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# Effect of sintering parameters on the densification and hardness of NiAl-CNTs composite

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**Abstract.** Nickel aluminides have attracted considerable interest in the past few decades owing to its unique properties. In this work, nickel aluminide (NiAl) was formed in-situ during spark plasma sintering of admixed powders of nickel, aluminium and carbon nanotubes (CNTs) after ball milling. 1 wt % CNTs was incorporated into the intermetallic matrix and the effect of varying sintering parameters investigated, particularly the sintering temperature and pressure. Results showed that a combination of higher sintering temperature with lower pressure yielded better results than lower sintering temperature and higher pressure. Thus the former parameters yielded better densification and subsequently higher micro hardness values of the NiAl-CNTs composites as compared to the latter.

## 1. Introduction

Aluminides have shown huge potential in high temperature applications due to their high temperature strengths, lightweight, high wear resistance and high oxidation resistance [1-3]. In particular, interest in nickel aluminides have progressively grown over the years because of the aforementioned properties in combination with their relative lightweights as compared to expensive nickel base super alloys [4]. The major limiting factor of these aluminides is their room temperature brittleness due to insufficient slip systems within the lattice [5, 6]. Efforts have been made in the research community to eliminate this challenge mostly by introducing an additional element into the dual system to enhance the ductility and toughness [7]. Recently, carbon nanotubes (CNTs) have proven to be ideal reinforcing candidates owing to the enhanced mechanical properties exhibited by the CNT reinforced metal matrices. They have been widely incorporated into metallic matrices like copper, titanium and aluminium. The mechanical properties of metal matrices have been seen to depend on the microstructure [4]. The microstructures of these metals however, also depend on their process history. The most popular fabrication route for these aluminides have been the powder metallurgy route due to its ease of fabrication coupled with the advantage of producing near net shape components [8]. During sintering, the metallurgical interactions occurring within the matrix are responsible for the microstructure, and consequently the resulting



mechanical properties. The fabrication parameters, especially sintering parameters play a critical role in determining the resulting mechanical properties and consequently, material performance. In this work, 1 wt % CNTs were incorporated into nickel aluminide intermetallic matrix via ball milling and the sintering parameters varied. The resulting mechanical properties from each combination of sintering parameters were thus investigated.

## 2. Methodology

### 2.1. Starting materials

Commercially available nickel, supplied by Weartech Limited with particle size of 0.5-3.0 microns and 99.5% purity, spherical aluminium powder with average particle size of 25 $\mu$ m was supplied by TLS Technik GmbH & Co., and MWCNTs supplied by Nanocyl, Belgium with average diameter of 9.5 nm and average length of 1.5 $\mu$ m were used in this study.

### 2.2. Low energy ball milling

The nickel, aluminium and CNTs powders were ball milled together to effectively disperse the CNTs in the nickel and aluminium powders using a planetary ball mill, PM 100 with milling speed of 150rpm. Nickel and aluminium powders were set at a weight ratio of 1:1 in all the sample mixtures for the synthesis of the NiAl composites. Ball to powder ratio of 10:1 was used and a 10 min break was taken after every 10 min milling to prevent excessive over heating of the powders. Weight percent of CNTs was set at 1 wt % for all samples for accurate comparison and evaluation. The powders were milled for 6 h after which the mixed powders were consolidated by spark plasma sintering.

### 2.3. Spark plasma sintering

The admixed powders were compressed in a graphite die and the whole die assemble was placed in the SPS system (model HHPD-25, FCT Germany). To investigate the effect of sintering parameters on the mechanical properties, 20 mm diameter with 5 mm height discs were sintered with two different sets of sintering parameters namely: 1) sintering temperature of 800 °C and pressure of 32 MPa and 2) sintering temperature of 600 °C and pressure of 50 MPa. Holding time was 5 min and heating rate was set at 100 °C per min for both regimes.

