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ASSESSMENT OF MODIFICATION LEVEL IN EN AC-46000 ALUMINUM CASTING ALLOYS USING THERMAL ANALYSIS AND MICROSCOPIC EVALUATION

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Abstract

The quantitative methods for controlling and predicting the level of Si modification in EN AC-46000 aluminum cast alloys were examined using thermocouples (thermal analysis) and optical microscopy (image analysis). A wide range of Sr, from 35 to 500 ppm, was added to the alloy. The alloys were cast using three different molds providing different cooling rate and consequently varied microstructure coarseness. Large difference in nucleation and growth temperature of unmodified and modified alloy was found irrespective of coarseness of microstructure. The depression in growth temperature of eutectic Si found to be strongly correlated to content of modification agent as well as modification level. Thermal analysis technique was realized as a non-biased, accurate and inexpensive approach for on-line prediction of Si modification level in the EN AC-46000 alloy cast under different cooling rate.

Introduction

Al-Si-Cu-Mg alloys are light weight alloys having high specific strength, high fatigue resistivity, good machinability and excellent castability which make them popular for different industries especially automotive industry [1, 2]. It is well-known that the mechanical properties of these alloys are primarily governed by secondary dendrite arm spacing (SDAS) as well as size, morphology and distribution of secondary phases (e.g. eutectic Si particles and other intermetallic phases) [3]. Increasing cooling rate during casting process results in refining the coarseness of all microstructure features including Si particles. However the polyhedral geometry of Si particles remains unaffected [4]. On top of cooling rate modification, addition of chemical modifier (like Sr, Na, Sb and etc.) changes the morphology of Si particles from coarse plate-like to fine fibrous. Sr is the most widely used chemical modifier among the others since it has high modification rate and long fading time. However excess amount of Sr leads to overmodification and generation of deleterious phases such as Al_2Si_2Sr, and hence degrades mechanical properties of the alloy [5]. Therefore it is essential for the sake of quality assessment of the castings to control the Sr content and modification level (ML). Assessment of ML has traditionally been done through destructive sampling and microscopic analysis of the microstructure. The given microstructure is qualitatively compared with the American Foundry Society (AFS) chart for microstructure control in hypoeutectic alloys. The AFS charts contain six different classes; from unmodified (class 1) to fully modified (class 6). Based on visual comparison given specimen is located where the microstructures and AFS chart are similar [6, 7]. This approaches have been applied by the author in another study [4], in order to assess the level of modification in an Al-Si cast alloy. However it was found that it is inaccurate and subjective approaches. Shabestari et al. [8] assessed effect of modification level on formation and undercooling temperature of different phases in 319 Al alloy cast in permanent mold. Djurdjevic [9] and MacKay [10] assessed modification level of 319 Al cast alloy through cooling curve analysis. The principle for determining the Si modification level is measuring the suppression of Al-Si eutectic growth temperature in modified structure compared to unmodified one. Francis et al. [6] introduced a model based on neural network using cooling curves experimental data in order to predict modification level in W319 Al alloy without using data of unmodified structure. In previous works thermal analysis has been performed using single solidification condition applying permanent TA cup (mold). However, varied thicknesses in the cast components as well as different types of casting process (e.g. high pressure die casting, permanent mold casting, sand casting and etc) lead to obtain diversified solidification rates and accordingly varied coarseness of microstructure. Therefore it seems an essence to find a reliable and precise approach in order to control the modification level and predict the optimum level which is valid for different solidification conditions and applicable for other Al-Si based casting alloy. In this paper Si modification level in EN AC-46000 alloy at various cooling conditions is assessed through microstructural evaluation and thermal analysis. Eventually an accurate approach for on-line prediction of optimum modification level in aluminum alloys, irrespective of solidification condition, was presented.

Experimental Procedure

Material preparation

Six kilograms of EN AC-46000 Al-alloy ingots were melted in a 8-kW resistance furnace with a silicon carbide crucible at 750°C. Then melts were modified with Sr using Al-10%Sr master alloy. In order to create different percentage of Strontium in the alloy, Al-10%Sr master alloy was cut into small pieces, wrapped in an Aluminum foil and preheated to 250°C prior to being added to the melt. The addition was carried out by placing Al-10%Sr master alloy particle on the melt surface and twenty minutes was considered for dissolution of strontium. Chemical composition of each sample was obtained by optical emission spectroscopy, and the data is summarized in Table 1.

