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Effect of Mo Addition on Nanostructured $Ni_{50}Al_{50}$ Intermetallic Compound Synthesized by Mechanical Alloying

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The mechanical alloying process was used to synthesize the $Ni_{50}Al_{50-x}Mo_x$ nanocrystalline intermetallic compound using pure Ni and Al elemental powder. This process was carried out in the presence of various Mo contents as a micro-alloying element for various milling times. Structural changes of powder particles during mechanical alloying were studied by X-ray diffractometry (XRD) and scanning electron microscopy (SEM). Results showed that mechanical alloying in various combinations was completed after 48 h of milling time. Minimum crystallite size of the as-milled powders (~10 nm) was achieved after introducing Mo and milling for 128 h. Also, lattice strain decreased with increasing milling time up to 48 h and again increased after 48 h of milling time. On the other hand, the presence of Mo significantly affected variation intensity of the lattice parameter and morphology of the powder particles.

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1. INTRODUCTION

The intermetallic compound NiAl which has drawn great attention owing to its physical properties can be exploited for high-temperature structural applications. The NiAl compound possesses high melting point (2184 °C), low density (5.86 g/cm^3), excellent thermal conduct, excellent oxidation, and corrosion resistance. However, lack of room temperature ductility and poor strength at high temperatures hinder its utilization as a candidate for replacing Ni-based superalloys. Therefore, many efforts have been made to reduce the brittleness of NiAl intermetallic compounds which can be classified as the modification of slip system by ternary or higher-order alloying [1], grain refinement [2, 3], and incorporation or precipitation of a ductile phase [4]. Also, improvement of the mechanical properties at high temperature has been reported through the second-phase incorporation [5]. Mechanical alloying (MA) can provide all the above-mentioned points simultaneously; therefore, this technique has been extensively applied to synthesize nickel aluminide intermetallic. Although extensive studies have been carried out on these alloys, new investigations still continue to reveal attractive properties of the NiAl alloy system [2, 6-8].

NiAl intermetallic compound have some potential applications; therefore, it has been greatly considered by researchers and experts in many applications; such as hot sections of gas turbine engines for aircraft propulsion systems, electronic applications, coated superalloy, connecting semiconductors in the electronic and catalyst industry, electric heating elements, structural components of energy conversion systems, medical engineering, chemical industry, magnetic materials, hydrogen storage materials, and rolling rollers [9-12]. Mechanical alloying has been successfully applied for making nanocrystalline materials. Many researchers have reported making nanocrystalline NiAl intermetallic compound by MA [13, 14]. However, there are a few reports in the literature on the MA of ternary Ni–Al–Mo powders. Recently, great attention has been paid to the investigation of ternary systems by mechanical alloying. Zamora et al. [15] studied the Ni-Al-Mo system and reported that milling intensity had a major effect on the evolution of the microstructure and

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phase formation. Alizadeh et al. [16] investigated the formation of $Ni_{50}Al_{50-x}Cr_x$ intermetallic compounds by MA and reported that the presence of Cr in NiAl system led to faster formation of nickel aluminide in the air. Albiter et al. [17] reported that addition of Mo, Fe, and Ga to NiAl can improve its mechanical properties. Darolia et al. [18] reported that the alloying elements can be used to improve the nickel aluminide ductility; but, maximum ductility was achieved when the additive value was less than 0.5 %. According to their results, ductility was raised from the interstitial defects. It has been reported that applying 0.1 to 0.2 % of Mo, Ga, and Fe would be useful for increasing the RT ductility of soft single-crystalline nickel aluminide, while these micro-alloying elements do not considerably affect the polycrystalline nickel aluminide. Noebe et al. [5] reported that a rod structure of NiAl eutectics with Mo caused a sufficient increase in the creep properties of NiAl. One of the elements that overcome shortcomings of the intermetallic compound is Mo, the most useful effect of which is increasing room temperature ductility of this compound [19].

In the present work, MA technique was used to synthesize the NiAl-Mo compound containing various Mo contents as a micro-alloying element.

