Extrusion of aluminum alloys prepared from mechanical alloying powder

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Abstract

The aluminum alloys were produced by means of mechanical alloying and subsequently hot extrusion, as well as microstructure were studied and properties determination. Two Al-Fe-X-Si (X=V or Nb) compositions, Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} and Al_{90.8-Fe_{6.2}-V_{1.0}-Si_{2.0}} (at. %), were prepared by mechanical alloying from the respective metallic powders. The powder mixtures and extruded materials were characterized by X-ray diffraction, differential scanning calorimetry, scanning (SEM) and transmission (TEM) electron microscopy, and energy dispersive spectrometry (EDS). Extruded materials were also characterized for hydrostatic density, hardness and tensile strength.

1. Introduction

Powder metallurgical process (PM) using mechanical alloying technique (MA) is able to produce a material with a superior strength [1]. The solid interaction by MA have attracted a large amount of study [1-4]. Mechanical alloying method has been recognized as complex process which can be applied to processing of advanced materials at low cost. In general, both stable and metastable phases can be produced by mechanical alloying [4]. MA takes place under high energy milling balls agitation, resulting in cold welding and fracturing of the metallic powder mixture, and some non-metallic powders can also be added [5]. The mechanisms of the state reactions during high energy milling have been intensively studied. This technique permits to get an alloy by mean of a mechanical processing completely in solid-state for subsequent consolidation [5,6]. The nanostructures produced by mechanical attrition are not caused by cluster assembly, but by the structural decomposition of coarser - grained structures, as the result of severe plastic deformation. The thermal stability of nanocrystalline microstructure is of major importance both for hot consolidation of the powders and for any possible elevated temperature applications. Mechanical alloying offers many advantages over the processes used for conventional aluminum alloys, including alloying with a fine microstructure and a high-volume fraction of thermally stable dispersoids. Al-Fe-Mn alloys and some rare earth elements produced by MA, are present for high-temperature applications. Mechanically alloyed (MA) aluminum alloys such as AlFeMn [7], AlTiNb [8], AlMnCe [9] and the other binary and ternary alloys have been studied but there are only a few papers on MA Al-Fe-V-Si alloys [10].

This paper presents the microstructure and properties of alloys MA by milling of elemental powders of Al, Fe, X, and Si, (X= V or Nb) followed by vacuum hot pressing and hot extrusion.
2. Experimental

Two Al-Fe-X-Si (X=V or Nb) compositions, Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} and Al_{90.8-Fe_{6.2}-V_{1.0}-Si_{2.0}} (at. %), were prepared by mechanical alloying from the respective metallic powders. The mean particle sizes of the starting elemental powders were: Al-50µm, Fe-100µm, Nb-100µm, V-1mm size flakes and Si-100µm. Preliminary, powder mixture for composition Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} milling experiments were conducted for 5, 10, 20, and 40 hours only at 1400 rpm to observe the powders morphology evolution and possible new phase formation. The powder mixture, Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} composition were also attrition milled for 10 hours at 800 rpm. The Al_{90.8-Fe_{6.2}-V_{1.0}-Si_{2.0}} (at. %) composition was attrition milled at 800 and 1400 rpm for 10 hours. Additionally the Al_{90.8-Fe_{6.2}-V_{1.0}-Si_{2.0}} (at. %) composition, was also attrition milled for 5 hours at 1400 rpm, and for 20 hours at 800 rpm.

The powders were mechanical alloying (MA) in an attritor mill with a vertical shaft in a jar under a protective nitrogen atmosphere. The milling vial of the alloy was made of polyethylene with 81mm in diameter. The balls were made of chrome steel with 7mm in diameter.

After milling, the powders were removed in air atmosphere and vacuum hot pressed (6x10^{-3} torr) under a pressure of 530MPa and a temperature at 340°C. The compacted billet of 31mm in diameter by 30mm in length, was hot at 500°C and extruded in a laboratory extrusion machine by indirect extrusion. Indirect extrusion was done with an extrusion ratio of 6:1. Hand- mixed composition (0hour) were also vacuum hot pressed and extruded, and subsequently used as reference materials.

The morphology evolution of the MA powders in the Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} composition were examined in a scanning electron microscope (SEM) and samples of the extruded bars in the two compositions were examined in a transmission electron microscope (TEM). TEM specimens were 3mm disks approximately 200µm in thickness. The center of these discs was thinned to approximately 300nm by double electrolytic jet polishing in Tenupol 3 (at 25V and 300mA) with a 10% perchloric acid 90% methanol solution at minus 10°C.

X-ray diffraction measurements were carried out in a Rigaku diffractometer with CuKα radiation. The morphology of the powder were observed in a Leo Steroscan 440 SEM and a Philips CM 200 TEM containing a lithium/ silicon X-ray detector. The DSC experiments were performed in a Shimadzu DSC 50, under dynamic nitrogen atmosphere with a heating of 5°C/min.

