Room and high temperature tensile behaviour of a P/M 2124/$MoSi_2$ composite at different heat treatment conditions

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Abstract

In the present work, a 2124/15vol%$MoSi_2$ composite was obtained by powder metallurgy. Its microstructure and mechanical properties were investigated at room and at high temperature (up to 200ºC) in conditions T351, T4 and after heat treatments at 495ºC for up to 100 hours. Up to 150ºC, tensile properties of 2124/$MoSi_2$ in T351 resulted similar to those of a ceramic reinforced 2124/SiC composite. Yield stress of the 2124/$MoSi_2$ material, after heating at 495ºC for up to 100 hours, resulted higher than that of the monolith 2124 alloy heated for the same periods. No diffusion reaction phases were formed surrounding the $MoSi_2$ reinforcing particles during such long exposures to high temperature. Only at 100 hours, large plate-like precipitates that contain Al, Cu, Mg and Si appeared. The high thermal stability of this 2124/$MoSi_2$ composite and its good mechanical properties at room and at elevated temperature makes $MoSi_2$ intermetallic a competitor of ceramic reinforcements.

Keywords

Aluminium alloys; Composites; Intermetallic compounds; $MoSi_2$; Powder metallurgy; Mechanical properties.

1. Introduction

During the last few years, aluminium matrix composites reinforced with intermetallic powder particles (AMCIPs) processed by powder metallurgy (P/M) have emerged as a possible substitute for ceramic reinforced composites [1-6]. This is mainly due to the lower abrasiveness of intermetallics compared to ceramics, which would lead to a longer service life of counterfaces in tribological applications and of machining tools. The higher coefficient of thermal expansion of intermetallics than ceramics could also be considered as an advantage when thermal fatigue resistance is required, as the corresponding lower mismatch between Al alloy matrix and reinforcement coefficients would result in less stress concentration at matrix/reinforcement interfaces. Also, recycling of intermetallic reinforced composites is more straightforward than that of ceramic reinforced materials because it is not necessary to make any separation of the components before melting. On the other hand, powder metallurgy has already proved to be a suitable processing route for AMCIPs because it allows a wide combination of Al alloys and intermetallics by controlling diffusion reactions between them [7-9], better than casting routes [2-4], where much higher temperatures are involved.

First studies on P/M AMCIPs were performed on extruded Al powder reinforced with 5 vol. % of gas atomised Ni_3Al particles [10]. This material presented a sound matrix/reinforcement bonding and good wear properties compared to unreinforced Al [6,11], and was thermally stable up to 300°C. For higher treatment temperatures, deleterious diffusion reaction products appeared [7,12], that hindered the use of age hardening Al alloy matrices such as those of the 2xxx and 6xxx Al series. When this type of Al alloys are required, a different intermetallic reinforcement should be found,

that withstand solid solution treatments without catastrophically reacting with Al or any other solute element of the matrix.

In a previous work [12], a PM 2124 aluminium alloy matrix was reinforced with four different intermetallic powder particles: two nickel aluminides, Ni_3Al and NiAl, and two silicides, Cr_3Si and $MoSi_2$, and their tensile properties were studied in tempering conditions T1 and T4, the latter consisting of a solid solution treatment at 495ºC for 30 minutes followed by water quenching and 48 hours of natural ageing. The best mechanical response was obtained with the 2124/$MoSi_2$ composite, that was also the only one that did not present reactivity between matrix and reinforcement during consolidation or during thermal treatment at 495ºC for 30 minutes. This composite also showed similar tensile properties than 2124/SiC age hardened following the same T4 heat treatment.

In this work, the microstructure and mechanical properties of the P/M 2124/15vol%$MoSi_2$ composite has been further investigated. Tensile properties in condition T351 (solid solution treatment at 495ºC for 60 minutes, water quenching and 1.5% stretching) have been studied at room temperature and up to 200ºC and compared to those of a 2124/SiC composite processed by the same P/M route. Tensile behaviour of the 2124/$MoSi_2$ material has also been investigated after heat treatments at 495ºC for up to 100 hours. In this case, results have been compared with those of the monolith P/M 2124 alloy submitted to the same heat treatments. Thermodynamic stability of the 2124/$MoSi_2$ system at solid solution temperature has also been assessed. $MoSi_2$ particles were obtained by self-propagating high-temperature synthesis (SHS).

2. Experimental procedure

Fig. 1 shows schematically the P/M procedure followed to obtain the aluminium matrix composites. The 2124 alloy matrix powder (chemical composition in weight %: Cu = 4.24, Mg = 1.4, Mn = 0.85, Si = 0.03, Fe = 0.06, Zr, Cr and Ti < 0.01 and Al = bal.) was prepared by argon atomisation by Alpoco, Sutton Coldfield, UK. The mean particle size of the matrix powder was 27 μm and the maximum size was less than 60 μm, with a spherical morphology typical of gas atomised particles. The $MoSi_2$ intermetallic reinforcement powder was produced by SHS at Fundación INASMET, San Sebastian, Spain, from pure elemental molybdenum particles of 3 to 7 μm and silicon particles of < 20 μm in size. SHS was followed by jet milling of the porous product, that gave rise to a median $MoSi_2$ particle diameter of 5.1 μm. A first batch of $MoSi_2$ powder was obtained sieving the milled $MoSi_2$ powder to < 8 μm in size, a second batch was obtained by also removing the <3 μm in size particles. The shape of the jet-milled particles was irregular. The 2124 matrix powders were mechanically blended with 15 vol.% $MoSi_2$ using a Turbula® mixer.