2. MATHEMATICAL MODEL

High purity Ni (>99.9%), Al (>99%), and Mo (>99.99%) elemental powder blends with the initial particle size of <200μm were mechanically mixed in high energy planetary ball mill using a sealed tempered steel container under atmosphere at the milling speed of 250 rpm. Various elemental blends with the composition of $Ni_{50}Al_{50-x}Mo_x$ (where x = 0, 2.5, 5) were studied in the present investigation. Hardened chromium steel balls (12 balls-1cm and 4 balls-2cm in diameter) were also used. Different ball sizes with more accidental collisions [19] gave more energy to the powder particles [20] and also resulted in less adhesion of powders on ball surfaces and more homogeneity of product mixtures [21]. The ball-to-powder ratio was 15 in mass. A small amount of ethanol (3 wt%) was added to prevent excessive welding of the powders to the steel balls and container. The milling times were 8, 16, 48, and 128 h. To control and prevent temperature rise, 15 min stop was applied after every 30 min of milling.

Structural changes of the powder particles were determined by X-ray diffraction (XRD) with Cu-Kα (λ = 0.154 nm). Morphology and microstructure of the mechanically alloyed powder particles were characterized by scanning electron microscopy (SEM) in a Philips XL30. Lattice parameters were determined by the separation of $K\alpha_1$ and $K\alpha_2$ diffractions at high angles of (2 0 0), (2 1 1), and (2 2 0) with extrapolation function method. The measured lattice parameters were plotted versus Cotgθ*Cosθ and the best fit with least square deviation was obtained. The cross point of the obtained line with vertical axis gives the best value for lattice parameter [22, 23]. Crystallite size and internal strain in the milled samples were calculated from the XRD line broadening using the Williamson–Hall equation [24]:

$$\beta \cos\theta = K\lambda / D + 2\varepsilon \sin\theta \quad (1)$$

where, ε is internal strain, β is peak broadening due to crystal refining and internal strain, the Scherrer constant, k, is 0.9, and λ = 0.1542 nm.

3. MATRIX GEOMETRIC SOLUTION

3. 1. Morphology NiAl intermetallic compound was successfully synthesized through milling Ni, Al, and Mo powder mixtures in a planetary mill. Kubaski et al. [25] showed the temperature of the vial during milling for this composition, at which the MSR can be noticed to occur between 62 and 65 min of milling; i.e. there was incubation time before the beginning of the reaction. This exothermic reaction can be attributed to a mechanically induced self-propagating reaction (MSR) [26, 27] and account for the compound formation during MA. The starting powders (Figures 1a, 2a, and 3a) had both spherical and flake shapes of aluminum with tiny nickel particles. Powder particles suffered from severe plastic deformation and, because the powder particles tended to more cold welding, size of particles increased compared to their initial state, indicating that rate of the cold welding process was dominant compared to the fracturing process [28]. After 16 h of milling time, the powders form the particles with irregular shape and size (Figures 1b, 2b, and 3b) due to the success of the cold welding. At this stage, a sintering occurred between the powder particles due to the flame and (Ni, Al) solid solution was transformed into the NiAl intermetallic compound; the powder particles were severely affected by this process. Also, effects of cold working were very low. The NiAl compound formed from nickel and aluminum which is extant stick together in the effect of sintering and the particles with shape bulking is created. Milling up to 48 h led to more uniform particle size distribution; but, the particle size was coarser (Figures 1c, 2c, and 3c). At this stage, the particles that formed the (Ni, Al) solid solution (due to cold working) were transformed into the NiAl intermetallic compound and sintered due to the heat from strong exothermic reaction. As can be seen in Figure 8, the internal strain decreased at the milling time of 48 h which corroborated the above mentioned point. In addition, before opening the vial lid and completing the explosion reaction, a small amount of the NiAl intermetallic

