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To cite this article: Mikheil Chikhradze *et al* 2019 *IOP Conf. Ser.: Earth Environ. Sci.* **221** 012105

Shock-Wave Consolidation of Boron and Carbon Containing Ultrafine Powders and Investigation Their Structure/Properties

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Abstract. The unique properties of the ultrafine grained composites, makes them attractive for aerospace, power engineering, machine and chemical and other practical applications. Carbon and Boron based composites are important and on high demand because of their specific properties and wide areas of application. It must be noted that SiC, TiC, TiB_2 and B_4C are characterized with high hardness, wear resistance, corrosion resistance, they have high melting temperatures. Therefore they find wide application for preparing the details to working at high temperatures and aggressive media. According to the phase diagrams in the selected systems, the composites/intermetallics may be obtained with wide spectrum of phase composition, in crystalline and amorphous structures. Depending on the composition and structure, the synthesized composites exhibit different specific properties. The potential of the system for development of new structural/composite materials in different thermodynamic conditions is very attractive. Nano structured composite materials of Ti-Al-Si-B-C system, prepared in the form of micromechanical blends, solid solutions and intermetallic compounds are of great practical interest because of improved mechanical properties in comparison with coarse grain material (>1 μm). The methodology and technology for the fabrication of bulk materials from ultrafine powders of Ti-Al-B-C and Si-B-C systems are described in the paper. The crystalline coarse Ti, Al, Si, C powders and amorphous B were used as precursors, and blends with different compositions of Si-C-B, Ti-Al-B-C and Ti-Al-C were prepared. The powders were mixed according the selected ratios of components to produce the blend. For Mechanical alloying (MA) the high energetic "Fritsch" Planetary premium line ball mill was used. Ratio ball to powder by mass was 10:1. The time of the processing was varied from 2 to 5 hours with rotation speed of 500 rpm. For the consolidation and bulk sample formation Explosive Compaction (EC) technology was used. The experiments were performed at room temperature. The shock wave pressure was varied in the range of 5-20 GPa. The ultrafine powders and bulk compacts of different compositions were prepared for investigations. The microstructure was studied by SEM and the effective regimes for obtaining nanopowders and nanocomposites in Ti-Al-Si-B-C composition has been established.

IOP Conf. Series: Earth and Environmental Science 221 (2019) 012105 doi:10.1088/1755-1315/221/1/012105

1. Introduction

New findings and discoveries in applied materials science is directly connected to the development of advanced materials and the technologies for their production. The variety of those advanced materials covers metallic, ceramic, metal-ceramic and composite materials. Number of investigations has been done in order to fabricate ceramic and metal-ceramic composite materials by different conventional technologies [1-3]. The above mentioned advances materials are expected to be characterized with unique mechanical and physical properties which can work at high temperature, under high intensity dynamic loadings and aggressive media. Majority of the materials are attractive for application in modern machine building, airspace, chemical and metallurgical industry as well as in energetics and nuclear fields. Although, it must be mentioned, that the wide application of composite materials is restricted due to the either disadvantages of traditional technologies or the absence of effective technologies for the production of such advanced materials. The improvements of the properties of nanomaterials depend on the improvement of synthesis methods to manufacture nanocrytalline materials in a large scale. Widespread application of nanocrytalline materials requires the low cost production of the industrial applicable quantity of nanopowder and efficient technology for the consolidation of nanoparticles for obtaining bulk materials. Therefore, it is very important to develop technologies which are orientated on industry and are resource-saving, environmentally friendly methods.

Several conventional methods are known for obtaining bulk ultrafine grained/nanostructured materials [4], in particular: Self –Propagating High-temperature Synthesis (SHS), hot isostatic pressing (HIP), spark plasma syntheses (SPS), laser engineered net shaping (LE), Mechanical Alloying (MA) and etc. Some of the mentioned methods require use of high temperature for extended period of time. Because of significant coarsening of the ultrafine grains, nanostructure effects are decreased.

MA involves repeated cold welding, fracturing, and re-welding of powder particles in a high-energy ball mill. Because of the specific advantages, MA is used to synthesize a variety of ultrafine grained materials and nanocomposites. Nanocomposites have also been obtained when the amorphous phases obtained by MA are crystallized at relatively low temperatures. An important attribute of these nanocomposites is in preventing or minimizing grain growth till very high temperatures [5].

