



Investigation of chromium effect on synthesis behavior of nickel aluminide during mechanical alloying process

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ABSTRACT

The mechanical alloying process was used to synthesize the $\text{Ni}_{50}\text{Al}_{50-x}\text{Cr}_x$ nanocrystalline intermetallic compound using the pure Ni and Al elemental powder. The mechanical alloying was carried out in the presence of various Cr contents as a micro-alloying element for various milling times. The synthesis behavior of final product was investigated by using XRD, SEM and EDS. Results confirmed that synthesis behavior of nickel aluminide intermetallic depends on the Cr content and milling time. The results revealed that for an inadequate milling time, the intermetallic phase is generated after opening the vial lid. The EDS results depict that the samples synthesized in the air atmosphere, i.e. after opening the vial lid, include a considerable value of oxygen. The X-ray map results reveal that, after opening the vial lid, the oxidation of milled aluminum particles will result in the generation of final product in the air atmosphere. According to XRD results, when the milling time is constant, increasing the Cr content leads to acceleration in the nickel aluminide formation in the air atmosphere.

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1. Introduction

Nickel aluminide intermetallics have been studied previously as a potential high-temperature structural material due its many attractive properties such as low density, high melting point, good temperature stability, high-temperature oxidation and corrosion resistance. Nickel aluminide is also known as a high thermal conductive compound which its conductivity is approximately eight times higher than those of typical nickel based super alloys [1–7]. These key advantages make this material to be suitable for extensive applications especially at elevated temperatures. However, the poor ductility at the room temperature (RT) limits its industrial applications. Hence, numerous attempts have been made in recent years to overcome this problem. The common methods employed to improve the RT ductility of nickel aluminide intermetallics include: grain refining, improving the slip systems, and alloying [8–15]. Mechanical alloying (MA) can provide all those mentioned above simultaneously, therefore, this technique has been extensively applied to synthesize nickel aluminide intermetallic.

Many alloying attempts have been made to improve the RT ductility by altering the slip vector from (1 0 0) to (1 1 1) in nickel

aluminide which has high ordering energy [16–19]. Darolia et al. have reported that the alloying elements can be used to improve the nickel aluminide ductility but the maximum ductility is achieved when the additive value is less than 0.5 at% [20]. According to their results the ductility is raised from the interstitial defects. It has been reported that applying 0.1–0.2 at% of Mo, Ga and Fe would be useful to increase the RT ductility of soft single-crystalline nickel aluminide while these micro-alloying elements do not considerably affect the polycrystalline nickel aluminide [21].

Among the various micro-alloying elements examined to improve the mechanical properties of nickel aluminide, Cr can be a suitable additive. The theoretical calculations indicate that the anti-phase boundary (APB) energy can be significantly reduced by Cr additive [22–24], thus, it enhances the possibility of (1 1 1) (1 1 0) slip system in nickel aluminide which is required to satisfy the *von mises* criterion for ductility of polycrystalline materials. Cr solute atoms which are mainly on Al sublattice sites substantially expand the lattice parameter and produce an unusual solid solution softening effect [24]. Cr is also thought to be effective at preventing hot embrittlement caused by oxygen enrichment at the grain boundaries [25].

As already mentioned MA is known as a proper tool for synthesis of nanocrystalline NiAl-x intermetallics. It has been reported that in the nanocrystalline materials obtained by MA, due to the extensive deformation, the internal energy of the particle lattice increases and consequently their reactivity is increased. The main factors that

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Table 1

Formation behavior of NiAl after the considered milling time: (☑) Formation of NiAl in vial under Ar atmosphere; (✓) Formation of NiAl in air.

Number	Composition	8 h	12 h	16 h
1	NiAl	✓	✓	☑
2	NiAl _{49.75} Cr _{0.25}	✓	✓	☑
3	NiAl _{49.25} Cr _{0.75}	✓	☑	☑
4	NiAl _{48.5} Cr _{1.5}	✓	☑	☑
5	NiAl _{47.75} Cr _{2.25}	✓	☑	☑

affect the mechanisms occurring in MA process are fracture and cold welding repetition of particles followed by an increase in their reactivity [26].

