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Application of ball milling in the synthesis of AA 7075–ZrO₂ metal matrix nanocomposite

S.E. Hernández-Martínez ^{a,*}, J.J. Cruz-Rivera ^a, C.G. Garay-Reyes ^b, C.G. Elias-Alfaro ^a, R. Martínez-Sánchez ^b, J.L. Hernández-Rivera ^c

^a Instituto de Metalurgia, Universidad Autónoma de San Luis Potosí, Sierra Leona # 550, Unidad de Posgrados, San Luis Potosí, Mexico

^b Centro de Investigación en Materiales Avanzados CIMAV, Av. Miguel de Cervantes # 120, Complejo Industrial, Chihuahua, Mexico

^c Instituto de Metalurgia, Universidad Autónoma de San Luis Potosí-Cátedras CONACyT, Sierra Leona # 550, Unidad de Posgrados, San Luis Potosí, Mexico

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ABSTRACT

Ball milling was used to reduce the particle size of yttria-stabilized ZrO₂ powders with the aim of introducing it into the AA 7075 matrix. It was observed that during the mechanical milling of the ZrO₂ powders a destabilization of the tetragonal structure occurred, which was confirmed by the X-ray diffraction results. A transition from tetragonal to monoclinic structure resulted as a consequence of the application of ball milling. The synthesis of the composite was carried out in the same way by applying mechanical ball milling in order to disperse 2 and 5 wt.% of zirconium oxide particles into the Al alloy matrix. It was found that longer times were needed for the 5 wt.% of ZrO₂ to achieve a complete dispersion into the matrix, unlike 2 wt.% of ZrO₂ sample, being 15 and 10 h, respectively. It was evident that the presence of ZrO₂ particles had a marked influence upon the powder morphology and particle size of the matrix during the milling process. Despite the structural change observed, a relatively good homogenous dispersion of ZrO₂ particles was found after ball milling.

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1. Introduction

Composite materials have gained great importance in the last years due to enhanced mechanical properties, which are obtained by combining different materials, such as metallic alloys and ceramics, which could be oxides, borides, nitrides and carbon nanotubes [1-4]. Aluminum alloys are of special interest to be used as matrix in composites due to their low specific weight, which is why they are very popular in the aeronautic and aerospace industry, especially the 2XXX, 6XXX, and 7XXX series, which can be heat treated in order to improve their strength. Among the mentioned alloys, the 7XXX series, whose main alloying elements are Zn and Mg, exhibit the greatest response to the precipitation hardening, thus, the AA 7075 is considered a high strength alloy because its yield strength is above 500 MPa in the optimal aging conditions [5,6]. This fact has motivated the investigation about how AA 7075 can be modified by introducing nanoparticles of partially yttria-stabilized ZrO₂ (hard and chemically stable ceramic) with the aim to obtain adequate oxide dispersion strengthening (ODS) by using ball milling processes.

Previous studies have used ZrO₂ as reinforcement with the aim to improve the properties of the alloy. Fuentes-Ramirez et al. [7] improved the wear resistance of pure Al, using a surface treatment employing

E-mail address: sergioeliseohernandezmartinez@hotmail.com

(S.E. Hernández-Martínez).

colloidal ZrO₂. In other research, Hemanth [8] used nanosized ZrO₂ to modify LM13 cast aluminum alloy, obtaining superior properties of hardness and tensile strength in comparison with the unreinforced alloy. In the same way, Abdizadeh et al. [9] incorporated partially yttria-stabilized ZrO₂ particles into an A365 Al alloy using the vortex method. The results showed a hardness of 70 HBN and a tensile strength of 240 MPa; in comparison, the unreinforced matrix exhibited 45 HBN and 45 MPA, respectively.

