Rheo-processing of an alloy specifically designed for semi-solid metal processing based on the Al-Mg-Si system

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Abstract

Semi-solid metal (SSM) processing is a promising technology for forming alloys and composites to near-net shaped products. Alloys currently used for SSM processing are mainly conventional aluminium cast alloys. This is an obstacle to the realisation of full potential of SSM processing, since these alloys were originally designed for liquid state processing and not for semi-solid state processing. Therefore, there is a significant need for designing new alloys specifically for semi-solid state processing to fulfil its potential. In this study, thermodynamic calculations have been carried out to design alloys based on the Al-Mg-Si system for SSM processing via the 'Rheo-route'. The suitability of a selected alloy composition has been assessed in terms of the criteria considered by the thermodynamic design process, mechanical properties and heat treatability. The newly designed alloy showed good processability with rheo-processing in terms of good control of solid fraction during processing and a reasonably large processing window. The mechanical property variation was very small and the alloy showed good potential for age hardening by T5 temper heat treatment after rheo-processing.

Keywords: Aluminium alloys, semi-solid processing, CALPHAD, alloy design, mechanical properties

1 Introduction

Semi-solid metal (SSM) processing is a promising technology for forming alloys and composites to near-net shaped products [1-3]. It is over 30 years since the concept of semi-solid metal processing was first discovered through the observation of thixotropic behaviour of a partially solidified melt which has a fluidity of machine oil when the normal dendritic structure is broken up [4, 5]. Semi-solid metal processing is different from the conventional metal forming technologies, which use either solid metals/alloys (solid state processing) or liquid metals/alloys (casting) as starting materials. SSM processing uses semi-solid metal slurries, in which non-dendritic solid particles are dispersed in a liquid matrix. There are two routes for processing in the semi-solid state; 'thixocasting', where the slurry is first cast as a billet and then reheated back to the semi-solid condition before component shaping, and 'rheocasting', which involves producing a non-dendritic slurry usually by the application of melt shearing during solidification followed by component shaping [6].

Since the 1990's, SSM processing has concentrated mainly on aluminium alloys and today semi-solid processed parts of aluminium and magnesium are quickly replacing the conventionally processed parts. In the past decade, SSM processing has experienced intensive research, development and commercialisation attempts [2, 3]. Today SSM processing has established itself as a scientifically sound and commercially viable technology for production of metallic components with high integrity, improved mechanical properties, complex shape and tight dimensional control and more importantly, it has demonstrated a great potential for further technological development and commercial exploitation [3]. There have been three comprehensive literature reviews on the SSM processing field [1-3]. Based on the current situation it appears that efforts should be directed to; development of new rheo (slurry-on-demand) based processes as cost efficient SSM processes for commercialisation; understanding the mechanisms of formation of globular structure and rheological behaviour of SSM slurries to tailor and control an ideal slurry generation; development of new alloys specifically designed for SSM processing to maximise the benefits of SSM processing; and the microstructure-property relationships in SSM formed materials that has been lacking in the past.

Aluminium cast alloys are still being used for SSM processing, which does not take full advantage of semi-solid state processing since casting alloys are designed specifically for liquid melt processing. Due to this, effective control during the slurry making process is lacking, efficient tailoring of microstructure is hindered and the complete understanding of structure-property relationship to modify or enhance the mechanical properties of the final parts, are still to be realised. Therefore, there is a significant need to design new alloys specifically for semi-solid state processing, to realise and exploit its full advantage.

In this study, thermodynamic calculations have been carried out to design alloys based on the Al-Mg-Si system for SSM processing. A selected alloy composition has been assessed upon its suitability to rheo-processing in terms of the criteria considered in the thermodynamic design process and also upon its mechanical properties and heat treatability.

