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Determination of dendrite coherency point characteristics using first derivative curve versus temperature

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Abstract The dendrite coherency point (DCP) temperature refers to the state of a solidifying alloy at which a coherent dendrite network is established during the formation of grains. Several relatively complex methods for detection of the DCP temperature have been developed. There are four main DCP temperature testing approaches: (i) the rheological technique, (ii) thermal analysis of the minimum temperature difference between two cooling curves, (iii) thermal analysis of the second derivative of one cooling curve, and (iv) the thermal diffusivity measurement technique. This paper follows up the proposed thermal analysis of one center cooling curve for the determination of the DCP characteristics such as: temperature, time, instantaneous solidification rate, and fraction solid. The first derivative of the cooling curve is plotted versus the temperature and time and the thermal characteristics of all metallurgical reactions, including the DCP are determined with the same accuracy achieved using the two thermocouple technique developed by Bäckerud et al. [4, 5]. Statistical analysis of the DCP temperature using the one versus two thermocouple techniques shows R^2 equal to 0.99. This research revealed that utilization of dT/dt versus the temperature curve methodology also allows for analysis of the α-Al dendrite nucleation and growth characteristics and consequent determination of the grain size. On-going work on this new methodology for characterization of other solidification events will be presented in subsequent papers.

Keywords Aluminum alloys · Cooling curve · Dendrite coherency point

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Introduction

A comprehensive understanding of ingot, melt quality, and casting process parameter expressed by various thermal characteristics is of paramount importance for the control and prediction of actual cast component properties. If process engineers can monitor the ingot, melt, and casting quality control online, then downtime costs and scrap levels can be reduced, thus resulting in better cast component engineering characteristics. The application of thermal analysis (TA) to study the evolution of the sample's microstructure was reported in earlier publications by Cibula [1] and Mondolfo [2]. TA can assist in the proactive on-line decision making process and therefore, has an important advantage over post-process testing and analytical methodologies which are destructive in nature. TA uses several different laboratory measurements and plant techniques such as differential thermal analysis (DTA), differential scanning calorimetry (DSC), and computer aided cooling curve (CACC) analysis. CACC analysis has been used for many years to construct binary phase diagrams and for fundamental metallurgical studies. Binary alloys of varying compositions were studied as they cooled and the arrest points were recorded and plotted on temperature–composition equilibrium (phase) diagrams.

The non-equilibrium cooling curve method is useful for commercial applications for a number of reasons: it is simple, inexpensive, and provides consistent rapid results for all important metallurgical events which contribute to the as-cast structure. This technique is a good choice for establishing functional engineering relationships between cooling curve characteristics (being used as common denominators) and ingot and melt quality, casting process parameters and resultant as-cast component metallurgical characteristics. A state-of-the-art thermal analysis platform is able to quantify metallurgical characteristics including: grain size (GS), dendrite arm spacing (DAS), and DCP, the level of silicon modification, the liquidus temperature, solidus temperature, nucleation and solidification temperature of the secondary and tertiary eutectics, as well as fraction solid and latent heat for a wide range of melt treatments and solidification conditions.

Cooling curve and its first derivative curve plotted versus temperature

Introduction of the new methodology for characterization of the DCP and GS based on the single center (T_C) cooling curve analysis technique requires a brief explanation of the fundamentals of the solidification process and its associated definitions. Definitions of the selected characteristic points associated with the individual non-equilibrium metallurgical events taking place during the solidification process are briefly described and presented in the form of specific formulas and are illustrated in the following figures.

The solidification process of a metal or alloy is accompanied by the evolution of latent heat. Recorded temperature–time data can yield quantitative information about the alloy's solidification process characteristics. A typical cooling curve obtained using a center thermocouple (T_C) and its first and second derivatives for an AlSi9Cu4 alloy is presented in Fig. 1. As can be observed, the test alloy does not start to solidify immediately at the equilibrium solidification temperature because no effective nuclei are present. Some undercooling is needed to supply the driving force for nucleation and growth of the aluminum dendrites. Evolving latent heat causes the temperature of the surrounding melt to rise. Dendrites continue to grow with further melt cooling.

