

Evaluation of Characteristics of Interfacial Phases Produced in Al/Ni₃Al Composite during Manufacturing

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ABSTRACT

Metal matrix composites (MMCs) are currently being investigated because of their superior properties. The properties are mainly attributed to the efficiency of the load transfer from the matrix to the reinforcements through the matrix-reinforcement interface. The aim of this study is to investigate the effect of manufacturing parameters on the microstructure and morphology of the interface and the aluminide phases formed at the matrix-reinforcement interfaces. The parameters are: milling time to fabricate Ni₃Al, method of mixing of Ni₃Al and Al powders, compaction pressure and sintering temperature. The composite studied in this research was Al/5 Vol% Ni₃Al made from two different types of Ni₃Al powders. The results showed that compacting and sintering at higher levels lead to the transformation of Ni₃Al particles to thin layers of Al₃Ni. It was also shown that the prolonged milling time to produce Ni₃Al reinforcements and the prolonged ball milling procedure for mixing the powders, both, promote the diffusion process at reinforcement/matrix interface.

Keywords: Metal-Matrix Composites (MMCs), Interface, X-Ray Diffraction (XRD), Powder Processing

1. Introduction

Aluminum-matrix composites (AMC) reinforced with intermetallic compounds have been proposed as substitutes for ceramic reinforced composites due to their adequate mechanical properties. The key challenge in traditional metal-matrix composite production is to obtain a good reinforcement-matrix interfacial bonding. Ceramic reinforcements are normally not well compatible with the metal matrix, which results in poor reinforcement-matrix interfacial bonding. Such interfaces reduce mechanical properties and increase corrosion sensitivity. The new class of reinforcements namely intermetallic compounds, with their extremely good mechanical properties offer new solutions to this problem.

There have been some studies on the properties of aluminum-matrix composites reinforced with intermetallic particles in the binary systems of Fe-Al [1], Ti-Al [2] and Ni-Al [3-7]. They showed Ni₃Al is one of the best known and characterized reinforcements amongst the intermetallics.

In composites, blending or mixing of reinforcement

and matrix powders is just as important. because it controls the final distribution of reinforcement particles and porosity in green compacts. This, in turn, strongly affects the mechanical properties of the composites. Segregation and clustering are the common problems associated with the present state-of-the-art blending or mixing methods. The segregation and clustering during blending can be overcome by a technique developed during the 1960s called mechanical alloying [8-10]. Mechanical alloying is a dry, high-energy ball-milling process for producing composite powders with a fine controlled microstructure [11].

Ball milling causes a uniform distribution of particles and metal powders with a satisfactory microstructure after compaction [12]. The distribution of reinforcements affects the compressibility of the composite powder. It has been reported that when mechanical milling is applied on aluminum powder, powder compressibility is reduced and the green density decreases with increasing milling time [13,14].

It is difficult to manufacture highly reinforced com-

posites because the green density decreases with increasing fraction of reinforcement particles. This often leads to insufficient strength for supporting subsequent processes such as sintering and machining. In other words, mechanically alloyed Al powder containing a fine and homogeneous distribution of submicroscopic intermetallics is very hard and, therefore, its compressibility is low. Also, surface oxide films and hardness make milled Al powders very difficult to sinter. On the other hand, the properties of composites are very sensitive to sintering temperature because the reinforcement-matrix interface changes by changing the sintering temperature [15-17]. The reinforcement-matrix interface has a strong influence on the mechanical properties of any composite.

2. Experimental Procedure

Aluminum-matrix composites were produced using pure aluminum as metal matrix and 5 Vol% Ni₃Al particles as reinforcement. Elemental Al powder (Merck-1056-99%, <160 μm, flaky morphology) and Ni (Merck-112277-99.5%, <10 μm, spherical morphology) were used as raw materials. The reinforcement particles (Ni₃Al) were produced under two different milling conditions of pure Ni and Al as shown in **Table 1**. As the table indicates, all parameters for production of the two types of intermetallic compounds are the same except for the milling time.

