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Computer-aided cooling curve thermal analysis used to predict the quality of aluminum alloys

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Abstract In this research, the effects of Al–5Ti–1B grain refiner and Al–10Sr modifier were studied on solidification characteristics and microstructural features of 319 aluminum alloy. Important solidification events such as recalescence and nucleation undercooling temperature and aluminum–silicon eutectic depression temperature have been evaluated using cooling curve and its first derivative curve obtained from thermal analysis of a sample. The aim of this article is to show the ability of the thermal analysis technique to predict some key parameters controlling solidification and casting process. It has been found that the thermal analysis is the identified method for a rapid on-line monitoring of metallurgical characteristics of aluminum alloy melts without conventional metallographic examination.

Keywords Solidification · Grain refinement · Silicon modification · Thermal analysis · Quality control testing

Introduction

The grain size and silicon morphology are the important quality characteristics for a sound casting in aluminum–silicon alloys. It is desirable to have small grains with a very fine and fibrous silicon network in order to improve the mechanical properties [1].

The determination of grain refining and modification conditions reliably prior to casting is a matter of great interest. Without doubt, optical microscopy is the best technique to evaluate changes induced by grain refining and modification. 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 and modifier in the melt is an attractive method. Grain size and silicon morphology depend not only on chemical analysis but also on the cooling rate. Also grain refiner and modifier, added in the form of a master alloy, have been found to have an incubation time of 1 to 2 h, with the degree of grain refining and modification improving with time. Therefore, composition analysis does not necessarily reflect the changes in structure [1, 2]. 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].

Thermal analysis as a technique is used to evaluate the melt quality. By this method, some characteristic values are extracted from a cooling curve and/or its derivative, and then a regression relationship is built up between the characteristics and quality indexes as grain size, eutectic structure, silicon morphology, and so on [4–6].

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 [7, 8], the latent heat of solidification [9, 10], the evolution of the fraction solid [7, 11], the types of phases that solidify [7], and even dendrite coherency [12]. There are also many other uses for thermal analysis, such as, determining dendrite arm spacing [13], degree of modification [4, 7, 14–16] and grain

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refining [4, 7] in aluminum alloys, the liquidus and solidus temperatures [7, 13], and characteristic temperatures related to the eutectic regions and intermetallic phase formation [7, 17–22].

The objectives of the present research are to study the effect of different amounts of Al–5Ti–1B grain refiner and Al–10Sr modifier on the macro- and microstructural features and the characteristic parameters of the cooling curve of 319 aluminum alloy.

Experimental procedures

The aluminum alloy used in the experiments was the 319 alloy. The composition of the alloy is shown in Table 1. 2 kg of the alloy was melted in an electric resistance furnace for each experiment and maintained at a temperature of 720 ± 5 °C. The melt was degassed for 5 min using argon inert gas. The melt was grain refined and modified by addition of Al–5Ti–1B (0.8–10 wt% Al–5Ti–1B, or 0.04–0.5 wt% Ti) and Al–10Sr (0.004–0.04 wt% Sr) master alloys, respectively. It should be noted that the grain refining was done without presence of modifier, and modification was also done without presence of grain refiner. The melt was maintained for 20 min after addition of master alloy 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 and Sr contents.

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. 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 study has a sensitive 16-bit converter (resolution of $\frac{1}{2^{16}}$ or 0.0015%), response time of 0.02 s, and a high accuracy

Table 1 Chemical composition of 319 aluminum alloy

Alloy composition/wt%	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

detection. The cylindric mold used for the CCTA in this study 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 and preheated to 300 °C.

The cooling curves and the first derivative curves were plotted using a thermal analysis program and Excel software. Solidification parameters such as, cooling rate, nucleation temperatures, nucleation undercooling, recalescence undercooling, and aluminum–silicon eutectic growth temperature were calculated from cooling curves and the first-derivative curves.

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. Optical microscope was used to characterize the microstructure and the macrostructure. Grain size measurements were carried out using an image analyzer.

Results and discussion

Cooling curve

A typical cooling curve and its first derivative for the 319 aluminum alloy are presented in Fig. 1. The shape of the cooling curve is the result of the heat lost to the surroundings by the cooling metal and the heat evolved in the mold during phase transformation.

The arrows marked on the cooling curve in Fig. 1 show three temperature arrest peaks that represent the various phase formation temperatures in the alloy system. The first peak shows the formation temperature of $\alpha - $ Al dendrite phase (liquidus region), the second peak indicates Al + Si eutectic temperature (Al + Si eutectic region), and the third one shows the formation temperature of the Al_2Cu phase (Cu-rich eutectic region).

