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Characterization and formation mechanism of nanocrystalline W–Al alloy prepared by mechanical alloying

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ABSTRACT

Tungsten and aluminum elemental powders with composition W–20 wt.% Al were mechanical alloyed in high energy planetary ball mill. Structural and morphological changes of powder particles after different milling times were studied by X-ray diffractometer, scanning electron microscopy and microhardness measurements. Mechanical alloying of this system led to the formation of W–Al alloy as a result of formation of W/Al layered microstructure having faceted interface between layers. This alloy indicated high microhardness value of about 570 Hv.

1. Introduction

Tungsten has a variety of industrial applications which stems from its unique properties. Tungsten alloys are used for kinetic energy penetrators, counter weights, radiation shields and electrical contacts [1]. W–Al alloys are expected to show improved mechanical properties compared to W [2].

Non-equilibrium processing of materials has attracted the attention of a number of scientists and engineers due to the possibility of producing better and improved materials compared to conventional methods [3].

There is a growing interest in the production of nanocrystalline and amorphous alloys because of their excellent physical and mechanical properties compared with coarse grained materials [4]. These materials are produced by the use of a variety of techniques including casting, powder metallurgy and self-propagation high temperature synthesis (SHS) and mechanical alloying (MA) [5–7].

An advantage of MA over many other techniques is that it is a solid state technique and consequently problems associated with melting and solidification are bypassed [8].

MA is the technique used to manufacture composites with immiscible components such as W–Cu and W–Ag [9]. The powders of the components can be mixed to obtain the conventional powder mixture or can be produced in high energy ball mill. The formation of alloys by the solid state reaction of powders, were assisted by severe plastic deformation that occurs during ball milling of elemental powders (MA). This process can yield an amorphous, crystalline or nanocrystalline structure [10]. Nanocrystalline materials are potentially attractive for many applications since the reduction of the crystallite size to the nanometer scale improves the physical and mechanical properties [11]. This solid state technique is especially useful for fabrication of those compounds that are difficult to prepare by conventional processes due to high vapor pressure and/or the large differences in melting points of components [7].

There have been little investigations in literature on the binary W–Al system. Oyang [12] investigated W–Al system by MA and suggested that Al dissolves into the W lattice at less than 50%. Tang et al. [2] investigated W–Al system and reported this alloys are lightened products and the hardness and oxidation resistance of the alloys are greatly improved compared to W and Al. Yifang et al. [13] studied Al–W binary system by MA and reported the solubility of A1 in W by MA is more than 50%. Following the theory of extending solubility by MA, the solubility of A1 in W was calculated with the embedded-atom method. The calculation is in good agreement with the experimental results. The extension of solubility by MA results in both thermodynamic and dynamic factors. The dynamic factor is the dominating one. In this paper, structural and morphological changes and formation mechanism of nanocrystalline W–Al alloy by MA, has been studied.

2. Experimental procedure

99.9% pure W powder and 99.8% pure Al powder were mixed to give nominal composition of W–20 wt.% Al. Fig. 1 shows the morphology of as-received W and Al powder particles. As seen the Al particles were irregular in shape with a size

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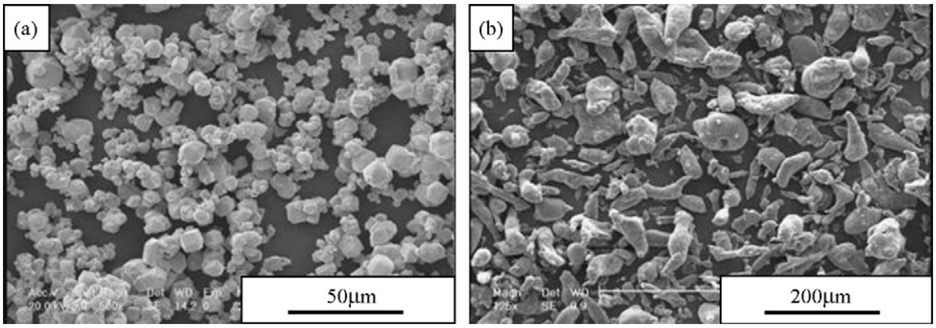

