EFFECTS OF COOLING RATE AND SILICON CONTENT ON Al/SiC$_P$ MMC

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Abstract: This paper reports the results of the study of the microstructural and mechanical properties of metal matrix composites (MMC) based on Al-Si matrix alloy and silicon carbide particulates. The scope of the study is to investigate the effects of cooling rate and silicon content on the microstructure and the mechanical properties of MMC. Samples were cast in moulds of different temperature in order to identify the effects of the cooling rate while silicon content was varied from 7 to 12.5 % in the matrix material. The conclusion is that the cooling rate has little effect on the properties unless it is taken to the extreme and the effects of the silicon content is no more pronounced than for non-reinforced aluminium.

Keywords: Al-Si alloy, Casting, Metal matrix composite, Microstructure

1. INTRODUCTION

This research project is part of a larger collaboration investigating problems with casting and machining aluminium alloy reinforced with silicon carbide particles, a metal matrix composite (MMC). The aim is to replace several products that are today manufactured from cast iron. Machinability tests identified problems during a turning operation that are associated with the structure of the matrix alloy and the reinforcement.

In all composite materials, it is desirable to have a matrix that is soft and ductile. Several materials are suitable, but aluminium is often chosen because of its affordable price, low weight and good mechanical properties as an alloy. For optimum strength of the composite material; the reinforcement particles should be harder than the matrix; particles should have small grain size and be evenly distributed in the matrix (Callister and Rethwisch, 2008). During solidification of cast material, the chaotic structure of the melt transforms into the ordered structure of the solid. For any alloy containing two or more alloying elements, during solidification a primary dendritic structure forms below the liquidus temperature and an eutectic structure forms during the later phase of solidification, once the material has been cooled below the solidus temperature. Different cooling rates produce different microstructures and properties of the finished product (Ståhl *et al.*, 1997). A faster solidification results in finer grain size and a harder, more brittle material. With faster cooling, there is an increase in the number of nucleation sites, as dendrite growth starts at nucleation sites, there are more dendrites growing at the same time. When the dendrites have grown to a given size they start interfering with each other and prevent further growth. This interference creates the finer structure. When decreasing the cooling rate, for example by increasing the mould temperature, the dendrite structure becomes coarser. A slow cooling gives the grains sufficient time to grow and produces a ductile material with large grains (Kalpkjian and Schmid, 2010; Iyengar, 2013).

The reference material matrix that is commercially produced consists of 9 wt% Si, 0.6 wt% Mg and the remainder aluminium. 21 wt% SiC particles are added to the matrix as a reinforcing components and a wetting agent is used to aid the inclusion. Principally, the addition of silicon to the matrix alloy improves the mechanical properties of the alloy, as does the small amount of magnesium, as well as increasing the casting properties of the alloy. This ideally should provide a combined effect creating a strong alloy, well suited for casting while maintaining a good machinability (Thyberg *et al.*, 1964). During solidification of the alloy, the material divides itself into areas of higher or lower concentration of silicon to obtain thermodynamic stability. It can be observed that grains with a high content of aluminium solidify first and are later surrounded by eutectic mixture of aluminium and silicon as the temperature drops. It is observed that SiC particles are pushed by the primary phase (α-Al) into the last solidification regions, i.e. they tend to amass on the grain boundaries. The SiC particles also work as a barrier preventing grain growth as well as an additional site of nucleation. This typically leads to a decrease of the matrix grain size with increasing amounts of SiC (Gertsberg, 2009). In the production of cast material, the casting process chosen will have a major impact on the material properties. To improve and optimize the properties of materials, it is also important to take into account such technological question as how the process (mould) can dissipate heat from the melt. Examples of factors affecting this are metallurgical treatment, alloy composition and solidification manner.

The aim of the project was to investigate the mechanical and microstructural properties of different matrix alloy compositions and the effects of the cooling rate in the casting process. The project was limited by the choice of matrix alloy and reinforcing particles. The silicon content of the alloy was varied while the proportions of the other constituents remained unchanged.

2. EXPERIMENTAL SETUP

The microstructure and mechanical properties of the components commercially manufactured at the collaborative company were studied to serve as a reference to the result from this project. The commercially produced material is composed of a matrix of aluminium with silicon, magnesium, and the particle reinforcement. The amount of reinforcement particles is 21 wt% SiC. A wetting agent is used to aid the introduction of the particles.