The methodology and technology for the fabrication of powders my Mechanical Alloying (MA) technique and synthesis of bulk materials from ultrafine grained powders of Ti-Al-Si-B-C system are described in the paper, including experimental results of MA and synthesis possibilities of MA bland by Shock wave compaction technique.

2. Experimental Investigations

The crystalline coarse Ti, Al, Si, C powders and amorphous B were used as starting elements and blends with different compositions of Si-C-B, Ti-Al-B-C and Ti-Al-C were prepared for MA procedures at the first stage.

Preliminary selection of blend compositions was made on the basis of theoretical investigations. Different initial compositions of Si-B-C and Ti–Al–B–C systems, including: 2Ti/Al/C, 3Ti/2Al/1C and 3Ti/6Al/4B/1C elemental molar ratios. Compositions were selected according to the phase diagrams for binary and ternary systems. Phase equilibrium system was determined based on the Gibbs's principle-minimization of total energy.

Precursors were classified by vibratory sieves. The particles size of Ti and Al powders was less than 200 μm. For MA, amorphization and nanopowder production, the high energetic "Fritsch" Planetary premium line ball mill was used. The mill was equipped with Zirconium Oxide jars and balls. Ratio ball to powder by mass was 5:1. The time of the processing was varied in range: 0.25, 1; 3; 5 hours. Rotation speed of the jars was 500 rpm.

After the MA process the Scanning Electronic Microscope (SEM) investigations has been carried out for the compositions in powder form. The SEM images on Figure 1 and Figure 2 shows the tendency of reduction of brain sizes during the mechanical alloying process.

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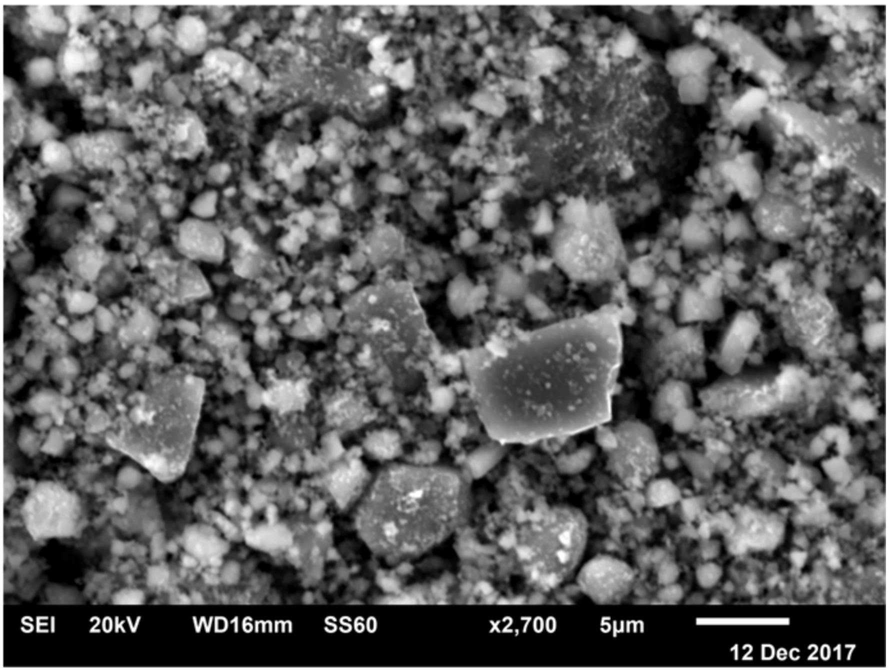