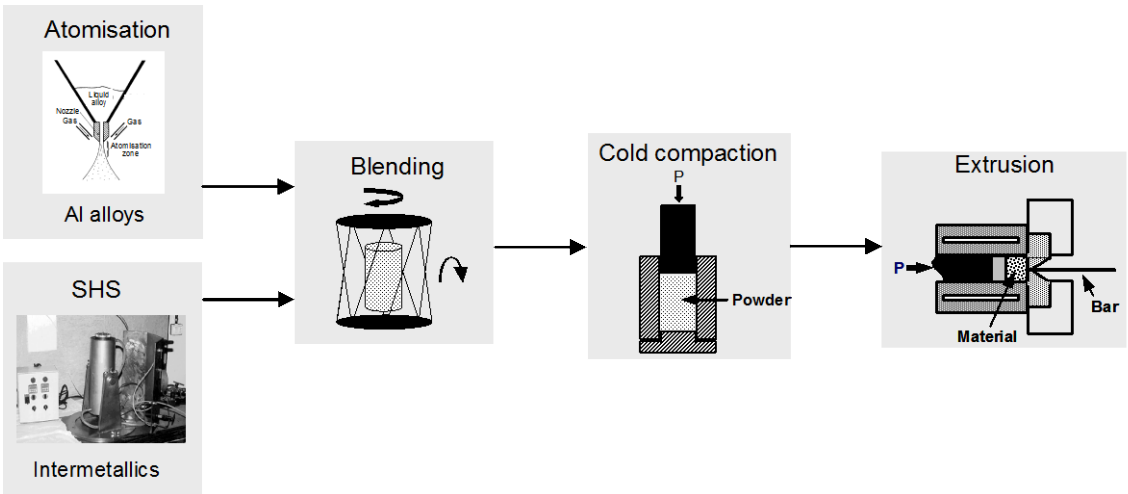


Fig. 1. Schematic representation of the P/M process

The blends of powders were uniaxially cold compacted at a rate of 90 MPa/minute up to 300 MPa with this pressure being sustained for 3 minutes. The cylindrical green compacts of 40 mm in diameter and 150 mm in length, were then heated for 30 minutes at the extrusion temperature and immediately hot extruded into bars of 8 mm diameter at 450°C, extrusion ratio of 37:1 and velocity of 11.1 mm/s and water quenched. Simultaneously, monolith 2124 and a 2124/15%SiC extruded bars were obtained by the same procedure.

The composite bars were studied as-extruded, i.e. T1 condition, after a solid solution treatment at 495ºC for 30 minutes, water quenching and 48 hours of natural ageing, i.e. T4 condition, and after solid solution treatment at 495ºC for 60 minutes, water quenching and 1.5% stretching, i.e. T351 condition. In addition, the effect of holding time at solid solution temperature for 0.5, 1, 3, 10, 30 and 100 hours, designated as TS treatment, was studied in order to examine the thermal stability of the composite and the interfacial reactions between the 2124 alloy matrix and the $MoSi_2$ reinforcement. All heat treatments were performed in air.

Microstructural characterisation was performed by scanning electron microscopy (SEM). The specimens for SEM observations were prepared by standard metallographic techniques without any chemical etching and were carried out in a JEOL 6500 unit. Microanalysis in the SEM microscope was undertaken using energy dispersive x-ray spectroscopy (EDS). X-ray diffraction was performed using a PHILIPS diffractometer with Cu radiation operated at 45kV and 40 mA.

Cylindrical tensile specimens of 3 mm diameter and 10 mm gauge length were machined from the extruded bars while maintaining the tensile axis parallel to the

extrusion direction. Yield stress (YS), ultimate tensile strength (UTS) and elongation to fracture (εf) were determined at room temperature and up to 200ºC at a strain rate of 10^{-4} s^{-1} employing at least two specimens for each material and condition. Scatter of results was less than 10%. Table 1 shows the processing conditions in which each material was tested.

Table 1.
Materials, reinforcing powder particle size ranges and tensile test conditions. T1: as-extruded; T4: solid solution treatment at 495ºC for 30 minutes, water quenching and 48 hours of natural ageing; T351: solid solution treatment at 495ºC for 60 minutes, water quenching and 1.5% stretching; TS: heat treatments at 495ºC for 0.5, 1, 3, 10, 30 and 100 hours.