During the non-equilibrium solidification of the test samples, some thermally weak events cannot easily be detected on the cooling curve alone. Therefore, first and higher level cooling curve derivatives are utilized. Previous work [3] shows that the complementary information about the solidification process including fraction solid and latent heat evolution can be readily obtained by calculation of the base line of the first derivative of the cooling curve that is

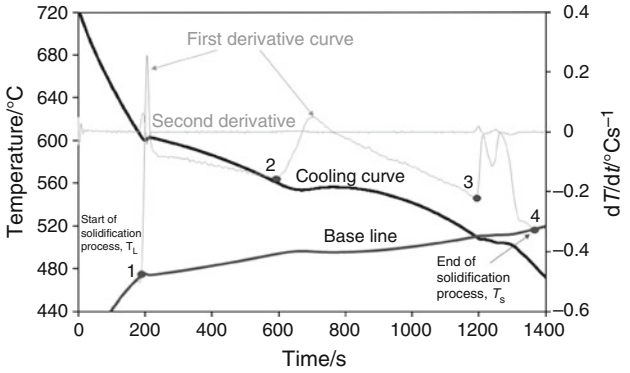

Fig. 1 328 alloys (AlSi9Cu4 in Table 1) cooling curve obtained from the center thermocouple (T_C) plotted as a time function. The first and second derivatives and the base line were calculated according to Kierkus and Sokolowski [3]. The *arrows* point out the major solidification events

plotted against the temperature instead of the time as the present predominant practice. The first derivative curve (d*T*/d*t* vs. time) in Fig. 1 intersects the "base line" and this signifies the beginning (event #1—liquidus temperature) and the end (event #4—solidus temperature) of the solidification process. The d*T*/d*t* curve also clearly identifies other characteristic temperatures like the nucleation of the main AlSi eutectic reaction (event #2) and the AlFeSiCuMg eutectics (event #3—convoluted peaks) that are present in the AlSiCu alloy. The first derivative represents the rate of cooling (solidification) of the test sample.

In order to illustrate additional vital information available from the first derivative versus the temperature, Fig. 2 shows the superimposed first derivative plotted versus both the temperature and time. As can be observed the plot of the d*T*/d*t* versus temperature shows two loops in the area of the primary α-Al dendrite nucleation and growth (event # 1) and one loop in the AlSi eutectic nucleation and growth (event # 2), while event # 3 represents the AlFeSiCuMg eutectics' similar to the convoluted peaks that are present

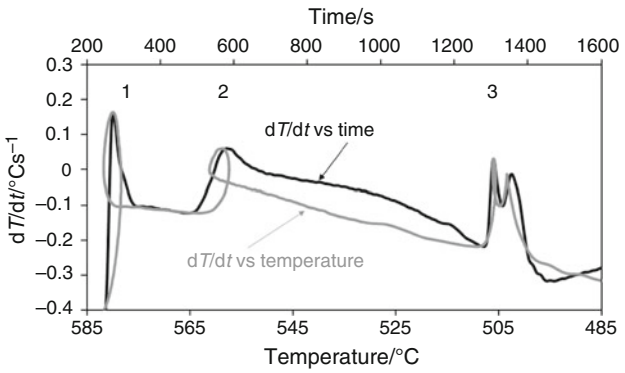

Fig. 2 Superimposed d*T*/d*t* versus time and temperature curves represent the T_C thermocouple data that corresponds to the cooling curve in Fig. 1. Data generated by the authors

for both the derivative versus time and temperature. Close examination (by the authors) of the loop (event # 1) associated with the formation of the dendrite structure revealed the possibility for determination of the α-Al dendrite minimum and growth temperatures and that the maximum width of the loop represents the respective undercooling that can be used for GS characterization. Using a similar methodology, (event # 2) characterization of the AlSi eutectic (i.e., Si modification level) will be addressed in a future paper. To date this analytical approach was not reported in the available literature.

The shape of the cooling curve and its first derivative at event #1 of the solidification process gives a good indication of the number of nuclei present in the melt, Fig. 3a, b. When there are a great number of nuclei, the shape of the cooling curve exhibits little under cooling. When there are a few nuclei, there exhibits more under cooling.

Definitions of the grain solidification process events, acronyms, and relationships

The α-Al dendrite nucleation temperature, ($T_{NUC}^{\alpha DEN}$)–non-equilibrium liquidus temperature

The $T_{\mathrm{NUC}}^{\alpha\mathrm{DEN}}$ signifies the start of the solidification process of the primary stable dendrites from the melt. This results in the change of the slope of the cooling curve and is determined by the first derivative inflection point. The equilibrium liquidus temperature is higher in comparison with the non-equilibrium temperature. At the liquidus temperature, the fraction solid is equal to zero.

