PROCEEDINGS OF THE INTERNATIONAL CONFERENCE NANOMATERIALS: APPLICATIONS AND PROPERTIES
Vol. **1** No 3, 03CNN12(3pp) (2012)

Formation of Nanocrystalline Solid Solution in Al-Fe-V-Si Alloys by Mechanical Alloying

H. Ashrafi*, M.H. Enayati†, R. Emadi‡

Isfahan University of Technology, Isfahan 84156-83111, Iran

(Received 18 June 2012; published online 22 August 2012)

In this study, the microstructural evolution of Al-Fe-V-Si alloy prepared by mechanical alloying starting from elemental powders was studied. X-ray diffraction results showed that by increasing the milling time, peak shifting and peak broadening of Al reflections occur due to dissolution of alloying elements and grain refinement, respectively. Reduction of Al lattice parameter by increasing the milling time indicates that an Al based solid solutions formed during mechanical alloying and solute concentration increased by increasing the milling time. After 60 h of milling, the microstructure consisted of a nanocrystalline Al solid solutions with a grain size of 19 nm and an internal strain of 0.55 in which Si phase was dispersed. In contrast to previous studies on rapidly solidified Al-Fe-V-Si alloys, there is no formation of trace of $Al_{12}(Fe,V)_3Si$ or other intermetallic compounds in the as-milled condition.

Keywords: solid solutions; Al-Fe-V-Si alloy; nanocrystalline; mechanical alloying.

PACS numbers: 81.20.Ev, 81.05.Bx

1. INTRODUCTION

Al-Fe-V-Si alloys are of interesting high temperature Al alloys due to their good combination of ductility, room and high temperature strengths and fracture toughness attributed to uniformly distributed $Al_{12}(Fe,V)_3Si$ intermetallic phase, which are resistant to coarsening [1]. The low coarsening rate of $Al_{12}(Fe,V)_3Si$ phase at elevated temperatures is a result of low solid solubility and low diffusivity of the alloying elements in Al, that are basic requirements for prevent Ostwald ripening [2,3]. Because of low coarsening rate and high volume fraction of $Al_{12}(Fe,V)_3Si$ intermetallic phase that inhibit recrystallization and limiting grain growth, the mechanical properties of Al-Fe-V-Si alloys do not changes significantly even after 100 hours annealing at temperatures as high as 500°C [1].

Al-Fe-V-Si alloys are produced by rapid solidification techniques such as melt spinning [4], gas atomization [5] and spray co-deposition [6]. The microstructure of rapidly solidified alloys often consisted of fine precipitates of $Al_{12}(Fe,V)_3Si$ phase in ultrafine grained Al matrix [4]. Recently, mechanical alloying (MA) become of widespread interest method for the producing of aluminium alloys [3,7,8]. MA is a non-equilibrium solid state processing which used to produce a variety of materials and alloys: super saturated solid solutions, amorphous materials, intermetallic compounds and metal-matrix composites [9]. It was found that MA led to formation of nanostructured solid solution in many Al-Fe base systems with high Al contents [3,7].

The aim of this study is to investigate the effect of MA on microstructure of Al-Fe-V-Si alloys starting from elemental powders. Then, a comparison has been performed between the as-milled products and those for rapidly solidified Al-Fe-V-Si alloys that found in the literatures.

2. EXPERIMENTAL

Mixtures of pure Al (99.5%, 10 – 40 μm, Khorasan powder metallurgy), Fe (99.8%, 100 μm, merck), V (99.5%, 500 μm, fluka) and Si (99.8%, 20 – 80 μm, merck) with a nominal composition of Al-11.7Fe-1.3V-2.3Si (wt.%) were used as starting materials. MA was performed in a high energy planetary ball mill (Fritsch P7 type) at room temperature and argon atmosphere with a rotation speed of 500 rpm and a ball- powder mass ratio of 10:1 for 60 h. In order to preventation of severe sticking of Al powders to balls and vial, 1.5 wt.% stearic acid was used as process controlling agent (PCA). Structural changes of powders during ball milling was determined by X-ray diffraction (XRD) in a Philips X' PERT MPD diffractometer using filtered Cu Kα radiation ($\lambda = 0.15406$ nm). The morphology and microstructure of as-milled powder was characterized by scanning electron microscopy (SEM) using a Philips XL30 at an accelerating voltage of 30 kV. The Grain size and internal strain of milled powders were evaluated from the XRD line broadening using the Williamson-Hall equation [10]:

$$\beta Cos\theta = \frac{0.9\lambda}{D} + 2\varepsilon Sin\theta \qquad (2.1)$$

Where β is peak breadth at half maximum intensity, θ is the Bragg angle, λ is the wavelength of the X-ray (0.15406 nm), D is the average grain size and ε is the mean value of internal strain.