Table 1 Chemical composition of the alloys (wt.% except for Sr, ppm)

	Si	Cu	Fe	Mg	Zn	Mn	Sr	Al
Alloy 1	8.02	2.11	0.78	0.26	0.99	0.17	**0**	Bal.
Alloy 2	8.02	1.89	0.92	0.26	1.03	0.21	**37**	Bal.
Alloy 3	8.05	1.90	0.91	0.26	1.05	0.21	**68**	Bal.
Alloy 4	8.10	2.02	0.90	0.25	0.96	0.24	**150**	Bal.
Alloy 5	8.30	1.99	0.98	0.27	1.06	0.21	**276**	Bal.
Alloy 6	8.21	1.95	0.98	0.27	1.07	0.21	**486**	Bal.

Thermal analysis

For each of indicated alloys, differential thermal analysis (DTA) tests were performed. Three cylindrical steel molds with same geometry (Figure 1) encased and sealed in a steel box, ceramic box and a fiberglass insulation box respectively in order to apply three different cooling rates. The prepared melt poured into the molds which was preheated to 700°C and the temperature was read by K-type thermocouples (Ni-Cr-Ni) located at two positions within the mold, mold wall and mold center, both at mid-height. The schematic view and dimensions of sealed mold has been demonstrated in Figure 1.

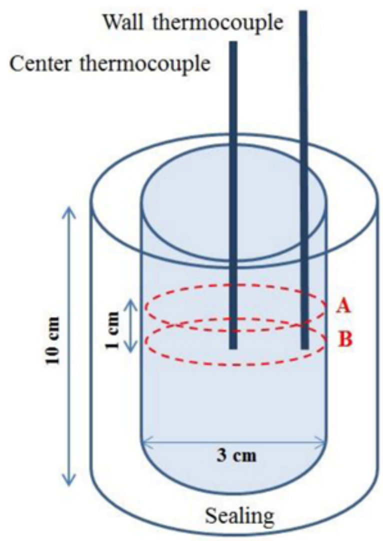

Figure 1. The schematic view of sealed cylindrical mold

The temperature-time data was collected via a high-speed data acquisition system logged on a computer through commercials interface which recorded temperature every 0.01 second. In order to allow a good comparison of results for each alloy, the same thermocouples were used for all tests on the given alloys. This was achieved by placing the thermocouples inside in a 1 mm diameter stainless steel sheath which allows removing the thermocouple from solidified samples.

Microstructural evaluation

Metallographic study was carried out by cutting samples from 2 different cross-sectional positions; position that the tip of thermocouple was located, 10mm upper than indicated position (see A and B in Figure 1). The cross sections of the cut specimens were ground and polished using standard metallographic procedures [11]. In order to evaluate qualitatively and quantitatively microstructural evolution, Olympus GX71 optical microscopy and Stream Motion image analyzer was employed. 12 optical microscopy images of each conditions from identical positions of specimens were probed.

Results and Discussion

Micrograph Analysis

The microstructural features (size and schematics) of the alloy cast under different cooling conditions and containing various Sr levels are depicted in Figure 2-5. The microstructure coarseness was measured through SDAS, which is a function of cooling rate. The measured SDAS for different solidification system are summarized in Table 2.

Table 2 Corresponding SDAS for different cooling systems

Mold type	Steel	Ceramics	Insulated mold
Cooling rate (°C/s)	3.8	1.4	0.4
SDAS	20 ± 4	49 ± 7	91 ± 6

It is clearly demonstrated that the fast cooling rate resulted in smaller SDAS and finer eutectic Si particles. However, no significant change in flaky morphology of Si was observed upon increased cooling rate. On the other hand, the optical micrographs clearly illustrate the tendency toward formation of fine fibrous eutectic Si particles upon initial addition of Sr and increasing level of Sr. It seems that the trend is quite similar for all coarseness of microstructure. Quantitative assessment of Si modification was done through analysis of 12 optical micrographs from two different cross section of each sample using image analysis system. Aspect ratio (AR), area and perimeter of eutectic silicon particles were measured and mean value of them as a function of Sr level are depicted in Figure 2-4.

