Microstructure and Microhardness of the Al-10Mg Alloy Processed by the Mechanical Alloying Technique

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Abstract:
In this work, the Al-10Mg nanostructured alloy was synthesized by high-energy mechanical milling. Subsequently, the powders consolidated under a uniaxial pressing in the air. The as milled powders were analyzed using scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and differential scanning calorimetry (DSC). SEM and XRD also characterized the sintered samples. Vickers microhardness of sintered Al-10Mg alloy was measured. SEM, XRD, and TEM characterizations show that the Al-10Mg alloy was synthesized after 10 h milling. The X-ray powder analysis of the structural parameters showed the increment with the time of the lattice parameter, strain as well as the solubility of Mg in Al. Besides, XRD and TEM studies showed that the crystallite size was reaching an average value of 19 nm after 10 h milling. The X-ray powder analysis of the structural parameters showed the increment with the time of the lattice parameter, strain as well as the solubility of Mg in Al. Besides, XRD and TEM studies showed that the crystallite size was reaching an average value of 19 nm after 10 h milling. XRD patterns of the all sintered specimens show the formation of the MgAl₂O₄ spinel phase. After powders compaction, the specimen sintered at 420 °C for 2 h shows microhardness of 125 HV.

Keywords: Al-10Mg alloy; High-energy mechanical milling; Structural characterization; Consolidation; Microhardness.

1. Introduction

Lightweight structural materials, such as Al, Mg and its alloys, have a high number of applications in the automotive and aerospace industries for body and chassis parts due mainly to its high strength/weight ratio, low density, ductility, good thermal and electrical conductivity, and corrosion resistance [1-6]. However, the low hardness of these alloys limits their use for some applications [7]. As a consequence of this, alloys of Al-Mg + M (M = Fe, Si, Cu, Cr, Mn, Zn, Zr, and Ti) have been developed to increase the hardness [3, 6, 8, 9].
Most Al-based alloys are synthesized by conventional casting [10], squeeze casting [11], and mechanical alloying (MA) [12]. Among them, MA is a non-equilibrium technique that offers advantages over others, such as low costs and control processing. During MA processing, a high quantity of crystalline defects is induced in the material that can increase the solubility of metals. Additionally, with this route, the obtained nanocrystalline alloy prevents the movement of the dislocations, which improves hardness, fracture tenacity, and wear resistance [13-16].

Several investigations using the MA technique improved the solubility of Mg in Al from 5 to 45 at.% [13-15]. On the other hand, few studies have been carried out employing high-energy mechanical alloying in Al(Mg) alloys with low concentrations of Mg, particularly the Al-10Mg alloy. Mg, as an alloying element of Al, is considered to increase its mechanical resistance due to its lower stacking fault energy. The Al-10Mg (11 at.% solid solution supersaturated can increase aluminum mechanical resistance without β-Al13Mg2 and γ-Al12Mg17 intermetallic phases’ precipitation. These phases have been reported in Al-Mg alloys processed by MA [17, 18].

Thus, in the present work, we report the synthesis of Al(Mg) 10 wt.% powders by MA as a function of time and their subsequent consolidation in the air. During the compaction of the powder, the temperature and time of sample sintering were varied and HV microhardness of the specimens evaluated.

2. Materials and Experimental Procedures

For the preparation of the Al-10Mg alloy by high-energy ball milling, Al (granular < 1 mm) and Mg (chips, 4-30 mesh) with a purity of 99.70 wt.% and 99.98 wt.%, respectively, were acquired from the Sigma Aldrich Co. The Al-Mg alloy was formed in a SPEX 8000M ball mill equipment at 1800 rpm, with a ball/weight ratio of 7:1. Stearic acid (3 wt.%) was used as the process control agent. The vial and the milling media were hardened stainless steel D2 type. The milling times used were: 0.5 h, 1 h, 2 h, 4 h, 8 h, and 10 h. The thermal stability of the mechanical alloy was investigated by differential scanning calorimetry (TA-Q600 instrument) using a heating rate of 10 ºC/min under an ultra-high purity argon atmosphere with a flow rate of 100 ml/min. The powders consolidation was carried out in the sample milled by 10 h. The experimental conditions were a uniaxial pressing with preheating at 300 ºC and a pressure of 600 MPa for 10 min. The sintering was carried out in a conventional oven at temperatures of 420 and 488 ºC for different durations (0.5, 1, and 2 h). The microhardness of the compacts was measured with a Mitutoyo model HM-200 Vickers indenter with a diamond tip applying a load of 100 g for 15 s. The morphological and structural characterization of the as-milled powders was carried out by employing scanning electron microscopy (JEOL JMS-6400), transmission electron microscopy (Phillips TECNAI F20), and X-ray diffraction (Bruker D8 ADVANCE). Step width and counting time were chosen as 0.02 º and 1 s, respectively. SEM and XRD also characterized the sintered samples.