Figure 1. Microstructure of Si-B-C powder composition after 5h MA process

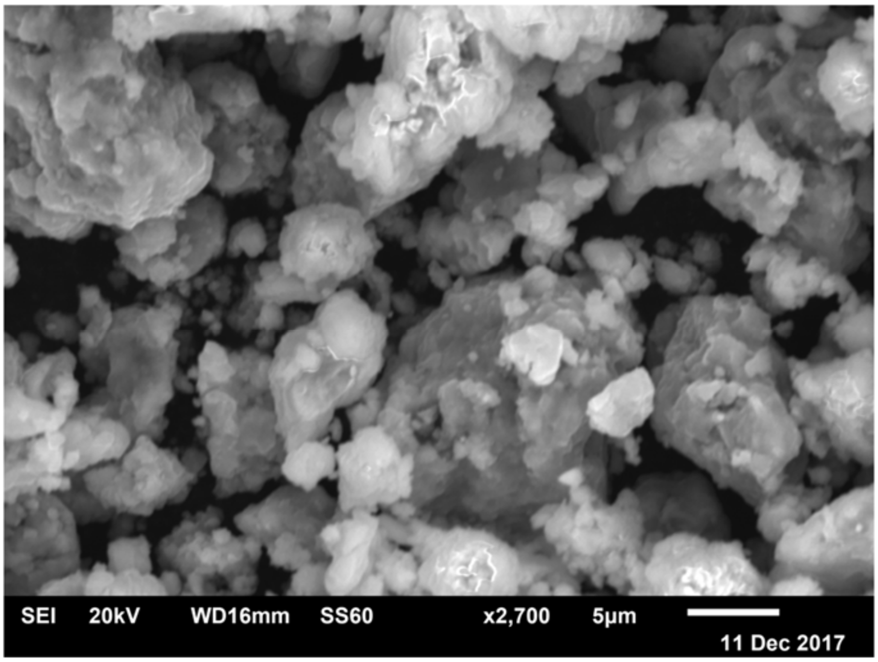

Figure 2. Microstructure of Ti-Al-B-C powder composition after 5h MA process

Preliminary works showed that the Shock wave compaction of metal-ceramic compositions is not only feasible but can produce materials of almost theoretical densities [6,7]. It was clear that the preliminary ball milling (due to fragmentation, mechanical alloying and critical reduction of particles sizes) should significantly increase the sintering ability of the blend and improve the compacting process and mechanical alloying of selected powder compositions. The major advantages of Shock wave compaction for bulk nanomaterials production are realization of high pressure, short processing time, and super high cooling rate (adiabatic cooling). The shock wave loading of high exothermic reactants allows generating in situ process of shock wave induced syntheses + shocking consolidation.
Consolidation of the samples was performed in two stages. The powder blend was loaded in the steel tube container (Figure3-c) and at the first stage the pre-densification of the mixtures was performed under static press loading. Cylindrical container/tube was closed from the both sides. A card box was filled with the powdered explosive and placed around the cylindrical powder container (Figure 3-a,b). The experiments were performed at room temperature. The shock wave pressure (loading intensity) was varied in the range of 5-20 GPa. The explosive was detonated by electrical detonator.

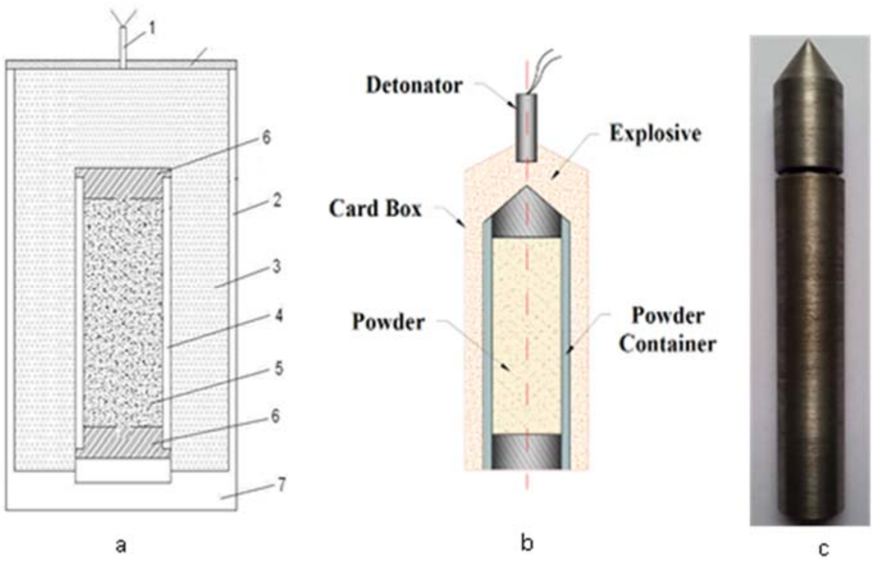

Figure 3. a) Schematic view of assembly for fabrication bulk rod: (1) electrical detonator, (2) explosive's container, (3) explosive, (4) steel tube, (5) reaction mixture, (6) steel plugs, (7) base table, (8) detonator seizer;
b) Schematic view of assembly before EC; c) Steel Container

The compaction experiments were performed at the underground explosive chamber. For shock wave generation (explosive compaction experiments) the industrial explosives were used in the experiments. The compacted samples were recovered, mechanically processed, cut and prepared for the structural investigations.