### 2.4. Density and hardness measurements

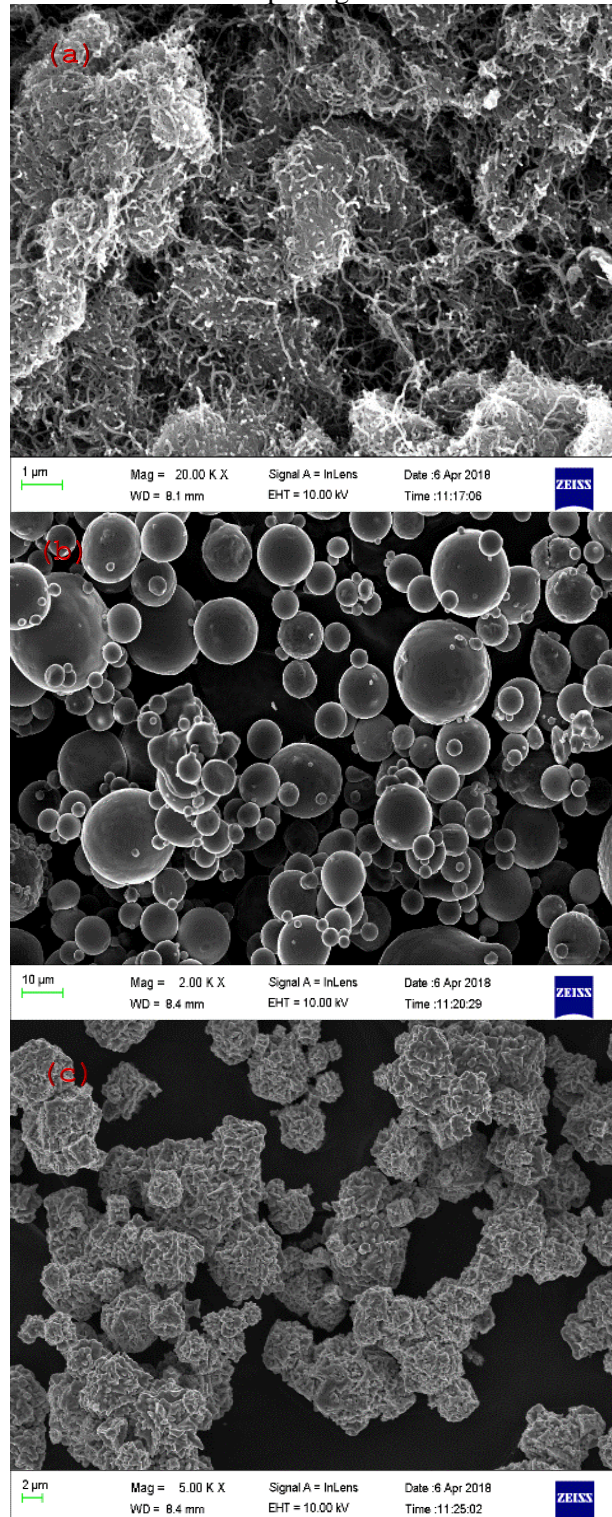
The densities of the sintered discs were measured using the Archimedes' principle according to ASTM standards. The recorded density was an average value of five repeated measurements taken from each sample and the relative densities calculated as percentages of the composites' theoretical densities. Vickers indentation microhardness tests were conducted on sectioned samples that had been previously polished to achieve mirror-like surfaces for all the samples. A load of 100 gf and 10 s dwell time was used for the hardness tests. The reported microhardness values are the arithmetic mean of five successive indentations made on the smooth sample surfaces.

