

# Syntheses of Ti-Al-Si-B-C nanocomposites by mechanical alloying and explosive compaction\*\*

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Nanocomposites of Ti-Al-Si-B-C systems are characterized by unique physical and mechanical properties. They are attractive and can be used in a wide range of areas including aerospace, power engineering, machines and chemistry. Coarse crystalline Ti, Al, Si and C powders and amorphous B were used as initial materials. Different compositions of Ti, Al and C were prepared for mechanical alloying. Selection of blend compositions was made on the base of phase diagrams. Powders were mixed accordingly to produce blends, which were processed in high energy “Fritsch” planetary premium line ball mills for mechanical alloying, synthesis of new phases and ultrafine particle formation. Processing time varied between 1 and 10 hours. Optimal regimens of blend preparation were determined experimentally. Ball-milled blends were investigated to determine mechanical alloying. Ultrafine blends were consolidated using explosive compaction technology for bulk ultrafine-grained composite formation and investigated structurally.

**Keywords:** composite materials, nanostructure, nanotechnologies

## 1. Introduction

The increasing role of and interest in advanced materials is promoted by the general development of engineering and materials science. Ceramic and metal–ceramic composite

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\*\* This paper was first presented at the eleventh Japanese–Mediterranean workshop (JAPMED’11), 15–19 July 2019 in Batumi, Georgia.

materials are being developed by various conventional technologies.<sup>1-3</sup> Composite nanostructured materials are expected to be characterized by exceptional mechanical and physical attributes including resistance to high temperatures and aggressive media. Many such materials are promising for applications in modern machine building, aerospace, the chemical and metallurgical industries and others. At the same time the wide application of composite materials, especially nanocrystalline ones, is restricted due to the absence of effective technologies for their production. Their industrial application requires low-cost production of large quantities of nanopowders and simple technologies for their consolidation to obtain bulk materials. It is very important to develop industry-oriented technologies characterized by resource-saving, environmentally friendly characteristics.

Several conventional methods are known for obtaining bulk ultrafine-grained/nanostructured materials.<sup>4</sup> Some of those methods require high pressure and temperature for long intervals. Because of significant coarsening of ultrafine grains, effects due to nanostructure are diminished. Mechanical alloying (MA) involves repeated cold welding, fracturing and rewelding of particles in a high-energy ball mill, with which the synthesis of a variety of ultrafine-grained materials and nanocomposites can be achieved. An important attribute of these nanocomposites is prevention or minimization of grain growth until very high temperatures.<sup>5</sup>

The technology for preparing powders by MA and the synthesis of bulk materials from ultrafine-grained powders of the Ti-Al-Si-B-C system are described in this work. The main problems in the production of bulk nanostructured materials arise from: constraints on the sizes and geometries of the final bulk material; high energy consumption; the need for complex manufacturing facilities; the difficulty of controlling grain sizes; and significant coarsening of structure under high temperature conditions prevailing for extended intervals.

The main objectives of this work are: (a) perfecting MA for obtaining nanopowders as precursors for the synthesis of bulk materials; and (b) explosive consolidation (EC) technology for the fabrication of bulk nanostructured materials.

## 2. Experimental

Preliminary selection of Si-C-B, Ti-Al-B-C and Ti-Al-C blend compositions was made on the basis of theoretical investigations. Different initial compositions of Si-B-C and Ti-Al-B-C systems were taken, including: 2Ti:Al:C, 3Ti:2Al:1 and 3Ti:6Al:4B:C atom ratios. Compositions were selected according to the phase diagrams for binary and ternary systems. Coarse powders of Ti, Al, C and Si and amorphous B were used as starting precursors. Different compositions

<sup>1</sup> R. Mania, M. Dabrowski et al. Some applications of TiAl micropowders produced by self-propagating high-temperature syntheses. *Intl J. Self-Propagating High-Temperature Synthesis* **12** (2003) 159–164.

<sup>2</sup> E.A. Levashov, B.R. Senatulin et al. Peculiarities of functionally graded targets in combustion waves of the SHS-System with working layer Ti-Si-B, Ti-Si-C, Ti-B-N, Ti-Al-B, Ti-C. *Abstracts IV Intl Symposium on SHS*, Technion, Haifa, pp. 17–21 (2002).

<sup>3</sup> A.G. Merzhanov and A.N. Pityulin. Self-propagating high-temperature synthesis in the production of functionally graded materials. *Proc. 3rd Intl Symposium on FGM*, Lausanne, pp. 87–94 (1995).

