PLANT SCALE INVESTIGATION OF LIQUID ALUMINIUM FILTRATION BY Al_2O_3 AND SIC CERAMIC FOAM FILTERS

Sarina Bao[1], Martin Syvertsen[1], Arne Nordmark[1], Anne Kvithyld[1], Thorvald Engh[2], Merete Tangstad[2]

[1]SINTEF Materials and Chemistry, Trondheim, N-7465, Norway
[2]Norwegian University of Science and Technology, Trondheim, NO-7491, Norway

Keywords: Aluminium, Filtration, Al_2O_3 filter, SiC filter, Ceramic foam

Abstract

Plant scale filtration experiments of 10``×10``×2``, 30PPi Al_2O_3 and SiC industrial filters were carried out. Wetting experiments show that the SiC filter wets better with molten aluminium than Al_2O_3. The assessments by LiMCA II and laser were employed to study the behaviour of the two filters. The Al_2O_3 filter shows improved time dependent behaviour, increasing filtration efficiency, during one hour filtration. This is not the case for the SiC filter. It decays faster than the Al_2O_3 one. The SiC filter requires less pressure drop to infiltrate the metal. The result suggests that the SiC can be a new filter choice in the aluminium industry.

Introduction

The presence of non-metallic inclusions is considered to be one of the most widespread causes of defects encountered in aluminium product: poor machinability, poor surface finish, reduced extrusion die life, cracks, reduced strength, ductility, and fatigue resistance, and pinholes [1]. Liquid metal filtration during the casting process is now a common technology to remove inclusions [2].

Ceramic foam filters (CFF) have an open pore reticulated structure with very high porosity and very high surface area to trap inclusions. The open foam structures are composed of ceramic material, such as alumina, mullite or silica. Alumina is the most common filter material. Ceramic foam filters operate in a deep bed filtration mode where inclusions smaller than the pore openings are retained throughout the cross-section of the filter [3]. Ceramic foam filters are produced by impregnating reticulated polyurethane foam with a ceramic slip, removing the excess slip by squeezing the foam, and then drying and firing the body. The result is a ceramic replica of the original foam [4].

Earlier tests have shown that pure SiC is significantly better wetted by molten aluminium than Al_2O_3 [5]. Improved wetting of aluminium on ceramics probably is an advantage in getting molten metal to infiltrate the filter during priming. Also better wetting should increase the filtration efficiency of inclusions during filtration due to better contact between filter and metal. Thus, SiC can be a filter choice and the use of SiC as an alternative filter material is investigated.

Therefore, plant experiments comparing SiC industrial filters and Al_2O_3 industrial filters have been carried out.

Experimental Procedure

Four filtration experiments were performed with two types of 10"×10"×2", 30ppi filters in the reference center of Hydro Sunndalsøra, Norway: one type high in Al_2O_3 and one high in SiC. These two types of filters were produced in the same line by the same supplier, giving similar porosity and wall thickness.

A top view of the filtration loop is shown in Figure 1. The melting furnace contains 15 tons of aluminium alloy, melted by a burner inside. A porous plug in the furnace bottom injecting argon gas is used to stir the metal. Mechanical stirring from gate 1 is employed, especially after standing a long night. The stirring is to increase the inclusion number in the metal from the melting furnace. The pump is used to control the mass flow of the metal.

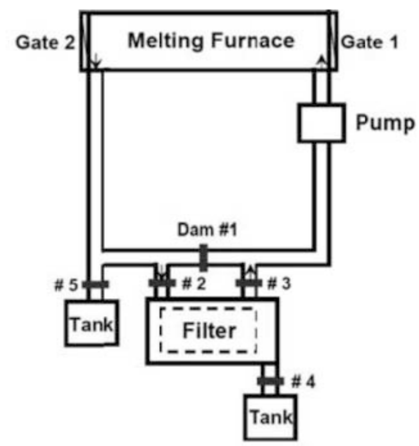

Figure 1 The schematic top view of the filtration loop

Initially metal runs in the launder from the outlet of the melting furnace bypassing the filter bowl, and goes back to the melting furnace. Dam 2 is opened when the LiMCA II reading before the filter shows a relatively stable inclusion level, and metal is led through the preheated filter, fills the lower space of the filter bowl, and goes out. Dam 3 is opened when the right side groove of the filter bowl is full of metal. Then dam 1 is closed to ensure a constant mass flow through the filter. After each experiment, dam 1 is opened and dams 2 and 3 are closed. Dam 4 is opened to drain the metal in the filter bowl after each experiment. Dam 5 is opened to drain the metal in launder at the end of the day. The filter in a filter bowl is preheated by a gas burner in the lid to avoid thermal shock and freezing of the metal when filtration starts.