## 3. Results and discussion

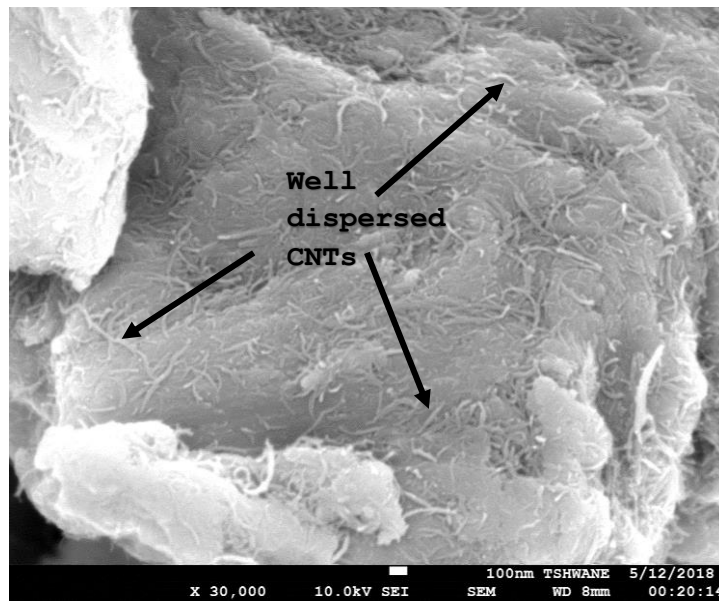
Figure 1 shows the powder morphologies of the starting powders that were milled together. This was done to effectively disperse the CNTs within the powders and secondly to trigger the reaction between nickel and aluminium to form an intermetallic powder matrix.

After 6 h of milling time, the CNTs clusters were effectively broken down and the CNTs uniformly distributed as observed in Figure 2. Low energy ball milling was used throughout to prevent significant damage of the CNTs in the matrix which would be detrimental to their mechanical properties [9]. This is because the mechanical properties of CNTs reinforced composites are largely dependent on the structural integrity of the CNTs. Hence the damage of these nanotubes would adversely affect the mechanical properties of the composites. Figure 3 shows the transmission electron microscopy (TEM)

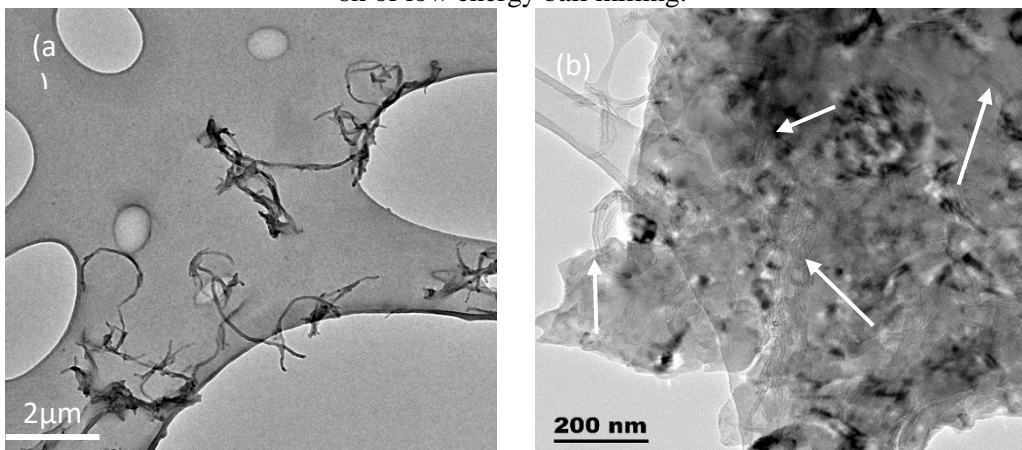
images of the nanotubes, depicting the homogeneous dispersion of the nanotubes and thus endorsing Figure 2. This confirms that the ball milling process employed was particularly effective in dispersing intact nanotubes with well retained tubular morphologies.



**Figure 1.** SEM images of starting powders (a) CNTs (b) aluminium and (c) nickel.



**Figure 2.** SEM image showing 1 wt % CNTs uniformly dispersed in nickel aluminide powders after 6h of low energy ball milling.



**Figure 3.** TEM images showing CNTs uniformly dispersed in nickel aluminide matrix powders after 6h of low energy ball milling.

