Materials Science Forum Vols. 416-418 (2003) pp 413-418
Online available since 2003/Feb/15 at www.scientific.net

doi:10.4028/www.scientific.net/MSF.416-418.413

Grain Growth and Mechanical Properties of Spray Formed Al/SiC Composites

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Keywords: grain growth, heat treatment, spray forming, composite.

Abstract. The objective of this work was to evaluate the grain growth behaviour and mechanical properties of metal matrix composites. The material was produced elsewhere by spray forming of an aluminium alloy AA 7475 and co-depositing silicon carbide particles (20 vol%). The microstructure was evaluated in the as received condition and after a series of heat treatments, annealing, ageing and overageing. Optical microscopy was used to characterise the composite microstructure. Hardness measurements and tensile testing assessed the mechanical properties. Accentuated grain growth occurred due to the solution heat treatment and subsequent ageing and overageing did not produce further changes in grain size. The hardness peak was attained after heat treatment of the composite at 150 □C for 10 hours. Ageing the composite at 120 □C for 24 hours lead to higher tensile strength.

Introduction

The attractiveness of metal matrix composites (MMC) has been extensively documented. MMC have been continuously progressing towards more prosaic uses, from aerospace to car industry. These materials show many advantages over monolithic metals, including high specific modulus, high specific strength, enhanced high temperatures properties, low coefficient of thermal expansion, improved wear resistance and toughness properties [1-3].

The detrimental effect of interfacial reactions, at room and elevated temperatures, has accelerated the development of novel processing techniques. Among the various processing routes which are under development to fabricate MMC, spray forming and co-deposition of the reinforcement, offer some advantages due to a number of factors [4]. First, because of the high efficient heat extraction associated to the atomisation, relatively low interface temperatures can be maintained during processing, and hence, deleterious interfacial reactions can be minimised. Second, spray-formed materials have been reported to exhibit characteristics similar to structures produced by rapid solidification, namely, fine microstructure, increased solid solubility, nonequilibrium phases, and the absence of macrosegregation. Third, spray forming and co-deposition are potential processes for near net shape manufacturing.

Spray forming and co-deposition involve the mixing of the reinforcement with the metallic matrix in the phase diagram region, where the matrix contains solid, semisolid and liquid phases. It appears that such approach will inherently avoid the exposition of the reinforcement to high temperatures at long times, with concomitant degradation of the interfacial properties and extensive macrosegregation, usually associated with casting process. Furthermore, this approach eliminates handling of fine reactive metal powders likewise with P/M [5,6].

Material And Methods

Material

The material used in this work was a spray formed (Osprey Process) aluminium AA 7475 (nominal composition in weight %: 6.0 Zn, 2.1 Mg, 1.4 Cu, Al balance) reinforced with silicon carbide particles at 20 % volumetric fraction. The material was supplied as 15 mm diameter extruded bars, at limited amount. The manufacturer did not supply the spray forming and extrusion parameters. MMC samples of the composite AA 7475/SiC were analysed in the as received condition and after a series of heat treatments.

Heat treatments

Table 1 gives a summary of all heat treatments used in this study.

- *Solution.* In order to be amenable to age hardening, the material was solution treated at 520 ± 5 °C for 2 h. The samples (size: 10 mm high by 15 mm diameter) were quenched in water at room temperature. This was carried out aiming to achieve the maximum supersaturation of alloying elements in preparation for subsequent ageing.
- *Ageing.* After solution treatment, samples were aged in a thermostatic oil bath at 120 ± 2 °C for 24 h. The samples were then cooled into water to room temperature.
- *Overageing.* Following ageing, an overageing heat treatment at 150 ± 2 °C for 10 h was carried out and the samples were then cooled into water to room temperature.

Table 1: Summary of the heat treatments used in this work.

heat treatment	condition
solution	520 ± 5 °C for 2 h
ageing	solution + 120 ± 2 °C for 24 h
overageing	solution + ageing + 150 ± 2 °C for 10 h

Microstructural characterisation

The microstructural characterisation was performed by means of optical microscopy - OM. Specimens for OM were cut either by a diamond wheel or by spark machining. Samples taken from the material, before and after heat treatment, were mounted in thermoplastic material and subsequently ground using SiC paper down to grit 800. Then, the samples were mechanically lapped to 1/4 μm diamond finish. Colloidal silica suspension was finally used to polish the surface of the samples.

In order to allow the observation of the grain boundaries by OM, the samples were anodised. The following chemical solution was used to anodise the metallographic samples: 4 vol%. of fluoboric acid in distilled water. The grain size was measured using the mean intercept length method (mean lineal intercept) in the optical microscope.

Brinell hardness and tensile testing

Hardness and tensile testing were undertaken, to characterise the material in both as received condition and after heat treatment. Brinell hardness measurements (load 62.5 kg and steel ball 2.5 mm) were performed for all conditions. Tensile tests measurements were performed for the materials

as received and heat-treated. Tensile testing was carried out according to DIN 50125 [7]. Averages of three specimens of each heat treatment condition were hardness and tensile tested.

