

Thermal analysis and microscopical characterization of Al–Si hypereutectic alloys

F.C. Robles Hernández*, J.H. Sokolowski

Light Metals Casting Technology (LMCT) Group, Room 212A, Essex Hall, 401 Sunset Avenue, Windsor, Ont., Canada N9B 3P4

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Abstract

In this research paper are presented the identified phases by thermal analysis and microscopy presented by four 3XX.X Al–Si hypereutectic alloys that were solidified under different conditions including natural heat exchange and quenching. In addition, a qualitative analysis of the phases was conducted by EDX scanning electron microscopy. The EDX results were used to identify the stoichiometry for the particular phases based on data reported in the literature. A total of nine reactions were detected by thermal analysis that were confirmed by optical and electron microscopy, where two additional phases (Fe and Pb enriched) were also detected. Above the liquidus temperature, the phase known as Si agglomerates was identified; the nature and principal characteristics of this phase are discussed in the present paper. Using thermal analysis, the phase identification, fraction solid and nucleation temperature for all the phases was conducted.

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1. Introduction

The Al alloys is a group of casting materials that are in tonnage terms the second most popular after ferrous castings. The Al alloys have been divided into several systems that were identified based on their alloying elements by the American Aluminium Association (AAA). Aluminium–Silicon (Al–Si) alloys are the most abundant among cast alloys and have wide-spread applications, especially in the aerospace and automotive industries and are identified by the AAA as the 3XX.X series of aluminiums alloys that constitutes from 80 to 90% of the total Al castings produced world wide [1–3]. The dominant group of Al–Si foundry alloys contain between 5 and 25 wt.% Si, with Mg, Ni and Cu additions. It is important to mention that until now Fe has not been considered as an alloying element; however, current research conducted by the LMCT demonstrates the opportunities to control cast component integrity by optimizing Fe with Mn additions to transform the detrimental Fe enriched intermetallics from its typical *needle* shape into the less harm-

ful *Chinese script* phase [1,5–9] and lowering the porosity level [10].

The presence of coarse primary Si particles in the microstructure of the Al–Si hypereutectic alloys has been identified as the main limitation for their industrial use. Even with the use of silicon modifiers and high cooling rates, the primary Si particles can only be reduced in size [11]. Nonetheless, Al–Si hypereutectic alloys have already been used in the automotive industry for engine blocks production (Vega 2300 engine), cylinder liners and pistons [6–9,12]. However, there are not reports of the performance of the Vega 2300 engine and is no longer available for regular production; which could be due to that the very hard primary Si particles certainly increase locally the wear resistance of the alloy, but unfortunately Si is brittle and is easy to crack exposing the soft Al matrix to extreme wear resulting catastrophic for the automotive or aerospace components [5,13–15]. The apparent solution to fully refine the primary Si particles are the liquid metal treatments that can modify the primary Si particles into fine Al–Si eutectic, that will contribute in a considerable increment of the wear resistance for these alloys [12,16].

Al–Si hypereutectic alloys for engine blocks or cylinder liners applications is the ideal solution to eliminate cast iron cylinder liners; additionally, Al–Si hypereutectic alloys possess low thermal expansion, excellent castability and low density which

* Corresponding author at: 7507 Wildfern Dr. Mississauga, Ont., Canada L4T 3P7. Tel.: +1 519 252 6728; fax: +1 514 370 3310.

E-mail address: fcrh20@yahoo.com (F.C. Robles Hernández).

make them attractive for automotive and aerospace applications [13,17,18]. In contrast, the use of other materials such as cast iron cylinder liners for aluminiums engine blocks applications have the following disadvantages: several steps machining, formation of residual stresses, preheating of the cylinder liners for cast-in applications, additional steps for engine block production (press in) technique, cast iron is limited to air quenching in order to avoid corrosion, formation of harmful phases and interfaces between the Al engine block and the cylinder liner, low heat conductivity and high density, different machining conditions, to mention just a few [12,16].

Thermal analysis is a powerful tool that has been successfully used to determine characteristic temperatures, fraction solids and latent heats during the solidification process. Thermal analysis has been applied effectively for Al–Si hypoeutectic alloys [4,19–27]; however, the Al–Si hypereutectic alloys have not been as investigated. Bäkerud et al. [4] have reported the most complete characterization using thermal analysis and SEM/EDX for two 390 Al–Si hypereutectic alloys. However, a more detailed analysis is required in order to develop techniques that can contribute to the use of Al–Si hypereutectic alloys for wear resistant applications.

