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Nanocrystallization of $Al_{88}Ni_{10}Nd_2$ Alloy by Mechanical Alloying

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Abstract

In the present study, mixtures of Al, Ni and Nd elemental powders with compound percentage of $Al_{88}Ni_{10}Nd_2$ were alloyed by mechanical alloying system for times up 110 h yields nanocrystalline products. The mechanically alloyed powders' structural changes and thermal behaviour of different stages of milling were characterized by X-ray diffraction (XRD), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). The SEM, XRD and DSC results demonstrated that solid state reaction occurred during mechanical alloying and nano-intermetallic compounds of the $Al_{88}Ni_{10}Nd_2$ powders could be obtained. Microstructures of the $Al_{88}Ni_{10}Nd_2$ mechanically alloyed powders were determined to show the formation of fine nanoparticles having a size of touching 10-20 nm. However, no amorphous phase was obtained in $Al_{88}Ni_{10}Nd_2$ powders after milled for times up 110 h.

1. INTRODUCTION

Nanocrystalline materials have got a special attention owing to their matchless combinations of mechanical and physical properties which are potentially excellent to those of their coarse-grained equivalent. These materials are generally manufactured by fast solidification of the alloying components from the melt state. The last decade, it is reported that the nanocrystalline materials can be also produced by mechanical alloying (MA) technique [1-3]. The MA system is a dry, high speed ball milling technique and has been used to produce several beneficial and scientifically attracted materials such as crystalline, nanocrystalline and quasicrystalline intermediate phase, amorphous alloy and supersaturated solid solutions materials [4]. It has been shown that a nano-sized grain structure can be obtained in most materials after sufficient milling time [5]. On the other hand, this technique can be combined with appropriate powder compression with ease and guarantee to allow preparation of bulk samples with extra larger dimensions than those existing through rapid solidification.

Among a large number of nanocrystalline alloys, Al-rich alloys have been admitted important research interest because of their technological and scientific importance [6-9]. Al-rich amorphous alloys have also attracted increasing interest as a manufacture nanocrystalline alloys by controlled crystallization of amorphous alloys due to the appearance of super high mechanical properties which have not been obtained for crystalline single phase or amorphous alloys [10-13].

In this work, the milling time effect on the microstructural changing, thermal treatment and the morphological variation of the powder particles obtained from MA of Al88Ni10Nd2 was investigated using XRD, DSC and SEM.

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2. MATERYALS AND METHODS

In this study, powders of Al (99.97 % purity, max. particle dimension 250 μm), Ni (99.5 % purity, max. particle dimension 10 μm) and Nd (99 % purity, max. particle dimension 100 μm) elementals were used as beginning materials. The powders were weighted in an argon glow box to acquire the percentage compound $Al_{88}Ni_{10}Nd_2$. The powders were then sealed under an argon atmosphere at 250 ml volume cylindrical stainless steel container together with 9-12 mm diameter stainless steel ball. Fritsch planetary ball mills, model Pulverisette 5, employing a ball-to-powder mass ratio of 10:1 and a rotating speed of 300 rpm with states of 20 min milling and 20 min rest to prevent the mixture from overheating. During mechanical alloying stearic acid (1 % mass) was used to reduce the adhering of ductile metals to the milling medium such as vial wall and the milling balls. To avoid oxidation, the vial was not opened during MA. Structural evolution and morphological change of the mechanically alloyed powders at different stages of milling was characterized by XRD and 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. For phase description, measurements were scanned for a broad range of diffraction angles (2θ) ranging from 20 to 100° with a scanning rate of 5 °/min. SEM analysis were 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 enhanced contrast. The crystallization attitude of the mechanically alloyed powders at different process of milling were analyzed by DSC using a Perkin-Elmer's Sapphire DSC-7 at a constant heating rate of 10 K/min from 50 to 675 ºC which corresponding to the limit of the DSC used.