2.1 Varying Cooling Rate

To vary the cooling rate of the composite, samples were cast in moulds preheated to different temperatures. The temperatures chosen for the experiment were room temperature as a lower extreme, 250°C, 350°C, 450°C and 750°C as the upper extreme. The collaborative company (Volvo Cars Corporation) is today casting in moulds being at 350°C; it was therefore chosen as the reference with the temperature adjusted a hundred degrees up and down and complemented with two extreme cases. The moulds for casting the samples were made from solid copper blocks. The mould cavity had the shape of a cylinder with diameter 25 millimetres and a height of 125 millimetres. The bottom of each mould was covered by an austenitic stainless steel plate, to obtain the same linear thermal expansion as the copper and to minimize the cooling rate at the bottom of the sample. Ideally this would lead to a more uniform cooling of the entire sample. The samples for casting were prepared in a top loaded furnace set at 775 º C in a graphite-clay crucible. Most of the materials used in the experiment were re-melted from commercial MMC produced by the contributing company. After a complete melting of the matrix alloy, the melt was agitated with a graphite paddle at 60 rpm for at least 10 minutes. The samples were all cast using gravity casting without shielding gas. To aid the casting, a stainless steel funnel heated to 750 º C was used to centre the liquid in the mould. The pouring time was roughly one to two seconds.

To monitor the temperature gradient, during solidification a thermocouple was placed in the mould about five millimetres from the cavity wall. Another thermocouple was placed in the centre of the mould cavity to be encased in the solidifying metal.

2.2 Varying Silicon Content

The silicon content was adjusted to investigate the effects on the matrix material. This was achieved by adding either aluminium with reduce the silicon content or by adding a commercial pure silicon to reach the desired silicon content. Addition of AA 5083 aluminium alloy was used for altering the matrix composition. Additionally, magnesium, wetting agents and silicon carbide particles were added to obtain the same composition as the reference, except from the silicon content. For the second part of the project, new moulds were manufactured, this time from medium alloy steel, using the same internal dimensions for the cast samples. New moulds were made to further simulate the current conditions at the production facility of the collaborating company. The samples were cast using the same technique as described previously for part one. The moulds were kept at 320 º C.

To prepare the melt, the commercially manufactured products were remelted in the furnace. The above mentioned ingredients were added to the melt to change the composition. The carbide particles were introduced shielded by the nitrogen gas through a Sialon tube to the bottom of the crucible in the course of the stirring action of the graphite paddle. A schematic view of the preparation setup is shown in Fig. 1. This process is a very slow method of adding carbide particles, at roughly 20 grams per hour. Several times during such process, the melt was left to rest without stirring for 30 minutes to aid the inclusion of particles. This process was also accompanied by a large-scale, manual stirring operation to equalize the carbide content of the entire mould.

The different matrix compositions tested are with 7, 9, 11 and 12,5 % silicon by weight. Today the alloy used by the collaborative company contains 9 % silicon. The amount of silicon was adjusted by two percent up and down from the reference in order to identify the influences. As an extra comparison point, the eutectic composition (12.5 wt%) was tested.

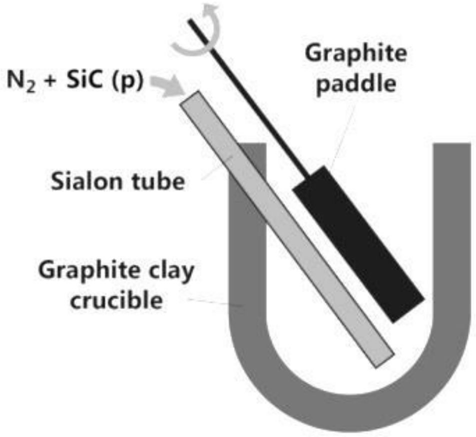


Fig. 1. A schematic view of the SiC introduction technique.