Fig. 1. Secondary electron SEM micrographs of as-received elementals powder particles: (a) W and (b) Al.

distribution of 50–100 μm. The W particles were equiaxed and had irregular shape with size distribution of 1–10 μm. MA was carried out at room temperature using a laboratory planetary ball mill (Fritch pulverisette5 type) under Ar atmosphere. The ball-to-powder weight ratio was 10:1 (the mass of used powder mixture was 18 g) and rotation speed of vial was approximately 500 rpm. A small amount of stearic acid was added to the containers as process control agent (PCA) in order to overcome the problem of sticking of the powder to the balls and the container wall. A small quantity of the milled powder was taken out of the mill at selected time intervals and was characterized by X-ray diffraction (XRD) in a Philips X'PERT MPD diffractometer using filtered Cu Kα radiation (λ = 0.1542 nm). Crystallite size in the milled samples was calculated from the XRD line broadening using the Williamson–Hall equation:

$$\beta \cos\theta = \frac{K\lambda}{D} + 2A\sqrt{(\varepsilon)^2}\,\sin\theta$$

Where θ is the Bragg diffraction angle, D the crystallite size, ε the average internal strain, λ the wave length of the radiation used, β the diffraction peak width at half maximum intensity, K the Scherer constant (0.9) and A is the coefficient which depends on the distribution of strain. Morphology and cross-sectional images of powders were investigated by scanning electron microscopy (SEM) in a Philips XL30 at an acceleration voltage of 30 kV. The average powder particle size was estimated from morphological SEM images by an image tool software. The average size of about 50 particles was calculated and reported as mean powder particle size (due to the shape of the Al particles, Al powder size was calculated longitudinally). The hardness of powder particles was also determined by microhardness test using a Vickers indenter at the load of 100 g and dwell time of 5 s. For measurement of microhardness, a small amount of powder was mounted. The average of five measurements for each sample was calculated and reported as hardness value.

3. Results and discussion

Fig. 2 shows XRD diffraction patterns of W–Al powder mixture at different milling times. As seen the intensity of W and Al diffraction peaks decreased and gradually weakened and broadened during milling. The weakening of W and Al peaks and the shift of W peak toward higher angels (Fig. 2(b)) indicates that Al atoms diffuse into W lattice during milling. Increasing milling time up to 60 h led to the disappearance of Al peaks in XRD patterns. This indicates that the solid–solution with a body centered cubic structure has completely formed after 60 h milling. However, as seen in Fig. 3 [14], the solubility of Al in W lattice at high temperature is very low and therefore Al element can not be dissolved in W lattice at ambient temperature, indicating that MA has extended the solid solubility of this system. Tang et al. [2] studied the W–Al system by MA and reported increased solid solubility of Al in W lattice during MA process. The large volume fraction of atoms in the grain boundaries in this system is expected to enhance diffusion and consequently the solid solubility levels in this system. This is in good agreement with the previous works [2,4].

The broadening of the peaks during milling indicates a decrease in the crystallite size of powder particles and internal strain induced by lattice defects. As seen in Fig. 4 the crystallite size of W(Al) solid–solution decreases with increasing milling time up to 60 h MA. After 60 h of milling time the crystallite size of W(Al) solid–solution was about 15 nm. This effect is due to the forma-

