EFFECT OF THE TiC PARTICLE SIZE ON THE EXTRUSION OF 7075 ALUMINIUM MATRIX COMPOSITE

Sylvia Cruz [a], Pilar Rey [a], Marta Cabeza [b], Marcela Lieblich [c], Pedro Merino[b]*

[a]*AIMEN Technology Center, Advanced Materials, C/Relva 27A, 36410. Porriño, Spain*
[b]*Escuela Técnica Superior de Ingenieros Industriales, Universidad de Vigo, Departamento de Ingeniería de los Materiales, Mecánica Aplicada y Construcción. Campus Universitario Lagoas-Marcosende, 36200. Vigo, Pontevedra, Spain.*
[c]*Centro Nacional de Investigaciones Metalúrgicas (CENIM), Centro Superior de Investigaciones Científicas (CSIC). Avda. Gregorio del Amo 8, 28040. Madrid, Spain.*
**e-mail address: prey@aimen.es*

Keywords:7075 matrix composite, extrusion, micrometric and nanometric particle reinforcement, TiC.

Abstract

In this study aluminium matrix composites were produced with pre-alloyed AA7075 powders and TiC nanometric and micrometric reinforcement particles by mechanical alloying and extrusion in laboratory and pilot plant using circular and L dies respectively. The aim of this work is to evaluate the effect of the process together with the TiC reinforcement and particle size on the microstructure and mechanical properties of the AlMMC. Mechanical alloyed samples show better mechanical properties and microstructure than the unprocessed prealloyed sample, as a consequence of the homogenization of the milling process. This effect is stronger than the addition of the reinforcement particles. The MA 7075+2%TiC nano sample shows better properties than its corresponding micro counterpart due to the fact that nanoparticles are more effective reinforcing agent as a consequence of a homogeneous dispersion of nano-TiC, allowing the effectively reinforcement of the matrix.

1. Introduction

7075 Aluminium alloys (Al-Mg-Zn-Cu) have been widely used as structural materials in aeronautic industry due to their attractive properties. In the last years ceramic reinforced aluminium metal matrix composites (AlMMCs) have acquire a great interest due to their improved properties as specific strength, stiffness, wear, fatigue and creep behaviour if compared with those of monolithic aluminium [1[3]

Initially SiC, B_4C and Al_2O_3 were used as ceramic reinforcements; recently NiAl, Ni_3Al, $MoSi_2$, TiB_2 and TiC have been shown to improve the wear resistance of aluminium and magnesium alloys [2, 3[3]]. TiC is recently investigated as a ceramic reinforcement in Al alloy matrix nanocomposites due to their high melting temperature, low thermal coefficient of expansion, chemical inertness and excellent wear and abrasion resistance [4, 5].

The size of reinforcement particle is known to affect hardness, wear and corrosion resistance. Both tensile strength and ductility decrease with increasing particle size, therefore, decreasing the ceramic particulate size can lead to substantial improvements in mechanical performance of MMCs, especially in the case of using nanoparticles [6-10].The extent of property improvement of MMCs at a given particle chemistry, size and volume fraction is related to the homogeneity of the particle distribution [11]. Particularly, fatigue crack initiation is largely dependent on clustering of particles. However, homogeneous dispersion of ceramic nanoparticles in metals is difficult to achieve [12].

These composites are synthesized by liquid or powder metallurgy routes. Mechanical alloying (MA) is a well-developed process for dispersing ex-situ nanoparticles uniformly in metal matrix [5].It has been successfully employed in this work to increase the quality of particle distribution in aluminium, showing that it should become an essential step within the powder metallurgy (PM) production route for manufacturing nano-AlMMCs [13-15].

The aim of this work is to evaluate the microstructure and the mechanical behavior of TiC reinforced AlMMC obtained by mechanical alloying and hot extrusion process both by laboratory and pilot scale, establishing the effect of the reinforcement and its particle size.

2. Materials and testing methods

Pre-alloyed 7075 powder aluminium alloy (90,53 Al; 5,14 Zn; 2,51 Mg; 1,46 Cu; 0,21 Cr;0,071 Fe y 0,024 Si) supplied by ALPOCO, with particle size nominally sub 90 µm and average size of 30 µm was used. TiC particles with purity of 99% and average particle size of 20 nm and sub 50 µm were used as reinforcement supplied by Iolitec (nano) and Kennametal INC (micro) respectively.

