ALUMINIO CON GRANO MECÁNICAMENTE REFINADO PREPARADO MEDIANTE MOLIENDA DE BOLAS DE ALTA ENERGÍA Y SINTERIZACIÓN RÁPIDA UTILIZANDO UNA RUTA ALTERNATIVA BASADA EN CALENTAMIENTO POR INDUCCIÓN DE ALTA FRECUENCIA

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RESUMEN

En este estudio, se presenta el uso de un proceso de sinterización rápida basada en calentamiento por inducción. Muestras de aluminio con grano refinado fueron obtenidas mediante molienda de bolas de alta energía a partir de aluminio puro en polvo. Debido a la alta velocidad de calentamiento y corto tiempo de sinterización (minutos en vez de horas) resultado de la sinterización por inducción con alta frecuencia, el tamaño de grano sub-micrométrico en las muestras se conserva; alcanzando con ello un aumento en la respuesta mecánica de las muestras obtenidas. El objetivo de este estudio es evaluar el efecto de los parámetros de sinterización sobre el desempeño mecánico de las muestras preparadas. La caracterización microestructural de los polvos fue realizada a través de microscopía electrónica y difracción de rayos X, mientras que la respuesta mecánica de las muestras se midió mediante pruebas de resistencia a la compresión y determinación de dureza a nivel micro/macro. Las muestras sinterizadas mediante inducción mostraron una mejorada respuesta mecánica comparada con la obtenida mediante una ruta de sinterización tradicional y valores reportados de una aleación comercial.

Palabras clave: Aluminio, Refinamiento de grano, molienda mecánica de alta energía, inducción de alta frecuencia.

Área temática: Materiales ligeros (Mg, Al, Ti, y sus aleaciones y composites).

Presentación: Oral

MECHANICALLY GRAIN REFINED ALUMINUM PREPARED BY HIGH-ENERGY BALL MILLING AND RAPID SINTERING USING AN ALTERNATIVE ROUTE BASED ON HIGH-FREQUENCY INDUCTION HEATING

ABSTRACT

In this work, the use of a rapid sintering process based on induction heating is presented. The grain refined aluminum samples studied in this work were obtained by high-energy ball milling using pure aluminum powders. Due the high heating rate and low sintering time (minutes instead hours) caused by induction sintering, the sub-micrometric grain size of samples is preserved; which leads to an increase on the mechanical response of samples. The objective of this study is to evaluate the effect of the sintering route on the mechanical performance of prepared samples. Microstructural characterization was developed through electron microscopy and X-ray diffraction, while the mechanical response was measured through compressive and micro/macro hardness tests. Induction sintered samples showed a better mechanical response in comparison to the samples prepared by conventional sintering and a commercial alloy.

INTRODUCTION

Strength is referred as an important material property related to the capacity to resist an applied stress until the breaking point. High-deformation level in metals is possible due to the presence of defects called dislocations; which can move along slip planes (under a force), causing deformation. Pure metals have low strength, but with alloying, metalworking or heat-treating methods is possible to increase their strength. From industrial modern metals, aluminum (Al) has a preponderant place due to their high technological applications [1], in fact Al is the second most important metal (after iron) due its unique properties: low density, high environmental resistance, low cost due its high recycling rate, high electrical and thermal conductivity, etc. Unfortunately, the above-mentioned characteristics are clouded by its low strength, thus Al strengthening is necessary for some applications. Normally, Al strengthening is obtained by strain hardening, solid solution and second phase dispersion. Wrought alloys are classified in two groups: non-heat treatable and heat treatable. Non-heat-treatable alloys have a high resistance to general corrosion and higher electrical conductivity, for electrical applications the addition of alloying metals to strengthen the material would not be feasible due to the amount of foreign atoms [2] causing falls on the electrical conductivity. Thus, electrical grade aluminum can be strengthen by two ways: a) work hardening (or cold working) caused by deformation at room temperature and b) fine-grain hardening, this by increasing the number of grain boundaries that act as barriers to the dislocation movement increasing strength. There is significant research into production of bulk materials with nanometer-scale grains for potential applications, beginning with the production of nanocrystalline (NC) or ultrafine grained (UFG) powders consolidated into a bulk [3,4]. Both can be obtained by two ways: changing of physical state by gas condensation or rapid solidification (forming nanometric particles) or refining micron scale particles by severe plastic deformation using ball milling [5,6,7,8]. NC and UFG [9] present outstanding macroscopic properties [10] because of the high volume fraction of the interfacial

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phase and the extremely small mean grain sizes; in fact yield stress and microhardness can be 2–10 times higher than coarse-grained counterparts [11,12,13,14]. To obtain bulk composites from mechanically alloyed powders, several consolidation techniques have been employed, such as sintering, hot-pressing, hot isostatic pressing and vacuum hot extrusion of pre-pressed powders [15]. Unfortunately, the grain size of conventional sintered samples becomes larger because of grain growth during sintering, missing intrinsic properties of NC and UFG. In this study, high-energy ball milling and high frequency induction sintering (HFIS) were used to produce bulk samples with refined microstructure.

