



Production of AA2124/MoSi₂/25p composites and effect of heat treatment on their microstructure, hardness and compression properties

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ABSTRACT: AA2124/25vol%MoSi₂ composites were processed by two powder metallurgy routes: high energy ball milling of the reinforcement and alloy powder (B composite) and wet blending with cyclohexane (W composite), both followed by extrusion to achieve full consolidation. As-extruded and heat treated composite bars were studied microstructurally and mechanically (hardness and compression tests under quasistatic loading). Microstructure and fracture profiles were observed by scanning electron microscopy and the reaction products formed in the matrix were identified by energy-dispersive X-ray spectroscopy and X-ray diffraction analysis. The results show that for both composites, the hardness of the specimens in solution and aged condition was higher than in the as-extruded condition. The hardness of the B composite was higher than that of the W composite whereas the age-hardenability of the B composite was significantly lower than that of the W composite. After heat treatments, small diffusion reaction phases appeared at the interface between matrix and reinforcements. Compressive yield strength and the ultimate strength of both composites improved considerably after the artificial ageing. The composite fracture surfaces exhibited microscopically a ductile appearance that consisted of dimples in the matrix and a fragile fracture of the MoSi₂ particulates.

KEYWORDS: Aluminium matrix composites; MoSi₂ intermetallic reinforcement; Powder metallurgy; Wear properties

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RESUMEN: *Producción de materiales compuestos AA2124/MoSi₂/25p y efecto del tratamiento térmico sobre su microestructura, dureza y propiedades a compresión.* En este trabajo se procesaron materiales compuestos AA2124/25vol% MoSi₂ mediante dos rutas pulvimetalúrgicas: mezcla de refuerzo y matriz mediante molino de bolas de alta energía (compuesto B) y mezcla húmeda con ciclohexano (compuesto W). Ambos polvos compuestos se consolidaron por extrusión. Los materiales recién extruidos y después de tratados térmicamente se estudiaron desde el punto de vista microestructural y mecánico (dureza y compresión bajo carga cuasiestática). Las microestructuras y los perfiles de fractura se observaron por microscopía electrónica de barrido y los productos de reacción formados en la matriz se identificaron por espectroscopía de dispersión de energía de rayos X y por difracción de rayos X. Los resultados indican que para ambos materiales la dureza es mayor después de los tratamientos térmicos. Por otro lado, la dureza del material compuesto B es mayor que la del W, mientras que la capacidad de endurecer de B es mucho menor que la de W. Después de los tratamientos térmicos aparecen pequeñas cantidades de fases de reacción entre la matriz y el refuerzo. La resistencia a compresión de ambos materiales compuestos mejora considerablemente a consecuencia del envejecimiento artificial. Las superficies de fractura exhiben una apariencia dúctil, con formación de cúpulas en la matriz y fractura frágil de las partículas de MoSi₂.

PALABRAS CLAVE: Desgaste; Materiales compuestos de matriz de aluminio; Pulvimetalurgia; Refuerzo intermetálico de MoSi₂

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1. INTRODUCTION

Discontinuously reinforced aluminum alloys (AMCs) possess superior stiffness, specific strength and wear resistance compared to unreinforced ones. Ceramic reinforcements have been preferentially used over the past few decades (Clyne and Withers, 1993; Chawla, N. and Chawla, K.K., 2006). However, they present a few drawbacks, such as high abrasiveness and brittleness, and recycling difficulties, together with more specific problems such as a high mismatch between the coefficient of thermal expansions (CTE) of matrix and reinforcement, which may result in poor thermal fatigue resistance. Some studies consider the possibility of adding intermetallics, which seems to be an interesting option in view of their high hardness, elastic modulus and compatibility with the matrix (Omura *et al.*, 1988; Zhou *et al.*, 1990; González-Carrasco *et al.*, 1994; Torres *et al.*, 2002; Pour *et al.*, 2007). Corrosion (Silva-Maia *et al.*, 1999) and wear behavior (Walker *et al.*, 2005; Sameezadeh *et al.*, 2010; Corrochano *et al.*, 2011) are also improved. AMCs reinforced with intermetallics are also, in principle, much easier to recycle because it is not necessary to separate both components of the composite before melting. Among intermetallics, MoSi₂ emerges as a promising candidate because of its high compatibility with aluminum and high Young modulus (440 GPa at room temperature) (Torres *et al.*, 2002; Tanaka *et al.*, 2001).

