Microstructure and Mechanical Behavior of Al-Si-Mg Alloys reinforced with TiB₂ particles

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MMCs of aluminum alloy matrix reinforced with TiB₂ particles were prepared in the laboratory by following a powder metallurgy route. The mixture of powders was compacted and, after lubrication, hot extrusion was employed as consolidation process. A homogeneous distribution of the reinforcement particles was achieved. Different studies were carried out in the composites developed. During fabrication and posterior heat treatments of MMCs, it is possible that reaction products and intherphases can appear in case the matrix is a high strength heat-treatable alloy. The effects of the TiB₂ reinforcement, heat treatments regarding temperature (of 350, 400, 450 and 500°C) were carried out at a fixed time. Tests were developed as much in MMCs fabricated as in aluminum matrix, to evaluate the changes due to the presence of reinforcement. In this study, optical and scanning electron microscopy with X-ray microanalysis, were used to determine the possible reaction products in the matrix/reinforcement interface and its composition. Microhardness tests were carried out on polished samples in order to evaluate the mechanical behavior. It was followed the Vickers microhardness evolution through the reaction layer formed in the different materials studied.

Ultimate strength and strain to failure were studied. There were performed mechanical tests at room temperature and at high temperature of the material in extrusion state. The type of damage was analyzed by scanning electron microscopy, viewing the effect in the sample surfaces. It was considered the microstructures changes due to the strain which samples were exposed. With this aim, longitudinal cuts in the strain were made examined by scanning electron microscopy.

Parole chiave: alluminio e sue leghe, compositi, prove meccaniche

INTRODUCTION

Aluminum-based composites reinforced with hard ceramic particles have received considerable interest because they can offer relative ease of processing and nearly isotropic properties in comparison to fiber-reinforced composites. In addition, these composites exhibit high strength and stiffness, creep resistance and superior wear resistance, whilst also provide good electrical and thermal conductivity. This suite properties makes particle reinforced MMCs attractive to a wide range of applications in automotive, aerospace and transport industries. Reinforcement particles used in the MMCs include nitrides, borides, carbides and oxides [1]. Among these particles, titanium diboride TiB₂ is particularly attractive because it exhibits high elastic modulus, strength and hardness as well as high thermal conductivity [2]. Titanium diboride also has high melting point with high chemical stability [3, 4] It is compatible with an aluminum matrix, and does not react with the aluminum at low temperatures. In this case, the formation of brittle interfacial products at

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Various aspects of ceramic particle reinforced aluminum MMCs have been investigated, especially in terms of the fabrication methods, interfacial and mechanical properties [11]. Current methods of fabrication can be divided into physical and chemical methods. The physical methods include spray deposition, powder metallurgy, mechanical alloying, liquid pressure forming and squeeze casting. Chemical methods result in in-situ formed Al MMCs. Each of these methods has its own advantages and disadvantages. The P/M route generally gives better mechanical properties whilst the in-situ methods, have lower fabrication cost.

The existing studies on reactive processing of Al-TiB₂ composites [12-17] agree that there is a lack of understanding of the reaction mechanisms, which hinders the optimization of the processes and the Al-TiB₂ composite product. This product optimization consists of controlling the composition, by avoiding the embrittling Al₃Ti phase; and the microstructure, by reducing the TiB₂ size particle. It is also favorable to reduce process temperature in order to avoid interfacial products. So a powder metallurgy process where the manufacture temperature used is the lowest possible, is a desirable method. To obtain MMCs with optimum properties is applied a secondary processing of the composites that assure uniform distribution of the reinforcing material in the matrix and the formation of a good interfacial bond. Amongst the various classical metal-forming procedures, extrusion and hot compacting have been used as the most secondary processing operation because of their excellent preferential axial alignment and their large compressive hydrostatic stress state [18, 19].

