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Production of Al-Fe-Nb-Si Alloys by Mechanical Alloying and Hot Extrusion

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Abstract. The present work shows the solid state interaction among Al, Fe, Nb and Si elemental powders, mixed by mechanical alloying (MA) and consolidated by hot extrusion. The MA powders and extruded materials were observed by scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and X-ray diffraction. The tensile tests and Vickers hardness were performed on the as-extruded material, as well as hydrostatic density measurement. Several kinds of dispersoids were detected in this alloying, suggesting that they are the main causes for the enhanced mechanical properties.

Introduction

Mechanical alloying (MA) is a high-energy ball milling process developed by INCO in 1960's to produce a material combining oxide dispersion strengthening in a nickel-based super-alloy purposed for gas turbine applications [1].

MA is important to establish the balance between cold welding and fracturing. Recently, MA has also been applied to produce supersaturated solid solutions and nanocrystalline material with novel properties. The application of milling produces a uniform fine refractory particle [2].

While most scientific attention in the use of mechanical alloying has been applied on metastable phases by this technique, intermediate phases and intermetallic compounds have been synthesized from the pure components in several alloy systems. MA has been known to be a versatile tool, as it enhances the limited solubility imposed by the equilibrium phase diagram.

A nonequilibrium phase was therefore also present, as a result of plastic deformation that is an integral part of MA. The nano-structures 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 [3].

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 alloys with the addition of transition elements and some rare earth elements produced by MA, are possible candidates for high - temperature applications [4].

Disordering of an ordered phase by mechanical alloying may occur with the formation of various kinds of defects in the ordered matrix. The lattice parameter of the disordered phase can be either larger or smaller than that of the ordered phases, depending on which kinds of defects are introduced in ordered phases by mechanical deformation [5].

Experimental Procedure

The mean size of the elemental powders of crystalline Al (40μm) with a purity of 99.99 %, Fe (100μm), Nb (100μm) and Si (100μm) were used for the experiments. The powders were mixed to the initial composition Al-11.7(wt%) Fe-3.2(wt%) Nb- 1.9(wt%) Si and milled in a high-energy mill (attritor) in a nitrogen atmosphere with the ball-to-powder weight ratio of 9:1 and impeller velocity of 800 rpm. The milling vial was made of polyethylene with 81 mm in diameter. The balls were made of chrome steel with 7mm in diameter. After milling for 10 h, the powders were removed in air atmosphere and vacuum hot pressed under a pressure of 530 MPa and a temperature at 340 ° C. The compacted billet of 31 mm in diameter by 30 mm in length, was hot at 500 ° C extruded with the rate of 20:1.

Microstructures of the powder and extruded bar were investigated by SEM, EDS and X-ray diffraction. The tensile properties of the extruded alloy were determined at room temperature and crosshead speed of 0.5 mm/min. Vickers hardness of the extruded material was measured with load, 0.1 Kgf with eight marks. Densities of specimens were measured by Archimedes's method. SEM was used to characterize the bending fracture surfaces of the specimens.

Results and Discussion

Fig. 1 presents size and morphology of the MA powders after 10 h of milling. It shows the typical flake shape of the powder particles, suggesting successive fracture and welding.

SEM image of the extruded material, in vertical to the hot extrusion plane, can be seen in the Fig.2. The microstructure of particles, which was mapping by EDS analysis, the elements Fe, Nb and Si distributed in the Al matrix were observed.

The X-ray diffraction of MA powders is presented in Fig. 3. The crystalline phase initial pure Al, Fe, Nb, Si elements remain the same and lattice parameter (a = 0.4049, 0.2866, 0.3307, 0.5431 ηm) respectively. The line intensity decreases and enlarges compared to that of simple mixture of Al, Fe, Si and Al, Fe, Nb X-ray diffraction patterns [6, 7]. It is evidenced that the material structure was mechanically deformed, facilitating the formation intermetallic phases. Fig. 4 presents X-ray diffraction patterns of the as-extruded material. The X-ray analysis revealed the presence of Al_8Fe_2Si, $Al_{0.5}Fe_3Si_{0.5}$ intermetallic compounds as well as other crystalline phases Al, Fe, Nb. The line intensity of the Si disappears, indicating that it may be in complete solid solution. The lattice parameter of the Al_8Fe_2Si, $Al_{0.5}Fe_3Si_{0.5}$ intermetallic phase and Al, Fe, Nb is shown in Table 1.

Fig.1- Morphology of MA powders after 10 h.

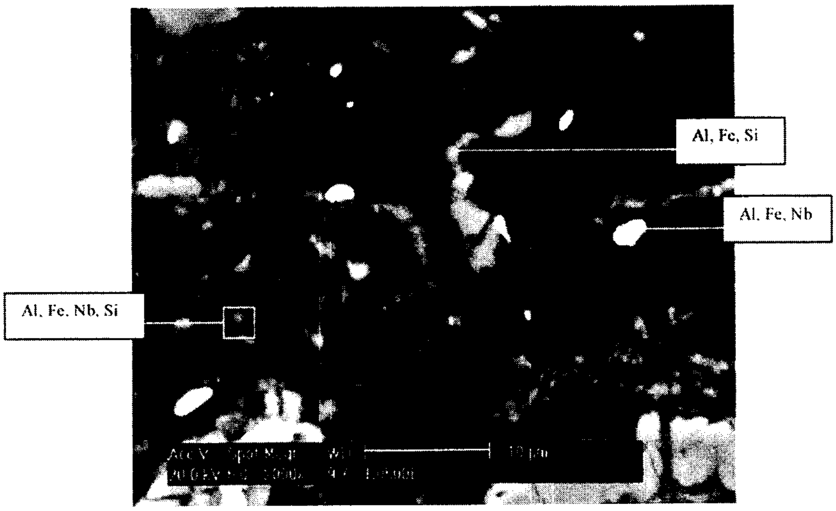

Fig.2- EDS analysis showing distribution of Al, Fe, Nb and Si in the extruded material.

