



Effect of CNT addition approach on the microstructure and properties of NiAl-CNT nanocomposites produced by mechanical alloying and spark plasma sintering



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ABSTRACT

An approach was offered to synthesize NiAl-CNT intermetallic matrix nanocomposite by mechanical alloying (MA) to ensure the effective distribution of carbon nanotubes (CNTs) within the matrix. For this purpose 0.5 and 1 wt% of CNTs were added to the powder mixture before the completion of reaction between Ni and Al. The resultant powders were compared to the powders obtained from MA of ex situ synthesized NiAl intermetallic compound with CNTs. Bulk samples were fabricated by spark plasma sintering (SPS) that retained the integrity of CNTs in the matrix. Structural evolutions were investigated by X-ray diffractometry (XRD). Field emission scanning electron microscopy (FESEM) micrographs showed that the offered MA approach caused the CNTs to uniformly embed in the in situ synthesized NiAl matrix. Meanwhile better distribution of CNTs resulted in higher density of SPSed bulk nanocomposite as well as higher hardness up to 5.6 GPa compared to 5.41 of NiAl intermetallic obtained from the same MA time. Fracture toughness showed more than 6.4% increase with bridging and pull out of CNTs and deflection of cracks as toughening mechanisms.

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1. Introduction

NiAl Intermetallic compound has been recognized as one of high temperature structural materials due to excellent physical and mechanical properties such as low density (5.86 g cm^{-3}), high melting point (1911 K) and excellent oxidation resistance up to 1573 K, as well as good thermal conductivity [1–3]. NiAl compound is also known for its low ductility and fracture toughness at room temperature [4]. Several methods such as grain refinement [3], second-phase strengthening [5] and alloying [6] have been offered to overcome this limitation. High energy ball mills have been used for mechanical alloying (MA) as a production method to synthesize and modify intermetallic compounds like NiAl [3,7]. It has been stated by several researchers that during MA of Ni and Al powder mixtures, NiAl intermetallic compound formation occurs either gradually along milling time, or suddenly through a mechanically self-propagating reaction (MSR), which occurs after a certain time

of MA called ignition time [7,8]. In the last decade MA has been widely used for the fabrication of intermetallic matrix composites containing ceramic or metalloid particles [9–11]. In recent years carbon nanostructures such as carbon nanotubes (CNT), fullerene and graphene have been the focus of significant research [12–15]. CNTs with exceptional mechanical and physical properties offer promising potential as a reinforcing material to enhance the mechanical, thermal and electrical properties of composites [12–14,16]. It is obvious that a successful dispersion of CNTs in the matrix is needed before any sort of significant benefits in the composite is realized. In fact, adequate dispersion of the CNTs in the matrix is still challenging [17]. It has been concluded from several researches that MA can be considered as an effective process to achieve homogenous distribution of CNT in metallic matrices [18]. It is known that CNTs are entangled between rewelded ductile particles during milling and after appropriate time are embedded within metal matrix [19]. Milling of CNTs with brittle powders like ceramics and intermetallic compounds has been reported to result in the distribution of CNTs on the surface of particles that can affect the compressibility and sinter ability of powder particles [20,21].

In the present study a route was proposed to synthesize NiAl-CNT by MA of elemental powders to achieve effective

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accommodation of CNTs within the NiAl matrix during in situ formation of intermetallic compound. The milling time with CNTs was limited to 5 h to reduce the damage of CNTs. In the present approach CNTs were added to the powder mixture before the completion of reaction between Ni and Al. therefore, CNTs could entangle between re-welding ductile powder particles before the complete evolution of particles to fully brittle intermetallic compound. The structural evolution during MA was investigated. In order to obtain fully dense composites, and minimize the decomposition of CNTs during sintering, spark plasma sintering (SPS) was employed in the present study. SPS, in which much lower sintering temperatures and shorter times are applicable, is an effective sintering technique for obtaining fully dense nanocrystalline composites [22]. Microstructure of powders and SPSed samples was studied and microhardness and fracture toughness of bulk samples were evaluated.

2. Materials and methods

Commercially available Ni (Merck, <20 μm, 99.8% purity) and Al (Sigma-aldrich, <20 μm, 99. % purity) powders and multiwalled CNTs (Iranian Research Institute of Petroleum Industry, about 10 nm outer diameter, 4 nm internal diameter and a few micrometers length, 99% purity) were used as starting materials. Powder mixtures were MAed, nominally at room temperature using a planetary ball mill. Stoichiometric ratio of 50 at.%Ni-50 at.%Al was considered for the preparation of powder mixtures to synthesize NiAl matrix by MA. CNTs were sonicated in ethanol for 30 min and MAed powder mixtures were added to this suspension and sonicated for further 20 min. The ball to powder weight ratio was chosen to be 10:1 with 20 mm diameter chromium steel balls. The hardened chromium steel vial was evacuated and filled with argon (99.99% purity) to prevent oxidation during the milling process. Two approaches were used for the distribution of 0.5 and 1 wt% of CNTs in the matrix powder as presented in Table 1. In the first approach CNTs were distributed in NiAl intermetallic powder obtained from 45 h of MA by further milling for 5 h. The second approach involved the addition of CNTs to Ni–Al powder mixture obtained from 15 h of MA and milled for further 5 h Table 1 presents the information of different powders produced by MA. MAed powders were consolidated by KPF-NANOSINT SPS equipment to obtain bulk samples of 25 mm diameter and 15 mm height. The powders were compressed in a graphite die up to 6 MPa uniaxial pressure using a manual press and the whole die was placed in the SPS system. Samples were sintered at 1000 °C with a holding time of 2 min. SPS process was performed under argon with a maximum uniaxial pressure of 50 MPa and a heating rate of 50 °C min⁻¹.