3. RESULTS AND DISCUSSION

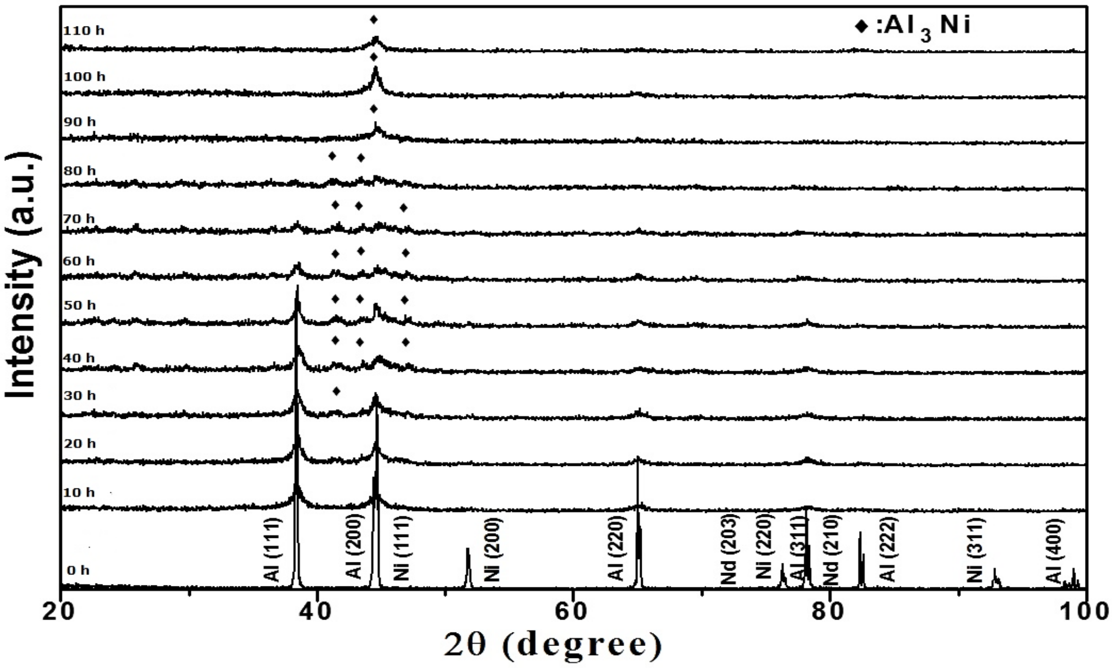

Figure 1. *XRD patterns from $Al_{88}Ni_{10}Nd_2$ powders after increasing milling time states*

The XRD samples of the mechanically alloyed $Al_{88}Ni_{10}Nd_2$ powder as-received and after increasing milling time states are showed in Figure 1. It can be seen that Nd peaks are sharply as certainable even for the powder mixture without milling because of its percentage concentration and fractional overlapping of its peaks with Al peaks. However, the sharply crystalline diffraction peaks of the as-received powder broadened progressively during MA because of the grain dimension refinement and internal strain accumulation. Next 10 h of milling, corresponding to Ni the diffraction peaks couldn't be well identified due to the broadening and reduction of the peak intensity. MA for 40 h led to alloying of the as-milled powder. This is evident with appearance of several new peaks as shown in Figure 1. This is related with the disappearance of the Al peaks. The new peaks are attributed to the orthorhombic Al_3Ni phases. Continued milling to 90 h led to the dissolution of all the other phase into a single Al_3Ni (131) phase. Between 90 to 110 h milling time, no significant change in the XRD pattern were seen. In this stage of milling (from 90 to 110 h), the XRD patterns showed only single intermetallic Al_3Ni (131) phase at around $2\theta=44°$. Therefore, this is an indicating that the intermetallic Al_3Ni phase is the steady phase for this alloy under the milling conditions studied. On the other hand, no amorphous phase has been obtained in $Al_{88}Ni_{10}Nd_2$ powders even after milled for times up 110 h. This probably because of the severe sticking of the aluminium powders to the milling tools, which causes a shift from desired stoichiometry to significantly lower Al content.