### 3.1. Relative density and micro hardness

The correlation between sintering parameters and densification cannot be downplayed. Densification, to a large extent, is dependent on sintering parameters. Sintering at higher temperatures or higher pressures tend to give better densification during the sintering process. As can be observed in Table 1, the effect of sintering parameters on the relative density and micro hardness of the NiAl-1 wt % CNT composites is quite significant in this study.

**Table 1.** Effect of sintering parameters on the densification and hardness of NiAl-1wt% CNT composite

Sample ID	Sintering temperature (°C)	Sintering pressure (MPa)	Relative density (%)	Porosity (%)	Hardness (HV/GPa)
A	800	32	97.5	2.5	839/8.23
B	600	50	96	4	691/6.78

Both samples A and B have the same composition of starting powders of CNTs, nickel and aluminium with the same milling history. Sample A was sintered at a higher sintering temperature with reduced pressure, while sample B was sintered at a reduced temperature and higher sintering pressure. The relative densities of the consolidated composites showed a higher density for sample A that experienced a higher sintering temperature. The relative density was 1.5% higher than that of sample B which was sintered at a lower temperature. The observed reduction in the density and subsequently microhardness of sample B depicts the presence of microvoids and microholes within the microstructure [10]. The presence of these flaws makes the compact bulk more susceptible to failure as these defects are potential sites for crack formation. This behaviour is similar to that which was observed in Cooke et al, [11] who also observed higher densification with higher sintering temperature of 550 °C compared to the sample sintered at 400 °C. Chen et al, [12] similarly in his work observed an improvement in densification with increase in sintering temperature.

The seemingly insignificant 1.5% increase in densification led to a substantial increment of 148HV in microhardness of sample A, suggesting that densification played a crucial role in the resulting mechanical properties of sintered composites. The reason for this increment can be attributed to improved bonding between the powder particles at higher sintering temperatures favouring better interfacial contact [12] thus reducing the porosity, leading to better densification and ultimately higher hardness. Given that diffusion is a thermally activated process, at higher sintering temperatures, diffusion is higher, facilitating the rapid diffusion of atoms between the matrix and reinforcement, leading to a cleaner interface between them [13]. This tends to improve the interfacial bonding strength between the matrix and reinforcement, consequently enhancing the load transfer mechanism leading to higher strengthening effects in the composites, hence the improved mechanical properties observed in the composite sintered at 800 °C.

Additionally, the hardness value obtained in this study for Sample A is significantly higher (almost 100% higher) than that documented in literature for NiAl-CNT composites. Ameri et al, [14] in a similar study, achieved a relative density of 85.6 % and corresponding microhardness value of 4.16 GPa in the NiAl-1 % CNT composite. The density and microhardness values observed in this study were 97.5 % and 8.23 GPa respectively, even though the sintering temperature used in Ameri et al, [14] was significantly higher (1000 °C) than that employed in this study. Groven et al, [15] also obtained microhardness values of 4.5 GPa with the incorporation of single walled CNTs and a much lower value of 3.5 GPa with the incorporation of CNTs. The significant increase in the microhardness value obtained in this study can be attributed to better dispersion of the CNTs (as depicted in Figure 2) which promoted better mechanical properties even at lower sintering temperatures as compared to literature.

#### 4. Conclusion

In this work, CNTs were uniformly dispersed in nickel aluminide matrix using low energy ball milling in a bid to preserve the structural integrity of the nanotubes. Sintering parameters were varied to determine their effect on the densification and subsequently hardness of the spark plasma sintered composites. The experimental results obtained clearly depict the importance of sintering parameters in the final mechanical properties of sintered composites. Both the density and the microhardness increased with increase in sintering temperature. Sample A sintered at higher temperature showed higher microhardness value of 8.23 GPa. While Sample B sintered at lower sintering temperatures exhibited lower densification and subsequently lower microhardness value of 6.78 GPa. In spite of the improved microhardness observed in this study in comparison with literature, higher microhardness values are still expected with higher sintering temperatures as can be seen from this study. However further increase of the sintering temperature led to the melting out of the admixed powders during sintering. Hence further work is still on going to overcome this challenge and thus sinter at higher temperatures and to investigate the fracture toughness of the NiAl-CNT composites.

