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Structural Evolutions of Al-Ni-Nd Alloy Prepared By Mechanical Alloying

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Abstract
Al-based nanocrystalline materials with average crystalline size of a few nanometers have attracted considerable attention due to their attractive potential in structural application. The proposed study presents nanocrystalline $Al_{88}Ni_8Nd_4$ alloy powders is produced by high-energy planetary ball milling, starting from elemental powders. The milling was carried out at speed of 300 rpm with intervals of 20 min. milling and 20 min. rest to prevent the mixture from overheating. In order to study the structural evolutions and thermal behaviour of the mechanically alloyed powders, X-ray diffraction (XRD), differential thermal analyser (DTA) and scanning electron microscopy (SEM) were used. Results show that Al_3Ni intermetallic compound is obtained after 10 hours of milling. Increasing the milling time up to 100 h leads to the formation of Al_3Ni with 15 nm crystallite size. In the present investigation, structural evolution and thermal behaviour of $Al_{88}Ni_8Nd_4$ alloy prepared by mechanical alloying have been discussed in detail.

Keywords: Al-Ni-Nd Powder Alloys, Mechanical Alloying, Phase Evolutions, Nanogranular Particles.

1. Introduction

Nanocrystalline materials, namely the poly-crystals with nanometer-sized crystallites (less than 100 nm), have attracted much scientific and technological interest recently. Both the structure of nanocrystalline materials, which is characterized by a large fraction of atoms located at the grain boundaries (generally called interfaces), and their properties, which are found to be appreciably different than those of the usual crystalline materials as well as of the amorphous solids, have given rise to much investigation [1-2]. Nanocrystalline material constituted of ultrafine grains, with a considerable volume fraction of interfaces, provides an opportunity to study the structure-property relationship and the nature of interfaces in solids [3]. Nano-materials are usually produced by rapid solidification of the alloying constituents from the liquid phase [4-6].

In recent years, an alternative way of preparing nano-materials is via solid state reaction, i.e. mechanical alloying (MA) of elemental powders [7].MA is a dry, high-energy ball milling technique and has been employed to produce a variety of commercially useful and scientifically interesting materials such as supersaturated solid solutions, crystalline and quasicrystalline intermediate phase, amorphous alloy and nanocrystalline materials [8].MA process leads to a refinement of the grains accompanied by the evolution of the system through different metastable phases, because of elemental powders interaction between ball-ball and ball-wall [9]. It has been shown that a nano-sized grain structure can be obtained in most materials after sufficient milling time [10]. On the other hand, this technique can easily be combined with suitable powder compaction, promise to allow preparation of bulk specimens with much larger dimensions than those available through rapid solidification.

Al-based especially amorphous alloys have been synthesized mainly in Al-TM-RE (TM: transition metal, RE: rare earth metal) [11] because they exhibit unusual mechanical properties with potentially good corrosion resistance and thermal stability. Al-Ni-Nd ternary amorphous alloys have been produced by melt-spinning [12]. However Al-Ni-Nd ternary powder alloys, which were produced by MA, have been little detailed work on their structural evolution and thermal behaviour with different stages of increasing milling time. The aim of this study is to investigate phase evolution and thermal behaviour of $Al_{88}Ni_8Nd_4$powder alloys by XRD, DTA and SEM.

2. Experimental

Ternary alloys with composition $Al_{88}Ni_8Nd_4$ were prepared by a high energy ball milling Fritsch Pulverisette 5 planetary ball mills using a 250 ml volume cylindrical stainless steel container together with 9-12 mm diameter stainless steel balls. The 88%Al-8%Ni-4%Nd alloy powders were produced by milling of aluminium powder (99.97 % purity**,** maximum particle size250 µm), Ni (99.5 % purity**,** maximum particle size 10µm) and Nd (99 % purity**,** maximum particle size 100 µm). Stearic acid (1 % mass) was used to reduce the adhering of ductile metals to the milling medium such as vial walls and the milling balls. Structural evolution of the mechanically alloyed powders at different stages of milling was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRD experiments were performed using a Philips X` Pert Pro diffractometer with filtered CuKα (λ = 0.154 nm) with 35 kV and 50 mA. The changes of the average grain sizes during mechanical alloying were estimated from X-ray diffraction line broadening. Debye-Scherrer's equation is expressed as the following [13]:

