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Improvement of the bonding interface of a sintered Al $2014-(Ti_5Si_3)_p$ composite by the copper coating of the reinforcement

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ABSTRACT

AA 2014 aluminium-based composites reinforced with (5-20 wt.%) Ti₅Si₃ intermetallic particles, with and without Cu coating, were obtained in a Turbula powder mixer from commercially-available prealloyed powders. Mechanical alloying was used for the deposition of Cu on the surface of the Ti₅Si₃ particles. Compaction of the specimens was performed using a hydraulic press and a floating die. The results show that the liquid formation and phase distribution are modified by the copper coating of the ceramic reinforcement, resulting in changes in the materials microstructure and the mechanical properties. The presence of the reinforcement particles improves densification of the composites. Improved densification was found for the 2014 + Ti₅Si₃ composites. 2014 + Ti₅Si₃-Cu composites exhibit superior mechanical properties compared to the 2014 + Ti₅Si₃ composites.

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1. Introduction

The recent development of metal-matrix composites (MMCs) has been receiving worldwide attention because of their superior strength and stiffness, wear resistance, corrosion fatigue behaviour and creep resistance. The need for new engineering materials with the advancement of modern technology in the areas of aerospace and automotive industries has led to the fast development of these materials.

These composites combine the great strength of ceramics and the ductility from the metallic matrix. The performance of the composite depends on the matrix microstructure, the nature of the ceramic reinforcement and, very critically, on the matrix-reinforcement interface [1,2]. The enhancement of the mechanical properties is not only a function of the volume fraction, size, shape and spatial distribution of the reinforcement, but is also dependent upon how the external applied load is transferred to the reinforcing phase. Stronger adhesion at the particle-matrix interface improves load transfer, increasing the yield strength and stiffness, and delays the onsets of the particle-matrix de-cohesion. [3–5]. The modification of the matrix composition and the chemical composition of the reinforcement has previously been employed to obtain desired interfaces with better properties [2,6]. Coating of reinforcement by chemical vapour deposition (CVD), physical vapour deposition (PVD), thermal spraying or sol gel processes are some of the successfully adopted techniques [7–9].

Among the various matrix materials available, aluminium and its alloys are widely used in the fabrication of MMCs. Aluminium metal-matrix composites have a good combination of stiffness and strength with potential applications in automotive components, particularly for parts where friction is a major issue. Their advantage over ferrous materials is the reduction in weight, leading to lower moment inertia and fuel consumption, and better corrosion resistance.

Silicon carbide, alumina, and zirconia have been added to aluminium metal-matrix composites to study the change in mechanical properties, corrosion and wear resistance. The major fabrication methods used for aluminium metal-matrix composites are stir casting, squeeze casting, compocasting, infiltration, spray deposition, the direct melt oxidation process and powder metallurgy (PM) [2–10]. Composite products manufactured by powder metallurgy (PM) are mainly used as structural components. Cast composites, on the other hand, are mainly considered as replacements for conventional materials used in wear resistant applications.

The mechanical properties of cast composites suffer from some drawbacks, such as the non-uniform distribution of reinforcement, undesirable chemical reactions and poor adhesion between the reinforcement and the matrix [11–13]. The low sintering temperature is very beneficial to the prevention of undesirable chemical reactions at the interface. The breakthrough of MMCs into large volume markets, such as the automotive and aerospace industries,





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is dependant on achieving the mechanical properties of PM materials at cost composites prices.

Different strategies have been attempted to improve the bonding between the matrix and the reinforcement. Mechanical alloying provides improves the microstructure by reducing the reinforcement size, refining the matrix microstructure and increases the adhesion between the reinforcement and the matrix [14–17]. Mechanical alloying has also been employed to increase the lattice strain that creates microstructural defects, such as vacancies and dislocations, to promote diffusion [18]. In this way, powder metallurgy can help ameliorate the interface problems of undesirable chemical reactions and the degradation of the reinforcement. Mechanical bonding between the matrix and the reinforcement is improved by the enhancement of diffusion and the formation of a liquid phase by the main alloying elements of the alloy. This way, the possible lack of wettability with the matrix is avoided.