2 Alloy design and selection by the CALPHAD approach

In the past few years there has been a strong desire for the development of new alloys tailored specifically for the needs of SSM processing [3, 7, 8]. Such alloys should have less temperature sensitivity of solid fraction, suitable freezing range, potential for age hardening, favourable rheological properties and mechanical properties. In this

work, thermodynamic calculation is used as the first step of aluminium alloy design for SSM processing to analyse the effects of alloy composition on the temperature sensitivity of solid fraction, freezing range and potential for age hardening, and to narrow the composition range with promising SSM processability.

Conventional alloy development has been primarily based on experimental approach and is frequently performed by trial and error methods. Such experimental approach to alloy design is cost intensive and time consuming. In the view of the large number of possible alloy systems, alloy compositions and processing parameters, computational thermodynamics can provide a useful guide to the experiments.

Computational thermodynamics, commonly referred to as 'CALPHAD', (CALculation of PHAse Diagram), is a modern tool that supplies quantitative data to guide the development of new alloys [9, 10]. In this approach, the Gibbs free energy of individual phases is modelled as a function of composition, temperature, magnetic critical temperature and sometimes pressure, and collected in a thermodynamic database. This enables the calculation of multi-component phase diagrams and the tracking of individual alloys during heat treatment or solidification by the calculation of phase distributions and compositions. These are essential data for understanding and controlling the behaviour of engineering alloys. Focused experiments can then be carried out on only the promising alloy compositions identified by thermodynamic calculations. Thus, it is a powerful tool to cut down the cost and time for alloy development.

Since Mg and Si are the most important alloying elements for both cast and wrought aluminium alloys, in this study, the basic considerations in alloy design for SSM processing are systematically analysed and applied to the Al-Mg-Si system. CALPHAD calculations have been performed at the Al-rich corner in the Al-Mg-Si

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system using the Thermo-Calc software [11], and the AL-DATA database provided by Thermo-Tech Ltd, (Guildford, UK).

A schematic binary eutectic phase diagram is shown in Fig. 1, which defines some of the terminologies used later on for thermodynamic alloy design. In Fig. 1, $T_{\rm m}$ is the melting point of pure element A, $T_{\rm E}$ is the eutectic temperature, $T_{\rm SS}$ is the SSM processing temperature, $T_{\rm A}$ is the aging temperature, and $T_{\rm L}$ and $T_{\rm S}$ are the liquidus and solidus temperatures respectively. In addition, ΔC is the difference in solubility of element B in the α phase between $T_{\rm SS}$ and $T_{\rm A}$, which is used here to measure the potential for age hardening of the T5 temper.

The phases present at the Al-rich corner of the Al-Mg-Si system are; liquid, α Al, Mg₂Si and (Si) with no ternary compounds. The following ternary eutectic reaction is predicted to occur at 557.2°C: L $\rightarrow \alpha$ Al + Mg₂Si + (Si)

2.1 Alloy design criteria

The main factors that influence SSM processability and properties of the final components are: temperature sensitivity of solid fraction, temperature range for solidification (freezing range), potential for age hardening, morphology of the solid phase in semi-solid state, rheological properties and castability of the semi-solid slurry. Among them the solid phase morphology, the rheological properties, and castability of the semi-solid slurry are sensitive to processing conditions and cannot be predicted with CALPHAD. In this work we use CALPHAD simulation to study the influence of compositional variation on temperature sensitivity of solid fraction, freezing range and potential for age hardening. Experiments for microstructure and mechanical properties can then be focused on the most promising alloys selected by thermodynamic calculations. The selection process and the selection criteria are described as follows:

a) SSM processing temperature and temperature sensitivity of solid fraction

Accurate evaluation of temperature sensitivity of the solid fraction (f_s) at SSM processing temperature (T_{SS}) is a critical factor to ensure a wide and stable SSM processing window. This parameter to a large extent controls the rheological behaviour and the evolution of microstructure in the semi-solid state [12].