Powder Metallurgical (PM) methods have been mainly employed to produce intermetallic reinforced AMCs in order to avoid the formation of deleterious interphases and because, through PM, the spatial distribution of the particles is normally more homogeneous than that obtained by casting methods (Lieblich *et al.*, 1997). High-energy ball milling has been used to further improve particle distribution throughout the matrix (Lu *et al.*, 1998; Parvin *et al.*, 2008; Corrochano *et al.*, 2009) because fracturing and cold welding of the powder particles occur, causing the reinforcement particles to be well embedded into each aluminum particle. Furthermore, the high degree of deformation involved reduces matrix grain size to nanometer level and produce a very fine distribution of oxides depending on the processing parameters (Corrochano *et al.*, 2009).

In the present work, 2124 aluminum alloys reinforced with 25 vol.% of MoSi₂, have been produced by two powder metallurgy routes. Hardness, microstructure and compression behavior of the 2124/MoSi₂ material on both as-extruded and heat-treated conditions have been investigated. The results have been compared to those of the monolith PM 2124 alloy, submitted to the same heat treatments.

2. MATERIALS AND METHODS

Argon atomized AA2124 aluminum alloy powder of <60 µm in diameter (a product of Alpmco, UK) was used as the matrix. The chemical composition of matrix alloy (AA2124) is shown in Table 1. The intermetallic reinforcing particles, MoSi₂, were produced from elemental powders by self-propagated high-temperature synthesis at INASMET (now TECNALIA), Spain, followed by jet milling of the porous product and disc milling. The resulting median diameter of the MoSi₂ intermetallic was less than 20 µm. AA2124 powder was blended with 25 vol.% of MoSi₂ by two methods: planetary ball milling operating in air at room temperature at 200 rpm for 4 hours (B composite) and wet blending with cyclohexane as liquid agent (W composite). The blends were encapsulated in 6063 aluminum cans and consolidated by extrusion in a horizontal direct hot extrusion press. Extrusions were performed at a temperature of 450 °C, a ram speed of 0.4 mm s⁻¹ and an extrusion ratio of 14:1 leading to 15 mm diameter bars that were left to cool down in air. The composite bars then were cut to small pieces ready for structural characterization. The materials were tested in as-extruded condition and after heat treatment. The heat treatment cycle was: solution treatment for 1 hour at 495 °C, water quenching and artificial aging at 190 °C for times between 1 and 12 hours. This artificial aging allowed to determine the T6 treatment for each material, i.e. the time of heat treatment that gives maximum hardness.

Microstructural characterization was performed by Scanning Electron Microscopy (SEM). The specimens for microscopy observations were prepared by standard metallographic techniques without any chemical etching. Microanalysis was undertaken using Energy Dispersive X-ray Spectroscopy (EDS). X-ray diffraction was performed using a Bruker D8 diffractometer with Cu radiation ($\lambda = 1.54 \text{ \AA}$) operated at 40 kV and 30 mA.

Brinell hardness measurements were taken at room temperature on all specimens using a 5 mm ball at a load of 1225 N (125 kgf). At least five indentations were performed on each specimen. Dispersion of data was less than 5%. Hardness data were collected from the as-received and solutionized specimens and then after each interruption in the heat treatment to assess ageing response.

Finally, cylinders with a length/diameter ratio of 2:0 (10 mm length and 5 mm diameter) were

TABLE 1. Chemical composition of the AA 2124 aluminum matrix alloy (wt.%)

Element	Al	Cu	Mg	Mn	Fe	Si	Cr, Ti, Zr
% Wt	Bal.	4.24	1.4	0.85	0.06	0.03	<0.01

prepared from both as-extruded and heat treated samples, according to the ASTM E9-09 (2009) standard for compression testing. The specimens were tested with a DARTEC 9500 testing facility under quasistatic loading (strain rate of $8 \times 10^{-4} \text{ s}^{-1}$) at room temperature. Both ends of the specimens were polished to make them parallel to each other prior to the compression test.