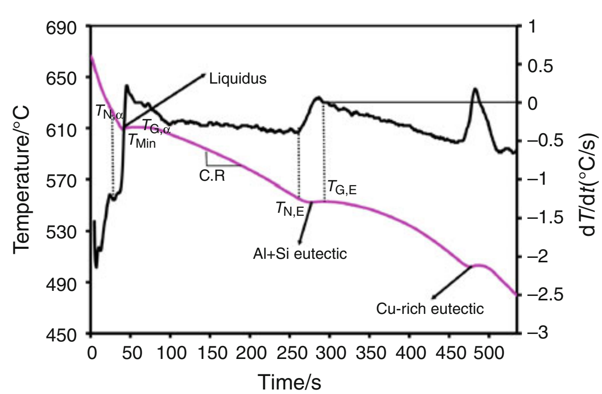

Fig. 1 Cooling curve and its first derivative curve, with major point labeled for the 319 aluminum alloy

At the beginning of solidification of any phase, the derivative increases in value, and decreases at the end of solidification. The main solidification parameters used in present work are shown in Fig. 1. The cooling rate in mushy zone (CR) for all samples is about 0.3 °C/s. Therefore, it can be suggested that the solidification conditions are constant at all experiments of grain refinement and modification.

Grain refining

The result of average grain size measurements is shown in Fig. 2 as a function of the percentage of Al–5Ti–1B master alloy added to the melt. The grain size decreases rapidly with increasing grain refiner into 319 alloy melt up to 3.4 wt% (0.17 wt% Ti). Addition of Ti more than 0.17 wt% does not affect the grain size significantly. Therefore, the optimum level of Ti and/or B additions for the grain refinement of 319 aluminum alloy is about 0.17 wt% Ti (3.4 wt% Al–5Ti–1B).

Grain refinement mostly affects the liquidus region on the cooling curve. Therefore, the changes of liquidus region parameters were evaluated by grain refinement.

In Fig. 3, a comparison of the cooling curves in liquidus region for unrefined and full-grain-refined 319 alloy is illustrated. The disappearance of the sinusoidal temperature undercooling (recalescence undercooling temperature) after grain refinement of the melt is evident. Effect of grain refinement on the recalescence undercooling temperature ($\Delta T_{R,\alpha} = T_{G,\alpha} - T_{Min,\alpha}$) is shown in Fig. 4. As seen, recalescence undercooling temperature decreases with increasing Al–5Ti–1B, and, its amount becomes zero in optimum level of grain refiner, and then it will be constant at higher Al–5Ti–1B levels. Figure 5 shows the effect of grain refinement on the dendrite nucleation undercooling temperature, $\Delta T_{N,\alpha}$. When the Al–5Ti–1B level is increased from 0 to 3.4% (optimum level), the nucleation undercooling temperature decreases from about 7 to 3 °C, and then increases with adding more Al–5Ti–1B grain refiner.

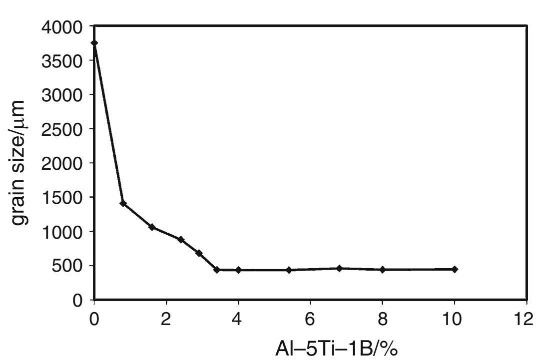

Fig. 2 Grain refining of 319 alloy as a function of Al–5Ti–1B wt% added to the melt

The grain structure in a casting is related to the number of nucleation sites present in the melt at the liquidus temperature. If the number of sites is large, many grains can be nucleated with very little undercooling and a fine-grained structure will result. However, if a few favorable sites are available at the liquidus temperature, significant undercooling can occur.