Due to the above-mentioned arguments, it is of crucial importance to conduct studies to understand the solidification pathways as well as liquid state characteristics of the Al–Si hypereutectic alloys. This will allow the development of novel technologies to fully refine the primary Si morphology, which in turn will increase the demand for the Al–Si hypereutectic alloys for wear resistance applications including the engine blocks, cylinder liners, brakes, clutches, etc. The present research investigation was conducted to understand in more detail the solidification pathways followed by the Al–Si hypereutectic alloys, nucleation temperature, fraction solid, microstructure and phases morphology among other characteristics. This can be used as a principle to determine the exact temperatures for chemical, mechanical, thermal, electromagnetic or combined techniques for the modification of the microstructure as well as the exact temperature determination for heat and/or solution treatments. By knowing the characteristics of the Si agglomerates, novel liquid and semi-solid melt treatments can be successfully developed and implemented [12,16,28–35].

2. Experimental methodology

Four Al–Si hypereutectic alloys were used for the present study; two of these alloys fell into the 390.1 AAA designation and the other two into the 393.2. The chemical composition of the above-mentioned alloys is presented in Table 1.

Ingots of 12 kg of the respective alloys were melted in a resistance furnace at a super heat (casting) temperature of 150 °C above liquidus. The alloys were held isothermally during the experiments. The melt was degassed blowing Ar gas by a degassing unit at a rate of 20 St. ft³/h, through a graphite impeller at a rotating speed of 120 rpm for 20 min. After degassing, the hydrogen level was in all cases below 0.1 ± 0.005 mL H₂/100 g of aluminiums as measured using a calibrated AISCAN unit.

After the hydrogen measurements were conducted, the molten alloys were poured into a stainless steel crucible with a capacity of 700 mL, which was then insulated on top and bottom using refractory bricks of alumina to minimize the gradient of temperatures. The poured samples had masses of 1300 ± 20 g of Al–Si hypereutectic alloy. Also, samples for each alloy composition were

Table 1

Chemical composition (in wt.%) of the investigated Al–Si alloys and their calculated liquidus (T_{LIQ}) temperature using the Si equivalency method (Si_{EQ}) [12,36]

AAA designation	Si	Cu	Fe	Mg	Mn	Ni	Calculated T_{LIQ}
390.1 ^a	13.14	4.11	0.51	0.99	0.21	2.19	600.2
390.1 ^b	13.80	4.00	0.61	1.30	0.23	0.45	599.7
393.2 ^a	25.0	1.18	0.39	0.05	0.12	0.4	752.5
393.2 ^b	28.64	2.43	0.90	0.15	0.36	0.57	794.9

poured into a die-cast bar system (reaching cooling rates >2 °C/s that minimizes Si macrosegregation), these bars were machined to obtain cylindrical specimens with 15 mm in diameter by 16 mm in height with masses of 7.5 g. Both types of samples were solidified under natural heat exchange conditions for all the alloys. During solidification, a K thermocouple previously calibrated according to the standard of the National Institute of Standards and Technology (NIST) was inserted in the centre of the sample to record the cooling curve from the superheat temperature until 400 °C. The thermal analysis was conducted three times for every alloy composition and solidification conditions to ensure the quality of the results. The results presented are the average of all the thermal analysis measurements. Samples for the 390.1^a and 393.2^a alloys were quenched in a salt-water solution (H₂O + 15 wt.% NaCl) from various temperatures from above and below the liquidus temperature.

Sections of the analytical samples were cut parallel and in close proximity to the thermocouple for metallographic analysis. The metallographic samples were mounted in cold resin and polished following the standard metallographic procedures. Optical microscopy, scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) spectroscopy were used for the identification of the phases. The stoichiometry of the phases was determined comparing the results of the EDX semi-quantitative chemical analysis with the results reported in the literature for alloys with similar composition.

3. Results and discussions

3.1. Thermal analysis

Fig. 2 shows the cooling, first derivative, baseline and fraction solid curves for the 390.1^{a,b} and 393.2^{a,b} Al–Si alloys as obtained from thermal analysis for the two types of samples. The first derivative of the cooling curve was determined to enhance slope changes that are related to the solidification reactions for the different phases; and facilitates the determination of the nucleation time, temperature, fraction solid and cooling rate of the phases. It is clear from this figure that both alloys present similar nucleation reactions and solidification characteristics, nonetheless, some differences that will be discussed were found. The use of the two thermal analysis methods allowed the precise determination of the characteristics for all the solidification reactions. For instance, using the small samples was assessed the exact temperature and fraction solid involved for the Si agglomerates; while for the Ni and Cu enriched phases, the thermal analysis conducted using the large samples was more appropriate.

The present investigation provides unique information for the solidification pathways followed by the Al–Si hypereutectic alloys; particularly, the identification of a reaction at temperatures above liquidus by thermal analysis and image analysis. This reaction can correspond to the transformation of the Si agglomerates into primary Si particles. The identification of this reaction was carried out using high-resolution thermal analysis

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