Thermal analysis of 6061 wrought aluminium alloy using cooling curve analysis-computer aided (CCA-CA) method

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Abstract. The objective of this work was to investigate the effect of different cooling rates on microstructure and solidification parameters of 6061 wrought aluminium alloy. Wrought Al6061 were heated and melted at temperature of 800°C in a graphite crucible. A thermocouple was placed at the centre of molten under different conditions. Solidification characteristics are recognized from cooling curve and its first derivative curves which have been plotted using thermal analysis technique. In this study, molten were solidify in different airflow conditions. The results of thermal analysis show that the characteristics of Al6061 are influenced by cooling rate. The cooling rates in this experiment ranged from 1.3 to 2.4 °C/s and increasing the cooling rate affects the solidification parameters. Images of microstructures were taken to present its relation with cooling rates.

1. Introduction

Aluminium plays an important role and extremely useful engineering material with very good physical properties [1]. Even though pure aluminium has low strength, it can be alloyed to gain high strengths, so that it finds many applications in modern technology [1]. As aluminium alloy widely used in industry, various research was done on improving its product. Few demands for high quality are fewer defects such as porosity, shrinkage and higher mechanical properties [2]. Based on rheology processing of aluminium alloy, alternative casting and forging process has developed for fuel-efficiency demands in automotive industry [3]. Alternative metal manufacturing is namely semisolid metal processing (SSMP). In order to gain successful SSMP, solidification curve of material data is the key aspect [4-6].

Thermal analysis is well-known in the casting process as a technique provides thermal profiles data. The information gained from TA widely used to ensure quality of metals in casting. TA consider as useful for commercial because it is simple, inexpensive and provides consistent results.[7-10]. This method capable to provide data about relationship between cooling curve characteristics and material melts. Meanwhile data of grain size, dendrite coherency point, solid fraction and latent heat can be quantified from an accurate thermal analysis system.[9].

Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA) and Cooling Curve Analysis (CCA) are several thermal analysis variations. It works with similar principles, to measure temperature changes of sample which occur as it is heated or cooled through phase transformation. For cooling process, latent heat releases will be interpreted as changes in the slope of
cooling curve. As phases appear in small amounts, it is difficult to detect directly from cooling curve. Thus, first derivative of curve is used to identify metallurgical characteristics [10].

DTA and DSC used a small (~200mg) sample in its method. DTA measure temperature difference during the heating or melting [11, 12]. DSC on the other hand measures the differences in heat flow. Useful information of metal alloys from both methods able to help understanding alloy process then results in high-quality production [13]. CCA used in this study is known as easy to implement, did not need an expensive instrument and complicated sample preparation. A macro-sized sample can be analysed and compared to thermal information.

DTA method used a small (~200mg) sample to measure temperature differences during the heating or melting [11, 14]. Meanwhile, DSC method measures the differences in heat flow. Both methods provide useful information for metal alloys, thus alloy process is well understood for high-quality production [13]. CCA, as used in this study, is known as easy to implement on foundry floor compared to the other two methods, which need expensive instruments and complicated sample preparation. Other than that, compared to DSC, macro-sized samples use in CCA can be analysed and compared to thermal information.

The objective of this research are to investigate effect of cooling rates on microstructure and solid fraction of 6061 wrought aluminium alloy. The liquidus temperature, solidus temperature, solid fraction and microstructure for different cooling rates were determined.

2. Method

Chemical composition of aluminium alloy 6061 as shown in table 1 was determined using Optical Emission Spectrometer.

| Composition | Wt (%) |
|-------------|--------|
| Al          | 96.300 |
| Si          | 1.250  |
| Fe          | 0.784  |
| Cu          | 0.040  |
| Mn          | 0.509  |
| Mg          | 0.680  |
| Zn          | 0.042  |
| Cr          | 0.150  |

Wrought aluminium 6061 alloy with a mass of 20 g was placed inside a 30 mm x 40 mm graphite crucible. The crucible was heated by using KX-5188 series auto control high-frequency induction heating machine at temperature of 800 °C. The aluminium 6061 molten alloy then allowed to solidify in three different conditions. The first condition was allowed the molten aluminium solidify at a normal ambiance temperature and this cooling condition is called normal cooling. The second condition was allowed the molten aluminium solidify with minimum fan blower air forced through crucible and this cooling condition is called medium cooling. The third condition was allowed the molten aluminium solidify with maximum fan blower air forced through crucible and this cooling condition is called fast cooling. In order to measure the cooling curve of the molten aluminium, K-type thermocouple was positioned at the centre of the crucible and immersed 20 mm from the top of crucible head. This thermocouple was connected to a GL-220 Data logger and linked to the computer with GL220 APS software for the data captured purposes. In order to get higher accuracy results, the data logger was set at 10 Hz/channel. The schematic for this experimental set-up is presented in figure 1 as follows:
Figure 1. Schematic for this experiment set-up used for normal cooling condition.

The microscopic samples were taken at the center and edge of solidified alloy. The sample was then mounted by using SimpliMet1000 Automatic Mounting press mounting machine and grinded by using Metkon Forcipol 2V grinding machine with the rotation of 240-300rpm and grit specification P240, P600, P800 and P1200 of abrasive paper respectively. The sample was then polished and etched with a Keller solution and the microstructure image of the sample were taken.