2.3 Material Characterization

The samples from both test series were tested for hardness, tensile strength and examined in an optical and a SEM microscope to determine its microstructure, as well as carbide content and distribution. The samples were tested for hardness with a hardness tester according to the Rockwell B-standard in ten different locations, well distributed over the cross section of the cast sample. For tensile testing, rods were machined from all the samples into a 10 mm diameter testing rod with 45 mm gage length. The rods were tested in a tensile testing rig with a set strain rate of 2 mm/min. The extension of the samples was recorded using an extensometer. To study the microstructure, all samples were ground and polished which was then followed by etching with 10% sodium hydroxide.

To analyse the carbide content, the scanning electron microscopy images of the microstructure of the samples were recorded and later were analysed using with a Leica QWin sowtware package for carbide content. The software results are presented as phase percentage in the cross-sectional area. This value had to be converted to a mass percentage for it to be used as a comparison to the theoretical carbide content used by the participating company during preparation of the material. **Error! Reference source not found.** was used to convert the results. The density for solid silicon carbide was assumed to be 3.21 g/cm^3 (German, 2005).

$$\%_{mass} = \%_{area} * \frac{\rho_{SiC}}{(\%_{SiC}*\rho_{SiC} + (1-\%_{SiC})*\rho_{Al})} \qquad (1)$$

Which can be simplified to:

$$\%_{mass} = \%_{area} * 1.1598$$

3. RESULTS AND DISCUSSION

3.1 Cooling rate

The temperature curves from the casting, used to monitor the cooling rate, are presented in Fig. 2.

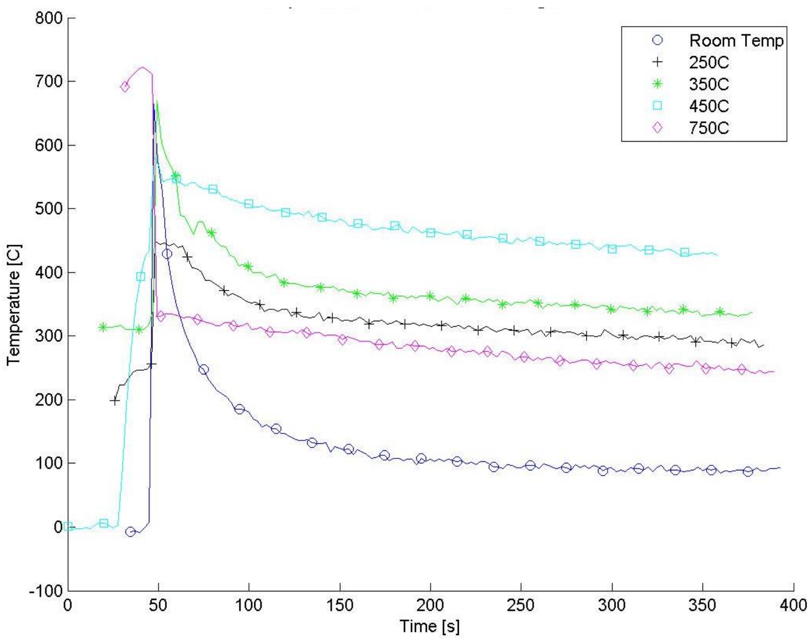


Fig. 2. A comparison between the temperature curves for the different cast samples.

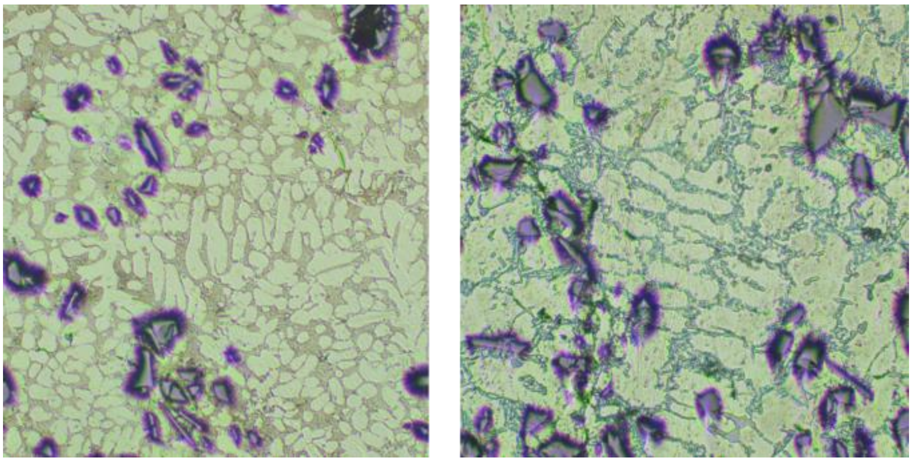

(a) (b)

Fig. 3. Microstructure of the sample cast at room temperature (a) and 350°C (b), both at 200X magnification.