compound was gradually formed within the vial, which caused a slight rise in temperature of the vial. Increasing temperature inside the vial during mechanical alloying increased the average particle size. Other forms of this issue have been also reported in other systems of mechanical alloying. Sharifati et al. [29] studied mechanical alloying in Fe-Co system and reported that, with starting the milling operation, the powder particles became flat. In addition, milling for longer time led to decrease in the grain size, but did not make an increase in the grain size at high times again. Youjun et al. [30] also examined the Ni-Al system and found a similar result. Excess milling (up to 128 h) produced more uniform size distribution of the particles and again decreased size of the particles (Figures 1d, 2d, and 3d). This figure indicates that the steady state for the size distribution of the particles was achieved after 128 h of milling due to the balance between cold welding and fracturing processes. It is generally known that high-energy ball milling processes reach a steady state in which particles have homogeneous size and shape [31]. At this stage, after the formation of (Ni, Al) solid solution with continuing milling process, as a result of topical warming, the powder particles were gradually transformed into the NiAl compound. Although this state caused coarsening the grain size, but milling operations after this formation again caused the cold working of the powder particles and decreased its grain size. In this step, cold working was dominated by fracturing.

At each stage, particle size for the powders containing molybdenum was smaller than the molybdenum-free samples. This subject was clear at the milling time of 48 h (Figures 1c, 2c, and 3c). According to Figure 7, the NiAl lattice parameter for the powders containing molybdenum was higher than the NiAl lattice parameter for the molybdenum-free samples, which confirmed the above points; i.e. with increasing the amount of molybdenum, more molybdenum was dissolved in NiAl intermetallic given the changes in lattice parameters.

As a result of Mo dissolution in NiAl intermetallic compound, stacking faults energy was reduced [32] and effect of work hardening became higher than that of the molybdenum-free sample, more dislocations and sub-boundaries were formed, and consequently grain sizes became finer. Furthermore, by the dissolution of Mo atoms in the Ni matrix, work hardening effects were increased [33, 34]; as a result, more powder particles were brittle and more failure occurred in them, which led to finer particles. In general, dissolution of alloying elements in metal crystals and generation of distortion in them increase work hardening in the cold working process [35, 36]. In fact, Mo saturation in Ni reduced such an effect. These results were in agreement with the previous results by Azimi et al. [37].

3. 2. XRD Analysis Figures 4 and 5 illustrate the XRD patterns of milled $Ni_{50}Al_{50-x}Mo_x$ (where x = 0, 5) powder mixture after different milling times. This figure shows only the presence of Ni, Al, and Mo in their elemental forms after 48 h of milling. On the other hand, reflections of NiAl intermetallic compound were already seen after 48 h of milling. The NiAl peaks can be seen in 16 h milled samples.

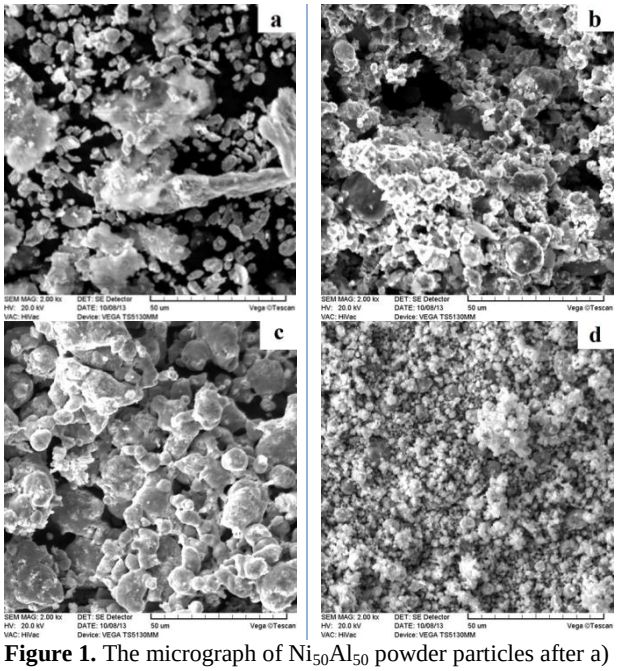

Figure 1. The micrograph of $Ni_{50}Al_{50}$ powder particles after a) 8, b) 16, c) 48 and d) 128 h milling times.