In the present work, MA technique is used to synthesize the NiAl–Cr compound including various Cr contents as a micro-alloying element. The main goal of this work is to investigate the mechanisms occurred during mechanical synthesis of NiAl–Cr, especially to describe the effects of Cr micro-alloying content on the synthesis behavior of final product during MA process.

2. Experimental procedure

High purity (>99.9%) Ni, Al and Cr elemental powder blends with initial particle size of <50 μm were mechanically mixed in high energy planetary ball mill (Fritsch Pulverisette) using a sealed tempered steel container under Ar atmosphere at a milling speed of 250 rpm. Various elemental blends with the composition of Ni₅₀Al_{50-x}Cr_x (where x=0, 0.25, 0.75, 1.5, 2.25) have been studied in the present investigation. 56 hardened chromium steel balls (50 balls-1 cm and 6 balls-2 cm in diameter) were used. The ball-to-powder ratio was 20 in mass. Small amount of ethanol was added to prevent excessive welding of the powders to the steel balls and container. The milling times were 8, 12 and 16 h. Samples were marked as A-B that A refers to the sample number and B refers to the milling time. XRD analysis was done using a Philips X'Pert diffractometer with Cu-Kα radiation. The nickel aluminide crystallite size (*D*) was determined using the Scherrer formula [27]:

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where *k* is a constant and generally assumed to be 0.89, λ the wavelength of Cu-Kα radiation, θ half of the diffraction angle, and β is the width (in radian) of half height for the diffraction peaks. To study the structural evolution during milling, a Philips XL30 scanning electron microscope (SEM) was used. The SEM was equipped with an energy dispersive spectrometer (EDS) for compositional analysis. X-ray mapping was also done to observe the distribution of the different elements in the milled powders.

3. Results and discussions

Following aspects can be interpreted from the XRD patterns of powder mixtures after MA at various milling times:

1. All XRD patterns show the nickel aluminide diffraction peak as the only phase existed in the final product.
2. The XRD patterns just differ together in (a): peaks displacement and (b): their intensity.

Table 1 shows the initial compositions selected for milling process and the environment in which the nickel aluminide compound is formed. According to Table 1, after stopping the milling process, two possibilities can be seen in relation to nickel aluminide synthesis as the milling product: (a) the nickel aluminide phase has been synthesized under Ar atmosphere through milling operation, i.e. before reaching the considered milling time. These samples were marked with ☑ symbol in Table 1. (b) The nickel aluminide compound would be generated after stopping the milling process and opening the vial lid, i.e. by an exothermic reaction in the air atmosphere followed by a flame. These samples are marked with ✓ symbol in Table 1. As Table 1 depicts, the mentioned possibilities depend on the milling time as well as the Cr contents. According to Table 1, 8 h of milling results in possibility of (b) for all Cr contents, 16 h of milling results in possibility of (a) for all Cr contents and



Fig. 1. Typical reaction followed by flame in the mixed powder after opening the vial lid (sample 1-8).

the formation behavior of nickel aluminide for 12 h of milling time depends on the Cr content. It can be seen that low Cr contents (samples 1-12 and 2-12) will result in possibility of (b) while increasing the Cr values (samples 3-12 up to 5-12) result in possibility of (a).

After opening the vial lid of the samples marked with (✓), mixed powders are exposed to air and a reaction takes place which is followed by a flame for a short duration. Fig. 1 depicts the reaction together with its flame for the sample 1-8 as well. This reaction begins in seconds after opening the vial lid followed by a flame for a short moment.

It was observed that the reaction in the samples 1-12 and 2-12 took place considerably faster than the ones in the samples with 8 h of milling. This observation can be related to decreasing the particle size which consequently enhances the surface area during MA process. Fig. 2 shows a SEM micrograph of the mixed powders in the samples 1-8 (Fig. 2a) and 1-12 (Fig. 2b). It can be easily seen that the sample with longer milling time (1-12) includes finer and more homogenous particles with respect to the ones with shorter milling time. Therefore, the samples milled for 12 h are more ready to react with the air with respect to the samples milled at 8 h.