$$d = 0.9\lambda / B\cos\theta \qquad (1^{st}) \quad \text{and}$$

$$B^2 = B_i^2 - B_o^2 \qquad (2^{nd})$$

where d is the grain size, λ the wavelength of X-ray, θ peak Bragg angle and B is the full width at half-maximum (FWHM). B_i is the FWHM of the mechanically alloyed powders peak and B_o is the instrumental broadening determined from an X-ray pattern of standard silicon sample used for instrumental calibration [14]. The lattice parameter a_o of mechanical alloyed powders was determined from the peak positions of the X-ray diffraction line broadening using Bragg's Law [13]:

$$2d\sin\theta_{hkl} = n\lambda \quad (n=1) \qquad (3^{rd})$$

$$d = \lambda / 2\sin\theta_{hkl} \qquad (4^{th})$$

Here, d is the interplanarspacing,θ_{hkl} is the diffraction angle of the (hkl) diffraction plane and λ is the X-ray wavelength. The lattice parameter a_o of cubic structure calculated using the equation

$$d^2 = a_o^2 / h^2+k^2+l^2 \qquad (5^{th})$$

from (4^{th}) equation becomes

$$\lambda^2 / 4\sin^2\theta = a_o^2 / h^2+k^2+l^2 \qquad (6^{th})$$

SEM analysis was performed with a JEOL JSM 5400 scanning electron microscope at an acceleration voltage of 20 kV after the specimen was coated with a vacuum-deposited gold layer in order to enhance contrast. The crystallization behaviour of the mechanically alloyed powders at different stages of milling were analyzed by differential thermal analyzer (DTA) using a Perkin-Elmer's Diamond TG/DTA at a constant heating rate of 20 ^oC/min from 100 to 1200 ^oC under flowing N_2.

3. Results and Discussion

The X-ray diffraction patterns of the mechanically alloyed Al88Ni8Nd4 powder as-received and after different milling times are shown in Fig. 1. In the un-milled sample, the Al peaks are more dominant than Ni, Nd peaks because of quantity of Al. The Al (111), (200), (220), (311), (222) peaks, Ni (111), (200),(220) peaks and Nd (203), (210) peaks are clearly observed. The Ni and Nd peaks are partial overlapping of its peaks with Al peaks. After 10 h of milling, the intensity of elemental peaks rapidly decreased and dissolved. However Al3Ni intermetallic phase observed. The Al(111), (200) and Ni(111) peaks are observed milling time up to 100 h but they are broadening and decreasing. After 100 h of milling, the Al(111), (200) and Ni(111) peaks disappear. Orthorhombic Al3Ni phases are clearly observed and stable phases for this composition. On the other hand, Al and Ni phases are replaced by Al3Ni intermetallic phases. This result show that solid state reaction occurred and solid solution completely in Al3Ni phases.