Tensile tests were carried out at room temperature, 200 and 350°C. Vickers hardness measurements were performed on extruded bars at room temperature. An Instron 4400R universal testing machine with a load cell of 5kN and at 1.67x10^{-3}mm/s was used in the tensile tests. In the hardness measurements, a load of 0.98N was used.

3. Results and discussion

The shapes of the powder particles Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} after 5, 10, 20 and 40 hours of milling at 1400rpm are shown in Fig. 1. The powder milled for 5 hours at 1400rpm, had the flake morphology, these time there was predominance of the welding. At 1400rpm after 10 hours , the powder particles are more round and smaller, was prevailed welding and fracture.

Fig. 2 show the X-ray diffractogram (XRD) of the powder particles Al_{90.8-Fe_{6.2}-Nb_{1.0}-Si_{2.0}} after 1, 5, 10, 20 and 40 hours. In the powder that was MA for 1 hours and 5 hours at 1400 rpm, no new phase was detected by X-ray diffraction. This is pattern showed peaks corresponding to the elements Al, Fe, Nb and Si. After 10 hours of milling, AlNb2 phase was formed. There were no other phases during milling. The decrease of intensity and enlargement of the diffraction peaks during the milling time is noted. It’s could be due to the fact of the increase number of defects in the elemental powders.
FIG. 1: Morphology of powder particles after (a)5, (b)10, (c)20 and (d)40 hours of milling at 1400 rpm.

FIG. 2: XRD of mechanically alloyed AlFeNbSi powder after 1, 5, 10, 20 and 40 hours at 1400rpm.

Fig. 3 shows the (XRD) of the extruded materials Al_{90.8-Fe_{6.2}}V_{1.0-}Si_{2.0} during 5 and 10 hours of milling at 1400rpm, during 10 and 20 hours at 800rpm and extruded materials Al_{90.8-Fe_{6.2}-Nb_{1.0-}}Si_{2.0} during 10 hours of milling at 800 and 1400rpm.
In the bar extruded from Al$_{90.8}$-Fe$_{6.2}$-X$_{1.0}$-Si$_{2.0}$ (X= Nb or V) powder for 10h at 800rpm, the hexagonal Al$_8$Fe$_2$Si phase, with cell parameters $a=1.243$nm and $c=2.626$nm, was found (Fig. 3a,b). In the same pattern, the Si peak disappeared, indicating reaction of this element to form the hexagonal phase.

In the bar extruded from Al$_{90.8}$-Fe$_{6.2}$-X$_{1.0}$-Si$_{2.0}$ (X= Nb or V) powder for 5 and 10 h at 1400 rpm, cubic phase $\alpha$-Al$_{12}$(Fe,X)$_3$Si was identified. This quaternary alloy has been obtained by rapid solidification [11-13] and is based on crystallographic studies carried out by Cooper on ternary alloys $\alpha$-(AlMnSi) and $\alpha$-(AlFeSi) [14,15]. It’s can bee observed in the XRD of the Al$_{90.8}$-Fe$_{6.2}$-Nb$_{1.0}$-Si$_{2.0}$ material, the AlNb$_2$ phase to stay remained in steady state. The intermetallic phase AlNb$_2$ present cell parameter $a=0.997$nm and $c=0.517$nm.

It is important to note, the extruded bar from MA powder for 10 hours of milling at 800rpm from two composition, the phase formation is Al$_8$Fe$_2$Si. This is suggest that the rotation of the mill, and the time of process, influence in the increase of the interface energy among the powder particles for formation of a phase or other phase.

Fig. 4a show the transmission electron micrograph of the Al$_8$Fe$_2$Si phase in the extruded bar of the alloy milled for 10h at 800rpm. This phase is irregular and nearly spherical with diameter approximately 500nm.

Fig. 4b show the $\alpha$-Al$_{12}$(Fe,V)$_3$Si phase in the transmission electron micrograph of the extruded bar of the alloy milled 10h at 1400rpm. This phase also has a nearly spherical form, with diameter in the range, 50 to 100nm. Microanalysis of this phase by EDS in the TEM presented the composition Al$_{77.29}$Fe$_{10.53}$V$_{1.23}$Si$_{6.95}$ (at%). This represents only an approximate composition of the quaternary $\alpha$-Al$_{12}$(Fe,V)$_3$Si phase.