The temperature sensitivity of solid fraction can be defined by the slope of the solid fraction versus temperature curve and represented as $|df_s/dT|$. Since $|df_s/dT|$ is usually negative, the absolute value is used here. When $|df_s/dT|$ is large, a slight variation in temperature induces a large change in the solid fraction. Thus, a minor decrease in temperature can lead to a considerable increase in f_s , and therefore a non-favourable microstructure for SSM processing due to inhomogeneous deformation and liquid segregation during mould filling. Therefore a smaller $|df_s/dT|$ at T_{SS} indicates better SSM processability.

It is proposed here that for good processability, $|df_s/dT| < 0.015$ at the SSM processing temperature, (T_{SS}) . This means, that a 1.5% change in f_s of the slurry is permitted for every 1°C change in temperature, *T*. A temperature accuracy of $\pm 3^{\circ}$ C is the achievable temperature control under SSM processing conditions. Further improvement in the accuracy of the temperature control during SSM processing is difficult and expensive, especially on an industrial scale. With a temperature accuracy of $\pm 3^{\circ}$ C, under well-controlled large scale processing conditions, the f_s change will be restricted to $\pm 4.5\%$.

For a given alloy, T_{SS} determines f_s and $|df_s / dT|$. However, during alloy design, $|df_s / dT|$ at different temperatures dictate the selection of T_{SS} . Since T_{SS} differs

greatly from one alloy to another, a typical solid fraction of $f_s = 0.3$ for rheo-route SSM processing is chosen here to analyse the influence of alloy composition on $|df_s/dT|$.

b) Freezing range

Freezing range is usually defined as the temperature range between the liquidus and solidus of a given alloy, ΔT_{L-S} . It is mainly determined by alloy composition and affected to a certain extent by the processing conditions such as cooling rate. Pure metals and eutectic alloys are not suitable for SSM processing due to the lack of freezing range. On the other hand, too wide a freezing range may lead to poor resistance to hot tearing, [13]. Therefore, the temperature range between the SSM processing temperature T_{SS} and solidus T_S , ΔT_{SS-S} , is important for SSM processing. It is proposed here that for good processability and high resistance to hot tearing, the freezing range should be, $10 \,^{\circ}\text{C} < \Delta T_{SS-S} < 150 \,^{\circ}\text{C}$.

c) Potential for age hardening

In order to achieve high strength, the alloys designed for SSM processing should be amenable to age hardening. Such alloys may have the advantage to utilise the more cost effective T5 temper heat treatment rather than the expensive T6 temper heat treatment. The potential for age hardening may be achieved by careful selection of alloy composition.

For alloys based on the Al-Mg-Si system, only the precipitation of Mg₂Si leads to age hardening. For T5 temper, an alloy is quenched from T_{SS} and then artificially aged at temperature T_A . The potential for age hardening of the α Al phase is defined by the equilibrium concentration difference, ΔC , of the Si and Mg in the α Al phase between T_{SS} and T_A (see Fig. 1). The maximum amount of Mg₂Si precipitates formed during the aging process, ΔC_{Mg_2Si} , is determined by the super-saturation of the Mg and Si in the α Al phase according to the stoichiometric ratio between Mg and Si in the Mg_2Si phase, i.e., Mg:Si = 0.634:0.366 in weight percent. If ΔC_{Mg_2Si} for both T5 and T6 tempers is less than 0.5 wt. %, then the alloy is deemed to be non-heat treatable, therefore, ΔC_{Mg_2Si} should be larger than 0.5 wt.%.

The ageing temperature for both the T5 and T6 temper is assumed to be $\sim 200^{\circ}$ C, while the solution temperature for T6 temper is assumed to be slightly below the solidus temperature.