Data extract from GL220 APS software were used to plot the cooling curve graph of temperature against time from different cooling rate condition. The first derivative graph was calculated from cooling curve graph. The first derivative was used to enhance accuracy for alloy feature characteristics determination. The baseline is a zero-curve of cooling curve by assumption of no phase transformation during solidification [8, 15]. The baseline was gained from the differential temperature of liquidus and solidus from the first derivative graph using third-order polynomial [16]. The solid fraction was calculated from accumulative area between first derivative and baseline based on following equation (1):

$$f_s = \frac{\int_{t_1}^{t_2} \left[ \frac{dT}{d\tau} \right]_cc - \left[ \frac{dT}{d\tau} \right]_{Bl} dt}{\int_{t_1}^{t_2} \left[ \frac{dT}{d\tau} \right]_cc - \left[ \frac{dT}{d\tau} \right]_{Bl} dt}$$

(1)

where CC is cooling curve and Bl is baseline.

3. Results and discussion

3.1 Thermal analysis

Cooling curve analysis will able to provide information on latent heat releases because it is corrospective to metallurgical phases within the alloy [10]. Types of phases and morphology are dependent on chemical composition. Other than that, cooling rate is known to have an effect on the morphology and distribution of phases in microstructure of an alloy.

Cooling and first derivative curve with a baseline for a normal cooling are plotted in figure 2. The cooling rate for this condition was at 1.3 °C/s. From the graph (Figure 2), it is shown that liquidus and solidus temperature occurred at 642 °C and 576 °C respectively. Meanwhile, the result for medium cooling condition is shown in figure 3. The cooling rate for medium cooling condition was at 2.0 °C/s (Figure 3). The liquidus temperature for this medium cooling condition was at 640 °C and the solidus temperature was at 579 °C. The fast cooling condition with fastest air fan blown cooling and first curve with a baseline is shown in figure 4. The results in figure 4 shows that the cooling rate was at 2.4 °C/s and the liquidus and solidus temperature were at 647 °C and 585 °C respectively.
Figure 2. Normal cooling condition for cooling rate of 1.3°C/s with cooling curve, first derivative curve and baseline.

Figure 3. Medium cooling condition for cooling rate of 2.0°C/s with cooling curve, first derivative curve and baseline.
Prior studies have noted the importance of liquidus and solidus determination for SSM research. The liquidus and solidus temperature are quite crucial factors as it will become a reference temperature for SSM feedstock billet processing [17]. In this current research work, the results show that the formation of liquidus and solidus temperature were slightly different compared to other findings [16]. It was found in previous research [16], the liquidus temperature for aluminium 6061 in normal cooling was at 663.6 °C meanwhile for solidus temperature at 551.2 °C.

3.2 Solid fraction
The solid fraction of a solidifying melt is defined as the percentage of the solid phase(s) that have stimulated at a point in time between the liquidus and solidus points [10]. It is crucial to have an accurate solid fraction data for computer simulations of solidification processes. Suitable solid fraction range for semisolid metal processing is 30% to 70%. The large solid phase would reduce shrinkage and due to its high viscosity, parts can be produced with easy governable flow tips. [18]. Based on the graphs, solidification rates are high at the beginning of solidification and reduce by the end of solidification.
Figure 5. Graph of temperature against solid fraction for normal cooling condition (1.3 °C/s).

Figure 6. Graph of temperature against solid fraction for medium cooling condition (2.0 °C/s).
Figure 7. Graph of temperature against solid fraction for fast cooling condition (2.4 °C/s).

3.3 Microstructure
Microstructure results for each cooling rate condition are shown in figure 8 until figure 13. Based on table 2, circularity comparison between center and edge for all condition, did not show significant differences between center and edge of sample. Other than that, fast cooling condition microstructure has finer grain size compared to other condition microstructure. This is constant with findings from previous research [16, 19, 20].

Figure 8. Microstructure of normal cooling condition at (a) center of sample (NCC).
Figure 9. Microstructure of normal cooling condition at edge of sample (NCE).
Figure 10. Microstructure of medium cooling condition at center of sample (MCC).

Figure 11. Microstructure of medium cooling condition at edge of sample (MCE).

Figure 12. Microstructure of fast cooling condition at (a) center of sample (FCC).

Figure 13. Microstructure of fast cooling condition at edge of sample (FCE).

Table 2. Grain size area and circularity for each part of microstructure.

|            | Grain size area / μm² | Circularity | Aspect Ratio |
|------------|-----------------------|-------------|--------------|
| NCC        | 175411.846            | 13.826      | 32.913       |
| NCE        | 223908.43             | 13.059      | 30.976       |
| MCC        | 24416.327             | 11.113      | 42.452       |
| MCE        | 30833.897             | 10.599      | 36.665       |
| FCC        | 55275.278             | 11.249      | 33.662       |
| FCE        | 5232.892              | 12.393      | 38.510       |

4. Conclusion
Effect of different cooling rates on thermal profiles and microstructure on 6061 wrought aluminium alloy was successfully investigated. The liquidus and solidus temperature for normal cooling
conditions (1.3°C/s) were 642°C and 576 °C respectively. Meanwhile, medium cooling conditions (2.0°C/s) have liquidus temperatures at 640°C and solidus temperature 579°C. For fast cooling conditions (2.4°C/s), liquidus and solidus temperature were 647 °C and 585 °C respectively. There was no significant difference for liquidus temperature for all cooling conditions. However, solidus temperatures increase constantly from normal to fast cooling conditions. This study also has proof that cooling rate has significant effect on microstructure of 6061 aluminium alloy. The fast cooling condition has finer microstructure compared to other conditions.

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