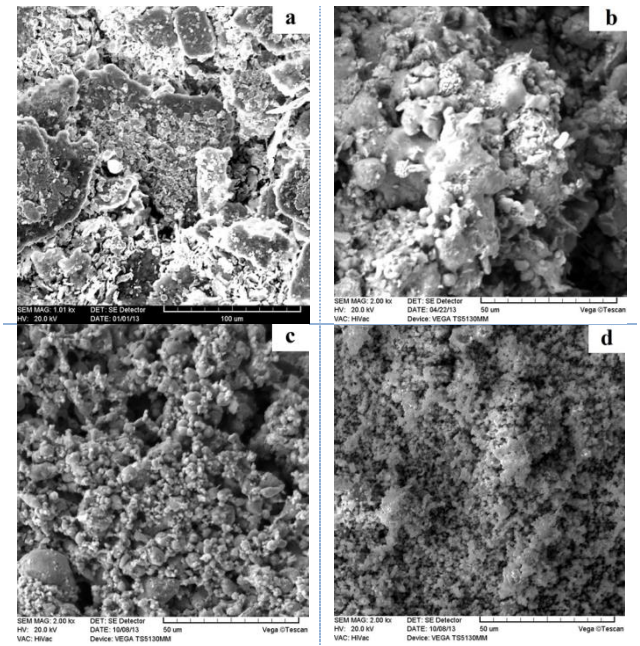

Figure 2. The micrograph of $Ni_{50}Al_{47.5}Mo_{2.5}$ powder particles after a) 8, b) 16, c) 48 and d) 128 h milling times.

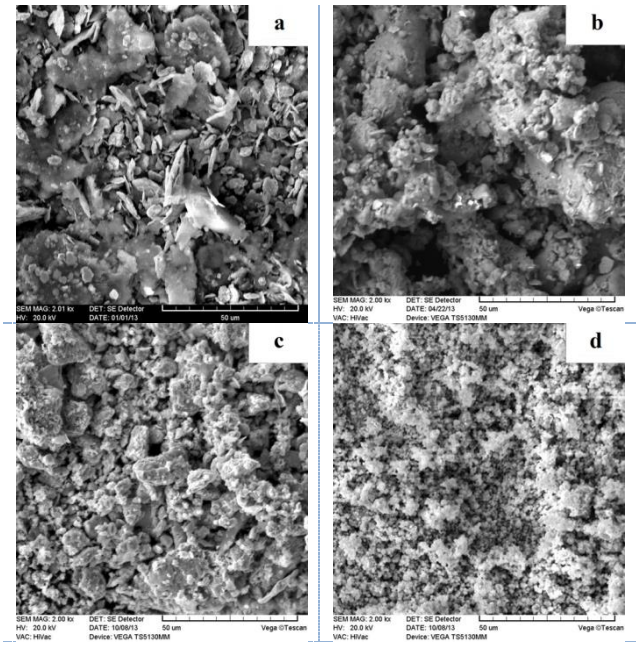

Figure 3. The micrograph of $Ni_{50}Al_{45}Mo_5$ powder particles after a) 8, b) 16, c) 48 and d) 128 h milling times.

At this stage, a sintering occurred between the powder particles due to the flame and (Ni, Al) solid solution was transformed into the NiAl intermetallic compound. In fact, analysis of these XRD patterns and the abrupt temperature increase [25] provided the evidence that NiAl formation took place during a sudden exothermic reaction (MSR) which occurred inside the milling vial and was mechanically activated. After 128 h of milling, only NiAl peaks were found in the XRD patterns. During MA, the sharp crystalline diffraction peaks of the as-received powder were broadened and their intensity was progressively decreased with increasing processing time. Mo, in addition to the solid state solution, tended to form second phase compounds such as Al_5Mo, Al_8Mo_3 and Mo_2C. Albiter et al. [17] reported that the presence of these phases was increased with the increase in the Mo contents. Mo_2C seemed to be formed through the reaction of molybdenum with the small amounts of ethanol added to the powders as a process control agent.