At the present, the two major obstacles to the application of such materials are high cost and chemical reaction at reinforcement/matrix interfaces during materials processing and service at elevated temperatures. Reinforcement/matrix interfaces undergo diffusion reaction [20], which can vary widely with diffusion of the individual components of the system [20, 21]. The study interface exhibits diffusion of its components when being submitted to high temperature. The diffusion at microscopic level could modify the structure of the interface altering the macroscopic, mechanical and thermal properties of the composite materials [20]. The importance of interfacial reactions has become one of the most active study fields. Develop of reaction layers in the reinforcement/matrix interface takes place when applying thermal processes to the composites at temperatures above 300°C. This occurs during the solution treatment in heat treatable aluminum alloys. These heat treatments have an important influence over the matrix-reinforcement interface as shown in a previous work by the authors [22].

The aim of this experimental work is to produce discontinuous AMCs with uniform, un-clustered spatial distributions of the reinforcing phase and assess their microstructural and mechanical properties.

EXPERIMENTAL

This investigation was developed in aluminum matrix composites, employing the alloy AA6061 as metal matrix. It was obtained by atomization of the molten metal by argon. There were used titanium diboride, TiB₂, ceramic particles as reinforcement. Titanium diboride powders were supplied by Advanced Refractory Technologies, INC. The nominal composition of the matrix as well as the reinforcement particles is given in table1. The average reinforcement size was roughly 9.4 μ m for all the composites fabricated, figure 1. It were employed different weight fraction of reinforcement particles: 5, 10 and 15%. The samples were obtained by P/M route: powders were mechanically blended in an alumina lab mixer during 2 hours at 90 rpm (1.5 s^{-1}) . The obtained mixture was uniaxially cold compacted by slowly increasing pressure up 250 MPa. Cylindrical samples with 25 mm of diameter, 34 mm of thickness and 86% of density were achieved. A secondary process of hot extrusion was chosen as the consolidation process. After graphite lubrication and a ram speed of 2 m/s, the samples were hot extruded at 530°C (803K) with a ratio of 25:1. Final composites had a 5 mm diameter and a length of about 400 mm. All the samples fabricated by this route presented a homogeneous distribution of the titanium diboride particles with the surrounding matrix, figure 2.

Heat Treatment

In order to emphasize the diffusion processes between matrix and reinforcement, heat treatments at a fixed time of 1 hour and temperatures of 350, 400, 450 and 500°C (623, 673, 723 and 773K) in a controlled atmosphere, were performed in samples with 5 mm thickness.

To analyze the influence of varying matrix microstructure, on mechanical behavior, another set of heat treatment were carried out. Some extruded samples for tensile tests were cut from the bars, and a T6 heat treatment was performed on them. The T6 heat treatments performed for the composites included a solid solution treatment at 530°C (803K) for 1 hour followed by water quenched. Afterwards, they were artificially aged at 175°C (448K) during 8 hours to reach the peak-aged condition.

Microhardness test

Vickers microhardness tests were carried out in determined zones of different samples to evaluate the hardness differences between matrix, reinforcement and possible reaction products. Tests were preformed in a Matsuzawa MHT2 microhardness tester, on polished samples. The applied charge was 10 gf (9.8 mN) during 15 seconds.

Mechanical Testing

All extruded samples were cut in bars of 5 mm diameter and 60 mm length. They were mechanized with a calibrated dia-

	%Cr	%Fe	%Si	%Mg	%Cu	%C	% O	%B	%AI	%Ti	Table 1. Raw materials composition employed in
Matrix A2 (AA6061)	0.21	0.63	0.63	1.04	0.23	-		_	Bal.	_	composites.
Reinforcement TiB2	•	0.9	-	-		0.8	1.3	30.2	iko <u>i</u> ne moble	Bal.	Tab. 1 Composizione dei materiali base utilizzati nei compositi.



Fig. 1. SEM micrograph of TiB₂ reinforcement powders Fig. 1. Micrografia SEM del rinforzo TiB₂, in polvere.



Fig. 2. SEM micrograph of A2 reinforced with 5 % TiB₂
Fig. 2. Micrografia SEM della lega A2 rinforzata con 5% di TiB₂.

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meter of 4 mm and calibrated length of 20 mm. The tensile test specimen design used in this investigation didn't satisfy the ASTM E-8 [23] requirements of specimen for tension testing because the bar obtained after extrusion was to small. Two tensile tests were carried out in accordance with the E8 ASTM standard, by means of an INSTRON 4204 tensile machine. It was chosen a speed of 5 mm/min (0.75 mm/s) to develop tensile experiments. The average of both tests was taken as the final result. To perform tensile tests at high temperatures, a thermal chamber was connected to the tensile machine. Samples were tested at room temperature in different precipitation state: T1 (state after extrusion) and T6. There also were tested samples at temperatures of 100, 200 and 300°C (373, 473 and 573K) on T6 samples.