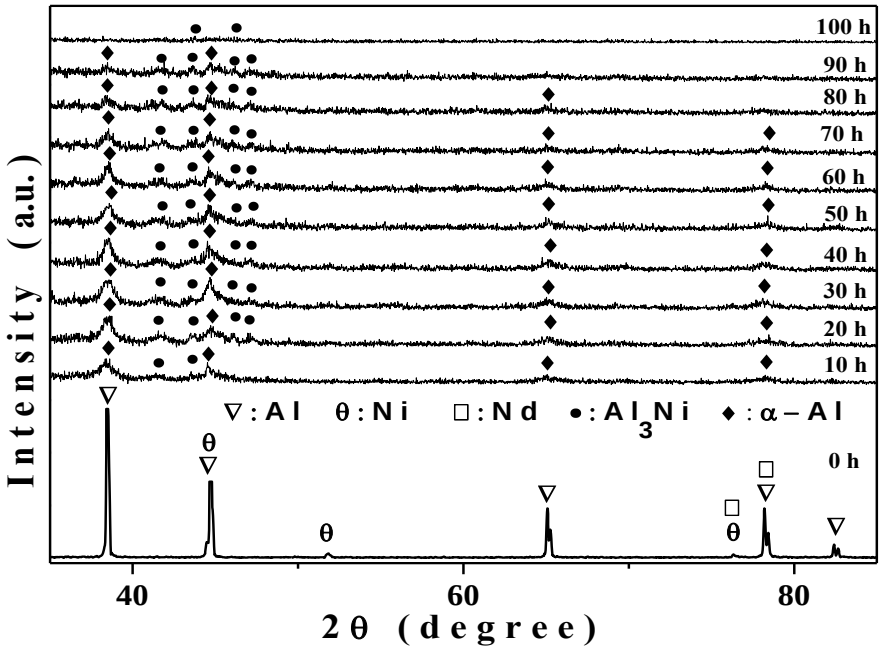

Fig. 1. XRD patterns from $Al_{88}Ni_8Nd_4$ powders after different milling time intervals.

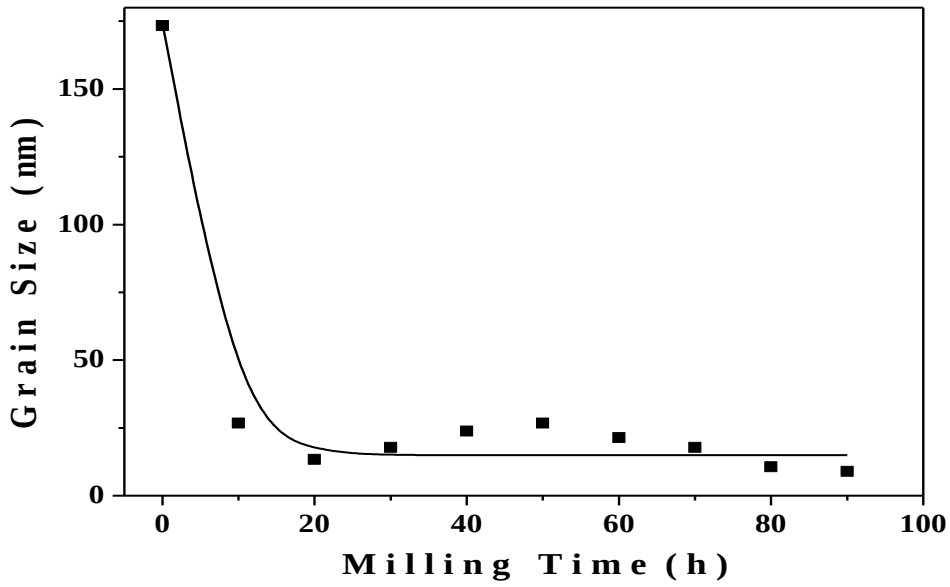

Fig. 2 Variation of grain size of the powders after different milling time intervals**.**

In Fig. 2, grain sizes of the fcc-Al (111) peak are calculated using the Debye-Scherer's equation by X-ray diffraction [13]. Debye-Scherer's equation was applied for the Al (111) peak since there is no overlap with another diffraction peak. Fig. 2 shows the variation in grain size of the Al particles after different milling time intervals. From Fig. 2, it is seen that un-milled Al powders are found to be 170 nm. After 10 h of Al powders, the grain size decreased rapidly approaching a constant value at longer milling times. Similar grain refinement behaviours were previously reported [4-5,15]. However, after 90 h of milling the average grain size of Al powders were about 15 nm. Therefore, mechanical milling has the inherent advantage of grain refinement.

