Journal of Metastable and Nanocrystalline Materials Vols. 20-21 (2004) pp. 207-212
online at http://www.scientific.net

Al-Fe-X-Si (X = V or Nb) Alloys Powders Prepared by High Energy Milling in an Attritor Mill

Rodrigo Estevam Coelho[1], Ricardo Mendes Leal Neto[2]
Priscilla Alves Camargo[3] and Francisco Ambrozio Filho[2]

[1]Escola Técnica Federal da Bahia, CEFET, Salvador, Brazil
[2]Centro de Ciência e Tecnologia de Materiais, IPEN, São Paulo, Brazil
[3]Escola de Engenharia Mackenzie, São Paulo, Brazil

Keywords: Aluminium Alloy, Attritor Mill, High Energy Milling, Mechanical Alloying

Abstract. This paper presents the solid state interaction of an alloy, $Al_{90.8}Fe_{6.2}(V \text{ or } Nb)_{1.0}Si_{2.0}$ (at%), prepared by mechanical alloying elemental powders of Al, Fe, V or Nb and Si in a horizontal attritor mill. The powder mixture was attrition milled in an nitrogen atmosphere with 0,5% of polyethylene, speed of 1500 rpm (rotation per minute), and milling time 1, 3, 5, 7, 10 hours. The powders milled for 10 hours were heat treated 1 hour at 300°C, 400°C and 500°C.
The evolution of the as milled powders size and shape with milling time was shown by laser difraction and scanning electron microscopy. The phases in the as milled and heated treated powders were determined by X-ray diffraction. New phases were detected after heat treating the milled powders: Al Nb_2 above 300°C and Al_{12} (Fe, V or Nb)$_3$ Si above 400°C.

Introduction

The use of cast aluminum alloys is normally limited to a maximum temperature of 150 °C. Even age hardening of these alloys does not assure their use at/or above this temperature. The addition of a third or fourth alloying element to binary alloys, with low solubility and diffusivity in Al, and the use of new preparation methods could promote the formation of new intermetallic precipitates that are harder and more stable, guaranteeing thus higher operation temperatures for these alloys[1].

The alloys of the quaternary system Al-Fe-V-Si have a good combination of strength, ductility, and toughness at room and high temperatures (up to 450 °C). Vanadium is an essential element to stabilize the cubic phase $Al_{12}(Fe,V)_3Si$ in the Al-Fe-V-Si alloy. The volume percent of the $Al_{12}(Fe,V)_3Si$ precipitate can be between 17 and 40%, depending on the processing method and composition of the alloy.

The rapidly solidified Al-Fe-V-Si alloy obtained by melt spinning inhibits the formation of intermetallics like Al_8Fe_2Si and Al_3Fe and favours $Al_{12}(Fe,V)_3Si$ precipitation. This precipitate is homogeneously distributed inside grains and at grain boundaries in the aluminum matrix, and its size varies between 40 and 80 ηm. The main advantage of this alloy compared to other aluminum alloys is the formation of this stable precipitate, which does not coalesce and inhibits recrystallization even after long periods at high temperatures [1-6].

This quaternary alloy has been studied [2,4] and is considered that the intermetallic $Al_{12}(Fe,V)_3Si$ changes the cristallyne system due to the substitution of Fe by V and a consequent modification of the interphase energy matrix/precipitate. [7], [8].

Mechanically alloyed (MA) aluminum alloys such as AlTiNb [9], AlFeMn [10], AlMnCe [11] have been studied but there are only a few papers on MA Al-Fe-(V or Nb)-Si alloys [12-14].

This paper presents the mechanical alloying of an alloy, $Al_{90.8}Fe_{6.2}(V \text{ or } Nb)_{1.0}Si_{2.0}$ (at%) , prepared from elemental powders of Al, Fe, V or Nb and Si in an horizontal attritor mill.

Experimental Procedure

Two Al-Fe-X-Si (X=V or Nb) alloys in the composition $Al_{90.8}Fe_{6.2}$(V or Nb)$_{1.0}Si_{2.0}$ (at%) or in weight percentage as 83,18Al-11,7Fe-3,15Nb-1,91Si wt % and 84,38Al-11,93Fe-1,75V-1,93Si wt %were prepared by milling in an attritor mill with horizontal shaft from the respective metallic powders. The mean particle sizes of the starting elemental powders were: Al= 50μm, Fe= 100μm, Nb=100μm, V= 1mm and Si=100μm. The milling was performed for 1,3, 5, 7, and 10 hours at 1500 rpm.

The processing parameters are shown in Table 1.