Microstructure Characterization

To study the reaction layer developed in the interface matrix/reinforcement, samples were metallographic prepared by conventional techniques. It were taken samples of 5 mm diameter and 2 mm thickness. Microstructure observations were made by optical microscopy (Nikon Microphot FX) and scanning electron microscopy (JEOL 6300) with X-ray microanalysis (Link Isis EDX). With this technique it was possible to determine the microcomponents variation in the reactivity layer.

Once broken, the tensile specimens fracture surfaces were analyzed using a scanning electron microscopy JEOL 6300. Analysis of the fracture surfaces was performed by matching surface fractography, as well as by quantification of the area fraction and particle size distribution of intermetallic particles present on the fracture surface. Observations were made at magnifications of 250X-1000X. Particular attention was directed toward examining for evidence of titanium diboride particle cracking vs. decohesion, and the effects of the matrix microstructure on the fracture morphology.

RESULTS AND DISCUSSION

Heat treatment-reaction layer study

After heat treatment, reaction layer developed in reinforcement/matrix interface was studied in all samples as a func-

tion of temperature. Qualitative microanalysis were performed, figure 3 a) and b) in order to have a notion of the interdiffusion of the microcomponents present in the samples. No evidence of reaction products was observed in samples until a heat treatment temperature of 500°C (773K), where a slight reaction layer was developed in reinforcement/matrix interface, as shown in figure 3. Quantitative microanalyses were performed in different composites to established diffusion profiles of microcomponets. To perform this analysis, there were chosen particles with a similar section to the equatorial theoretic. No variation in profiles of major components was observed in temperatures of 350, 400 and 500°C (623, 673 and 723 K). Analyzing samples with a heat treatment of 500°C (773K) during 1 hour were found intermetallic reaction products. Also, the presence of silicon in the reaction layer was verified. The possible explanation of this fact is: the silicon presents in the aluminum alloy matrix that is no combined with magnesium as Mg₂Si compound, goes to the reaction layer and there, it combines with aluminum and titanium to form intermetallic phases of Ti₂(Al, Si) as indicates the composition of these elements at the reaction laver.

Taken in account the weight fraction of reinforcement employed, high reactivity between reinforcement particles and matrix was found for low contents of reinforcement. The presence of more reinforcement particles, make difficult interdifusion of microcomponents and less reaction products can be found.

Microhardness evolution

As commented before, a slight reaction layer of intermetallics is formed between matrix and reinforcement at high temperatures. In order to determine the relative resistance of the different phases present in the samples, microhardness tests were made in determinate samples. It was easy measure the microhardness of the matrix. For the reinforcement, due to its small size, measuring these values was more difficult but still possible. In the case of the reaction layer formed the microhardness value could not be taken. The results are shown in figure 4. It can be observed that the reinforcement ceramic particles are much harder than the aluminum matrix.

Fig. 3. Qualitative microanalysis of the reaction layer developed in A2 reinforced with 5% TiB₃, after heat treatment at 500°C during 1h. (a) SEM micrograph (b) EDX analysis. Microcomponents evolution in the reinforcement particle, reaction layer and matrix.

Fig. 3. Microanalisi qualitativa dello strato di reazione sviluppatosi nella lega A2, rinforzata con 5% TiB₂,dopo un trattamento termico di 1h a 500°C. (a) Micrografia SEM (b) analisi EDS.

Fig. 4. Optical micrograph showing Vickers identation; A2 matrix reinforced with 5% TiB₂, after 1 h of heat treatment at 500°C (773K). Microhardness values for matrix and reinforcement.

Fig. 4 Micrografia ottica di un'impronta Vickers; matrice A2 rinforzata con 5% TiB₂, dopo un trattamento termico di 1h a $500^{\circ}C$ (773 K). Valori di microdurezza per matrice e rinforzo.