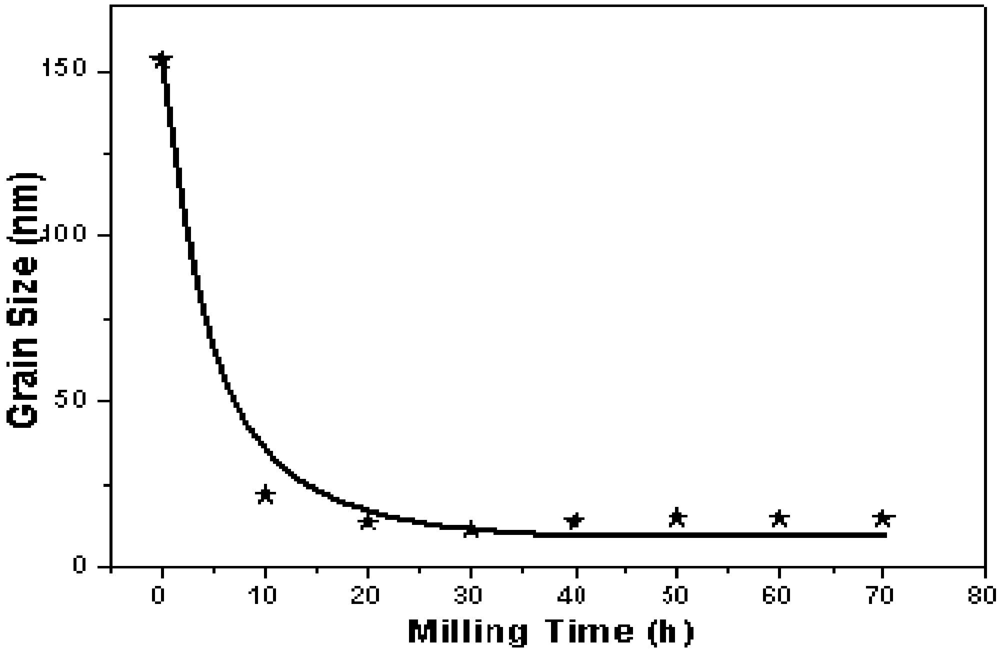

Figure 2. *Variation of grain dimension of the powders after increasing milling time states*

The changes of the average grain (crystal) dimensions during mechanical alloying were estimated from XRD line broadening. Scherrer`s equation [14] was applied for the fcc-Al (111) peak since there is no overlap with other diffraction peak. Figure 2 shows the change in grain dimension of the Al particles after increasing milling time states. Based on Scherrer`s equation, the average grain dimension of as-received Al powders were found about 150 nm. However, as it can be seen from Figure 2, after 10 h of milling the grain dimension decreased sharply and then approach a constant value at longer milling time. Similar grain refinement behaviours were previously reported [1-2, 8, 15]. However, after 70 h of milling the average grain dimension of Al powders were about 10-20 nm. Therefore, mechanical milling has the natural advantage of grain refinement.

In order to investigate the structural change during mechanical alloying of $Al_{88}Ni_{10}Nd_2$ powders, the samples were investigated by SEM. The morphology of as-received and milled powder particles after 10, 40 and 110 h are shown in Figure 3. As-received powders had a nearly spherical morphology and average particles dimension were about 10-100 μm. For the powders subjected to 10 h of MA, the change of particle dimension was not significant. In this case, the presence of isolated flat particles with layered substructure indicates that the mechanical alloying process is not yet completed. With increasing milling time to 40 h, the average particle dimension decreases and cold-welded by the colliding balls, leading to the formation of rather large agglomerates. As it is seen in Figure 3, 110 h of mechanical alloying results in the formation of submicrometer particles with an average particle dimension of about 5 μm. This means that the scale of the microstructure decreases with increasing milling time.

