Journal of Applied Sciences

ISSN 1812-5654

science alert

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Journal of Applied Sciences 8 (20): 3727-3732, 2008
ISSN 1812-5654

Effect of Silicon on Nanostructure TiAl(γ) Formation Kinetic via Mechanical Alloying Method

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Abstract: With the aim of studying the effect of Si on Mechanical Alloying (MA) process of TiAl (γ) intermetallic compound, two compositional powder blends of Ti-50Al(at%) and Ti-48Al-2.4Si(at%) were mechanically alloyed in a planetary ball mill. The structural evaluation in these powders was done by X-Ray Diffraction (XRD) technique and Scanning Electron Microscopy (SEM). It was found that elemental powders were progressively transformed into nanocrystalline Ti solid solution and amorphous phases during MA process. The time required for Ti solid solution or amorphous phase formation was longer in Si containing powders. While annealing of the mechanically alloyed Ti-50Al powders at 900°C for 10 min leads to the nanocrystalline phase of TiAl(γ), nanocrystalline phases of mainly TiAl(γ) together with some silicides and $Ti_3Al(\alpha_2)$ phase were formed in Si containing powders. Annealing at higher temperature of 1150°C leads unstable silicides to be transformed into more stable ζ-Ti_5Si_3 phase.

Key words: Mechanical alloying, titanium aluminide, silicon, nanostructure, intermetallic

INTRODUCTION

Intermetallic titanium aluminides, particularly TiAl (γ) based alloys, have received a great deal of attention as candidate materials for high-temperature applications such as aerospace and automobile industries because of their low density, high specific strength, good oxidation and corrosion resistance at elevated temperatures (Szewczak *et al*., 1997; Polmer, 2005; Lu *et al*., 2002; Westbrook and Fleischer, 1995). However the poor ductility and fracture toughness at ambient to intermediate temperatures are the major obstacles for practical use of these materials (Kumaran *et al*., 2008; Chau-Jie *et al*., 2006). During recent years, many attempts have been made to improve the ductility. Attempts adapted include techniques such as grain refinement to nano scale structure (Imayev *et al*., 1999; Froes *et al*., 2001) and microstructure modification.

Mechanical alloying process has been shown to be an effective means to achieve grain refinement. MA produces powders with a fine nanoscale grain size and a homogeneous distribution of dispersoids. The process involves repeated flattening, welding, fracturing and rewelding of powder particles in a dry high energy ball mill, which is now extensively employed to produce a great variety of structures, including supersaturated solid solutions, amorphous alloys and nanocrystalline materials (Suryanarayana, 2001; Soni, 1999).

It has also been found that addition of some ternary elements such as Nb, V, Cr and Si improves the high temperature properties of TiAl based alloys. Among these elements Si appeared to be one of the most attractive candidates which raises the creep resistance of the alloy due to ζ-Ti_5Si_3 phase formation as the strengthening constituent (Koch, 1998). While a number of studies have been dedicated to the investigations of reinforcement effect of silicides formed in TiAl based composites, only a few works have been reported on the effect of Si on phase transformations kinetic during MA process. In the present work, the compositional Ti-50Al and Ti-48Al-2.4Si powder blends were used to perform MA process. The study aimed to investigate the structural evaluation of the powders during MA process and also after annealing treatments of mechanically milled powders.

MATERIALS AND METHODS

A planetary mill with two tempered steel vials and Cr-steal balls was employed in this research. The capacity of each vial was 250 mL and diameter of balls were 15 and 20 mm. The powders of Ti, Al and Si with purity of 99.90, 99.98 and 99.82(wt.%) and average particle sizes of 130, 100 and 65 μm, respectively were used for making the powder blends of Ti-50Al and Ti-48Al-2.4Si. The initial weight of powder blends was 10 g which

