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Obtaining NiAl intermetallic compound using different milling devices

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ABSTRACT

NiAl intermetallic compound was synthesized by mechanical alloying technique in planetary and attritor mills. The starting powders consisted of elemental mixtures of Ni and Al at $Ni_{50}Al_{50}$ (at%) composition. In the planetary mill, compound formation occurred gradually during mechanical alloying, while the occurrence of a mechanically induced self-propagating reaction (MSR) can be suggested in the attritor mill. The NiAl obtained in both mill types was partially disordered with long-range order parameter not inferior to 0.66. Quantitative phase analysis using the Rietveld method was performed in as-milled samples, and this method was also employed to estimate changes in crystallite size and lattice strain of the NiAl produced during mechanical alloying.

1. Introduction

Nanocrystalline intermetallic compounds are said to have enhanced ductility and yield strength as compared to conventional grain-sized materials [1], and mechanical alloying in ball mills has been considered a suitable processing method capable of attaining the goal of producing nanostructured materials [2]. Not only is mechanical alloying capable of reducing grain size but it can also lead to the following changes in the material: disordering of the lattice and modification of the crystalline structure of crystals into a more symmetric, for example, cubic shape [2].

It is suggested that the work by Ivanov et al. [3] is the first report on the synthesis of nickel aluminides using mechanical alloying technique. These authors processed the compositions Ni_xAl_{100-x} ($32 < x < 90$), and milling runs were conducted in a laboratory ball mill with a ball-to-powder ratio of 10:1, and the milling media was made of hardened steel. Depending on the initial composition, the following phases were obtained: NiAl, Ni_2Al_3 and metastable solid solutions of Al in Ni. Also, the presence of amorphous NiAl was reported in the range of 65–73% (at%) of nickel.

The obtaining of NiAl intermetallic compound in attritor mill was described by Nash et al. [4] using an elemental powder mixture of $Ni_{49.5}Al_{49.5}W_1$. Afterwards, Nash and co-works consolidated and characterized powders based on NiAl [5–13] and also composite powders of NiAl reinforced with AlN and/or Al_2O_3 [14–16]. The research was conducted in a three-shaft attritor mill, employing elemental powder mixtures, and the ball-to-powder ratio was changed from 15:1 to 40:1; also, in some cases, cryogenic milling was employed at the beginning of the process, and milling times as long as 70 h were utilized.

An extensive research on synthesis of Ni–Al system intermetallics was conducted by Murty and co-workers [17–24]. Mechanical alloying of Ni and Al powder elemental mixtures in the proportions of Ni_xAl_{100-x} ($x = 10, 18, 21, 25, 40, 50, 65, 68, 70, 75$ and 90) was performed in a planetary mill. Single phase NiAl was found as a product after mechanical alloying of $Ni_{40}Al_{60}$ and $Ni_{50}Al_{50}$ (at%) compositions. Phase field extension of NiAl intermetallic compound was also observed; in the mechanically alloyed material, NiAl was present in the range from 25% to 65% of Ni (at%), whereas the equilibrium range goes from 46% to 59% (at%) of Ni [18]. In addition, the authors reported that contamination with iron from the milling media can promote intermetallic disordering, which would improve material ductility [20].

During mechanical alloying and depending on the milling parameters employed, NiAl intermetallic compound can take place either gradually or through a mechanically induced self-propagating reaction (MSR), which was first documented by Atzmon [25]. In fact, the time necessary for the gradual formation of NiAl and the occurrence or not of an MSR is strongly affected by process variables such as type of mill, ball-to-powder ratio, the use of process control agent (PCA) and the milling media.

The type of mill employed in the mechanical alloying process accounts for different milling mechanisms, that is, the way in which the available energy is transferred from de milling media to the material.

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The planetary ball mill has one or more milling vials arranged on a rotating plate; the vials and the plate rotate around their own axes. Since vial and plate rotate in opposite directions, the milling balls can either slide on the inner vial walls transferring energy to the milling material through friction events or travel inside the vial and colliding against the vial wall; in the latter, the energy is transferred to the material through impact events [2].