EXPERIMENTAL PROCEDURE

The used material was a commercial aluminum powder (99.5% purity, -325 mesh); Al was milled in a high-energy ball mill (Spex 8000M) for 2h to produce the microstructural refined samples. Steel balls were used as milling media in a vial sealed with a protective Ar atmosphere. To obtain bulk samples from the above powders we followed two routes: a) Conventional method (C), powders were uniaxially cold compacted under 900 MPa in a die, producing cylindrical bars. Green compacts were sintered in a furnace with an Ar flow during 3h at 550°C followed by natural cooling until room temperature. b) High-frequency induction sintering (HFIS), the powders were placed in a steel die pressed in air at 450 MPa, after reaching the pressure, an induced current (70 kHz and 0.8 kW) was activated reaching 450°C with a heating rate of 135°C/min and hold for 3 min. After sintering, the electric current was turned off and the die was cooled down using a forced airflow. To avoid oxidation, the sintered samples were unloaded at room temperature. The morphological studies and EDS analyses of powders were done using a scanning electron microscope (SEM) JEOL-JSM 7201F and X-ray diffraction (XRD) analyses were performed in a Bruker D8 advance diffractometer. The mechanical characterization of the sintered samples was done based on hardness determinations and compression tests.

RESULTS AND DISCUSSION

Morphological analysis. Original powders particles present an irregular rounded morphology characteristic of atomized metals with a broad particle size distribution as can be seen in the Fig. 1a. During milling, some metallic particles are trapped between the highly energized clashing balls, inducing a severe plastic deformation in the powders. Resulting flattening increase the surface to volume ratio and larger aggregates formation is evident; in Fig. 1b the presence of flakes and cracks on the surface of a large agglomerate is noticed. Further milling causes additional welding and plastic deformation narrowing the lamellae of the starting particles with fragments of smaller particles into the created “new surfaces”; in this way, the internal structure is improved by thinning of the internal lamellae. As the deformation progresses, the particles harden and the tendency to fracture predominates over welding resulting in the refining of particle size [16] as can be seen in the Fig 1c. In other words, this starts with the deformation into shear bands with high dislocation density followed by recombination of dislocations, forming sub grains [5].

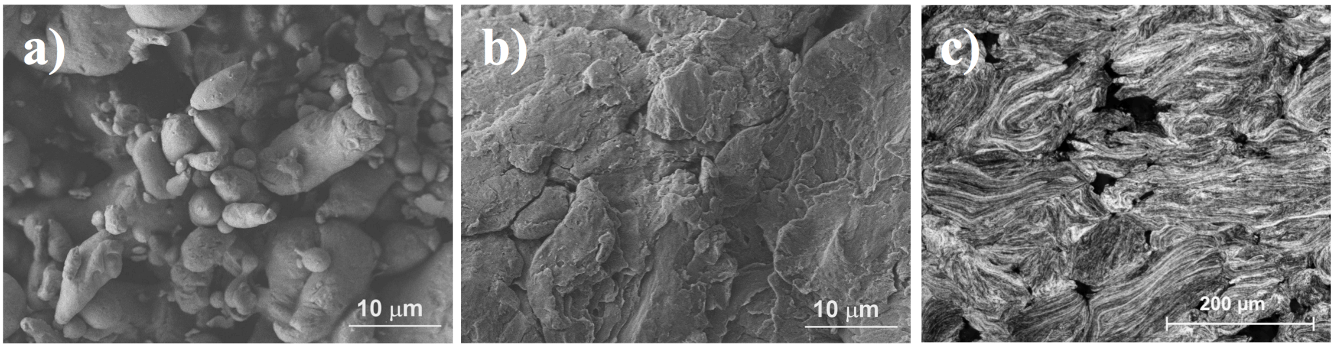
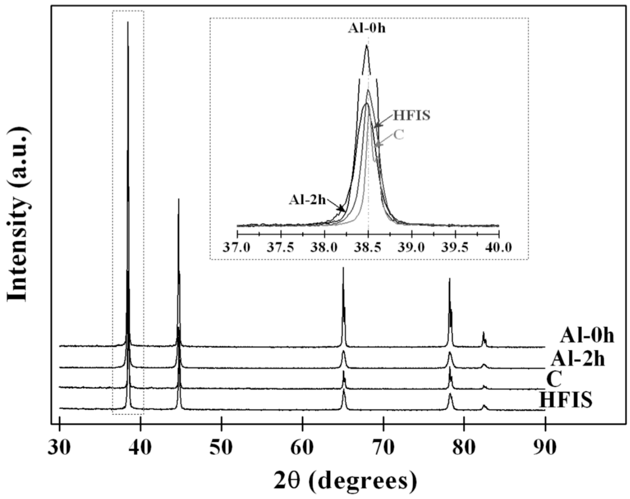