In order to evaluate the effect of mixing conditions on the properties of composites, two types of mixing processes such as blending and high energy ball milling (planetary ball mill) were applied. The blending process was carried out in a tumbling mixer for 30 minutes. The ball milling of Al and Ni₃Al powders was conducted using a planetary apparatus (model FP2) to produce composite powder particles. The powders and hardened 20 mm diameter steel balls were sealed in a hardened stainless steel vial at room temperature. The ball-to-powder weight ratio and rotational speed were 20:1 and 300 rpm, respectively. To prevent excessive welding of particles in the vial, 1 wt% stearic acid was added to the system. To avoid oxidation, the entire process was performed in argon atmosphere. After 12 hours of milling, the powders were analyzed for morphology and structure.

Table 1. Milling condition parameters for producing Ni₃Al powders.

Speed of milling	550 rpm
Milling time	15 and 55 h
Weight ball/powder ratio	20:1
Ball diameter	20 mm
Ball material	stainless steel
Process control agent	Stearic acid (1 wt%)
Atmosphere	Argon

The blended and milled powders were cold compacted uniaxially in a floating die at two compacting pressures of 400 and 800 MPa. After ejecting the specimens from the die, their densities were measured by volumetric method. Weight and dimensions of the compacts were measured by an accurate balance (± 0.1 mg) and a micrometer (± 0.1 mm). The error of measurement was found to be less than 1%.

The compacted test specimens were sintered at three temperatures of 580°C, 620°C and 650°C in a vacuum furnace for 30 minutes followed by furnace cooling. Densities of the sintered parts were determined according to Archimedes principle (DIN ISO 3369).

Microstructure observations were made by an optical microscope and a scanning electron microscope model LEO 440 i. In addition, size distribution of powder particles was evaluated by Clemex Image Analyzer. In order to study the distribution of elements within the interfacial layers, chemical composition was measured by the energy dispersive X-ray (EDX) method. The interfacial phases under different manufacturing conditions were characterized by Bruker; Advance-D8 X-ray diffractometer.

Vickers macro-hardness tests were performed on the sectioned and polished specimens. The values reported are the average of at least 10 indentations by applying 30 kg load for 10 s.

3. Results and Discussion

3.1. Production of Particulate Reinforcement

Diffusion of aluminum atoms into the nickel system during ball milling process resulted in the formation of Ni(Al) solid solution and then Ni₃Al compound as a final product after 15 hours of milling. X-ray diffraction pattern of the milled powders is shown in **Figure 1**. The point analysis of chemical composition of powders using energy dispersive X-ray (EDX) method at random sites indicated a 75:25 atomic percentage ratio of Ni to Al, which corresponds to Ni₃Al compound. It has been claimed that the same composition has been produced after

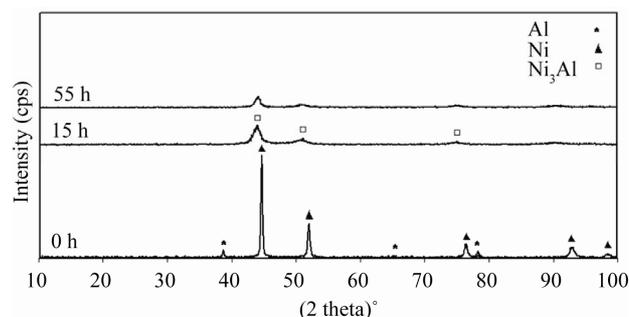


Figure 1. X-ray diffraction of milled powders.

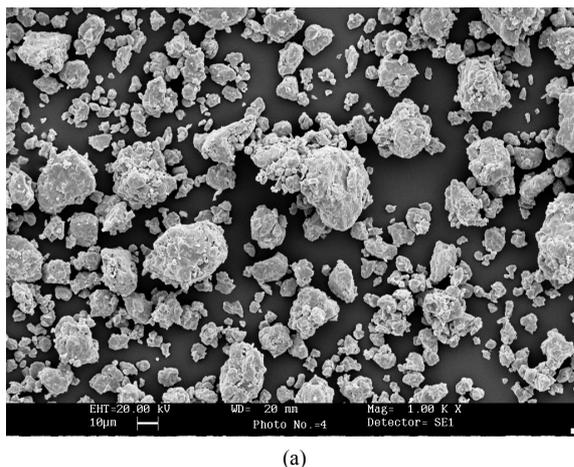
prolonged ball milling treatment (up to 55 hours) [18].

XRD peak broadening represents the variation in the crystallite size of nanostructure Ni₃Al and accumulation of lattice strain with increasing time of milling. The XRD peaks were analyzed by Williamson-Hall method. The size and lattice strain of 15-hour milled powders were 4.81 nm and 0.005%, respectively. While the values of these parameters reach 19.19 nm and 0.88% at 55 hours of ball milling time.