Fig. 3 shows the X-ray maps for elements of Al, Ni and oxygen taken from a desired part of the sample 1-8. These maps were provided after generation of nickel aluminide phase in the sample 1-8. Also Table 2 shows the results of EDS analysis representing the oxygen percent in the samples synthesized in the air (✓) and Ar (☑) atmosphere. Fig. 3c clearly confirms the existence of oxygen more than it was expected (i.e. more than the alumina amount generated on the pure Al in the air atmosphere). These maps show that in spite of homogenous distribution of Ni atoms, Al has similar distribution with oxygen (see Fig. 3b and c). This implies that

Table 2

The results of EDS analysis representing the oxygen percent in the samples synthesized in the air (✓) and in the Ar (☑) atmosphere.

Samples	1-8 (✓)	1-12 (✓)	4-8 (✓)	4-12 (☑)
at% O	16.71	16.39	17.52	1.5

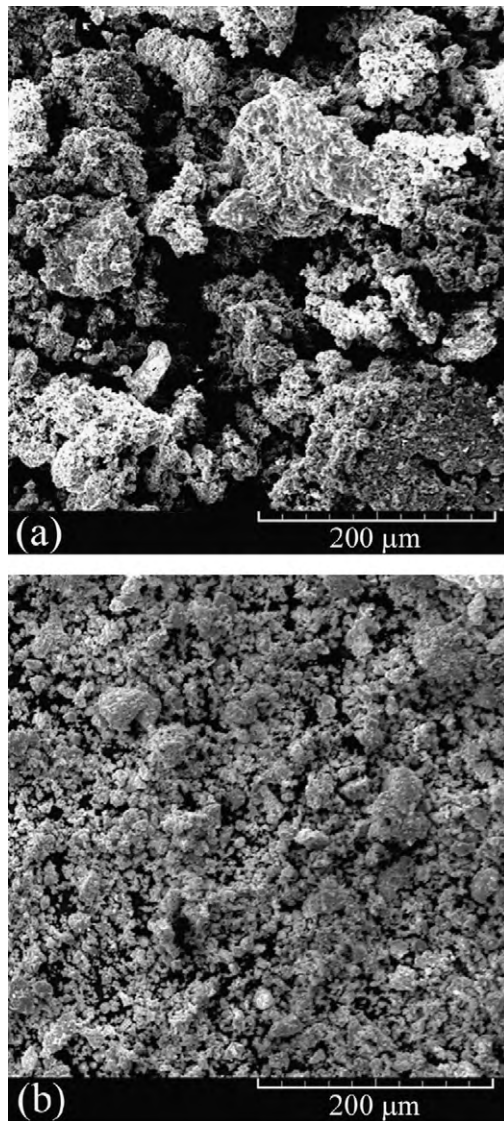


Fig. 2. The SEM micrograph of NiAl particles without Cr content generated in air atmosphere for the samples 1–8 and 1–12.

wherever there is a lack of Al atoms, there is also a lack of oxygen atoms. The presence of oxygen atoms can be used to explain the mechanism of nickel aluminide formation after milling stops. After opening the vial lid, the Ar gas is replaced by the oxygen which exists in the air. Contacting the oxygen with the Al particles activated through MA which have reached many high energy surfaces during MA causes a strong exothermic reaction by which a considerable heat is released. In other words, opening the vial lid causes oxidation of Al particles in some parts of the samples marked with (✓) (Table 1). Comparing the Fig. 3b and c reveals that the oxygen map coincides to the Al map. This coincidence confirms that Al particles have been reacted with the oxygen and it is well known that this reaction is extremely exothermic. The oxidation heat helps the formation of nickel aluminide phase by reduction of energy barrier against nickel aluminide formation. This process is similar to SHS (self-propagating high-temperature synthesis). Ignition occurs at a hot spot where the Al oxidation is taken place, and the combustive reaction propagates from there into other parts of the mixed powders and formation of nickel aluminide is finished after seconds.

To investigate nickel aluminide synthesis behavior more in-depth, study of the structural changes of Ni and Al lattice during

MA in the presence of Cr as micro-alloying element was attempted. For this purpose, it is necessary to investigate the XRD pattern of some milled samples before generation of nickel aluminide phase. Therefore the samples (2–8 and 5–8) in which the nickel aluminide phase is not formed under Ar are selected and their XRD pattern is achieved before reaction with air.