Decreasing the intensity of Al diffraction peaks was more severe than the Ni peaks, which was not due to the lack of Al atoms in the system, rather can be due to the severe plastic deformation that aluminum powder incurred during the milling process and thus its crystal size reached the nanometer range. This phenomenon caused a decrease in the intensity of Al peaks and increase in their width [38]. Peaks related to elements with lower atomic number and lower atomic scattering factor had a less degree and consequently their peaks disappeared faster than the elements with greater atomic number [16]. Positions of the crystalline Ni peaks showed a shift towards larger angles. The reason for more increase of the angles with increasing milling time could be attributed to increased crystal defects and then dislocations as a result of severe plastic deformation of the particles. These phenomena themselves caused an increase in elastic strain and also refinement of the grain size of Ni and Al, both of which led to the increased peak width of Ni and Al [39, 40].

3. 3. Crystal Size Variations of crystal sizes in the three samples of 0, 2.5, and 5% at Mo contents during milling are given in Figure 6. According to the figure, after 8 h of milling in all the samples, crystal sizes were in the range of 10 to 170 nm (XRD method is suitable for the determination of particle size less than 100 nm, Therefore, this measurement is not very accurate). At the same time, temperature may rise locally and especially at particle surfaces enough for diffusion of Mo atoms into Ni matrix. Diffusion can be also depend on other factors [16, 34, 41-43]. Crystal size in NiAl molybdenum-free samples after 128 h of milling was 26 nm and, with increasing molybdenum up to 5%, this value was decreased to 10 nm. With increasing molybdenum, crystallite size suddenly decreased for the powders containing 2.5 and 5% Mo; but, this trend reached saturation with more increase of molybdenum, which can be due to the solubility saturation of Mo atoms in nickel lattice. Albiter et al. [17] reported that addition of Mo tended to slightly refine the grain size of the NiAl alloy.

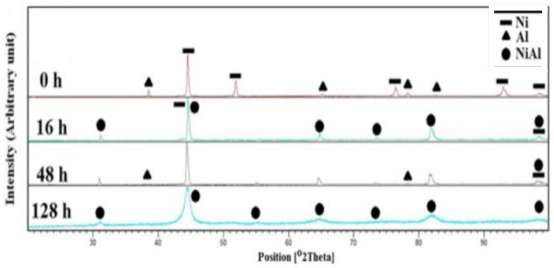

Figure 4. X-ray diffraction patterns of $Ni_{50}Al_{50}$ powder mixture after different milling times.

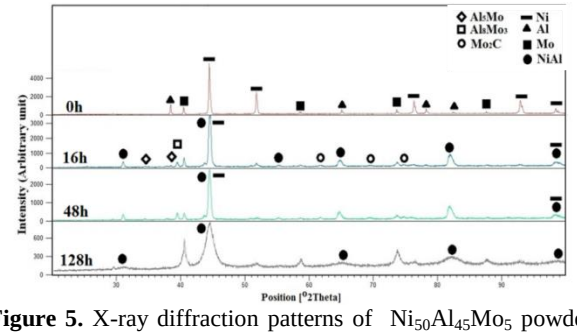

Figure 5. X-ray diffraction patterns of $Ni_{50}Al_{45}Mo_5$ powder mixture after different milling times.

They also reported that average grain size was less than 20 nm in comparison to the NiAl alloy, where the grain sizes were between 20 and 50 nm. Dislocation networks are suitable paths for high diffusion rates of Mo atoms. However, more plastic deformation in the pre-cold-worked matrix may move dislocations without moving their diffused Mo atoms. Therefore, Mo atoms may be left inside Ni matrix. Repeating this process by continuing milling operation will lead to more homogeneous distribution of dissolved Mo atoms with much higher concentration than the equilibrium value. At longer milling times, higher dislocation densities and their pile-ups at obstacles may lead to the formation of new boundaries and fragmentation of initial crystals into nano-scale crystals [37]. Formation of nanocrystals and diffusion of Mo atoms into Ni matrix will continue even more rapidly, since more boundaries facilitate the diffusion process. As a result, work hardening effect was reduced and consequently dislocations and sub-boundaries were formed to some extent and then the intensity of crystallite size reduction was decreased [10]. Intense grain size reduction is one of the methods for improving ductility so that nanometric dimensions of grains lead to further improvement in ductility and creeping resistance than the micron grains [44, 45].