Material	Size, μm	Condition
2124/$MoSi_2$	3-8	T1,T4, T351, TS
2124/SiC	<5	T351
2124	-	T351, TS

3. Results

Data on yield strength (YS), ultimate tensile strength (UTS) and elongation to failure (εf) of 2124/$MoSi_2$ in T1 and T4 are shown in Table 2.

Table 2. YS, UTS and εf of 2124/$MoSi_2$ composite.

Condition	T1	T4
YS (MPa)	345	430
UTS (MPa)*	520	610
εf (%)	5	6

*Broken before necking

In order to study thermal stability of the composite at solid solution temperature, specimens were submitted to heat treatments at 495ºC for times varying between 0.5 and 100 hours. The microstructure and tensile properties were characterized after each

treatment. As can be seen in Fig. 2, no diffusion reaction products were detected between matrix and reinforcement that surround the intermetallic $MoSi_2$ particles.

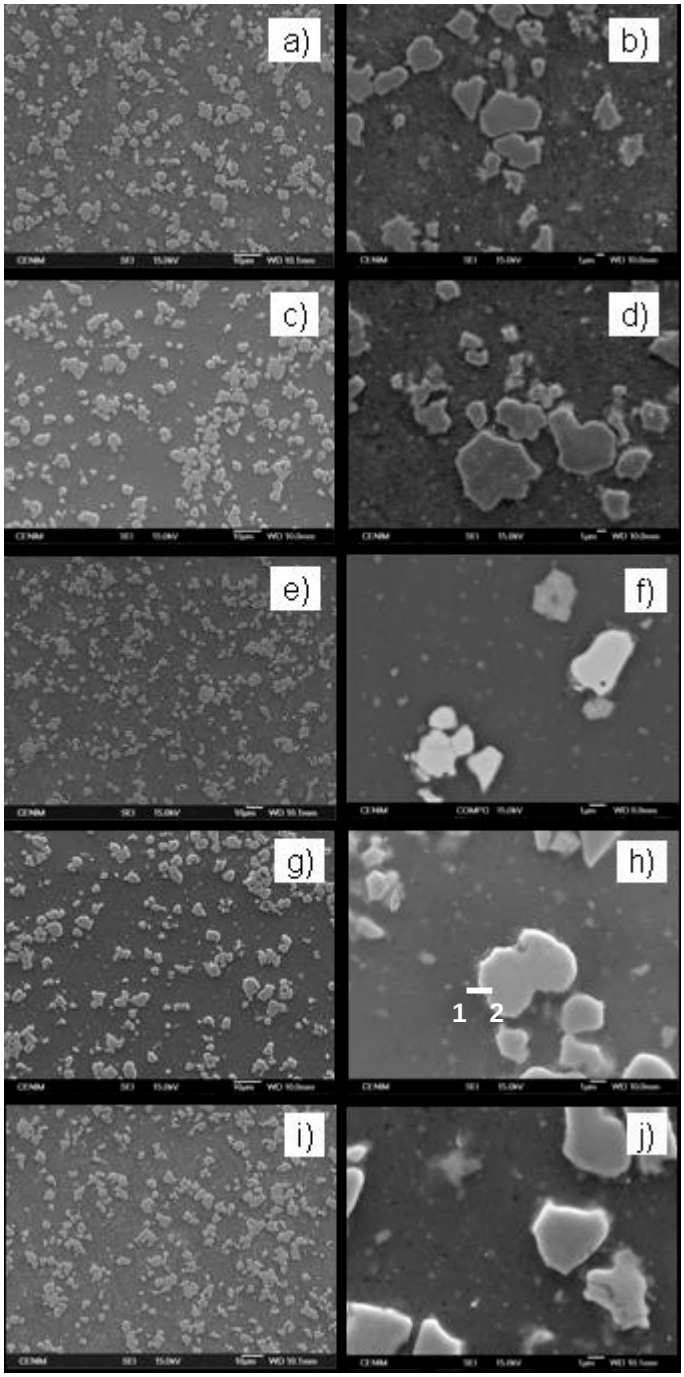
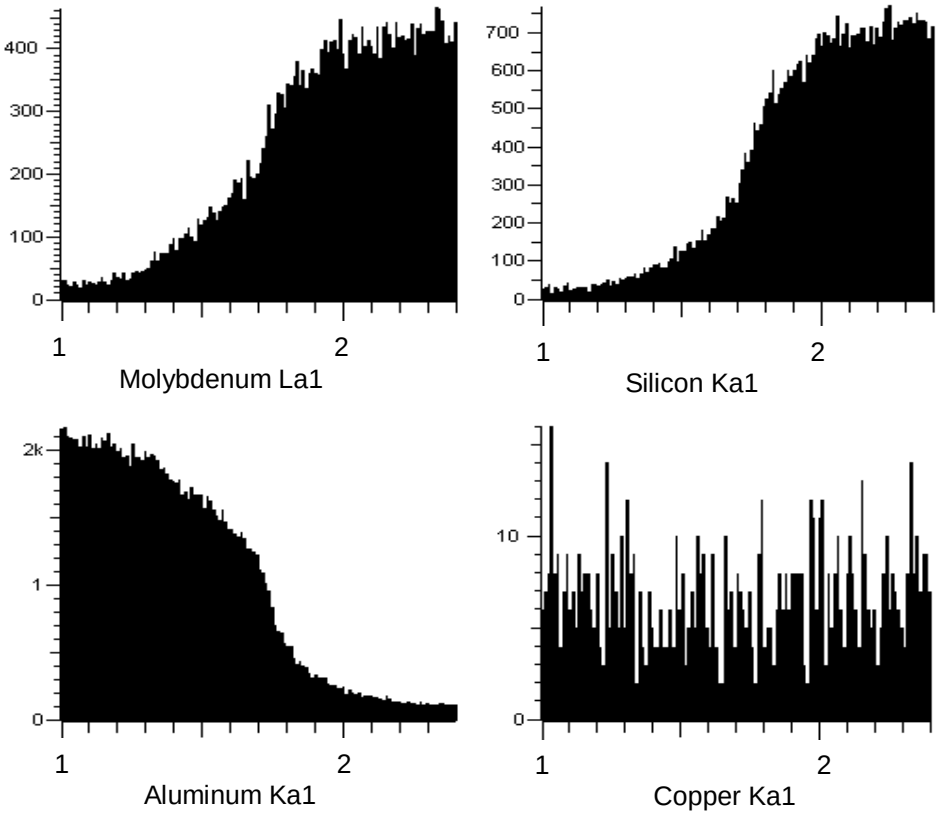