The attritor mill consists of a vertical vial with one or more impellers (shafts) inside it, and as these impellers rotate, they account for energizing the ball charge so that the mechanical alloying process can take place [2]. Likewise, the planetary mill, the milling process in the attritor mill occurs by friction and collision events; however, the collision events take place only in some regions of the vial, that is, only some portion of the powder is subjected to collision events. Consequently, only a small fraction of the powder suffers collision events, which are mainly responsible for the mechanical alloying process [26,27]. Table 1 summarizes the milling mechanisms of planetary and attritor mills.

There are several reports on the synthesis of NiAl intermetallic compound using mechanical alloying technique in different mill types. Nevertheless, descriptions of NiAl formation in different mill types in the same report were not found in the literature. In this work, NiAl intermetallic compound was synthesized in two different types of milling devices: attritor mill and planetary mill.

2. Experimental

Ni and Al powders, with a purity level of 99.9% and 99.8%, respectively, were mixed in the proportion $Ni_{50}Al_{50}$ (at%). Powder mixtures were processed under argon atmosphere using planetary (Fritsch Pulverisette P6) and attritor (Union Process Attritor 01 HD) mills. Mechanical alloying was performed for several times (4, 8, 12, 18, and 24 h) in both mill types, and stearic acid was employed as process control agent (PCA).

In the planetary mill, a 10:1 ball-to-powder ratio, and a stainless steel milling media were employed; 500 g of balls with 10 mm of diameter were used in a 500 mL grinding bowl so that 50 g of powder were processed in each milling run. Two different amounts of PCA were employed: 0.75%, and 1.00% (wt%).

On the other hand, millings in the attritor mill were conducted using a ball-to-powder ratio of 20:1, chromium steel balls, and a 750 mL stainless steel vial coated with ethylene–tetrafluoroethylene polymer. The milling balls were, in fact, a mixture of balls with diameters of 6.35 and 4.76 mm at a number fraction of 0.5 [27]. A stainless steel shaft with arms manufactured in nickel-based alloy was employed. An amount of 0.75% of PCA was used in all attritor milling runs. A summary of the experimental conditions employed in each mill type is shown in Table 2.

The vial temperature in both mills was monitored during milling runs; in the planetary mill, the temperature data was recorded using a Fritsch gas pressure and temperature measuring system (GTM), which allows physical and chemical reactions to be monitored *in situ* in the grinding vial. However, in the attritor mill, vial temperature was indirectly estimated by measuring the water inlet and outlet temperatures of the mill cooling jacket with the use of two K-type thermocouples, and a DO 9416 Delta Ohm acquisition data system.

Table 1
Main milling mechanisms of the planetary and attritor mills [26,27].

Milling device	Milling mechanism
Planetary	Friction and collision events
Attritor	Friction and collision events, but only a small portion of the powder is subjected to the latter mechanism

Table 2
Experimental conditions used in both milling devices.

Characteristic	Attritor mill	Planetary mill
Mill type	Union Process, Attritor 01HD	Fritsch P6
Vial capacity	750 mL	500 mL
Vial material	Polymer-coated stainless steel	Martensitic stainless steel
Shaft material	Stainless steel with Colmonoy arms	–
Material of the balls	SAE 52100 steel	Martensitic stainless steel
Diameter of the balls	6.35 mm + 4.76 mm, number fraction of 0.5	10 mm
Ball mass per milling run	2000 g	500 g
Ball-to-powder ratio (BPR)	20:1	10:1
Sample mass	100 g	50 g
Rotation	500 rpm (shaft)	300 rpm (plate)
Process control agent (PCA)	Stearic acid	Stearic acid
Amount of PCA (wt%)	0.75%	0.75% and 1.00%
Milling atmosphere	Argon (dynamic)	Argon

NiAl intermetallic compound formation was detected by X-ray diffraction (XRD) analysis using a Philips MPD 1880 diffractometer with CuKα radiation. Qualitative phase analysis was performed using the Powder Diffraction File (PDF) database, and the software PANanalytical X'Pert HighScore Plus, release 2.2a (2.2.1). The same software was employed to perform the Rietveld analysis of XRD patterns; quantitative phase analyses, estimation of crystallite size (L) and lattice strain (ε) were performed using the Rietveld method [28]. Yttrium oxide (Y_2O_3) was used as a standard sample to estimate the instrumental broadening for crystallite size and lattice strain calculation. The long-range order parameter (S) of NiAl was also estimated from the XRD profiles using the following expression [21]:

$$S = \sqrt{\frac{(I_{100}/I_{110})_{obs}}{(I_{100}/I_{110})_{std}}} \tag{1}$$

where $(I_{100}/I_{110})_{obs}$ and $(I_{100}/I_{110})_{std}$ are, respectively, the ratio of 100 superlattice and 110 fundamental reflections of the sample, and that of the standard Powder Diffraction File database. Moreover, contamination of as-milled powders was estimated using X-ray fluorescence (Philips PW 2404).