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weighted in a glove box with argon atmosphere. The vials were designed to allow pumping and subsequent filling by high purity (99.9999%) inert argon gas. The final gas pressure in the vial was kept to be 0.1 MPa. The MA process was carried out at a rotation speed of 550 rpm and ball to powder weight ratio of 20:1. To avoid temperature increases during MA, periods of 0.5 h were alternated with an equal rest time. After alloying for various lengths of time up to 90 h the milled powders were withdrawn from the vials for subsequent analysis. The crystal structure of the powders was characterized by a Bruker-D8-Advanced diffractometer, using Cu-K_α radiation at 30 kV and 20 mA. Analysis of powder morphology and particle size measurements before and after milling was achieved using a Cam Scan MV-2300 SEM equipped with an EDS analyzer at an accelerating voltage of 25 kV. The crystallite size and lattice strain of the powder particles were determined using the X-ray peak broadening techniques (Scherrer and Williamson-Hall formulas):

$$d = 0.9\lambda/B \cos \Theta \quad (1)$$

$$B \cos\Theta = 0.9\lambda/d = \eta \sin \Theta \quad (2)$$

where, d is the crystallite size, λ is the wavelength of the X-radiation used, B is the peak width at half the maximum intensity, Θ is Bragg angle and η is the strain. Some of the as milled samples were annealed using an Alcatel CFA-222 vacuum furnace.

RESULTS AND DISCUSSION

In order to study the effect of Si on kinetic of structural changes during MA process, two compositional Ti-50Al and Ti-48Al-2.4Si powder blends in the presence of 2 wt.% stearic acid ($C_{18}H_{36}O_2$) as a process control agent were mechanically milled for different times at a vial speed of 550 rpm and ball to powder ratio of 20:1.

Comparison of the XRD patterns shown in Fig. 1 and 2 indicates that addition of Si to powder blends leads the MA process rates at various stages to be considerably reduced. For example, in Ti-50Al powder mixture, the Al peaks disappeared after 10 h milling, indicating that the starting powders have completely transformed into Ti solid solution, whereas for Si containing powders longer times up to 40 h is required the Al peaks to disappear and Ti solid solution to be formed. With increasing milling time further, the Ti solid solution phase begins to collapse into the amorphous state so that, after milling the Ti-50Al powder blends for 20 h, only a broad peak of Ti solid solution phase was achieved, indicating that the starting

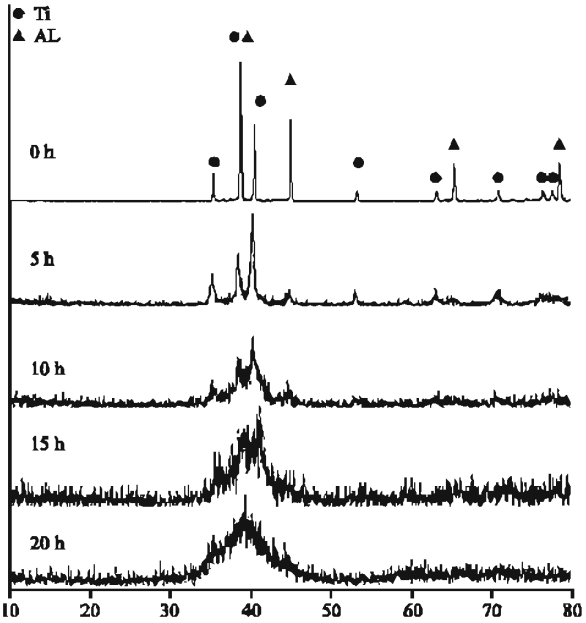

Fig. 1: X-ray diffraction patterns of Ti-50Al powder mixtures at various stages of milling process