3. RESULTS AND DISCUSSION

Figure 1 presents a SEM micrograph of the synthesized micrometric MoSi₂ particles, which shows a quite broad size distribution of MoSi₂ particles. Figure 2(a and b) shows the microstructure of B and W composites in the as-extruded condition. Sample W shows the largest particles. The smaller, submicrometric MoSi₂ particles, in B composite are the result of the ball milling process, where larger intermetallic particles break more easily than smaller ones. It is also obvious from the micrographs of Figure 2 that MoSi₂ distribution is more homogeneous in B than in W composite, with some agglomeration being more evident in the latter. The poorer distribution in W composite should be ascribed to the large difference between particle size of reinforcement, $<20 \mu\text{m}$, and aluminum alloy, $<60 \mu\text{m}$, that, due to geometrical constraints, makes good distribution impossible (Bhanu Prasad *et al.*, 2002).

Matrix grain size (d_m) and mean MoSi₂ particle size (D_{rf}) of B and W composites are shown in Table 2. The determination of aluminum matrix grain size was performed using X-ray diffraction line broadening. It is obvious that matrix grain size, or at least the diffraction volume, is reduced during the ball milling process, reaching a value of 43 nm

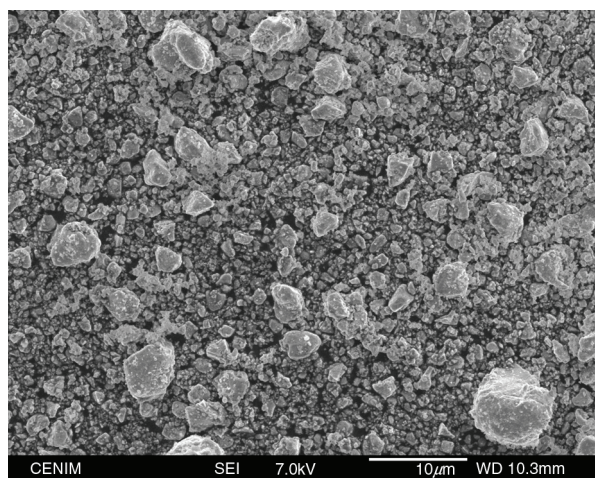


FIGURE 1. SEM micrograph of micrometric MoSi₂ produced after disc milling.

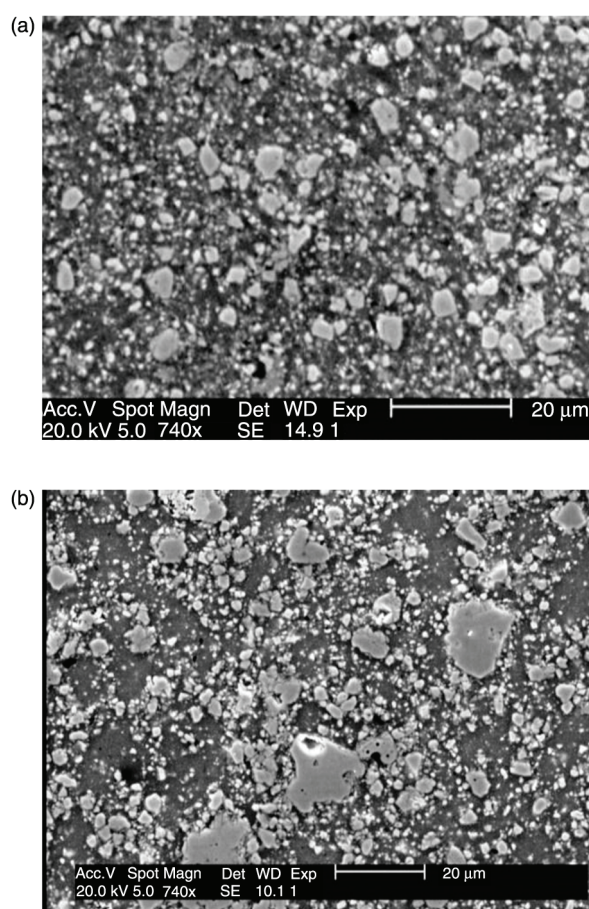


FIGURE 2. Cross section of as-extruded AA2124/25vol%MoSi₂ composites: (a) B composite and (b) W composite.

for B composite, 30% lower than the 60 nm of W composite. It was found that after heat treatments, no further changes in the grain size of the matrix occurred. This was true for both composites. The particle size of the reinforcement (Table 2) was evaluated by image analysis, measuring at least 1200 particles per material. As a result of the more energetic ball milling process, reinforcement particles were much smaller in B composite than in the wet blended W composite.

