



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

Mostafa Alizadeh^{a,*}, Ghaffar Mohammadi^a, Gholam-Hosein Akbari Fakhrebadi^b, Morteza Mirzaei Aliabadi^a

^a Department of Materials Science and Engineering, International Center for Science, High Technology & Environmental Sciences, Kerman, Iran

^b Department of Materials Science and Engineering, Shahid Bahonar University, Kerman, Iran

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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

* Corresponding author. Tel.: +98 342 6226611; fax: +98 342 6226617.

E-mail addresses: alizadeh@icst.ac.ir, mostafa.alizadeh56@yahoo.com (M. Alizadeh).

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

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