Effect of hot isostatic pressing on cast A357 aluminium alloy with and without SiC particle reinforcement

A. ZULFIA, H. V. ATKINSON, H. JONES Department of Engineering Materials, Hadfield Building, University of Sheffield, Mappin St., Sheffield, S1 3JD, UK E-mail: h.v.atkinson@sheffield.ac.uk

S. KING

Bodycote HIP Ltd., Carlisle Cl., Sheffield Rd., Sheepbridge, Chesterfield, S41 9ED, UK

The effect of Hot Isostatic Pressing (HIPping) treatments on porosity in the aluminium casting alloy A357 and stir-cast A357/15 vol % SiC particulate Metal Matrix Composite has been investigated. Densitometry and image analysis have been used to determine the apparent percentage porosity. Four HIPping temperature profiles have been investigated (103 MPa pressure) and mechanical testing has been carried out by single notch four point bending. The bend strength was increased after HIPping relative to as-received. The optimum HIPping cycle involved HIPping for a short time in the semi-solid region followed by a sustain below the solidus. © 1999 Kluwer Academic Publishers

1. Introduction

Ceramic particle reinforced Metal Matrix Composites (MMCs) give improved specific stiffness, specific strength, wear resistance and tailorable thermal expansion characteristics [1]. They are already in use for some transportation applications. They can be made by both solid-phase (powder metallurgy) and liquidphase (casting) processes. Casting methods are more economic but give poorer ductility and toughness than solid state routes, mainly because of porosity and particle clusters [2, 3].

Hot Isostatic Pressing (HIPping) is a well-established route for the elimination of porosity from a wide range of metal and non-metal parts including castings. It involves the simultaneous application of a high-pressure (usually inert) gas and an elevated temperature in a specially constructed vessel [4]. The pressure applied is isostatic because it is developed with a gas, so that, at least as a first approximation, no alteration in component geometry occurs. Under such suitable conditions of heat and pressure, internal pores or defects within a solid body collapse and weld up.

Several previous studies have been carried out on HIPping of cast MMCs. Loh *et al.* [5] HIPped two ascast SiC A359-based composites at pressures in the range 100–150 MPa and temperatures in the range 450–550 °C. HIPping increased the ductility but reduced the yield strength drastically. This is in sharp contrast with the results obtained for Al-Li based MMCs [6] and alumina reinforced A356 MMCs [7]. The decrease in strength obtained by Loh *et al.* was thought to be largely

due to matrix softening with the high HIP temperatures they used, and could be restored by age hardening. Pagounis *et al.* [8] found that rigid-rigid contacts and the formation of ceramic particle networks increased the pressure required for densification. There is a critical volume fraction of reinforcement above which continuous networks start to form. Percolation theory suggests this is around 16% [9].

The aim of the work described here was to find optimum conditions for the removal of porosity from a particulate MMC by HIPping and the consequent effect on mechanical properties.

2. Experimental

The materials used (supplied by Norsk Hydro) were based on the aluminium foundry alloy A357 (Al6.7 Si0.3Mg0.2Ti0.1Fe unreinforced, Al5.7Si0.5Mg0.3Ti 0.1Fe reinforced). Unreinforced material was compared with stir-cast MMC having A357 as the matrix and 15 vol % SiC particle size fraction as reinforcement. The average particle size was around 30 μ m.

Four cycles of HIPping were carried out in an Autoclave Engineers laboratory unit at Bodycote HIP Ltd., Chesterfield, UK. Firstly, (HIP cycle 1), HIPping was attempted at 550 °C/103 MPa/2 h, i.e. below the solidus. (The solidus for Al7Si is at 555 °C and the liquidus at 615 °C). The results were not satisfactory, as porosity was only slightly reduced relative to as-received. Secondly, (HIP cycle 2), HIPping was attempted at 575 °C/103 MPa/2 h i.e. between the solidus and the



Figure 1 Optical micrographs (unetched): (a) A357 as-received; (b) A357 after HIP cycle 3 (565 $^{\circ}$ C/103 MPa/15 min followed by 535 $^{\circ}$ C/103 MPa/2 h). Note the higher magnification than in Fig. 1a so as to illustrate the spheroidisation of eutectic silicon; (c) Stir-cast A357/15 vol % SiC as-received; and (d) Stir-cast A357/15 vol % SiC after HIP cycle 3.

liquidus. This was too severe a treatment in the semisolid region, in that the unreinforced specimen distorted and porosity was significantly increased relative to asreceived. In the third cycle, (HIP cycle 3), HIPping was carried out at 565 °C/103 MPa/15 min followed by 535 °C/103 MPa/2 h.

