

Fabrication of bulk AlN–TiN nanocomposite by reactive ball milling and underwater shock consolidation technique

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Abstract

Reactive milling of aluminum nitride and titanium powders corresponding to the stoichiometric reaction $\text{Ti} + \text{AlN}$ resulted in the formation of the ceramic matrix composite AlN–TiN. Prolongation of the milling process led to a microstructure with nanosize range of crystallites of both AlN and TiN, evidenced through XRD measurements, SEM and TEM observation. Further, underwater shock compaction with a pressure level of about 10 GPa was applied to the nanocomposite powders to obtain bulk nanostructured sample. The effect of this shock compaction on the prolonged milled powder resulted in a 22% reduction in crystallite size. The average microhardness of the consolidated nanocomposite was 656 HV and 840 HV for 40 h and 100 h MA samples, respectively, with densities 98% of theoretical values in both cases as well as no change in chemical composition.

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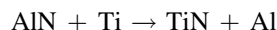
1. Introduction

Nanocrystalline structures poses superior properties such as high strength, hardness and elastic modulus [1,2], and thus have attracted attention as high-performance materials. The process of mechanical alloying (MA) is a common method for the synthesis of ceramic matrix based nanocomposites [3]. The advantage of MA is that both matrix and reinforcement formation are an in situ process [4]. The in situ composites produced by MA possess clean, contaminant free matrix–reinforcement interfaces, which promote good bonding between matrix and reinforcement. Moreover, MA allows a homogeneous distribution of nanosized reinforcing particles in the matrix [5–8].

MA includes a process called mechanochemistry [9], in which chemical reactions and phase transformations occur due to the application of mechanical energy.

This process includes exchange reactions, reduction/oxidation reactions, compound decomposition and phase transformations.

Usually, these reactions are characterized by a large negative free energy change that can occur as a gradual transformation or as a self-propagating combustion according to milling conditions [10]. The resulting product depends on the composition of the starting powder mixture. For example, a reduction reaction of aluminum nitride by titanium can result in the ceramic matrix:



with a varying ceramic phase as a matrix together with nanocrystalline structures.

Commercial applications, however, require that these powders be compacted to obtain bulk nanocomposites, a process that demands high pressure and temperature [11]. It has been observed that the elevated temperature at conventional method require, cause excessive grain growth [4] leading to degradation of the nanocrystalline structures. Earlier work [12–18], though, has shown that this grain growth can be inhibited by very high pressure applied during consolidation.

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Explosive compaction, an alternative method for compaction of nanocrystalline powders [22] utilizes a very rapid and intense deposition of shock energy to the powder particle surfaces to produce consolidation [19,20]. Shock waves originating from an explosive detonation can create pressure from a few to tens of GPa, resulting in inter-particle bonding in microseconds [21]. Still, explosive compaction also creates very high temperatures, which may cause melting and excessive grain growth leading to partial depletion of nanocrystalline structures [23,24]. Underwater shock compaction, an underwater shock wave generated by the detonation of an explosive for compaction [24,25], was envisioned as a technique to overcome the temperature problem. During pressurization and acceleration by the underwater shock wave, an intensive deformation of the powder surface is induced which causes the powder surfaces to undergo melting and solidification in microseconds. Material which is fully dense yet retains its nanocrystalline structure is possible due to the absence of overheating and the associated excessive grain growth. Hence, the aim of the present work is to produce “in situ” nanocrystalline AlN–TiN ceramic matrix nanocomposite powders through reactive milling and to consolidate the same using underwater shock compaction technique.

2. Experimental procedure

2.1. Milling details

Mixtures of aluminum nitride and titanium powders with a molar ratio of 1:1 were prepared, from starting powders of Ti (99.7% pure, max. particle size 45 μm), and agglomerated AlN powder. A Fritsch Pulverisette P6 planetary ball mill was used for the reactive ball milling processes under the milling conditions given in Table 1. Milling was interrupted at regular intervals for characterization. Powder handling was performed in a glove bag under an argon atmosphere.