Fig. 4c show the $\alpha$-Al$_{12}$(Fe,Nb)$_3$Si and AlNb$_2$ phase also in the transmission electron micrograph of the extruded bar of the alloy milled for 10h at 1400rpm. The $\alpha$-Al$_{12}$(Fe,Nb)$_3$Si phase also has a nearly spherical form, with diameter in the range, 20 to 40nm. Microanalysis of this phase by EDS in the TEM presented the composition Al$_{77.95}$Fe$_{13.55}$Nb$_{2.97}$Si$_{5.53}$ (at%). This represents only an approximate composition of the quaternary $\alpha$-Al$_{12}$(Fe,Nb)$_3$Si phase. The AlNb$_2$ phase has a nearly polygonal morphology, with diameter in the range 50 to 65nm.

The formation of the intermetallic $\alpha$-Al$_{12}$(Fe,X)$_3$Si phases observed in extruded bars takes place during heating of the milled powder for pressing and extrusion. Formation of the $\alpha$-Al$_{12}$(Fe,V)$_3$Si phase in powder heated after mechanical alloying has been reported [10].

**FIG. 3:** XRD of the extruded bar, (a) AlFeVSi-10h at 800rpm, (b) AlFeNbSi-10h at 800rpm, (c) AlFeVSi-20h at 800rpm (d) AlFeVSi-5h at 1400rpm (e) AlFeVSi-10h at 1400rpm (f) AlFeNbSi-10h at 1400rpm.
Vickers microhardness (HV$_{100}$) at room temperature, the yield strength, tensile strength and elongation of the extruded bars at room temperature are presented in Table 1. The value of the Vickers microhardness is the mean of 12 determinations. The yield strength, tensile strength and elongation values are the means values of three determinations on samples from different extrusions.

Fig. 5 shows the ultimate tensile strength measured as a function of test temperatures of the extruded bar of the AlFeXSi (X=V or Nb) alloy processed with MA powders milled for 10h at 800 and 1400rpm. It can be observed in the figure that the UTS of the AlFeXSi alloy decrease with the increase rotation of milling.

The alloy AlFeXSi have a typical ductility. At room temperature, elongation is 5.8%. It’s decrease with increasing test temperature. At 200°C elongation is 3.4% and at 350°C it is 3.0%. The mechanical properties of the material is probably due to the nanometric size precipitates of the Al$_{12}$(Fe,V)$_3$Si phase.

Fig. 4: TEM bright-field micrographs showing: (a) extruded material from powder for 10h at 800rpm consisting of Al$_8$Fe$_2$Si phase and α-Al, (b, c) extruded material from powder for 10h at 1400rpm consisting of α-Al$_{12}$(Fe,X)$_3$Si (X=V or Nb) and α-Al.

<table>
<thead>
<tr>
<th>Material</th>
<th>HV$_{100}$ (Kgf/mm$^2$)</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>Elong. (%)</th>
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<tbody>
<tr>
<td>AlFeNbSi (0 h)</td>
<td>32 (±6)</td>
<td>145(±14)</td>
<td>135</td>
<td>12.5</td>
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<tr>
<td>AlFeNbSi (5h 800rpm)</td>
<td>50 (±5)</td>
<td>196(±12)</td>
<td>169</td>
<td>11.5</td>
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<tr>
<td>AlFeNbSi (10h 800rpm)</td>
<td>115 (±3)</td>
<td>337(±18)</td>
<td>286</td>
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<tr>
<td>AlFeNbSi (10h 1400rpm)</td>
<td>215 (±3)</td>
<td>547(±11)</td>
<td>497</td>
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<tr>
<td>AlFeVsSi (10h 800rpm)</td>
<td>114 (±4)</td>
<td>316(±9)</td>
<td>262</td>
<td>6.2</td>
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<tr>
<td>AlFeVsSi (5h 1400rpm)</td>
<td>145 (±4)</td>
<td>364(±12)</td>
<td>332</td>
<td>6.0</td>
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<tr>
<td>AlFeVsSi (20h 800rpm)</td>
<td>183 (±5)</td>
<td>507(±10)</td>
<td>421</td>
<td>5.8</td>
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<tr>
<td>AlFeVsSi (10h 1400rpm)</td>
<td>209 (±4)</td>
<td>524(±10)</td>
<td>478</td>
<td>5.8</td>
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</table>
Fig. 5: UTS measured as a function of test temperatures of the AlFeXSi

4- Conclusions

The addition of control agents like stearic acid, methanol or polyethylene based wax are not necessary when the milling is performed in a polyethylene jar.

The AlNb₂ phase were detected in the mechanical alloying powder and the cubic $\alpha$-Al₁₂(Fe,X)₃Si phase were detected only after extrusion of the mechanical alloying powders, due to the reaction between the elements during heating of the alloy.

The ultimate tensile strength, higher in the extruded material from powder milling for 10h at 1400rpm, can be attributed to the process of mechanical alloying that contributed in the formation of the cubic $\alpha$-Al₁₂(Fe,X)₃Si phase.

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References