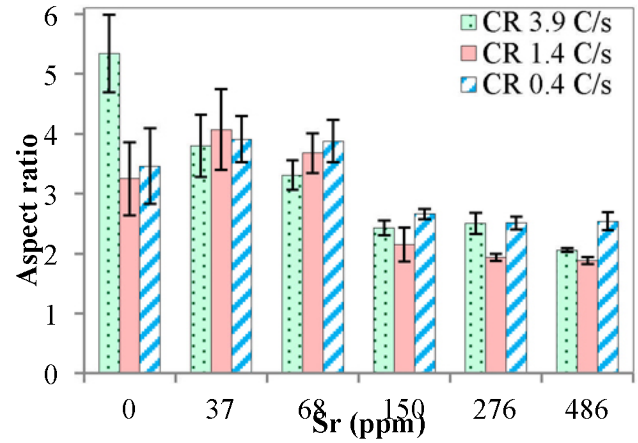

Figure 2. AR as of function of Sr content in the alloy

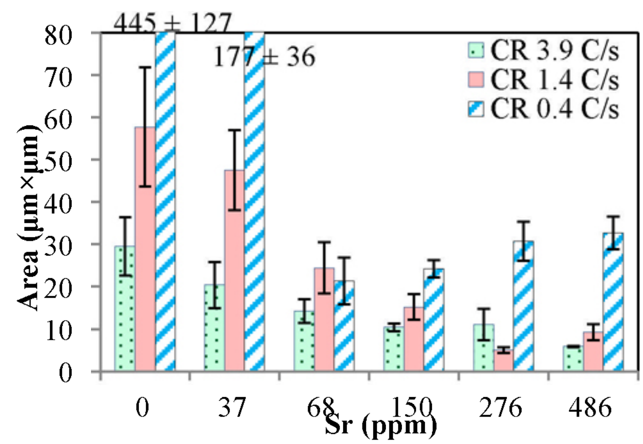

Figure 3. Area as function of Sr content in the alloy

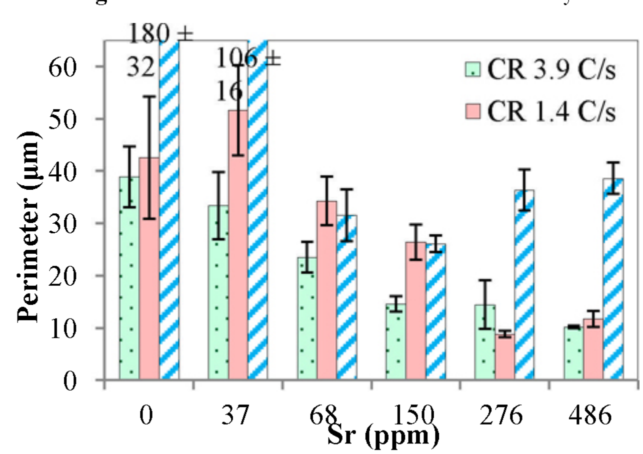

Figure 4. Perimeter as function of Sr content in the alloy

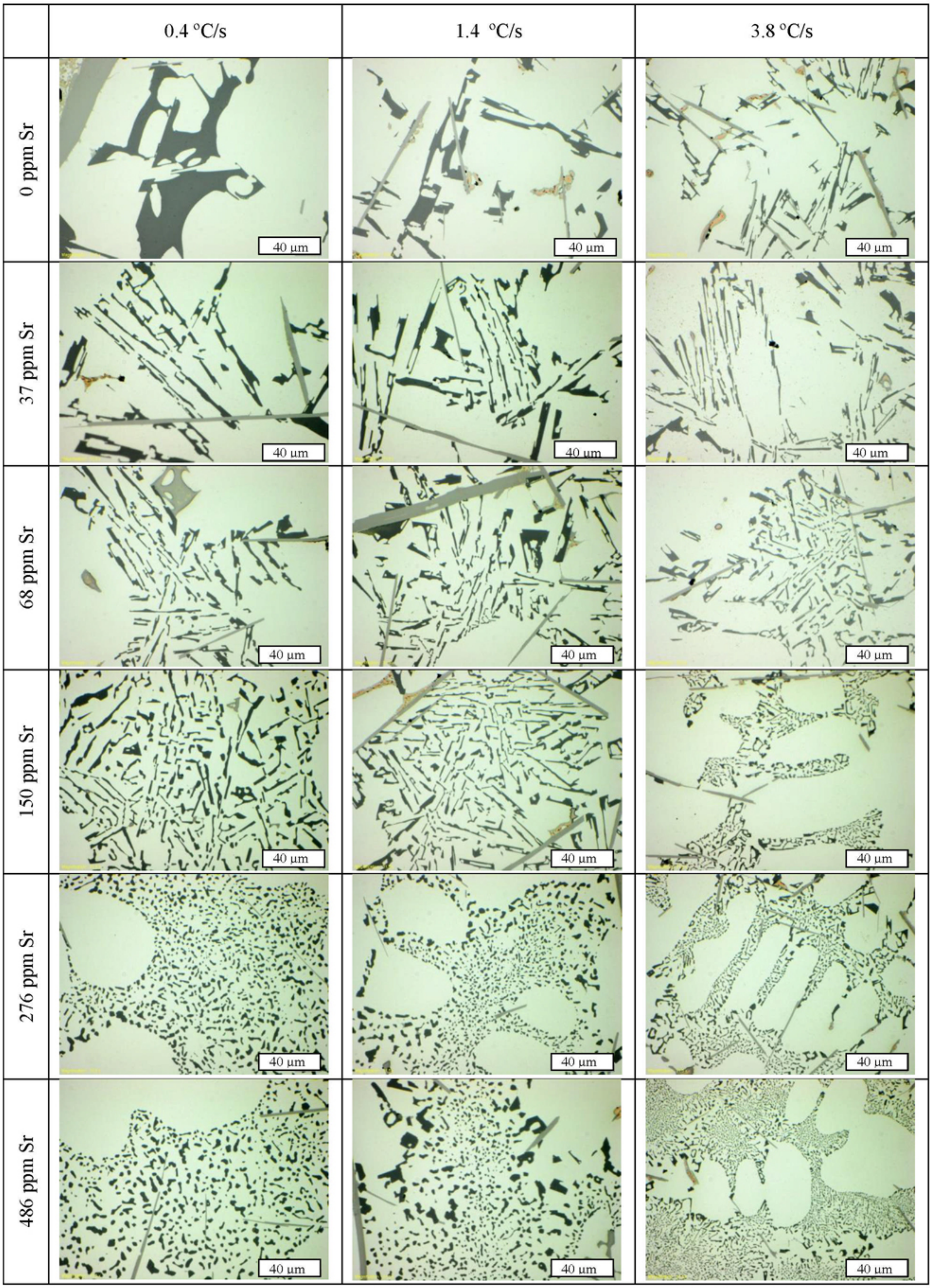

Figure 5. Micrographs showing Si structure cast under different cooling conditions and contain various Sr levels

The evolution of Si structure from coarse plate-like towards fine fibrous as a function of Sr content are depicted in Figure 5. As a result, AR, area and perimeter of particles are expected to be decreased as Sr level increases. The results based on aspect ratio of Si particles showed negligible changes with changing Sr level from 0 to 486 ppm. It may be due to essence of microscopic analysis which is two dimensional. Hence this parameter has proven to have poor validity in expressing level of modification. This was also pointed out by Djurdjevic [9]. Compared to AFS charts on the relationship between Si morphology and ML [4], the current measurements on area and perimeter yielded similar trends which are an overall reduction of Si area and perimeter as a function of Sr content. Djurdjevic et al [9] has done an identical assessment for the 319 aluminum alloy cast in a graphite mold. The perimeter of eutectic Si particles was introduced as the most promising index to describe Si modification level through metallographic analysis which is in agreement with the present investigation. However the results of this study show that the trend is not consistent for slow cooling rates. The decreasing trend in area and perimeter is accompanied with a slight increase when the Sr content exceeds 150 ppm. This behavior could be due the presence of coarse particles which enlarged the mean values of area and perimeter. Increasing the population of counted particles (e.g. multiple surfaces) may yield a more unified trend for all cooling conditions. The solution however requires more operational time and at same time increases the subjectivity of the results.

