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Invited article

Thermodynamic evaluation of the thixoformability of Al–Si alloys

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ABSTRACT

The goal of this work is to introduce a simple method for evaluating the thixoformability of Al–Si alloys based on semisolid transformation curves obtained by differential thermal analysis (DTA), differential scanning calorimetry (DSC) and Thermo-Calc® simulation. The Al–Si alloys evaluated had a silicon content ranging from 2 to 7 wt% Si and were produced by grain refining with the addition of Al5Ti1B master alloy. The semisolid transition for each alloy was analyzed according to classical thermodynamic thixoformability criteria, i.e., by looking at the highest "knee" on the liquid fraction *vs.* temperature curve, the sensitivity of liquid fraction ($df_L/dT_{0.4-0.6}$) at 40/60% liquid fraction and the solidification interval of each alloy. The results showed that thermodynamic characterization using DTA, DSC and Thermo-Calc® was effective in predicting semisolid behavior but that use of the commonly adopted criteria reduces the number of alloys considered suitable for semisolid metal (SSM) processing. The criterion proposed here, "*The sensitivity of the liquid fraction, dfL/dT, at the desired liquid fraction, fL, exclusively for the primary phase must be as low as possible ($< 0.03\ °C^{-1}$)*", increases the number of thixoformable alloys by focusing on the control of SSM processing. Thixoforming or Rheocasting must be avoided at temperatures at which the common secondary phase, i.e., usually the eutectic phase present in SSM raw materials, undergoes transformation as this transformation is more complex and less stable than the corresponding one for the primary phase. The paper also demonstrates that kinetics parameters, such as heating (or cooling) rate, have a major effect on semisolid transformation and should be incorporated in the control of SSM processing so that the best processing window can be determined more accurately.

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1. Introduction

Semisolid materials (SSM) processing involves the forming of metallic materials in high-pressure die-casting (HPDC) machines with the alloy in the semisolid temperature range [1,2]. It was established by Flemings in 1991 [2] that, as with any manufacturing process, SSM processing has certain advantages and disadvantages. The most important advantages include energy efficiency; lower processing temperatures, reducing the thermal shock to which the die is subjected; fine, uniform microstructures, which confer enhanced properties; and reduced solidification shrinkage, resulting in dimensions closer to near-net dimensions, justifying the removal of certain machining steps and reducing costs and material losses.

SSM processing is usually divided into two categories: *rheocasting*, which involves controlled cooling of the melted liquid down to the semisolid temperature range, and *thixoforming* (or *thixocasting*), which involves controlled heating of a solid billet up to the semisolid temperature range [1,3]. In both processes the material is shaped while it is in the semisolid state. This constitutes an advantage in HPDC machines as the presence of solid within the liquid increases the viscosity of the slurry, leading to less turbulent die filling and reduced porosity, the main problem in die-cast products [2,3].

To determine whether a particular alloy can be used as raw material for SSM processing, it is essential to understand first the solid-to-liquid and liquid-to-solid transitions; second, the morphology, size and distribution of the remaining solid at the semisolid temperature; and third, the effect of both these factors on the rheological behavior. Only then can the final mechanical properties of the rheocast or thixoformed part be determined. A knowledge of the thermodynamics of the solid-to-liquid transformation is thus of key importance in SSM processing [3–7].

According to Tzimas and Zavaliangos [4], "*the use of thermodynamic data is a fast and reliable tool for alloy design*". In their groundbreaking paper they compared three evaluation methods (thermodynamic prediction, DSC and quantitative metallography

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of quenched alloys) to determine the real solid fraction in mush metal, laying the foundation for future studies. They have established that a key to the process control would be the evaluation of the variability of the solid fraction (or liquid) as a function of process temperature [5]. Liu, Atkinson and Jones [6] then proposed thermodynamic thixoformability criteria for alloys based on parameters obtained from semisolid transformation curves such as the liquid fraction vs. temperature curve. These criteria were later modified by Uggowitzer and Uhlenhaut [7]. The present study proposes to discuss these thermodynamic thixoformability criteria using CALPHAD simulation software, differential thermal analysis (DTA) and differential scanning calorimetry (DSC) to investigate partial solidification and melting of low-silicon Al–Si alloys so that more alloys are available for use in SSM processing.

2. Experimental procedure

The experimental procedure involves two steps: the production of several Al–Si alloys and characterization of the thermal transition.

The aluminum alloys were produced by conventional casting from a mix of raw materials including A356 alloy, commercial pure Al alloy and Mg–10.0 wt% Al alloy. These were melted in the appropriate proportions in a 7 kg-capacity SiC crucible in an electrical furnace at 750 °C and then poured into metallic molds to produce sixteen 220 mm-long 30 mm-diameter ingots of each alloy. Grain refining was used to obtain a non-dendritic structure by adding Al–Ti–B master alloy (Al–5.0 wt% Ti–1.0 wt% B) until a Ti content of approximately 0.2 wt% Ti was reached. Eight new alloys were produced, and an acceptable deviation range of ± 0.3 wt% Si and ± 0.15 wt% Mg was adopted. The chemical composition was determined with a BILL OES optical spectrophotometer.