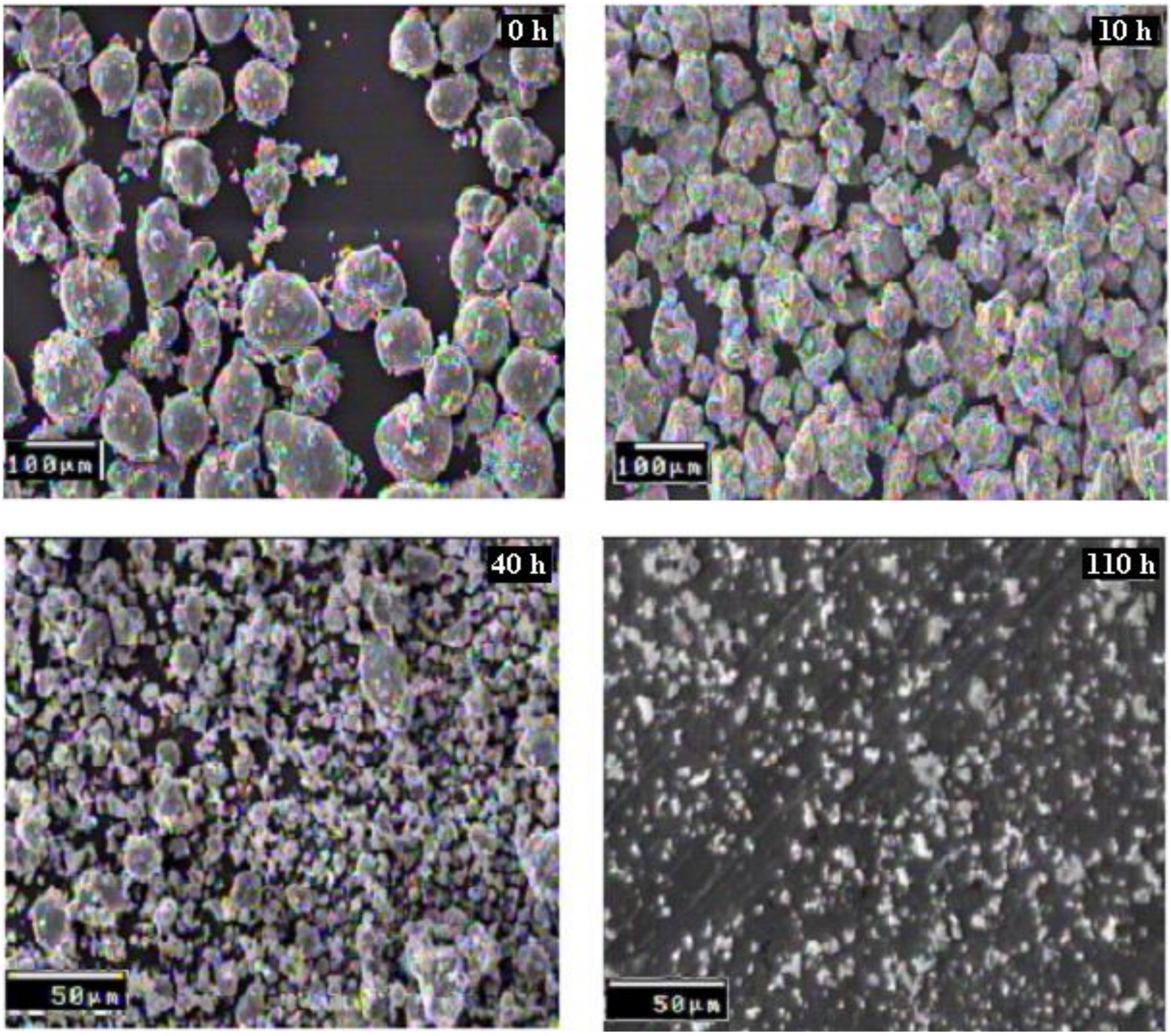

Figure 3. *SEM images of the $Al_{88}Ni_{10}Nd_2$ powders milled for different hours*

Physical and mechanical properties of materials violently depend on their grain sizes. A lot of studies have been exhibited the relationship between the microstructurel, physical and mechanical properties. For this aim, determination of grain size has a great importance. The SEM investigation is basically based on direct observations and counting of the particles, provides particle dimension and its distribution, which is closer to reality of the morphology. However, due to the ease of XRD, Scherrer's method has been used extensively to determine the grain (crystal) size of the coherent domains in nanomaterials. Usually, there is a large discrepancy between the results obtained by SEM and XRD. This discrepancy originates from the difference between the type of the information obtained by XRD and SEM. In fact, the size values obtained by XRD and SEM corresponds to grain (crystal) size and coherently scattering of domain size of particles [16-24].

Figure 4 shows DSC marks from the $Al_{88}Ni_{10}Nd_2$ powders as-received and after increasing milling times. For milling time shorter than 40 h, the DSC traces showed a broad exotherms around 250-300 ºC. This is due to the reaction between Al, Ni and Al, Nd powders, which are not completely alloyed to form intermetallic Al_3Ni phase. This result is consistent with the XRD results. However, the intensity of this exotherms decreased with increasing milling time from 10 to 30 h and completely disappeared after 40 h of milling. Another important feature that can be deduced from DSC curves is the endothermic peak at 635 °C. The equilibrium phase diagram of Al-Ni alloy indicates that the melting temperature of Al-phase is at 640 °C [25,26]. Thus, it is clear that the endothermic peaks at around 635 °C correspond to the melting of the Al-phase. However, the intensity of this endothermic peak decreases with increasing milling time and completely disappeared after milling of 80 h. This result is again consisted with XRD observation. As mentioned above, XRD patterns of the $Al_{88}Ni_{10}Nd_2$ powders after milling of 80 h show only single intermetallic Al_3Ni phase and the melting temperature of this phase is higher than 800 °C [26].