Figure 1. SEM micrographs of Al powders a) raw material 0h, b) 2h milled powder and c) optical microscope image in cross section of Al powders after milling and cold compaction.

X-Ray diffraction. The figure 2 shows the XRD pattern of the Al samples in powder form and bulk samples after sintering. It is evident that the intensity of Al reflections decreases notably and peaks become broader after milling (Al-2h). This is related with severe plastic deformation, usually accompanied by strain hardening and grain refining. Lattice parameter changes were measured based on the XRD peaks shifting; shifting absence and new peaks generation suggests low milling contamination or alumina formation during sintering.



Sample	Grain size [nm]	μ-strain [%]	Lattice Param. [A]
Al-0h	880 ± 78	0.024 ± 0.010	4.045
Al-2h	560 ± 88	0.149 ± 0.028	4.048
C	1160 ± 108	0.060 ± 0.005	4.048
HFIS	585 ± 42	0.118 ± 0.017	4.046

Figure 2. XRD patterns with a close up on Al (111) peak and summary chart.

Grain size and micro-strains determinations were taken using the Williamson-Hall method [17] and are presented in the summary chart of Fig. 2. During conventional sintering is evident an important grain growth and microstrain reduction, contrary to HFIS which maintains a sub micro grains in bulk samples. Although HFIS process was performed in an air atmosphere, oxidation evidence was not found: XRD results did not show alumina formation (or it is below detection limit); same happened with EDS analyses performed on sintered samples (not shown), which exhibited a low concentration of oxygen in comparison to conventional sintered sample (processed under Ar).

Mechanical Testing. Hardness tests were performed following the ASTM E18 standard, HRF scale was selected (1/16” ball indenter and 60 kgf of load) measuring each sample five times and obtaining the average and deviation. Al-0h sample presents a very low hardness response (below of minimum scale value) as the Fig. 3a shows. After milling, the ductility of samples was modified and the hardness value could be measured. As a milling result, the increase of high volumetric density of grain boundaries impedes dislocation movement to adjacent grains; strengthening the material [4]. The assumption is that grain boundaries act predominantly as sumps for dislocations [11]. Some authors assume that grain boundary depends upon local structure and chemistry establishing the bulk properties [10]. To compare the hardness reached, an Al-1350 commercial alloy (99.5% Al) after the highest hardened treatment (H19) is included in the plot, alloy has a hardness value of 50 HB (equivalent to 53 HRF). There is an increase of 62% with HFIS sample compared with the Al-1350-H19 alloy meanwhile C sample has a 53 ± 3 HRF value (very similar to the commercial alloy). To compare the hardness of all samples, some micro hardness measurements were done (100g load during 15s); the results are illustrated in Fig. 3b. Is noticeable the increase of strength after milling; after conventional sintering, part of hardness is lost due to grain growth, unlike HFIS that retains refined structure, thereby its hardness is high.