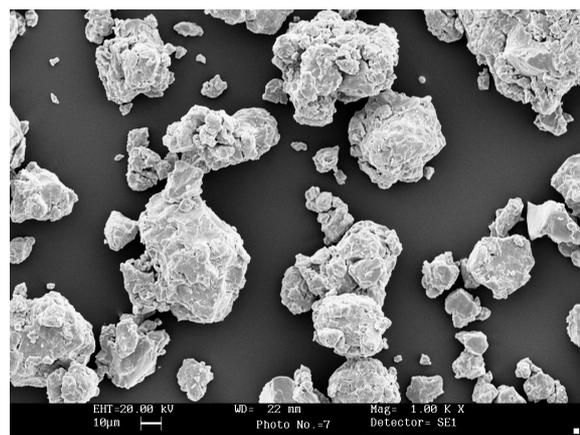
Figure 2 shows morphology of the two different types of Ni₃Al produced after 15 and 55 hours of milling. The particle size distribution of the powders is illustrated in **Figure 3**. It is observed that the size of 55-hour milled powder is greater than that of the 15-hour milled powder. During initial milling times, sequences of welding and fracturing are perceived. After passing 15 hours of milling process, welding dominates fracturing. Cold welding between powder particles occurs during long milling time (55 hours) which causes increased average particle

size. Image analysis indicated that spherical diameter of Ni₃Al powders after 15 and 55 hours of milling were 6.7 and 10.9 μm and their aspect ratio were 1.96 and 2.61, respectively.

Since Ni₃Al particles are brittle, it is expected that during milling fracture should predominate over cold welding leading to particle refinement instead of particle coarsening. However, as mentioned earlier, it was observed that the particle size of 55-hour milled powder is larger than 15-hour milled powder. Therefore, in the case of Ni₃Al particles the results showed to be the opposite. This is due to the fact that mechanical milling causes formation of nanocrystalline powder particles which results in grain boundary sliding or migration during milling. As a result, nanocrystalline structure develops ductility in Ni₃Al compound and encourages cold welding. Such line of reasoning has been postulated by Kumaran *et al.* [19] for coarsening of TiAl powders during high milling process.

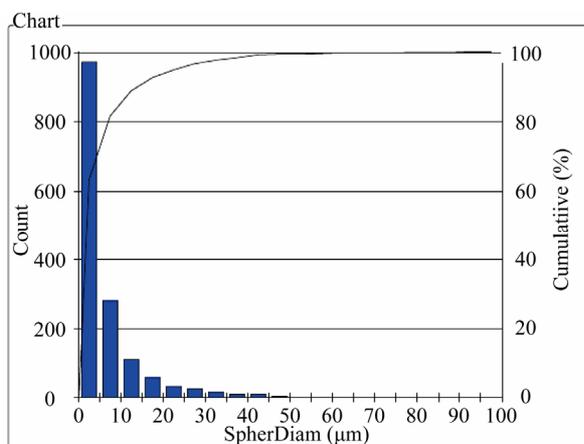


(a)

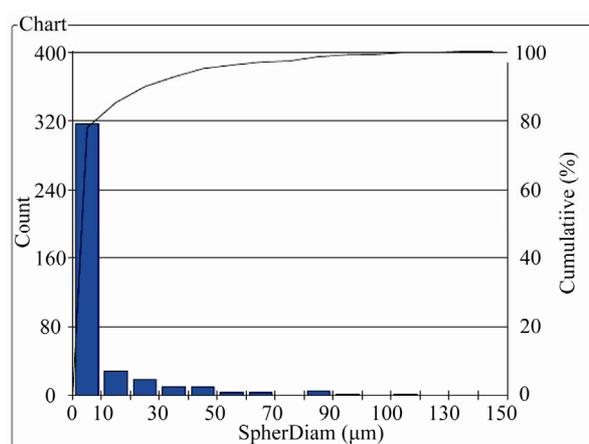


(b)

Figure 2. Morphology of Ni₃Al powders obtained by different milling time; (a) 15 hours, and (b) 55 hours.



(a)



(b)

Figure 3. The particle size distribution of Ni₃Al powders obtained by different milling time; (a) 15 hours, and (b) 55 hours.