Results And Discussion

Grain growth behaviour

The microstructures of the MMC samples as received, solution, aged and overaged are showed in Fig. 1. The grain boundary of the as received material was not revealed by the current used metallographic technique. However some very fine grains or sub-grains with a mean size around 3 μm were delineated. This composite microstructure was obtained by hot extrusion of the composite. According to Srivatson and Lavernia [8], the co-injection of ceramic particles during atomisation leads to an accentuated reduction in composites' grain size, if the parameters of the production process are kept constant. Unfortunately, a correlation between spray forming process parameters and the composite grain size could not be established, once the hot extrusion condition of the as received material was not known.

The solution treated MMC showed a microstructural change, that is, accentuated grain growth (size of 29 ± 6 μm) and the grain boundaries were etched. The solution treatment promoted recrystallization and grain growth of the composite material. It is well known that recrystallization occurs in the presence of a critical level of previous work hardening, below which it does not take place. During recrystallization, the grain orientation changes. The free energy of the material decreases due to the formation of new non-deformed grains. The growth of these new grains occurs at the expense of the deformed structure. Subsequently, the grain boundaries continue to migrate, although more slowly than during grain growth. The process occurs either by migration of original grain boundaries or sub-grains growth. Usually, migration of grain boundaries occurs at approximately a same rate, leading to the formation of equiaxed grains, as it can be seen in the optical micrographs of Figs. 1b to 1d.

One of the consequences of spray forming can be the formation of equiaxed grains. Several authors [8-10] have observed this equiaxial morphology of grains. It has been proposed that the formation of equiaxial grain during spray forming result from three simultaneous process [11]. The first is the fragmentation of the dendrite arms. The second is the multiplication of grains nucleation. Finally, constrained growth. Due to the repeated impacts of the partially solidified droplets, firstly on the surface of the substrate and subsequently among them, an extensive dendrite fragmentation occurs during deposition. It has been proposed that dendrite fragments, the development of liquid convection during the droplets impact and the formation of a great number of solid nuclei, are factors that contribute to the development of equiaxed grains during spray forming.

Some researchers [12] studied the grain size change in Al-Li/SiC_p composites and Al-Li alloys, both produced by spray forming. The grain size of the composites was visibly smaller than those of the monolithic alloy, for the same heat-treatment. The authors suggested that this was due to SiC particles at the grain boundaries. SiC in the grain boundaries promote a decrease in the total free energy of the system due to the decrease of the available grain boundary surface area. Similarly, the grain boundaries movement force (migration) decreases. It can also be observed in micrographs, Figs. 1b to 1d, that the SiC particles are located both inter and intra granularly. Then besides reducing the grain boundary area, the SiC outlines the grains structure. SiC in the grain boundaries causes a decrease in their movement. These facts are also related to the high dislocation density at the Al/SiC interface, due to the difference between thermal expansion coefficient of SiC and aluminium. These are preferential sites for heterogeneous nucleation and leads to solute segregation in these areas, thus reducing grain boundary mobility. The preferential segregation of solute at the interface matrix / reinforcement has been documented by several researches [9,13]. The presence of a fine distribution of porosity in the grain boundary has a similar effect to that of the SiC, reducing the movement of the grain boundaries.

The change of grain size for the solution, aged and overaged MMC are not seen in Figs. 1b to 1d. Quantitatively, solution, aged and overaged presented mean grain sizes of 29 ± 6 μm, 28 ± 6 μm and 32 ± 4 μm, respectively.