3. Results and Discussion

3.1. Al-10Mg mechanical alloying

Figure 1 shows SEM images corresponding to the 0.5 h, 4 h, 8 h, and 10 h milling times. The mechanical alloying (MA) induced morphological changes in the particles due to the plastic deformation. For the first 0.5 h of MA, the particle size was approximately 2.5 mm (see Figure 1a). Deformed flake-type particle morphology is observed, attributed to the ductile character of the starting materials. After 4 h milling time (Figure 1b), the processes


hardening and fragmentation predominate, which leads to a particle size reduction of approximately 210 μm.

Figure 1c shows that the particle size decreases, showing semi-spherical particles after 8 h of milling time. At this stage, the fracture process dominates over the cold welding process is attributed to the processing control agent (PCA). The presence of PCA leads to an efficient process that avoids the excessive union of the particles and improves the solute diffusion.

In the SEM micrograph corresponding to the 10 h milling (Figure 1d), a minimal decrease in particle size was observed associated with the equilibrium between fracture and cold welding near to the end of the solid solution. In the past, it has been widely mentioned that during mechanical alloying, this equilibrium is achieved in different alloy powder systems [17]. Also, it is found that for this milling time, a narrower particle size distribution is obtained with an average size of 16 μm and a semi-spherical morphology. However, the alloy structure cannot be refined indefinitely due to the increase in hardness with milling time and crystal size reduction. On the other hand, during mechanical alloying, the reaction is not a purely mechanical process because that involves interdiffusion driven by the mixing enthalpy of the Al-Mg [13, 18]. As the milling time elapses, crystallites continue to decrease in size, giving rise to Al(Mg) nanostructured alloy.

The EDS technique was used to find the elemental chemical composition of the milling powders. EDS spectrograms (inserts Figure 1) show the Al and Mg elements from the starting material, confirming no signs of contamination. The O element attributes to the superficial metal oxide formed in both elements.

**Fig. 1.** SEM images corresponding to mechanical alloy samples for different times as well as their respective EDS patterns: a) 0.5 h, b) 4 h, c) 8 h, and d) 10 h.

Figure 2 shows the XRD patterns of the samples corresponding to 0.5 h, 1 h, 2 h, 4 h, 8 h, and 10 h of milling. The X-ray patterns belonging to the milling times of 0.5 h and 1 h clearly illustrate the presence of Al and Mg reflections (JCPDS no. 00-004-0787, JCPDS no. 01-089-4894). As the milling time increases, the diffraction peaks of the aluminum decrease in intensity and become broadened, while the reflections corresponding to the magnesium disappear. This behavior indicates a progressive solid solution formation, as well as a reduction in crystal size accompanied by an increase in the lattice strain. According to the X-ray theories, the decrease in the crystal size and the rise of crystalline strain are attributed to
crystalline defects such as vacancies, linear, and superficial defects, causing a local deformation (micro-stress) during the mechanical alloying.

Also, displacements of the Al peaks towards smaller angles are seen, which indicates the increment in the lattice parameter attributed to the Al substitution by Mg because the atomic radius of Mg (0.160 nm) is higher than Al (0.143 nm). After 10 h of milling time, the Mg reflections entirely disappear, indicating the formation of the Al-10Mg solid solution.

![XRD patterns corresponding to the mechanical milling times of 0.5 h, 1 h, 2 h, 4 h, 8 h, and 10 h.](image)

**Fig. 2.** XRD patterns corresponding to the mechanical milling times of 0.5 h, 1 h, 2 h, 4 h, 8 h, and 10 h.

Figure 3 shows the refinement of the structural parameters of the Al(Mg) phase synthesized by MA as a function of milling time [19]. Figure 3a illustrates that as the milling time increases, the Mg disappears, while the lattice parameter of Al increases from 4.03718 ± 0.00166 Å to 4.07319 ± 0.00018 Å, associated with the substitution of Al atoms by Mg. These results confirm the formation of a solid solution of Al(Mg) after 10 h of the MA process. The solid solubility for Mg in Al at 100 ºC is 1.9 wt.% (2.1 at.%), while at room temperature it is 1 at.%. It has been reported that solid solubility extends beyond equilibrium concentrations up to 45 at.% by MA [13]. The solid solubility is attributed to the constant fracture and cold welding, where the latter minimizes the distance of atomic diffusion between the layers formed by both components. Besides, it is attributed to the high temperatures generated locally during the impacts between the balls and dust (≈ 400 ºC) [13, 20-23].