Fig. 2. SEM micrographs showing the microstructure of the 2124/$MoSi_2$ composite after 0.5 hour (a and b), 1 hour (c and d), 10 hours (e and f), 30 hours (g and h) and 100 hours (i and j) of heat treatment at 495ºC.

The absence of diffusion reaction products can be better stated from element line profiles, as those presented in Fig. 3 for the 2124/$MoSi_2$ composite after 30 hours at 495ºC, performed along the white line drawn in Fig. 2(h). Mo and Si follow the same profile, just symmetric to the Al one and Cu profile seems to reflect only background noise. Some accumulation of O and Mg exists at the interface probably forming $MgAlO_3$ or MgO [13]. Oxigen may come from physically and chemically absorbed water on Al powder particle surface [14] or from SiO_2 coating on $MoSi_2$ particles [15].



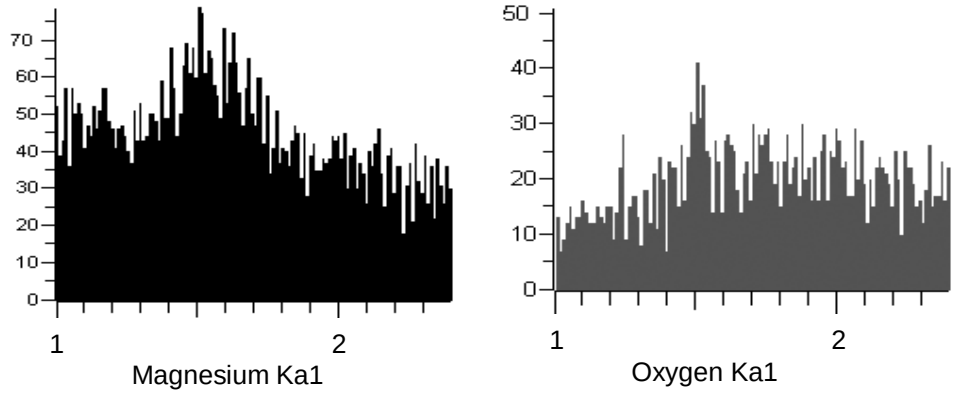


Fig. 3. EDS concentration lines of Mo, Si, Al, Cu, Mg and O at particle/matrix interface of 2124/$MoSi_2$ composite after 30 hours of heat treatment at 495ºC (see Fig. 2(h)).

Fig. 4, shows YS, UTS and εf of 2124/$MoSi_2$ and P/M 2124 alloy as a function of time of heat treatment at 495ºC. In both materials, YS and UTS remain constant up to 30 hours. Only after 100 hours, a significant decrease in YS of 35 and 50 MPa and in UTS of 80 and 55 MPa is observed for the composite and monolith 2124 alloy, respectively. Ductility remains quite constant independently of the time of the heat treatment. Comparison of composite and monolith materials indicates that YS are similar, whereas UTS and εf are higher for the unreinforced alloy. The lower ultimate tensile strength and ductility of the 2124/$MoSi_2$ composite should be related to increasing damage (either by particle fracture or interface decohesion) as plastic deformation progresses, thus decreasing the stress carried by the reinforcing particles.

