



# Assessment of the effect of grain refinement on the solidification characteristics of 319 aluminum alloy using thermal analysis

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## ARTICLE INFO

### Article history:

Received 3 August 2009

Received in revised form 5 November 2009

Accepted 5 November 2009

Available online 22 November 2009

### Keywords:

319 aluminum alloy

Thermal analysis

Grain refinement

## ABSTRACT

The effect of different amount of Al–5Ti–1B grain refiner on the macro and microstructural features and characteristic parameters of the cooling curve of 319 aluminum alloy were studied using thermal analysis. Important parameters in liquidus, eutectic Si and eutectic Cu-phase regions have been calculated using the first derivative cooling curves. The results indicated that, the solidification parameters such as nucleation and growth temperatures of various phases, undercooling temperatures, solidification range and total solidification time were affected by grain refining. Also, latent heat and fraction solid were determined for the different amount of grain refiner for 319 alloy. It has been found that the fraction solid is completed fast by addition of Al–5Ti–1B grain refiner.

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## 1. Introduction

Among aluminum–silicon alloys, the 319 family of alloys are popularly used in automotive applications. Based on the aluminum–silicon system, the alloy contains copper as the main alloying element, and Mg is often added in the alloy. Other impurity elements, such as iron and manganese are present in small amounts. 319 alloy has excellent casting characteristics and good mechanical properties.

Control of the cast structure is one of the most fundamental requirements in the foundry industry. The grain size is an important quality characteristic for a sound casting. It is desirable to have small grains with a dendrite morphology that is as globular as possible in order to fill the mold more completely and avoid unfavourable micro- and macroporosity [1].

Grain refinement is one the effective treatment used widely to improve the quality of the castings [2]. A fine-grained structure in a casting ensures uniform mechanical properties, reduces hot-tearing, improves feeding to eliminate shrinkage porosity and distributes the second phases and microporosities [1–4].

The grain refining inoculants commonly used in the aluminum industry are usually masteralloys of Al–Ti or Al–Ti–B [1]. A substrate with low interfacial energy is placed into the melt, either by adding a nucleus or by generating the nucleation takes place on the substrates, and is aided by growth restriction and constitutional factors to produce grain refinement [2].

The control of grain size, prior to casting, is a matter of great interest. Without doubt, optical microscopy is the best technique to evaluate changes induced by grain refining. However, the main drawbacks of this technique are the time required to prepare a sample and the fact that the casting must be destroyed. Chemical analysis to determine the levels of grain refiner in the melt is an attractive method. Grain size depends not only on chemical analysis but also on the cooling rate. Also grain refiner, added in the form of a masteralloy, has been found to have an incubation time of 1–2 h, with the degree of grain refining improving with time. Therefore, composition analysis does not necessarily reflect the changes in grain size [1]. It would, therefore, be advantageous if a rapid on-line melt monitoring technique could be developed to replace metallography as the primary control tool [3–8].

Thermal analysis as a technique is used in the determination of grain size [5,6] and microstructure [7,8]. Graphical changes in cooling curves have been related to macro- and microstructural changes in Al–Si alloys [8].

Conventional thermal analysis is often used in order to assess the degree of grain refinement of Al alloys [3–8]. A molten sample is poured into a cup, and a thermocouple is used to record temperature changes of the sample, as it solidifies. The measured temperature is then plotted versus the time elapsed during solidification, and a cooling curve is obtained. Different parameters may be obtained from the cooling curve, and these can be related to the grain size [8]. A study of the first derivative cooling curve provides information about the cooling rate of the sample, as well as the beginning and end points of reactions which cannot easily be observed on the cooling curve itself [8–10].

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In metal casting industry an improvement of component quality mainly depends on better control over the production parameters. Thus, computer-aided cooling curve thermal analysis (CA-CCTA) of alloys is extensively used for the evaluation of several processing and material parameters. Thermal analysis of alloys can provide information about the composition of the alloy [8], the latent heat of solidification [11,12], the evolution of the fraction solid [8,13], the types of phases that solidify [8,14], and even dendrite coherency [15]. There are also many other uses for thermal analysis, such as, determining dendrite arm spacing [9], degree of modification [16,17] and grain refining [3,5,18] in aluminum alloys, the liquidus and solidus temperatures [8,9], characteristic temperatures related to the eutectic regions and intermetallic phase formation [19,20].