Once the cooling rate increases, the gap between value of the parameters for each level of Sr decreases and thus the trend seems to become constant. The behavior was observed for both perimeter and area results, see Figure 3 and 4. In addition, the values of those parameters scattered significantly, especially in unmodified structures. For instance, the area varied from 100 to 2500 μm^2 and perimeter from 50 to 600 μm for the unmodified structure with cooling rate of 0.4 ℃/s. Thus, area and perimeter of adjacent Sr levels applying 95% confidence interval were overlapped in higher cooling rates (3.9 ℃/s), while it was conceived that the structure visually changed significantly upon addition of Sr, see Figure 5. Thus, the mean value is assumed not to be a promising measure to report the parameters and relate them to modification level especially where the cooling rate is decreased. In a similar work [9] median value of perimeter of Si particles was used in order to predict modification level of structure. However, virtually median value is assumed as an imprecise measurement.

The scattered values of perimeters are clearly depicted in Figure 6 which is confirming inaccuracy of using means value measurement. Accumulation of the smallest perimeter range (< 10 μm) shows the portion of structure which is fully modified. The general trend is ascending over the addition of Sr. A similar trend was observed for the 0.4 and 1.4 ℃/s. It also shows distinguished gap between each adjacent Sr level. Despite of an increase in portion of refined particles, the coarser still remained unaffected upon addition of 37 and 278 ppm Sr. This also confirms that the presence of remained coarse particles in selected regions may significantly affect the trend. Accordingly, the perimeter and area frequency of fully refined structure in histogram graphs appeared to be the best index to be employed to express modification status of the structure. However, the descriptor still excludes several particles and also unable to offer the optimum Sr level for the alloy. All in all, characterizing the modification level through micrographs analysis was realized to be subjective and time consuming method. Hence it is not an appropriate approach to be employed in foundries in order to predict the optimum modification level in the Al-Si alloys.

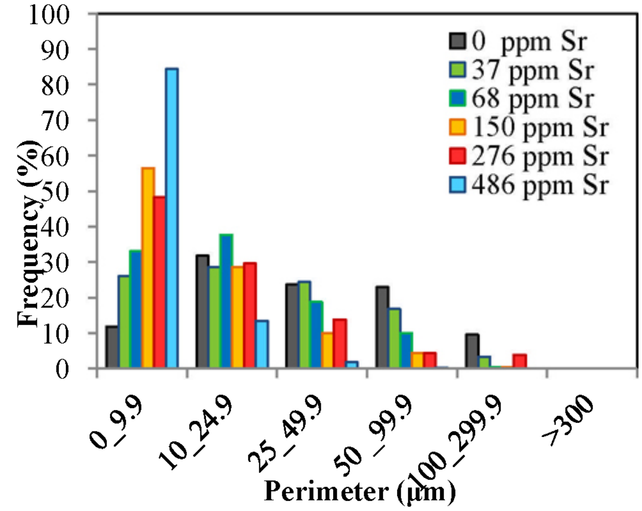

Figure 6. Histograms graph for the perimeter of at 3.8 ℃/s cooling rate.

Thermal Analysis

Figure 7 shows cooling curves of the alloy in unmodified condition solidified in different molds. The alloy exhibits three main reactions during the solidification processes as pointed out in the figure, see arrows in figure 7. The solidification starts with formation of α-Al dendritic networks at temperature range of 620-580℃ followed by the development of eutectic phases; Al and Si at temperature range 0f 580-555℃. Eventually other intermetallic compounds are formed below 550 ℃ [12-14]. In order to evaluate the modification level through cooling curves, nucleation and growth temperatures of Al-Si eutectic are important parameters.