3. Results and discussion

XRD patterns of samples mechanically alloyed in the planetary mill for different times, and two amounts of PCA are shown in Fig. 1. The qualitative analysis of these patterns demonstrates that superlattice reflection of NiAl (at 2θ about 31°) is already visible after 12 h of milling in samples with 0.75% of PCA. On the other hand, this reflection can be seen only after 18 h of milling in samples with 1.00% of PCA; as a result, until 12 h of milling only Ni and Al reflections are observed in these samples. Also, the presence of Ni or Al reflections is verified until 24 h of milling for both PCA contents, indicating that some powder fraction was not alloyed and phase formation did not occur in the entire sample. A detailed observation of Fig. 1(a) and (b) also indicates that for 1.00% PCA samples longer milling times are necessary for alloy formation.

It has been known that when powder mixtures of Ni and Al are mechanically alloyed, NiAl intermetallic compound formation occurs either gradually along milling time [18], or suddenly through

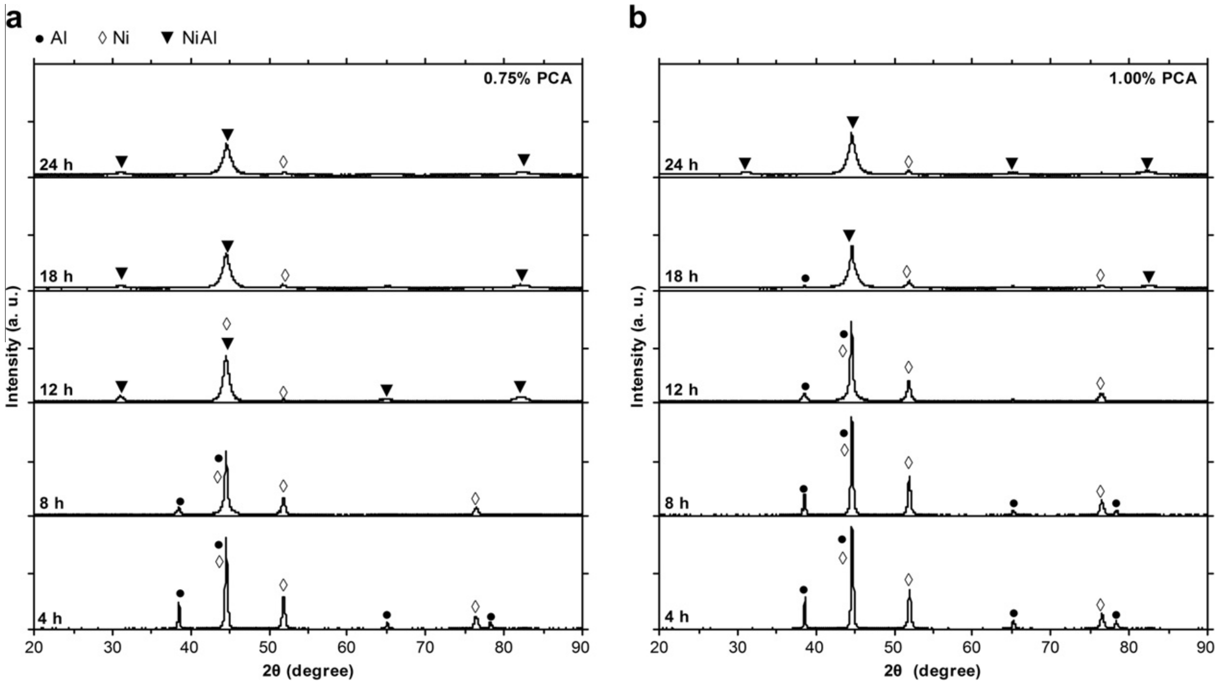

Fig. 1. XRD patterns showing the formation of NiAl intermetallic compound during MA in $Ni_{50}Al_{50}$ powder mixtures in the planetary mill. (a) 0.75% PCA; (b) 1.00% PCA.