A cross-sectional XRD pattern related to the most intense peak of Ni is plotted in Fig. 4. This figure reveals the structural variations of Ni lattice during MA before generation of nickel aluminide phase. Three peaks are present in this figure: the lower peak is related to as received Ni powder and other peaks show the XRD patterns of Ni milled together with the Al powder for 8 h in the presence of Cr as micro-alloying element. The middle and upper peaks are related to the powder blend including 0.25 and 2.25 at% Cr, respectively. The middle and upper peaks are related to the samples in which there is not any nickel aluminide intermetallic yet. It is evident from Fig. 4 that the middle and upper peaks are slightly shifted toward higher 2θ angles with respect to the lower peak. As a result, the peak of Ni for the sample with higher Cr contents (upper peak) moves less than that one for the samples with lower Cr contents (middle peak).

Considering the atomic size of the elements existed in the powder blend (Ni = 124 pm, Cr = 128 pm and Al = 143 pm), the behavior observed in Fig. 4 is being explained as follows:

It is clear that during MA, heavy deformation, welding and fracturing is introduced into the particles. This is manifested by the presence of a variety of crystal defects such as dislocations, vacancies, stacking faults, and increased number of grain boundaries. The presence of this defect structure enhances the diffusivity of solute elements into the matrix. Furthermore, the refined microstructural features decrease the diffusion distances. In addition, the slight rise in temperature during milling further aids the diffusion behavior. Displacement of Ni peaks is caused by the stress induced by the structural defects such as vacancies created during MA. Since, the Cr atomic size is approximately close to the Ni atomic size, situation of Cr in some of the Ni vacancies reduces the stress induced by MA. In other words, solution of Cr in the Ni lattice compensates the stress stored in the Ni lattice raised by the defects induced during MA. For this reason, the peak of Ni for the sample with higher Cr contents (upper peak in Fig. 4) has been displaced less than the sample with lower Cr contents (middle peak in Fig. 4). However, the amount of Cr is not adequate to compensate the stress stored in the Ni lattice completely. It has been reported that Cr dissolution in Ni is an exothermic process. The heat released from this reaction accelerates the reaction of Ni with Al. Hence, increasing the Cr content accelerates formation of nickel aluminide. This is the reason for the observed difference in formation behavior of nickel aluminide in the 12 h milling time (see Table 1).

The highest peaks of elemental Al resulted by XRD analysis of the powder blend milled for 8 h is shown in Fig. 5 and can be used to study the structural variations of Al lattice during MA before generation of nickel aluminide phase. Similar to Fig. 4, there exist three peaks in Fig. 5: the lower peak is related to as received Al powder and other peaks show the XRD patterns of Al milled together with the Ni and Cr powder for 8 h. The middle and upper peaks are related to the Al in the powder blend including 0.25 and 2.25 at% Cr, respectively. These peaks are related to the samples in which there is not any nickel aluminide intermetallic yet. According to Fig. 5, both middle and upper peaks have been moved toward higher 2θ angles with respect to the lower peak (no milled peak). Unlike Ni peaks, the upper peaks of Al have been moved similar to the middle peak. This means that the Cr existed in the powder blend does not have any effect on the displacement of Al peaks. Since, solubility of Cr in Al lattice is low; the Cr cannot compensate the stored stress in Al lattice induced by the defects created during MA. Therefore, displacement of Al peaks is only due to severe plastic deformation and generation of many defects resulted from milling media.

