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EFFECT OF DIFFERENT MECHANICAL MILLING PROCESSES ON MORPHOLOGY AND MICROSTRUCTURAL CHANGES OF NANO AND MICRON Al-POWDERS

WPŁYW RÓŻNYCH PROCESÓW MIELENIA NA MORFOLOGIĘ I MIKROSTRUKTURĘ NANOMETRYCZNYCH I MIKRONOWYCH PROSZKÓW Al

In this research, effect of the various mechanical milling process on morphology and microstructural changes of nano and micron Al-powders was studied. The milling of Al-powders was performed by both high energy and low energy ball milling process. The influence of milling (pulverizing) energy on the structural changes of Al-powders was studied. Al-nanoparticles were agglomerated during the MA and its size was increased with increasing milling while micron Al-powder gets flattened shape during high energy ball milling due to severe plastic deformation. Meanwhile, structural evolution during high energy ball milling of the nano powder occurred faster than that of the micron powder. A slight shift in the position of X-ray diffraction peaks was observed in nano Al-powders but it was un-altered in macro Al-powders. The variation in lattice parameters was observed only for nano Al powders during the high energy ball milling due to lattice distortion.

Keywords: Al-alloys, High-energy ball milling, Low energy ball milling, Plastic deformation

1. Introduction

Recent advances in global technologies allowed the production of aluminum and related alloy powders, which are attractive structured materials for huge applications such as for most aerospace and defense applications [1]. These materials possess the unique properties such as high specific strength and stiffness, good wear resistance and excellent electrical conductivity, low weight and high thermal conductivity, which are encourages that the researchers are foot forwarded to enhance such properties [2, 3].

It is well known that, nanopowders which are the bridge between bulk materials and atomic structures and have the favorable attractive attention owing to their large surface area compare to its volume ratio. The large surface area can significantly improve the properties of powder such as the mechanical, chemical and physical properties [4]. So, the synthesis of nano and micron Al powders is of challenging and a number of powder metallurgy (PM) methods such as pulsed wire evaporation and high energy ball milling were employed [5-8]. Among the all of nanopowder synthesizing powder metallurgy methods, mechanical alloying is an attractive solid state nano powder fabrication method, which explores the simple operations for mass production and low economical processing method [9-12]. Mechanical milling is a process that involves repeated collision between the balls to wall and gives the deformation, welding and fracture. Many parameters such as type of miller, grinding medium and speed, milling atmosphere, ball to powder weight ratio, and process control agent can influence the stages of milling [10, 13]. So far, many works were carried out for the milling of micron and Aluminum composite powders, however the effect of powder size (nano and micron) and the milling energy (pulverization energy) on the morphology and microstructure of Al-powders are very rare and interesting. Recently, our previous work, carried out by Al-nano particles were synthesized using low energy ball milling process, and the effects of various processing parameters on the resultant porosity and microstructure were investigated [14]. However, studies related to influence of powder size and the milling energy on the morphology and microstructural changes of the aluminum powders have not been well studied.

The present paper well addressed the morphology and structural changes during the mechanical alloys as well as milling energy on the structural changes of Al-powders were studied.

2. Experimental

Initially, we have taken two types of Al powder with mean particle size of 100 nm (nano powder) and 200 μm (micron powders) and proceeded. The Al-nano particles are in spher-

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ical shape whereas the Al-micron poweders were in irregular shape. As received Al- nano powder was loaded into a stainless steel vial and sealed under argon atmosphere. Stainless steel balls of 5.54 mm diameter corresponding to a ball to powder weight ratio (BPR) of 15:1 with 400RPM were used for milling. The high energy ball milling was carried out for both Al-nano and micron powder for different milling times with appropriate conditions. The milled powder was collected at the end of milling and weighed in order to determine the yield of the process. More than 90% yield was achieved in all the milling processes.

X-ray diffraction (XRD) patterns were obtained using an X-ray diffractometer (MiniFlex-600, Rigaku, Japan) with Cu Kα radiation. The analysis of the XRD peaks was performed via Bragg's law is given by eq (1),

$$n\lambda = 2dsin\theta \tag{1}$$

Where d is space between the planes in atomic lattice, and λ is wavelength of incident beam [15]. The lattice parameter can be measured by following eq (2),

$$a = \left(\frac{h^2 + k^2 + l^2}{d^2}\right)^{1/2}. \tag{2}$$

Where (*hkl*) are miller indices of the Bragg plane in a crystal. For Aluminum case, it is a face centered cubic system and (*hkl*) = (111). The morphology and microstructure of both low and high energy milled Al powders was analyzed by scanning electron microscopy (SEM-MIRA LMH II (TESKAN), Czech Republic).