3. 4. Lattice Parameter Variations of NiAl lattice parameter in powder mixtures with 0, 2.5, and 5% Mo against milling time are illustrated in Figure 7. In all the cases, lattice parameter increased with milling time [46]. On the other hand, in the same figure, in the presence of molybdenum, variation trend of lattice parameter became more intense than the molybdenum-free sample and, the more the milling time increase, the more the increase in the lattice parameter would be. Large amounts of molybdenum in the primary powder mixture caused higher density gradient, which led to more penetration of molybdenum atoms into the nickel lattice and thus its further expansion. Hence, at the identical time of milling, there was higher variation of NiAl lattice parameter in the primary mixture with more molybdenum; consequently, a more supersaturated solid solution was formed. Albiter et al. [17] added molybdenum to $Ni_{56}Al_{44}$ compound and reported similar results. The reason for more increase of lattice parameter with increasing molybdenum could be attributed to increased crystal defects and then dislocations as a result of severe plastic deformation of particles. Thus, sub-grains were formed; sub-grains and dislocations made vaster pathways for the movement of molybdenum atoms and were displaced by more plastic deformation of particles; however, molybdenum atoms were not displaced. By repetition, there would be an increased possibility for the penetration of Mo atoms [36]. Azimi and Akbari [36] also reported a complex behavior of the lattice parameter changes as a result of the dissolution of zirconium atoms in copper during the mechanical alloying process. Entry of larger zirconium atoms in the copper matrix led to increased copper lattice parameter, which indicated the formation of zirconium-copper solid dissolution.

3. 5. Internal Strain Variations of crystal sizes in the three samples of 0, 2.5, and 5% Mo contents during milling are given in Figure 8. As can be seen, with increasing milling time, in both samples, internal strain received by the powder particles within 16 to 48 h of milling had a falling trend. Lattice strain followed an increasing trend with increasing milling time up to 128 h. In addition, the NiAl intermetallic compound containing molybdenum demonstrated higher internal strain than the molybdenum-free sample. In other words, it can be said that, with increasing the amount of Mo, more internal strain of the milling tool was transferred to the powder particles. With increasing molybdenum and dissolution of its atoms in Ni matrix, more defects, specifically dislocations, were formed in the materials.

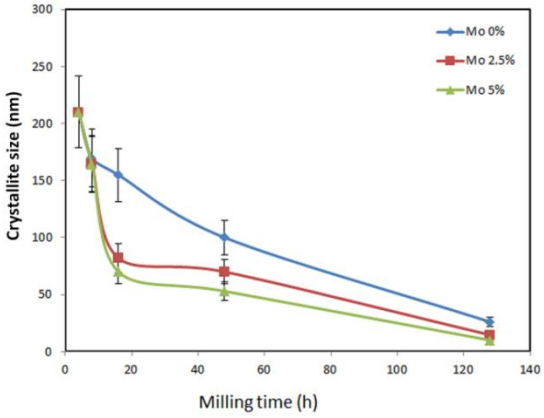

Figure 6. Crystallite size variations during milling in $Ni_{50}Al_{50-x}Mo_x$ (X = 0, 2.5 and 5 %) powder mixtures.

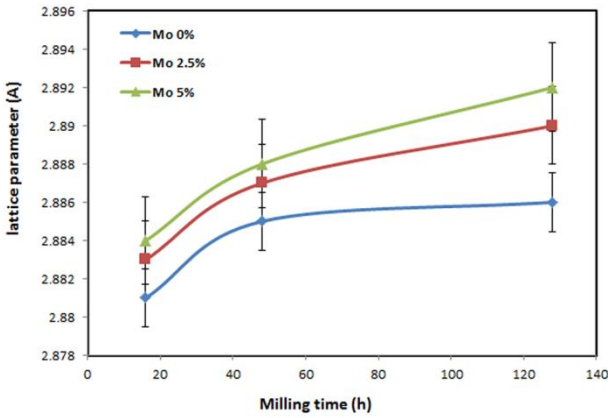

Figure 7. Lattice parameter variations during milling in $Ni_{50}Al_{50-x}Mo_x$ (X = 0, 2.5 and 5 %) powder mixtures.