3.2. Distribution of Ni₃Al Particles in Al Powders

Blending of aluminum and reinforcement powders (Ni₃Al) results in heterogeneous size distribution of the reinforcement particles (**Figure 4(a)**). The largest size is about 50 μm which is dispersed heterogeneously between aluminum powders. It is certain that the homogenization brings about improved physical and mechanical properties. In the milling process, impact of milling balls on the powders causes plastic deformation and formation of flake-shaped soft aluminum powders and brittleness and fracturing of Ni₃Al particles. Thus the fine reinforcement particles are cold welded to aluminum powder flakes. Continuation of milling causes cold welding of these flakes together. Consequently, a composite powder consisting of aluminum and Ni₃Al is formed. More uniform distribution of reinforcement particles in Al powder is shown in **Figure 4(b)**, which is the result of milling for 12 hours.

Another factor influencing composite properties is the compaction pressure. To investigate the effect of this parameter, two different compaction pressures were applied. Although milling enhances the distribution of reinforcements, it results in lower compressibility of ball milled powder as compared to the blended powder. Therefore, compaction of ball milled powders needs more pressure. Therefore, compression of the powders at 400MPa was impossible.

As a general rule, due to the hardening effect of the mechanical milling, green density of milled powders decreases. The other reason for the incompressibility of the high energy milled powder at the lower pressure is the flat shape of the powder [13,14]. The applied pressure (400 MPa) is not high enough to activate higher plastic deformation due to the flattened morphology. At higher compaction pressure (800 MPa), green density of

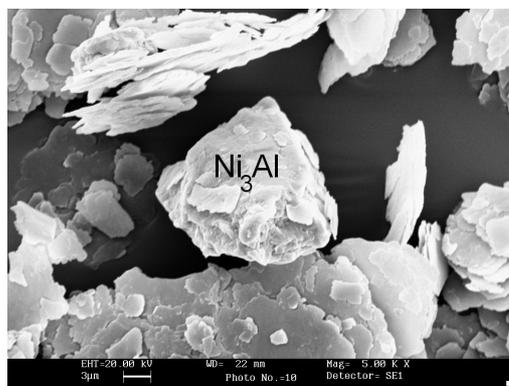
flattened composite (due to plastic deformation of powders) comes up to only 2.65 g/cm³ which equals to 90.5% of the theoretical density. Whereas, compaction of blended powders is easier and green density reaches 89% and 95% of the theoretical density at 400 and 800 MPa, respectively.

3.3. Effect of Manufacturing Parameters on the Interfacial Bonding

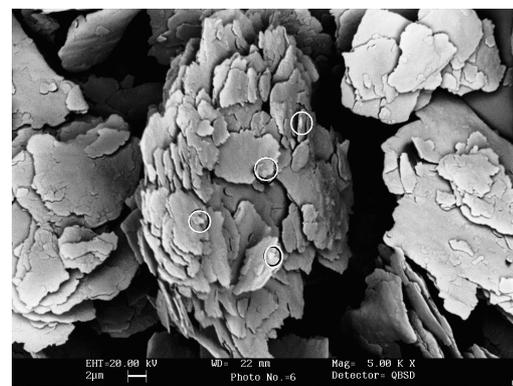
The main parameters such as: duration of mechanical alloying, mixing conditions, compacting pressure and sintering temperature strongly affect the constituent phases and thickness of multi-layers around reinforcement particles.

Figure 5 shows microstructure of Al/5 Vol% Ni₃Al samples compacted at 400 MPa and sintered at 620°C. The powders were produced by milling at different times (15 and 55 hours). As it is observed, the size of 55-hour milled powder is greater than the powder milled for 15 hours due to cold welding of the flakes together.

In the case of Al/5 Vol% Ni₃Al (15-hour milled) composite sintered at 620°C, the effect of strain accumulation on the diffusion, phase transformation and interfacial bonding is less pronounced as compared to the composite with its reinforcement powder mechanically alloyed for 55 hours and then sintered at the same sintering temperature. The comparison of microstructures of the composites (**Figure 5**) shows that in 15-hour milled specimen, Ni₃Al particles are surrounded by diffusion layers and Ni-aluminide phases. While, in the specimen with 55-hour milled Ni₃Al, diffusion and reaction rates are so high that Ni₃Al particles are mostly replaced by Al₃Ni and Al₃Ni₂. The stored elastic energy and higher dislocation densities due to longer milling times are the main parameters in creating the condition which leads to an increase in the reaction rate by providing short diffu-



(a)



(b)

Figure 4. Morphology of Al/5 vol% Ni₃Al; the indicated phases show reinforcement particles (a) Blended powders, (b) 12-hour Milled powders.