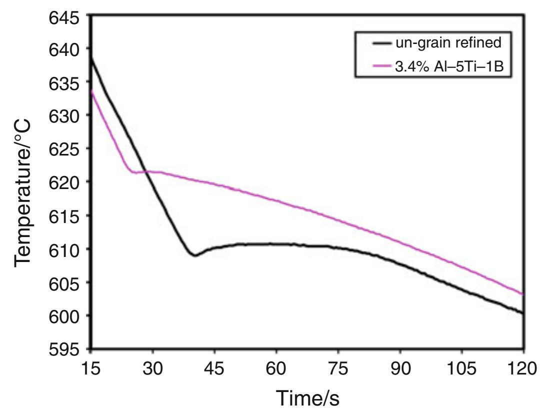

Fig. 3 Effect of grain refinement on liquidus arrest of cooling curve of 319 alloy

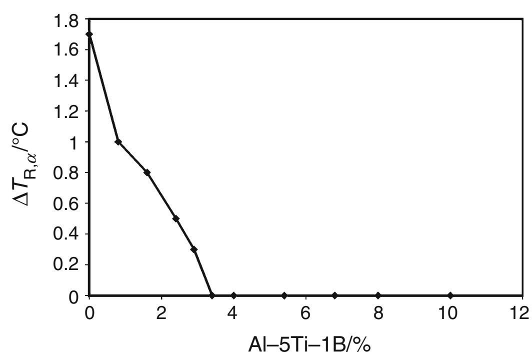

Fig. 4 Effect of grain refinement on the recalescence undercooling temperature

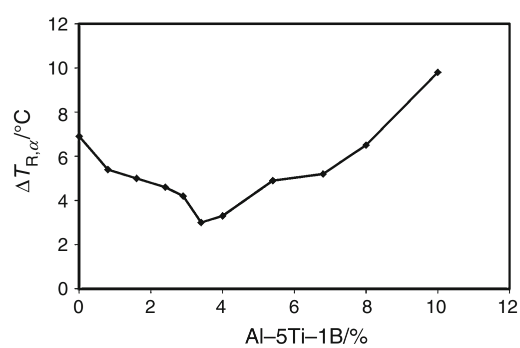

Fig. 5 Effect of grain refinement on the dendrite nucleation undercooling temperature

The change of liquidus parameters in the cooling curve can be used to on-line quality control of grain refinement. Therefore, it will predict the soundness of the casting before pouring the melt.

Modification

Effect of strontium modification on cooling curve of the alloy has been shown in Fig. 6. This figure shows that the addition of strontium depresses the aluminum–silicon eutectic growth temperature ($T_{G,E}$). When the strontium level was increased from 0 to 0.012 wt%, the $T_{G,E}$ temperature decreased from 553 to 547.6 °C. Then it increases and has a constant rate in strontium contents higher than 0.018 wt% Sr. Figure 7 indicates the changes of $T_{G,E}$ versus strontium concentration.

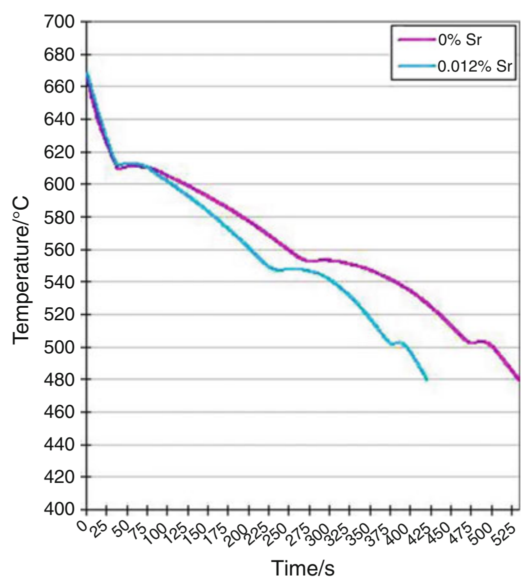

Fig. 6 Thermal analysis cooling curves for unmodified and modified 319 alloy

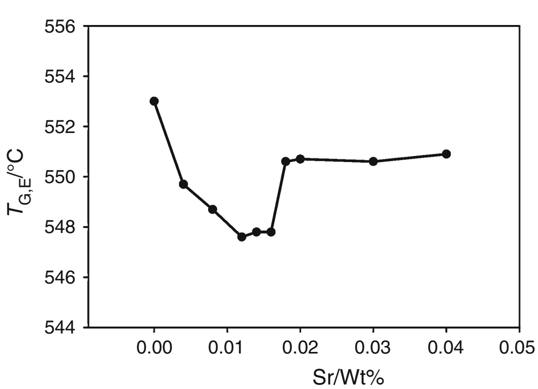

Fig. 7 Changes of eutectic growth temperature ($T_{G,E}$) versus strontium concentration