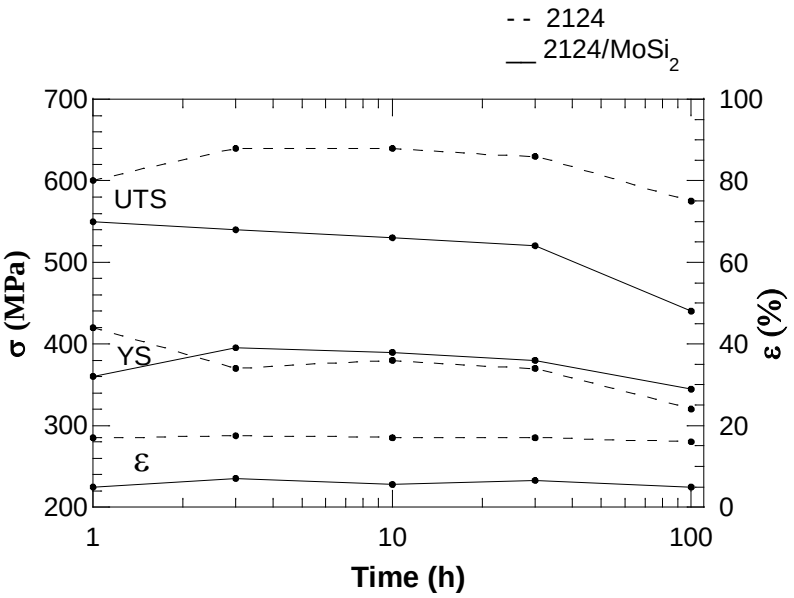


Fig. 4. YS, UTS and εf of 2124 matrix and 2124/$MoSi_2$ composite after submission to heat treatments at 495ºC for 0.5 to 100 hours.

Fracture surface of 2124/$MoSi_2$ tensile specimens were observed by SEM. Fig. 5a and 5b show dimples developed around $MoSi_2$ powder particles, with the corresponding EDS spectra of matrix and $MoSi_2$ particle, Fig. 5c and 5d, in specimens heat treated for 3 hours at 495ºC. After 100 hours of heat treatment at 495ºC, Fig. 6, the existence of large (>30 μm long) plate-like precipitates was detected, Fig. 6b, which clearly have a weak interface with the matrix. Fig. 6c shows a typical spectrum of this phase together with semi quantitative analysis of three plate-like precipitates that contain Al, Mg, Si and Cu. X-ray diffraction patterns of this sample only revealed peaks corresponding to Al and $MoSi_2$. A different aspect was presented by the fracture surface of monolith 2124 alloy heat treated for 100 hours at 495ºC, Fig. 7, where no sign of these precipitates was evident.

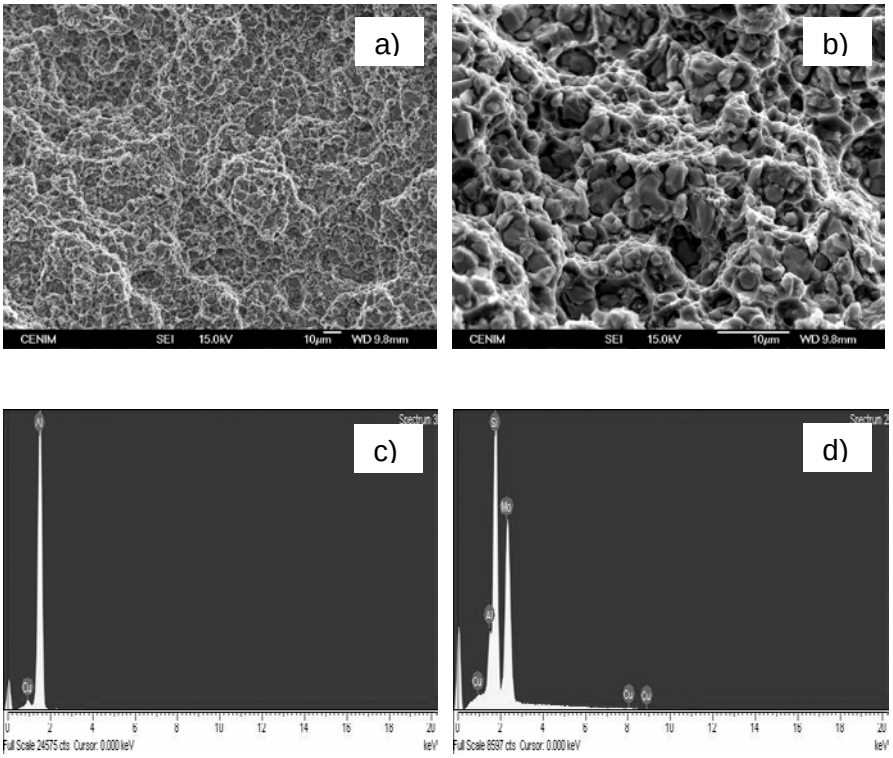
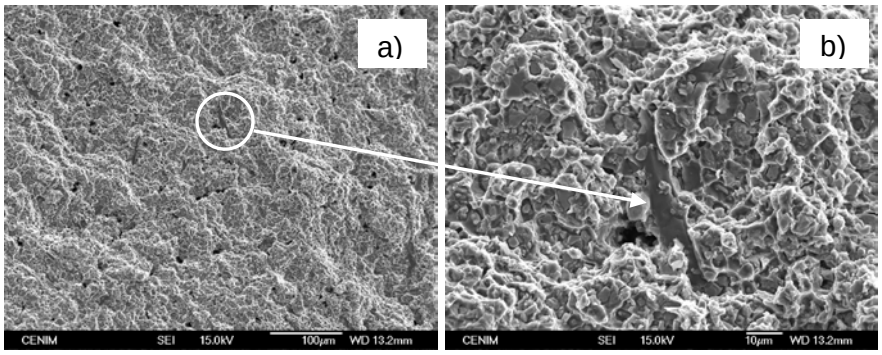