Table 1: Milling parameters

Jar volume (l)	2
Powder mass (g)	150g
Ball mass (g)	1500
Ball diameter (mm)	7
Shaft speed (rpm)	1500
Process control agent	0,5% of polyethylene
Flowing gas	Nitrogen
Cooling media	Water
Processing time (h)	1,3,5,7,10

The $Al_{90,8}Fe_{6,2}X_{1,0}Si_{2,0}$ (at.%) (X=Nb ou V) powders milled for 10 hours were heat treated 1 hour at 300°C, 400°C and 500°C.

The characterization methods utilized were:

X-ray diffraction – to identify the phases after milling and after heat treating of the powders. X-ray diffraction measurements were carried out in a Rigaku diffratometer with CuK_α radiation (wavelength of 15,42 ηm), scanning angles from 10 to 100°.

Scanning electron microscopy – to observe the morphology of the powders after milling. The powders were dispersed over a conducting tape on the sample holder. The powders were observed in a scanning electron microscope XL-30.

Granulometric analysis – to verify the milling time effect on the powder particle size. The results were obtained as a cumulative mass versus particle size. The mean particle size was obtained from the 50 weight % accumulated. The powder in water with detergent was analysed in a laser scattering Cilas 1064 after dispersion by ultrasonic agitation.

Fig
3h,

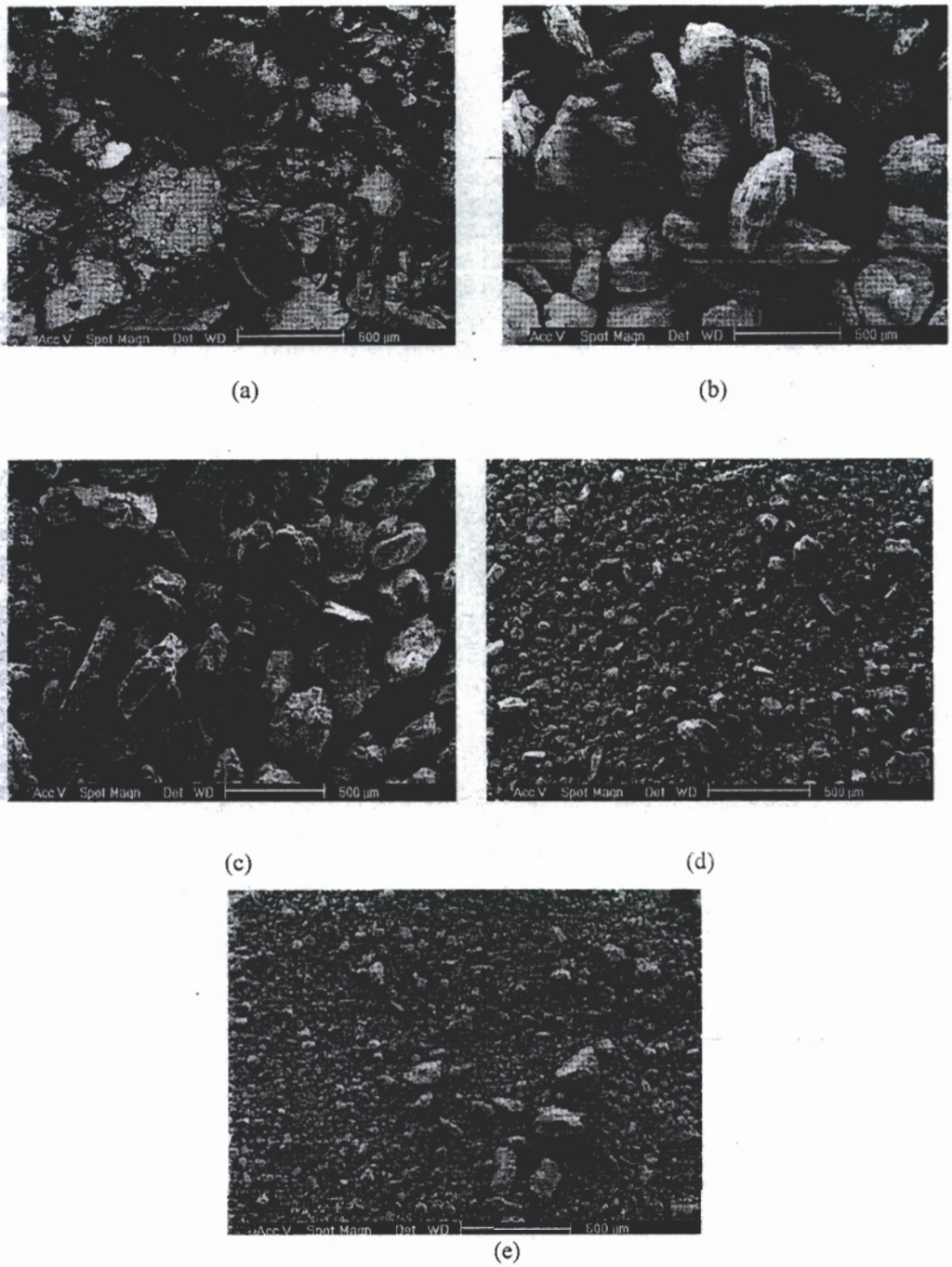

Figure 1: Scanning Electron microscopy of the as milled powder of the $Si_{2.0}$ (at.%) alloy. (a) 1h, (b) 3h, (c) 5h, (d) 7h e (e) 10 h, at 1500 rpm.