The composite materials investigated were prepared by a powder metallurgical route. A cyclic process of mechanical alloyed (MA), typical in ductile materials, was carried out with a ZOZ Simoloyer CMO1 attritor mill, during 480 minutes, with 10:1 relation between mass of powder and balls, in presence of argon atmosphere and Licowax as process controller agent (PCA).The powder composition was adjusted for 2% weight either for TiC micro and nanometric reinforcement. The details of the process were described before [15]. The resulting powder composites are identified as: 7075- prealloyed, MA 7075, MA 7075+2%TiC micro and MA 7075+2%TiC nano.

The powder blend is extruded with two different geometries and routes, laboratory and pilot plant scale. In the laboratory route, the powder is uniaxially cold pressed at 370KN during 15 minutes and extruded at 480ºC and 2 mm/s, without caning and degassing, with a cylindrical geometry die (extrusion ratio 5:1).
For the pilot plant tests, a horizontal lab-scale computerized extrusion press, 300 ton capacity, was used for this work. The powders were encapsulated in 6063 cans and consolidation was achieved during the extrusion process. The extrusions were conducted at a temperature of 480ºC, an extrusion ratio of 37:1 and extrusion speed of 2 mm/s. A die with L geometry (17.7 mm length and 1.2 mm width) provided by Iberia Dies Phoenix, Zaragoza, Spain was used.

The microstructure of the cylindrical composites were examined in transversal section by light optical microscopy –LOM-(GX51 Olympus microscope) and FEI Field Emission Gun Scanning Electron Microscope (FEI Helios Nanolab) equipped with electron back scattered diffraction (EBSD) detector at the CACTI laboratories of the University of Vigo, Spain. For the EBSD measurements, the FESEM were operated at 20keV and a step size 0.04 µm-0.08µm. The grain size was calculated from EBSD data at different areas –border and central-in the transversal section in order to determinate the homogeneity of the samples (ASTM E 2627-13)

The tensile test of the L aluminium profiles was performed on a MTS 250 kN test machine according to UNE-EN ISO 6892-1:2010. The tensile direction is longitudinal, and parallel to extrusion direction.0.1 kg Vickers hardness tests were performed in a Durascan Emcotest durometer according to UNE-EN-ISO 6507-1:2006.

3 Results and Discussion

All the laboratory extruded samples were obtained with surface quality and without appreciable defects. Figure 1 shows the microstructure of the samples after extrusion at the laboratory scale. It seems to be homogeneous and free of defects. The aluminium grains in the milled materials with and without reinforcement were more difficult to visualize than in the unprocessed 7075-prealloyed. In the pre-alloyed sample the grain size is both bigger and more heterogeneous, alternating greater grains with smaller ones. It is a consequence of the initial microstructure that have non-processed aluminium particles. On the other hand, Figure 1-C shows that TiC micro particles appear sometimes as agglomerates with a smaller size (10 µm) than the initial. The Figure 1-D shows a homogeneous microstructure and no grain or particle of TiC is observed, probably due to the size –submicron-. Figure 2 and Table 1 show that prealloyed 7075 presents a fully dendritic microstucture with alloying elements (Mg, Cu and Zn) segregated into the inter-dendritic spaces. Mechanical alloying is able to homogenize the microstructure although reinforcement particles are not added.