One important fabrication route of composites, is the mechanical milling (MM) process, since the dispersion of the reinforcement is carried out in solid state, unlike casting routes, where the density difference between the matrix and the reinforcement commonly makes difficult to obtain a homogeneous dispersion of the later [2,3]. The MM process consists in repetitive collisions of the milling media (vials and grinding balls) with the matrix and the reinforcement particles. The process refines the structure of the ductile matrix powders because of the repetitive steps of deformation, welding, and fracture. On the other hand, the brittle ceramic particles mostly fracture during the MM and remain trapped between the matrix powders [10]. Nevertheless, reinforcement can form small clusters, generating a nonhomogeneous microstructure, a situation which adversely affects the expected mechanical properties of the composite [11]. Therefore, to obtain a desirable microstructure and to improve mechanical properties it is essential for all the particles to be homogeneously distributed in the mixture [12], Kuruvilla et al. [13] argued that strengthening observed in an AlTiB₂ based composite is mainly attributed to fine dispersion of







^{*} Corresponding author. Tel.: + 52 4442615515.





the high-modulus TiB₂ particles which are well bonded to the matrix, Kang et al. [14] showed that the major strengthening mechanism in aluminum matrix composites reinforced with Al₂O₃ nanoparticles is the Orowan strengthening by ceramic particles; however, he found that when clusters of these ceramic particles were present, the particle strengthening effect diminished.

This is the reason why the parameters of the MM process need to be studied and optimized in order to obtain a fine dispersion of partially yttria-stabilized zirconia through the AA 7075 matrix by using mechanical ball milling and to characterize its microstructural evolution through the process.

2. Materials and experimental procedure

The matrix powders were obtained from a commercial bar AA 7075-T6. The bar was annealed for 48 h at 415 $^{\circ}$ C \pm 5 $^{\circ}$ C in a Felisa furnace followed by a slow cooling in order to remove the high hardness caused by the T6 heat treatment. During this heat treatment it is promoted the presence of stable η (MgZn₂) phase instead of η' (MgZn), lowering the hardness of the alloy by this way. The matrix powders were obtained by

Table 1	
Average particle size of ZrO ₂ powders after ball milling with two milling media.	

Milling time	Average particle size of ZrO ₂ (nm)		
Hours	Balls	Balls and rods	
0	37,000 (±11,000)	37,000 (±11,000)	
33	134 (±53)	-	
80	110 (±45)	142 (±63)	
140	108 (±45)	123 (±43)	

grinding the bar and then they were sieved with a 50 size mesh. On the other hand, the yttria-stabilized zirconia powders were mechanically milled to reduce their average size in a Fritsch Pulverisette 5 highenergy planetary ball mill at 180 RPM for 33, 80, and 140 h using methanol as a process control agent (PCA). Two milling media were used in processing this reinforcement: the first consisted of zirconia balls and the second consisted of 50-50 wt.% of zirconia balls and rods. Balls-to-



Fig. 2. Effect of milling time on X-ray diffraction patterns of ZrO₂.

Table 2

Effect of milling time on the content of crystalline structures of ZrO₂.

Structure →	Monoclinic		Tetragonal		Cubic	
Condition ↓	Crystallite size (nm)	wt.%	Crystallite size (nm)	wt.%	Crystallite size (nm)	wt.%
ZrO ₂ as received ZrO ₂ 80 h of milling	32 25	44.1 65.7	55 49	47.5 16.9	77 66	8.3 17.2

powder weight ratio was 20:1 in the two modes. According to the results of average particle size of ZrO₂, 80 h were selected as the optimum time for milling the reinforcement because in shorter times the average particle size was higher, and further processing time did not show significant reduction in particle size. The composite was produced by milling the AA 7075 powders with 2 and 5 wt.% yttria-stabilized zirconia particles in a planetary ball mill at 120 RPM using stainless steel balls and vials. Balls-to-powder weight ratio was set at 8:1.

The morphology, size and microstructure of matrix powders were characterized at 2, 5, 10 and 15 h by scanning electron microscope (SEM) Phillips XL-30 operated at 20 kV and the images were acquired with backscattered electrons (BSE) detector, also the SEM was equipped with an energy dispersion spectrometer (EDS). The reinforcement distribution was evaluated gualitatively in a transmission electron microscope (TEM) JEOL 1230 operated at 100 kV. X-ray diffraction tests were obtained in a Rigaku diffractometer in the 2θ range of 25-85° using Cu radiation K α ($\lambda = 0.154$ nm) with a step-size of 0.02° and speed of 1°/min. Average crystallite size of matrix was obtained from the XRD peaks broadening when the diffractograms were refined and analyzed using MAUD software. The structural parameters that were refined by this way were diffractogram background, texture and strain. Iterative refinement finished when the indicators sigma and Rw converged to acceptable values according to Ferrari and Lutterotti [15]. This software was developed to perform a simultaneous refinement of material structure described through the measurement of lattice parameters, phase contents, crystallite size, microstrain and crystallographic texture. The Rietveld method basically consists of fitting a large portion of the experimental X-ray diffraction pattern with analytical functions to describe the profiles whose intensity is directly related to the structure of each studied phase [16].