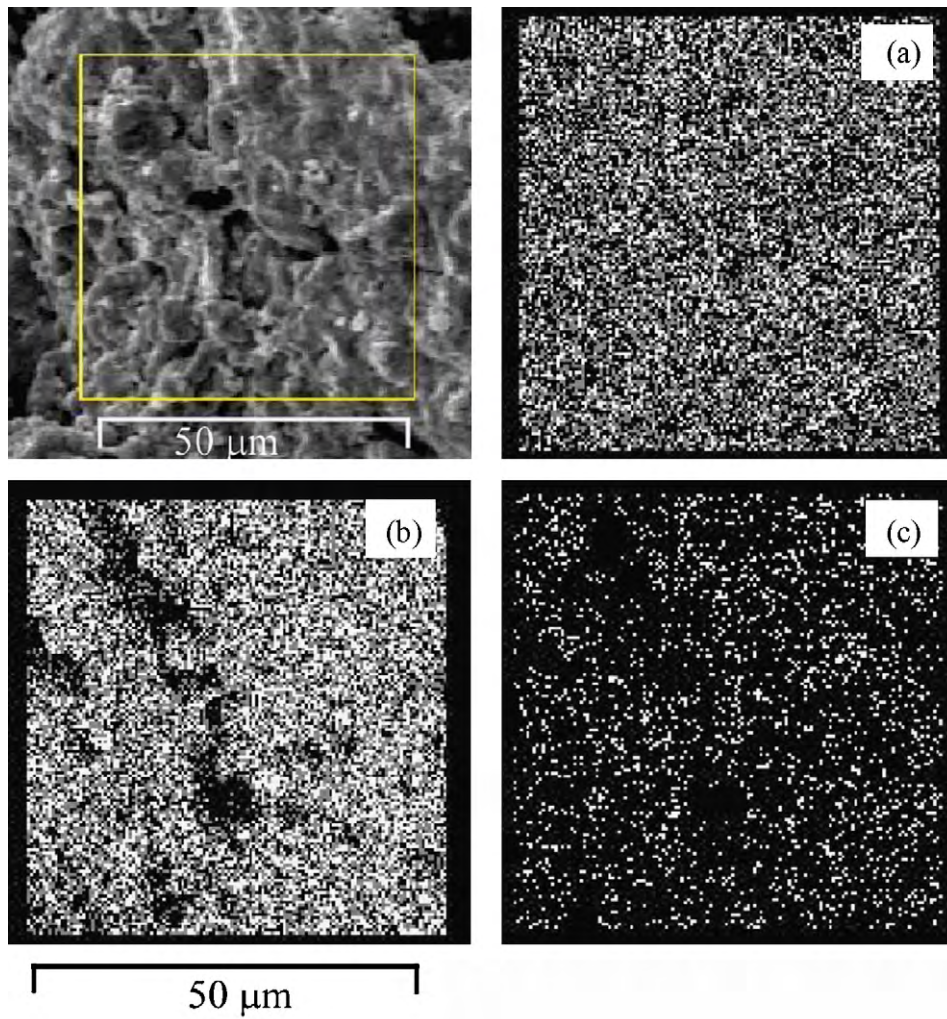


Fig. 3. EDS X-ray mapping of sample 1-8: (a) nickel; (b) aluminum and (c) oxygen.

Fig. 6 depicts the main XRD peaks of nickel aluminide phase synthesized in the presence of 0 and 0.25 at% Cr. The two upper peaks are related to the samples 1-16 and 2-16 that were milled until 16 h and the nickel aluminide compound completely formed in the Ar atmosphere before milling stops. Other peaks show the

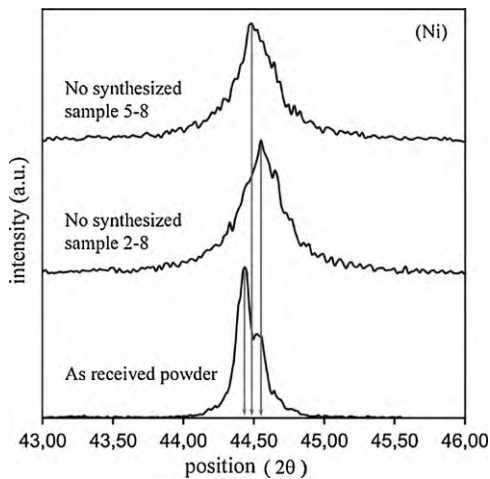


Fig. 4. Comparison between three most intense Ni peaks (the XRD pattern provided before generation of nickel aluminide phase in samples 2-8 and 5-8).