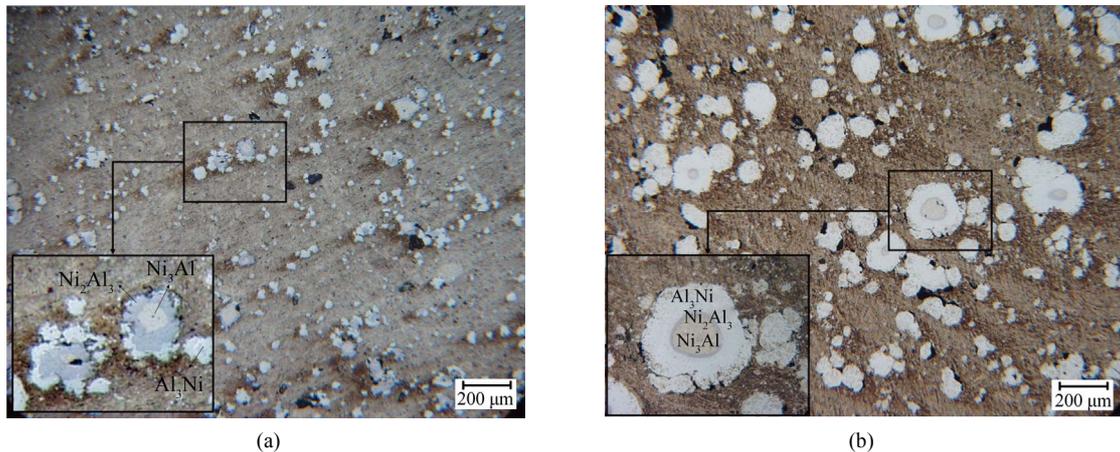


Figure 5. Microstructures of Al/5 vol% Ni₃Al composite sintered at 620°C for 30 min. (a) after 55 hours; and (b) after 15 hours milling.

sion paths. The results are in good agreement with findings of Lieblich *et al.* [20]. They have claimed that the reaction of Ni₃Al and aluminum matrix and dissolution of Ni₃Al particles follows a parabolic law and occurs during three steps. In the first step, nucleation and growth of Al₃Ni phase occurs which surrounds the dissolving Ni₃Al particles; in the second step and at longer times, the Al₃Ni₂ phase nucleates and grows between the Ni₃Al and the Al₃Ni layers; and in the third step, the Ni₃Al and Al₃Ni₂ phases completely dissolve and the stable Al₃Ni phase remains.

It has also been reported [21] that increasing milling time generates more defects such as vacancies and stored strain energy in the powders. The defects formed due to the large plastic deformation result in an increase in reaction rate by providing short circuit diffusion paths.

Figures 6(a) to (c) show microstructures of mixed Al/Ni₃Al powders compacted at 400 MPa and then sintered at three different temperatures (580°C, 620°C and 650°C). The microstructure of compacted powder at 400MPa and sintered at 580°C shows that Ni₃Al reinforcement particles remain almost separated, and the bonding between matrix and the intermetallic particles is not strong (**Figures 6(a)**). Also, the thickness of the diffusion layers around the reinforcements is negligible.

Higher sintering temperatures facilitate the diffusion of elements at matrix/reinforcement interface. Sintering at 620°C causes the formation of multilayer bonds and conjunction between matrix and reinforcements (**Figure 6(b)**). Chemical analysis of samples by the energy dispersive X-ray (EDX) method on reaction layers at reinforcement/matrix interface of composite sintered at 620°C has shown the formation of thin layers Ni₂Al₃ and Al₃Ni around the remaining Ni₃Al nucleus.

At 650°C sintering temperature, the Ni₃Al and Al₃Ni₂ phases completely dissolve and the equilibrium Al₃Ni

phase remains (**Figure 6(c)**). The reinforcement size produced by sintering at 650°C is larger than the one created at 620°C. Furthermore, the microstructure of reinforcements in sintered composite at 650°C is lamellar. Also, the voids in the sintered composite at 650°C are more or less rounded as compared to the other samples sintered at lower temperatures (**Figures 6(a)** and **5(b)**). Because of the decrease in number of voids with increasing sintering temperature, the density of the composite sintered at 650°C is 2.6 g/cm³ which is higher than the other ones (2.57 and 2.59 g/cm³ corresponding to 580°C and 620°C sintering temperature, respectively).