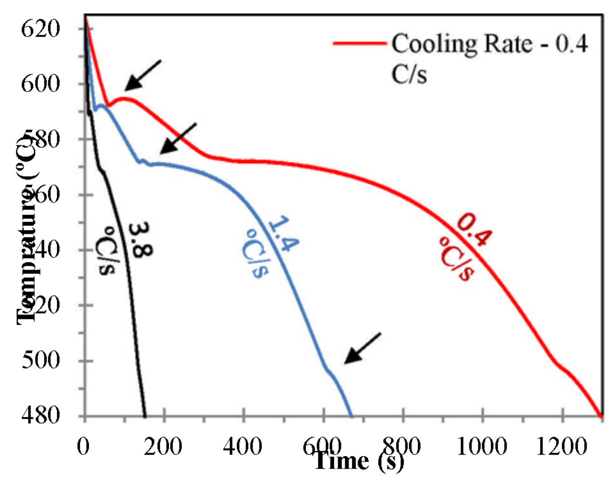

Figure 7. Cooling curves of the unmodified alloy (0 ppm Sr) at different cooling rates.

Generally two notable changes occur in the eutectic regions by the addition of Sr; 1. a decrease in eutectic growth temperature (T_G) which means increased undercooling of eutectic growth and 2. an increase in recalescence undercooling of eutectic region [8]. Figure 8 indicates that minimum eutectic growth temperature was decreased with addition of Sr, up to a certain level and then increased once the Sr content increased. It is well known that, Sr restricts the growth of Si acting as a impurity atoms which poisons the growing layers [15]. Blocking the growth of Si increase nuclei numbers and as a result reduces the nucleation and then growth temperature of Si [8], Figure 8.

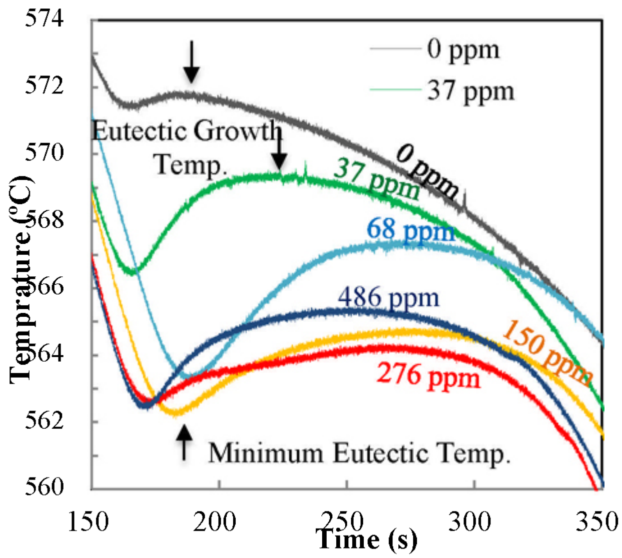

Figure 8. Cooling curves of the alloy at eutectic region for different level of Sr. Cooling rate is 1.4 ºC/s.

However, once Sr content exceeds a particular content, the excess amount forms deleterious Al_2Si_2Sr intermetallic phase which also degrades the modification effect [5]. Hence, looking at thermal curves of the Al-Si alloys during solidification, the maximum eutectic growth temperature shifts to a lower value upon addition of Sr and this effect starts to diminish as the Sr content exceed than a particular level [8, 13, 16]. Figure 8 shows that increasing the Sr concentration in the alloy from 280 to 486 ppm leads to an increase in eutectic growth temperature. The trend is quite identical for all cooling conditions. It is suggesting that the excess amount of Sr does not additionally contribute to the modification of the Si particles and will exclusively form deleterious intermetallic compounds. Shabestrai et al. [8] studied effect of Sr content on eutectic nucleation and growth temperature of a Al5Si3CuMg cast alloy. It was pointed out that addition of 160 ppm, decreases the minimum eutectic temperature up to 6ºC compare to unmodified one. While 180 ppm Sr and on (to 800 ppm) resulted in an almost constant 3ºC decrease in minimum eutectic temperature. This is in agreement with finding of this study, and suggesting that lower Si containing alloy demands less Sr to obtain the fully modified structure. One have also to put in mind that the optimum Sr content, yielding the highest modification level without formation of Al_2Si_2Sr phase, is a function of Si level in the alloy.