Two Liquid Metal Cleanliness Analysers (LiMCA) II [6] which give on-line information for inclusion level in k/kg were positioned before and after the filter.

Two lasers positioned before and after the filter bowl give the metal height in the launder in mm. Finally a thermocouple positioned in the launder measures the temperature after the filter in °C.

The Al_2O_3 industrial filter (for aluminium filtration) contains ~85-90% Al_2O_3, ~6% P_2O_5, ~6% SiO_2, and ~1% K_2O+Na_2O, while the SiC industrial filter contains 5-9% Al_2O_3, 58-64% SiC, and 29-33% SiO_2. This SiC filter is normally used for DC casting and continuous casting of copper and copper alloy. The data are given by the supplier. The densities of filter materials were tested by AccuPyc 1330 [1] at SINTEF. Al_2O_3 and SiC industrial filters have average porosity of 88.2% and 85.0%, respectively. The porosity is calculated on the basis of mass and volume measurements using the relation:

$$\boldsymbol{porosity} = \mathbf{1} - \frac{\boldsymbol{the\ bulk\ density}}{\boldsymbol{the\ material\ density}} \quad (1)$$

Using the same procedure, each filtration experiments lasted for 1 hour. The filters were Al_2O_3 in Exps.1 and 3 and SiC in Exps.2 and 4. 3 groups of disk samples for spectrographic analysis from both before and after the filter were taken in each experiment at approximately 0 min, 30 min, and 60 min.

The wettability of the filter materials had been tested in a furnace as described in [7] in a high vacuum of 10^{-8} bar using the same procedure. The received flat filter material was cut into a small tablet and used as the substrate.

Results

The alloy contained approximately 1.00 wt% of Mg, 0.14 wt% of Fe, 0.07 wt% of Si. Other elements are all less than 0.05 wt%. All four experiments have relatively stable inlet and outlet metal compositions, except for a sudden increase at inlet level at 60min in Exp.1 and outlet level at 0 min in Exp.3 for Ca. As an example, Figure 2 shows the composition of the alloying elements in Exp.2 from spectrographic analysis.

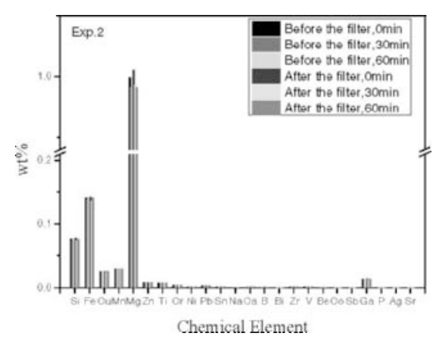

Figure 2 The content of alloying elements before and after the filter in Exp.2 using a SiC filter

Figure 3 to Figure 6 give the time dependent behaviour of the contact angle of pure aluminium on filter materials at higher temperatures. The first degree exponential decay fittings show the contact angles approaching 84° and 44° at 1100°C and 1200°C for the Al_2O_3 industrial filter, and 39° and 28° at 1100°C and 1200°C for the SiC industrial filter.

Due to settling, the inclusion concentration declines with time. Since the rate of reduction in concentration is proportional to the concentration, the concentration will follow an exponential function. It is necessary to take the time dependency of a narrow size range into account when the filtration efficiency is calculated.

Using a narrow size range is more accurate when taking settling into account. Figure 7 to Figure 10 show the number density of inclusions in the size range of 25-30 μm before and after the filter, as well as filtration efficiencies in the 4 experiments.

We can calculate the 68% confidence intervals (dashed lines) for the fitted curves and use those curves to give errors for propagation. The confidence lines give the range where the true value for a given measurement is likely to be given that the fitted curve is of correct form. Approx. 68% of values drawn from a normal (or Gaussian) distribution are within 1 standard deviation away from the average. In this way it is possible to calculate both the filtration efficiency as a function of time for the various size ranges, and also the uncertainty for the filtration efficiency. For more details of this statistical treatment, please refer to [8]. Note that the filtration efficiency is defined as the inclusion number left in the filter divided by the initial inclusion number.