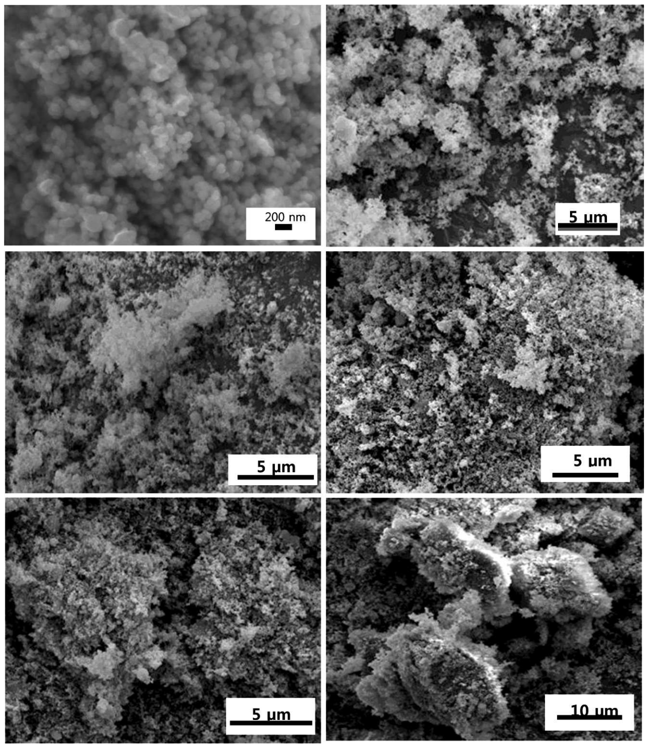

Fig. 1. Morphology of the nano Al-powders by low energy ball milling with the milling time. SEM micrographs of (a) 0 min, (b) 10 min, (c) 30min, (d) 1h, (e) 5 h and (f) 30 h. (inset: high magnified SEM images)

3. Results and discussion

Figure 1 shows SEM micrographs of Al-nanopowders milled by low energy ball milling with various milling times. Figure 1(a) represents Al- nano powder in as-received (un-milled) condition. As it can be seen from the Fig. 1(b) and (c), at low milling times (10 min and 30 min respectively), nanopowders exhibits partially agglomerated state and look likes sponge shape, while increasing milling time, Al particles were agglomerated and its size was increases. After increases milling time from 1 to 5h, initial particles were deformed and then formed into full agglomerated state. At early stage of milling, the rate of mechanical bonding caused by plastic deformation of powders becomes higher than the fragmentation owing to higher rate of cold welding. As milling time advances, the powders get work hardened, and easily initiate the crack formation. For more increasing milling time (up to 30 h), the fracture of cold welded powders takes place heavily and then cause to minimize the powder agglomeration size. In order to study the influence of milling energy on Al-nanopowders during mechanical alloying, high energy ball milling was carried out with same conditions as previous.

Figure 2 illustrates the morphology of Al-nano powders by high energy ball milling. As we know that, during the high energy ball milling process, powder particles were subjected to high energetic impact. So, the microstructure of Al-nanopowders by high energy ball milling was totally different as compared with low energy ball milling. Fig. 2(a) represent as received initial Al- nano powder. At the initial stage of high energy ball milling, micro-forging and cold welding led in individual particles or clusters of particles being impacted repeatedly by milling balls with huge kinetic energy, and powder gets agglomerated irregular shape. As increased the milling time from 30 min to 3h, significant changes occur in comparison with those powder in initial stage. Here, cold welding takes place significantly. In this stage, fracturing and cold welding are predominant milling processes. Al-powder get considerable refinement and reduction in particle size at the final stage of milling (3h) during the mechanical alloying process. However, the agglomerated particles were irregular shape and get more plastic deformation shown in high magnified figure Fig. 2(f). As it can be observed that the morphology of Al-nanopowders by high energy ball milling was fairly different compare to that of Al-nanopowders by low energy ball milling (Fig. 1). High impact energy was applied to aluminum nano powders during high energy ball milling, so that powder gets severe plastic deformation than Al nanopowders under high energy ball milling. For comparison of morphology of Al-nano powders, micron Al- powder (~200 μm) was performed by high energy ball milling. As shown in Figure 3, after 10 min of milling, the micron Al-particles were deformed and observed as irregular shape was noticed. Whenever we have proceed for long milling times, the aluminum particles were flattened and flake-like particles were formed (Fig. 3b) [16, 17]. Moreover, micro welding between the particles was also observed. It can be noticed that the morphology was affected by different milling processes such as high energy and low energy ball milling and detailed explanation was given in last further session (Fig. 6 sessino).