This short burst in the semi-solid region followed by a sustain below the solidus gave significantly reduced porosity relative to as-received. In the final cycle, (HIP cycle 4), which was less effective, there was a longer period in the semi-solid region of $570 \,^{\circ}\text{C}/103 \,\text{MPa}/40 \,\text{min}$ followed by the same sustain of $535 \,^{\circ}\text{C}/103 \,\text{MPa}/2 \,\text{h}$ below the solidus.

The apparent percentage porosity was found by densitometry using Archimedes Principle according to British Standard 5600. The density ρ is given by:

$$\rho = \frac{w_{\rm a}\rho_{\rm L} - w_{\rm L}\rho_{\rm a}}{w_{\rm a} - w_{\rm L}}$$

where w_a is weight in air, w_L weight in liquid, ρ_L the density of the liquid and ρ_a the density of air. The percentage porosity is then found, for the unreinforced by comparing ρ with a handbook theoretical density for A357 (2713 kgm⁻³), and for the composite by comparing with the theoretical density for A357 containing 15 vol % SiC (2788 kgm⁻³). The difficulty here is that the composite may not contain exactly 15 vol % SiC and thus the results show only trends in percentage porosity rather than absolute values. Porosity measurements by image analysis can also be unreliable as smearing of matrix over pores occurs during mechanical polishing. Reassuringly, the trends from the image analysis results in the present work were the same as found by densitometry.

After HIPping, the specimens were heat treated. The T6 condition was achieved by: solution treatment at $530 \,^{\circ}$ C for 17 h, followed by quenching in hot water, then aging at $170 \,^{\circ}$ C for 9 h. Mechanical testing was carried out by four point bending on as-received material and specimens from HIP cycle 1 and HIP cycle 3. The specimen geometry followed that in [10], in the absence, as yet, of standards for four point bending of these materials.

The two side faces of each bend bar were polished to 6 μ m diamond finish prior to testing on a Universal Screw Driven Mayes instrument. Fracture surfaces were examined by Scanning Electron Microscopy (SEM) and sections were taken perpendicular to the fracture surface in order to obtain more information from the sub-fracture surface region.

3. Results and Discussion

Fig. 1a–d show a comparison between the unreinforced and the reinforced material, before and after HIP cycle 3 (illustrated in Fig. 2), which was found to be the



Figure 2 Temperature versus time profile for HIP cycle 3. The solidus is at 555 $^{\circ}$ C and the liquidus for A357 at 615 $^{\circ}$ C.



(a)



Figure 3 (a) Optical micrograph of stir-cast A357/15 vol % SiC after HIP cycle 2 (575 $^{\circ}$ C/103 MPa/2 h). (b) Back scattered scanning electron micrograph of the same specimen as in Fig. 3a. The white phase between the SiC particles contains Si-Fe-Cu and the grey phase silicon.



Figure 4 Apparent porosity values (a) from densitometry and (b) from image analysis.

optimum in terms of reduction in porosity. Fig. 1a shows evident porosity as-received, particularly in the interdendritic regions. After HIPping, the interdendritic eutectic silicon has spheroidised but there is no indication of new phases, in contrast with the more severe treatment in the semi-solid region in which the specimen distorted (HIP cycle 2). For HIP cycle 2, globules of material which was liquid in the semi-solid region are evident in the grains and contain particles of silicon and Mg-Si-Fe phases when analysed by Energy Dispersive Spectrometry (EDS) in the SEM. The interdendritic material contains Chinese script phases based on Al-Si-Fe-Mg, along with magnesium silicide and Si.

As-received MMC is shown in Fig. 1c. Where porosity is present it is almost always between SiC particles. After HIP cycle 3, there is reduced porosity (Fig. 1d). The extent of SiC clustering is similar to that in the asreceived state. The microstructure after the most severe HIPping treatment (HIP cycle 2) is shown in Fig. 3a and b. The SiC is severely clustered and there is gross porosity. Secondary phases between the SiC particles include silicon particles and a phase based on Si-Fe-Cu. There is also a question as to whether any aluminium carbide has been formed. This could not be analysed for in the SEM available and is usually sought by TEM. It is notable that some SiC particles have fractured.

The apparent porosity levels from densitometry and from image analysis are shown in Fig. 4a and b. The error on the results from densitometry is between 5 and 10%. As commented earlier, the results from image analysis may underestimate the percentage porosity because of smearing during polishing. The trends from image analysis are however, consistent with those from densitometry. HIP cycle 2 gives the highest porosity levels and HIP cycle 3 the optimum for reinforced material. For unreinforced material, HIP cycle 4 produces the lowest porosity but the difference from HIP cycle 3 is marginal. Pores were not completely removed. This may be because of the presence of some particle networks resisting deformation, as the volume fraction of



Figure 5 Bend yield strength for A357 and A357/15 vol % SiC before and after HIPping and T6 heat treatment.