Thermal characterization involved determining transformation, solidus and liquidus temperatures as well as the solid-to-liquid transition using DTA, DSC and Thermo-Calc® simulation software. DTA and DSC were performed with a NETZSCH STA 409C and samples weighing approximately 70 mg. The STA 409C has excellent resolution ($\pm 20{,}000$ digits), a data acquisition rate of 20 data s^{-1} and good sensitivity (± 10 mV), giving a measurement accuracy of more than 0.5 °C. The samples were heated to 750 °C at heating rates of 5, 10, 15, 20 and 25 °C min^{-1} and then cooled to room temperature at the same rate. Both the heating and cooling curves were analyzed. The solid-to-liquid transition was evaluated using the method first introduced by Flynn [8] based on partial integration of the heat flow curve to determine the solid fraction transformed into liquid as a function of temperature and vice versa.

Rheocasting and thixoforming processes normally use cooling and heating rates close to those used in this paper. In rheocasting, the molten metal is usually cooled at 10–25 °C min^{-1} under intense stirring (mechanical stirring, electromagnetic stirring, stirring with inert gas bubbling etc.) until the solid fraction reaches 10–30%. The semisolid slurry is then transferred to the HPDC equipment and injected into the die at a liquid fraction of between 50% and 80% [1,3–7]. Thixoforming processes use grain-refined solid billets, produced by several techniques, heated to the semisolid state at higher heating rates (up to 100 °C min^{-1}). However, to ensure a uniform billet temperature, the heating rate is reduced to 10–25 °C min^{-1} when melting begins. The semisolid transition therefore usually occurs at the same heating rate as that used in this paper. The semisolid billet containing 30–60% liquid is then transferred to the HPDC equipment and injected into the die [1,3–7].

For the Thermo-Calc® simulation, the percentages of copper, magnesium, iron and titanium were taken into account, but residual elements, such as manganese, chromium, nickel and zinc, were not. Routines based on the Scheil model for non-equilibrium solidification were used. The Thermo-Calc® software was also used to produce the phase diagrams for the alloys based on the iron, magnesium and titanium content.

3. Results and discussion

All the aluminum alloys were produced within the acceptable deviation range of ± 0.3 wt% Si and ± 0.15 wt% Mg adopted. The intention was to produce semisolid raw material using the lowest-cost method, i.e., grain refining. Table 1 shows the composition of the alloys produced.

Using the Thermo-Calc® software, the phase diagram for those alloys can be plotted. Fig. 1 shows the expected phase diagram based on an alloying-element content of up to 0.42 wt% Mg, 0.27 wt% Fe, 0.13 wt% Cu and 0.14 wt% Ti. A previous study [8] discussed thermodynamic prediction of the thixoformability of Al–Si alloys but disregarded the effect of the common elements usually found in these alloys. In Fig. 1 those areas of interest for rheocasting and thixoforming, i.e., the interval from 2 wt% Si to 7 wt% Si and high temperatures, are highlighted in color. The compositions relating to white areas are outside the scope of this work.

In Fig. 1 the red area represents the presence of liquid without any solid phase. In the upper left (very low silicon content), there is an area highlighted in white where small particles of stable $TiAl_3$ and, depending upon the manganese content, Ti_4MnAl_3 can form. These phases are identified by the Thermo-Calc® software as AL3_MDO2 and AL3_MDO22 (or approximately Al–37.3 wt% Ti and Ti–16.8 wt% Mn–24.7 wt% Al). These particles may act as heterogeneous nuclei in the molten metal for compositions with up to 2 wt% Si and serve as grain refiners in materials with low silicon content. However, this phase, which is temporary and exists only at high temperatures and with low silicon content, should disappear as the temperature decreases.

With decreasing temperature, the first solid expected to form is α phase aluminum with an FCC lattice structure known as Al_{FCC} or Al_α. This consists of a solid-solution phase of aluminum rich in iron, copper, magnesium and silicon that coexists with liquid in the area highlighted in yellow, the most important area for thixoforming and rheocasting operations, where only Al_α and liquid-rich silicon coexist. In fact, all the semisolid processing should be done in this area to avoid temperatures where Al_XM_Y precipitates and silicon particles could form, adversely affecting the characteristic semisolid rheological behavior [10].

As the temperature decreases further, the Al_α solidifies, segregating the secondary elements into the liquid-silicon-rich phase and causing small dendrites of Al_α and liquid to form. In the upper-left corner of the diagram two areas with Al_α plus liquid and α

Table 1
Al–Si alloys for use in rheocasting and thixoforming processes (wt%).

Alloy	Si	Mg	Fe	Ti	Cu	Res*	Al
Al2Si	2.00	0.48	0.12	0.14	0.05	0.10	Bal
Al3Si	2.82	0.48	0.16	0.15	0.08	0.17	Bal
Al4Si	4.04	0.47	0.22	0.15	0.10	0.17	Bal
Al5Si	4.86	0.42	0.27	0.14	0.13	0.19	Bal
Al6Si	5.94	0.42	0.30	0.14	0.15	0.21	Bal
Al7Si**	6.80	0.42	0.35	0.14	0.18	0.23	Bal

* The sum of residual elements such as Mn, Cr and Ni.
** Despite the high iron content, this composition can be classified as similar to that of commercial A356 and A357 alloys, in which the Fe content is limited to 0.2 wt% Fe and 0.15 wt% Fe, respectively.

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