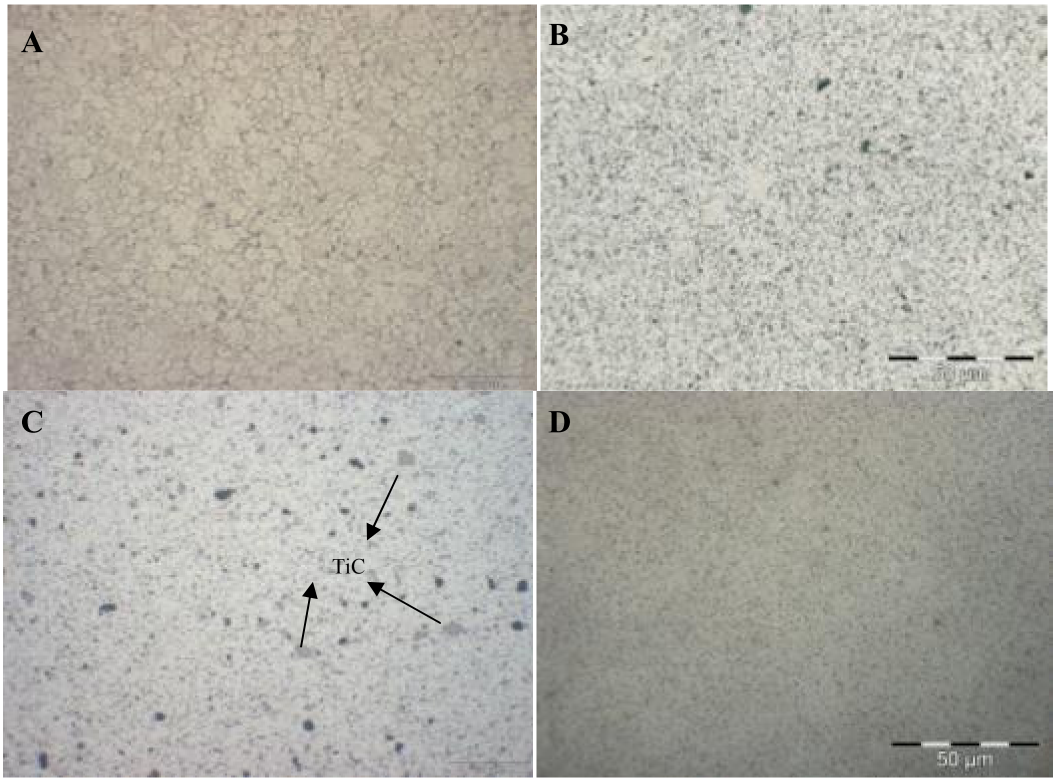

Figure 1. Light Optical Microstructure of the samples A) 7075 prealloyed B) MA 7075 C) MA+2%TiC micro D)MA+2%TiCnano. Etching reactive: Keller's (2.5ml Acetic Acid, 0.5ml de HNO_3 and 47ml H_2O)

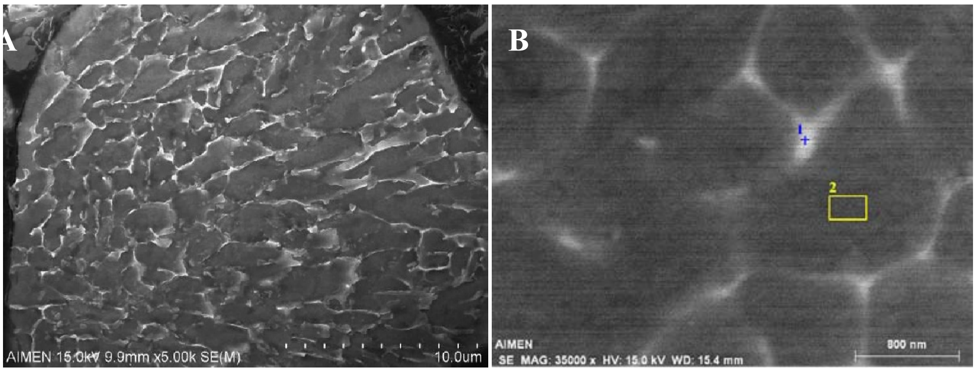

Figure 2. SEM micrographs of A) the dendritic microstructure of the original 7075 aluminium alloy and B) detail showing the zones where EDS analysis were made.

Spectrum	**O**	**Mg**	**Al**	**Cu**	**Zn**
1	0,74	5,62	77,11	5,58	10,94
2	0,65	2,49	90,69	0,77	5,41

Table 1. EDS analysis for the 7075-pre-alloyed.