Fig. 3 can be used as an example of how the microstructure changes with the cooling rate. For the experiment with moulds at room temperature, the dendrites have a rounder shape because the dendrites had insufficient time to grow prior to solidification. When increasing the mould temperature to 350 degrees, a more typical cast structure can be seen with significantly larger number of fine dendrites. No distinct increase in the dendrite size was seen even as the mould temperature was further increased. In the experiment with moulds at 350 degrees, it can also be observed that the dendrites are placed more at random. One explanation for this is that the dendrites might have followed the stirring movements in the melt and then solidified.

The results from the tensile tests can be seen in Fig. 4. The results from the 250°C, 350°C and the 450°C were similar. Room temperature stands out with a higher Young's modulus and overall a higher strength. The sample cast at 750°C shows a more ductile behaviour with a much higher elongation at fracture. As with all cast material, the samples produced in our experiment show a high degree of inclusions and porosities in the produced material. This could have resulted in the large spread in data among samples cast at the same temperature that is observed on the Fig. 4. Stress and elongation data for the tensile tests have been calculated as the engineering stresses and strain; that is force divided by original area and elongation divided by original length. Tensile testing for all samples cast a t varying temperature shows no conclusive influence of the cooling rate on the properties of the produced material. A somewhat large uncertainty can be seen in the test data with some of the samples cast at one temperature showing similar properties to samples cast at other temperatures. Looking at the larger-scale tendencies visible in test results, it is possible to sum up that materials cast into higher temperature moulds show a more ductile behaviour with a large elongation at the same stresses. The largest spread within a group of samples was with the samples from 250°C moulds.

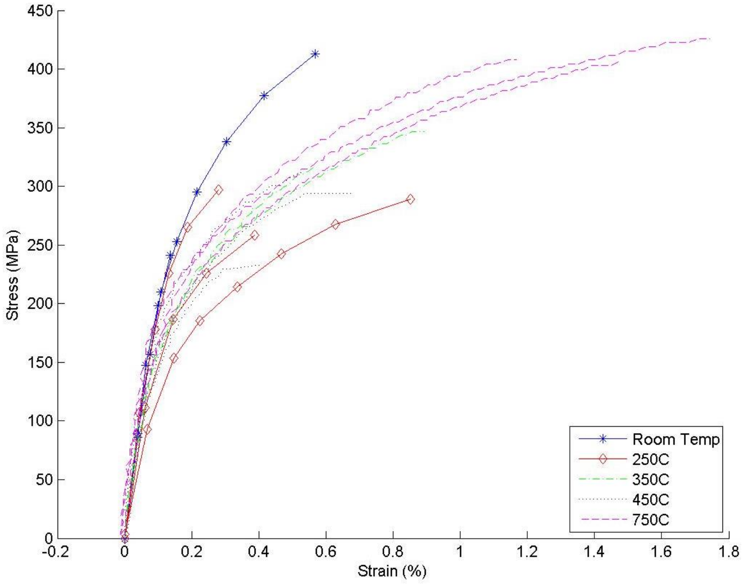


Fig. 4. Tensile strength dependent on cooling rate.

Table 1. Carbide content for samples with varying mould temperature.

	% SiC
Room Temp	10,21
250°C	5,80
350°C	9,55
450°C	10,00

The analysis of the carbide percentage showed a fairly even content for all samples, except for the sample cast at 250°C. This is expected to be the main factor of larger variation of tensile strength data.

When comparing the hardness test to the carbide content of the samples, a connection to the structure could also be seen. All samples except the 250 °C have around 10 wt% SiC and fit the theoretical trend between a slower cooling rate and a lower hardness. The samples cast at 250 °C however, had only approx. 6 wt% SiC, and were also softer than expected (Fig. 5).