The objective of this work is to study an aluminium-based metal-matrix composite reinforced with Ti_5Si_3 particles, with and without Cu coating. It includes a brief analysis of the interface formed, its chemical composition, properties and how this interface nature can affect the sintering of the material and the mechanical behaviour of the consolidated composite. Ti_5Si_3 is known to have better mechanical properties at elevated temperatures than ceramics or conventional superalloys [19].

Moreover, it is expected to impede the motion of dislocations and to control the grain size growth of the Al matrix. Cu was added to the system with the goal of increasing the sinterability of the composite. The role of Cu in promoting densification of Ti_5Si_3 has been confirmed in earlier works [20–22].

2. Experimental procedure

2.1. Production of powders and composite materials

Composite materials were prepared by blending and homogenizing a commercially-available prealloyed powder (AA 2014), Ti_5Si_3 and mechanically alloyed Ti_5Si_3 -Cu as the particle-reinforcements for 30 min in a Turbula powder mixer. The powders' composition is indicated in Table 1.

The Ti₅Si₃ ceramic reinforcement particles (dp50 = 25 μ m) were covered with copper (5 wt.%) by mechanical alloying (MA) and studied before mixing. The coated Ti₅Si₃ powder revealed homogeneous and well adhered Cu coatings on the Ti₅Si₃ particles. The milling was performed in a planetary ball mill with hardened steel container and balls, under an argon atmosphere. The ball to powder weight ratio was 20:1 and the rotation speed was 300 rpm. Milling was performed for up to 10 h. Ti₅Si₃ and MA Ti₅Si₃-Cu powders were added to 2014 prealloyed powder in 5%, 10%, 15% and 20% (wt.%) quantities.

2.2. Consolidation of samples

The specimens were compacted using a hydraulic press and a floating die to fit MPIF standard 41 (ISO 3325:1996). The compaction

Table 1

Powder	Source	Size fraction (µm)	Element	%
AA2014	Aluminium powder company	<75	Cu Mg Si	4.4 0.4 0.7 Bal
MA Ti ₅ Si ₃	Cerac	<44	Ti	99.5
Cu	Cerac	<74	Si Cu	99.5

pressure was 300 MPa. Delubrication and sintering occurred in a vertical furnace with a high purity N₂ atmosphere. The heating rate was 5 °C min⁻¹ up to 300 °C. The temperature was maintained for 30 min for dewaxing. The samples were heated again at 5 °C min⁻¹ up to 590 °C. This temperature was maintained for 60 min followed by a water quench to study the liquid phase distribution.

2.3. Characterization

The sintered density was determined using the Archimedeś method, following the standard ISO 327:1985. The densification, ψ , was calculated to determine the amount of shrinkage or expansion according to MPIF 42 and the following expression:

$$\psi = \frac{\rho_s - \rho_g}{\rho_t - \rho_g} \tag{1}$$

where ρ_s , ρ_g and ρ_t are the sintered, green and theoretical density, respectively. A positive value of ψ indicates shrinkage; ψ approaches unity as full density is attained. Hardness was evaluated by Vickers indentation with a 30 kg load (MPIF 51). Bending strength was carried out by the three-point test with a loading rate of 1 mm/min, following the MPIF standard 41 (ISO 3325:1996). A complete microstructural study was made by scanning electron microscopy (SEM), DRX and EDX.

3. Results and discussion

3.1. Densification

As explained above, the matrix-reinforced interface is critical to the performance of metal-matrix composites. The interface between the matrix and the reinforcement determines the final properties of the composite [1–5]. The density obtained for the different composites are shown in Fig. 1. It can be observed the green density of 2014 + MA Ti₅Si₃-Cu composites after the pressing step is closer to the theoretical density than the 2014 + Ti₅Si₃ one. The green density values obtained are similar for both series of materials when small quantities of reinforcement are added. However, the addition of copper to this hard phase increases the reinforcement's elastic and plastic deformation, raising the green density when 15% and 20% of MA Ti₅Si₃-Cu is added. The copper ductility helps during the pressing step, increasing global deformation of the composite material, matrix and reinforcement when compared to the uncoated ceramic reinforcement. There is very little deformation in the uncoated ceramic samples. The sintered density of the uncoated reinforcement composite materials is not dependant on reinforcement content over a 5% (Fig. 1a). However, a continuous increase of sintered density is attained from the 5-15% of additions when the reinforcement is coated (Fig. 1b). The best result is obtained for the 15% sample. Good sintering behaviour combined with the higher green densities shows that the coated samples are superior to the uncoasted reinforcement. However, the densification values in Fig. 2 show that there is swelling during the sintering step. Densification values above zero for each composite studied are the main result obtained from Fig. 2. These positive values indicate composite shrinkage during the sintering step. The aluminium alloy selected in this work belongs to the 2xxx series. The sintering causes the formation of a liquid from the melting of an Al-Al₂Cu(θ) eutectic. The spreading of the sintering liquid phase causes swelling during the first part of the cycle. With the diffusion of the alloying elements within the aluminium, densification and shrinkage take place and positive ψ values are obtained. Comparing both composites series with the optimal reinforcement addition (10–15%), the $2014 + Ti_5Si_3$ composites have the higher densification values.