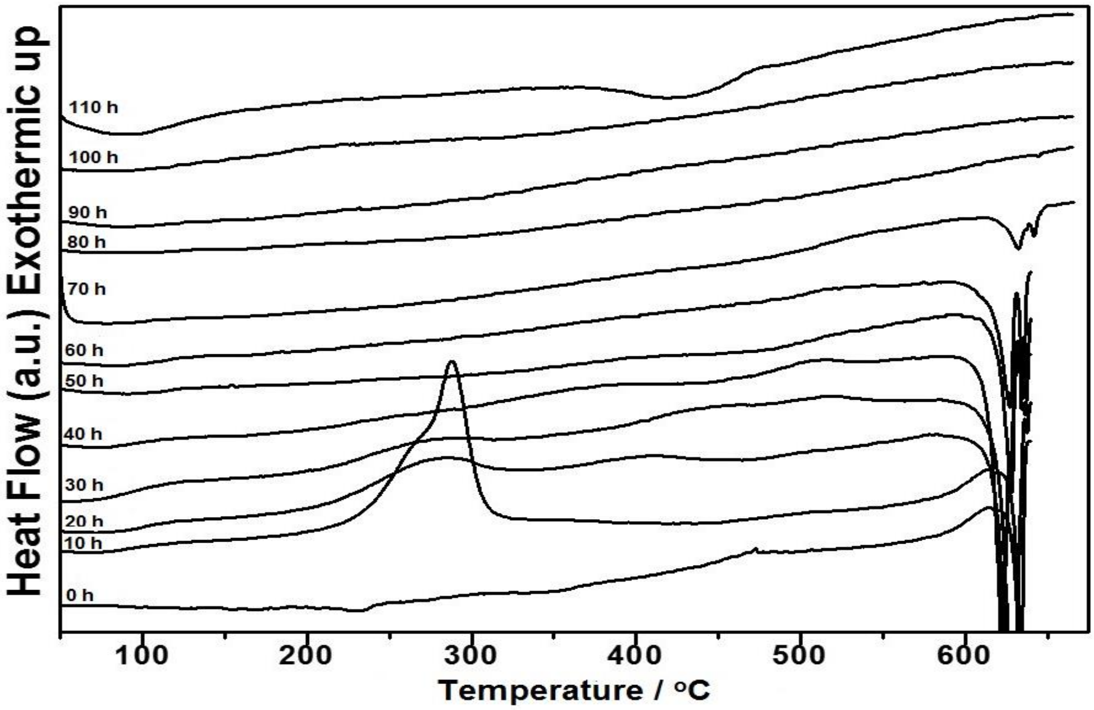

Figure 4. *DSC curves of the* $Al_{88}Ni_{10}Nd_2$ *powders after increasing milling time states*

4. CONCLUSIONS

In this research, the impact of the milling time on the structural and thermal changes of $Al_{88}Ni_{10}Nd_2$ milled powders were investigated and the main results obtained are summarized as follows:

1. Mechanical alloying of $Al_{88}Ni_{10}Nd_2$ powders resulted in the formation of nanoparticles with a dimension of about 10-20 nm. No amorphous phase was obtained even after milled for times up 110 h.
2. In the initial milling stages, the average grain dimension of as-received Al powders reduced with increasing MA time since the grain dimension refinement and the internal strain storage.
3. After 40 h of milling, the intermetallic Al_3Ni and phases were observed.
4. Milling time is shorter 40 h, the DSC traces display a broad exotherms nearby 250-300 ºC corresponding to the reaction between Al, Ni and Al, Nd powders. DSC traces of the mechanically alloyed $Al_{88}Ni_{10}Nd_2$ powders also display an endothermic peak at around 635 ºC which correspond to the melting of the Al-phase. However, the intensity of this endothermic peak decreases with increasing milling time and completely disappeared after milling of 80 h.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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