Tensile tests

A representative stress vs strain plot obtained from a tension test is shown in figure 5. The 0.2% offset yield strength σ_v , the ultimate tensile strength σ_{μ} and the strain to failure ϵ_{μ} were calculated from this plot and the calculated values of the tensile properties are listed in table 2 and 3. Table 2 summarizes the tensile properties obtained in both the unreinforced and composite materials at different precipitation state (T1 or T6) and tested at room temperature of about 22°C (295K). In table 3, are listed the obtained results for the samples tested at T6 state and high temperatures of 100, 200 and 300°C (373, 473 and 573K).

To have a clear understanding of the calculated values, some graphs were drawn. Figures $\overline{6}$, 7 and 8 show the evolution of tensile parameters as a function of precipitation state of the matrix, for the unreinforced material as well as some reinforced samples. It was found that the yield strength, fi-

gure 6, as well as the ultimate tensile strength, figure 7, of both monolithic alloy and the composite improved considerably after the artificial aging as a consequence of the presence of small intermetallic precipitates. Coherent precipitates increase the material flow strength trough the wellknown mechanism of dislocation-precipitate interaction. The elongation variation with heat treatment, figure 8, was more complex.

The weight fraction of TiB₂ particles was found to have significant effect on tensile properties of the matrix.

This is illustrated in figure 9 and 10, which represent the variation of the three tensile values calculated: the offsets yield strength, the ultimate tensile strength and elongation with weight fraction of TiB, reinforcement particles. A single one of all these graphs exposed, with two or three data points, would be inadequate to draw even a trend line; however since the data trends are monotonic in the same sense for all

> Table 2. Tensile properties at room temperature for several studied composites.

Tab. 2 Proprietà tensili a temperatura ambiente di alcuni dei compositi studiati.

Sample	Precipitation State	(MPa)	σ (MPa)	е (%)
A2	TI T6	197.6	100	23.76
A2 + 5% TiB2	TI T6	210.05	82.14 119.63	12.3 14.66 8.66
A2 + 10% TiB2	T1 T6	224.76 397.02	90.28 113.39	13.4 16.2
A2 + 15% TiB2	TI T6	316.99	- 116.19	21.45

Sample	(°C)	о (MPa)	σ (MPa)	е, (%)	
A2	100	350		-	
	200	285		-	
	300	200		-	
A2 + 5% TiB2	100	212.99	126	18.54	
	200	210.21	121.42	15.43	
	300	127.21	74.28	6.62	
A2 + 10% TiB2	100	201.66	120.2	19.20	
	200	263.05	117.85	7.92	
	300	141.39	88	10.25	
A2 + 15% TiB2	100	356.95	117.73	15.72	
	200	268.24	114.28	9.29	
	300	122.72	70.77	3.11	



Tab. 3 Proprietà tensili ad alta temperatura di alcuni dei compositi studiati.



Fig. 5. Representative tensile curve for A2 alloy reinforced with 10% TiB, in T1 state.

Fig. 5 Tipica curva di trazione per una lega A2 rinforzata con 10% TiB_{2} , nella condizione T1.



Fig. 6. Differences on ultimate tensile strength σ_{μ} for several materials as a function of matrix precipitate state.

Fig. 6. Differenze di carico a rottura per vari materiali in funzione dello stato di precipitazione della matrice.

ALLUMINIO E LEGHE



Fig. 7. Differences on offset yield strength σ_y for several materials as a function of matrix precipitate state

Fig. 7. Differenze del carico di snervamento per vari materiali in funzione dello stato di precipitazione della matrice.



Fig. 9a. Evolution of tensile parameters as a function of weight fraction reinforcement. A2 reinforced with 5 and 10% of TiB_2 in T1 state.