Structural evolution during MA and after consolidation was investigated by a Philips X'PERT diffractometer employing monochromatic Cu K_α radiation (λ = 0.15406 nm). X-ray diffraction (XRD) scans were performed between 20 and 90° in 2θ with a step size of 0.05° and a dwell time of 20 s per step. Powder diffraction files (PDF) of the International Center for Diffraction Data (ICDD) were used to identify the existing phases during MA and after high

temperature exposure. Morphology and cross-sectional microstructures of powder particles and SPSed samples were studied by scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM). Powders were mounted within a conductive mounting resin and the mounted samples were prepared by metallography techniques.

Fracture toughness of the samples was determined by the indentation method. A Vickers indenter was used on metallographically prepared SPSed samples with a 20 kgf load. The lengths of the cracks (where present) were measured from scanning electron micrographs imaged in the secondary electrons (SE) mode. At least 10 indentations from each sample were examined. The fracture toughness (K_c) values were calculated using cracks showing 0.6 ≤ c/a ≤ 4.5. Evans and Wilshaw equation was selected for the indentation fracture toughness for the current work due to its applicability to systems generating short cracks as given by Ref. [23]:

$$K_c = 0.079 \left(P/a^{3/2} \right) \log(4.5 a/c)$$

where, P is the applied indentation load, a is the indentation half diagonal and c is the crack length from the center of the indent.

3. Results and discussion

3.1. Powder preparation

The formation of NiAl intermetallic compound by MA of Ni₅₀Al₅₀ powder mixture was investigated to determine the mechanism and time of reaction between elements. Fig. 1 shows the XRD patterns obtained from Ni₅₀Al₅₀ powder mixture after different times of MA. It can be observed in the XRD pattern that after 10 h of milling both peaks relating to Ni and Al were broadened and decreased in intensities. A slight peak shift occurred in Ni peaks while Al peaks seem unmoved. After 15 h of milling peaks corresponding NiAl intermetallic compound (matched with ICDD PDF# 00-002-1261) appeared in the XRD pattern and Al peaks disappeared, while Ni peak at 2θ value of 51.5° still was observable. The reason for the disappearance of the Al XRD peaks can be either the crystallite refinement of Al or the atomic scattering factors of the elements. Considering XRD results it can be concluded that NiAl has been synthesized by gradual diffusion of elements through the point defects, dislocations and the fine lamellar structure induced by MA as mentioned by previous researchers [7]. According to the literature the synthesis of NiAl by MA process may accomplish also through self-propagating or diffusional mechanism depending on the milling conditions [8]. The Existence of (100), (111) and (210) superlattice diffraction peaks at 2θ values of 31.3°, 55.4° and 73.9° suggests that the crystalline NiAl phase has B2 ordered structure. Transformation of Ni₅₀Al₅₀ powder mixture to NiAl intermetallic compound seems to be completed after 40 h of MA, since no sign of residual Ni can be observed in the XRD pattern.

Cross-sectional SEM micrographs of powder particles obtained from MA of Ni₅₀Al₅₀ powder mixture are presented in Fig. 2. After 20 h of milling (Fig. 2a) powder particles showed a fine lamellar morphology of Ni-rich and Al-rich layers, which is typical of MA. However, in some small particles, the lamellar structure has been disappeared (arrows in the figure). By increasing the milling time up to 40 h that resulted in the completion of Ni–Al reaction, the layered structure disappeared as can be seen in Fig. 2b.

The first approach for the fabrication of NiAl-CNT nanocomposite was the addition of 0.5 and 1 wt% CNTs to NiAl after the completion of reaction. Therefore, the powder obtained from 45 h of milling was considered as the matrix to be milled with CNTs for

Table 1
MA routes for the synthesis of NiAl and NiAl-CNT powders.

No.	Specimen code	MA of Ni ₅₀ Al ₅₀	MA of Ni ₅₀ Al ₅₀ + CNT
1	NiAl(50 h)	50 h	–
2	NiAl(45 h)-0.5%CNT	45 h	5 h
3	NiAl(45 h)-1%CNT	45 h	5 h
4	NiAl(20 h)	20 h	–
5	NiAl(15 h)-0.5%CNT	15 h	5 h
6	NiAl(15 h)-1%CNT	15 h	5 h

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