The α-Al dendrite minimum temperature, ($T_{MIN}^{\alpha DEN}$)

At $T_{\mathrm{MIN}}^{\alpha\mathrm{DEN}}$ the nucleated dendrites have grown to such an extent that the liberated latent heat of fusion balances the heat extracted from the test sample. After passing this point, the melt temperature increases to a steady state growth temperature ($T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$). The $T_{\mathrm{MIN}}^{\alpha\mathrm{DEN}}$ as the local minimum, is determined by the point at which the first derivative intersects the zero line ($\mathrm{d}T/\mathrm{d}t = 0$). The time period required for heating up of the test sample to the $T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$ is called recalescence.

The α-Al dendrite growth temperature, ($T_G^{\alpha DEN}$)

The $T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$ represents the local maximum temperature. The $T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$ corresponds to the second zero point on the first derivative curve ($\mathrm{d}T/\mathrm{d}t = 0$) following the start of nucleation. In the case that the $\mathrm{d}T/\mathrm{d}t$ curve does not intersect the zero line, $T_{\mathrm{MIN}}^{\alpha\mathrm{DEN}}$ the $T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$ temperatures are identical and correspond to the maximum point on the first derivative curve.

The dendrite undercooling temperature, ($\Delta T_U^{\alpha DEN}$)

The relationship between $T_{\mathrm{MIN}}^{\alpha\mathrm{DEN}}$ and $T_{\mathrm{G}}^{\alpha\mathrm{DEN}}$ is graphically presented as the hatched area in Fig. 3b and is mathematically expressed by Eq 1 as the undercooling temperature $\Delta T_{\mathrm{U}}^{\alpha\mathrm{DEN}}$. This equation has been used as a criterion for estimation of the efficiency of grain refinement additions into aluminum melt.

$$\Delta T_{\mathrm{U}}^{\alpha\mathrm{DEN}} = T_{\mathrm{G}}^{\alpha\mathrm{DEN}} - T_{\mathrm{MIN}}^{\alpha\mathrm{DEN}}\ (^{\circ}\mathrm{C}) \quad (1)$$

The above equation reflects on the observation that after the liquidus, the latent heat of evolution from the test

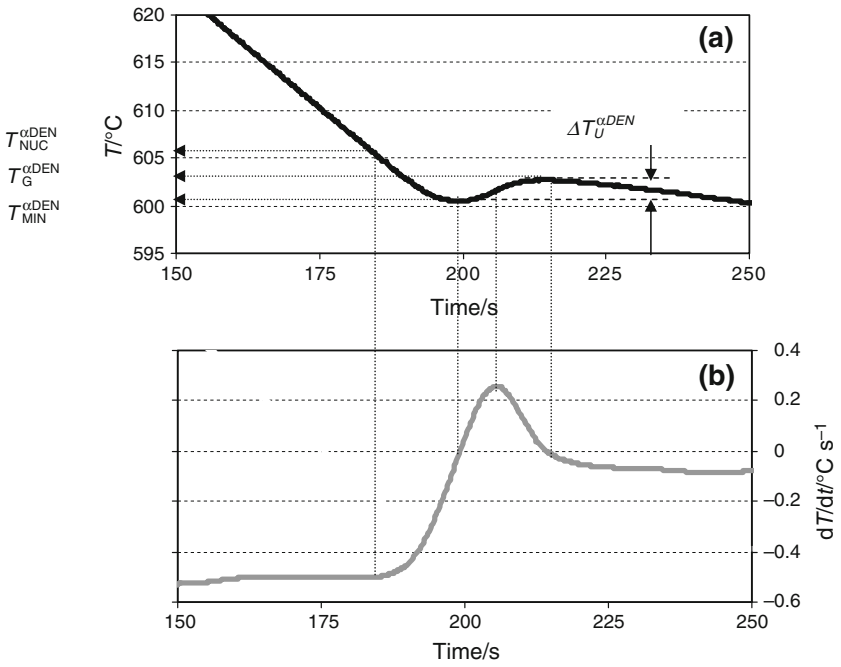

Fig. 3 Part of the cooling curve (**a**) and its first derivative (**b**) showing the definitions for the under cooling temperature and under cooling time for the 328 melts

sample temporarily equals the heat loss of the sample ($dT/dt = 0$), and then reaches the local maximum (next $dT/dt = 0$) before losing heat again.

The undercooling associated with the formation of grains is manifested as a peak on the dT/dt versus time curve (Fig. 3b), and as a loop of the dT/dt versus temperature curve (Figs. 2, 8, 10).