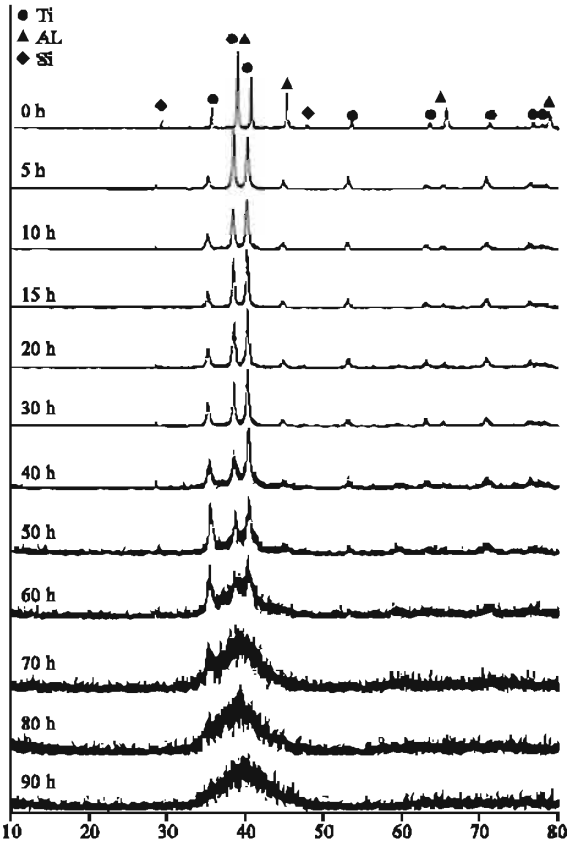

Fig. 2: X-ray diffraction patterns of Ti-48Al-2.4Si powder mixtures at various stages of milling process

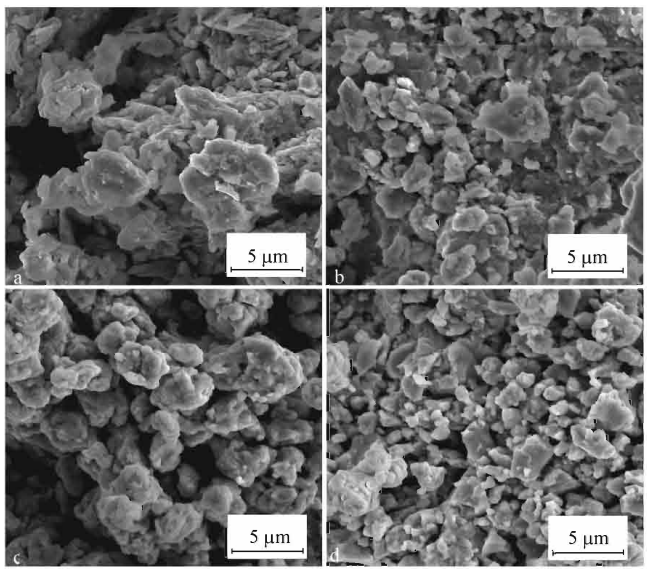

Fig. 3: SEM micrographs of as milled Ti-50 Al powder mixtures for (a) 5, (b) 10, (c)15 and (d) 20 h at vial rotation rate of 550 rpm and ball-to-powder weight ratio of 20:1

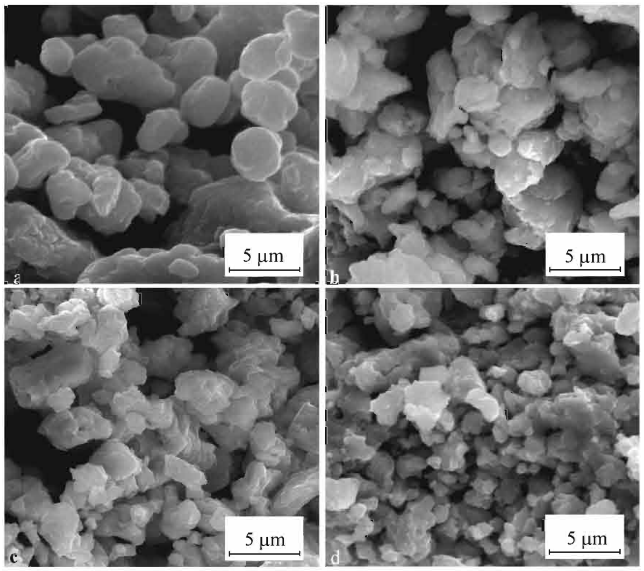

Fig. 4: SEM micrographs of as milled Ti-48Al-2.4Si powder mixtures for (a) 20, (b) 40, (c) 60 and (d) 80 h at vial rotation rate of 550 rpm and ball-to-powder weight ratio of 20:1

powder has been completely transformed into an amorphous phase. Whereas in the case of Si containing powders, longer times up to 90 h was required for the same phase formation process. This might be resulted from lower diffusion rate of Al in Ti in the presence of Si than those of Si free powders.

Comparison of the micrographs indicates that at the early stages of milling the stearic acid was not completely melted and large particles were achieved due to domination of cold welding over fracturing process. But with the gradual melting and adsorption of stearic acid on the surface of particles, the cold welding rate reduces. In both cases, after alloying for a certain lengths of time a balance between welding and fracturing rates was achieved (Fig. 3, 4).