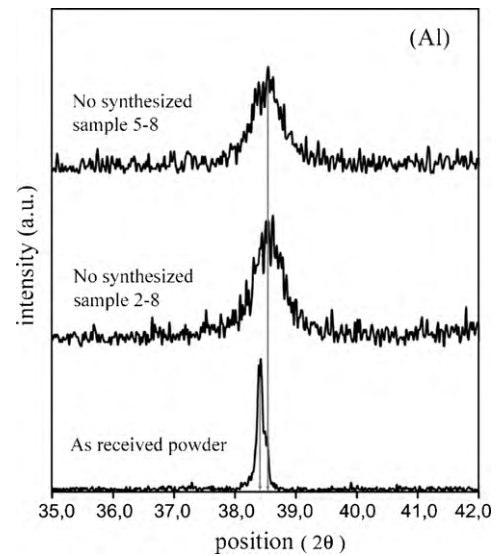


Fig. 5. Comparison between three most intense Al peaks (the XRD pattern provided before generation of nickel aluminide phase in samples 2-8 and 5-8).

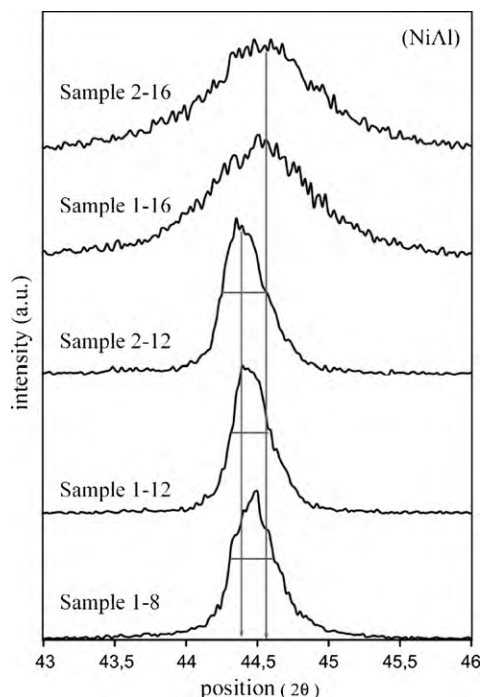


Fig. 6. Comparison between various most intense NiAl peaks formed on different milling times.

XRD pattern of nickel aluminide phase generated after milling stops in the air atmosphere (the samples 1-8, 1-12 and 2-12).

As it can be observed from Fig. 6:

1. The nickel aluminide peaks for the samples 1-16 and 2-16 have been appeared in similar angles. It means that when the generated nickel aluminide is milled under Ar, increasing Cr from 0 up to 0.25 at% does not have any effect on the peaks displacement.
2. The nickel aluminide peaks for the samples 1-8 and 1-12 have been appeared in similar angles. It means that the milling time of the powder blend does not have any effect on the diffraction behavior of nickel aluminide compound synthesized after milling stops (in the air atmosphere).
3. In relation to nickel aluminide phases generated in the samples 1-8 and 1-12, although the intensities and diffraction angles are similar to each other, they have been slightly displaced to lower angles with respect to the samples 1-16 and 2-16.
4. The main peak of nickel aluminide phase formed in the sample 2-12 is appeared in the lowest diffraction angles with respect to that one formed in the other samples.

The above observations can be explained as follows:

When the milling time is adequate, the nickel aluminide intermetallic is synthesized under protective gas (here is Ar) before milling stops. Continuing the milling process after generation of nickel aluminide causes an increase in the internal crystal stress in the nickel aluminide lattice. In other words, the nickel aluminide phases synthesized in the samples 1-16 and 2-16 have been milled after generation while those synthesized in the samples 1-8 and 1-12 do not have any milling experience after generation. Therefore, the main peak of nickel aluminide formed in the samples 1-16 and 2-16 is located in the higher angles with respect to that one formed in the samples 1-8 and 1-12.