d) Proportion of different phases during solidification

Using thermodynamic calculations, the proportion and distribution of different phases during the solidification processing can also be tracked. The proportion of the α Al phase, f_{α}^{p} , formed before the ternary eutectic reaction, (liquid $\rightarrow \alpha$ Al + Mg₂Si + (Si)), should not be less than 0.6 in order to ensure that α Al is the primary (major) constituent and continuous phase in the final casting, to promote ductility and toughness. A large proportion of eutectic and intermetallic phases will result in poor mechanical properties therefore the proportions of Mg₂Si and (Si) phases formed before the ternary eutectic reaction, $f_{Mg_2Si}^{p}$ and f_{Si}^{p} , should be as small as possible for good mechanical properties. Hence, it is intuitively set here that, $f_{a}^{p} > 0.6$, $f_{Mg_2Si}^{p} < 0.05$ and $f_{Si}^{p} < 0.05$ in order to achieve good mechanical properties of the final components.

e) Composition tolerance

The composition of alloys should be allowed to vary slightly around their nominal compositions without causing any processing difficulties. If a minor variation in composition leads to poor processability, the alloy is not applicable in practice. Thus, a small variation in the composition of the alloy should only slightly vary its f_s - T

curve, in other words, the change in $|df_s/dT|$ at $f_s = 0.3$ must be reasonably small and its absolute value still less than 0.015 to satisfy the criteria of good processability.

Based on the thermodynamic selection criteria discussed above, the alloys with promising processability and improved component properties can now be selected. These alloys should have less temperature sensitivity of solid fraction, suitable freezing range and potential for age hardening, a large proportion of the primary α Al phase and a small proportion of (Si) and Mg₂Si phases before ternary eutectic reaction and good composition tolerance. The selection results for the Al-Mg-Si system are shown in Fig. 2 for typical rheo-route SSM processing for $f_s = 0.3$. The alloy compositions in the Al-Mg-Si system that satisfy all the criteria are selected as the most promising alloys for SSM processing. The rest of the compositions, which do not obey one or more or all of the design criteria, have been eliminated.

In addition to the above thermodynamic considerations, other factors that influence the castability, the rheological properties and the solid phase morphology, should also be considered. The content of Si influences the castability of a given alloy, the higher the Si content, the better the castability, and minor additions of other alloying elements greatly improve the processability and mechanical properties of the final components. For example, minor additions of Fe reduces die-soldering, Ni, Cr, Co, Mo and other high melting elements all improve the resistance to hot tearing by improving the alloy's high temperature strength [13]. It should be pointed out that addition of minor alloying elements may not substantially affect the basic thermodynamic characteristics of the base alloy. Therefore, conventional wisdom for the selection of minor alloying elements for aluminium alloys is applicable to the alloys designed here for SSM processing.

2.2 Selection of a suitable alloy composition for rheo-processing

The compositions predicted to be most suitable for rheo-processing at $f_s = 0.3$ are shown in Fig. 2. The alloy compositions are listed in Table 1 and vertical sections from the Al-Mg-Si ternary phase diagram at fixed Mg contents of 1, 2, 3 and 4 wt. %, are presented in Fig. 3.

Alloys of group 1 in Table 1, are similar in composition to commercial aluminium cast alloys A356 (Al-7Si-0.3Mg) and A357 (Al-7Si-0.5Mg). All alloy compositions are presented in wt. % unless specified otherwise. Thus, group 1 alloys will result in similar processability and mechanical properties to A356 and A357 alloys and therefore they are not chosen for further experimental study.

Alloys of group 2 in Table 1, have increased Mg content of 2 wt. % compared to A356 and A357 alloys, which will result in higher strength but lower ductility. Silicon in aluminium alloys improves castability but at the same time it drastically reduces the ductility. If the three alloys, 6, 7 & 8 wt. % Si, from group 2 are compared on the 2 wt. % Mg vertical section in Fig. 3, then the 6 wt. % Si alloy shows the least amount of primary Si upon solidification and therefore will have better ductility than the 7 & 8 wt. % Si alloys. Thus, from group 2, the Al-6Si-2Mg alloy looks the most promising candidate to exhibit a good compromise between strength and ductility.

Alloys of group 3 and 4 in Table 1, have higher Mg contents and from the vertical sections of 3 and 4 wt. % Mg, these alloys are observed to have primary Mg₂Si forming before the solidus line, which is thought to be non- favourable for mechanical properties. This is due not only to its brittle nature but also as it will not fully contribute towards precipitation strengthening when the alloy is heat treated for T5 temper.