Figure 5 shows the variation in the amounts of powders recovered from the vial as a function of milling time. In general, it has been found that the addition of Si

seems there is a correlation between sticking tendency of powder mixtures to milling tools and their phase transformation from elemental powder to Ti solid solution phase. The same observations has been reported by Takasaki and Furuya (1999) when they studied MA process of three kinds of $Ti_{72}Al_{28}$, $Ti_{57}Al_{43}$ and $Ti_{48}Al_{52}$ powder blends.

X-ray diffraction patterns from mechanically milled amorphous powders of Ti-50Al and Ti-48Al-2.4Si after annealing at 900°C in a vacuum oven for 10 min are shown in Fig. 6 and 7, respectively.

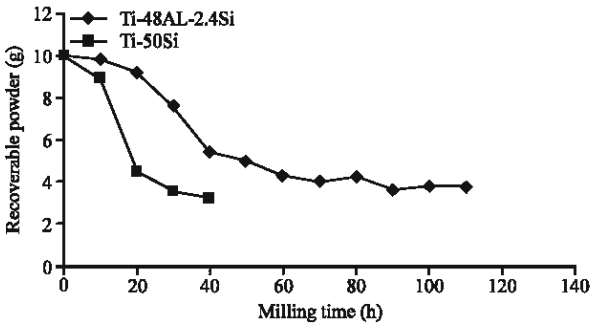

Fig. 5: The amount of recoverable powder products as a function of alloying time

As indicated, while in Si free powders only nano crystalline phase of TiAl(γ) with an average grain size of 25 nm was formed, nanocrystalline phases of mainly to powder blends leeds the amount of recoverable powders to be increased as compared with the Si free powders. This might be due to settling of harder Si particles between powders and milling tools and preventing them from sticking together which results in higher amounts of recoverable powders as compared with those of Si free powders. The Fig. 5 also shows that during structural changes from elemental powder mixtures to Ti solid solution phase, the amount of recoverable powders has been decreased in both types of powders. It TiAl(γ) with mean grain size of 15 nm together with some silicides and TiAl(α_2) phase were formed in Si containing powders. SEM micrographs of both annealed powders are shown in Fig. 8.

Annealing at higher temperature of 1150°C for the same time leads unstable silicides to be transformed to more stable ξ-Ti_5Si_3 phase (Fig. 9). The formation of the same ξ-silicides during consolidation of the amorphous as-milled powders of Ti-45Al-2.4Si (at %) has also been reported by Fanta *et al.* (2001). They reported that during consolidation of the as compacted powders, a homogeneous microstructure of eqiaxed TiAl(γ) grains with about

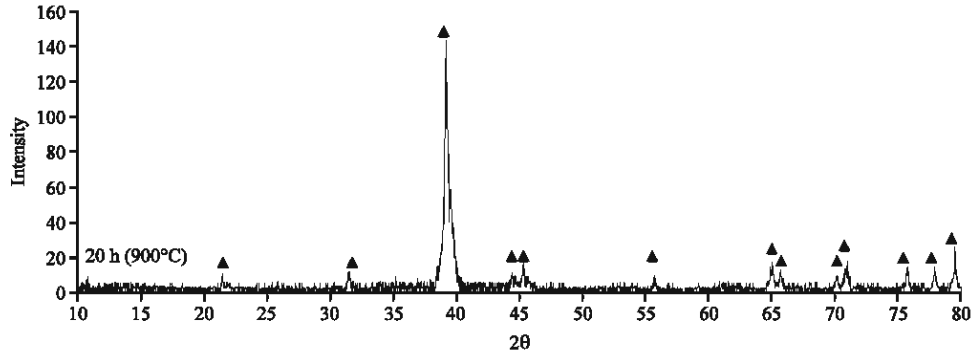

Fig. 6: X-ray diffraction pattern of mechanically milled Ti-50Al powder mixture for 20 h after annealing at 900°C for 10 min