Therefore, the depression amount of eutectic growth temperature from unmodified to modified structure ($\Delta T = T_{G/unmodified} - T_{G/modified}$) seems to be a suitable parameter expressing the modification status in Al-Si alloy. Figure 9 shows ΔT as function of Sr level for different cooling rates. Djurdjevic et al. [9] used ΔT in order to correlate modification level to Sr content in W319 Al cast alloy through a curve fitting equation. Francis et al. [6] used thermal analysis parameters as inputs to develop neural networks models which enables prediction of Si modification level in a W319 Al mold-cast alloy.

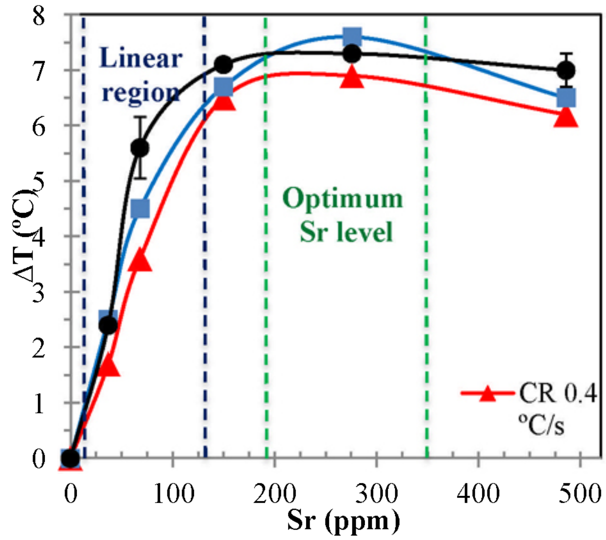

Figure 9. Changes in eutectic growth temperature as a function Sr content at different cooling rates.

The trends in figure 9 are initially ascending till reaching a maximum value, followed by a slight reduction. In the linear region (pointed out in Figure 9), the relation between depression in eutectic growth and Sr content is linear with rough estimation. The curve fitting equations of these regions are a promising mean for online prediction of modification level of castings [9]. However it seems that the optimum Sr content is where ΔT obtains its highest value irrespective of cooling condition. Prior to reaching the peak point, the Sr content is not enough to modify all Si particles, Figure 9. While after the peak point, Sr tends to form intermetallic compounds and its modification role diminishes. Therefore it is believed that the indicated region in Figure 9 (Sr level where maximum ΔT is obtained, plus minus a few tens of ppm) is the optimum range of Sr levels offering excellent modification. Since the online prediction approach is through recording of thermal history of the melt, the acquired parameter (for example ΔT) is unique value based on activity of Sr in the melt and represents the whole batch. However, results of image analysis technique is based on evaluation of 2D microscopy images of few hundreds Si particles. It was already pointed out in another work [4] that, surprisingly the most appreciate improvement in tensile properties (in particular elongation to fracture) of the alloy was achieved by addition of 276 ppm Sr [5]. Figure 9 shows that the thermal history of alloys at different cooling rates are quite similar suggesting that online prediction of optimum Si level in the alloy is independent of casting condition. It means that the size and type of the mold do not play any role in the experimental set-up which makes this method proper for the foundries. Thus, it is believed that employing thermal analysis for the online assessment of optimum level of Sr in the Al-Si cast alloys is inexpensive, accurate and objective approach.

Conclusion

The present work was carried out in order to assess the level of Sr modification in EN AC-46000 aluminum casting alloy through evaluation of as cast micrographs and cooling curves. The following conclusions were pointed out :

1. Frequency of refined Si particles (expressed as by their area and perimeter) are found to be the best descriptors of assessing modification status in the alloy.
2. The microscopic analysis approach was realized as operator-biased and subjective method.
3. Optimum level of Sr which offers the best modified structure was found to be strongly correlated with the depression of the eutectic growth temperature.
4. Thermal analysis technique was realized to be a reliable, fast and inexpensive approach in order to predict online the optimum Sr content which required to modify Al-Si casting alloys.

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