Fig. 9a. Variazione delle caratteristiche meccaniche in funzione della frazione in peso di rinforzo. Matrice A2, con 5 e 10% di TiB_2 nella condizione T1.

graphs, they are presented as a guide to trends. The ultimate tensile strength slightly increases with volume fraction of composite, until reinforcement content of 10% of ceramic particles. After that, lower values are achieved by 15% weight fraction in the studied T6 state. The addition of the ceramic particles improves the strength mainly by the load transfer from the matrix to the reinforcement due to the differences in the elastic constants. Offset yield strength was found to fall down with the addition of reinforcement. The reinforcement content seemed to not influence the offset yield strength value. Regarding the elongation, it can be said that at TiB, content of 5% the elongation values were the lowest in both precipitation states of the matrix (T1, T6). In T1 state, the elongation value was found to decrease when increasing TiB, content. Nevertheless, analyzing material in T6 state, other tendency was observed. When the TiB, surpasses 5% content, the elongation increased gradually, reaching superior values even to ones of the matrix. The more uniformly TiB₂ particles distribute the higher elongation of the composite. So better distribution and less particle aggregates for the high reinforcement contents, can be the explanation of these higher elongation values found.

Figure 10 shows the mechanical behavior of studied composites when testing at high temperatures, by represented the



Fig. 8. Differences on elongation \mathcal{E}_{f} for several materials as a function of matrix precipitate state

Fig. 8. Differenze di allungamento a rottura per vari materiali in funzione dello stato di precipitazione della matrice.



Figure 9b. Evolution of tensile parameters as a function of weight fraction reinforcement. A2 reinforced with 5, 10 and 15% of TiB_2 in T6 state.

Fig. 9b. Variazione delle caratteristiche meccaniche in funzione della frazione in peso di rinforzo. Matrice A2, con 5,10, 15% di TiB2 nella condizione T6.

ultimate tensile strength.

Similar trend was achieved for unreinforcement alloy and composite tested. When higher temperatures were employed, important decreases of tensile properties were achieved, mainly owing to an atomic relax of the material that favor the dislocations movement. The effect of temperature on tensile properties was much more aggressive for the composites, where lower tensile values were reached. It was observed the similar behavior for 5 and 15% reinforcement contents.

Tensile values undergo ever below matrix tensile values, whatever temperature. Samples with 10% reinforcement content, as shown above, presented at room temperature higher tensile values to those of the matrix. Nevertheless, when tensile tests were performed at high temperatures a rapid decrease of strength appeared and at 100°C (373K) the founded values were very similar to those of the matrix. Increasing the temperature caused its behavior was the same as the other reinforcement contents and lower tensile values were achieved.

Some fractographic analysis were performed on fractured samples, figure 11 and 12. The composite fracture surfaces exhibited microscopically a ductile appearance by means of matrix/reinforcement interface (dimples), and a fragile frac-



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Fig. 10. Evolution of ultimate tensile strength as a function of temperature for matrix and A2 reinforced with 5, 10 and 15% of TiB_2 particles. All samples in T6 precipitation state.

Fig. 10. Variazione del carico a rottura in funzione della temperatura per la matrice A2 e con 5, 10, 15% di TiB_2 nella condizione T6.



Fig. 11. Fracture surface of the A2 matrix at T1 precipitation state. Fig. 11. Superficie di frattura della matrice A2 nella condizione di precipitazione T1.



Fig. 12. Fracture surface of the A2 reinforced with: a) 5% TiB_2 and b) 10% TiB_2 ; both in T1 precipitate state. Fig. 12. Superficie di frattura di A2 con: a) 5% TiB_2 e b) 10% TiB_2 , entrambe nella condizione di precipitazione T1.

ture of the particulates. The dimples were due to the aluminum matrix fracture, figure 11. It was also observed, that fracture did not growth by particles, figure 12, that remain its initial shape after fracture. The fracture grows by the matrix/reinforcement interfaces.

CONCLUSIONS

As a brief summary of all the previously exposed, it can be indicate:

- No evidence of reaction products was observed in samples until a heat treatment temperature of 500°C. At this temperature were found second intermetallic phases at the reinforcement/matrix interfaces, formed mainly by Ti₃(Al, Si) intermetallic.
- It was not possible to measure microhardness value of reaction layer formed due to its small size. It could be. The greater hardness of the reinforcement could be corroborated by microhardness analysis.
- It was found an improvement of the ultimate tensile strength with an artificial aging (T6), in all the samples studied. However, low offset yield strength and elongation

values were achieved.