<sup>4</sup> J. Hebeisen, P. Tylus, D. Zick, D. K. Mukhopadhyay, K. Brand, C. Suryanarayana and F.H. Froes. Hot isostatic pressing of nanostructured g-TiAl. *Powders Metals Mater.* **2** (1996) 71–74.

<sup>5</sup> C. Suryanarayan. Mechanical alloying and milling. *Progr. Mater. Sci.* **46** (2001) 1–184.

(with different ratios) of Si-C-B, Ti-Al-B-C and Ti-Al-C were prepared for MA, again selected according to the phase diagrams for binary and ternary systems. Precursors were size-classified by vibratory sieves. Particle sizes of the Ti and Al powders were less than 200  $\mu\text{m}$ .

For MA and nanopowder production, the high-energy “Fritsch” planetary premium line ball mill was used. The mill was equipped with zirconium oxide jars and balls. The ball to powder ratio was 5:1 by mass. Processing times were 1, 3, 5 and 10 hours. Rotation speed of the jars was 500 rpm. MA was applied for the Ti-Al-B-C powders for processing times of 1–10 h in preparation for applying the EC technique. Preliminary work showed that EC of metal–ceramic compositions is not only feasible but can produce materials of almost theoretical densities.<sup>6</sup> The major advantages of EC for bulk nanomaterial production are realization of high pressure, short processing time and superhigh cooling rate (adiabatic cooling).

The powder blend was loaded into a cylindrical steel container (Fig.1), which was then closed at both ends. A cardboard box was filled with powdered industrial explosive and placed around the cylinder.<sup>7,8</sup> Operations were carried out at room temperature in an underground explosive chamber. The shock wave loading pressure was varied in the range 7–10 GPa, which is optimal in the sense that the configuration of loading/unloading waves in the powder and container allows the syntheses to be initiated in the reaction mixture, for it to be simultaneously consolidated, and for the phase composition to be fixed under adiabatic cooling.

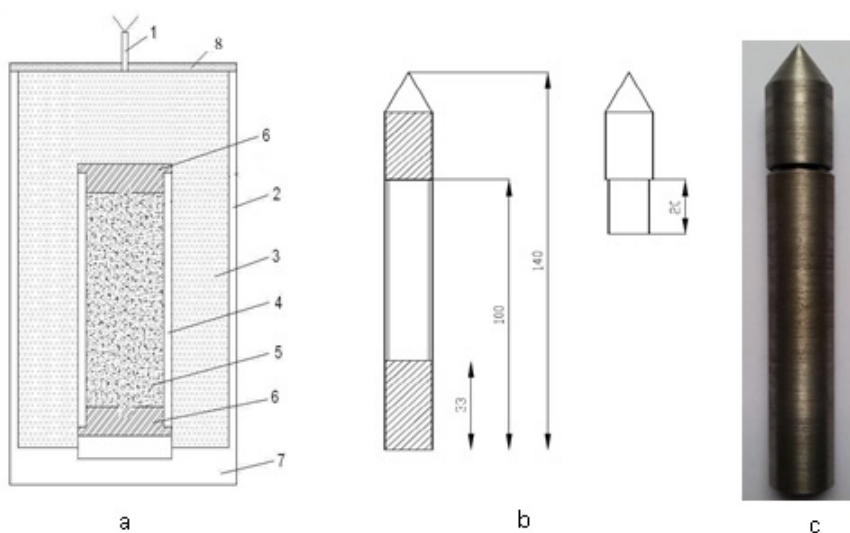


Figure 1. (a) Schematic view of assembly for fabrication of bulk rod: (1) electrical detonator; (2) container for explosive; (3) explosive; (4) steel tube (container); (5) reaction mixture; (6) steel plugs; (7) base table; (8) detonator holder. (b) Schematic of container (dimensions in mm). (c) Photograph of container.

<sup>6</sup> L.J. Kecskes, R.H. Woodman, N. Chikhradze and A. Peikrishvili. Processing of aluminum nickelides by hot explosive consolidation. *Intl J. Self-Propagating High-Temperature Synthesis* **13** (2004) 89–92.

<sup>7</sup> R. Prummer. *Explosive Working of Porous Materials*. Springer: Berlin (1987).

<sup>8</sup> N. Chikhradze, A. Gigineishvili, M. Chikhradze and G. Oniashvili. Explosive consolidation of ultrafine Ni-Al-Ti powders, *SGEM-2010*, vol 1, pp. 1173–1180 (2010).