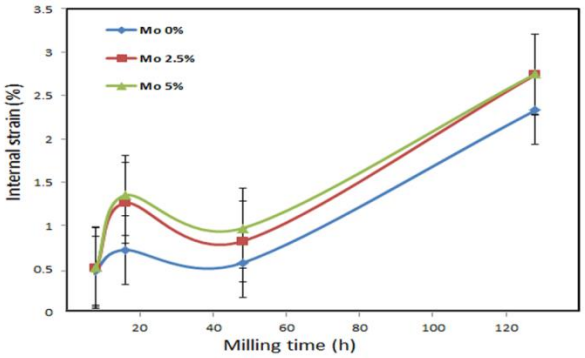

Figure 8. Internal strain variations during milling in Ni50Al50-xMox (X = 0, 2.5 and 5 %) powder mixtures.

Decrease of the lattice strain at time of 48 h was due to the excessive heat of combustion reaction for transformation of the (Ni, Al) solid solution into the NiAl intermetallic compound. Within the time interval of 48 to 128 h of milling, the NiAl intermetallic compound was almost formed and this intermetallic compound was in the middle of the plastic deformation as the agent for increasing lattice strain. Sharifati et al. [47] investigated Fe–Co mixed powders containing 0, 5, and 9 wt% Cu for longer milling times and reported an increasing trend in the internal strain rate.

4. CONCLUDING REMARKS

The mechanical alloying process was used to synthesize the nickel aluminide intermetallic using the pure Ni and Al elemental powder. The MA was carried out in the presence of various Mo contents as a micro-alloying element for various milling times. Mo addition led to second phase formation of the Al_5Mo, Al_8Mo_3 and Mo_2C types. Mo_2C phase can be related to the reaction between the Mo and methanol. The micro-alloying additions refined the microstructure of the intermetallic NiAl. Thus, for example, the average grain size obtained from NiAl was of the order 26 nm. However, with ternary additions, the grain size was reduced up to less than 10 nm. Also, internal strain of the powder particles increased with increasing Mo. Broadening peaks of X-ray diffraction pattern due to molybdenum increase was mainly due to decreased crystallite size and increased lattice strain. In the presence of molybdenum, the produced alloy's lattice parameter showed higher values.

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Effect of Mo Addition on Nanostructured $Ni_{50}Al_{50}$ Intermetallic Compound Synthesized by Mechanical Alloying

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چکیده

در این تحقیق خواص ریزساختاری ترکیب بین فلزی $Ni_{50}Al_{50-x}Mo_x$ تهیه شده به روش آلیاژسازی مکانیکی بررسی گردیده است. این فرایند در حضور مقادیر مختلف مولیبدن به عنوان یک عنصر میکروآلیاژ در زمان های مختلف انجام شد. تغییرات ساختاری ذرات پودر در طول آلیاژسازی مکانیکی توسط الگوهای پراش اشعه ایکس (XRD) و میکروسکوپ الکترونی روبشی (SEM) مورد مطالعه قرار گرفت. نتایجنشان می‌دهدآلیاژسازیدرترکیب های مختلف بعدازحدود ٤٨ساعتآسیاب‌کاری تکمیل گردید. کمترین اندازه کریستالیت پودرهای آسیاب شده (١٠ نانومتر) بعد از افزودن مولیبدن در ١٢٨ ساعت آسیاب کاری به دست آمد. همچنین کرنش شبکه با افزایش زمان آسیاب کاری تا ٤٨ ساعت کاهش یافت و مجددا بعد از ٤٨ ساعت آسیاب‌کاری روند افزایشی نشان می‌دهد. ازسویدیگرحضورمولیبدنروند و شدتتغییراتپارامترشبکه، کرنش داخلی و مورفولوژی ذرات پودر را در حین آسیاب‌کاری به طور محسوسی تحت تاثیر قرار داده است.

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