Figure 3 shows Brinell hardness of extruded AA2124/25vol% MoSi₂ composites (B, W) after heat treatments at 190 °C. Artificial ageing of the AA2124/25vol%MoSi₂ extrudates results in a

TABLE 2. Matrix grain size (d_m) and mean MoSi₂ particle size (D_{rf}) of extruded composites

Material	d_m (nm)	D_{rf} (μm)
B composite	43	0.36
W composite	60	1.10

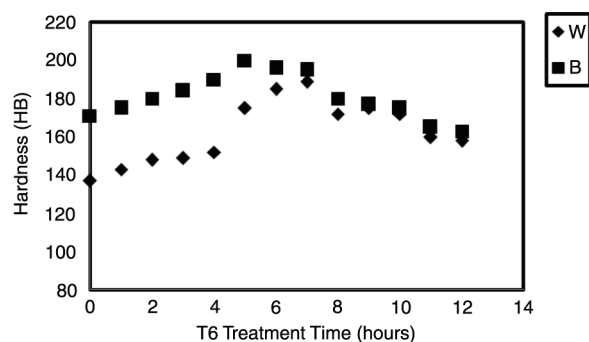


FIGURE 3. Brinell hardness of extruded AA2124/25vol%MoSi₂ composites (B, W) after heat treatments at 190 °C. 0 hours is the as-extruded hardness whilst 1 hour is that of solution treated samples.

rapid increase in hardness, so that maximum hardness is achieved after only 5 hours treatment for B and 7 hours for W. The shorter peak age time in B can be associated with its smaller grain size and reinforcement size (Corrochano *et al.*, 2009). Comparison of the ageing response of the monolithic (not shown in the graph) and the two reinforced AA2124 alloys (B, W) at 190 °C indicates that the composites follow the expected general trend of higher as-solutionized hardness and shorter peak age time than the unreinforced 2124 alloy (Chawla, N. and Chawla, K.K., 2006).

In Figure 4, peak age hardness (i.e. after a T6 treatment), HT6, and in solutionized condition, HS, have been represented for the composites and the monolithic alloy, together with their age-hardenability, $\Delta HT6$, which is the difference between the former quantities, $\Delta HT6 = HT6 - HS$. According to the literature, the ball milling process increases the dislocation density of the ductile materials and this effect is intensified if hard reinforcements are present, as occurred during ball milling of AMCs (Suryanarayana, 2001; Khakbiz and Akhlaghi, 2009).

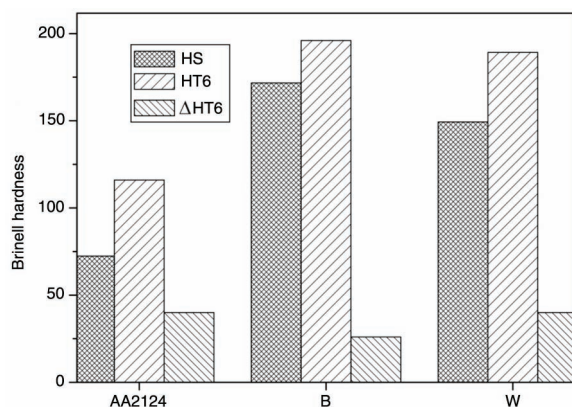


FIGURE 4. Hardness and age-hardenability of heat treated samples.

Mechanically alloyed materials not only have high dislocation density around hard reinforcing particles but they also have smaller grain structures which promote precipitation sites. The age-hardenability decreases with increasing the precipitation of stable phases on grain boundaries, because these phases contribute very little to the hardening effect (Parvin *et al.*, 2008). In addition, higher density of grain boundaries, that act as sinks for the solute atoms and vacancies (which are necessary to form age-harden precipitates), promote more precipitation free zones (Corrochano *et al.*, 2009). The preferential precipitation of the stable phases on grain boundaries may occur in the earlier stage of aging and contribute to a significant reduction of the age-hardenability effect (Kang and Chan, 2004).

