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Microstructural Characterisation of Spray Formed Aluminium Matrix Composite

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Abstract: The material used in this work was produced by spray forming AA 7475 aluminium alloy and co-depositing silicon carbide particles (20% volume fraction). The spray formed composite billets were hot extruded into round bars. The microstructure was examined in the as received and heat treated (annealed, aged, and overaged) conditions by scanning electron microscopy. A comparison was made with published works on similar composite materials. Scanning electron microscopy revealed an extensive Mg_2Si phase precipitation at the Al/SiC interface due probably to Mg segregation from the matrix to the interface, during the heat treatments.

Introduction

Metal matrix composite (MMC) reinforced with particles may have some advantages over monolithic metals alloys, as: low cost, increase of high temperatures properties, low coefficient of thermal expansion, relatively simple processing, and now some can be even commercially manufactured. These materials are obtained by a mechanical mixture of different constituents, that differentiates them from conventionally obtained alloys by solidification or reaction in the solid state, as precluded by phase diagrams [1]. In conventional production processes of metal matrix composites, particulate or fibrous, the microstructure presents dispersed and isolated phases of the reinforcement surrounded by the matrix material [2,3].

Several processes for obtention of metal matrix composite do exist. The processes that are outstanding are those that involve casting (compocasting, squeeze casting, conventional foundry, infiltration, etc.), or powder metallurgy and spray forming (Osprey Process) [1]. There are many variations of processes that often turn them complementary. The processes that involves dispersed reinforcement in the matrix, demand great attention and care regarding quality of the metallurgical bond between matrix and reinforcement, to guarantee appropriate stress transfer. Undesirable reactions in the interface of the composite, which degrade the reinforcement element, need to be avoided under the used processing conditions. Reactions that improve the adhesion between constituents, without however to cause degradation of the reinforcement, are ideally considered and wilfully induced when possible, in the search for a composite with optimised properties.

Under excellent conditions, the microstructure of the material deposited by spray forming

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exhibits little or any individuality evidence. Due to high efficiency of heat extraction and massive number of particles that arrives in the preform, associated to the spray atomisation, low temperature can be maintained during the process and deleterious interfacial reactions can be minimised. It is usual to obtain a uniform microstructure, with fine grains and with little segregation, contrasting strongly with the coarse structure, segregated and dendritic of the materials conventionally cast. Further mechanical work of the spray formed billets, turns the microstructure still more uniform minimising the segregation [4].

The aluminium series 7XXX alloys frequently undergo complex sequence of thermomechanical treatments to attain maximum strength (among many other mechanical properties). This sequence consists of rolling (cold or hot), solution, quenching, natural ageing to room temperature and artificial ageing at higher temperatures This results in a structure with finely dispersed precipitates, which modifies the mechanical properties of the material.

In alloys of the series 7XXX, where Zn is the main alloying element, the phases more common to be found are: $MgZn_2$, $Mg_3Zn_3Al_2Cu$, Al_3Fe and $CuAl_2$ and inclusions of $FeAl_3$, Mg_2Si, besides other possible phase [5].

The objective of this work, was to analyse by scanning electron microscopy, the microstructure of a composite Al/SiC produced by spray forming. The composite been analysed before and after heat treatments aiming to verify the possible phases that are formed during heat treatments. The aluminium matrix alloy and the interface Al/SiC were analysed.

Experimental

The material used in this work was a spray formed (Osprey Process) aluminium AA7475 (nominal composition in weight %: 6.0 Zn, 2.1 Mg, 2.1 Mg, 1.4 Cu, Al balance) reinforced with silicon carbide particles at 20% volumetric fraction. The composite was supplied by Peak-Germany, in the form of round bars, with diameter of 15 mm. The manufacturer did not supply the spray forming and extrusion parameters. The material was supplied in limited amount.

Heat treatments

Solution treatment – In order to be amenable to age hardening, the samples of material samples (size: 7 mm high by 15 mm diameter) were solution heat treat in thermostatic oil bath at 520 ± 5 °C for 2 hours. Soon afterwards these samples were quenched in water at room temperature, aiming to achieve maximum supersaturation of alloying elements in preparation for subsequent ageing. A glass thermometer accurately gauged to 2 degrees monitored the solution temperature.

Ageing and overageing treatments – After solution treatment, samples were aged in thermostatic oil bath at 120 ± 5 °C for 24 hours. The samples were quenched in water at room temperature. Following ageing, the samples undergone an overageing heat treatment at 150 ± 5 °C for 10 hours. Soon afterwards the samples were quenched in water to room temperature. Again a glass thermometer accurately gauged to 2 degrees monitored the ageing and overageing temperatures.