EBSD maps were obtained at the centre and border for the transversal cross sections of the laboratory cylindrical compacts, extruded from the samples MA 7075 and MA 7075+2%TiC nano and are shown in Figure 3.The mean grain size of the cross sections calculated from EBSD maps are in the Table 2.

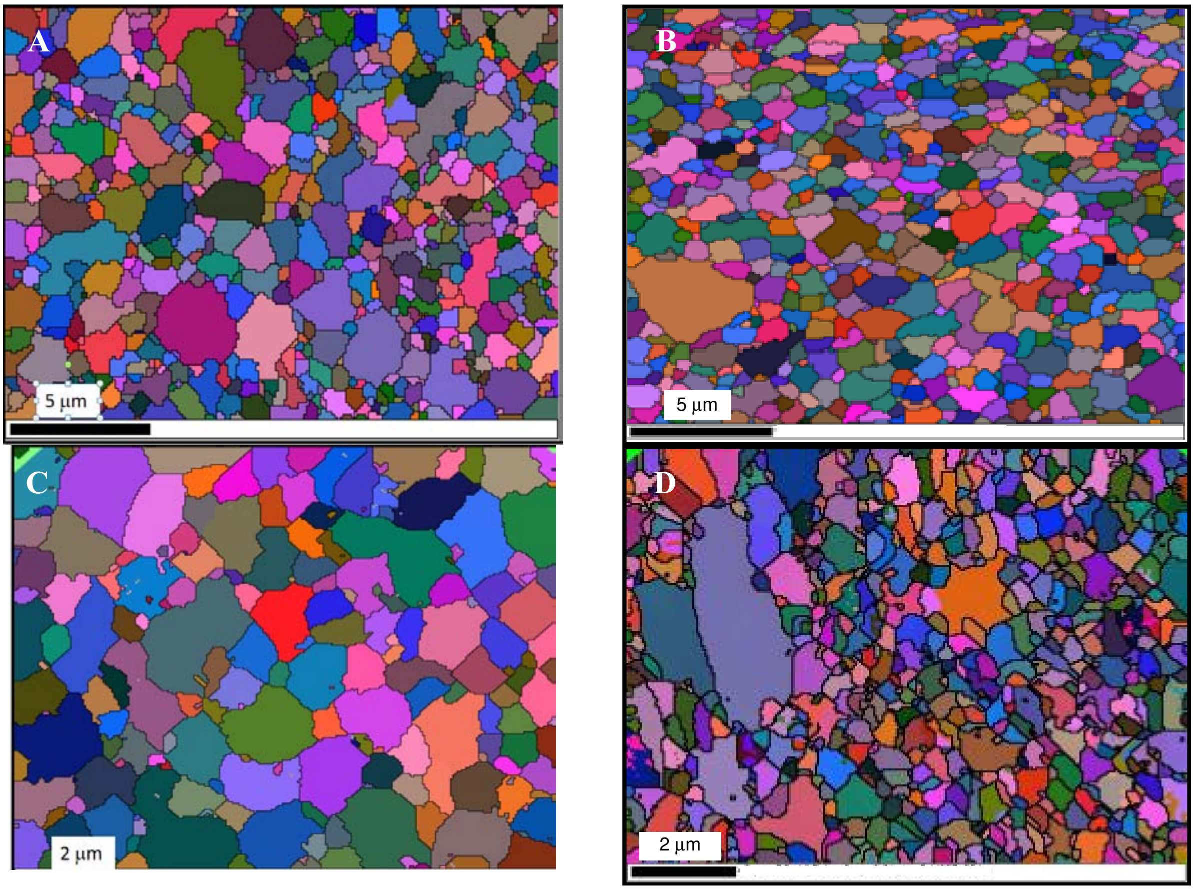

Figure 3. EBSD Images at the border and centre of the samples A) MA 7075-border B) MA 7075-Centre C) MA 7075+2%TiC nano. D) 7075+2%TiC Centre.

Both mechanically alloyed samples, with and without reinforcement, show fine and equiaxed grains. However, the presence of TiC nanoparticles promotes different velocity in the extrusion process due to the more friction with the walls because of the higher hardness of the reinforced alloy. As a consequence, the grain size at the center of the MA 7075+2%TiC nano is nearly half than in the border as can be seen in Table 2.