Another important feature that is observed in Figure 4 is that the addition of MoSi₂ particles has brought a grand increase in the hardness of composites compared to the matrix alloy, which amounts to about 100% in the solutionized condition and 80% in T6. An increase was expected because hard particulate reinforcements act as a barrier to the dislocation movement within the matrix and exhibit greater resistance to indentation of the hardness tester. From Figure 3, the B composite exhibits higher hardness than that of the W composite. This is mainly due to the fact that the inter-particle distance is smaller in using smaller reinforcement (at constant vol.% comparison), which results in more effective action of Orowan mechanism (Arakawa *et al.*, 2000). In this mechanism, the dislocation bends between the particles leaving a dislocation ring around each one. Energy must be supplied to increase the total length of the dislocation line; the stress required is, neglecting a numerical factor, roughly $(Gb)/L$ where G is the shear modulus, b is the Burgers vector, and L is the spacing between obstacles.

The improve of hardness in Al-Cu alloys is due to the presence of Cu in solid solution in the matrix, which during the age treatment gives rise to the formation of Al₂Cu precipitates (Smith, 1998). Figure 5(a and b) shows the microstructure of MoSi₂ reinforced composites in overaged condition. Phases at the interface between matrix and reinforcements that appeared only after the heat treatment are arrowed in these micrographs. According to the literature (Smith, 1998), the composition of these interphases can be identified with S' (CuMgAl₂) and θ' (CuAl₂) as they contain Al, Cu and, some of them, small amounts of Mg. XRD pattern of the composite samples, in heat treated condition, is shown in Figure 6, where the presence of the CuAl₂ phase is evident in both samples.

Generally speaking, at ageing temperatures corresponding to the precipitation of a semi coherent phase the effect of introducing the reinforcement is to accelerate the kinetics of hardening. This acceleration