Fig. 5. a) and b) Fracture surface of 2124/$MoSi_2$ heat treated for 3 hours at 495ºC and EDS spectra of c) matrix and d) a $MoSi_2$ intermetallic particle.



c)

	Mg	Al	Si	Cu
Spectrum 1	36.12	21.47	31.72	10.69
Spectrum 2	35.99	22.86	30.61	10.54
Spectrum 3	36.31	20.80	31.96	10.93

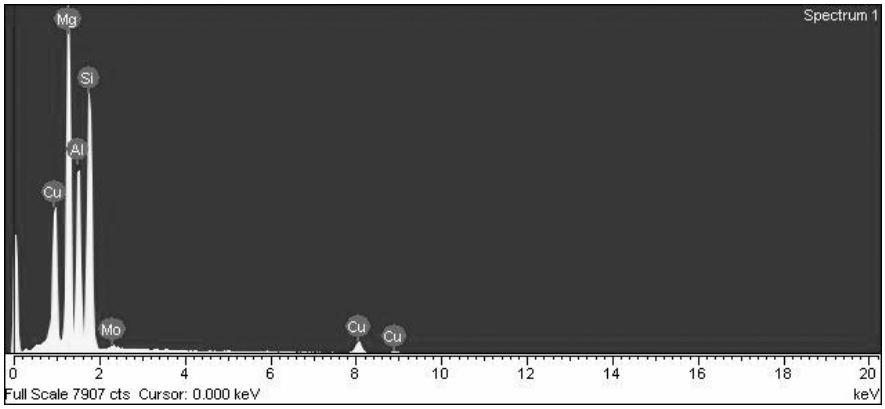


Fig. 6. a) and b) Fracture surface of 2124/$MoSi_2$ heat treated for 100 hours at 495ºC and c) EDS spectra ofthree Mg-Al-Si -Cu-containing precipitates.

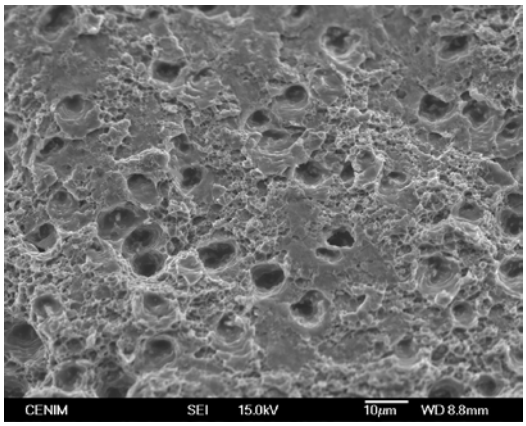


Fig. 7. Fracture surface of 2124 alloy heat treated for 100 hours at 495ºC.

Finally, tensile properties of 2124/$MoSi_2$, 2124/SiC and monolith P/M 2124 alloy in T351 were studied from room temperature up to 200ºC. Fig. 8 shows these results. It can be seen that the intermetallic reinforced composite presents properties similar to the ceramic reinforced one and that up to 150ºC, YS is higher in both cases than that of the unreinforced alloy. On the contrary, UTS and elongation to failure is always higher for the monolith material in the whole temperature range.