The most promising composition for the first experimental attempt with rheoprocessing in this work is selected to be the Al-6Si-2Mg alloy. The calculated equilibrium solid weight fraction f_s and $|df_s/dT|$ of the Al-6Si-2Mg alloy are plotted in Fig. 4, as a function of temperature. The liquidus temperature of the Al-6Si-2Mg alloy is 615.7°C and the solidus temperature is 557.2°C, providing a freezing range of 58.5°C.

The solidification path of the alloy is summarised as follows: at 615.7°C, the primary α Al phase solidifies from the liquid phase, as the temperature decreases to 559.3°C, the (Si) phase begins to form with a corresponding equilibrium solid fraction of the α Al phase at 559.3°C of 0.6181, when the temperature decreases further to the ternary eutectic temperature of 557.2°C, the remaining liquid phase decomposes to the α Al + Mg₂Si + Si ternary eutectic and the solidification process finishes. The maximum solid fraction of the α Al phase directly formed from the liquid phase above the ternary eutectic temperature is 0.6681, while that of the (Si) phase above the ternary eutectic temperature is 0.0067. The proportion of the ternary eutectic is 0.3252.

3 Experimental study

3.1 Experimental procedures

The Al-6Si-2Mg alloy was prepared using a Carbolite VCF 12/23 electric resistance furnace. The starting materials were commercial purity Al (LM0), Al-50Si master alloy, supplied by KBM Affilips Ltd, (Alcester, UK) and pure Mg (99.89%), supplied by Magnesium Elektron, (Manchester, UK). The Al-6Si-2Mg alloy was later modified with up to 0.5wt. % Fe to avoid die-soldering during rheo die-casting. Iron was added in the form of Al-46Fe master alloy, supplied by KBM Affilips Ltd, (Alcester, UK).

Rheo-processing was performed by the 'Rheo Die-Casting' (RDC) technology, which is a combination of a 'twin-screw slurry maker' and the 'high pressure diecasting' (HPDC) process, [14]. In this process, a liquid alloy is converted into a semisolid slurry under high shear rate and high intensity of turbulence, provided by the twin-screws. The temperature field and composition field in the twin-screw slurry maker are expected to be very uniform, [15]. The RDC process allows the semi-solid slurry produced from the twin-screw slurry maker to be cast into any shape or component. In this work, the semi-solid slurry was cast into four round tensile bars for microstructure analysis and for evaluation of the tensile properties in both the as-cast and heat treated states.

Samples for microstructural observation were mounted in bakelite, ground using 120, 800, 1200, 2400, 4000 grit SiC papers and polished on a cloth with 0.04µm diamond suspension. The microstructures were recorded using a light microscope fitted with a digital camera and image capture software.

Tensile testing was performed on a hydraulic 'Instron 8501' system connected to a PC for automated testing and calculation of tensile results. The dimensions of the test pieces were 6.3mm in diameter with a gauge length of 63mm. A crosshead speed of 2.5mm/min was used at all times and an external extensometer of 50mm gauge length was manually attached to each test piece during testing to obtain accurate elongation results. All samples were tested at room temperature. The yield strength was measured at an offset of 0.002mm/mm (0.2% proof strength) from the origin of the stress-strain curve.

Heat treatments were performed in an electrical resistance furnace in an air atmosphere. Solution treated samples were water quenched to room temperature and artificially aged samples were allowed to cool naturally in air to room temperature. The heat treatment cycles were designed by taking hardness measurements at various periods of time of solution and ageing treatments. The samples were water quenched to

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room temperature in-between the solution or ageing time intervals prior to taking hardness measurements.

3.2 Results of experimental evaluation

The heat treatability and processability of the Al-6Si-2Mg-0.5Fe alloy is evaluated along with microstructure and mechanical properties. More importantly, the rheo-processability of the alloy is compared with the thermodynamic considerations and the criteria that were used in the selection of suitable alloy compositions from the Al-Mg-Si system.