Grain refining criteria

According to Cibula [1], to characterize the grain nucleation and growth process, it is important to relate the equilibrium liquidus (T_L), $T_{NUC}^{\alpha DEN}$ and $T_G^{\alpha DEN}$ one to the other. The effect of grain refiners on the cooling curve parameters can be divided into three types of regimes:

Type 1: Heterogeneous dendrite nucleation is rather inefficient. Grain refinement is not possible.

$$T_L > T_G^{\alpha DEN} > T_{NUC}^{\alpha DEN}$$

Type 2: Heterogeneous dendrite nucleation is more efficient and grain refinement is possible.

$$T_L > T_{NUC}^{\alpha DEN} > T_G^{\alpha DEN}$$

Type 3: Nucleation and growth start from the local nucleus with a chemical composition different from that of the bulk melt. Grains are well refined.

$$T_{NUC}^{\alpha DEN} > T_L > T_G^{\alpha DEN}$$

Existing techniques for determination of the DCP

The DCP characteristics are important features for understanding and for consequent control of the alloy solidification process. This point marks the transition from mass feeding to interdendritic feeding in the solidification process [4–15]. During the early stages of the aluminum alloy solidification process, dendrites are separate and move freely in the melt. However, as the melt cools, the growing dendrite tips begin to impinge upon one another until a coherent dendritic network is formed. Casting defects such as macro segregation, shrinkage porosity, and hot tearing begin to develop after the DCP event [10–13]. Therefore, a thorough understanding of the solidification characteristics of the DCP and the factors that influence them are crucial for process quality control and for the engineering of a new alloy(s) and the development of its' manufacturing processes for high performance cast components.

The DCP is a physical phenomenon; however, its direct detection is virtually impossible. There are four main approaches for detection of the DCP:

(i) the mechanical (or rheological) method,
(ii) the TA method based on the application of two thermocouples and based on determination of the minimum temperature difference,
(iii) the single thermocouple method using the minimum of the second derivative of the cooling curve and,
(iv) the thermal diffusivity method (based on three thermocouples).

The mechanical method is based on the fact that the shear strength of the solidifying melt only begins to develop at the DCP [13]. This is usually measured by monitoring the torque required to rotate a paddle [4, 12–14] or a disk [4] in the melt at a constant rate. At the DCP the required torque rapidly rises, and this measured point is known as the DCP.

The TA method utilizes the two thermocouple technique developed by Bäckerud et al. [4–6, 12]. One thermocouple is located at the center (T_c) of a test crucible, and the other at a nearby inner wall (T_w). The DCP is determined by identifying the local minimum on the ΔT versus time curve ($\Delta T = T_w - T_c$) and its projection on the T_c cooling curve and the reading of the corresponding temperature as shown in Fig. 4. The reasoning that DCP occurs at this minimum ΔT versus time curve is based on the fact that the heat removal from the solid is faster than from the liquid phase. This is due to the significantly higher thermal conductivity of the solid dendrites (forming the network) in comparison to the surrounding liquid metal.

Sokolowski et al. [7, 15, 16] applied one (T_c) thermocouple located in the center of the TA cup to determine the DCP temperature characteristics. The DCP is defined as the minimum point on the second derivative curve, Fig. 5b. At that time, the inventors of the one thermocouple technique proved that this is a convenient way to obtain the required information. However, later work has shown that the thermal signal is sometimes so weak that it cannot be properly detected on the second derivative curve. This paper demonstrates that the first derivative of the center cooling curve (T_c) alone can be used successfully to accurately predict the DCP temperature characteristics.

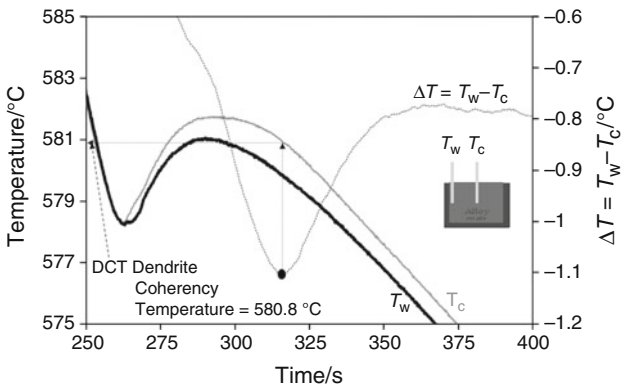

Fig. 4 T_C and T_W cooling curves versus the time and temperature difference ($\Delta T = T_w - T_c$) curve with identified DCP being the minimum of ΔT according to the Bäckerud [4, 5] method