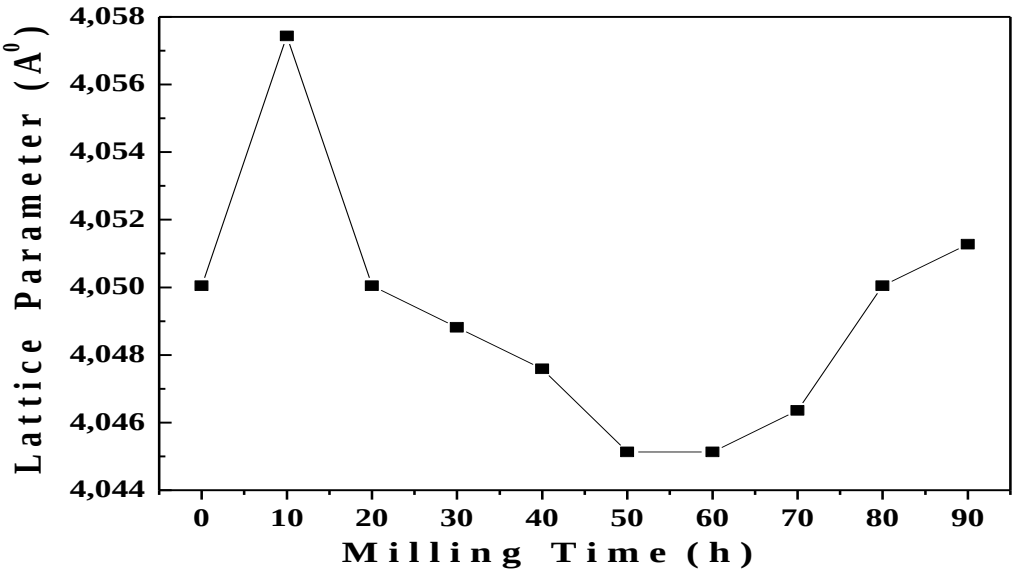

Fig. 3 Variation of lattice parameter (a_0) of the powders after different milling time intervals.

In Fig. 3, the lattice parameter (a_0) are calculated using the Bragg equation [13], is plotted as a function of milling time. Diffusion of atoms and atomic radius are very important to form lattice parameter (a_o). Aluminum (Al), Nickel (Ni) and Neodymium (Nd) atomic radius are 1,43 A^0, 1,24 A^0 and 1,82 A^0 respectively. From Fig. 3, it is seen that lattice parameter increased after 10 h of milling because diffusing of Nd atoms in Al lattice leads to increase in its lattice constant. This result is consisted with X-ray patterns after 10 h of milling while Nd peaks are dissolved completely. From 20 h to 60 h of milling, lattice parameter decreased because of diffusing of Ni atoms in Al lattice. Al_3Ni intermetallic compounds of the $Al_{88}Ni_{10}Nd_2$ powders observed. This result is again consisted with XRD observation. From 60 h to 90 h of milling, the Al lattice parameter increased because dominant Al and Ni peaks are broadening and dissolving. Mechanical alloying can promote the formation of Al_3Ni intermetallic phases, based on XRD results.