Microstructural characterisation

The microstructural characterisation was performed by means of scanning electronic microscopy

(SEM) and by energy dispersive spectroscopy (EDS). Samples taken from the composite material, before and after heat treatments, were mounted in thermoplastic material and subsequently ground using SiC paper down to grit 1200 Then, the samples were mechanically lapped to 15 µm diamond finish. Colloidal silica suspension was finally used to polish the surface of the samples. The mechanical polishing was done in semiautomatic equipment using diamond pastes 6, 3 and 1 µm, following by final polishing in colloidal silica of 0.25 µm.

Results and discussion

Received composite material

The Fig. 1a) shows general aspects of the microstructure of the composite material in the longitudinal section. It is observed the presence of many second phase particles in the matrix (light grey phase) and some dark areas inside SiC_p agglomerates.

The microstructure of the traverse section of the sample of the composite material in the as received condition is shown in the Fig. 1. It can be observed with more clarity the great amount of second phase particles in light grey tones distributed in the matrix and some adjacent to the SiC_p, see Fig 1c). The dark grey phase of approximate 5 µm size is mainly associated to the SiC_p, see Fig. 1d).

By EDS, it was verified that the light grey phases associated to the SiC_p particles are either rich in Fe and Cu or rich in Zn, Cr and Mg. This implies that this phase could be intermetallics such as $FeAl_3$ and Al_7CuFe_6 or $Al_{12}MgCr$ and $MgZn_2$. The dark phase was rich in Mg and Si and is probably Mg_2Si.

In published works with alloys AA7475 and AA7050, the presence of intermetallics was verified (sizes varying from 5 to 20 µm). It was verified that those phases were Al_7CuFe, Mg_2Si and $FeAl_6$, resulting from the presence of impurities such as Fe and Si. The phase Al_2CuMg was not found in alloys AA7475, however the precipitation of this phase occurred in alloys AA7050 during slow cooling during processing deformation. The non intermetallics precipitation in alloys AA7475, were due to the low amount of Cu in this alloy when compared to alloy AA7050 and to the used homogenising heat treatment [6,7].

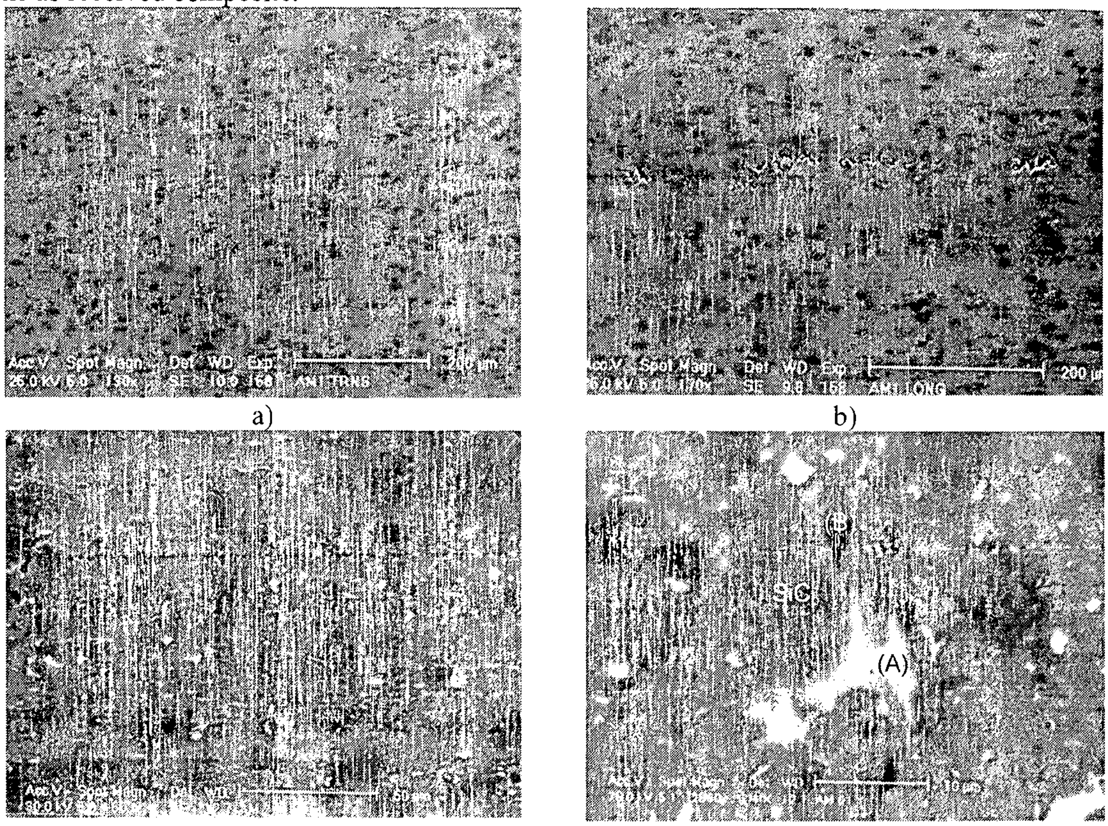
In the present work, regarding the light grey second phase particles distributed in the matrix and distant away from the SiC_p, the EDS revealed that they are rich in Zn, Cr and Mg, similarly to those observed associated to SiC_p. This implies that this could be $Al_{12}MgCr$. Another observed phase was rich in Mg and Zn, implying that this could be the $MgZn_2$ phase.