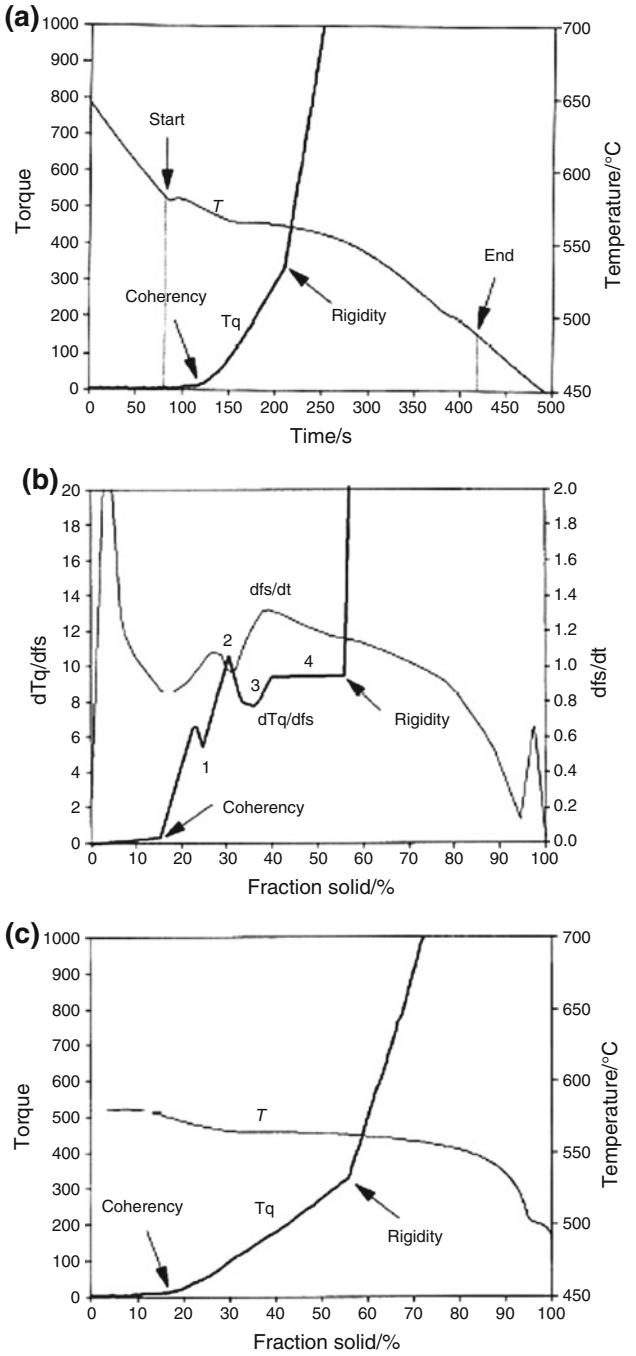

Fig. 5 Comparison techniques for determination of the DCP temperature and time using the two thermocouple ΔT technique, d^2T_C/dt^2 and using the thermal diffusivity method [18]

A third method was recently developed by Colas and a group of authors from Mexico [17]. They measured the variation in the thermal diffusivity of A319 alloy (AlSi7Cu3) during the solidification process using three thermocouples located at the center, middle, and close to the wall of the steel and graphite crucibles. Comparison of this method with the two thermocouple and other methods mentioned above concluded that all three methods yield similar results and can be used to evaluate the DCP temperature, see Fig. 5c.

Development of a new methodology for determination of DCP characteristics

Figure 6 presents the superimposed ΔT versus temperature and dT/dt versus temperature curves for the tested secondary 328 alloy. Two sets of coinciding loops are visible on both curves. As can be observed the temperatures corresponding to the respective ΔT and dT/dt maximum of these curves are identical. In order to examine more closely the first set of loops associated with the determination of the DCP characteristics, Fig. 7 presents a "zoom up" of the narrower temperature range (570–590 °C) in comparison with Fig. 6. Using the ΔT versus temperature curve approach, the temperature of the DCP (point A) was determined as being equal to 580.8 °C. As can be seen, the DCP temperature determined using dT/dt versus temperature is identical to the ΔT versus time methodology.