The filtration efficiency for N25-30 in Exp.1 are given from 15% to 32% according to the confidence lines, and from 33% to 11% (decreasing), from 10% to 38%, and from 35% to 51% for Exp.2, Exp.3, and Exp.4, respectively. The same increasing trend for the Al_2O_3 filter and decreasing trend for the SiC filter are observed for inclusions until 40 μm, except 25-30 μm inclusions in Exp.4 (Figure 10). See Table I. Only less than 13.2% of the inclusions are larger than 40 μm in all four experiments, as shown in the last row of Table I, which results in a huge uncertainty (more than 100%) for filtration efficiencies in that range. Thus the results larger than 40 μm are unreliable.

Table I Filtration efficiencies (%) with inclusions until 40μm and % of inclusions larger than 40 μm

Exp.	1	2	3	4
N20-25	21-28	33-9	20-32	46-33
N25-30	15-32	33-11	10-38	35-51
N30-35	4-47	40-11	16-51	52-21
N35-40	1-76	76-0	0-66	70-46
% of inclusions > 40μm				
N40-100	8.2	5.3	11.1	13.2

Note data such as 21-28 means it increases from 12% to 28%; and 33-9 means it decreases from 33% to 9%.

Figure 11 shows the pressure drop with the metal temperature. At the same temperature around 720-730°C, Exp.4 (SiC) has a lower pressure drop than Exp.2 (SiC) due to the warmer bowl. Exp.3 (Al_2O_3) with a higher metal temperature experiences a lower pressure drop than Exp.1 (Al_2O_3). The warmer the metal or the bowl, the less pressure drop is required.

[1] AccuPyc 1330 is a density analyser from Micromeritics, 4356 Communications Dr. Norcross, GA 30093-2901, U.S.A.

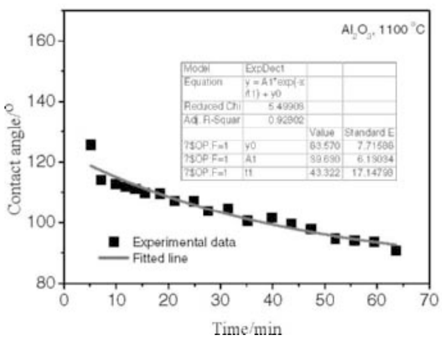

Figure 3 Contact angle at 1100℃ for the Al_2O_3 industrial filter

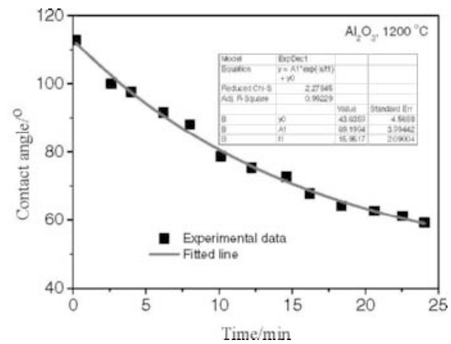

Figure 4 Contact angle at 1200℃ for the Al_2O_3 industrial filter

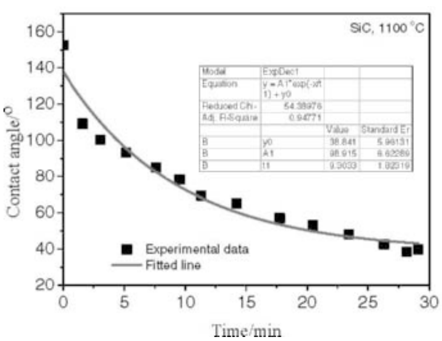

Figure 5 Contact angle at 1200℃ the SiC industrial filter

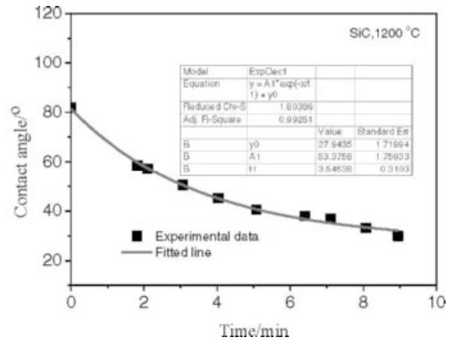

Figure 6 Contact angle at 1200℃ for the SiC industrial filter

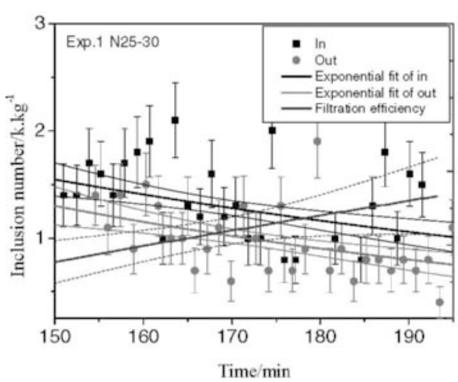

Figure 7 The inclusion number (density) and filtration efficiency for inclusions 25-30 μm in Exp.1 using an Al_2O_3 filter