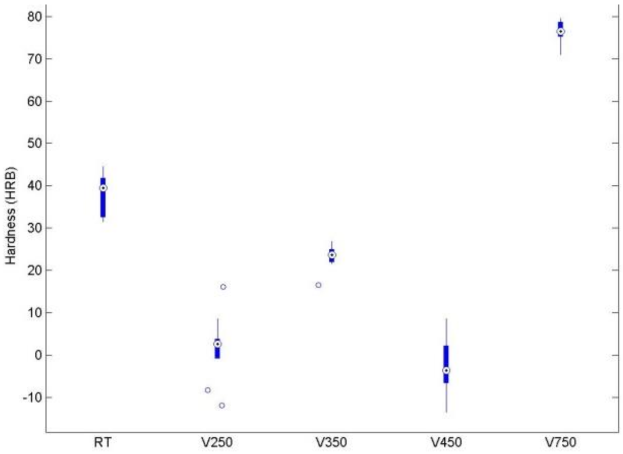
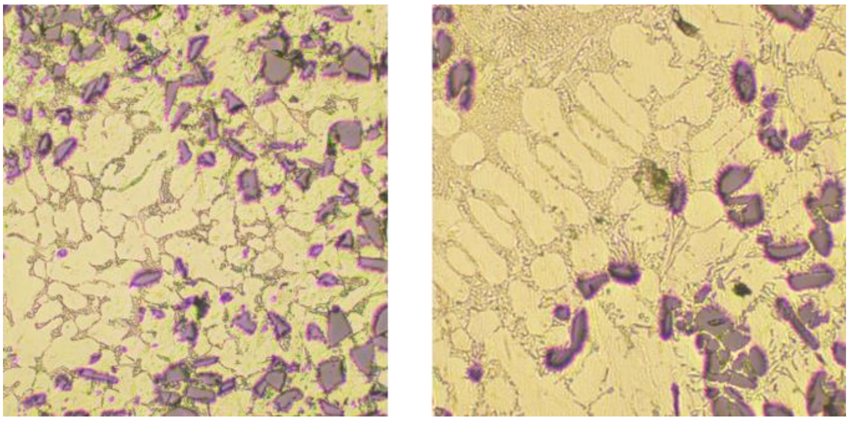


Fig. 5. Hardness for varying mold temperature.

3.2 Silicon content

Fig. 6 shows an example of how the microstructure changes with the increasing silicon in the matrix alloy. With added silicon, there is a change in the structure to a more eutectic lamellar in between the aluminium-rich light areas. With increasing silicon content, a less than optimum distribution of the particles in the matrix is also clear. This is consistent with expectations as the particles are pushed by the solidifying eutectic structure to the grain boundaries. With increasing silicon, there is an increase in the eutectic portion of the matrix, leaving less room for the particles.



(a) (b)

Fig. 6. Microstructure 7 % Si (a) and 11 % Si (b), both at 200x magnification.

Table 2. Carbide content with varying silicon content.

Sample	% SiC
7 % Si	23,02
9 % Si	7,13
11 % Si	10,73
12.5 % Si	30,27

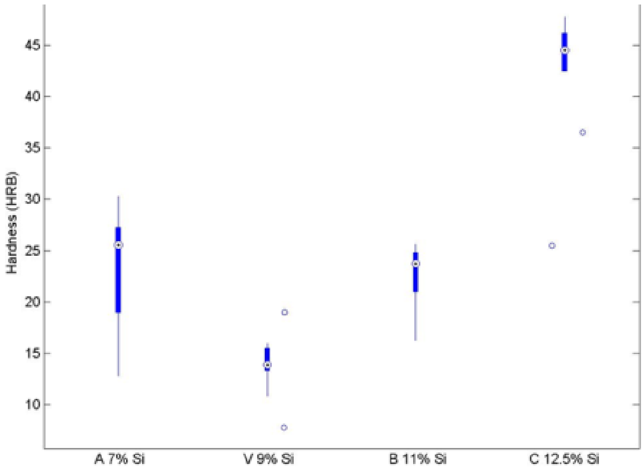


Fig. 7. Hardness for varying silicon content.