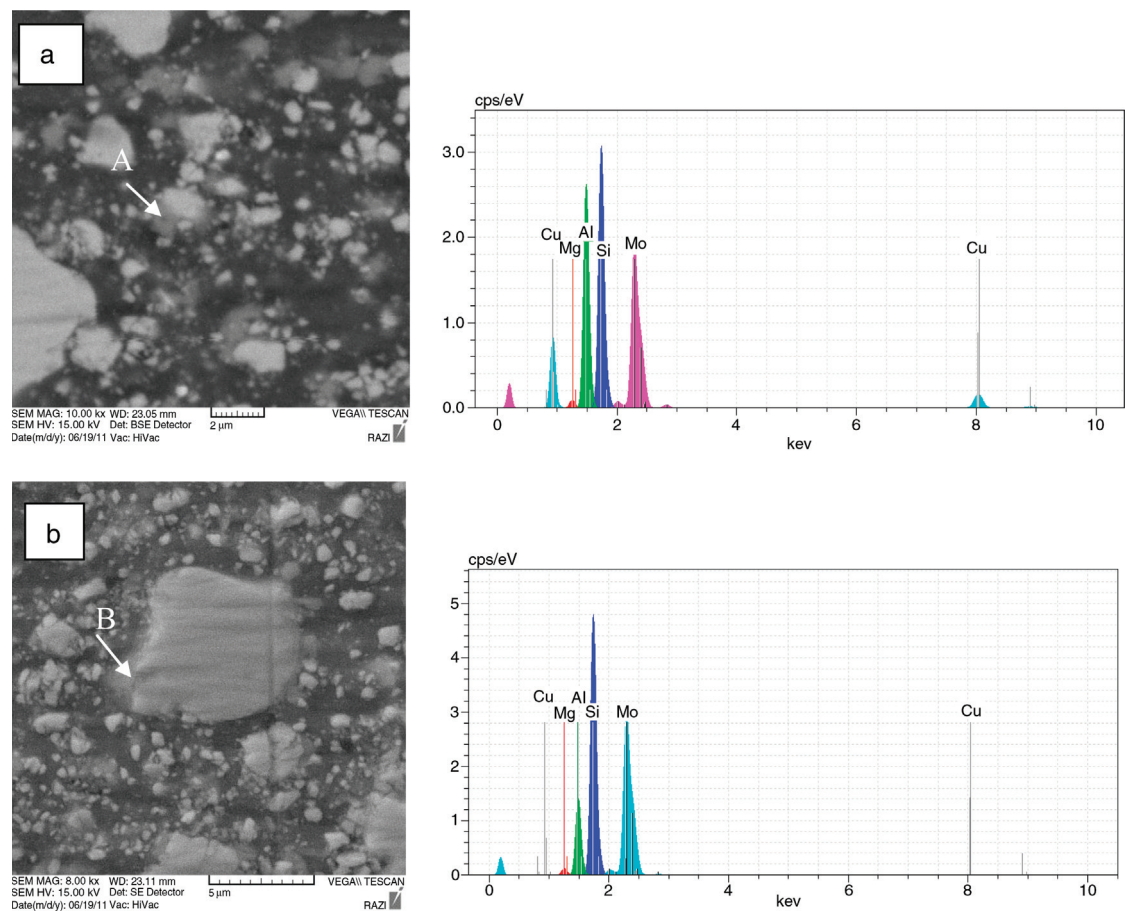


FIGURE 5. SEM micrograph and EDS analysis of AA2124/25vol%MoSi₂ composites after 12 hours ageing: (a) B composite and (b) W composite, showing small Cu-rich precipitates (A,B) next to MoSi₂ particles.

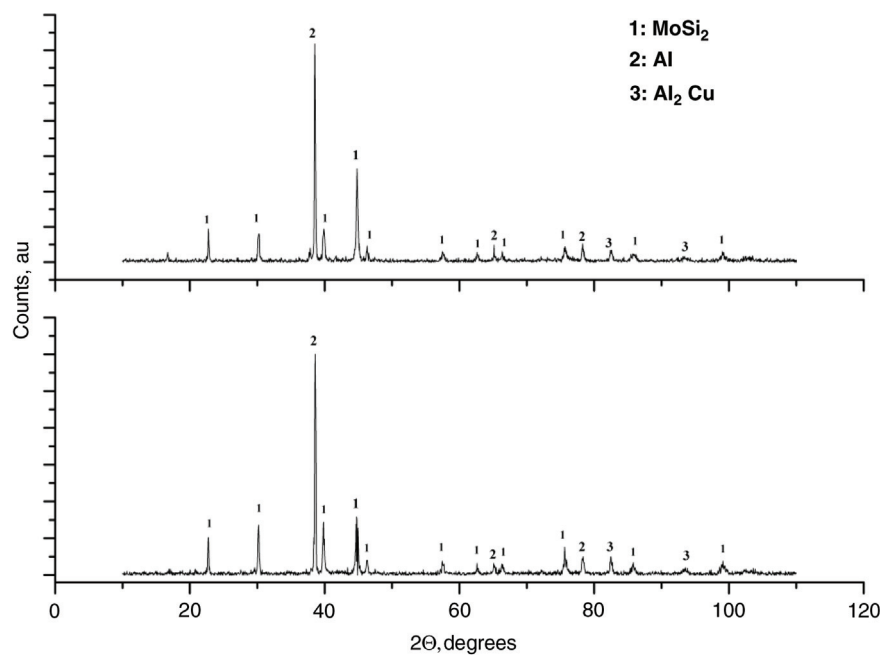


FIGURE 6. XRD pattern: (a) B composite and (b) W composite after 12 hours ageing.

TABLE 3. Compression properties in as-extrude and heat treated 2124 composites reinforced with 25 vol.% of MoSi₂

	B		W	
	As-extruded	Heat treated	As-extruded	Heat treated
YS (MPa)	300	350	250	300
UTS (MPa)	480	570	440	550

is determined by the ratio $R = t_{pHm}/t_{pHc}$, where t_{pHm} is the time to peak hardness in the unreinforced matrix and t_{pHc} is the time to peak hardness in the composite (Walker *et al.*, 2005). R depends on the volume fraction of the reinforcement and, to a lesser extent, on the size of the particles and on the homogeneity of their distribution (Merle, 2000). R is calculated as $16/5 = 3.20$ for B composite and $16/7 = 2.28$ for W composite.

The decrease in particle size eliminates the formation of GP- I zones and promotes the direct formation of GP-II zones. The time required for peak ageing is a function of the reinforcement size, and the hardening kinetics is different according to the size of the reinforcing particles (Merle, 2000). This explains why the time required for peak ageing is shorter for B composite, reinforced with smaller particles, than for W composite.