a mechanically induced self-propagating reaction (MSR) [29,30], which occurs after a certain time of milling called ignition time [31]. The occurrence or not of such an exothermic reaction can be easily detected by recording the temperature of the milling vial [31]. In samples processed in the planetary mill, the curves of the vial temperature as a function of milling time (Fig. 2) show no sudden temperature increase, and this evidences that intermetallic compound formation took place gradually under those mechanical alloying conditions. In fact, Fig. 2(a) and (b) show gradual temperature rises starting at about 4 h of milling, and, in this case, they can be due to the alloying process, but cannot be associated to an MSR. An MSR is characterized to be finished just a few seconds after its ignition [32].

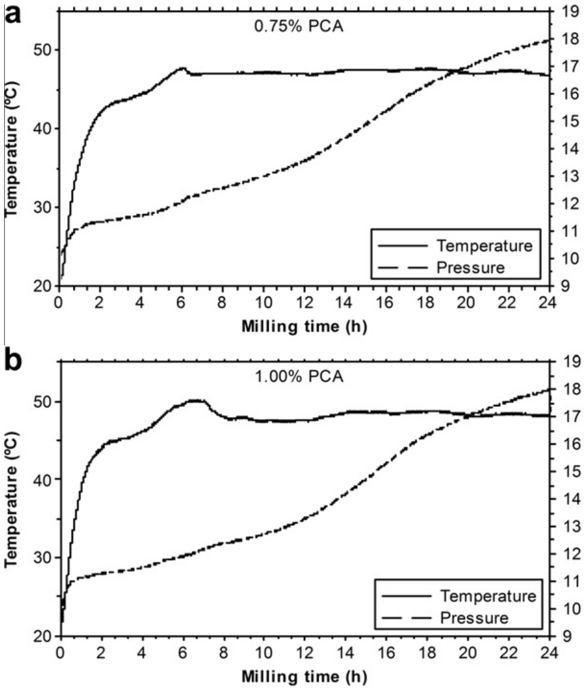

Fig. 2. Temperature and internal pressure of milling vial as a function of milling time for $Ni_{50}Al_{50}$ powder mixture processed in the planetary mill. (a) 0.75% PCA; (b) 1.00% PCA.

Furthermore, the internal pressure of the vial as a function of milling time (also in Fig. 2) indicates no abrupt pressure increase either, which would be expected if a MSR had taken place in this system.

Samples processed in the attritor mill also show NiAl intermetallic formation after 8 h of milling (Fig. 3(a)). The curve of vial temperature as a function of milling time showed an abrupt temperature increase of about 1 °C at approximately 5 h of milling and, in this case, the temperature change suggests the occurrence of an MSR (Fig. 3). It must be highlighted that the method employed for temperature measurements is an indirect one and this may explain the noise in the curve. Possibly, this also explains the relatively long duration of the temperature peak.

Although the temperature increase detected in the measuring system is relatively low (about 1 °C), this temperature peak can be suggested as MSR. This affirmation is possible and plausible since the vial of the attritor mill has a cooling jacket in order to avoid the superheating of the mill, and to prevent the occurrence of damage in the vial and milling shaft. This cooling system has a great mass and volume as compared to the powder that is processed; also, there is a continuous water flow in the cooling jacket. Moreover, the temperature increase of 1 °C was measured in the water outlet of the cooling jacket. Thus, the occurrence of a temperature increase of 1 °C, which was measured in the water outlet, means that a much greater temperature increase happened inside the milling vial during mechanical alloying. Finally, the occurrence of MSR in the attritor mil can be suggested based upon these facts added to the appearance of NiAl reflections in samples mechanically alloyed for 8 h.