Fig. 1. Green, sintered and theoretical density for (a) 2014 + Ti₅Si₃ and (b) 2014 + MA Ti₅Si₃-Cu composites.



Fig. 2. Composites densification values for different amounts of reinforcement.

For the MA Ti₅Si₃–Cu composites, despite the higher content of reinforcement, higher densification is obtained (10–15%). The presence of the copper coating reduces densification and lower values are obtained. When the reinforcement is coated, composites are supposed to have a higher copper content in solid solution. A higher quantity of the eutectic Al + Al₂Cu(θ) liquid phase is formed from the diffusion between the aluminium matrix and the copper reinforcement coating. This phenomenon could explain the matrix swelling.

Nevertheless, the decrease in densification (ψ) is not drastic enough to reduce the benefit derived from the improved pressing step. Higher sintered density is obtained for the 15% reinforcement addition.

3.2. Mechanical properties

Analyzing the evolution of bending strength and hardness of both series of composites (Fig. 3), it can be observed that higher



Fig. 3. Composites (a) bending strength and (b) hardness.

values are obtained for the mechanically copper coated reinforced composites. Moreover, the results show the improvement in bending strength for the 10% Ti₅Si₃ reinforcement (Fig. 3a). The higher

quantity of copper in the composite has two affects. In one sense, a major quantity of copper means a higher quantity of liquid phase is expected to form, which could diffuse between particles and grain boundaries during the sintering step. The liquid phase usually enhances the rate of interparticle bonding during sintering [23]. On the other hand, the copper content could probably modify the reaction in the interface matrix–reinforcement. As a consequence, in the case of the 2014 + MA Ti_5Si_3 –Cu composites, the copper could help by creating a chemical bond between the ceramic reinforcement and the aluminium matrix [1,2].

The influence of the reinforcement content on the bending strength is more clear for 2014 + MA Ti₅Si₃–Cu composites. For these materials, the differences show that when the reinforcement is increased from 5% to 10% or 15% to 20% the reinforced composites are stronger compared to 2014 + Ti₅Si₃ composites. For the first series, when a 10% of reinforcement is added, a strong increase in the bending strength occurs. This behaviour has been previously observed by the authors when the reinforcement percentage is raised over a content that is specific for each composite [24]. In addition, the presence of higher Cu content in the matrix increases the liquid phase quantity, causing matrix swelling, which can be the reason for the decrease in properties as explain below. In the same way, when 20% of the reinforcement is added, the decrease in bending strength is clear, which might mean that the optimal

reinforcement addition has been exceeded. In the copper samples, there is a large decrease in bending strength, accompanied by a smaller decrease in hardness, in contrasts to the growth in hardness when a 20% of uncoated reinforcement is added to the alloy. For these materials, a slightly higher bending strength is obtained when 10% of Ti_5Si_3 is added [24]. It is clear that the differences in densification for both composites with the same quantity of reinforcement have a strong influence on bending strength and hardness (Figs. 2 and 3).

Analyzing the hardness in more detail (Fig. 3b), it can observed that the values for $2014 + MA Ti_5Si_3$ -Cu are higher for 5-15% of reinforcement content. However, when 20% of uncoated Ti_5Si_3 is added, the hardness is raised over $2014 + MA Ti_5Si_3$ -Cu one.