	Border/nm	**Centre/nm**
MA 7075	659 ± 15	658 ± 9
MA 7075+2%TiC nano	537 ± 12	371 ± 5

Table 2. Grain size –Equivalent circle diameter- from EBSD data.

The extruded L profiles of 7075-prealloyed, MA 7075, MA 7075+2%TiC nano and MA 7075+2%TiC micro are shown in the figure 4.

Figure 4. L aluminium profiles extruded in pilot plant

It can be seen that 7075 pre-alloyed sample present serious defects as a consequence of the temperature. As we said previously, unprocessed aluminium particles show a dendritic microstucture with alloying elements segregated into the inter-dendritic spaces. This kind of microstructure promotes the local melting of segregated intermetallics, generating an unusable profile. On the other hand, the mechanical alloyed samples only show small tearing defects on the surface due to the homogenization of the alloying elements with the matrix as a consequence of the high energy of the process. These defects are not appreciated in the laboratory extruded samples due to the lower extrusion ratio and the geometry of the die.

The hardness, tensile and yield strength, elongation and elastic modulus of the L profiles are presented in Table 3.

	Vickers Hardness	**Ultimate tensile strength (MPa)**	**Yield Strenth (MPa)**	**% Elongation**	**Elastic modulus (GPa)**
7075-prealloyed	79	---	---	---	---
MA 7075	133	439	353	10	74.7
MA 7075+2%TiC nano	140	463	372	9	78.7
MA 7075+2%TiC micro	137	442	354	9	75.6

Table 3. Mechanical properties of the L-profiles extruded at the pilot plant scale.

The hardness and tensile and yield strength of the mechanically alloyed samples are significantly higher than those of the 7075 prealloyed material. On the other hand, the composites show hardness and mechanical properties better than those of the unreinforced

mechanically alloyed sample. In addition, the MA 7075+2%TiC nano shows better properties than its micro counterpart, with the same elongation. Again, elastic modulus is higher for the mechanical alloyed reinforced samples, with the best value for the MA 7075+2%TiC nano. Nanoparticles are more effective reinforcing agent as a consequence of a homogeneous dispersion of nano-TiC in the powder observed in previous work [15], allowing the effectively transference of the applied load and reinforcement of the matrix due to a nanoparticle-dislocation interaction by means of the Orowan bowing mechanism. In the MA 7075+2%TiC micro, the agglomerations of TiC promote a strength reduction compared with the nano-reinforced counterpart and only a slight improvement respect to the MA 7075.
Although only hardness values for 7075 prealloyed are shown, it can be affirmed that the largest increase in performance and mechanical properties is due to the mechanical alloying process that promotes homogenization of the prealloyed particles and a grain size reduction more than the addition of reinforcement particles.

Conclusions

Al/TiC composites were produced using a powder metallurgy technique and extrusion at laboratory and pilot plant scales and their microstructure and mechanical properties were studied. The most important results are the following:

- 7075 prealloyed sample shows a heterogeneous microstructure in the lab extruded samples as a consequence of its dendritic microstructure with alloying elements segregated at the interdendritic spaces. This microstructure generates serious defects in the L profile extruded samples at pilot plant, preventing that tensile tests could be done.
- Mechanical alloying process promotes homogenization of the microstructure, even in samples without reinforcement and promotes a significantly reduction in the grain size.
- EBSD maps reveal significant differences in the grain size between the border and centre for the MA 7075+2%TiC nano sample as a consequence of the higher stress and friction caused by the TiC reinforcement particles.
- Mechanical alloyed samples show better mechanical properties than the unprocessed prealloyed sample. However, largest increase in performance and mechanical properties in processed samples is due to the mechanical alloying process effect more than the addition of reinforcement particles
- Hardness and mechanical properties for the reinforced samples are larger than for the unreinforced mechanically alloyed sample
- The MA 7075+2%TiC nano sample shows better properties than its corresponding micro counterpart due to a good distribution of the reinforcement nanoparticles.

Acknowledgments

The authors gratefully acknowledge the financial support from the *Xunta de Galicia* under program *INCITE* for the development of the project 09TMT001CT.

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