In other work [8], the microstructure and mechanical properties at room temperature of spray formed Al alloys series 7XXX were investigated in the extruded an peak aged conditions. To elucidate the influence of Zn additions on the microstructure and mechanical behaviour of deposited materials, three alloy compositions were selected: 7075 (96.62 wt. % Zn), 7150 (6.62 wt. % Zn) and 7150X (12.4 wt. % Zn). The results obtained from X-ray and TEM studies revealed the presence of secondary phase particles such as $Mg(Zn,Cu)_2$ in the extruded condition, and $Al_3(Ti,Zr)$ and Mg_2Si in the extruded and peak aged condition for the 7150X alloy. Similar results were obtained for the 7075 alloy, although the latter material (in the peak-aged condition) also revealed the presence of the Al_7Cr phase. Also evident in the spray deposited microstructure was the presence of $MgZn_2$ precipitates, with an average size of 0.25 µm

The presence of porosity can be observed in the microstructure of the as received composite sample, see Fig. 1d). This porosity can be due to the SiC_p agglomerates. A microstructural characteristic frequently associated with the atomised microstructures and spray formed, is the presence of a finite amount of non-interconnected porosity. That porosity depends [9]: of the thermodynamic properties of the material; (b) of the thermodynamic properties of the atomised gas and (c) of the process parameters. As the droplets can be put upon to the previously deposited this can take the formation of cavities in the order of micron size. Regarding the great amount of turbulence due to the gas, the smaller drops solidify during the flight and upon deposition they can impede the presence of liquid phase to fill up the present cavities, causing the formation of irregular pores.

Solution heat treatment

In the SEM micrograph, see Fig. 2, it is observed that the solution heat treated (520 °C for 2 hours) composite showed a microstructure where many intermetallics phases, present in the as received material, were dissolved. The observed remained intermetallics after this solution heat treatment, are probably insoluble intermetallics usually present in the series 7XXX alloys, or intermetallics that has not been dissolved during the present solution heat treatment condition. Probably, the temperature and span of time for the solution have not been enough for to achieve the total dissolution of these primary phases. By EDS analysis of these intermetallics, it was verified the presence of Zn, Cr and Mg, implying that this phase could be $Al_{12}Mg_2Cr$ and $MgZn_2$, similar to the observed in the sample of the as received composite.



a) b)

c) d)

Fig. 1. SEM micrographs of the as received composite material at different magnifications. Fig. 1d) shows the aluminium alloy matrix, the SiC_p and (A) light grey phase, (B) dark grey phase.

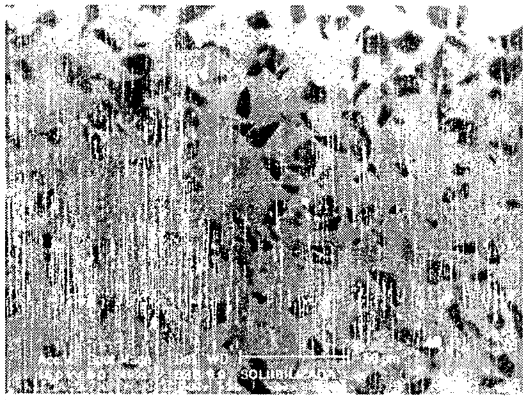


Fig. 2. SEM micrograph of the solution treated composite (520 °C for 2 h).