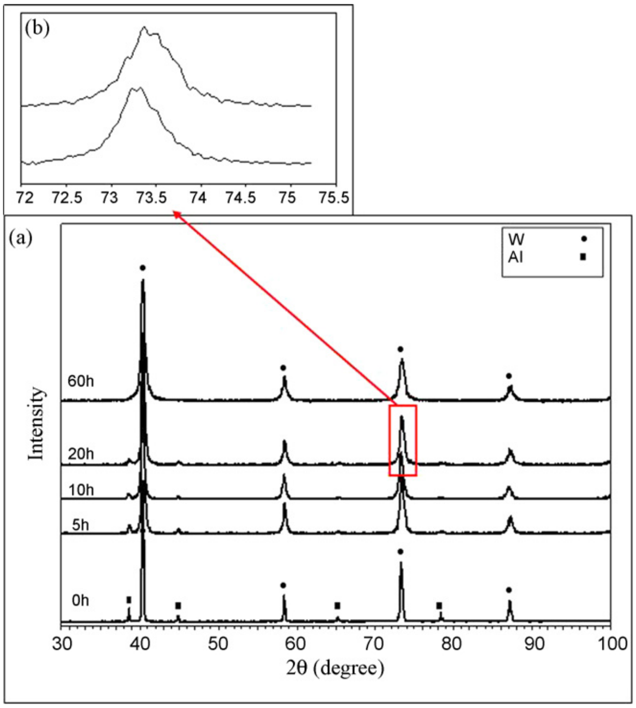

Fig. 2. (a) XRD patterns of W–Al powder mixture after different milling times and (b) displacement of (2 1 1) W diffraction peak.

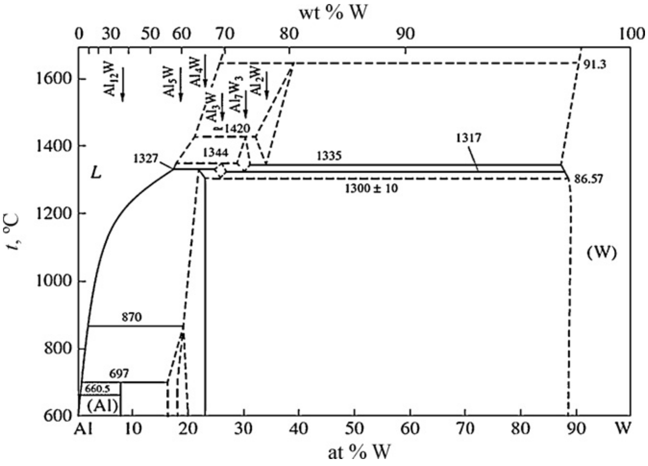

Fig. 3. Phase diagram of W–Al system [14].

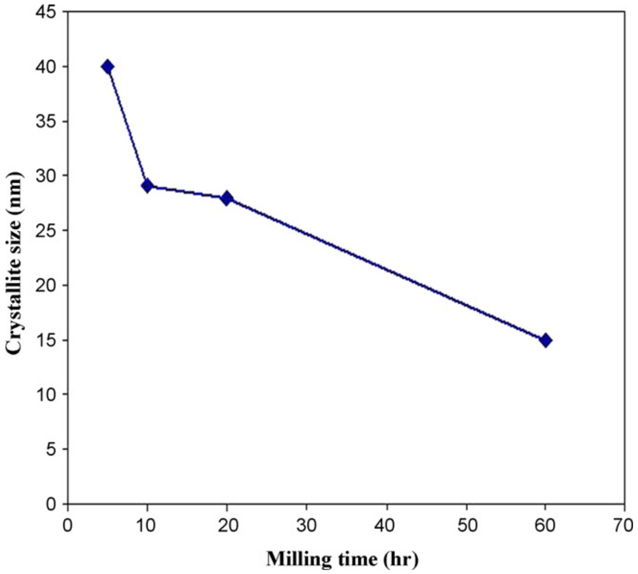

Fig. 4. Crystallite size of W(Al) solid–solution at different milling times.

tion of high density of dislocations during milling and consequently formation of subgrain structure.

It has been reported that [15], the formation of subgrains is attributed to the decrease of the atomic level strain. Further ball milling time leads to excess deformation occurring in the shear bands located in the unstrained parts of the powders which leads to subgrain size reduction. Thus, the orientation of final grains becomes random in crystallographic orientations of the numerous grains and hence, the direction of slip varies from one grain to another.

Morphological evolution of powder particles after 2, 5, 20 and 60 h of milling times are shown in Fig. 5. After 2 h milling the size distribution of powder particles is nearly uniform and the powder particles exhibit flake morphology with an average size of about 130 ± 20 μm.