This trend in mechanical properties, both in bending strength and hardness, is derived from the higher sintered density but is also related to the copper diffusion. The addition of copper aids the material pressing and higher green density values can be reached (Fig. 1). Moreover, it is known that copper diffuses faster in aluminium than aluminium in copper ($D_{Cu in Al}$: $5.01 \times 10^{-13} \text{ m}^2/\text{s}$, $D_{Al in Cu}$: $1.14 \times 10^{-16} \text{ m}^2/\text{s}$) [25]. This diffusion phenomenon increases aluminium hardness by the distortion of the fcc lattice. However, the copper coating, which contributes to the enhancement of the mechanical properties by increasing the amount of copper in the solid solution, also increases the amount of liquid



Fig. 4. Microstructures of (a) 2014 + 5% Ti₅Si₃, (b) 2014 + 10% Ti₅Si₃, (c) 2014 + 15% Ti₅Si₃ and (d) 2014 + 20% Ti₅Si₃.



Fig. 5. 2014 + 10% Ti₅Si₃ microstructure detail.

phase formed during sintering that decreases the densification due to the microstructure swelling.

3.3. Microstructural analysis

The differences observed for both series of composites continue in the microstructure. In Fig. 4 the microstructures of the composites reinforced with Ti_5Si_3 are shown. Different areas in the microstructure can be identified by back scattering scanning electron microscopy (BSE). A zoomed in scan and the chemical composition performed by EDX in each area is shown in Fig. 5. This technique can perfectly distinguished the aluminium matrix, the copper rich zones forming pools inside the aluminium particles and surrounding the grains, the former particles boundaries [26] and the Ti_5Si_3 reinforcement particles. A DRX analysis was carried out after sintering that confirmed that new intermetallic compounds have not been formed (Fig. 6).

Two different reinforcement particles can be found in Fig. 5. One of them presents two colour areas in the same particle. The EDX analysis revealed more Ti and Si than Al and Cu. However, a reinforcement can also be found with a homogeneous colour, in which Ti and Si content is reduced compared to Al and Cu. The results of EDX analysis show higher values compared to Ti. The changes observed in Si and Cu quantities are not as large. The increase in the Al quantity, detected in the reinforcement particles, could be explained by the diffusion of Al atoms from the matrix into the reinforcement particles during the sintering process.

Al is a faster diffuser in Ti than Ti in Al. At room temperature, the diffusivity of Al in Ti is $1.06 \times 10^{-21} \text{ cm}^2 \text{ s}^{-1}$ compared to $2.9 \times 10^{-23} \text{ cm}^2 \text{ s}^{-1}$ for Ti in Al [27]. The equilibrium solubility of Al in Ti is only 11% at room temperature and increases to a maximum of 47.5% at higher temperatures; meanwhile the solubility of Si in Ti is much smaller, with a maximum of 3.75% at high temperature [28].

The microstructures of 2014 + MA Ti₅Si₃-Cu composites (Fig. 7) shows important differences when compared to the 2014 + Ti₅Si₃ composites (Fig. 4). The aluminium matrix, copper rich zones and reinforcement particles can also be identified from the microstructure (Fig. 8). After the sintering step, DRX analysis was also carried out to confirm the absence of new intermetallics compounds (Fig. 9). However, the differences found between Figs. 4 and 7 are significant. One difference is related to the amount of copper rich zones. When the reinforcement has been mechanically alloyed the copper rich zones are more plentiful when compared to 2014 + Ti₅Si₃ ones. These are located in pools, inside particles and in the former particles' boundaries. They are also found surrounding the reinforcement particles, net-shaped, connecting the reinforcement through the aluminium grains and particles boundaries. This phenomenon was expected from the reinforcement copper coating, but this copper is not exclusively surround-



Fig. 6. 2014 + 20% Ti₅Si₃ XRD diffraction pattern.



Fig. 7. Microstructures of (a) 2014 + 5% MA Ti₅Si₃-Cu, (b) 2014 + 10% MA Ti₅Si₃-Cu, (c) 2014 + 15% MA Ti₅Si₃-Cu and (d) 2014 + 20% MA Ti₅Si₃-Cu.



Fig. 8. 2014 + 10% Ti₅Si₃ MA microstructure detail.



Fig. 9. 2014 + 20% MA'ed Ti₅Si₃-Cu XRD diffraction pattern.

ing the former reinforcement particles. It has diffused into the aluminium matrix and formed the liquid phase Al–Al₂Cu(θ), spreading in between the particles and grain boundaries. This phenomenon causes the microstructure swelling that is clearly detected when the composite densification is analysed (Fig. 2).