3. Results and discussions

The formation of bulk nanocomposites from nanopowders requires the selection of the compaction technological parameters. For the selection of these main parameters the following three main factors were considered for optimization of EC regime: 1. selection of explosive, mass and geometry; 2. Selection and determination of powder container parameters; 3. powder related parameters (composition, charging density, particle sizes and their distribution). Number of investigations was dedicated by several researchers to investigation of shock compaction process. Professor R. Pruemmer [10] performed detailed analysis of explosive compaction of powders. But, for explosive compaction of nanopowders (with dramatically changed free surfaces, reactivity and exothermal rate) the topic under discussion requires additional experimental investigations for obtaining accurate data on technological parameters. Selection of the container material for particular cases needs the detailed investigations as well. For selection of container parameters in cylindrical axis symmetric experimental set up the criteria for selection of container (internal diameter d and wall thickness δ) can be expressed in the following form:

$$A < Ep + Econ; Edestr. = Adestr. \times M; M = \rho \times \pi h [d2 - (d-2\delta)2]/4 = \rho \times \pi h \delta (d - \delta), \quad (1)$$

Where: A – full energy of explosive; Ep - energy consumed on plastic deformation of the container; Econ.- energy consumed on consolidation of the powder; Adestr - energy for full destruction of material's unit mass; M – mass of container; ρ-density of material, h-length of cylindrical tube.

The optimal shock wave loading pressure is varied in the range of (7-10) GPa. In these conditions the configuration of loading/unloading waves in powder and container allows one to initiate the syntheses in the reaction mixture, to simultaneously consolidate it and to fix the phase composition under adiabatic cooling. The SEM images of Si-B-C and Ti-Al-B-C compositions are shown on Figure 4 and Figure 5.

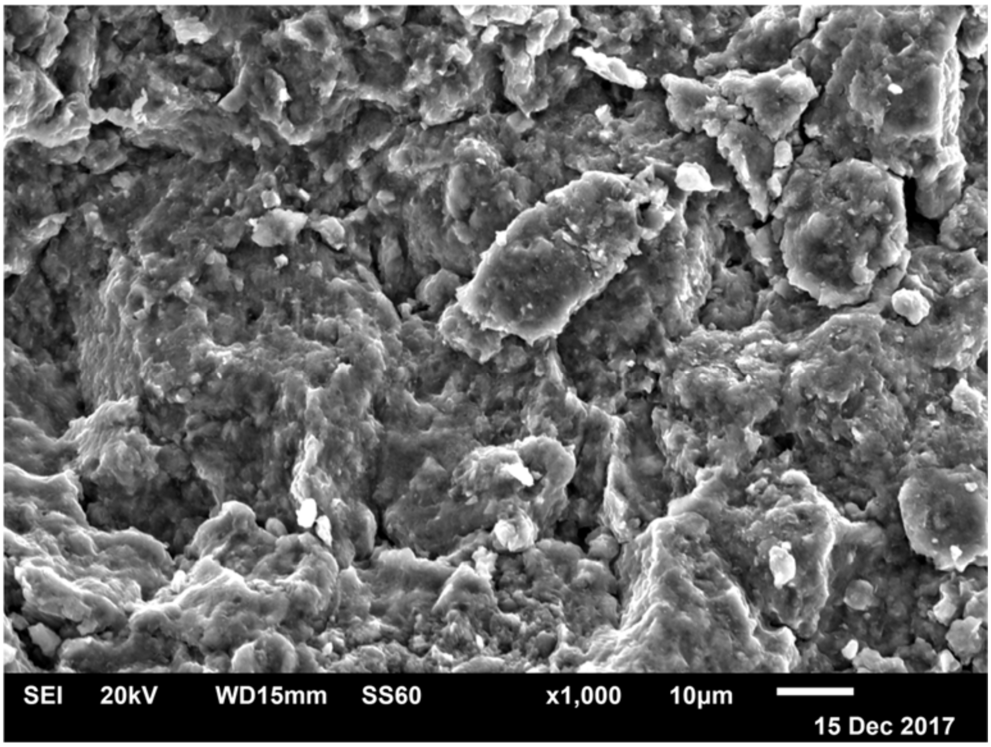

Figure 4. The microstructure of Si-B-C composition after shock wave consolidation