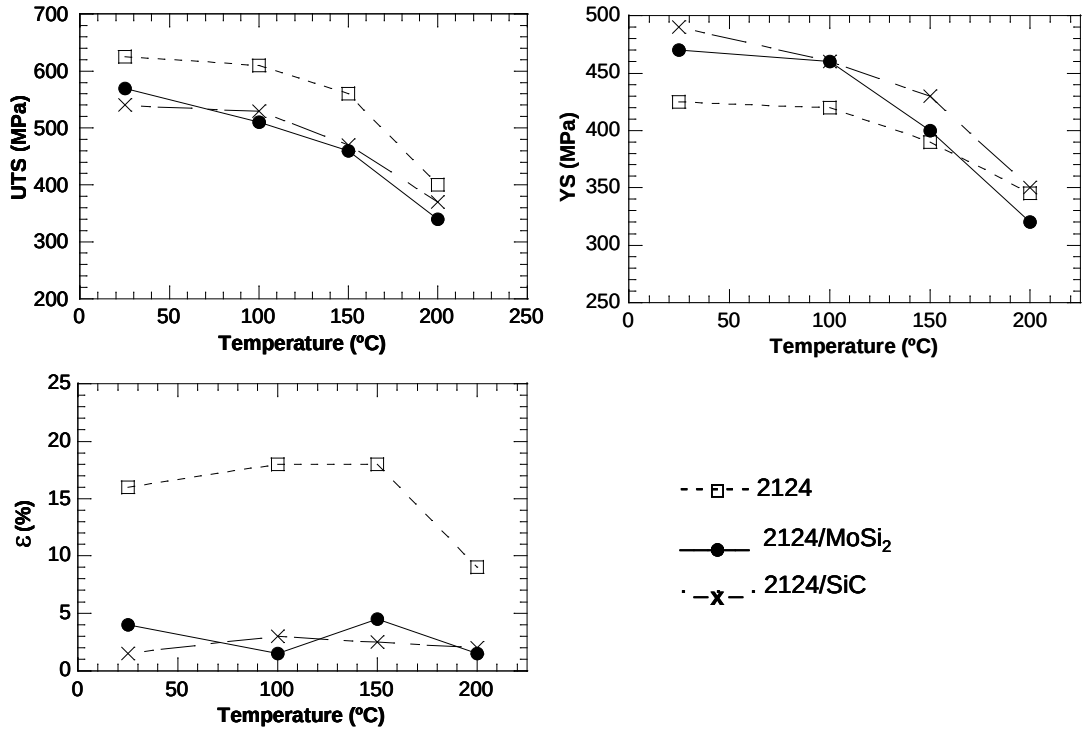


Fig. 8. YS, UTS and εf of 2124/$MoSi_2$, 2124/SiC and 2124 alloy, determined at room temperature and up to 200ºC.

4. Discussion

According to the literature [12], aluminium matrix composites reinforced with intermetallics present in general lower properties after T4 or T6 treatment than in T1. These treatments are intended to obtain maximum strength thanks to precipitation of solute atoms from the matrix in the form of small hardening particles that hinder dislocation movement. When ceramic reinforcement is introduced in the matrix, although some reactions may take place, these do not provoke, in general, catastrophic failure [16-17]. On the contrary, during solid solution treatment of intermetallic reinforced composites, diffusion reaction products are more easily formed between intermetallic particles and the Al alloy matrix. In the case of Ni-aluminide reinforcements, the Al_3Ni that nucleates and grows around the particles is brittle and the

interphase with the matrix becomes very weak [3,5,12]. In the 2124/$MoSi_2$ composite studied here, yield stress, ultimate tensile strength and elongation to failure are clearly higher in T4 than in T1, Table 2. This result indicates that matrix-reinforcement reactions either do not take place, or they are not deleterious. From microstructural observations, Fig. 2a and b for T4 condition, it is inferred that there is no interphase formed that surrounds reinforcing $MoSi_2$ particles.

Industrial application of this type of materials may require solid solution treatments of large components. In this sense it is important to characterise the thermal stability of the 2124/$MoSi_2$ composite at solid solution temperature and the influence of long heat treatments on mechanical properties. As can be observed in Fig. 4, yield stress remains high after at least 30 hours of soaking at 495ºC, and, most significantly, it remains higher than that of the monolith alloy. Moreover, as the time of heat treatment at 495ºC increases, yield stress of the composite suffers a less steeper decrease than the monolith alloy. This fact together with the higher elastic modulus of the intermetallic reinforced material (100 GPa [18]) in comparison to that of the 2124 matrix (72 GPa [19]) and the possibility of submitting large components to solid solution treatments makes this 2124/$MoSi_2$ composite technologically attractive. In addition, the absence of diffusion reaction interphases between 2124 and $MoSi_2$ makes $MoSi_2$ intermetallic a superior option as reinforcing material in comparison to other intermetallics formerly investigated [12].

However, after 100 hours of permanence of the 2124/$MoSi_2$ composite at 495ºC, large plate-like precipitates containing Mg, Al, Si and Cu were observed, Fig. 6, that did not appear in the unreinforced 2124 alloy after the same time of heating, Fig. 7. Taking into account their morphology and the semi quantitative analysis of their composition

(listed in Fig. 6 for three precipitates) this phase could be assigned to $Al_5Cu_2Mg_8Si_5$, which is typical of 2xxx alloys with high silicon content [20]. As the amount of silicon in the original 2124 matrix is very low, this seems to indicate that they form due to an interaction between the atoms of the matrix and some Si atoms coming from $MoSi_2$ intermetallic. However, neither EDS spectra nor X-ray diffraction patterns make it possible to detect a difference in Si content of $MoSi_2$ reinforcing particles.

The loss of UTS and YS observed in 2124/$MoSi_2$ at 100 hours of heat treatment at 495ºC can be obviously attributed to the Al-Cu-Mg-Si-containing precipitates. However, taking into account that the unreinforced 2124 matrix presents the same mechanical behaviour, Fig. 4, other causes such as increased grain size [21] may also play a significant role.