Figure 3b shows that the crystal size decreases, while the internal strain increases, as a function of milling time attributed to the lattice distortion. In MA processes, an excess of energy accumulation is released through the fracture of particles to reach nanometric sizes. The crystal size obtained for 10 h milling was 19 nm with a lattice strain of 0.079 %. These results were similar to those previously reported in samples treated by mechanical milling [18, 21].
Fig. 3. Different plots showing the variation of the structural parameters in the Al alloy as a function of the milling time: a) Mg content (ordinate left), lattice parameter (ordinate right); b) crystal size (ordinate left) and lattice strain (ordinate right).

Figures 4a and 4b show transmission electron microscopy (TEM) images, which confirm the formation of a nanostructured alloy obtained with an average crystal size between 10-25 nm after 10 h MA. From the selected area diffraction pattern, a series of concentric rings corresponding to the (111), (200), (220), (311), and (222) crystallographic planes of the Al-fcc crystal lattice (Figure 4c) are observed [24]. Figure 4d displays a high-resolution transmission electron microscopy (HRTEM) image, showing a crystal with a size of approximately 20 nm. The interspacing of the HRTEM micrograph corresponds to the interatomic distance of 2.28 Å belonging to the (111) plane of the Al-fcc crystal lattice. Also, the inset shows the FFT pattern confirming the (111) planes of Al.

Fig. 4. TEM micrographs of Al-10Mg alloy in a) bright field, b) dark field, c) selected area diffraction pattern, and d) high-resolution with its respective FFT (inset).
3.2. Alloy sintering

Figure 5 shows several SEM micrographs with their corresponding EDS chemical analysis of the sintered Al-10Mg alloy. Figure 5a shows a micrograph that corresponds to the preheating sample at 300 ºC for 3 min. Irregular particle morphology is seen. The densification value for this sample was 77.4 %. Figures 5b, 5c, and 5d correspond to the alloy sintered at 420 ºC for 0.5 h, 1 h, and 2 h, respectively. The densification for the three sintered samples was 80.5 %, 82.2 %, and 83 %, respectively. As observed, the particle size grows while the porosity decreases as the time of the thermal treatment increased attributed to the high diffusion of the elements with the temperature.

Figures 5e and 5f display images of the sintered material at 488 ºC for 0.5 h and 1h, respectively. Particles with irregular morphology can be seen in these images. EDS spectra indicate the increasing presence of O as the temperature increases due to the formation of the typical oxide layer that appears in the Al alloys. The densification values obtained for the two specimens here observed were 79.3 % and 80.2 %, respectively. These values are lower than those obtained at 420 ºC attributed to the oxide layer formation. However, they are similar to those reported by other authors under uniaxial pressure conditions [25, 26].

![Fig. 5. SEM images corresponding to the Al-10Mg alloy treated with different temperatures and sintering times: a) green sample (preheating at 300 ºC), b) 420 ºC-0.5 h, c) 420 ºC-1 h, d) 420 ºC-2 h, e) 488 ºC-0.5 h, and f) 488 ºC-1 h.](image)

Figure 6 shows the X-ray diffraction pattern of the Al-10Mg before and after sintering. Figure 6a shows the XRD pattern corresponding to the preheating material at 300 ºC for 3 min illustrating only the typical reflections of the Al alloy. The average crystal size determined was 22 nm. Figures 6b, 6c, and 6d correspond to the XRD patterns of the sintered material at 420 ºC during different times 0.5 h, 1 h, and 2 h, respectively. XRD sharp peaks of the solid solution Al(Mg) are observed and attributed to the crystal growth by the temperature effect. The measurements of the average crystal size for these conditions were 29 nm, 28 nm, and 29 nm, respectively.

In the same way, slight displacements of the peaks towards high angles are appreciated in the patterns, which means a decrease in the cell parameter, caused by the segregation of Mg. The Mg segregation favored the nucleation of the MgAl₂O₄ spinel phase.
(JCPDS no. 00-033-0853) because the sintering process is carried out in the air atmosphere. The amount of the spinel phase was quantified in 1.1 wt.%, 1.9 wt.%, and 2.5 wt.% for the residence times of 0.5 h, 1 h, and 2 h, respectively. It is essential to mention that the precipitation of intermetallic phases (i.e., \( \beta\)-\( \text{Al}_3\text{Mg}_2 \)) does not occur due to the absence of a controlled atmosphere.

XRD patterns of the sintered material at 488 °C for 0.5 h and 1 h are presented in Figures 6e and 6f. The crystal size determined for the two specimens was 39 nm and 34 nm, respectively. From the patterns, an increase in intensity and a decrease in the width of the peaks were observed. Besides, the presence of the \( \text{MgAl}_2\text{O}_4 \) phase was estimated at 4.1 wt.% and 5.4 wt.%, respectively.

As can be observed in the absence of a controlled atmosphere, the oxidation product obtained was \( \text{MgAl}_2\text{O}_4 \) spinel phase since it is more thermodynamically stable than others such as \( \text{MgO} \). These results are in agreement with those obtained in [27, 28].

