

Mechanical Properties and Microstructure of Al-Al₂O₃ Composites

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Abstract

Microstructural development in a powder, metallurgy 2014 aluminum alloy - Al₂O₃ particles composite subject to controlled and systematic aging treatments was investigated using analytical transmission electron microscopy and matrix microhardness measurements. In order to build a basis for comparison, the precipitation characteristics of the unreinforced matrix material with an identical processing history were also examined. The results indicate that the matrix of the composite material has a much greater density of dislocations than the control alloy. The increased dislocations density facilitates the nucleation of strengthening precipitates whereby the incubation time for precipitate nucleation and the aging time to achieve peak hardness in the matrix are significantly reduced for the composite as compared to the unreinforced matrix material.

Riassunto

Mediante analisi al microscopio elettronico in trasmissione e misure di microdurezza, è stata studiata l'evoluzione microstrutturale della lega di alluminio 2014 rinforzata con particelle di Al₂O₃ ottenuta per sinterizzazione.

Il meccanismo di precipitazione della lega rinforzata è stato confrontato con quello della lega senza rinforzo sottoposta allo stesso trattamento termico.

I risultati evidenziano una maggiore densità di dislocazioni nella lega rinforzata rispetto alla lega senza rinforzo.

All'aumentare della densità di dislocazioni risulta facilitato il processo di nucleazione dei precipitati rinforzanti la lega, cosicché sia il tempo di nucleazione che quello necessario alla matrice rinforzata per raggiungere il picco massimo di durezza risulta ridotto in modo significativo rispetto alla matrice non rinforzata.

Introduction

Aluminum alloys reinforced with fibres and particles offer attractive combination of properties for an extensive use in aircraft structures. In fact the metal matrix composites combine the high strength and the hardness of the reinforcing phase with the ductility and toughness of light metals.

However, the use of these materials is limited by their low fracture toughness and ductility.

These low fracture toughness has been associated to different thermal coefficient between the two phases which generated a high dislocations number in the matrix (1-2).

The increased dislocation density facilitates the nucleation of precipitates and the aging time to achieve peak hardness on the reinforced alloy is lower (3-5).

The majority of investigations conducted on whisker-reinforced alloy utilized heat treatment procedures which allowed to obtain primarily information on microstructural evolution for the unreinforced matrix material (6-8).

The objective of the present work is to correlate of microstructural development in a Al₂O₃ whisker-reinforced 2014 aluminum alloy in function of the aging heat treatment.

In order to build a basis for comparison, the precipitation characteristics of the unreinforced matrix material with an identical processing history were also investigated. Microstructural evolution during controlled aging of both the unreinforced material and the composite was studied using analytical transmission electron microscopy techniques, matrix microhardness measurements and quantitative analysis of precipitate growth.

Experimental Procedure

In the present investigation the alloy 2014 reinforced with 20 vol. % Al₂O₃ particles was used. The alloy

was produced by Alumina S.p.A. Novara (Italy), by powders metallurgy technology. The matrix composition of the composite is shown in Table 1.

TABLE 1 - Chemical composition of matrix, (wt%)

Cu	Si	Mn	Mg
4,55	0,86	0,78	0,60

The MCC microstructures in the extrusion direction and in a direction perpendicular to the extrusion axis are shown in Fig. 1.

After mechanically thinning the specimens down to 2 mm thickness, they were solution treated.

The mechanical thinning was necessary in order to remove non-homogeneous areas where the presence of oxide particles was found.

After the mechanical treatment, cubic samples of 1 cm side were obtained.

The samples were solution treated in an anhydrous nitrogen atmosphere at a temperature of 500°C for 3 hours and water quenched at room temperature; in order to prevent natural ageing of the composite, the specimens were kept at -20°C.

Subsequently, different microstructures were obtained ageing MMC samples in a thermostatically controlled silicone-based bath at temperature of 145°, 160°, 180° and 190°C, with times ranging from 1 to 280 hours. After ageing treatment, the specimens were rapidly cooled in water at room temperature and then tested with microhardness measurements.

Microhardness measurements were performed by means of microhardner type HMV 20000 produced by Shimatzu, using a 136°C Vicker diamond pyramid indenter and a 25 g. load.

In order to obtain reliable microhardness values, microhardness measurements must be taken very carefully in the matrix.