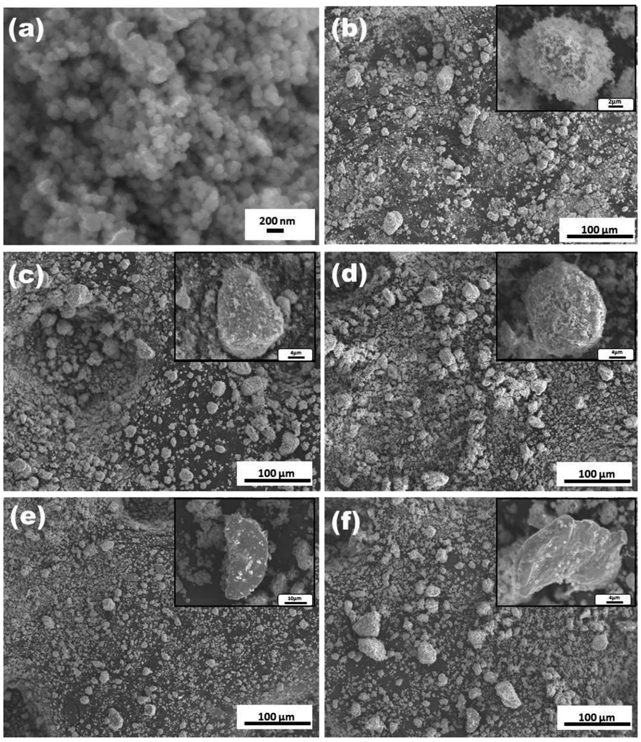

Fig. 2. Morphology of the nano Al-powders by high energy ball milling with the milling time. SEM micrographs of (a) 0 min, (b) 10 min, (c) 30min, (d) 1 h, (e) 2 h and (f) 3 h. (inset: high magnified SEM images)

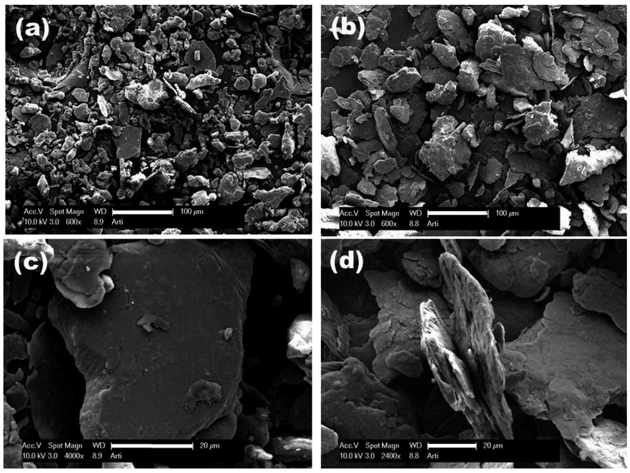

Fig. 3. SEM micrographs of micron Al-powders by high energy ball milling. (a) 10 min and (b) 3 h. (c) and (d) shows High magnification SEM images of 10 min and 3 h respectively