2.2. Assembly design of shock compaction apparatus

The underwater shock compaction apparatus, which its schematic is shown in Fig. 1, was composed of five parts: explosive lens, explosive container, water container, powder container and cover plate. Two types of explosives: SEP and HABW (both supplied by Asahi-Kasei Chemicals Corp., Japan); detonation velocity: 6.97 and 4.75 km/s, respectively; density: 1300 and 2200 kg/m³, respectively, were used in the

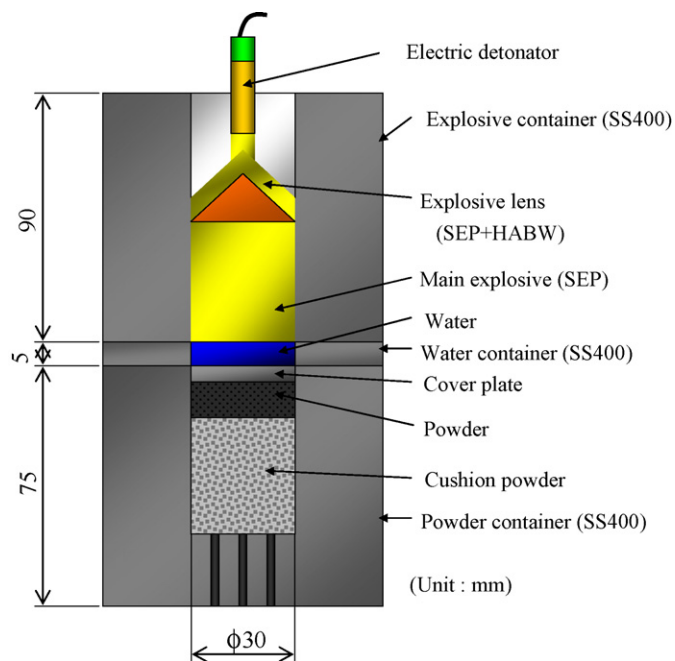


Fig. 1. Schematic illustration of underwater shock compaction apparatus.

explosive lens to create planar waves. The explosive SEP (PETN 65% mass and paraffin 35% mass) was set in the explosive container as the main explosive. The water container, made of mild steel with dimensions of 5 mm height and 70 mm diameter was filled with water for the underwater shock wave to propagate through it. Shock pressure magnitude was controllable by adjusting the height of the water container. The magnitude of shock pressure used in this work was estimated as 10 GPa based on numerical simulation [26], almost identical to the actual pressure magnitude as measured using a Manganin gauge developed by Mashimo and a co-worker [27]. The milled powder was filled and pressed into the powder container using a uniaxial press machine at a pressure of 50 MPa. The powder container, made of mild steel, had an inner diameter of 30 mm, a height of 75 mm, a powder charging depth of 50 mm, and a gas drain hole of 2 mm diameter to allow trapped air to escape during compaction. The thickness of the pressed powder was approximately 10 mm. The cover plate was made of stainless steel with a thickness of 2 mm and was placed on top of the powder.

2.3. Sample characterization

Observations and characterizations of the powders and the consolidated composite were carried out using optical microscope (OM), SEM, TEM, EPMA and X-ray diffraction (XRD). The XRD investigations were performed using a Rigaku Denki ultra X 18VB2-3 with Cu K α radiation ($\lambda = 0.15418$ nm). Scanning electron microscopy (SEM) observations of the powder particles size and morphology were performed using a JEOL (JCM-5700) at an accelerating voltage of 15 kV. Structural observations of the powders by transmission electron microscopy (TEM) were carried out with

Table 1
Milling conditions.

Mill	Planetary ball mill
Milling speed (rpm)	250
Ball to powder ratio “BPR”	10:1
Diameter of milling ball (mm)	10
Process control agent “PCA”	1% stearic acid
Volume of milling vial (ml)	500
Weight of each milling ball (g)	4
Milling time (h)	2.5, 5, 10, 20, 40, 100

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