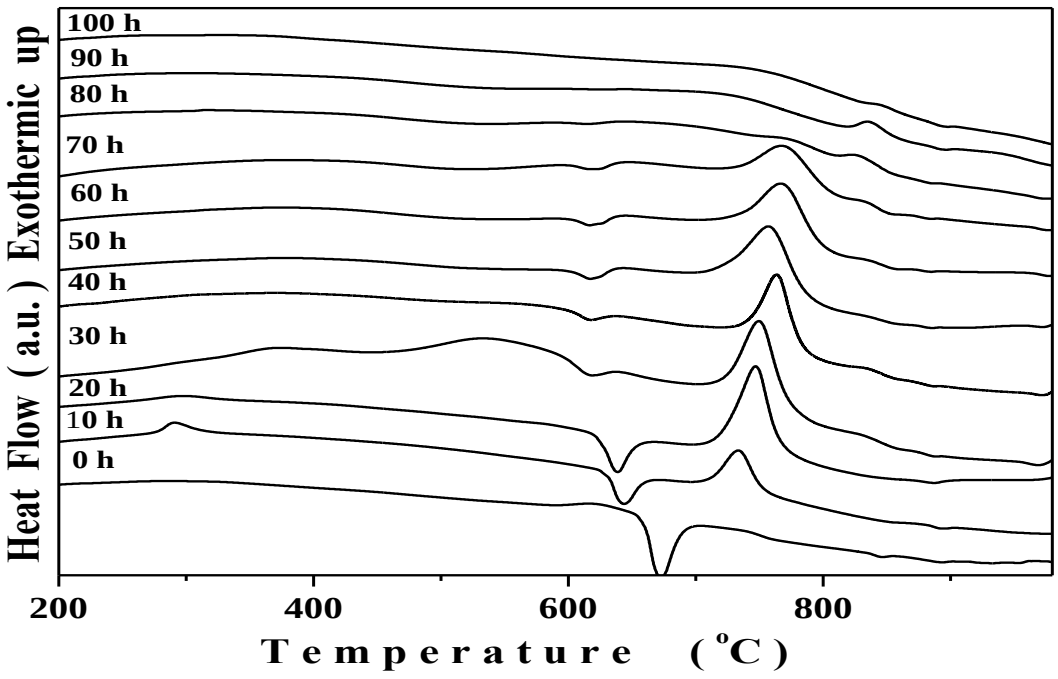

Fig 4. DTA curves of the $Al_{88}Ni_8Nd_4$ powders after different milling time intervals

Fig. 4 shows that DTA scans of the $Al_{88}Ni_8Nd_4$ powders prepared at different milling times. DTA scans of un-milled $Al_{88}Ni_8Nd_4$ powders show that only endothermic peak at 640 °C observed. This endothermic peak temperature indicates that the melting temperature of Al phase [16]. On the other hand, DTA curves showed two exothermic peaks around 280-320 ºC, 730-780 ºC respectively after 10 h. After 20 h, the intensity of first exothermic peak around 280-320 ºC is decreased and brodened. However the intensity ofsecond exothermic peak around 730-780 ºC increased. This is indication the reaction in between Al, Nd and Al, Ni particles. After 30 h of milling, the first exothermal peak around 280-320 ºC disappeared completely and second exothermic peaks around 730-780 ºCbrodened. Also, a little endothermic peakaround 850-890 ºC is observed and so on. The second exothermic peak around 730-780 ºC disappeared completely after 90 h of milling. After 100 h of milling, only a little endothermic peak around 850-890 ºC observed. This peak indicates the melting temperature of Al_3Ni intermetallic compound. This result is consistent with the XRD results and previously reported [16-18].