Results and Discussion

Figure 1 presents micrographs by scanning electron microscope of the powder particles after milling at 1,3,5,7 and 10 hours with 1500 rpm for the $Al_{90,8}Fe_{6,2}Nb_{1,0}Si_{2,0}$ (at.%) alloy. The evolution of the particle shape is shown in this figure and it can be seen after 10 hours and 1500 rpm a change from flake to a more round shape. Similar results were obtained for $Al_{90,8}Fe_{6,2}V_{1,0}Si_{2,0}$ (at.%) alloy.
Table 2 shows that the mean particle size does not change too much after 7 hours milling. So it is expected no great changes after milling for longer times.

Table 2 – Mean particle size (μm) – particle size corresponding to a mass accumulated of 50% for different milling times.

time / alloy	1 hour	3 hours	5 hours	7 hours	10 hours
Al-Fe-Nb-Si	49	135	100	53	40
Al-Fe-V-Si	178	277	86	64	66

Figures 2 and 3 show the X-ray diffractograms of the milled powders at 1500 rpm for different times and figures 4 and 5 the X-ray diffractograms of the milled powders at 1500 rpm for 10 hours and heat treated at different temperatures

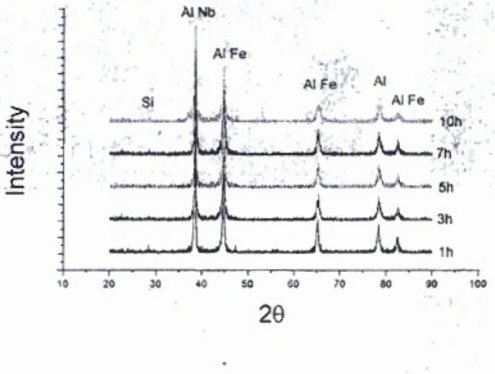

Figure 2: X-ray diffractograms of the powder AlFeNbSi milled at 1500 rpm for different milling times

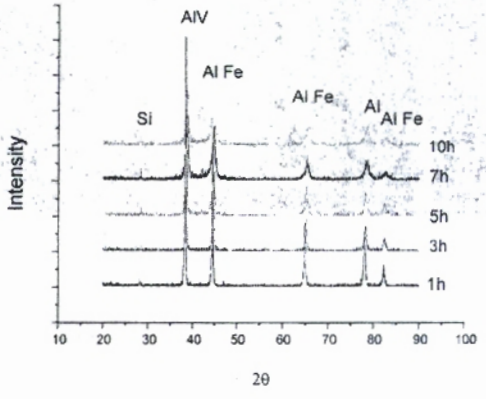

Figure 3: X-ray diffractograms of the powder AlFeVsi milled at 1500 rpm for different milling times

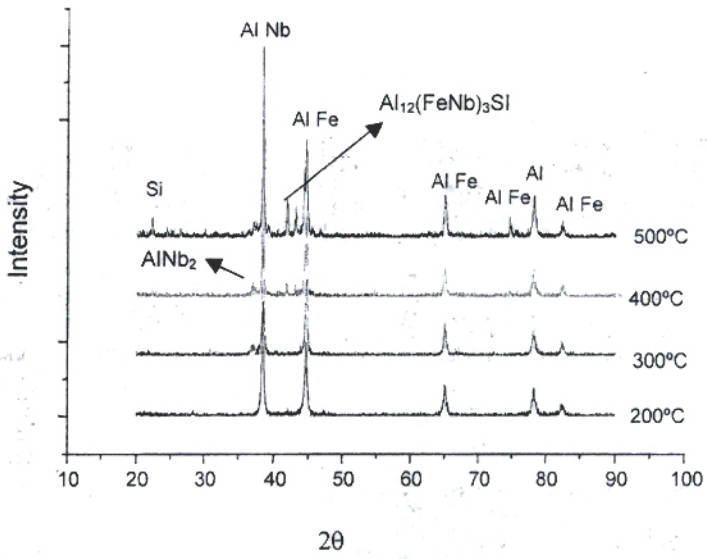

Figure 4: X-ray diffractograms of the powder AlFeNbSi milled at 1500 rpm for 10 hours after 1 hour heat treating in different temperatures