For silicon modification assessment, the aluminum–silicon eutectic depression temperature ($\Delta T_{G,E}^{Al-Si}$) is used to indicate the modification level [4, 14]. The aluminum–silicon eutectic depression temperature is calculated from the temperature difference between the unmodified and modified aluminum–silicon eutectic growth temperatures according to Eq. 1 [4, 5, 23]:

$$\Delta T_{G,E}^{Al-Si} = T_{G,E,UNMODIFIED}^{Al-Si} - T_{G,E,MODIFIED}^{Al-Si} \tag{1}$$

Changes of $\Delta T_{G,E}^{Al-Si}$ are shown in Fig. 8 as a function of strontium content. $\Delta T_{G,E}^{Al-Si}$ is increased from 0 to 5.4 °C where Sr increased to 0.012 wt%. It has a remarkable reduction in higher strontium content particularly, in more than 0.016 wt% Sr. This trend is constant (about 2 °C) at Sr content higher than 0.018 wt%.

It has been reported that eutectic growth temperature shifts to lower temperatures than the equilibrium curve by adding strontium to the melt [1, 4, 5, 7]. Its effect on modification of microstructure diminishes by adding more strontium to the melt, and the eutectic growth temperature starts to increase. As it is seen from Fig. 8, changes of $\Delta T_{G,E}^{Al-Si}$ are related to the eutectic growth temperature. As $T_{G,E}$ decreases with strontium addition, it increases $\Delta T_{G,E}^{Al-Si}$. There is a reduction in $\Delta T_{G,E}^{Al-Si}$ where adding strontium more than the optimum level of 0.012–0.016 wt% Sr. It may be related to the impingement and coalescence of strontium particles. Recalescence undercooling of eutectic reaction has been determined by the difference between eutectic growth temperature and the minimum temperature in each alloy ($\Delta T_{R,E} = T_{G,E} - T_{min,\ E}$). Effect of strontium on the eutectic recalescence undercooling has been shown in Fig. 9.

In general, two important changes happen in Al–Si eutectic region of the cooling curve during full modification. First, decrease of eutectic growth temperature $T_{G,E}$ or increase of aluminum–silicon eutectic depression temperature ($\Delta T_{G,E}^{Al-Si}$) comparing with unmodified state. Second,