3. RESULTS AND DISCUSSION

The XRD patterns of alloy powder mixture at different milling times are shown in Fig. 1. As seen, in the initial mixture of alloy powders, only the diffraction peaks of Al and Si can be seen. The absence of Fe and V diffraction peaks are due to overlapping and low concentrations, respectively. By increasing the

* h.ashrafi@ma.iut.ac.ir
† ena78@cc.iut.ac.ir
‡ remadi@cc.iut.ac.ir

milling time, two distinct features are noted in this figure, one is peak broadening of Al reflections and the other is peak displacement of Al to higher angles. The peak broadening is due to the grain refinement and introduction of internal strains, whereas peak displacement suggests formation of an Al based solid solutions. It must be noted that after 60 h milling, three small peaks of Si still existed on the XRD pattern, indicating that an amount of this element has been dispersed in Al matrix as small isolated particles.

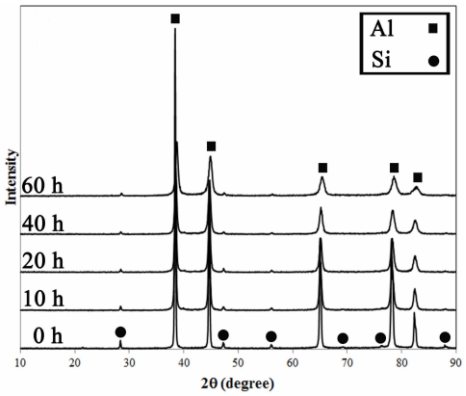

Fig. 1 – The XRD patterns of Al-11.7Fe-1.3V-2.3Si powder mixture after different milling times

Formation of an Al based solid solutions can better be understood from the changes in Al lattice parameter. Variations of Al lattice parameter as a function of milling time is presented in Fig. 2. It is noted that the lattice parameter decreases by increasing the milling time. The observed decrease in the lattice parameter indicates dissolution of smaller Fe, V and Si atoms (Table 1) into fcc Al and formation of an Al-based solid solutions. By increasing the milling time, solute concentration increased.

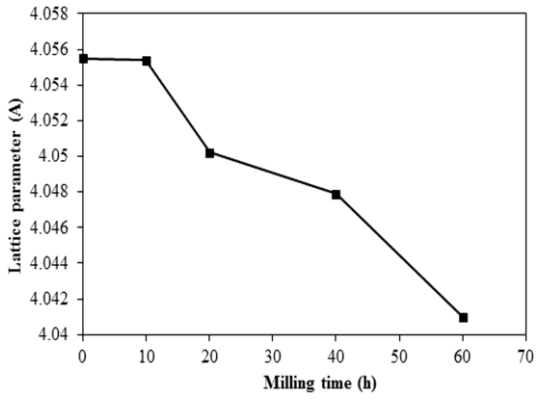

Fig. 2 – Variations of Al lattice parameter as a function of milling time

Table 1 – Atomic radius of alloying elements

element	Al	Fe	V	Si
Atomic radius (nm)	0.143	0.126	0.111	0.134

Table 2 presents the lattice parameter, grain size and internal strain of Al in the as-mixed state and after 60 h MA. As observed, the estimated grain size and internal strain of Al after 60 h of MA were 19 nm and 0.55, respectively.

Table 2 – Lattice parameter, grain size and internal strain of Al at different conditions

condition	lattice parameter (A)	Grain size (nm)	Internal strain (%)
as-mixed	4.06	350	-
60 h MA	4.044	19	0.55

Cross-sectional SEM image of 60 h mechanically alloyed powders are presented in Fig. 3. A nearly uniform microstructure is seen in the SEM image, but some bright particles with a size smaller than 1μm also observed in the matrix, indicates that all the alloying elements have not been dissolved in Al matrix. The uniform microstructure in this stage shows that alloying take place well and nearly a homogeneous microstructure has been obtained after MA.