Similarly, from these results, it can be seen that as the temperature and sintering time increases, the amount of spinel phase increases, which directly influences the obtained lower densification percent which is due to the limiting of the mass diffusion between the species.

![XRD patterns](image)

**Fig. 6.** XRD patterns corresponding to the Al-10Mg alloy treated with different temperatures and sintering times: a) green sample (preheating at 300 °C), b) 420 °C-0.5 h, c) 420 °C-1 h, d) 420 °C-2 h, e) 488 °C-0.5 h, and f) 488 °C-1 h.

Figure 7 shows the DSC-thermal analysis corresponding to the Al-10Mg alloy. The results indicate that the material exhibits a thermal behavior characterized by three events. The first corresponds to an exothermic peak at approximately 350 °C. The second peak situated at 480 °C is also exothermic. Finally, the DSC curve shows an endothermic peak at the maximum temperature of 651 °C, which is associated with the melting point of the alloy. In the literature, the first two events are continuously reported as the \( \beta\)-\( \text{Al}_3\text{Mg}_2 \) and \( \gamma\)-\( \text{Al}_{12}\text{Mg}_{17} \) intermetallic compounds formation, which appear under certain experimental conditions such as Mg weight percentage in Al alloy, controlled atmosphere, time, and temperature of sintering [13, 20]. The endothermic peak is attributed to the melting point of Al alloy, which temperature is lower than that of pure Al (660 °C), which also indicates the Mg solubility in Al [29].
3.3. Microhardness tests

Figure 8 displays the optical micrographs of the indentation made in the Al-10Mg alloy sintered at different temperatures. Based on the trace of indentation, an average Vickers microhardness of 125 HV was obtained for the sintering sample at 420 °C for 2 h (Figure 8a). Meanwhile, Figure 8b shows the indentation profile for the sintered material at 488 °C for 1 h determining a microhardness of 111 HV.

Contrary to expectations, it was observed from the SEM results (Figure 5) that as sintering temperature increases, the densification values decrease since with the increase in temperature there is an increase in the diffusion of the particles. From the XRD patterns (Figure 6), it was observed that with the increment in temperature, the presence of the MgAl₂O₄ spinel phase increased. From the above, it is deduced that the increase of the MgAl₂O₄ spinel causes a low diffusion, limiting the densification of the material under environmental conditions. Also, these results directly influence the microhardness values.

Thus, the best densification results (83 %) were obtained when the sintering temperature was 420 °C for 2 h, where is recorded 2.5 wt.% of MgAl₂O₄. Under these experimental conditions, a microhardness of 125 HV is obtained being higher than pure Al (45 HV) attributed to the formation of the Al(Mg) nanostructured alloy [30, 31]. Besides, the microhardness values reported here are slightly higher than those mentioned in [21, 32-34], who report microhardness values of 50 HV, 95 HV, 70 HV, and 60 HV which gives the possibility that the Al-10Mg alloy can be used in structural applications.
4. Conclusion

In summary, the Al-10Mg nanostructured alloy was synthesized by high-energy mechanical alloying in a 10 h milling time. The XRD technique observed that the lattice parameter, strain as well as the solubility of Mg in Al increases as the milling time increases. XRD determined a crystal size of 19 nm to 10 h of milling. TEM studies display a crystallite size distribution of 10-25 nm. SEM, XRD and DSC results show that the best sintering and densification (83 %) conditions were at 420 °C for 2 h. Under these compacts conditions, a crystal size of 29 nm, a lower proportion of the MgAl2O4 phase (2.5 wt.%) and relatively high microhardness value (125 HV) were obtained. This microhardness value is relatively higher than other Al-Mg alloys with similar compositions, which offers the possibility of structural applications.

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5. References

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Саметак: У овом раду је описана синтеза Al-10Mg наноструктурне легуре током млевења у високо-енергетском млину. Након тога, прахови су консолидовани униаксијалним пресовањем у атмосфери ваздуха. Млевени прахови су анализирани помоћу анализе SEM, TEM, XRD и DSC. SEM и XRD су такође коришћене ради характеристике синтерованих узорака. Мерена је и микротврдоћа по Викерсу. SEM, XRD, и TEM анализе показују да је легура Al-10Mg синтетисана након 10 сати млевења. XRD анализа праха указује на раст параметра решетке са временом млевења, микронапрезања и растворљивост Mg у Al. Поред тога, XRD и TEM резултати показују да је величина кристалита 19 nm након 10 h млевења. XRD шеме свих синтерованих узорака доказују формирање спинелне фазе MgAl₂O₄. Након пресовања, узорци синтеровани на 420 ºC током 2 h показују микротврдоћу од 125 HV.

Кључне речи: Al-10Mg легура, високо-енергетско млевење, характеристика, консолидација, микротврдоћа.