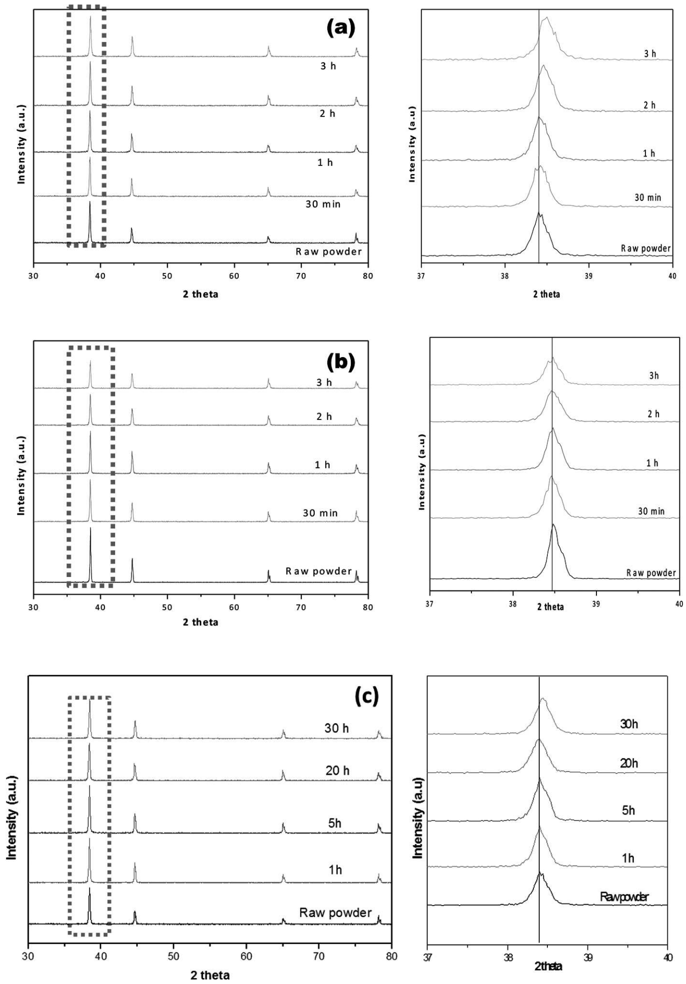

Fig. 4. XRD analysis of (a) nano Al-powders (b) micron Al-powders by high energy ball milling, and (C) nano Al-powders by low energy ball milling

Mechanical milling of nano and micron aluminum powder was accompanied by sub and micro-structural changes. Severe plastic deformation of the nano and micron particles can lead to grain refining, accumulation of internal stress and change of the lattice parameter. The X-ray diffraction pattern of initial and milled Al-nanopowders by high and low energy ball milling were shown in Fig. 4. It reveals from the figure, with increasing milling time from initial state to longer milling times, the XRD pattern was broadening and resembles decreases in powder size. It was perceptibly indicated from the nano Al-powder by high energy ball milling in Fig. 4(a), a slightly shift in the position of the XRD peaks. However, there was no change in the position of the XRD peaks for micron Al-powders by high energy ball milling. The XRD pattern of Al-nano powder by low energy ball milling was represented in Fig.4(c). The shifts in the position of the XRD peaks was not effected in the case of Al-nanopowder by low energy ball milling compared to that of high energy ball milling. However, only 30 h milling powder shows very little shift, and it may be neglegible. In addition, XRD pattern of Al-micron powder peaks were un-altered compare to nano Al- powder by high energy ball milling. The calculated lattice parameter values for nano and micron Al-powders by high energy ball milling was illustrated in Fig. 5. It was evidently observed from the Fig. 5, the values of lattice parameter of Al-nano powder was altered when compared to the Al- micron powders. The displacement of Aluminum nanopowder XRD peaks (111) towards higher angles and decreasing Al lattice parameter was observed with increasing milling time. Accordingly, the Al-lattice parameters were decreased from a = 2.147 Ao to a = 2.142 A° in mechanically alloyed powders after high energy ball milling for3 h. It might be expected that, during high energy ball milling of Al-nano powders, the high impact energy was applied on the nano particles and it attributed to a slight distortion in crystal lattice compared to micron power. In addition, the strain during the high energy ball milling was also the result of lattice distortion and structural disorder. Besides, the decrement of lattice parameter was might be due to the possible lattice shrinkage [18]. It is might be due to the high surface tension of the nano aluminum particles since nano particles have high surface to volume ratio. The strong internal strains in the lattice initiated by high energy ball milling may also influence the lattice shrink. In addition, contaminations (due to milling balls and jars) may also take place during the mechanical milling. It could be said that the lattice distortion can be as a result of entrance of contamination atoms to the lattice of nano powders under severe milling conditions.

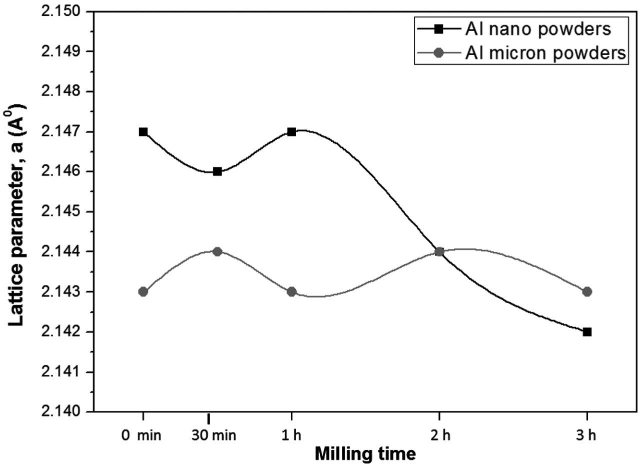

Fig. 5. Calculation of lattice parameter of Al-nano powders and Al-micron powders by high energy ball milling