The objectives of the present work are to study the effect of different amount of Al-5Ti-1B grain refiner on the macro and microstructural features and the characteristic parameters of the cooling curve of 319 aluminum alloy. Also, this paper describes the range of temperatures over which the latent heat is given off and the fraction solid can be estimated from cooling curves during solidification. Latent heats and fraction solid are determined for the different amounts of grain refiner for 319 alloy.

## 2. Experimental procedures

### 2.1. Melting and alloying

Commercial 319 aluminum alloy ingots were used in this study. The chemical composition is given in Table 1. Two kilograms of the alloy were melted in an electric resistance furnace for each experiment and maintained at a temperature of  $720 \pm 5^\circ\text{C}$ . The melt was degassed for 5 min using argon inert gas. The melt was grain refined by addition of grain refiner (0.8–10% of Al-5Ti-1B, or 0.04–0.5% Ti). The melt was maintained for 20 min after addition of masteralloy and was stirred at each 5 min to obtain a homogenized melt. After melting, the oxide layer was skimmed from the surface and the molten metal poured into the mold. Three samples were cast, in order to check the reproducibility. Spectrochemical samples were also produced to determine the Ti content.

### 2.2. Thermal analysis

Cooling curve thermal analysis (CCTA) was performed on all samples using high-sensitive K type thermocouples that is protected in a stainless steel sheath, and data were acquired by a high-speed data acquisition system (A/D converter) linked to a notebook computer. The signal was recorded each 0.5 s for all experiments. Thermocouple and mold were mounted on a test stand to avoid any vibration. The thermocouples were located in the center of the mold at a position of 25 mm from the bottom of the mold. In order to obtain reproducible results, the thermocouple was placed exactly at the same position for each experiment. All experiments were performed in a constant condition. Analog-to-digital (A/D) converter used in this work has a sensitive 16-bit converter (resolution of  $1/2^{16}$  or 0.0015%), response time of 0.02 s and a high accuracy detection. Thermal analysis program can simultaneously display the cooling curves, first derivative curves, temperature and time on the monitor of the computer for an instant observation.

The cylindric mold used for the CCTA in this work was made of thin wall (1.5 mm) steel having a diameter of 30 mm and a height of 40 mm. The mold was coated with Mica spray and preheated at  $350^\circ\text{C}$ .

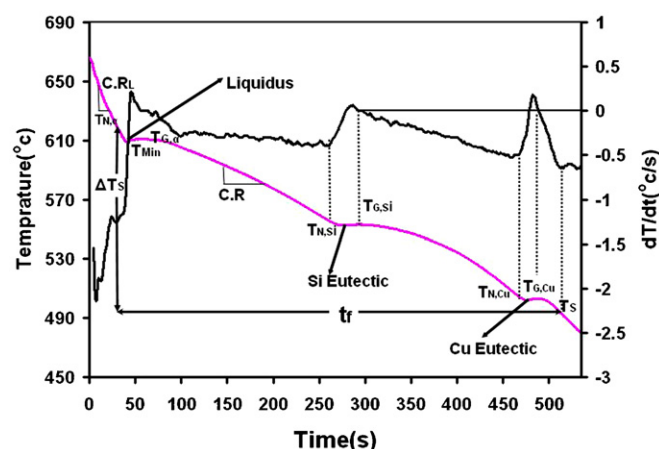
The cooling curve data was processed using a thermal analysis program and Excel software. The processing included smoothing, curve fitting, plotting the first derivatives, identifying the onset and end of solidification, determining solidification parameters such as, cooling rate, nucleation temperatures, nucleation undercooling, recalescence undercooling, solidification range and total solidification time.

There are some differences in defining the thermal analysis parameters in the literature. To avoid this, solidification parameters used in present work are shown in Figs. 1 and 2 and Table 2.