3. Results and discussion

3.1. ZrO₂ mechanical ball milling process

The objective of milling ZrO_2 powders was to reduce its average particle size. Initially this parameter was 37 µm (Fig. 1a). As it was mentioned in the experimental section, two variants of milling media were used with the purpose of comparing the convenience for each one. At first sight, the media exclusively composed of balls produced better results than the mixture of balls and rods, since the average particle size for 140 h of milling was 108 nm in the case of the former, and 123 nm after 140 h for the latter. These results can be seen in Table 1.

The convenience of using the ZrO_2 particles milled by 80 h employing only balls is evidenced in the micrograph shown in Fig. 1b. Since there is no great difference in the average particle size between the samples milled for 80 and 140 h, it was decided to use ZrO_2 powder milled for 80 h employing only balls to incorporate it to the composite material. The ZrO_2 is a polymorph material having 3 crystalline structures. At room temperature, the stable phase is the monoclinic structure, also known as Baddeleyite, which is stable up to 1450 K, where the monoclinic to tetragonal transition occurs while the cubic structure is stable at temperatures higher than 2650 K prior to melting [17-20]. It has been reported previously that tetragonal and cubic structures can be



Fig. 3. Influence of the milling time on the morphology of the AA 7075-2 wt.% ZrO2 composite a) 2, b) 5, c) 10 and d) 15 h.



Fig. 4. Influence of the milling time on the morphology of the AA 7075-5 wt.% ZrO2 composite a) 2, b) 5, c) 10 and d) 15 h.

stabilized at room temperature by introducing some cubic stabilizer oxides, such as Y₂O₃, MgO, CoO, or CeO₂, in the lattice of the zirconia [17]. In the diffractogram shown in Fig. 2, it can be seen that the asreceived ZrO₂ was a mixture of mainly monoclinic and tetragonal structures. The polymorphs of ZrO₂ were identified in the as-received state by X-ray diffraction with PDF# card 89-9068 for tetragonal P42/nmc structure, PDF# card 89-9066 for monoclinic P21/c structure and PDF# card 89-9069 for cubic Fm3m structure. The identification of each phase by X-ray diffraction can be difficult, especially when trying to distinguish between the tetragonal and cubic structures, because of the overlapping peaks (the difference between the main peaks of each phase is only 0.079° in 20 degrees).

One of the most accurate methods of structure refinement and quantitative analysis of ZrO_2 polymorphs is the Rietveld method [18], which was adopted in this case using the MAUD software. By using this tool, the contribution of each phase in the mixture is discriminated taking into account the crystallographic details earlier ascribed to the cubic and tetragonal structures. As mentioned earlier, the starting material was a mixture of monoclinic, tetragonal, and cubic structures, where the tetragonal and cubic phases were stabilized with 3 %mol of yttria. It was observed that ball milling seemed to destabilize those phases, since a structural change resulted, which is evident in the X-ray diffraction pattern shown in Fig. 2. This last result can be demonstrated by taking into account the main peak of tetragonal ZrO_2 (30.198°), whose

Table 3			
Average size of the matrix	particles during	mechanical	milling.

Composite	AA 7075-2 wt.% ZrO ₂		AA 7075–5 wt.% ZrO ₂	
Milling time (h)	Average particle size (µm)	Standard deviation	Average particle size (μm)	Standard deviation
2	130	93	213	131
5	82	54	98	72
10	64	48	92	66
15	51	48	75	48

intensity was markedly reduced after the milling process. A reduction in the content of the tetragonal phase and an increase of the cubic phase were determined by the quantitative analysis performed by Rietveld method; these results are shown in Table 2.