This homogeneous distribution of copper rich zones surrounding aluminium grains, the former aluminium particles and ceramic reinforcement, could also explain the better mechanical properties shown in this composites series (Fig. 3). A chemical bond is formed between the ceramic and the aluminium matrix aided by copper diffusion. The accumulation of structural defects, such as vacancies, dislocations, local stresses and grain boundaries, after the milling of the reinforcement with copper could result in a significant increase in diffusion and, consequently, in the solid solubility [28]. This increase could explain the higher Cu and Al contents in the reinforcement particles giving rise to a strong bond between the reinforcement and the matrix (Fig. 8).

The nature of the interface has a strong influence over the properties of the metal–matrix composites. In fact, as it was said above, the critical point in the performance of a metal–matrix composite is related to the matrix–reinforcement interface. The bonding can be mechanical or chemical. Mechanical bonding arises from mechanical interlocking between the matrix and the reinforcements in the absence of all chemical sources of bonding. This mechanical union could occur in the 2014 + Ti_5Si_3 composites. The extent of the chemical reaction has a strong influence over the physical and mechanical properties. Further, the reaction products formed during processing may continue to form during service as well, thereby resulting in progressive improvement or degradation of the properties.

Another difference observed in the microstructure is in the porosity level, which decreases when the reinforcement is mechanically alloyed with copper. This decrease is probably due to the higher quantity of liquid phase, which fills the pores during the sintering step. In these microstructures (Fig. 7), the porosity is reduced compared to $2014 + Ti_5Si_3$ ones. All these differences found and the chemical bonding between the interface matrix-reinforcement could explain the mechanical properties improvement shown in Fig. 3.

3.4. Fracture analysis

Fracture surfaces obtained after bending strength tests were analysed to study the differences found related to the matrix and the reinforcement bonding. In Fig. 10 shows an example of the fractography of the composites with 10% of the reinforcement.

The 2014 + Ti_5Si_3 composites fracture takes place in the matrixreinforcement interface. It can be seen how the fracture goes through the surface of the ceramic particles. If matrix-reinforcement bonding is strong enough, the applied load can be transferred to the reinforcement, improving the composite's mechanical properties. However, this link is not as strong as desired and the material breaks under stresses lower than the 2014 + MA Ti_5Si_3 -Cu samples (Fig. 3). In Fig. 11 it can be seen a image of one brittle ceramic surface.

The presence of fissures and cracks can be observed (Fig. 10b) in the reinforcement particles, which usually act as notches, causing the material failure. Consequently, the aluminium matrix could be the only source of the material strength.

Chemical bonding was observed in the 2014 + MA Ti₅Si₃-Cu composites, which could mean a strong link between the aluminium matrix and the ceramic reinforcement. This kind of matrix-reinforcement bonding is strong enough to avoid failure along the ceramic surfaces, promoting the breaking through the ceramic particles. This would explain the fracture surfaces shown in Fig. 10c and d.



Fig. 10. Fracture surfaces from (a) and (b) 2014 + 10% Ti₅Si₃, and (c) and (d) 2014 + 10% MA Ti₅Si₃-Cu composites.



Fig. 11. Detail of 2014 + 10% Ti₅Si₃ fracture surface.

Finally, the analysis of the fracture surfaces reveals the ductile behaviour of all of the samples, which provided by the ductile 2014 matrix alloy.

4. Conclusions

All the composites show shrinkage during sintering and densification is improved with the reinforcement content.

Improved densification was found for the $2014 + Ti_5Si_3$ composites, due to the smaller copper content.

Despite the presence of copper as a coating for the reinforcement that increases the swelling of the composites, the decrease in densification (ψ) is not strong enough to reduce the benefit derived from the improved pressing step. A higher sintered density is attained for 15% of coated reinforcement.

The 2014 + MA Ti₅Si₃-Cu composites exhibit higher mechanical properties compared to the 2014 + Ti₅Si₃ composites. This enhancement could be related to the bonding between the aluminium matrix and the copper reinforcement, promoted by the mechanical alloying process that aids diffusion between the matrix and reinforcement.

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