After 5 h milling the powder particles were irregular in shape and the size distribution of powder particles in this stage was not uniform with mean particle size of about 140 ± 70 μm. This indicates that from 2 until 5 h milling, there is no significant change in mean particle size. This is probably due to the same rate of fracturing and cold welding of powder particles in this stage, while there is a significant change in powder morphology. Flake morphology then transformed to nearly equiaxed morphology. Increasing milling time up to 20 h led to significant decrease in mean particle size due to increased volume fraction of W(Al) solid–solution and work hardening of powder particles during milling. In this stage the mean size of powder particles was about 50 ± 20 μm. As seen in Fig. 5(e), the powder size is reduced to about 10 ± 5 μm after 60 h MA. This is due to the predominance of fracturing over the cold welding processing in this stage. Fig. 5(f) revealed that the large particles in this stage are in fact an agglomeration of many smaller particles.

As seen in Fig. 6 a layered composite microstructure was formed at early stages of milling. Formation of this layered microstructure between metallic elements has been previously reported [16]. Increasing milling time up to 20 h led to refinement of layered microstructure and therefore formation of very fine microstructure comprise alternative W and Al layers due to the repeated cold welding and fracturing of powder particles. Formation of this microstructure with very faceted interface between layers and also decrease in thickness of layers with increasing milling time promote the interaction between layers and as seen in Fig. 6(e) led to the formation of homogenous microstructure at longer milling times.

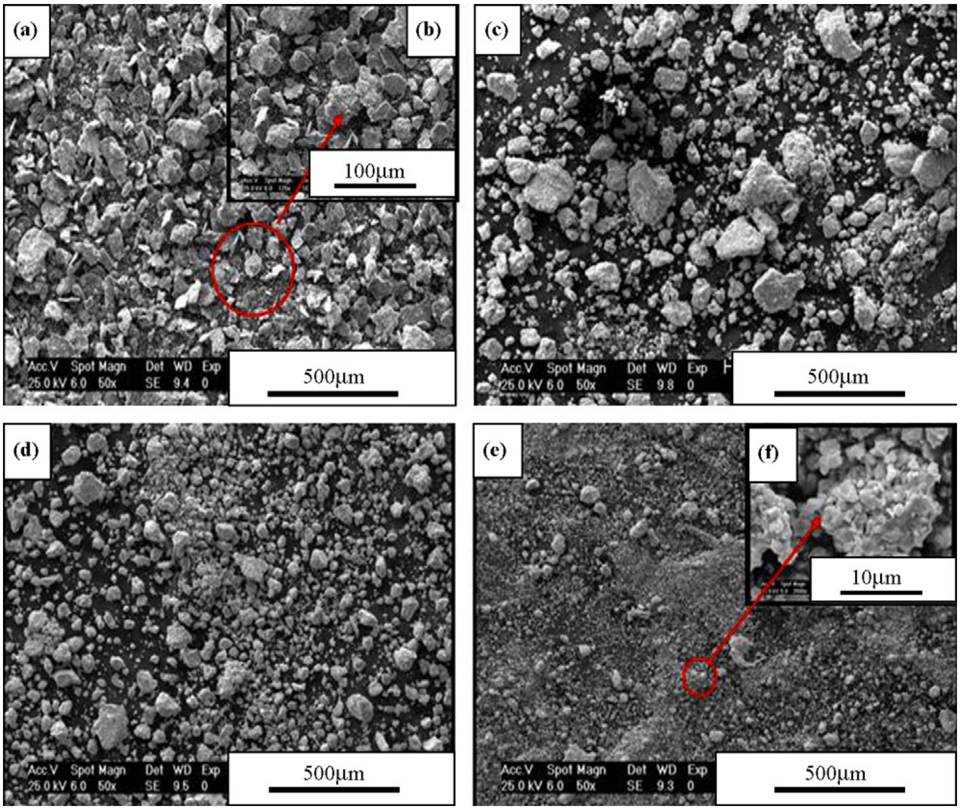

Fig. 5. Secondary electron SEM micrographs of powder particles after (a and b) 2 h, (c) 5 h, (d) 20 h and (e and f) 60 h of milling times.