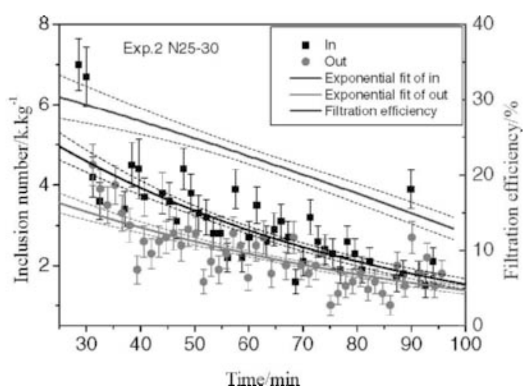

Figure 8 The inclusion number (density) and filtration efficiency for inclusions 25-30 μm in Exp.2 using a SiC filter

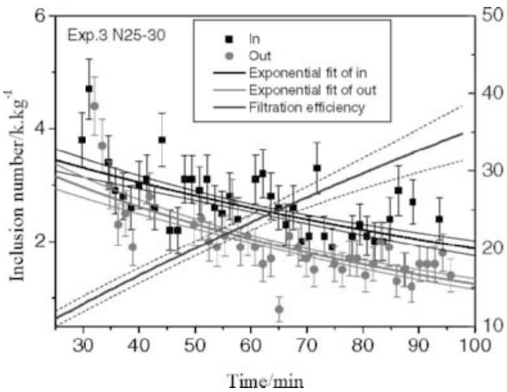

Figure 9 The inclusion number (density) and filtration efficiency for inclusions 25-30 μm in Exp.3 using an Al_2O_3 filter

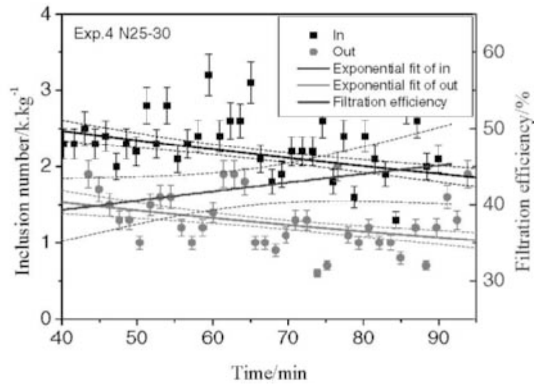

Figure 10 The inclusion number (density) and filtration efficiency for inclusions 25-30 μm in Exp.4 using a SiC filter

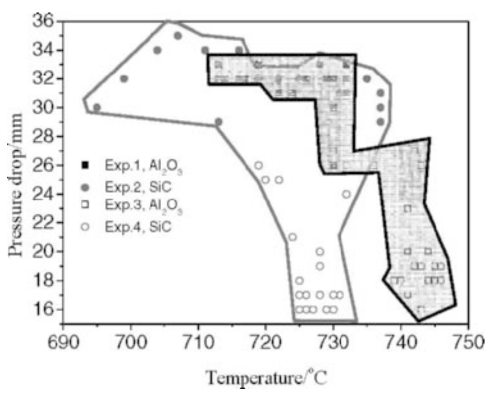

Figure 11 The pressure drop vs. metal temperature

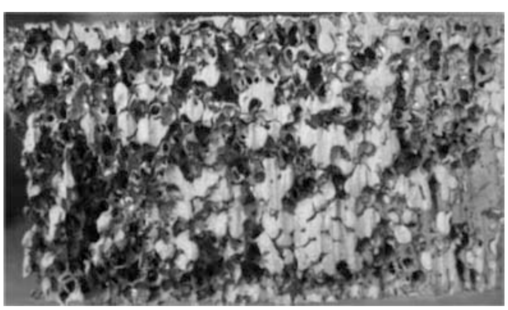

Figure 12 Cross sectional view of the Al_2O_3 spent filter in Exp.3
White parts are filter materials

As examples, the cross sectional views of the spent filters in Exp.3 and Exp.4 were shown in Figure 12 and Figure 13. Filters were partly infiltrated by the metal. Part of the metal may have drained away during solidification due to gravity and the cohesion work of the metal, especially for non-wetting filters. We also obverses that reddish materials cover the exposed Al_2O_3 spent filter. No obvious foreign objects in the SiC filter were found.