X-ray diffraction patterns shown in **Figure 7** confirm the formation of the above mentioned phases at reinforcement/matrix interface at different sintering temperatures.

Figures 6(d) to (f) illustrate the influence of compaction pressure on microstructure of interface layers of the blended composites, compacted at 800 MPa, and sintered at different temperatures. As it was expected the increasing number of contacts of the reinforcement to matrix, and consequently their reaction with each other, facilitated phase transformation of reinforcement at the interface. Comparing **Figures 6(b)** with **Figure 6(d)** shows that at the higher compaction pressure, the microstructure of composite sintered at 580°C is similar to that compacted at 400 MPa and sintered at 620°C. Increasing the compaction pressure to 800MPa for blended grade powders caused the highest sintered density of 2.7 g/cm³ after sintering at 620°C.

The effect of dispersion condition of Ni₃Al particles in composite powder on phase transformation of reinforcements is noticeable. Microstructural homogeneity of reinforcement is strongly affected by the milling operation. The comparison of the XRD patterns of blended and milled starting powders sintered at different temperatures

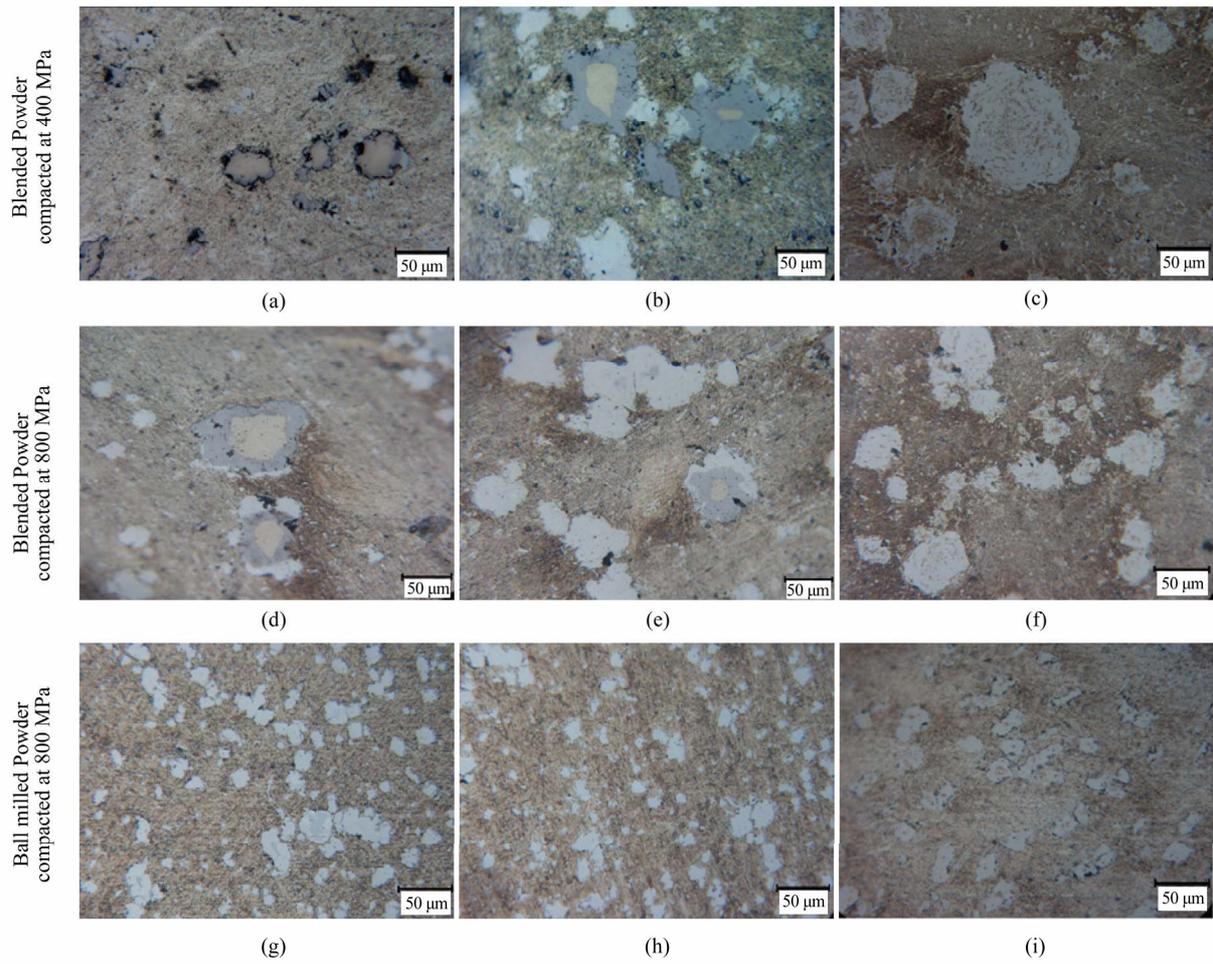


Figure 6. Microstructures of the composites compacted and sintered at different conditions. (a)(d)(g) Sintered at 580°C; (b)(e)(h) Sintered at 620°C; (c)(f)(i) Sintered at 650°C.