Fig. 5 shows the resulting microstructure after gravity casting from a temperature of 700°C into a cold cylindrical cavity steel mould (20mm in diameter and 130mm in length). The microstructure clearly demonstrates typical dendritic growth morphology with long columnar dendrites observed in a matrix of a fine eutectic.

The RDC process utilises high pressure die-casting, offering a high cooling rate for solidification of the semi-solid slurry produced after intensive shearing from the twin-screw slurry maker. The barrel of the twin-screw slurry maker was set at a temperature of 605°C (calculated $f_s \sim 0.25$) with a shear time of 60s and screw speed of 300 rpm, which is equivalent to a shear rate of 534s⁻¹. The resulting cross-sectional microstructures from one of the tensile specimens produced by RDC are shown in Fig. 6.

The semi-solid microstructure obtained by RDC consists of globular primary α Al solid particles (~50µm in diameter) uniformly distributed amongst a much finer secondary microstructure. The larger primary α Al particles originated from the primary solidification, which took place inside the twin-screw slurry maker, while the very fine secondary microstructure was solidified inside the die-cavity from the remnant liquid.

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The secondary microstructure is made of fine α Al dendrites/particles distributed in a eutectic matrix. It should be noted that, some solid particles are formed during the transfer of the slurry along the shot sleeve, these particles are considered in the secondary solidification.

Rheo die-casting of Al-6Si-2Mg-0.5Fe alloy resulted in yield strengths of 250–260 MPa, tensile strengths of 320–335 MPa and percentage strain (ductility) of 2.0–3.0 %. The thermodynamic alloy design criteria for the potential for age hardening was assumed to be an ageing temperature of 200°C for T5 temper, with a solution treatment temperature assumed to be slightly below the solidus temperature, (545°C). The optimum condition for heat treatment by T5 temper was found to be two hours of ageing at 200°C followed by natural cooling in air to room temperature. The resulting tensile properties after heat treatment are given in Table 2.

The T5 temper heat treatment resulted in a very high increase of yield strength from 260 MPa to 323 MPa, an increase of tensile strength from 335 MPa to 360 MPa and a percentage strain (ductility) decrease from 3.0 % to 1.2 %. The T4 temper heat treatment resulted in a decrease of both the yield strength and tensile strength, but increased the percentage strain (ductility) greatly from 3.0 % to 8 %. The T6 temper increased the yield strength from 260 MPa to 312 MPa with little change in the tensile strength and decreased the elongation to 1.6 %.

In SSM processing, the solid fraction/volume of a melt is significant to successful processing in terms of fluidity during die-filling and, in turn, formation of defects such as hot-tears, cold shuts, solid/liquid segregation and gaseous porosity. On the other hand, it is quite difficult to control the solid fraction of semi-solid slurries especially in thixoforming processes. Therefore, to investigate the control of solid fraction in the RDC process, tensile specimens were prepared at shearing temperatures

of 600°C, 605°C and 610°C, which theoretically correspond to solid fractions of 30%, 20% and 10%, respectively. The same shearing time of 60s and the screw speed of 300 rpm were set for RDC processing at all temperatures. The resulting cross-sectional microstructures from a tensile specimen produced at each RDC processing temperature of the Al-6Si-2Mg-0.5Fe alloy are shown in Fig. 7 with their corresponding solid fractions measured by use of an optical image analysing software.

The actual measured solid fractions resulting from the microstructures of each temperature are higher than the calculated solid fractions set in terms of temperature during RDC processing. The resulting tensile properties obtained after testing the tensile specimens produced by RDC processing at 600°C, 605°C and 610°C are plotted against shearing temperature which represents their equilibrium solid fractions, as shown in Fig. 8.