In addition, for longer milling times, the XRD patterns show only a change in reflections area and intensity. An important point that can be highlighted is the difference between the two mechanisms of alloying formation in the planetary and attritor mills. In

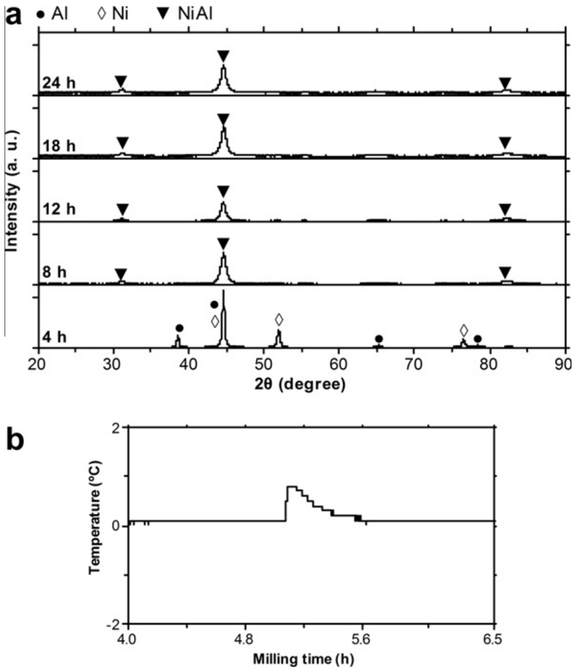

Fig. 3. (a) XRD patterns showing the formation of NiAl intermetallic compound during MA in $Ni_{50}Al_{50}$ powder mixtures in the attritor mill. (b) Vial temperature as a function of milling time.

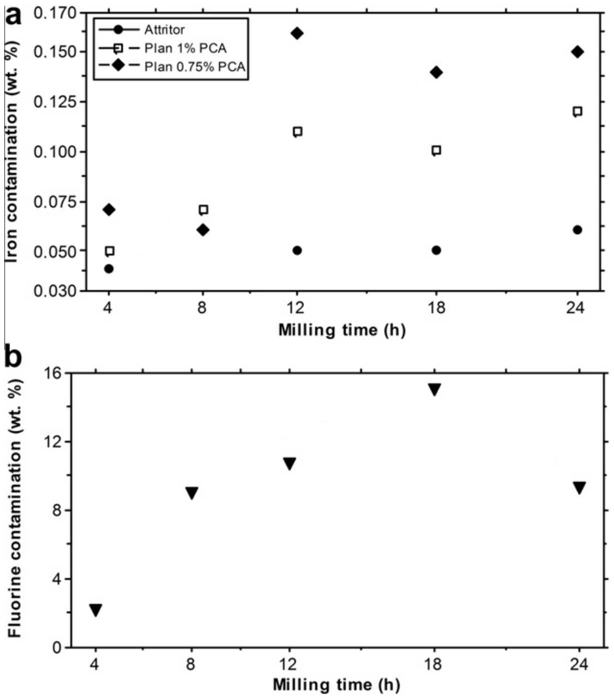

Fig. 4. (a) Iron contamination in samples mechanically alloyed in planetary and attritor mills. (b) Fluorine contamination in samples mechanically alloyed in the attritor mill.

the former, the results suggested that alloying formation occurred in a gradual manner; on the other hand, in the attritor mill, the temperature elevation suggested that NiAl intermetallic formation took place through a MSR.

Although the ball-to-powder ratios used in the planetary and the attritor mill are different, the comparison between the results of the two milling devices is significant since the ball-to-powder ratio is not a very useful variable to compare results from different milling devices. The action of ball-to-powder ratio during the milling process is strongly dependent on the size of milling balls and constructive details of the mill used. Also, the ball-to-powder ratio is dependent on the milling capacity; higher ball-to-powder ratios are used in high-capacity milling devices [2]. Furthermore, the ball-to-powder ratio is directly proportional to the energy provided by the milling device to the processing powder [33,34], and this energy would be a much more useful parameter for comparing different milling devices. Equations for predicting the milling energy of a planetary mill are found in the literature [33,35]. However, for the attritor mill the equations found in literature lead to figures that are not in agreement with experimental results [36]. In addition, depending on the type of mill that is used, prediction of the milling energy may involve complex computational modeling and calculus, which are beyond the scope of the present work.