Table 3 shows results of compression tests performed for each AA2124/MoSi₂ composite and condition studied here. Yield stress and ultimate compression strength are clearly higher in the aged sample than in as-extruded condition. As can be seen, the yield strength as well as the ultimate tensile strength of both composites improve considerably after the artificial ageing. This increase is attributed to the presence of small intermetallic precipitates. Coherent precipitates increase the material flow strength through the well-known mechanism of dislocation precipitate interaction.

Figure 7 shows the fracture surface of the heat treated composite materials. In both cases (B,W) the micro mechanism of fracture is similar with decohesion between reinforcing particles and the matrix more evident in W composite. The composite fracture surfaces revealed microscopically a ductile appearance with dimples in the matrix and a fragile fracture of the particulates. The cracks follow the matrix/reinforcement interfaces.

4. CONCLUSIONS

AA2124/25vol%MoSi₂ composites were produced by two powder metallurgy routes followed by hot extrusion. The effects of MoSi₂ reinforcement and ageing treatment on mechanical properties and structure of the composites were investigated. The results of this research can be summarized as follows:

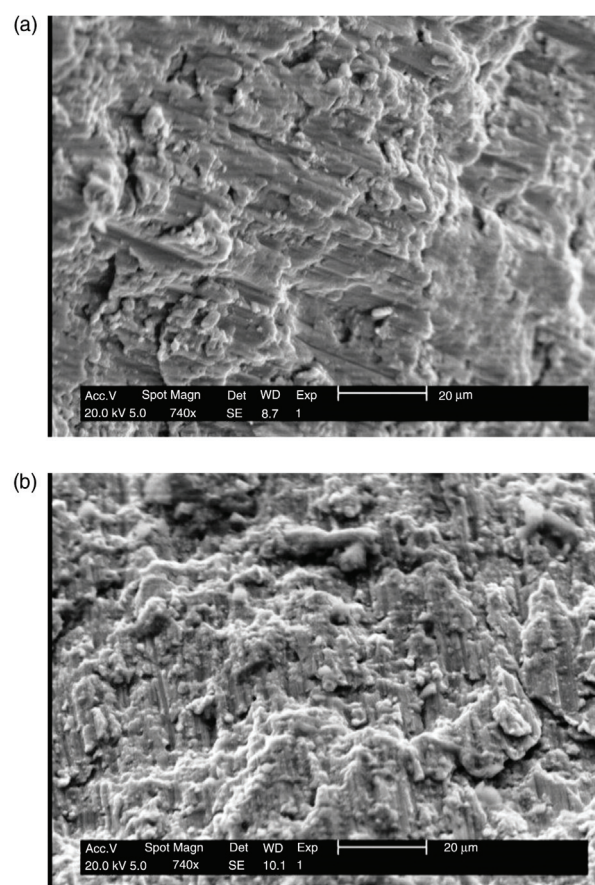


FIGURE 7. Fracture surface of composites after heat treatment: (a) B composite and (b) W composite.

- Microstructural studies have confirmed that all the extruded composites have a quite homogeneous distribution of intermetallic particles and that no reaction appears at matrix/particle interfaces in the as-extruded condition.
- For both composites, the hardness of solution treated and aged condition was higher than for the as-extruded condition. The hardness of B composite was higher than that of W composite whereas the age-hardenability of the B composite was significantly lower than that of W composite.
- Compared to the monolithic alloy, the addition of MoSi₂ particles has brought a 100% increase in hardness in the solutionized condition and 80% increase in T6 condition.
- The time required for peak ageing is shorter for B composite reinforced with the smallest particles in comparison with W composite.
- Small diffusion reaction phases appeared at the interface between matrix and reinforcements after heat treatment. The XRD pattern of heat treated composite samples shows the presence of CuAl₂ phase in both composites.

- It was found that compressive yield strength and the ultimate strength of both composites improved considerably after the artificial ageing, and this is attributed to the presence of small inter-metallic precipitates.
- The composite fracture surfaces exhibited microscopically a ductile appearance that consisted of dimples in the matrix and a fragile fracture of the MoSi₂ particulates.

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