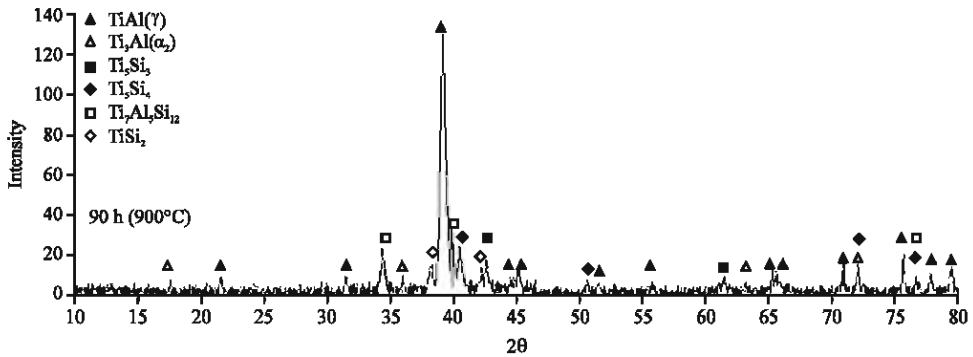

Fig. 7: X-ray diffraction pattern of mechanically milled Ti-48Al-2.4Si powder mixture for 90 h after annealing at 900°C for 10 min

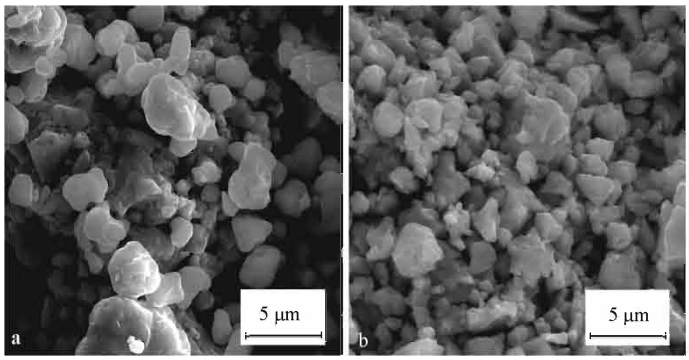

Fig. 8: SEM micrographs of mechanically milled (a) Ti-50Al powder for 20 h and (b) Ti-48Al-2.4Si powder for 90 h, after annealing at 900°C for 10 min

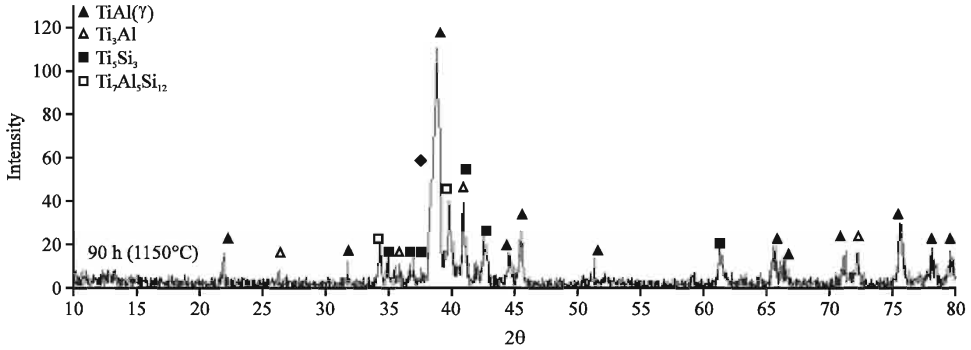

Fig. 9: X-ray diffraction pattern of mechanically milled Ti-48Al-2.4Si powder mixture for 90 h after annealing at 1150°C for 10 min

10 vol% of ξ-phase finely dispersed along grain boundaries of the matrix, was formed.

CONCLUSIONS

Mechanical alloying of powder blends by a high energy ball mill under argon atmosphere leads the Ti solid solution to be formed. This was gradually transformed to an amorphous and nano sized mixture by increasing milling time. The time required for structural changes was longer for Si containing powders.

Adhesion tendency of powder mixtures to milling tools was considerably increased during transformation from elemental powder form into solid solution phase. In general, the adhesion tendency of Si containing powders was considerably lower than those of Si free powders.

Annealing of the mechanically alloyed Ti-50Al powders 900°C for 10 min gave rise to nano scale grain sized TiAl(γ) phase. Nanocrystalline phases of mainly TiAl(γ) together with some silicides and $Ti_3Al(\alpha_2)$ were formed after annealing Si containing powders at the same annealing treatment. Some of the unstable silisides were transformed into more stable ζ-Ti_5Si_3 phase when annealing temperature was increased to 1150°C.

ACKNOWLEDGMENT

The financial support of this study by Sahand University of Technology is gratefully acknowledged.

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