RDC processing of the Al-6Si-2Mg-0.5Fe alloy; at 600°C resulted in yield strength of 255 MPa, tensile strength of 320 MPa and tensile strain (ductility) of 2.1 %; at 605°C resulted in 258 MPa, 330 MPa and 2.5 %; and at 610°C resulted in 265 MPa, 335 MPa and 3.8 %, respectively. The trend of tensile property with shearing temperature proves that the Al-6Si-2Mg-0.5Fe alloy can be processed successfully in the window of 15°C (600–615°C) and solid fraction range of 0-30% f_s with nominal change in tensile properties. The tensile strain (ductility) is observed to be higher (3.8 %) at a shearing temperature of 610°C (calculated $f_s \sim 10\%$), indicating that the Al-6Si-2Mg-0.5Fe alloy has a reasonably large processing window. It should be noted that the slurry transferred to the die is of uniform temperature and composition due to the turbulent and distributive shearing from the twin-screws, which results in a fine nondendritic structure during secondary solidification. A T5 temper was carried out on further tensile specimens obtained from RDC processing at 610°C, which resulted in yield strength of 321 MPa, tensile strength of 362 MPa and tensile strain (ductility) to fracture of 2.1 %.

4 General discussion

This investigation was performed to design new alloys to optimise the processing and property benefits of the RDC process, and to explore the rheoprocessing of the designed alloys to achieve better control of semi-solid microstructure, maximum microstructural refinement and uniformity, and improved mechanical properties. The work aimed at exploring the feasibility of using computational thermodynamics to design alloys for SSM processing, rather than the trial-and-error approach. It is envisaged that through a proper design and selection of alloys with optimised rheo-processing parameters, aluminium alloy components can be manufactured with RDC, to offer better microstructural control, integrity and performance, than achievable through conventional casting.

For the Al-Si-Mg based alloys, we identified a set of CALPHAD criteria on the basis of RDC processability and heat treatability. Our case study of the RDC processed Al-6Mg-2Si-0.5Fe alloy showed that this set of CALPHAD alloy design parameters were valid for effective development of RDC alloys. Being consistent with the CALPHAD prediction, the rheo-processability of the selected new Al-6Si-2Mg-0.5Fe alloy was found to be reasonably good in terms of accurate control of solid fraction in the semi-solid slurry. The alloy had a large semi-solid processing window of 15°C from below its liquidus temperature in which the solid fraction could be controlled fairly well due to the low $|df_s/dT|$ values. The experimentally observed solid fraction was consistently higher than the CALPHAD prediction. This could be attributed to possible

errors of melting temperature about 3 to 4 °C, either arising from the experimental control or due to inaccuracy in the thermodynamic database. The use of twodimensional micrograph data for three-dimensional volume fraction could be another source of statistical error, considering that the solid particles were of globular morphology. In addition, the RDC samples of selected Al-6Si-2Mg-0.5Fe alloy also demonstrated significant response to age hardening (T5 temper), which verified the feasibility for the identified criterion for heat treatability via controlling ΔC_{Mg_2Si} .

The yield strength and tensile strength of the RDC samples were quite stable and the ductility was higher with decreasing solid fraction as a result of the novel, extremely refined, secondary solidification structure as determined in this investigation. This was also of great importance in providing better mechanical properties compared to other casting processes.

5 Conclusions

As a first step of aluminium alloy design for SSM processing, thermodynamic calculation by the CALPHAD approach was used to analyse the effects of alloy composition on the temperature sensitivity of solid fraction, freezing range and potential for age hardening. These three major criteria taken into consideration for successful semi-solid processing were systematically analysed and applied to the Al-Mg-Si system, from which the composition range was narrowed down to the eight most promising alloy compositions. These were further analysed from their respective pseudo-binary equilibrium phase diagrams, from which the Al-6Si-2Mg alloy composition was selected for the first experimental attempt with semi-solid rheo-processing (RDC). The alloy was modified by minor addition of Fe which improved its processability with RDC.