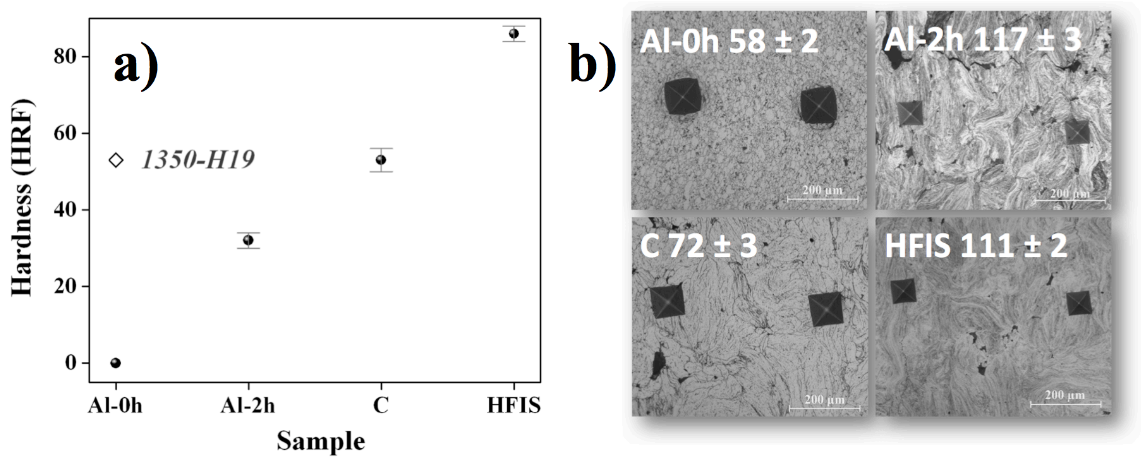


Figure 3. a) Hardness plot of studied samples compared with a highly strengthened commercial alloy b) Microhardness determinations in μVickers scale and indentation marks.

To complement the mechanical study, some compressive tests were done on sintered samples obtaining the curves showed in the Fig. 4a, while Fig. 4b presents the yield strength obtained from compression curves, again the Al-1350-H19 commercial alloy is compared. The Al-2h HFIS sample presents the highest value of 317 MPa (90% above commercial alloy). The mechanical characterization indicates that HFIS constitutes a promising technique to strengthen aluminum by retaining the fine microstructure reached by MM using low temperature (450 vs. 550°C) and low processing times (9 min. vs. 9 hours) in comparison with the conventional sintering route. If we compare the mechanical response of samples represented as hardness and yield strength a similar behavior is observed: unmilled samples have a low mechanical response, milling increases the mechanical response of samples and HFIS samples have better mechanical response in comparison with the conventional sintering route. Although some authors establish that there is no simple relationship, between hardness and yield strength in metals and alloys [11], evidence shows a clear tendency that grain refinement further improves the strength of pure Al alloys; a precise determination of the strengthening mechanisms has been hindered by the complexity of the possible ways: grain-boundary strengthening (Hall–Petch effect), solid-solution strengthening, dislocation strengthening and precipitation strengthening [4].

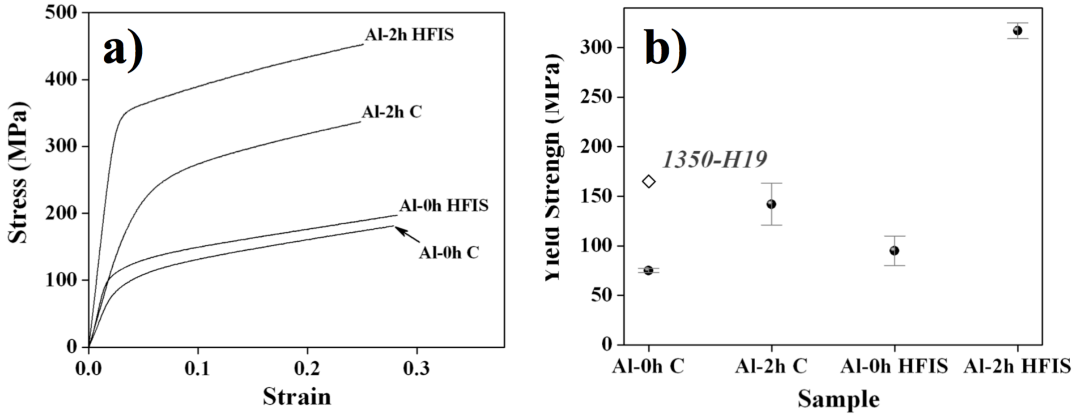


Figure 4. (a) Strain-stress curves (average) and (b) yield-strength of studied samples.

Some authors argued that is generally true that for UFG materials, the grain size refinement is the fundamental strengthening mechanism [18,19] and others state that different strengthening mechanisms may contribute simultaneously to the overall strength of real materials [1,13]. Bibliography on this subject mentions that the yield stress of a NC material is the sum of stresses of grain interiors and grain boundaries, deformation mechanisms are described by lattice dislocations and creep at atomic level [11].

CONCLUSIONS

SEM-XRD studies revealed an important grain refinement after high-energy ball milling, this condition was kept after sintering using HFIS. Although HFIS was done in air, the sample oxidation was low. Our described method produced samples with enhanced mechanical properties: HFIS samples had a hardness of 86 HRF with 317MPa of elastic limit, compared to a commercial hardened alloy (Al-1350-H19) this means 60 and 90% of increase, respectively. The described method constitutes a promising technique to produce strengthened samples, retaining the microstructure reached by milling, using low temperature and shorter processing times (minutes instead hours).

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