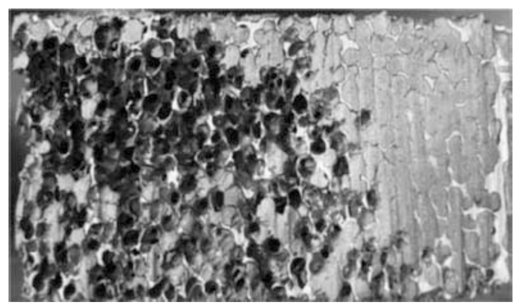

Figure 13 Cross sectional view of the SiC spent filter in Exp.4
Dark parts are filter materials

Discussion

The contact angle is believed to decrease exponentially with time during isothermal holding, approaching a stable angle at the end [5]. Thus, first degree exponential decay fittings in Figure 3 to Figure 6 should closely describe filter behaviour. The final contact angles indicate that the SiC industrial filter has better wetting with aluminium then the Al_2O_3 industrial filter. The Al_2O_3 industrial filter does not wet aluminium, the contact angle >90°, at the casting temperature of 700°C; while the SiC industrial filter might wet aluminum at the same temperature. The wetting becomes poor at lower temperatures.

As indicated in Figure 14, we assume that the metal travels closer to the wall when the filter- Al wettability increases. The inclusions carried in the metal will get more chances to collide with the wall and be captured by it. The liquid prefers rough surface (it gives better wetting) then a flat one. From these points of view, the SiC industrial filter gives a better filtration efficiency.

According to the thermodynamics Mg will react with Al_2O_3 in the filter to $MgAl_2O_4$ spinel at the interface:

$$3Mg(l) + Al_2O_3 = 3MgO(s) + 2Al(l) \quad (2)$$
$$3Mg(l) + 4Al_2O_3 = 3MgAl_2O_4(s) + 2Al(l) \quad (3)$$

The reddish materials in the spent Al_2O_3 filter in Figure 12 may be the products from reactions (2) and (3). Chemical analysis is requested in future work.

The phase diagram of the Al-Si-C system [9] shows that the following ternary quasi-peritectic reaction [10] occurs isothermally at 650°C (923K):

$$3SiC + 4Al(l) = Al_4C_3 + 3[Si] \quad (4)$$

However, no apparent change of Si and Mg content was detected. The total amount of silicon available in the filter is probably too little to give a significant change in the alloy composition.

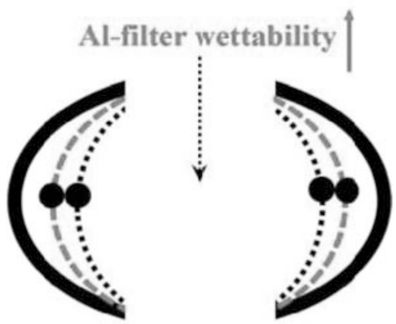

Figure 14 The schematics of Al-filter wettability in a filter cell

The SiC and Al_2O_3 industrial filters did not change the metal composition. Moreover, there is no indication that carbide was formed or entered the metal from the filters. An aluminium alloy with less than 10 at% Si [11] at 700°C could allow Al_4C_3 to be produced according to reaction (4).

However, reaction (4) is probably slow. In the current one hour filtration with 0.10 wt% of Si alloy, no significant increase of Al_4C_3 was measured[2] in Exps.2 and 4 (SiC filters). The SiO_2 and Al_2O_3 components in the SiC industrial filters may slow down the kinetics of reaction (4).

LiMCA results are influenced significantly by micro bubbles. However, no gas bubbling refining unit was involved.