TABLE I Summary of results of four point bend testing for unreinforced A357

0 1 2 4 0 0 1 6 2		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccc} \pm 10 & 367 \pm 22 \\ \pm 4 & 400 \pm 10 \\ \pm 9 & 385 \pm 20 \\ \pm 10 & 408 \pm 23 \\ \pm 13 & 406 \pm 29 \\ \pm 0 & 468 \pm 21 \end{array}$	
	$\begin{array}{cccc} 00 \pm 1500 & 178 = \\ 00 \pm 3300 & 171 = \\ 00 \pm 3100 & 181 = \\ 00 \pm 4400 & 180 = \\ 00 \pm 3200 & 207 = \\ \end{array}$	$\begin{array}{llllllllllllllllllllllllllllllllllll$

Note: M: Bending moment; σ_y : Bend yield strength; σ_{nom} : Bend nominal strength.

reinforcement is close to the percolation threshold. Alternatively, it could be due to residual hydrogen in the aluminium alloy, which has combined with carbon liberated during the formation of aluminium carbide to give hydrocarbons which are difficult to remove in the HIPping process.

Bend yield strength results increased after HIPping and T6 heat treatment gave a further slight improvement. HIP cycle 3 plus T6 gave the highest strength results for both unreinforced and reinforced material. The unreinforced material had relatively ductile fracture surfaces when examined in the SEM. There is still some ductility evident in the fracture surface of reinforced material after HIP cycle 3. A transverse section through the same specimen revealed relatively few broken SiC particles beneath the fracture surface, whereas after the T6 treatment there were considerably more. The results for bending moment and bend nominal strength are given in detail in Tables I and II. They follow exactly the same trend as those for bend yield strength. The enhancement in yield strength with HIPping of up to 30% is consistent with the enhancement in properties obtained in other HIPping applications.

The results for the reinforced material are consistently lower than for the unreinforced (even taking into account the errors), contrary to expectation. For example, in a survey of 0.2% yield strength results for particulate Al/SiC Metal Matrix Composites in [11], only

TABLE II Summary of results of four point bend testing for reinforced A357/15 vol $\% SiC_p$

Condition	<i>M</i> (N/mm)	$\sigma_y \ (\text{N/mm}^2)$	$\sigma_{\rm nom}~({\rm N/mm^2})$
As-received	32600 ± 880	96±3	214 ± 7
As-received + T6	38100 ± 2200	112 ± 7	252 ± 15
HIPped 1	38100 ± 1700	112 ± 5	251 ± 12
HIPped $1 + T6$	39500 ± 1300	115 ± 4	262 ± 8
HIPped 3	41400 ± 1800	121 ± 5	274 ± 12
HIPped $3 + T6$	43700 ± 3700	128 ± 11	289 ± 24

Note: M: Bending moment; σ_y : Bend yield strength; σ_{nom} : Bend nominal strength.

one value is below that for the unreinforced material, and then only marginally. Our result could be due to brittle phases occurring in conjunction with the reinforcement, or to higher levels of porosity in the reinforced material relative to the unreinforced for the HIP cycles 1 and 3 which were investigated in mechanical testing (see Figs 4 and 5). In addition, when the porosity occurs in the composite, it tends to be present in conjunction with the reinforcement rather than distributed randomly as in the unreinforced material, and it is perhaps this which is decreasing the strength relative to the unreinforced A357. This is an issue which requires further investigation.

4. Conclusions

For A357 reinforced with 15 vol % SiC particle volume fraction, the optimum HIPping treatment, of those investigated, was 565 °C/103 MPa/15 min followed by 535 °C/103 MPa/2 h. This is a short burst in the semisolid region followed by a sustain below the solidus. There was no indication of any new phases being formed with this treatment and porosity was reduced relative to the as-received condition. Optimum HIPping enhanced the bend yield strength, bending moment and bend nomial strength by around 10–30%, levels which are similar to those achieved in other HIPping applications. However, the mechanical properties were consistently lower than for the unreinforced material, contrary to expectation, and this is an issue which requires further investigation.

Acknowledgements

We are grateful to Bodycote HIP for carrying out the HIPping and to Norsk Hydro for the supply of material. We would also like to thank the Indonesian Government for support for A. Zulfia.

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Received 16 September 1997 and accepted 24 February 1999