Table 1- Lattice parameter of the phases as extruded material.

Phase	Lattice Parameter (ηm)		Deviate (%)
	Experimental	Calculated	
Al	a=0.4049	a=0.4053	0.10
Fe	a=0.2866	a=0.2866	0.00
Nb	a=0.3307	a=0.3309	0.06
Al_8Fe_2Si (hexagonal)	a=1.2405, c=2.6236	a=1.2429, c=2.6256	a=0.19, c=0.08
$Al_{0.5}Fe_3Si_{0.5}$ (cubic)	a=0.5721	a=0.5733	0.21

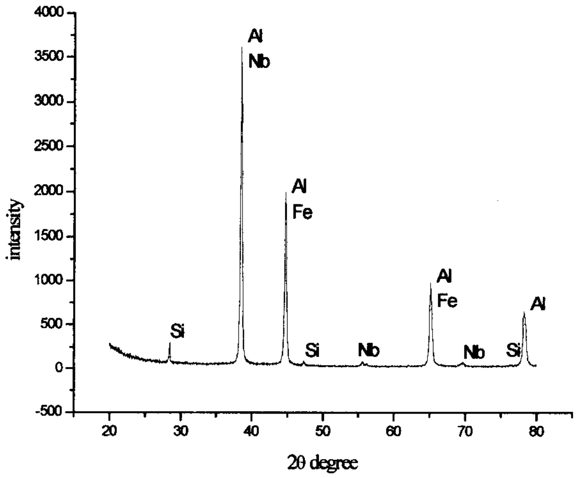

Fig.3- X-ray diffraction pattern of the powder after 10 h MA.

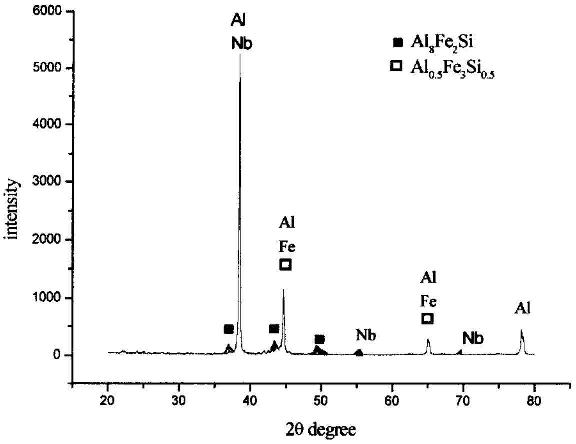

Fig.4- X-ray diffraction pattern of the extruded material.

The tensile properties at room temperature, hardness (HV_{100}) and density of as extruded material are summarized in Table 2.

Table 2 - Room temperature mechanical properties of the as - extruded alloy

Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Hardness (HV_{100})	Density (g/cm^3)	
				Billet	Extruded bar
286	337	5.25	128.9	2.88	3.00

Figure 5 shows the fractography of the as-extruded alloy. All the essential detail was discernible for a microstructure interpretation of the fracture process. The presence of microdimples, caused by small dispersoids in the as-extruded material, reveal a ductile fracture around the dispersoids. Microplasticity at separation, was clearly evident on all fracture surfaces. The same particles are observed at the base of the dimples.

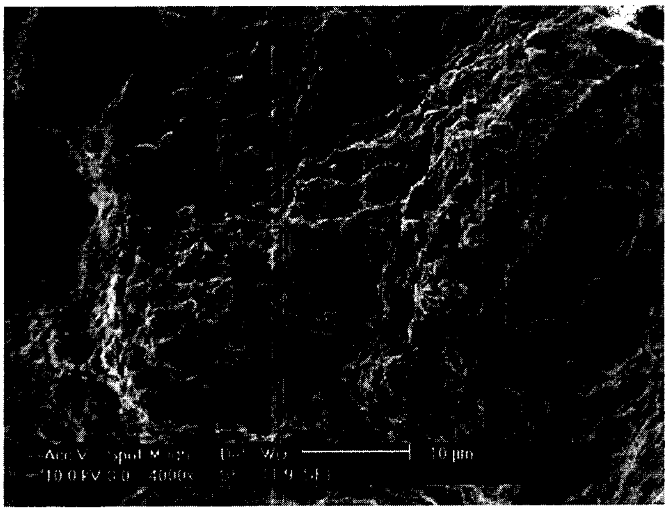

Figure 5- SEM fractograph of $Al_{83.2}$-$Fe_{11.7}$-$Nb_{3.2}$-$Si_{1.9}$ alloy in the extruded.

Conclusions

Mechanical alloying produces a fine particle that facilitates a solid solution of elements in the Al matrix, but the elemental Al-Fe-Nb-Si powder mixture in 10 hours of milling at 800 rpm, no phase change occurs.

The formation of intermetallic phase (Al_8Fe_2Si and $Al_{0.5}Fe_3Si_{0.5}$) occurs during the hot extrusion at 500 ^{0}C.

The degassing temperature of powder at 340 ^{0} C, during the pressing, is important to improve mechanical properties.

The flake morphology powder particles do not impede the consolidation by hot extrusion.
After extrusion the calculated density is equal to the theoretical density.

The SEM fractograph reveals that the formation of the cracks is the main cause for the reduction in tensile stress.

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