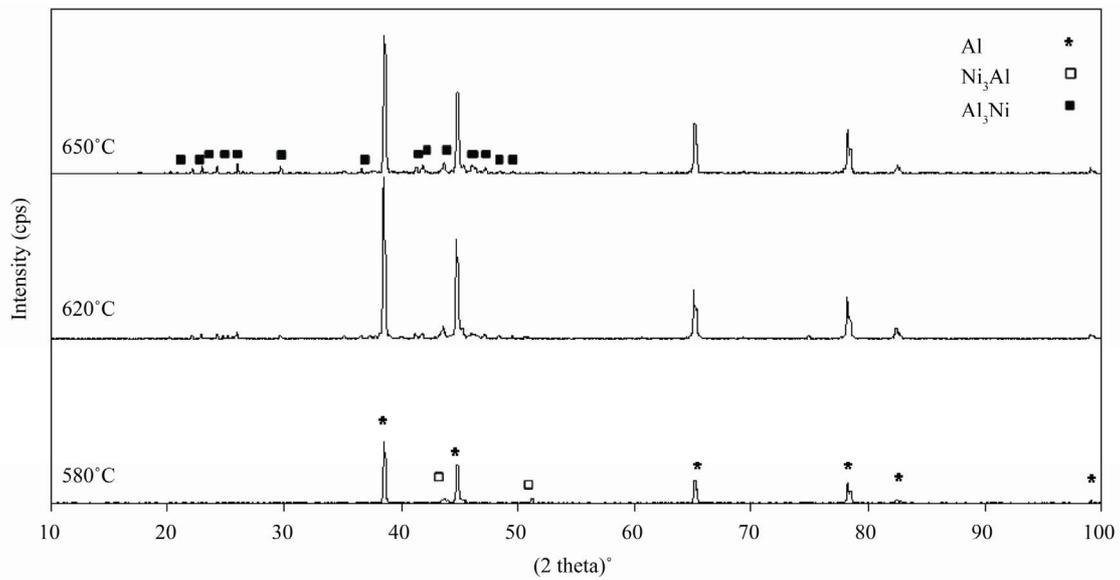


Figure 7. X-ray diffraction pattern of as sintered blended grade samples compacted at 400 MPa.

reveal that milling promotes phase transformation of Ni₃Al to Al₃Ni with respect to the blended one so that the two phases can be observed at all sintering temperatures (see **Figure 7** and **Figure 8**).

It is a well known fact that milling operation can cause the decrease in density of composites. As expected, the lower green density resulted in the drop of sintered density values. The maximum value of density in milled grade was 2.65 g/cm³. The results presented so far indicate that the density of blended and milled grade samples sintered at 650°C is 2.62 g/cm³. This can be attributed to the composites with Al₃Ni reinforcement particles.

Hardness measurement revealed sticking of reinforcement to matrix at the interface. The results presented in **Table 2** followed the same pattern described above. The data shows that the hardness values of sintered specimen produced from ball milled powder are markedly higher than those made of the blended powder grade. Work

hardening of powders and the uniform distribution of reinforcement on Al powder introduced by ball milling process are considered to be the main reasons for such a trend, especially at temperature at which multilayer forms at interface (620°C).