The schematic Fig. 6 shows how the different mechanical milling process will have an effect on the surface morphology and agglomeration of Al nano powders. At initial stage, aluminum nano powders were in the order of 100 nm size and spherical in shape. On the milling for 1h under low energy ball milling, the initial nanopowders become moderately bonded by deformation and cold welding, resulting neck formation within bonded region like a sponge shape. And, due to acquiring high surface energy of nano particles; it causes to form them into agglomerated powders [19]. Resulting, the powder showed coarsening due to aggregation of soft Al particles. On the other hand, during high energy ball milling, the particles get trapped between the grinding medium and undergo plastic deformation. For long milling times under low energy ball milling, nano particles become more adhesion to each other and fully bonded due to further growth of necking and continuously arise of cold welding. In addition, few small particles (satellite particles) were also observed on the surface of agglomerated powders. In case of high energy ball milling, particles acquire more impact energy and get the severe plastic deformation than the low energy ball milling. In general, the impact energy or pulverization force of the balls E_w (J/kg.s) can be calculated from the balls motion, as follows

$$E_w = \sum_{j=0}^{n} \frac{1}{2W} m v_j^2. \qquad (3)$$

Where m is the mass (kg) of ball, n and mv_j^2 denotes the number of collision of a ball or the mill wall within a second an square of the relative speed of the balls, respectively. W denotes the weight (kg) of samples charged in mill [20]. Due to the continuing high impact energy on nano aluminum powders, the particles get severe plastic deformation and the structure of the powder was totally changed. It was evidently observed from the XRD pattern of nano powders by high energy ball milling, the lattice parameters were slightly shifts due to the effect of high impact energy on the Aluminum nano powders. However, in case of Aluminum micron powders, the particles were turned deformation and get flatten, but not similar than that of nano powders during the high energy ball milling. So, the lattice parameters were just fluctuate but not stronger than the aluminum nano powders during the high energy mechanical alloying.

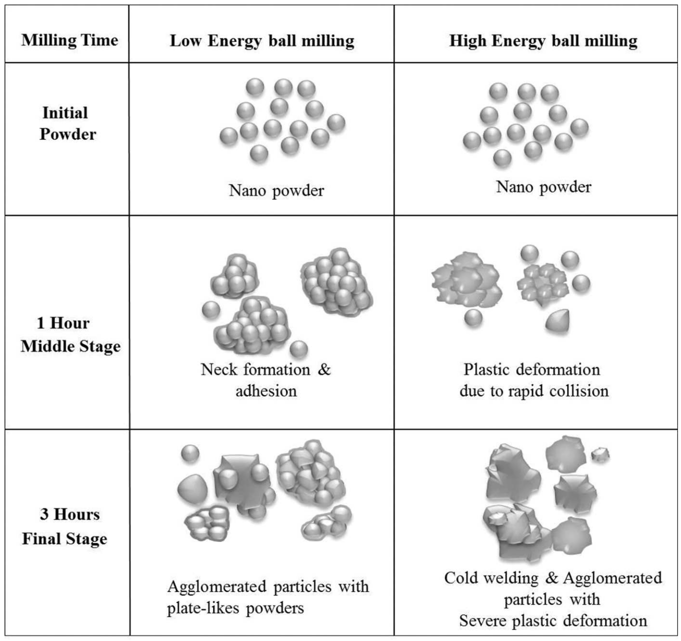

Fig. 6. Schematic diagram showing effect of the surface morphology and agglomeration of Al nano powders by different mechanical milling process

4. Conclusions

In this conclusion, we studied the effect of different mechanical milling processes on morphology and microstructural changes of nano and micron Al-powders. The influence of milling energy on the structural changes of Al powders was studied. Al-micron powder gets flattened shape and was observed via high energy ball milling due to severe plastic deformation and repeated cold welding. Slight shifts in the position of XRD peaks was observed in Al-nanopowders during high energy ball milling and it was almost un-altered for low energy ball milling of Al- nano powders. The variation in lattice parameters was observed only for nano Al powders during high energy ball milling due to lattice distortion while not observed for other cases (low energy ball milling of Al nano powder and high energy ball milling of micro powders).

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