83

The precipitation sequence was the cause of the compositional differences between the main and secondary phase domains in the SSM raw material block. Because the sample was quenched in a water that was at ambient temperature, the DTM solidification process could not produce a complete precipitation sequence. As a result of the metastable β precipitation, the excess Si element in the processed aluminium alloy generates stoichiometric Mg_2Si . The mechanical properties of the alloy are improved by the formation of stoichiometric Mg_2Si [27]. The ratio of Si to Mg components increases during the early precipitation process and changes the agglomeration structure [28,29]. The alloy properties are degraded by the small amount of Si elements precipitating in the alloy, making the β -metastability less stable.

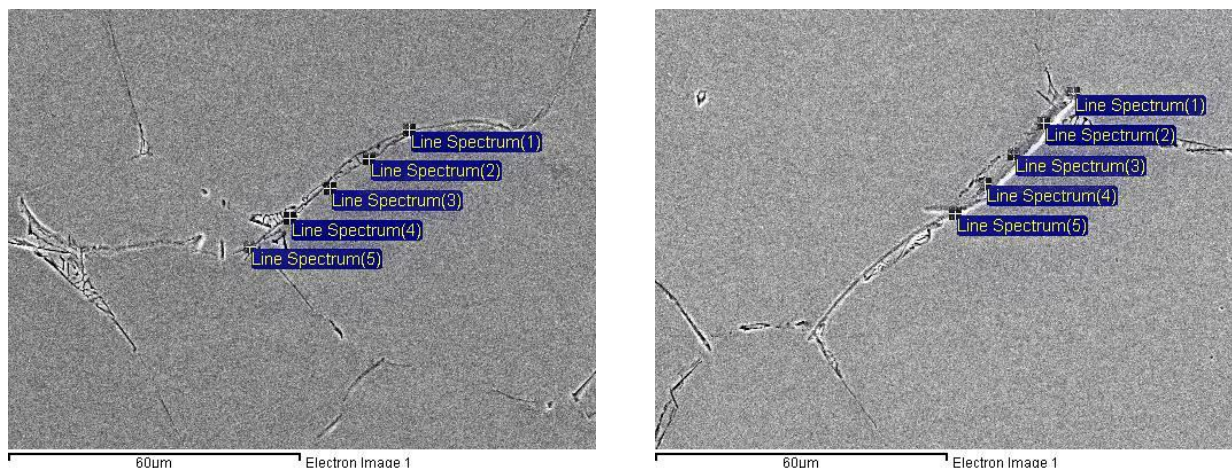


Fig. 6. The selected secondary phases area

Table 4

Composition of primary phases area			
Elements	Aluminium	Magnesium	Silicon
Spectrum (wt%)	88.77-74.89	1.49-10.53	10.26-25.04

4. Conclusions

The experimental work investigated the effect of different combinations of processing parameters on the formation of the microstructure of the wrought aluminium alloy produced by the DTM process. S660-20 produced a tiny grain size of $2507.87 \mu\text{m}^2$ with a pouring temperature of 660°C and a holding time of 20 s. The largest grain size was $5254.69 \mu\text{m}^2$ in S680-60 with a pouring temperature of 680 and a holding time of 60 s. Due to the effects of varying pouring temperatures, S660-20 and S680-60 differ significantly from each other. The low pouring temperature of alloy 6061 (S660-20), which is slightly above the liquidus temperature, leads to slow cooling, which favours the formation of nuclei and results in smaller grain size. Compared to the other parameters, S660-20 had the most spherical grain structure, with a circularity of 0.75. The variation in grain size distribution is a consequence of the different holding times during solidification. The prolonged time period stimulated grain development in the alloy, resulting in higher grain size. The heat convection of the alloy when it is extracted from the copper mould is liable for the formation of the spherical microstructure. In order to achieve the spherical microstructure required for SSM processing, heat must be rapidly extracted from the molten alloy, hence slowing the formation of the dendritic microstructure. The lower pouring temperature improves nucleation during solidification and leads to a more spherical microstructure and smaller particle sizes. The fine and most globular grain structure increase the fluidity of the alloy. Small and spherical grain particles produce better formability due to the better movement and less collisions between the particles. Thus, S660-20 produces small and globular grain structure is suitable for SSM processing.

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