It is apparent from Table 2 that there is a rise in the content of monoclinic and cubic structures, while a decrease of the tetragonal phase is observed; these findings partially agree with results of Gupta et al. [19], who stated that a tetragonal/monoclinic transformation occurs during fracture; thus, this phase transformation is possible during the milling since the mechanism of reduction of average particle size is the fracture event. This phase transformation, and details of the specific mechanism can be found elsewhere [20]. On the other hand, the mechanism to obtain the cubic structure by means of ball milling has been previously reported by Gateshki et al. [21], who described it in terms



Fig. 5. Matrix crystallite size as a function of the milling time.



Fig. 6. X-ray diffraction difractograms of AA 7075–5% ZrO₂ composite.

Table 4 Increment of weight percentage of monoclinic structure as the ball milling time increased in the 5 wt.% ZrO₂ composite.

Milling time (h)	wt.%
2	0.9
5	0.3
10	1.8
15	3.7

of a local atom ordering process. It is important to mention that the exact mechanism of provenance of each phase (monoclinic and cubic) is beyond of the scope of the present study. Table 2 also shows the average crystallite size of the ZrO_2 polymorphs. It is evident that the reduction of the diffracting domains in the monoclinic (until 25 nm) was greater, compared with the tetragonal structure (until 49 nm), but the highest reduction belongs to the cubic structure (until 66 nm). These results can indicate that the cubic phase might display a more ductile behavior during milling. Hence, it is proposed that the differences observed in the average crystallite size may be due to the relative hardness of each phase.

3.2. Ball milling of the composite material

SEM micrographs of the AA 7075–2 wt.% ZrO₂ composite milled for different times are shown in Fig. 3. The influence of the milling process on the morphological evolution of matrix powders can be seen in these micrographs. It was found that the morphology of the matrix particles changed from irregular to flake shape and the average particle size decreased slightly. On the other hand, a white layer on the surface of the matrix particles can be spotted at early milling times (2 and 5 h). According to the outcome of chemical analysis (EDS), this white layer seems to be an agglomeration of ZrO₂ particles due the high counts of Zr registered in the EDS spectrum. These white zones can be interpreted as evidence that adequate dispersion of the reinforcement was not achieved at earlier times. Furthermore, it was observed that for 10 and 15 h of ball milling the white layer did not appear anymore in the surface of matrix particles.

Fig. 4 exhibits the SEM micrographs of the AA 7075–5 wt.% ZrO₂ composite. A more intense white layer on the matrix particles is perceptible in Fig. 4a-c; as a consequence, it can be inferred that it was more complicated to obtain a homogeneous dispersion of the ceramic particles in these samples owing to the higher content of ZrO₂. Around 15 h of mechanical milling were necessary to make this white layer no longer visible on the powder surface, in contrast to the 2 wt.% ZrO₂ composite, for which the time required was approximately 10 h. In the case of the 5 wt.% sample the flake shape was not markedly evident even in the later times of processing, and the size of the particles seemed to remain practically constant. This fact can be associated to the influence that ZrO₂ could have on the powder surfaces, since the unique difference between the composites was the content of ZrO₂. To confirm this assumption, an average particle size measurement was performed, and the corresponding results can be seen in Table 3. It was found that there is a considerable drop in particle size in the 2 wt.% of ZrO₂ composite achieving an average particle size of 51 µm at 15 h of MM; meanwhile the 5 wt.% of ZrO₂ composite achieved 75 µm at the same time. Hence, it is stated that the reinforcement acted as a process control agent, causing a softer milling process as the ZrO₂ content was increased; it means that the plastic deformation of matrix particles was promoted while the fracture was inhibited. To support this assumption, X-ray diffraction data of samples were analyzed by Rietveld method in the MAUD software with the aim of computing the average crystallite size of the matrix particles since this parameter is dependent on the strain imposed on the material during the processes [22]. In Fig. 5, a plot of the crystallite size against the milling time is presented for the



Fig. 7. TEM micrographs of the AA 7075-2 wt.% ZrO₂ mechanical milled for a) 2, and b) 15 h.