Comparison of several dT/dt versus temperature and ΔT versus temperature curves revealed that the elbow point (point B) on the dT/dt versus temperature curve and the minimum point (point A) of the ΔT curve versus temperature curve consistently occur at the same temperature. The point where dT/dt curve suddenly deviates from the horizontal tangent has been chosen as DCP (point B on the Fig. 7). Therefore, the one thermocouple method that utilizes the dT/dt versus temperature curve can replace the ΔT versus time (or temperature) two thermocouple method. The one thermocouple method simplifies and lowers the costs of the platform and speeds up data analysis. Experimental DCP data for a wide range of chemical compositions are presented in Fig. 10 in the 'Results and discussion' section.

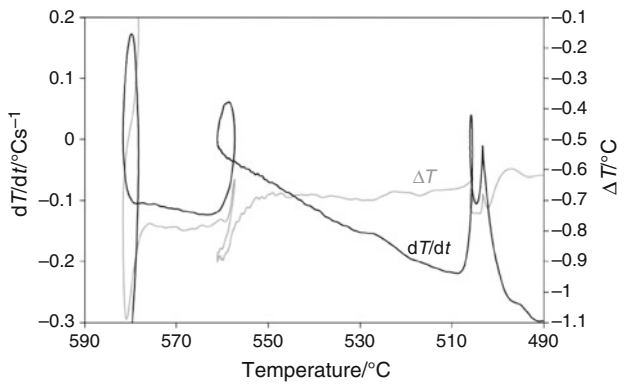

Fig. 6 Superimposed ΔT and dT/dt curves versus temperature. Data generated by the authors

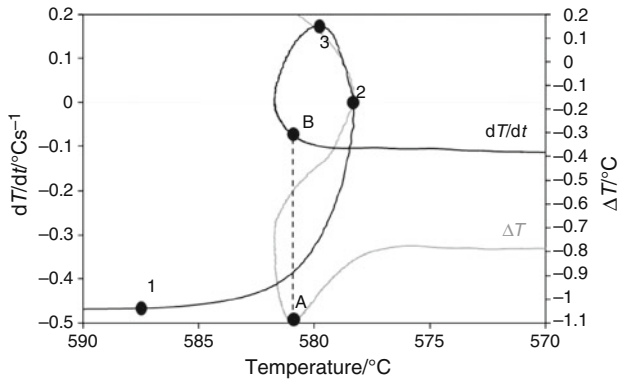

Fig. 7 "Zoom up" of the DCP event for determination of characteristics and comparison using the ΔT and dT/dt curves versus the temperature curves from Fig. 6

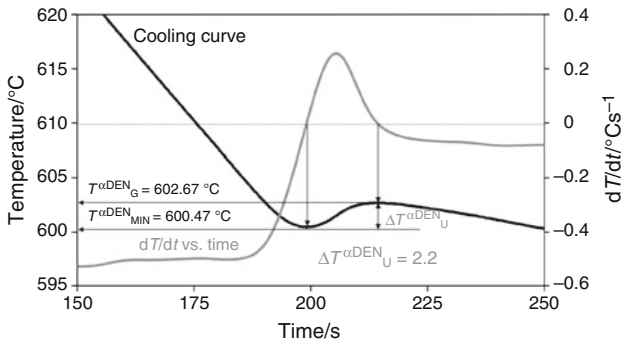

Fig. 8 Determination of the $\Delta T_{\mathrm{U}}^{\alpha\mathrm{DEN}}$ using the cooling curve and the dT/dt versus time curve

Assessment of the grain refinement efficiency using dT/dt versus Temperature

Thermal analysis methodology

Grain refinement improves strength, density, ductility, castability, machinability, pore size, its distribution and amount, increases resistance to hot tearing, and enhances feeding properties [1–3]. Fluidity, solidification rate, secondary dendrite arm spacing (SDAS), and grain refinement are interrelated. In addition, according to Arnberg et al. [9, 10] grain refinement is used to achieve a reduction in the solution treatment time, improvement of the distribution of the secondary phase particles, and porosity on a fine scale.

Increasing the silicon level can lead to coarser grains resulting from the Si poisoning effect in the melt as refined using the AlTiB master alloy(s) [8]. It was observed that Al–Si alloys having more than 3 wt% Si respond poorly to grain refinement [9]. Several mechanisms were proposed to explain the Si (and other elements like Zr, Li, and Cr) poisoning phenomena and this includes the surface tension mechanism, the ternary aluminide mechanism, the segregation mechanism, and the velocity mechanism when Al5Ti1B is used [12–14].