Fig. 7 displays the results from the hardness measurements on the samples with varying silicon content in the matrix alloy. The predicted result is that the material with higher silicon content in the matrix should be harder, however the samples with 9 and 11 % are softer than expected, possibly due to the lower carbide content, as seen in Table 2. When adjusted for the missing carbide content, the hardness tests show a general trend consistent with established knowledge, with higher silicon content showing a greater hardness. It is worth noting that the samples with 7 wt% silicon show the largest spread in sample data, which can be explained by the porosity of the sample.

The results from the tensile tests can be seen in Fig. 8. The results partly support the theory that a material with higher silicon contents exhibiting an increase in strength and a lower elongation at fracture, however there is a peak in tensile strength for 9 % silicon.

As an additional test, the different manufacturing methods of the MMC material were compared, all with the same material composition. The methods compared were squeeze casting, gravity casting and forging. The results, Fig. 9, show a major difference between the resulting material properties. The squeeze casting method that is commercially used today by the collaborative company, and forging has been evaluated as a possible production method for the future. Both of these methods have been compared to the material cast during this research project.

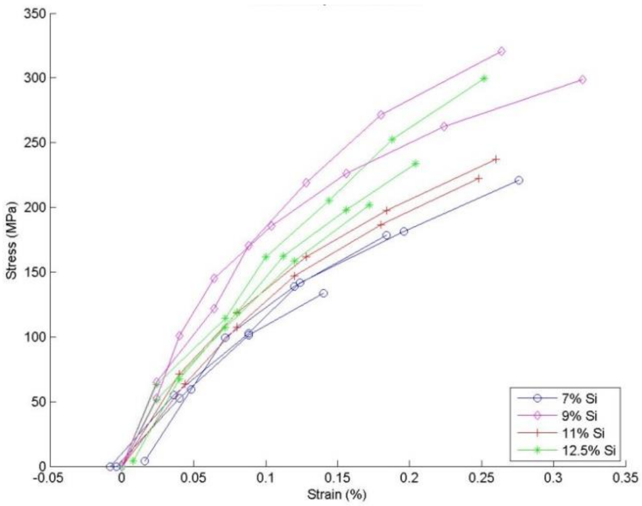


Fig. 8. Tensile strength depending on matrix silicon content.

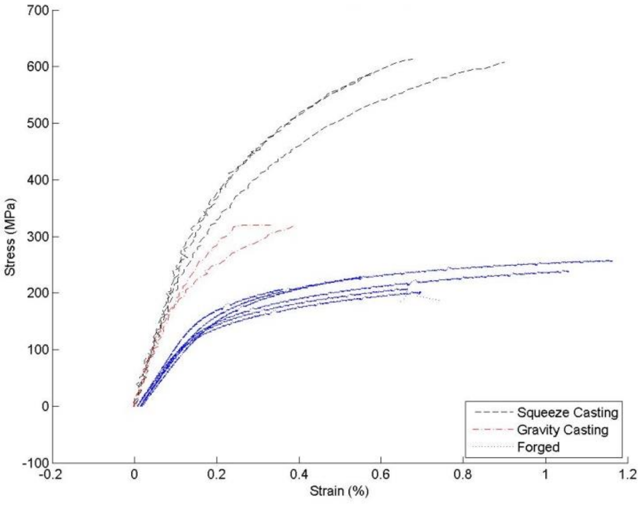


Fig. 9. A comparison material strength manufactured by different methods.

4. CONCLUSIONS

Only a minor difference between the cooling rates, regarding the microstructure and the mechanical properties of the resultant material, were identified. A clear difference is only seen when the solidification time is taken to its extremes, with the samples cast in the moulds at room temperature or moulds heated to 750 ºC. As for the hardness, there is also a not so significant influence from the varying cooling rate.

While varying the silicon content, the matrix material has shown behaviour close to the expectation. With increasing amounts of silicon, a decreasing amounts of dendrites and more lamellar structure was observed. The tensile tests show a clear peak in tensile strength at 9 % silicon, with higher silicon content showing a more brittle behaviour and lesser strength. The method used for the production of the metal matrix composite was found to have a great effect on the mechanical properties of the material. Among the tested methods, squeeze casting provided better strength and ductility; forged material had reduced strength yet high ductility; while gravity casting resulted in a material being excessively brittle.

5. ACKNOWLEDGEMENTS

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