The newly designed Al-6Si-2Mg-0.5Fe alloy showed good processability with rheo-processing (RDC) in terms of accurate control of solid fraction during processing. It also had both a reasonably large processing window, in which the mechanical property variation was very small, and also showed good potential for age hardening by T5 temper heat treatment following rheo-processing.

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Group 1	Group2	Group3	Group 4
Al - 6Si - 1Mg	Al - 6Si - 2Mg	Al - 6Si - 3Mg	Al - 5Si - 4Mg
Al – 7Si – 1Mg	Al – 7Si – 2Mg		Al – 6Si – 4Mg
	Al – 8Si – 2Mg		

Table 1:

The list of alloys resulting from the thermodynamic alloy design for SSM processing in the Al-Mg-Si system, presented in Fig. 2.

Temper	Yield Strength at 0.2% Offset MPa	Ultimate Tensile Strength MPa	Elongation to Fracture %
F	260	335	3.0
Т5	323	360	1.2
T4	164	270	8.0
T6	312	342	1.6

$\underline{Al-6Si-2Mg-0.5Fe-RDC}$

Table 2:

Tensile properties resulting after heat treatment of rheo die-cast Al-6Si-2Mg-0.5Fe alloy by T5, T4 and T6 tempers. (The properties reported are best achieved under present work and not average value). {**F:** as-cast, **T5 temper:** 2 hours at 200°C followed by natural cooling, **T4 temper:** 2 hours at 545°C followed by water quench, **T6 temper:** 3 hours at 545°C followed by water quench and 24 hours at 155°C followed by natural cooling}





A schematic binary eutectic phase diagram illustrating relevant terminologies





Projection of compositions that satisfy $|df_s / dT| < 0.015$ in the Al-Mg-Si system at $f_s = 0.3$. Round coloured spots represent compositions which have been eliminated due to the lack of one or more or all of the listed design criteria. Crossed squares represent alloy compositions which are suitable for SSM processing for $f_s = 0.3$.



Fig. 3:

Calculated equilibrium vertical sections from the Al-Mg-Si ternary phase diagram at fixed Mg contents of, (a) 1wt. %, (b) 2wt. %, (c) 3wt. % and (d) 4wt. %.





Calculated equilibrium $|df_s / dT|$ and $f_s - T$ curve for the Al-6Si-2Mg alloy.



Fig. 5:

The microstructure produced upon final solidification to room temperature of the Al-6Si-2Mg-0.5Fe alloy cast from 700°C into a cold cylindrical cavity steel mould presented at two different magnifications. (no etchant used)



Fig. 6:

Optical micrographs of the Al-6Si-2Mg-0.5Fe alloy at different magnifications of a cross-sectioned tensile specimen from the RDC preform produced after shearing at 605°C for 60s at screw speed of 300 rpm (shear rate of $534s^{-1}$) with the die heated to 200°C. (etched with a solution of 1g NaOH in 100mL H₂O, for 10secs)

Al-6Si-2Mg-0.5Fe - RDC



(a) Sheared at 600°C, Actual $f_s \sim 36\%$



(b) Sheared at 605°C, Actual $f_s \sim 25\%$



(c) Sheared at 610°C, Actual $f_s \sim 17\%$

Fig. 7:

Optical micrographs at two different magnifications of a cross-sectioned tensile specimen obtained from the RDC preform of the Al-6Si-2Mg-0.5Fe alloy produced after 60s of shearing with the screw speed set at 300 rpm (*shear rate of* 534^{s-1}) at shear temperature of: (a) 600°C corresponding to 30% f_s , (b) 605°C corresponding to 20% f_s , and (c) 610°C corresponding to 10% f_s , with their actual measured solid fractions. (etched with a solution of 1g NaOH in 100mL H₂O, for 10s)



Fig. 8:

Plot of tensile properties against shearing temperature obtained from RDC processing of the Al-6Si-2Mg-0.5Fe alloy at 600°C, 605°C and 610°C, which theoretically correspond to calculated solid fractions of 30%, 20% and 10%, respectively.