Since the effective mechanism of Si (and other elements) poisoning is not widely accepted and there is still a lot of uncertainty in terms of adding an efficient amount of grain refiner for a given melt and solidification conditions, therefore, the TA methodology seems to be the best for determination of these factors and their optimization. Considerable improvement in assessing grain refinement using the new methodology was recently presented by Easton and StJohn [18]. Assessment of the effect of Al–B–Ti–Sr additions to Al–Si alloy systems needs to be addressed since mutual poisoning between B and Sr and also between Ti and Sr has been reported [19, 20].

TA provides an exceptional opportunity to overcome the above problems associated with quantitative evaluation of the grain refining efficiency of the various master alloys that are used for a wide range of casting and solidification conditions. This paper provides some analytical tools for determination of the pertinent cooling curve characteristics that can be used for determination of the efficiency of the grain refinement additions into aluminum melts.

Figure 8 shows the procedure for the determination of the $\Delta T_{\mathrm{U}}^{\alpha\mathrm{DEN}}$ using the cooling curve and dT/dt versus time curve. The minimum and maximum temperatures can also be determined by finding the temperatures at which the first derivative crosses the zero axes for the dT/dt versus temperature curve.

In Fig. 9 dT/dt is plotted against the temperature, while the corresponding time axis is added for complementary analysis of the cooling curve. Using this cooling curve, the minimum and maximum temperatures can be read directly from the tangent drawn through points A and B.

Temperature is an extensive property that does not depend on the size of the TA test samples. On the other hand, time is an intensive property that is very sensitive to small change in thermal mass. Very often the mass of the

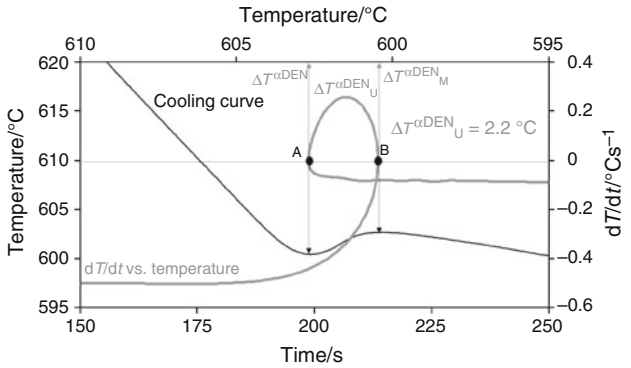

Fig. 9 Determination of the $\Delta T_{\mathrm{U}}^{\alpha\mathrm{DEN}}$ using cooling and dT/dt versus temperature curves

TA test sample is a variable parameter (affecting interpretation) that cannot be kept constant (or constant in a very narrow range) during the TA experiments. Therefore, using the temperature as an independent variable is more practical.

To determine the DCP and grain characteristics using the new methodology based on the single T_C thermocouple analysis technique, a series of experiments were performed on synthetic aluminum alloys to prove that the results were virtually identical to the traditional TA technique using two thermocouples (T_C and T_W).

Experimental procedures

The alloys used in the experiments were designed to cover a wide range of Si and Cu present in the industrial 3XX series alloys. Nine synthetic 3XX compositions were produced by melting a charge of Al 5, 7, and 9 wt% Si with 1, 2, and 4 wt% Cu levels. The chemical compositions of the experimental alloys, as determined using optical emission spectroscopy are presented in Table 1.

The 3XX aluminum alloys were melted in a reverberatory furnace. Using a rotary degasser supplied with argon, the melts were degassed to obtain the lowest possible hydrogen level (0.10 ml H/100 g Al melt) and were covered with a protective nitrogen gas blanket to prevent hydrogen and oxygen contamination. No grain refining or silicon modification master alloys were added to the melts. Melt temperatures were kept at 745 ± 5 °C. TA test samples with masses of ~550 ± l0 g were poured into stainless steel cups having a thickness of 1 mm. Low thermal mass two K-type thermocouples (T_C and T_W having 0.5 mm wires) were inserted into the melt and temperatures between 700 and 400 °C were recorded. The data for TA was collected using a high-speed National Instruments Data Acquisition System linked to a personal computer equipped with proprietary software for automated data collection, processing, and analysis. Each TA trial was repeated four times for calculation of the mean value and standard deviation of the measured and calculated metallurgical characteristics. The solidification rate during all experiments was kept at ~0.1 °C/s. The solidification rate was calculated as the ratio between the temperature difference between the liquidus and solidus temperatures and the total solidification time.