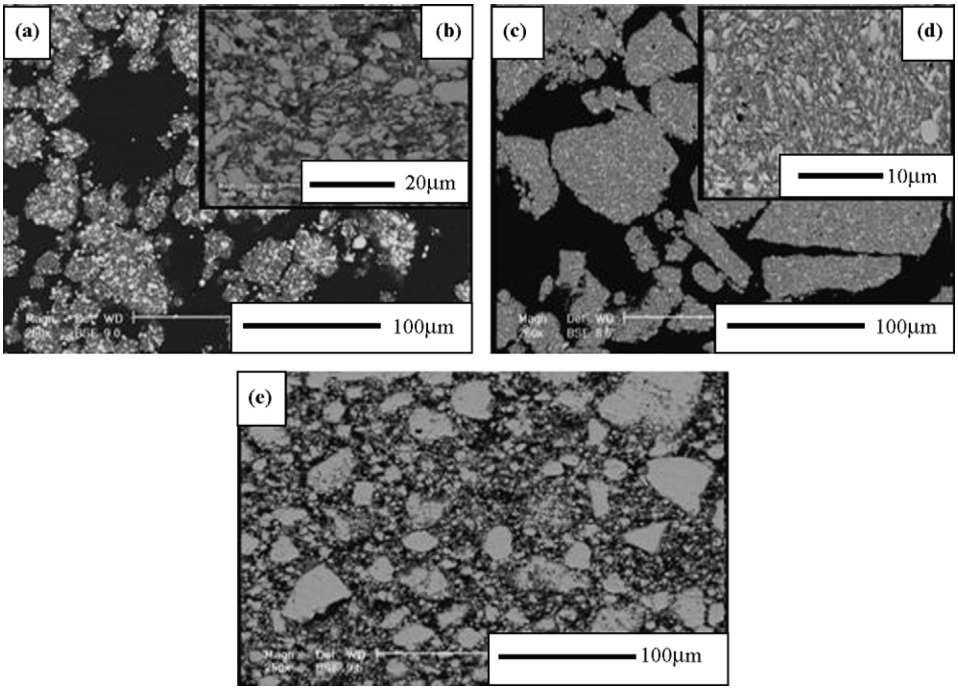

Fig. 6. Cross-sectional back scattered SEM images of powder particles after (a and b) 5 h, (c and d) 20 h and (e) 60 h of milling times.

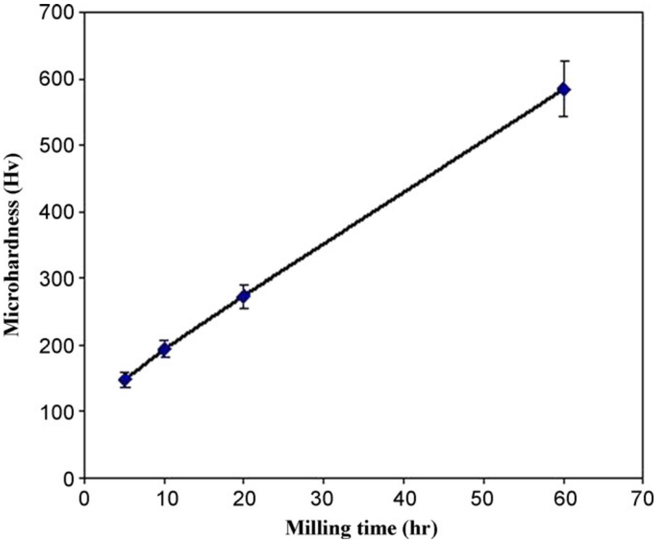

Fig. 7. Microhardness value of W–Al powder mixture at different milling times.

Fig. 7 shows the microhardness values of powder particles at different milling times. As seen the microhardness value increases with increasing milling time. This is due to increased volume fraction of W–Al alloy and refinement of crystallite size by increasing milling time. The W–Al alloy exhibited a high value of microhardness of about 570 Hv after 60 h MA.

4. Conclusion

The nanocrystalline W–Al alloy was successfully synthesized by mechanical alloying of W–20 wt.% Al powder mixture in a high energy ball mill. During milling W–Al solid–solution was formed. It was found that the formation of very fine layered microstructure between elements with very faceted interface area promote the diffusion rate of Al in W lattice in ambient temperature. This phase exhibited a significant microhardness of about 570 Hv.

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