Fig. 8. TEM micrographs of the AA 7075-5 wt.% ZrO2 mechanical milled for a) 2, and b) 15 h.

two composites. As the milling time was increased, a reduction tendency of the crystallite size was observed in both samples, except at 10 h of milling, at which the crystallite size appeared to increase slightly. It is evident that the composite with 2 wt.% of ZrO₂ exhibited a smaller average crystallite size compared to 5 wt.% of ZrO₂. If it is taken also into account that the former sample exhibited a more markedly morphological change in comparison to the latter, it can be stated that 2 wt.% ZrO₂ sample was subjected to a higher strain during MM.

Another interesting feature viewed in the X-ray diffractograms shown in Fig. 6 is the increase in the intensity of ZrO₂ peaks after composite milling, which suggests a probable increase in the phase content of monoclinic and cubic structures. This proposes that ball milling of the composite promoted an additional phase transition in the reinforcement. Assuming that the tetragonal structure was further destabilized, a quantitative Rietveld analysis was performed in the composite and the results are presented in Table 4. It can be seen that the monoclinic structure increased its weight percentage up to 3.7% in the case of 5 wt.% ZrO₂ composite. It is important to mention that this feature was not found in the 2 wt.% ZrO₂ composite due to a lower content of reinforcement.

The dispersion of the ceramic particles was characterized by TEM. Fig. 7a shows the micrographs of the ceramic particles and their distribution in the matrix powders. It can be seen that there are agglomerates of the ZrO₂ particles inside the matrix powder in the AA 7075–2 wt.% ZrO₂ composite subjected to 2 h of milling. In some areas it is difficult to distinguish the size of the ZrO₂ particles because there was not a good resolution of their boundaries, due the dense clustering. On the other hand, the sample shown in Fig. 7b was subjected to 15 h of ball milling. It was found that the reinforcement deagglomeration occurred for this milling time, which is evident in the micrograph that clearly shows the difference in contrast between the ceramic and metal particles. The appraisal of the reinforcement distribution indicates that 10 h of milling can be sufficient to avoid the agglomeration of them, a fact that could lead to harmful properties in the consolidated composite, since these clusters can act as stress concentrators and crack initiation sites, characteristics which are undesirable in subsequent processing [2,11].

Fig. 8a and b shows the AA 7075–5 wt.% ZrO₂ composite at 2 and 15 h of milling time, respectively. A denser clustering of ZrO₂ particles can be seen on the surface of the matrix powders at 2 h. In this figure it is not possible to discern the boundaries of the reinforcement, and the agglomeration seems to be greater because the ZrO₂ content in this sample is higher. Otherwise, the composite processed by 15 h of ball milling is shown in Fig. 8b, here the situation seems to be completely different, because there was no heavy agglomeration of the reinforcement exhibited and its distribution is more homogeneous.

The interaction between the reinforcement and the matrix powders can be spotted in Fig. 9, where a ZrO_2 particle seems to be trapped by the matrix in the AA 7075–2 wt.% ZrO_2 composite milled by 15 h, confirming that the main mechanism of the mechanical milling in this system is based on the trapping of reinforcement particles in the grain boundaries of ductile matrix powders as they collide themselves and with milling media [2,3].

4. Conclusions

- 1. Two metal matrix composites (AA 7075–2 wt.% ZrO₂ and AA 7075– 5 wt.% ZrO₂) were produced via powder metallurgy. To achieve a complete dispersion of ZrO₂ particles into AA 7075 matrix, 15 h of mechanical milling are needed for the 5 wt.% ZrO₂ composite, in contrast to 10 h for the 2 wt.% ZrO₂ composite.
- 2. Ball milling was used to reduce the average particle size of ZrO₂, and two variants of milling media were tested. It was found that milling using only balls gave better results since the average particle size was lower than milling with a mixture of balls and rods.
- 3. After 80 h of ZrO₂milling, an average particle size of 110 nm was achieved. It was confirmed that further milling time did not produce additional reduction of the particle size, because at 140 h the average particle size was 108 nm.



Fig. 9. TEM micrograph of the AA 7075-2 wt.% ZrO₂ composite mechanical milled for 15 h.

- 4. It was found that the ZrO₂ milling produced a destabilization of the starting tetragonal structure and the process promotes the monoclinic and cubic phases.
- 5. Good reinforcement–matrix bonding can be obtained by mechanical milling since the ZrO₂ particles were trapped in the fine grain boundaries of the matrix at 15 h of mechanical milling for both ZrO₂ contents

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