In blended grade, increasing the sintering temperature from 580°C to 620°C causes an increase in hardness values, while sintering at 650°C results in the lower hardness. This can be attributed to the formation of a softer intermetallic phase such as Al₃Ni. A similar drop in hardness of the blended grade compacted at 400 MPa is also expected. Approximately the same values of hardness were observed in samples sintered at 620°C and 650°C, and is considered to be the consequence of void elimination that occurred at 650°C. In other words, hardness drop due to the formation of softer intermetallic phase can be compensated by increasing hardness resulting from void elimination.

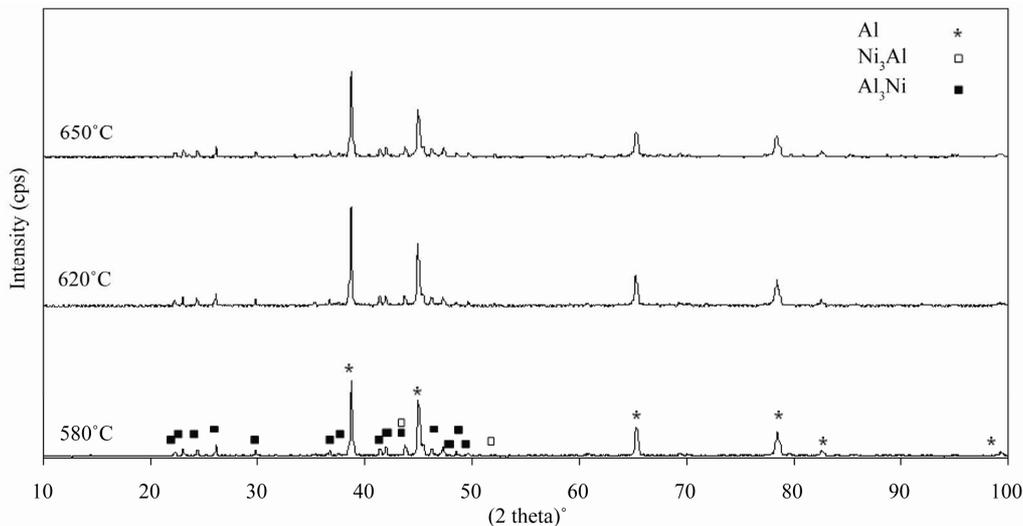


Figure 8. X-ray diffraction pattern of ball milled grade samples compacted at 800 MPa and sintered at three different temperatures.

Table 2. Influence of manufacturing parameters on hardness of sintered Al/Ni₃Al.

Mixing condition	Compacting pressure	Sintering temperature		
		580 (°C)	620(°C)	650(°C)
Blended grade	400 (MPa)	20 HV30	30 HV30	30 HV30
Blended grade	800 (MPa)	35 HV30	45 HV30	34 HV30
Ball milled grade	800 (MPa)	87 HV30	95 HV30	61 HV30

4. Conclusions

Interfacial bonding at matrix/reinforcement interface is significantly affected by mechanical milling time in producing Ni₃Al particles, mixing condition of Al and Ni₃Al powders, compacting pressure and sintering temperature.

- Mechanical milling produces Ni₃Al reinforcement powders that promote the diffusion process owing to large deformation of powder particles as well as more stored strain energy.
- The 55-hour mechanically milled reinforcements provide an effective means of thicker diffusive layers formation around the reinforcements during sintering,

as compared to the thinner interfaces in the composite containing 15-hour mechanically milled intermetallic particles at the same sintering temperature.

- It was shown that finer distribution of reinforcement is significantly more pronounced in ball milled grade than blended Al/Ni₃Al. This apparently improved mechanical properties of sintered composite and encouraged phase transformation of reinforcement.
- In blended grade samples compacted at 400 MPa, low sintering temperature (e.g. 580°C) cannot eliminate the pores around intermetallic particles that generated primarily during compacting by decohesion of the matrix-particle interface. Therefore, the bonding between the matrix and intermetallic particles is not strong and thickness of diffusion layer around the intermetallic is negligible.
- Microstructure of the composite sintered at 580°C shows that Ni₃Al particle reinforcements remain approximately intact. With increasing sintering temperature, diffusive layers will be extended and reaction phases improve the bonding.
- More contacts between reinforcements and matrix caused by higher compaction pressure enhance the reaction at interface; therefore, at higher compaction pressures similar multilayers around reinforcements form at relatively lower sintering temperature.

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