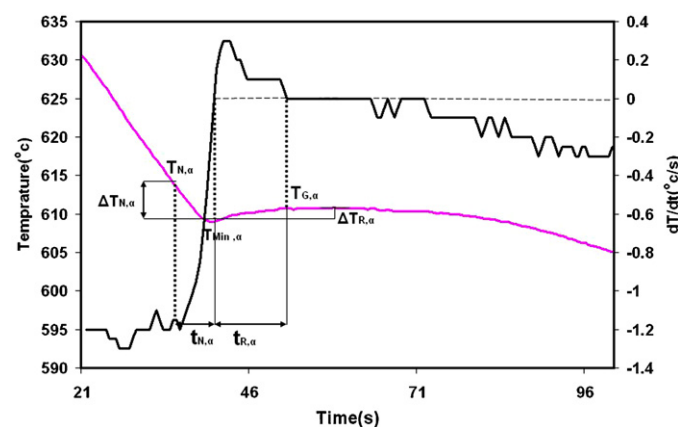
**Table 1**

Chemical composition of 319 aluminum alloy.

| Alloy composition   | Elements |     |      |      |      |      |
|---------------------|----------|-----|------|------|------|------|
|                     | Si       | Cu  | Mg   | Fe   | Mn   | Zn   |
| 319 (AA standard)   | 5.5–6.5  | 3–4 | <0.1 | <0.8 | <0.5 | <1   |
| 319 (actual sample) | 5.7      | 3.5 | 0.1  | 0.18 | 0.24 | 0.01 |



**Fig. 1.** Cooling curve, first derivative curve and representation of characteristic parameters used and analysed in the present study for 319 alloy.



**Fig. 2.** Cooling curve, first derivative curve and representation of characteristic parameters for 319 alloy at the beginning of solidification (liquidus region).

### 2.3. Microstructural analysis

The thermal analysis samples were sectioned horizontally through the place that the tip of the thermocouple was located. Metallographic specimens were prepared via standard grinding and polishing procedures. The final stage of polishing was done using commercial silicon oxide slurry. Optical microscope was used to characterize the microstructure and intermetallic phases. For the microstructural analysis, samples are etched on the same surface using 10% HF-5% CuCl<sub>2</sub> solution, and the average grain size is measured by the line intercept method and image analyser system. The effect of Al-5Ti-1B on the micro and macrostructures was

**Table 2**

Solidification characteristic parameters shown in Figs. 1 and 2.

| Characteristic symbol | Characteristic description  |
|-----------------------|---|
| $T_{N,\alpha}$        | $\alpha$ -Al dendrite nucleation (liquidus) temperature   |
| $T_S$                 | Solidus temperature (end of solidification process)   |
| $\Delta T_S$          | Solidification range ( $\Delta T_S = T_{N,\alpha} - T_S$ )                                      |
| $t_f$                 | Total solidification time   |
| $C.R_L$               | Cooling rate in liquidus region   |
| $C.R$                 | Cooling rate in mushy zone  |
| $T_{Min,\alpha}$      | $\alpha$ -Al dendrite minimum temperature   |
| $\Delta T_{N,\alpha}$ | Nucleation undercooling temperature ( $\Delta T_{N,\alpha} = T_{N,\alpha} - T_{Min,\alpha}$ )   |
| $t_{N,\alpha}$        | Nucleation undercooling time  |
| $T_{G,\alpha}$        | $\alpha$ -Al dendrite growth temperature  |
| $\Delta T_{R,\alpha}$ | Recalescence undercooling temperature ( $\Delta T_{R,\alpha} = T_{G,\alpha} - T_{Min,\alpha}$ ) |
| $t_{R,\alpha}$        | Recalescence undercooling time  |
| $T_{N,Cu}$            | Si eutectic nucleation temperature  |
| $T_{G,Cu}$            | Si eutectic growth temperature  |
| $T_{N,Cu}$            | Cu-rich eutectic nucleation temperature   |
| $T_{G,Cu}$            | Cu-rich eutectic growth temperature   |

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