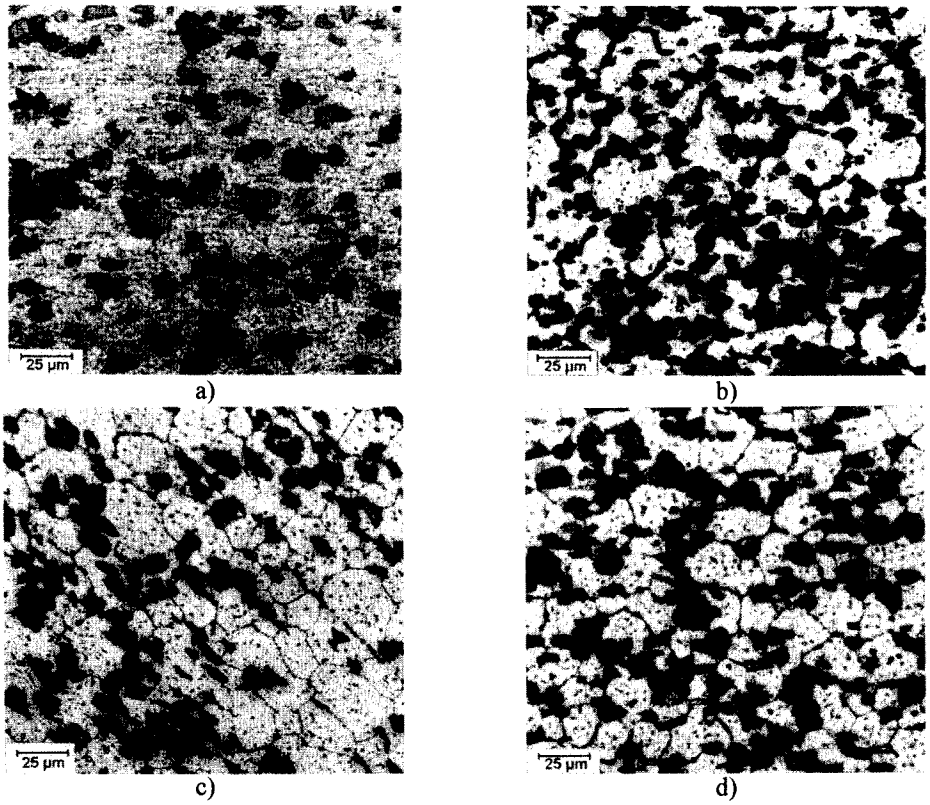

Fig. 1. Optical micrographs of the longitudinal direction of the composite material after anodising. a) As received. b) Solution treated. c) Aged condition. d) Overaged condition.

Mechanical properties

The mechanical properties yield strength ($YS_{0.2}$), ultimate tensile strength (UTS), elongation (e), area reduction (ψ) and Brinell hardness (HB), for the as received composite material and after a series of heat treatments are presented in table 2. There are some plausible explanations for the mechanical strength variation of the composite namely, dislocation density change, precipitates arrangement and grain growth.

Table 2 shows that the values of $YS_{0.2}$ and UTS for the solution treated composite are smaller than that of the material as received. Besides, the hardness of the solution treated composite was smaller comparatively to the received material. Barriers such as precipitates and existent intermetallic second phase particles present in the as received material were dissolved during the solution treatment, promoting a smaller tension against the movement of the dislocations [14-17]. This may have contributed to the decrease in the mechanical strength. Furthermore, in the solution treated composite many dislocations that had been generated during the extrusion of the material were annihilated [14-17]. The dislocations in the cells walls formed during the thermal mechanical process disappeared [14-17]. The ductility increased slightly, and the elongation was uniform, since

there was little variation of the specimen cross-section area. Besides recovery, recrystallization and grain growth also occurred in the composite material after solution treatment. These have promoted the reduction of the mechanical strength in the material. The grain boundaries also offer resistance to dislocation movement. Smaller grain size leads to higher $YS_{0.2}$. Conversely, the solution treated composite, in which there was an accentuated grain growth, showed a diminution of $YS_{0.2}$.

Table 2. Mechanical properties of the composite Al/SiC as received and after various heat treatments.

heat treatment	$YS_{0.2}$ (MPa)	UTS (MPa)	e (%)	ψ (%)	hardness (HB)
as received	340	449	5.4	2.0	133
solution	327	430	6.7	2.0	84
ageing	528	555	1.8	1.0	107
overageing	...	520	2.7	1.6	142

... Not measured.

The UTS and $YS_{0.2}$ of the composite increased considerably after ageing in comparison to the solution treated material. These increments of the UTS and $YS_{0.2}$ are explained by a larger number of precipitates and of Guinier Preston's area (GPZ), which impairs dislocation movement as previously reported [14-17]. Besides, grain growth, which are competitive phenomena. It can also be observed, that hardness increased. Some researchers suggested that the variation of $YS_{0.2}$ in composite materials are due to the difficulty of plastic relaxation at the interface matrix / reinforcement. Consequently, a formation of voids would occur at these interfaces, implying in a decrease of ductility [18].

The overaged composite showed an increase of hardness, without the corresponding improvement of $YS_{0.2}$. In fact, $YS_{0.2}$ of overaged composite decreased in comparison to the aged. Ductility (elongation and area reduction) increased in relation to the aged composite, likely due to the large precipitation of phases and solute impoverishment at the grain boundaries contributing to the decrease of $YS_{0.2}$.

Conclusions

Optical microscopy showed that very fine grains related to the as received composite. The used solution treatment allowed composite recrystallization and the grain boundaries were etched (well-delineated grains). Subsequent heat treatment of ageing and overageing did not produce further changes in grain size.

The mechanical properties of yield and ultimate tensile strength were improved for the composite material aged at 120 □C for 24 hours. This was related to the larger number of precipitates and to Guinier-Preston zones that interferes with dislocation movement. The hardness of the aged composite decreased comparatively to the as received material, due to larger grain sizes.

Acknowledgements

The authors acknowledge the support received from the Brazilian Government- CNPq in the provision of a scholarship to E. G. G. and to Peak - Germany for material supply.

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Advanced Powder Technology III
doi:10.4028/www.scientific.net/MSF.416-418

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