In relation to the samples 1-8, 1-12 and 2-12 it can be expressed that the heat released during formation of nickel aluminide compound (in the air atmosphere) causes recovery and annealing of the milled particles, thus, the peaks of nickel aluminide phase gener-

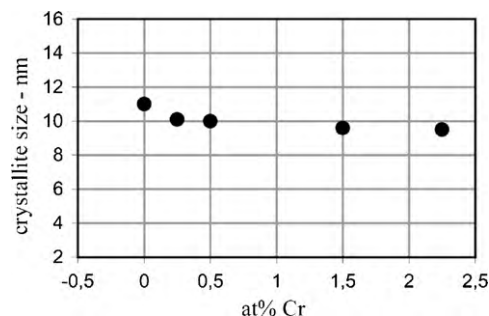


Fig. 7. Variation of crystallite size vs. atomic percent of micro-alloying Cr.

ated in the samples 1-8 and 1-12 are independent of the milling time. For the samples 1-12 and 2-12, it can be seen that the peak of nickel aluminide formed in the sample 2-12 appears in the lower angles, while it was already mentioned that increasing Cr from 0 at% up to 0.25 at% does not have any considerable effects on the peaks displacement (see the peaks of 1-16 and 2-16). It can be explained that the Cr increases the slip systems and decreases the APB energy. Therefore, the critical shear stress for dislocation generation would be decreased and as a result, the dislocation mobility is increased. Obviously, the new dislocations generated by the new activated slip systems affect the recovery and annealing process, i.e. Cr can influence the recovery and annealing process of nickel aluminide phase. Thus, when the nickel aluminide intermetallic is synthesized in the air atmosphere, increasing the Cr from 0 up to 0.25 at% has a considerable effect on its crystal lattice. The stress is released due to recovery and annealing, therefore, the peak of nickel aluminide generated in the sample 2-12 appears in the lower angle with respect to the sample 1-12.

Fig. 7 shows the crystallite size variation of nickel aluminide phase by increasing Cr content for a constant milling time. Apparently, increasing the micro-alloying Cr does not have any considerable effect on the crystallite size. Therefore, more broadening of the peak of 2-12 with respect to the peak of 1-12 (Fig. 6) is not caused by the more dislocation size. Activation of new slip systems followed by the more dislocation mobility, results in more micro strain in the nickel aluminide lattice generated in the sample 2-12. Therefore, it can be explained that the more broadening of the peak of 2-12, can be related to the more micro strain stored in the nickel aluminide phase formed in the sample 2-12.

4. Conclusion

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 Cr contents as a micro-alloying element for various milling times. The synthesis behavior of $Ni_{50}Al_{50-x}(x-Cr)$ phase was investigated and the following results were observed briefly:

1. Synthesis behavior of nickel aluminide intermetallic depends on the micro-alloying of Cr contents and milling time.
2. In an adequate milling time, the nickel aluminide phase is generated during MA under protective gas which here is Ar.
3. If the milling time is not enough, the nickel aluminide intermetallic is not generated during milling process. In this case, the milled powder blend is activated during MA and after air exposure the nickel aluminide phase is generated by a process similar to SHS (self-propagating high-temperature synthesis).
4. Longer milling time accelerates the nickel aluminide formation in the air atmosphere.

5. The samples synthesized in the air atmosphere include a considerable amount of oxygen (compared to those synthesized in the Ar atmosphere). Therefore the oxygen plays a considerable role in the formation of nickel aluminide phase.
6. Coincidence of Al and oxygen X-ray maps reveals that after opening the vial lid, the oxidation of milled aluminum particles results in the generation of the final product in the air atmosphere.
7. When the milling time is constant, increasing the Cr content accelerates the nickel aluminide formation in the air atmosphere.
8. The Cr compensates the defects created in the Ni lattice during MA.
9. When the nickel aluminide phase is formed during MA, the Cr content does not affect the peak displacement. Though, the peak of nickel aluminide generated in the air atmosphere is displacing by changing the Cr contents.
10. The milling time of the powder blend does not have any effect on the diffraction behavior of nickel aluminide compound synthesized in the air atmosphere.
11. The crystallite size of nickel aluminide does not vary considerably with Cr contents. Therefore, broadening of nickel aluminide peaks is caused by the micro-strain raised by activation of new slip systems followed by the more dislocation mobility which is a result of Cr content.