### 3. Results

After MA the powders were examined in a scanning electronic microscope (SEM). The images presented in Fig. 2 show the tendency of grain sizes to be reduced. We infer that ball milling, which causes fragmentation and critical reduction of particle sizes, can significantly increase the sintering ability of the blend and improve the compaction process and MA of selected powder compositions.

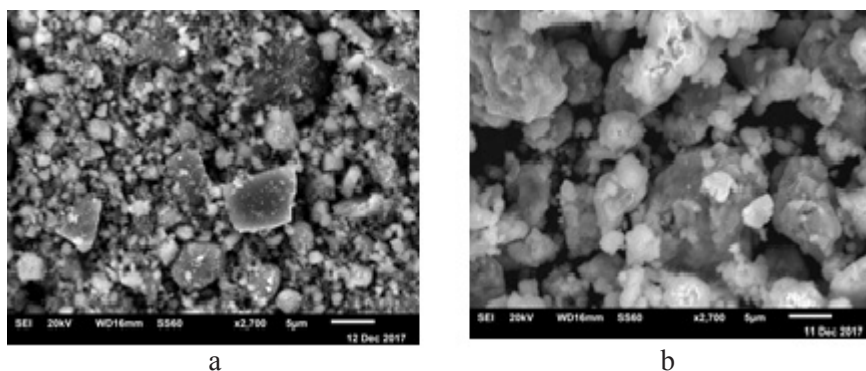


Figure 2. Scanning electronic micrographs of: (a) Si-B-C powder after 5 h mechanical alloying (MA); (b) Ti-Al-B-C powder composition after 5 h MA. The scale bars at the lower right of each image represent 5 µm.

EC enables experiments at high pressure with a short processing time and high cooling rate to be realized, which is a very convenient route for the production of bulk nanocomposites. SEM images of Si-B-C and Ti-Al-B-C after EC are shown in Fig. 3. Some amorphization of structure is observed.

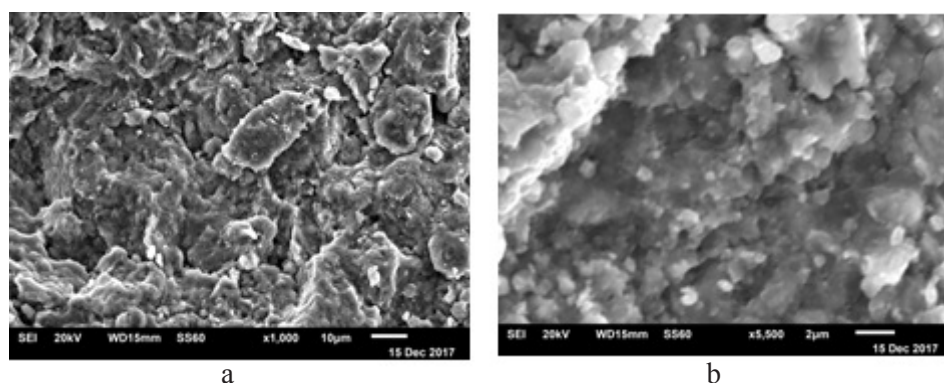


Figure 3. Scanning electronic micrographs of: (a) The microstructure of Si-B-C after explosive consolidation (EC) (scale bar at the lower right represents 10 µm); (b) The microstructure of Ti-Al-B-C after EC (scale bar at the lower right represents 2 µm).

#### **4. Summary and conclusions**

- Different compositions of Ti-Al-Si-B-C were selected for formation of nanostructured composites. Optimal régimes for MA of powders, such as ball:powder ratio, rotation speed and medium, were established.
- The optimal shock wave loading pressure was found experimentally.
- It was confirmed that if the shock wave pressure and released energy exceeded the strength limits of the container, it was destroyed.
- Microstructure and particle sizes were examined after MA and before EC. It was established that the structure is not uniform, with both nanosized and coarse grains being present.
- Compaction was observed after EC.
- A régime for obtaining Ti-Al-Si-B-C nanocomposites has been elaborated.
- Scanning electron microscopy is a useful technique to evidence the successful synthesis of nanocomposites.

#### **Acknowledgment**

This work was supported by Shota Rustaveli National Science Foundation (SRNSF) via Grant #218008, Project title “Syntheses of Ceramic Nanocomposites in the Si-B-C System by Mechanical Alloying and Explosive Compaction”.