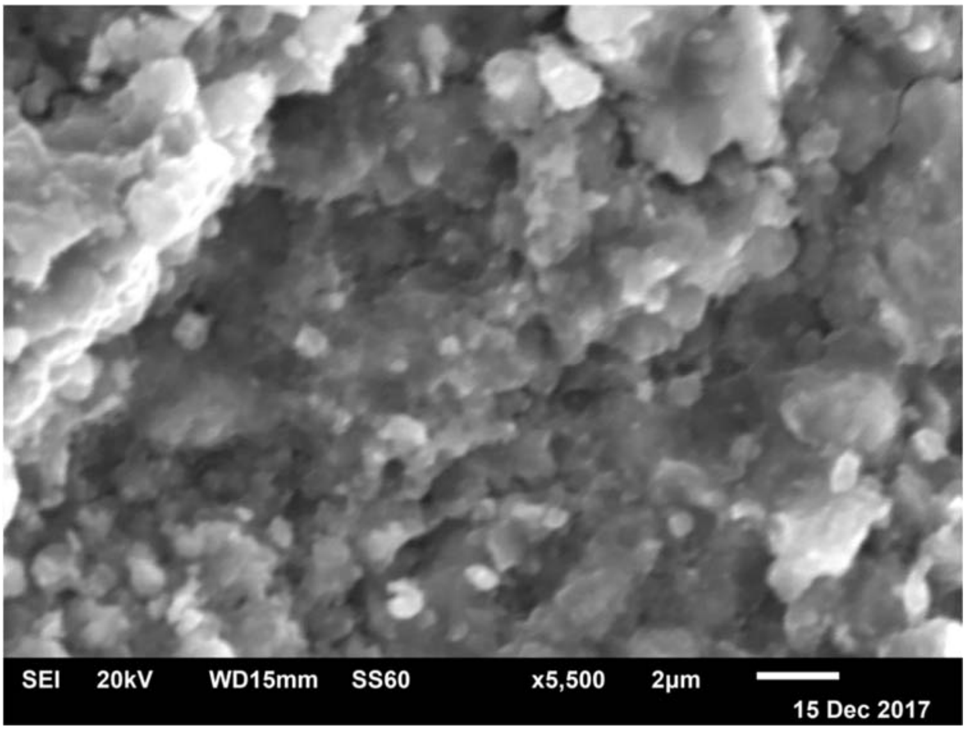

Figure 5. The microstructure of Ti-Al-B-C composition after shock wave consolidation

The results of SEM investigations confirm the successful realization of synthesis of compositions during MA and shock wave composition. The results of structural investigations give possibility to have evidences of the reduction of grain sizes in structure and amorphization of structure is observed as well. As a result, the main objects of the investigations were realized: a) the ball milling technology/MA for obtaining nanopowder materials as precursors for synthesis of bulk materials and b) Shock wave Consolidation technology for fabrication of the bulk nanostructured materials.

4. Conclusions

Ultrafine powders of Si-B-C and Ti-Al-B-C system were prepared for Mechanical alloying and realized in planetary ball mill with Zirconium oxide chars and balls. The duration and optimal rotation speed were established experimentally. For the consolidation of powders, the shock wave compaction technology was selected. For preliminary selection of explosives and configuration around the sample, computer modeling was used. The calculated results were validated experimentally.
The optimal shock wave loading pressure was selected and tested experimentally. The pressures are generated by the selected explosive materials, which provide to initiate the syntheses in the reaction mixture, to simultaneously consolidate it.

The microstructure and particle sizes were studied after MA and before shock wave compaction by using the Scanning Electronic Microscope. The microstructure and particle sizes were studied after shock wave compaction using the Scanning Electronic Microscope. By preliminary investigations is established, that the structure of bulk samples is not uniform. Structure is presented by nanosized and coarse grains. The effective technology/regimes for obtaining nanopowders and nanocomposites in Si-B-C and Ti-Al-B-C compositions has been elaborated and the rational Compaction technology for fabrication of bulk nanostructured materials by shock wave induced syntheses has been selected.

Acknowledgment(s)
This work was supported by Shota Rustaveli National Science Foundation (SRNSF) [grant #218008, Project Title: "Syntheses of Ceramic Nanocomposites in Si-B-C System by Mechanical Alloying and Explosive Compaction"]

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