## 5. References

- [1] Liang W, Jiang Y, He Y, Xu N, Zou J, Huang B and Liu CT 2011 The corrosion behaviour of porous Ni<sub>3</sub>Al intermetallic materials in strong alkali solution. *Intermetallics* **11** 1759-65.
- [2] Sulka GD and Józwick P 2011 Electrochemical behaviour of Ni<sub>3</sub>Al-based intermetallic alloys in NaOH. *Intermetallics* **7** 974-81.
- [3] Zhu S, Bi Q, Yang J, Qiao Z, Ma J, Li F, Yin B and Liu W 2014 Tribological behavior of Ni<sub>3</sub>Al alloy at dry friction and under sea water environment. *Trib. Inter.* **75** 24-30.
- [4] Liu E, Jia J, Bai Y, Wang W and Gao Y 2014 Study on preparation and mechanical property of nanocrystalline NiAl intermetallic. *Mat. & Des.* **53** 596-601.
- [5] Noebe RD, Bowman RR and Nathal MV 1993 Physical and mechanical properties of the B2 compound NiAl. *Inter. Mat. Rev.* **4** 193-232.
- [6] Povarova KB, Drozdov AA, Kazanskaya NK, Morozov AE and Antonova AV 2011 Physicochemical approaches to designing NiAl-based alloys for high-temperature operation *Russ. Metall. (Metally)*. **3** 209-20.
- [7] Geist D, Gammer C, Rentenberger C and Karnthaler HP 2015 Sessile dislocations by reactions in NiAl severely deformed at room temperature. *J. of all & comp.* **621**: 371-77.
- [8] Sarmasti AB, Yazdanirad M, Nouri Khezrabad M and Karbalaie M 2011 Effect of alumina particle size and thermal condition of casting on microstructure and mechanical properties of stir cast Al–Al<sub>2</sub>O<sub>3</sub> composites. *Mat. Sci & Tech.* **11**: 1653-66.
- [9] Munir KS and Wen C 2016 Deterioration of the Strong sp<sup>2</sup> Carbon Network in Carbon Nanotubes during the Mechanical Dispersion Processing—A Review. *Crit. Rev. in Sol. St & Mat. Sci.* **5** 347-66.
- [10] Liu ZF, Zhang ZH, Lu JF, Korznikov AV, Korznikova E and Wang FC 2014 Effect of sintering temperature on microstructures and mechanical properties of spark plasma sintered nanocrystalline aluminum *Mat & Des.* **64** 625-30.
- [11] Cooke RW, Kraus NP and Bishop DP 2016 Spark plasma sintering of aluminium powders pre-alloyed with scandium additions *Mat. Sci. & Eng. A.* **657** 71-81.
- [12] Chen B, Kondoh K, Imai H, Umeda J and Takahashi M 2016 Simultaneously enhancing strength and ductility of carbon nanotube/aluminium composites by improving bonding conditions. *Scri. Mat.* **113** 158-162.
- [13] Kurita H, Estili M, Kwon H, Miyazaki T, Zhou W, Silvain JF and Kawasaki A 2015 Load-bearing contribution of multi-walled carbon nanotubes on tensile response of aluminum. *Comp. Pt A: App. Sci & Man.* **68** 133-39.
- [14] Ameri S, Sadeghian Z and Kazeminezhad I 2016 Effect of CNT addition approach on the microstructure and properties of NiAl-CNT nanocomposites produced by mechanical alloying and spark plasma sintering. *Intermetallics* **76** 41-8.
- [15] Groven LJ and Puszynski JA 2012 Combustion synthesis and characterization of nickel aluminide–carbon nanotube composites. *Chem. Eng. J.* **183** 515-25.

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