- A major improvement of mechanical behavior was assessed by composites reinforced with 10% TiB₂ due to a better distribution of the reinforcement phase in the matrix.
- At high temperatures, important decreases of tensile properties were achieved, mainly in the reinforced matrix. The presence of reinforcement particles gave worse tensile values.
- The elongation is important in all cases, so fracture surfaces exhibited microscopically a ductile appearance by means of matrix/reinforcement interface (dimples), growing the fracture by means of this interface. Reinforcement particles conserved its shape after fracture.

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MICROSTRUTTURA E COMPORTAMENTO MECCANICO DI LEGHE Al-Si-Mg RINFORZATE CON PARTICELLE TIB,

Sono stati preparati in laboratorio materiali compositi a matrice metallica in lega di alluminio di rinforzata con particelle di TiB₂ mediante un metodo di metallurgia delle polveri. La miscela di polveri è stata pressata e, dopo lubrificazione, è stata utilizzata l'estrusione a caldo come processo di consolidamento. Si è ottenuta una distribuzione omogenea delle particelle di rinforzo. Sono stati effettuati diversi studi sui compositi sviluppati.

Durante la fabbricazione e i successivi trattamenti termici dei compositi se la matrice è una lega ad alta resistenza trattabile a caldo, è possibile che possano apparire prodotti di reazione e interfasi. Gli effetti dei rinforzi TiB, sulla formazione di uno strato di reattività dei composti a matrice metallica di Al TiB./6061 sono stati studiati. Per valutare gli eventuali prodotti di reazione e intefasi fra matrice e rinforzo sono stati effettuati trattamenti termici per un tempo fissato a diverse temperature (350, di 400, di 450 e di 500°C). Le prove sono state efffettuate sia sulla matrice di alluminio che sui MMC ottenuti, allo scopo di valutare i cambiamenti dovuti alla presenza di rinforzo. In questo studio sono state utilizzate la microscopia elettronica a scansione e ottica con microanalisi a raggi X, per determinare gli eventuali prodotti di reazione all'interfaccia matrice/rinforzo e la relativa composizione.

Sono state effettuate prove di microdurezza su campioni lucidati per valutare il comportamento meccanico. In seguito è stata analizzata l'evoluzione di microdurezza Vickers attraverso lo strato di reazione formato nei diversi materiali studiati.

Sono stati studiati il carico massimo e la deformazione a rottura.

Sono state effettuate prove meccaniche a temperatura ambiente e a temperature elevate del materiale allo stato estruso. E' stato investigato il tipo di danneggiamento mediante microscopia a scansione elettronica, osservando gli effetti nelle superfici dei campioni. Sono state considerati i cambiamenti microstrutturali dovuti alle tensioni alle quali i campioni sono stati sottoposti. A questo scopo sono stati esaminati mediante microscopia a scansione elettronica tagli longitudinali, nella direzione delle deformazioni. Sono state indicate le seguenti conclusioni:

- Non è stata osservata alcuna evidenza di prodotti di reazione nei campioni fino ad una temperatura di trattamento termico di 500°C. A questa temperatura sono state rilevate seconde fasi intermetalliche all' interfaccia rinforzo/matrice, formate pricipalmente da intermetallici Ti₃(Al, Si).
- Non è stato possibile misurare il valore di microdurezza dello strato di reazione formatosi a causa della sua piccola dimensione. La maggiore durezza del rinforzo potrebbe essere confermata da analisi di microdurezza.
- Si è riscontrato un miglioramento della massima resistenza alla trazione con un invecchiamento artificiale (T6), in tutti i campioni studiati. Tuttavia, a ciò sono corrisposti minor carico di snervamento e minor allungamento.
- Si è evidenziato un considerevole miglioramento del comportamento meccanico nei compositi rinforzati con TiB₂ al 10% dovuto a una migliore distribuzione della fase di rinforzo nella matrice.
- Alle temperature elevate sono state ottenute notevoli diminuzioni delle proprietà tensili, soprattutto nella matrice rinforzata. La presenza delle particelle di rinforzo ha comportato un peggior comportamento meccanico.
- L'allungamento è notevole in tutti i casi, quindi le superfici di frattura hanno mostrato al microscopio un aspetto duttile dell'interfaccia matrice/rinforzo (dimples), in cui la frattura si sviluppava per mezzo di questa interfaccia. Le particelle di rinforzo hanno conservato la loro forma dopo la frattura.