The optimum loading conditions were determined through microhardness tests performed with loads varying between 5 and 100 g.

Results and Discussion

Fig. 2 shows that microhardness values up to 25 g. test load are constant; at higher test loads the microhardness measurements show a strong dependence load, that can be attributed to impression size.

As a matter of fact, at test loads higher than 25 g. microhardness measurements were partially achieved an reinforcement.

In Figs. 3 and 4 are reported respectively, at different temperatures, the microhardness values of the metal matrix as a function of ageing time for the alloy with and without reinforce.

The curves show that the necessary time to reach the maximum hardness peak decreases with increasing ageing temperatures, and that the time necessary to reach the maximum hardness peak is lower for the composite material.

The difference is minimum at lower ageing temperatures, while it increases at high ageing temperatures.

This behaviour has been attributed to microstructural modifications in the metallic matrix during the ageing treatment.

S¹ (Al₂CuMg) precipitates, which are responsible for the materials hardening at high ageing temperatures, nucleate heterogeneously in the dislocations and grow along a straight line while at lower temperatures there

is homogeneous nucleation in Cu rich GP zones.

In fact, as the ageing temperature increases, the GP zones nucleation is obstructed by the microvoids produced during the cooling process. The presence of microvoids into the metallic structure favours the dislocation formation and consequently the S' phase precipitation.

This is confirmed by scanning calorimetric analyses performed at low temperatures on similar materials (9). In the composite material, owing to the different thermal expansion coefficients (CTE) between matrix and reinforcement, there is a remarkable plastic deformation near the interface and consequently a tension increase.

The high number of dislocations (about $4 \times 10^{14}/m^2$) (10), present in these zones is due to the above mentioned tensions.

Therefore, as the dislocations density increases at higher temperatures, the heterogeneous nucleation and the fast growth of the S' phase will be favoured, resulting in an accelerated ageing process of the composite material.

Furthermore we have observed (11,12) that, not only the S' phase, but also the possible oxides formation at the matrix-reinforcement interface, contributes to a remarkable ageing of the composite material.

The microstructural modifications related to the ageing process, has been investigated by Transmission Electron Microscopy (TEM).

TEM Analysis

3 mm diameter disks mechanically thinned with grinding paper were obtained from the aged samples. Final thinning was performed in a double-jet unit using a 30% HNO₃-methanol solution at -40°C and 12V.

A TEM Philips CM12 equipped with LaB6 cathode at 120Kv was used. In both alloys at low aging temperature, the presence of GP zone and S' phase precipitates (Al₂CuMg) is revealed.

The phase S' volume fraction, responsible of the hardening is much larger in the reinforced alloy, due to the great number of dislocations present at the interface between the matrix and the surface; which for the S' phase, represent preferential sites of heterogeneous nucleation. Fig. 5 show the S' phase heterogeneously nucleated on dislocations.

Conclusions

By comparing the results of the experimental tests of the investigation on the ageing kinetics of the alloy Al 2014 and of that reinforced with whiskers Al₂O₃, a substantial difference of the ageing time necessary to two samples to reach the maximum hardness peak is evidenced.

In fact, the time necessary, to the reinforced alloy to reach the hardness peak is shorter and this difference increases with the ageing time.

This difference in the ageing kinetics, is related to different thermal expansion coefficients between the fibers and the matrix, and forms, at the fiber-matrix interface, the formation of the a high number of dislocations which act as preferential sites for the S' phase nucleation, responsible of the alloy hardening.

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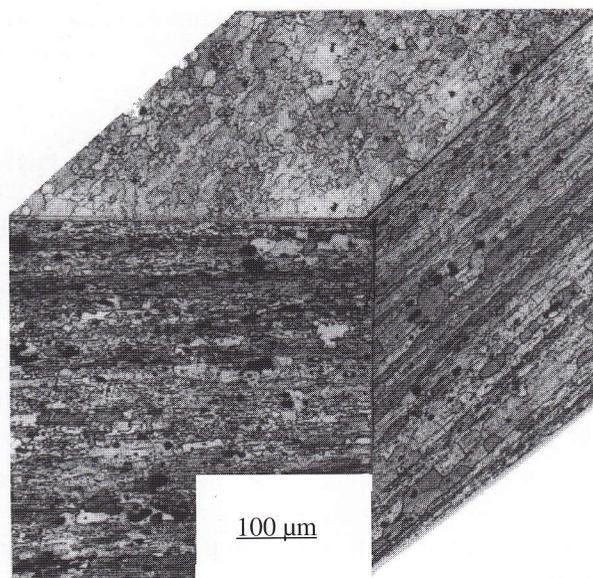