Materials processed by mechanical alloying technique may present a high level of contamination due to the wear of the milling media. The level of iron contamination in the as-milled powders was estimated using X-ray fluorescence; also, the level of fluorine contamination was estimated in materials that were mechanically alloyed in the attritor mill. Fig. 4(a) shows that the iron contamination is less than 0.17% (wt%) for all milling conditions, and, for the attritor mill, the amount of iron detected is about 0.05%. These contamination levels are relatively low as compared to other reports from the literature, in which iron contamination as high as 18% (at%) is found [20,23]. In addition, it is suggested that iron contamination from the milling media increases the intermetallic compound ductility due to the fact that the presence of iron would favor the formation of a disordered intermetallic compound [20], and, according to the authors of that study, the disordering of the intermetallic lattice enhances the material ductility after its consolidation. On the other hand, an elevated amount of fluorine can be verified in attritor milled samples (Fig. 4(b)), and the presence of such contaminant is due to the vial coating. Unlike iron contamination, no report relating the presence of fluorine to the mechanical properties of NiAl was found in the literature.

Phase quantification during mechanical alloying was performed for both milling devices, and is shown in Fig. 5. As expected, NiAl intermetallic compound formation occurs at longer milling times in high PCA content samples. As can be clearly seen in Fig. 5, after 12 h of mechanical alloying in the planetary mill, the results indicated no NiAl in samples with 1.00% of PCA; on the other hand, for the other condition, 80% of NiAl is present in the as-milled powder. This delay in alloying formation due to a higher PCA content is in agreement with other reports found in the literature. For example, the use of PCA delays the formation of an amorphous phase in the Ti–Al system [37], and according to these authors, the presence of PCA changes the mechanism of amorphization; nevertheless, the authors presented no reasons to explain this fact. Shaw et al. [38] observed that the presence of PCA also delays the formation of $Al_{93}Fe_3Ti_2Cr_2$ alloy, when elemental mixtures of theses metals are mechanically alloyed in a Spex mill. The alloying formation was followed measuring the decrease in the aluminum lattice parameter, and the presence of PCA diminished the rate of lattice parameter reduction, and, consequently, delayed the alloy formation process. The authors suggested that the PCA adsorption on metal surface may prevent the dissolution process which is necessary for alloying formation, and, in this case, for the reduction of the aluminum lattice parameter. Also, the refinement of Fe, Ti and Cr in the Al matrix is related to the level of plastic deformation in the matrix and the dissolution rate of these elements inside the matrix; the level of plastic deformation is decreased due to the lubricating function of the PCA, while the PCA adsorption on metal surface diminishes the dissolution rate. Rocha et al. [39] verified that alloy formation occurred at longer mechanical alloying times

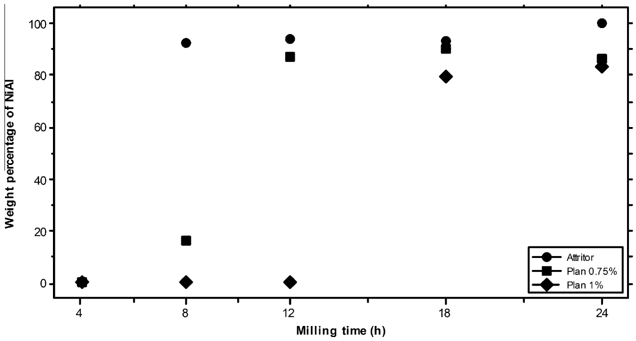

Fig. 5. Comparative quantitative phase analysis for attritor and planetary mills. Errors in the measurements are, respectively, 0.5%, 4.2%, and 3.0% for Attritor, Plan 0.75% and Plan 1%.

when wax was used as PCA; the authors considered the welding events during mechanical alloying process as an important factor in alloy formation kinetics, and, as a result, the presence of wax impeded such events, delaying the mechanical alloying process.

In the attritor mill, as described previously, at about 5 h of milling, it is suggested that the MSR of NiAl formation takes place and the occurrence of this exothermic reaction explains the variation of the percentage of the phase. Also, after 8 h of milling, the presence of more than 80% of NiAl intermetallic compound was verified in the as-milled powder (Fig. 5). In addition, although the chemical analysis of attritor milled samples detected fluorine, no reflections of fluorine-based compounds were found in XRD patterns (Fig. 3(a)).

Finally, a comparative of the quantitative phase analysis performed in both milling types is also shown in Fig. 5. As can be seen in this figure, a higher amount of NiAl (80%) is verified in attritor milled samples after 8 h, and this is due to the MSR that promoted intermetallic formation in this milling device. In the planetary mill, compound formation occurred gradually during the milling process, and longer milling times were necessary to produce 80% of NiAl.