Once the high compatibility of the system 2124/$MoSi_2$ has been asserted, properties of the intermetallic reinforced composites have been compared with those of the SiC reinforced one. Ceramic reinforced Al alloys are already being applied in the industry, but some characteristics of ceramics, such us extreme brittleness and hardness, make them not completely suitable for machining steps and specific applications, mainly for parts submitted to wear. Although ceramic reinforced materials would be more resistant to wear than those reinforced with intermetallics, the counterface is much less damaged in the latter case [6,22]. On the other hand, $MoSi_2$ was selected among other intermetallics because of its high elastic modulus [23,24], quite close to that of SiC, and in this sense $MoSi_2$ can be considered as a possible substitute for ceramics, not only for tribological applications, but also for other applications where high modulus of composite is required [25]. As observed in Fig. 8, tensile properties of both composites are quite similar in the whole temperature range, which indicates that the intermetallic

reinforced composite would be also suitable in applications where tensile properties of SiC reinforced Al alloys are appropriate, with the advantage that the 2124/$MoSi_2$ composite is easier to machine than 2124/SiC. The main drawback of the 2124/15%vol. $MoSi_2$ is its higher density, 3.2 g/cm^3, compared to 2.8 g/cm^3 for 2124/15%vol.SiC. The values of tensile properties of 2124/SiC in this work are in the same range as others reported in the literature [26,27].

As expected [28-30], ultimate tensile stress and yield strength of both composites and the monolith alloy, all of them in T351 condition, diminished as the temperature of tensile test increased, Fig. 8. Elongation to failure, however, behaves in a different way, being quite constant during the whole temperature range for the composites and diminishing only at 200ºC in the case of the unreinforced alloy. This contradicts the expected results. Normally, it would be accepted that ductility increases with increasing temperature due to recovery processes [16,28,29]. A possible explanation is that during high temperature exposure some depletion of solute elements occurred at the surface of Al powder particles which weakened their bonding. The comparison of the results of the tensile tests conducted on the composites and the monolith alloy specimens, which were heat treated at 495ºC, has shown that the yield strength is higher in case of composites. On the other hand, the 2124 Al alloy has demonstrated higher ultimate tensile strength and elongation to failure, compared to the composite

5. Conclusions

A P/M 2124 aluminium alloy was reinforced with 15% volume of $MoSi_2$ powder particles and its mechanical and thermal stability properties studied at various conditions.

In tensile tests from room temperature up to 200ºC, tensile properties of 2124/$MoSi_2$ composite in T351 are similar to those of the ceramic reinforced 2124/SiC composite.

In specimens submitted to heat treatments at 495ºC up to 100 hours, yield stress of the intermetallic reinforced composite is higher than that of monolith 2124, whereas UTS and deformation to failure are lower. At 100 hours, large plate-like precipitates that contain Al, Cu, Mg and Si appear, which seem to indicate some interaction between matrix and reinforcing $MoSi_2$ particles.

The promising properties obtained with the 2124/$MoSi_2$ composite are a consequence of the high chemical compatibility of the 2124-$MoSi_2$ system, that can be subjected to the elevated temperature of the solid solution treatment at up to 100 hours without forming deleterious interdiffusion reactions between matrix and intermetallic reinforcing powder particles. This high thermal stability makes of $MoSi_2$ intermetallic a superior reinforcing option in comparison to other intermetallics studied up to now, and a real competitor for ceramic reinforcements.

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Figure legends

Fig. 1. Schematic representation of the P/M process.

Fig. 2. SEM micrographs showing the microstructure of the 2124/$MoSi_2$ composite after 0.5 hour (a and b), 1 hour (c and d), 10 hours (e and f), 30 hours (g and h) and 100 hours (i and j) of heat treatment at 495ºC.

Fig. 3. EDS concentration lines of Mo, Si, Al, Cu, Mg and O at particle/matrix interface of 2124/$MoSi_2$ composite after 30 hours of heat treatment at 495ºC (see Fig. 2(h)).

Fig. 4. YS, UTS and εf of 2124 matrix and 2124/$MoSi_2$ composite after submission to heat treatments at 495ºC for 0.5 to 100 hours.

Fig. 5. a) and b) Fracture surface of 2124/$MoSi_2$ heat treated for 3 hours at 495ºC and EDS spectra of c) matrix and d) a $MoSi_2$ intermetallic particle.

Fig. 6. a) and b) Fracture surface of 2124/$MoSi_2$ heat treated for 100 hours at 495ºC and c) EDS spectra of three Mg-Al-Si -Cu-containing precipitates.

Fig. 7. Fracture surface of 2124 alloy heat treated for 100 hours at 495ºC.

Fig. 8. YS, UTS and εf of 2124/$MoSi_2$, 2124/SiC and 2124 alloy, determined at room temperature and up to 200ºC.