Fig. 1:
Microstructures of Al-Al₂O₃ composite

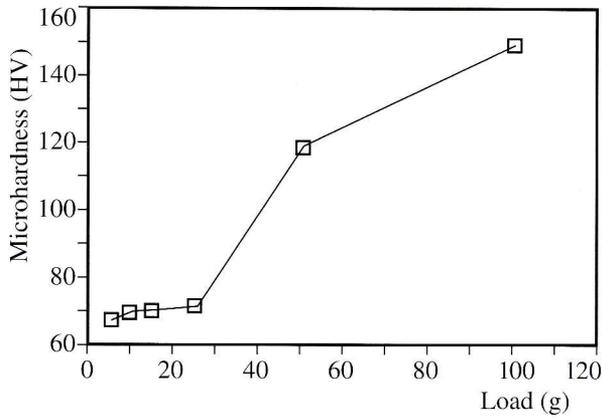


Fig. 2:
Effect of load on microhardness value

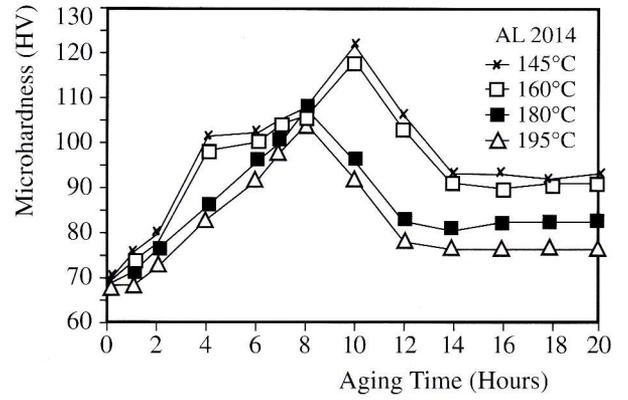


Fig. 3:
Variation in matrix microhardness for alloy without reinforce as a function of aging time at different temperatures

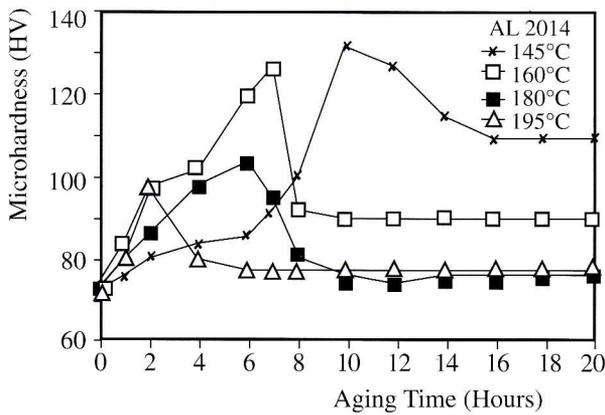


Fig. 4:
Variation in matrix microhardness for alloy with reinforce as a function of aging time at different temperatures

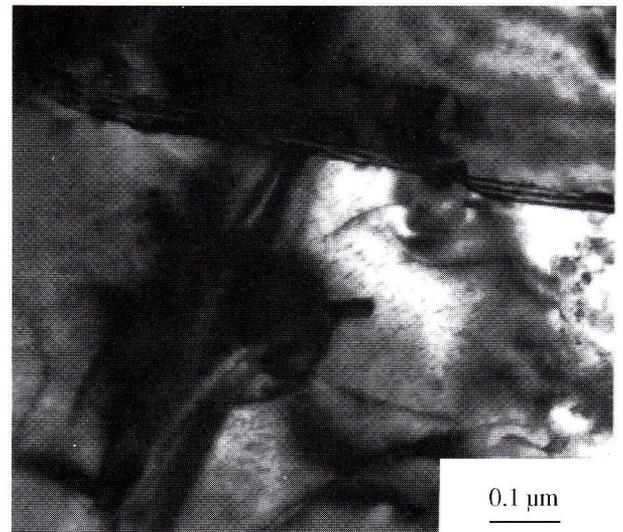


Fig. 5:
S' phase nucleated on dislocations