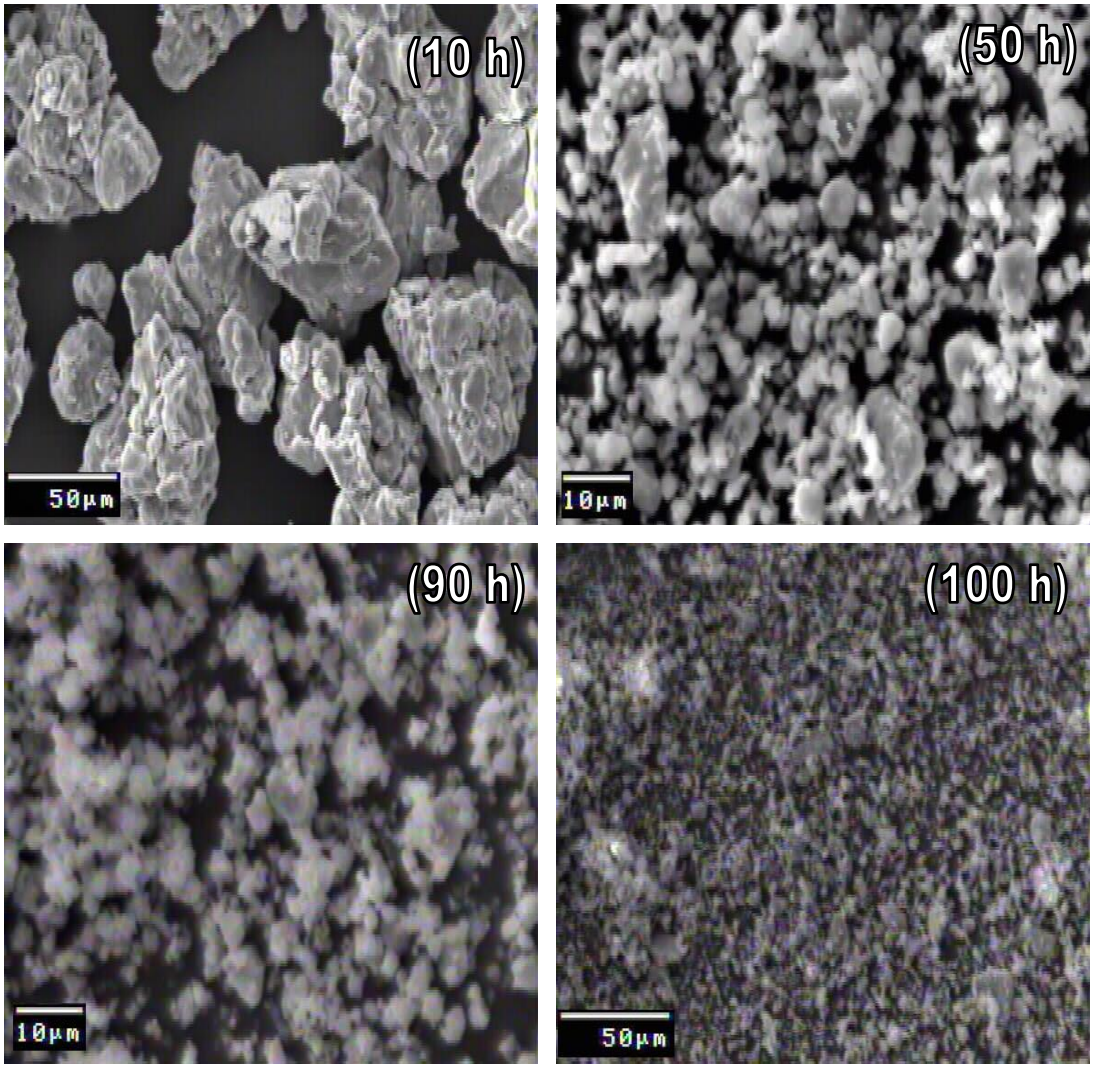

Fig. 5. SEM images of the $Al_{88}Ni_8Nd_4$ powders milled for10 h, 50 h, 90 h and 100 h.

Fig. 5 shows SEM images of $Al_{88}Ni_8Nd_4$ powder alloys after 10, 50, 90 and 100 h of milling. After 10 h of MA, composition of $Al_{88}Ni_8Nd_2$ powders were cold welding by MA process. After 50 h of milling, average grain size decreases and solid state reaction are not completed.This result is based on XRD results (After 50 h of milling, X-ray patterns show that elemental phases and Al_3Ni intermetallic phases). From 90 to 100 h of milling, solid-state reactions are completed and the average grain size decreased with milling time. This results are cohered XRD observation (XRD patterns show only Al_3Ni phases) and previously reported [17].

4. Conclusions

In this study, in order to investigate the microstructural evolutions and thermal behaviour during mechanical alloying of $Al_{88}Ni_8Nd_4$powdersthe samples examined by XRD, DSC and SEM. The main results obtained are summarized as follows:

1. Mechanical alloying of $Al_{88}Ni_8Nd_4$ powders resulted in the formation of nanoparticles with a size of about 15 nm.
2. After 90 h of milling, solid state reaction completed and only Al_3Ni intermetallic phases observed.
3. For milling time shorter than 30 h, the DTA scans showed a broad exothermsinbetween 275 and 325 ^{0}C, corresponding to the reaction between Al, Ni and Al, Nd powders.
4. For powders milled 30 h and longer prevailing of Al_3Ni intermetallic phase was detected, supported by the decreasing intensity of the endothermic peak at 635 ^{0}C (melting of the Al-Ni eutectic left) tand it disappeared by milling for 80 h.

5. References

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