The long-range order parameter (*S*) values calculated for NiAl obtained in the mechanically alloyed samples indicated a partially ordered intermetallic compound because *S* values of about 0.7–0.8 were estimated (Table 3). The *S* value may vary from 0 (completely disordered) to 1 (completely ordered) [40]. In XRD patterns, the presence of superlattice reflections indicates ordering in the lattice; for NiAl structure (B2 type), in the superlattice reflections the sum $(h + k + l)$ is odd, and on the other hand, this sum is even for fundamental reflections [2]. Therefore, (1 0 0) and (1 1 0) are, respectively, superlattice and fundamental reflections for the NiAl intermetallic compound. There are several reports in the literature about the disordering of intermetallics during the mechanical alloying process, and the review by Suryanarayana [2] provides detailed information about the subject.

Table 3
Long-range order parameter (*S*) of NiAl intermetallic compound.

Milling condition	Milling time (h)	*S*
Attritor	8	0.77
	12	0.81
	18	0.76
	24	0.76
Planetary 0.75% PCA	12	0.75
	18	0.66
	24	0.71
Planetary 1.00% PCA	24	0.78

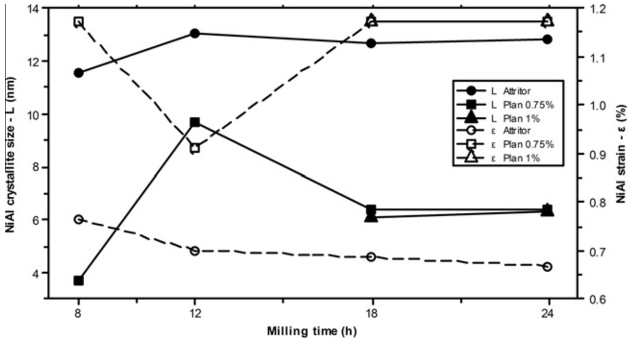

Fig. 6. Comparison of crystallite size and lattice strain of NiAl obtained in planetary and attritor mills. The curves used to connect the experimental points are just a guide for the reader to follow the evolution of the crystallite size and lattice strain. Typical errors in the measurements are ± 1.2 and ± 0.1 for crystallite size and lattice strain, respectively.

A comparison of crystallite size (*L*) and lattice strain (ε) of NiAl obtained on planetary and attritor mills is shown in Fig. 6. This figure shows a smaller lattice strain and a greater crystallite size in NiAl compound formed in the attritor mill in comparison to the compound produced in the planetary mill. The milling mechanisms are suggested as an explanation to this difference on lattice strain values; in the planetary mill, the milling mechanism is based mainly on friction forces and collisions among milling balls and between balls and vial wall; on the other hand, in the attritor mill, the largest powder amount occurs under friction (attrition) forces and only a small powder fraction occurs under collision events [26,27]. Also, in samples mechanically alloyed in the planetary mill, the crystallite size became practically stable after 18 h of milling.

As expected, there is a decrease in crystallite size and an increase in lattice strain as mechanical alloying is conducted for longer milling times. For the planetary mill, the lattice strain of reactants diminishes as the PCA content is increased. On the other hand, a higher PCA content leads to an increase of crystallite size of reactants. Consequently, when higher PCA contents are used during mechanical alloying, there is the need of providing higher energy levels to allow powder particles to achieve a certain level of crystallite refinement and lattice strain. Also, these results are in agreement with a previous report [38]; these authors concluded that the presence of PCA diminishes the occurrence of excessive cold welding, the crystallite size refinement and the increase of lattice strain. The lubricating function of PCA reduces the level of plastic deformation during ball impacts; as a result, there is a reduction in crystallite refinement and formation of solid solutions. On the other hand, differences in crystallite size and lattice strain of NiAl were not significant when the PCA content was changed.

4. Conclusions

NiAl intermetallic formation can be suggested to occur through an MSR in the attritor mill; on the other hand, alloy formation gradually took place in samples mechanically alloyed in the planetary mill.

The increasing of PCA content delayed the process of alloy formation in milling runs conducted in the planetary mill. Moreover, a greater amount of PCA promoted smaller lattice strain and less crystallite refinement.In all milling conditions, the NiAl compound obtained was partially disordered (long-range order parameter over 0.66).

The quantitative phase analysis showed a maximum of 80% of NiAl in the planetary mill while 100% was obtained in the attritor mill after mechanical alloying for 24 h.

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