Ageing heat treatment

The microstructure of the composite after ageing at 120 °C for 24 hours is shown in Fig.3. This SEM micrograph indicates that the amount of intermetallics did not change much in relation to the solution heat-treated material. It can also be observed that the light grey phase associated to the SiC_p is also observed in the aged condition. Probably, these intermetallics are insoluble phases in Al alloy or intermetallics that were not dissolved in the solution heat-treated material. These coarse intermetallics associated to the particles of SiC_p were analysed by EDS, and it was detected the presence of Fe and Cr, implying that this phase could be Al_7Cr_2Fe. It was also observed in the Fig. 3b, dark phases associated to the particles of SiC_p, rich in Mg and Si, implying that this phase could be Mg_2Si, whose size increase was favoured by the segregation of Mg from matrix to the Al/SiC_p interface.

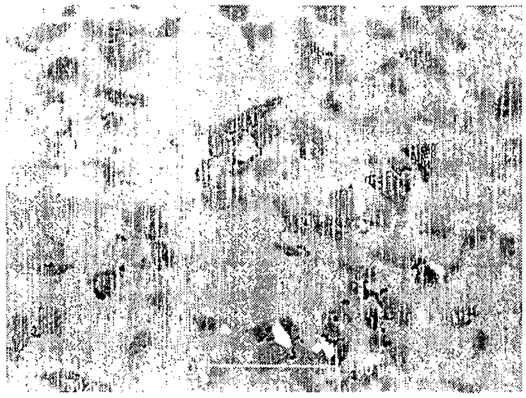
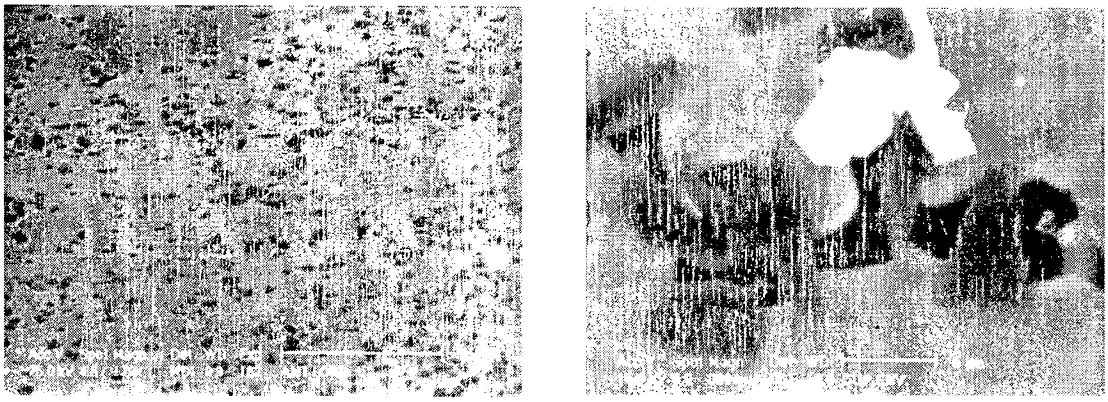


Fig. 3. SEM micrograph of the aged composite material (120 °C for 24 h).

The microstructure of the composite after the overageing heat treatment 150 °C for 10 hours (after solution and ageing), see Fig. 4, shows the presence precipitate in the matrix, light grey phase and dark phases associated to the SiC_p. It was also verified the absence of matrix amid the SiC_p agglomerates. The Fig. 4b a higher magnification of Fig. 4a, evidences a light grey intermetallics associated to a SiC_p, whose EDS obtained in the SEM, showed the presence of Fe and Cr, that could be Al_7Cr_2Fe phase. The EDS from the dark phase associated to the SiC_p, showed mainly the presence of Mg and Si that could be probably a Mg_2Si phase present in the interface. The overageing heat treatment provided an intense diffusion of Mg to the interface, and that together with the Si originating from the dissolution of SiO_2 presents in the surface of the SiC particles and the presence of Al_2O_3 in the interface Al/SiC formed the phase Mg_2Si in the interface Al/SiC. The Mg_2Si is a hardening phase and is usually present at the Al/SiC interface of composites, whose matrix is aluminium alloy of the series 6XXX due to the high amount of Mg in this alloy matrix.



a) b)

Fig. 4. SEM micrograph of the overaged composite material (150 °C for 10 h).

Conclusions

It was verified by energy dispersive spectroscopy (EDS) the presence of some insoluble intermetallics rich in Zn, Cr and Mg, and other rich in Cu and Fe, associated to the SiC_p, before and after heat treatments.

It was observed the presence of the Mg_2Si phase in the Al/SiC interface, after heat treatments, mainly in the overaged condition. This was probably due to segregation and Mg of the matrix to the interface.

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