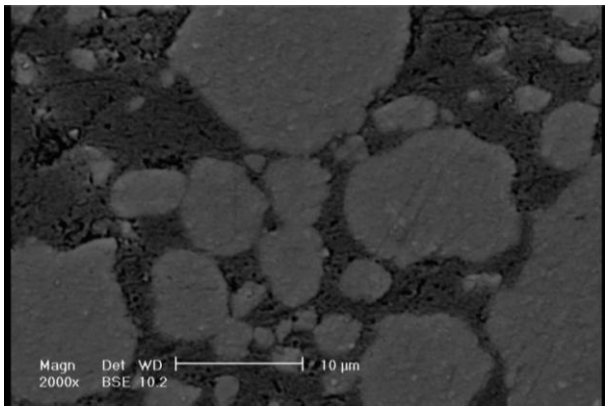

Fig. 3 – Cross-sectional SEM image of 60 h mechanically alloyed powder mixture

Fig. 4 illustrates the morphology of alloy powder particles after 60 h of ball milling. As seen, after 60 h milling the particles became very fine due to the ball-powder-ball collisions and fracturing of brittle powder particles during MA. The milled powder particles exhibit equiaxed morphology with an average particle size of 12 μm.

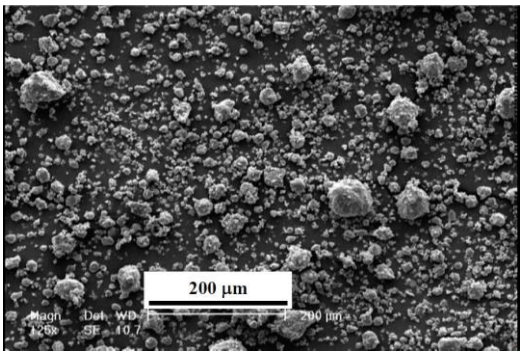

Fig. 4 – Morphology of alloy powder mixture after 60 h milling

Table 3 compares the phases presented after MA of Al-Fe-V-Si alloys with those obtained by rapid solidification techniques. As seen, rapid solidification techniques lead to formation of intermetallic phases in Al matrix, while in the present work no trace of any intermetallic compound

was detected after MA. The microstructure of as-milled product consisted of nanocrystalline Al solid solution in which Si phase was dispersed.

Table 3 – Comparison between presented phases after MA and various rapid solidification techniques of Al-Fe-V-Si alloys

process	phases after process	reference
MA	Al+Si	This work
Melt spinning	$Al+Al_{12}(Fe,V)_3Si$	[4]
Spray co-deposition	$Al+Al_{12}(Fe,V)_3Si+Al_{13}Fe_4$	[6]
Gas atomization	$Al+Al_{12}(Fe,V)_3Si$	[5]

4. CONCLUSIONS

The present study of Al-11.7Fe-1.3V-2.3Si alloy shows that nanocrystalline Al-based solid solutions can be formed by MA of elemental powders. XRD results showed that during mechanical alloying, peak broadening of Al reflections occurs as a result of grain refinement and enhancement of internal strains. Reduction of Al lattice parameter indicates dissolution of smaller Fe, V and Si atoms into the Al lattice and formation of a nanocrystalline Al solid solution. In contrast to previous studies on rapidly solidified Al-Fe-V-Si alloys, there is no formation of $Al_{12}(Fe,V)_3Si$ or other intermetallic compounds in the as-milled condition.

REFERENCES

1. R.E. Franck, J.A. Hawk, *Scripta Metall.* **23**, 113 (1989).
2. S.C. Khatri, A. Lawley, M.J. Koczak, *Mater. Sci. Eng. A* **167**, 11 (1993).
3. M. Zawrah, L. Shaw, *Mater. Sci. Eng. A* **355**, 37 (2003).
4. J.Q. Wang, M.K. Tseng, X.F. Chen, B.J. Zhang, Z. Xianyu, *Mater. Sci. Eng. A* **179**, 412 (1994).
5. S. Hariprasad, S.M.L. Sastry, K.L. Jerina, *J. Mater. Sci.* **31**, 921 (1996).
6. Y. Tang, D. Tan, W. Li, Z. Pan, L. Li, W. Hua, *J. Alloys Compd.* **439**, 103 (2007).
7. T.T. Sasaki, T. Mukaib, K. Hono, *Scripta Mater.* **57**, 189 (2007).
8. S. Mula, S. Ghosh, S.K. Pabi, *Mater. Sci. Eng. A* **472**, 208 (2008).
9. C. Suryanarayana, *Prog. Mater. Sci.* **46**, 1 (2001).
10. G.K. Williamson, W.H. Hall, *Acta Metall.* **1**, 22 (1953).