As shown in Table I, Exps.1-3 have similar filtration efficiencies. However, Exp.4 has a higher value. The reason may be the improved wetting for SiC at metal higher temperature [2]. The filtration efficiency tends to increase with time for Al_2O_3 filters in Exps.1 and 3, and to decrease for SiC filters in Exp.2 and 4. The same trends are found for larger inclusions (up to 40 μm). It is more obvious for larger inclusion load. For example, Figure 9 (started with 4k/kg inclusions in Exp.3) has a more obvious increasing trend than Figure 7 (started with 1.5k/kg inclusions in Exp.1) for Al_2O_3 filters.
Figure 11 indicates that Al_2O_3 requires higher temperatures than SiC to achieve the similar pressure drops in overall. For example, Exp.3 (Al_2O_3) and Exp.4 (SiC) show similar pressure drops at around 735-745°C and 720-730°C, respectively. The explanation is that SiC industrial filters need less pressure drop than Al_2O_3 industrial filters to let the metal run through the filter.

Priming is an issue in the aluminium industry, especially when metal freezes with a too low filter or metal temperature. The SiC filter requires less pressure drop to infiltrate the filter.

Conclusions

1. The SiC filter has better wetting with aluminium then the Al_2O_3 filter. The latter one does not wet aluminium at the casting temperature of 700°.
2. Both filters did not influence the metal composition.
3. The Al_2O_3 filter filter shows better time dependent behaviour, with increasing filtration efficiency, during one hour filtration. But the SiC filter does not. It decays instead. This SiC filter is now used in the copper industry. Changes of composition and properties are required for use in aluminium refining.
4. The SiC filter requires less pressure drop to infiltrate the metal. This gives improved priming.

Acknowledgment

This research was carried out as part of the Norwegian Research Council (NRC)-funded BIP Project (No. 179947/I40) Remelting and Inclusion Refining of Aluminium (RIRA). It includes the following partners: Hydro Aluminium AS, SAPA Heat Transfer AB, NTNU and SINTEF. Fundings by the industrial partners and NRC are acknowledged gratefully. Thanks are also owed to Bjørn Rasch at hydro, for support in the industrial experiments.

References

1. S.K. Ås, *Fatigue Life Prediction Of An Aluminium Alloy Automotive Component Using Finite Element Analysis Of Surface Topography*. 2006, Norges teknisk-naturvitenskapelige universitet: Trondheim.
2. S. Bao, *Filtration of aluminium-experiments, wetting and modelling*. PhD Thesis. 2011, Norges teknisk-naturvitenskapelige universitet: Trondheim. p. 204 s.
3. Z. Taslicukur, *Production of ceramic foam filters for molten metal filtration using expanded polystyrene.* Journal of the European Ceramic Society, 2007(27): p. 637.
4. S.F. Ray, S. Pyrotek. *Recent Improvements in The Measurement and Control of Ceramic Foam Filter Quality*. 2001.
5. S. Bao, A. Kvithyld, T.A. Engh, M. Tangstad, *Wettability of Aluminium with SiC and Graphite in Aluminium Filtration.* Light Metals, 2011: p. 775-782.
6. L. Liu, F.H. Samuel, *Assessment of melt cleanliness in A356.2 aluminium casting alloy using the porous disc filtration apparatus technique: Part I Inclusion measurements* Journal of Materials Science, 1997. **32**: p. 5907-5925.
7. S. Bao, K. Tang, A. Kvithyld, M. Tangstad, T.A. Engh, *Wettability of Aluminium on Alumina.* Metallurgical and Materials Transactions B, 2011. **42**(6): p. 1358-1366.
8. S. Bao, M. Syvertsen, B. Rasch, A. Kvithyld, T.A. Engh, M. Tangstad, *Performance Evaluation of Al_2O_3*

[2] It has been measured by PoDFA. PoDFA (Porous Disc Filtration Apparatus) is an off-line method to detect inclusion types and concentration area in mm^2/kg on a polished surface.

and SiC Ceramic Foam with the Use of LiMCA II Data. Metallurgical and Materials Transactions B, 2012. Submitted.

9. J.C. Viala, P. Fortier, J. Bouix, *Stable and Metastable Phase Equilibria in The Chemical Interaction between Aluminium and Silicon Carbide.* Journal of Materials Science, 1990. **25**(3): p. 1842-1850.

10. V. Laurent, D. Chatain, N. Eustathopoulos, *Wettability of SiC by aluminium and Al-Si alloys* Journal of Materials Science, 1987. **22**(1): p. 244-250.

11. D.J. Lloyd, *The solidification microstructure of particulate reinforced aluminium/SiC composites.* Composites Science and Technology, 1989. **35**: p. 159-179.