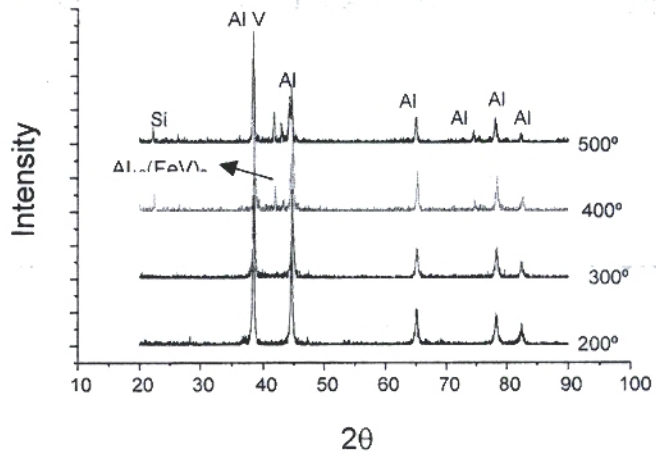

Figure 5 X-ray diffractograms of the powder Al FeVSi milled at 1500 rpm for 10 hours after 1 hour heat treating in different temperatures

No new phase was detected by X-ray diffraction after milling. The pattern showed peaks corresponding to the elements Al, Fe, V or Nb and Si. The diffraction peaks of Fe and Nb or V are in the same position of the Al, not clearly identifying these elements.
After heat Treating above 400°C for 1 hour the cubic phase $Al_{12}(Fe,X)_3Si$ (X=V or Nb) was identified (12,13). The phase $AlNb_2$ was clearly identified after heat treating above 300 C. This phase was found just after high energy milling in an attritor with the same alloy composition (13).

A previous work (13) has shown that the $AlNb_2$ phase has a nearly polygonal morphology, with diameter in the range of 50 to 65 μm and $Al_{12}(Fe,X)_3Si$ (X=V or Nb) phase has a nearly spherical form, with diameter in the range of 20 to 80 μm.

Conclusions

In the attrition milling with horizontal shaft of Al, Fe, V or Nb and Si elemental powders with the composition $Al_{90.8}Fe_{6.2}(V \text{ or } Nb)_{1.0}Si_{2.0}$ (at%) it was obtained:

- The particle size does not change after 7 hours milling at 1500 rpm
- It was not observed new phases after milling for 1,3,5,7 and 10 hours at 1500rpm
- The phase $Al_{12}(Fe,X)_3Si$ (X=V ou Nb) is formed during heat treating above 400C in the powders milled for 10 hours at 1500 rpm
- The phase $AlNb_2$ was detected by X ray diffraction after heat treating above 300^0C

Acknowledgements

The authors wish to thank CNPq and Fapesp for the financial support of this project and the following laboratories for the use of their equipment: Geo-Ciências-USP (SEM) and IFUSP (TEM).

References

[1] R. Rainen; J Ekvall, J. Metals, (1988) p.16-18.
[2] R. L Bye, N. J. Kim, D. J. Skinner, D. Rayboud, A. M. Brown, Allied-Signal Co. (1987) 283-289.
[3] D. J. Skinner, R. L. Bye, D. Raybould, A. M. Brown, Scr. Metall 20 (1986) 867-972.
[4] V. R. V. Ramanan, D. J. Skinner, M. S. Zedalis, Mater. Sci. Eng. (1991) 912-916.
[5] C. Surynarayana; F.H.Froes; S.Krishnamurthy; Y.W.KIM, v.26, n.2, 1990, p.117-29,.
[6] D. J. Skinner, J. Min. Metals Mater. Society, (1988) 181.
[7] F.H Froes; J.J.Barbadillo; & C.Surynarayana, - Development, Technology Transfer, and Application of Advanced Aerospace Structural Materials. 1991, pp. 01-14.
[8] R.E.Frank, & J.A. Hawk, - Effect of Very High Temperatures on The Mechanical Properties of Al-Fe-V-Si, Alloy, Scripta Metallurgica, 1989, vol. 23, pp. 113-118.
[9] D. C. Jia, Y. Zhou, T. C. Lei, Mater. Sci. Eng. A232 (1997) 183-190.
[10] X. Niu, P. Brun, L. Froyen, C. Peytour, L. Delaey, Powder Metall. Int. v.25, n.3, (1993) 118-124.
[11] Yu Ji, Marke Kallio, Tuomo Tiainen, Scr. Mater. 42 (2000) 1017-1023.
[12] L. J Zheng, J. X. Lin, B. S. Zhang, M. K. Tseng, Mater. Sci. Forum. v.331-337 (2000) 1225-8.
[13] R. E., Coelho, F. Ambrozio, Lima, S. J. Materials Science Forum, v.416-418 (2003) 176-182.
[14] R. E., Coelho, F. Ambrozio, Key Engineering Materials, Vols 189-191, 2001, pp 555-560