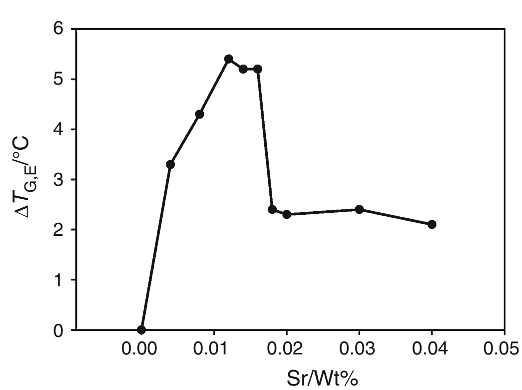

Fig. 8 Changes of $\Delta T_{G,E}^{Al-Si}$as a function of strontium content

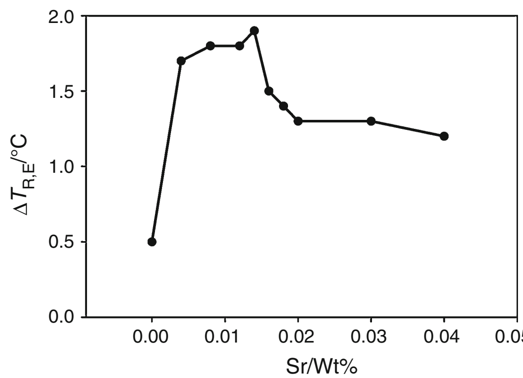

Fig. 9 Effect of strontium on the eutectic recalescence undercooling

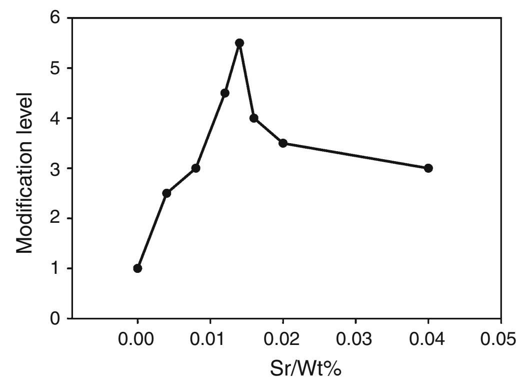

Fig. 11 Silicon modification level as a function of strontium content

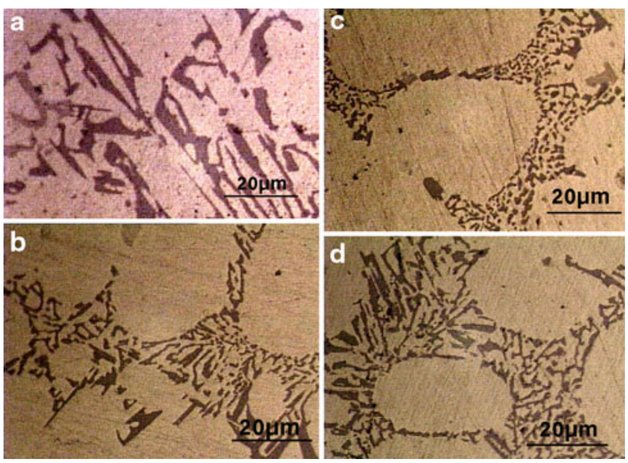

Fig. 10 Aluminum–silicon eutectic morphology of 319 alloy as a function of strontium level. **a** Unmodified microstructure (without Sr); **b** partially modified microstructure (0.008 wt% Sr); **c** modified microstructure (0.014 wt% Sr); and **d** coarse microstructure (0.04 wt% Sr)

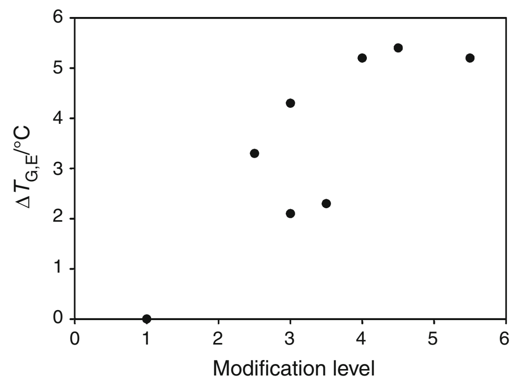

Fig. 12 The relation of modification level with $\Delta T_{G,E}^{Al-Si}$

increase in recalescence undercooling $\Delta T_{R,E}$ during full modification. The optimum level of strontium addition to achieve full modification can be obtained from these changes in cooling curve in on-line process control.

Microstructural changes that occur with increasing strontium content are shown in Fig. 10a–d. These micrographs depict the development of a more fibrous aluminum–silicon eutectic structure as strontium content increases to optimum level. Figure 10a–c shows the aluminum–silicon eutectic morphology has been changed from coarse acicular plates to a fine fibrous morphology with increasing the Sr content of the alloy from 0 to 0.014 wt%. Then, coarsening of silicon particles occur at high strontium content (Fig. 10d). The data obtained from the thermal analysis presented in Fig. 6, as well as the representative micrographs presented in Fig. 10, show that the addition of strontium decreases the aluminum–silicon eutectic growth temperature ($T_{G,E}$) and changes the degree of silicon modification.

Figure 11 shows the relation of modification level (ML) obtained according to AFS Standard Charts versus the Sr content. It is evident that the modification level increases with the Sr addition, and then reaches a maximum modification level of 5.5 when the Sr content is 0.014 wt% (optimum level). The modification level of eutectic silicon decreases with the further increase of Sr content. However, this kind of relationship cannot be utilized for the on-line prediction of ML. Before this can be accomplished, it is necessary to correlate the ML to one of the TA characteristics, so that the TA system can be utilized as a tool for the on-line measurement. In fact, a strong correlation exists between aluminum–silicon eutectic depression temperature ($\Delta T_{G,E}^{Al-Si}$) and ML because both are correlated with the strontium level (see Fig. 12). Figure 12 shows that the greater the level of modification is accompanied the higher the $\Delta T_{G,E}^{Al-Si}$.

Conclusions

This study was carried out to establish quantitative on-line method for controlling the level of grain refining and silicon modification in the 319 aluminum alloy. The on-line prediction of grain refinement and modification are important for controlling casting microstructures, and hence, mechanical properties. A technique has been developed which is based on the computer-aided cooling curve thermal analysis instead of laborious metallographic techniques to monitor melt quality. The changes of recalescence and nucleation undercooling temperatures, $\Delta T_{R,\alpha}$ and $\Delta T_{N,\alpha}$, in the liquidus region of cooling curve can be used to on-line quality control of grain refinement. Silicon modification due to the addition of strontium was found to be correlated with the depression of the aluminum–silicon eutectic growth temperature, $\Delta T_{G,E}^{Al-Si}$, that is controlled by the strontium content. Therefore, the TA measurement of $\Delta T_{G,E}^{Al-Si}$ can be used for on-line prediction aluminum–silicon eutectic ML.

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