Results and discussion

The DCP characteristics for each set of experiments were determined using both the one and two thermocouple methodology. The statistical relationship between the DCP temperatures obtained using both methods are shown in Fig. 10. As can be seen, the linear function where $R^2 = 0.994$ indicates the "perfect" relationship between these two methods. Therefore, it can be concluded that the one thermocouple method that is based on the analysis of the first derivative of the cooling curve (dT/dt) versus temperature can replace the two thermocouple method.

The one thermocouple method is based on the physical phenomenon that the liquid–solid transition affects the thermal conductivity between the liquid alloy and the solid Al dendrites that are reflected on the dT/dt versus temperature curve characteristics [1]. A solid and coherent Al dendritic network (heat transfer "channels") has approximately twice the thermal conductivity in comparison with the liquid melt, and therefore, enhances the heat transfer [2]. Acceleration of the dT/dt rate contributes to the increase in fraction solid (f_S%) during dendrite growth (Fig. 11).

A further increase in fraction solid is no longer able to accelerate the heat transfer, and the latent heat generated due to the decrease in solidification reaches a steady state behind point B (Fig. 7). After this point, the first derivative of the cooling curve is almost constant, and point B signifies the DCP.

Table 1 Chemical compositions (wt%) of the synthetic AlSiCu alloys

Alloy	Si	Cu	Mg	Ti	Sr	Ni	Al
AlSi5Cu1	4.85	1.03	0.14	0.057	0.001	0.009	Rest
AlSi5Cu2	5.01	2.06	0.15	0.062	0.001	0.009	Rest
AlSi5Cu4	4.89	3.85	0.16	0.057	0.003	0.009	Rest
AlSi7Cu1	6.80	1.05	0.28	0.098	0.003	0.008	Rest
AlSi7Cu2	6.62	1.91	0.29	0.091	0.003	0.009	Rest
AlSi7Cu4	6.83	4.03	0.28	0.094	0.003	0.009	Rest
AlSi9Cu1	8.95	1.06	0.31	0.096	0.006	0.007	Rest
AlSi9Cu2	8.92	2.05	0.31	0.100	0.004	0.007	Rest
AlSi9Cu4	8.92	4.04	0.27	0.090	0.003	0.009	Rest

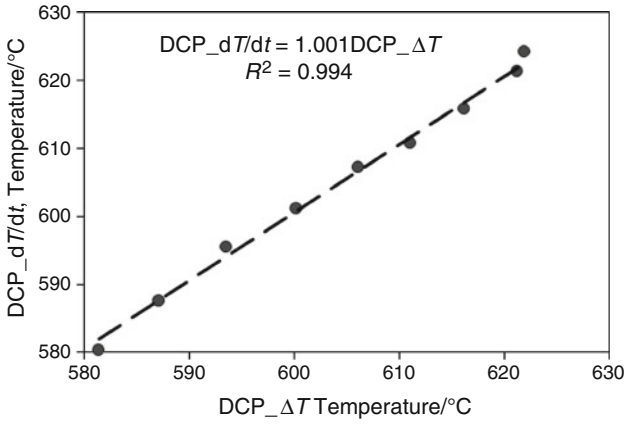

Fig. 10 Relationship between the DCP temperatures obtained using the dT/dt and ΔT methodologies, note the 1:1 correlation between this temperature characteristic determined using two techniques

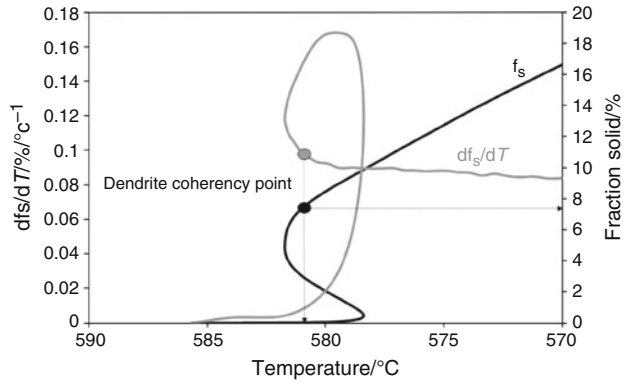

Fig. 11 Correlation between fraction solid and df_S/dT around dendrite coherency point

Conclusions

The DCP for the 3XX series of Al alloys was studied using TA. It was found that the DCP could be determined by the first derivative curve (using the one thermocouple method). The experimental results demonstrated that the DCP determined by the first derivative curve and the ΔT curve (the two thermocouple method) were identical.

The one thermocouple method simplifies the platform and lowers the costs of TA. It is, therefore, concluded that the one thermocouple method, based on the analysis of the cooling curve, can replace the two thermocouple method for determination of the DCP.

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