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References

- [1] C.K. Lin, S.S. Hong, V. Lee, *Intermetallics* 8 (2000) 1043–1048.
- [2] M. Krasnowski, A. Antolak, T. Kulik, *J. Alloys Compd.* 434–435 (2007) 344–347.
- [3] L.M. Peng, J.H. Wang, H. Li, M. Gong, *Mater. Sci. Eng., A* 425 (2006) 339–345.
- [4] L.D. Angelo, G. Gonzalez, J. Ochoa, *J. Alloys Compd.* 434–435 (2007) 348–353.
- [5] E. Arzt, P. Grahle, *Acta Metall. Mater.* 46 (1998) 2717–2727.
- [6] G. Sauthoff, *Intermetallics*, VCH publishers, New York, 1995.
- [7] D.B. Miracle, R. Darolia, NiAl and its alloys, in: J.H. Westbrook, R.L. Fleischer (Eds.), *Intermetallic Compounds*, John Wiley & Sons, New York, 2000.
- [8] L. Sheng, W. Zhang, J. Guo, F. Yang, *Intermetallics* 18 (2010) 740–744.
- [9] Y.C. Liang, J.T. Guo, Y. Xie, L.Y. Sheng, L.Z. Zhou, Z.Q. Hu, *Intermetallics* 18 (2010) 319–323.
- [10] L.Y. Sheng, J.T. Guo, L.Z. Zhou, H.Q. Ye, *Mater. Sci. Eng., A* 500 (2009) 238–243.
- [11] Y. Xie, J.T. Guo, Y.C. Liang, L.Z. Zhou, H.Q. Ye, *Intermetallics* 17 (2009) 400–403.
- [12] R. Darolia, R.D. Lahrman, D. Field, J.R. Dobbs, M. Chang, E.H. Goldman, D.G. Konitzer, Overview of NiAl alloys for high temperature structural applications, in: T.C. Liu, R.W. Cahn, G. Sauthoff (Eds.), *Ordered Intermetallics—Physical Metallurgy and Mechanical Behaviour*, Kluwer Academic Publishers, Dordrecht, 1992.
- [13] R.D. Noebe, A. Garg, *Scripta Metall.* 30 (1994) 815–820.
- [14] J. Jordar, S.K. Pabi, H.J. Fecht, B.S. Murty, *Philos. Mag. Lett.* 82 (2002) 469–475.
- [15] E. Bonetti, E.G. Campari, L. Pasquini, E. Sampaolesi, G. Scipione, *Nanostruct. Mater.* 12 (1999) 895–898.
- [16] G.K. Dey, J.A. Sekhar, *Trans. Indian Inst. Met.* 50 (1988) 79–89.
- [17] K.H. Hahn, K. Vedula, *Scripta Metall.* 23 (1989) 7–12.
- [18] D.F. Lahrman, R.D. Field, R. Darolia, In the effect of strain rate on the mechanical properties of single crystal NiAl, in: *High Temperature Ordered Intermetallics IV*, MRS Symp Proc, 1991, pp. 603–607.
- [19] T. Takasugi, S. Watanabe, S. Hanada, *Mater. Sci. Eng., A* 149 (1992) 183–193.
- [20] R. Darolia, R.D. Field, D.F. Lahrman, A.J. Freeman, in: S.H. Wang, C.T. Liu, D. Pope (Eds.), *High Temperature Aluminides and Intermetallics*, Metallurgical Society of American Institute of Mechanical Engineers, Warrendale, 1989.
- [21] R. Darolia, D.F. Lahrman, R.D. Field, *Scripta Metall. Mater.* 26 (1992) 1007–1012.
- [22] C. Jiang, *Acta Mater.* 55 (2007) 4799–4806.
- [23] J.S. Benjamin, D. Farkas, *Intermetallics* 12 (2004) 937–943.
- [24] C. Jiang, D.J. Sordelet, B. Gleeson, *Scripta Mater.* 54 (2006) 405–410.
- [25] W.H. Tian, C.S. Han, M. Nemoto, *Intermetallics* 7 (1999) 59–67.
- [26] C. Suryanarayana, *Mechanical Alloying and Milling*, Marcel Dekker, New York, 2004.
- [27] C. Suryanarayana, M. Grant Norton, *X-Ray Diffraction: A Practical Approach*, Plenum Press, New York, 1998.