ARPHA Proceedings

Proceedings CAUSummit 2024, 0272-0277
doi: 10.3897/ap.7.e0272

World Summit: Civil Engineering-Architecture-Urban Planning Congress

Structure and Composition of Ceramic Materials Obtained by SHS Compaction of Mechanoactivated Chasms of Ti-BN, Ti-BN-C and Ti-B_4C Compositions in Ti-B-C-N System

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Abstract. Using the original technological scheme of SHS-compaction, the scientific group of the Institute of Metallurgy and Materials Science obtained synthetic composite ceramic materials "constructed" from hard Synthetic Ceramic Composite Materials (SCCM) in Ti-B-C-N system: SCCM-1 on the basis of Ti-BN composition, SCCM-2 on the basis of Ti-BN-C composition and SCCM-3 based on Ti-B_4C composition. By changing the concentration of the initial components, it is possible to change the phase composition of the final product.The synthesis process can be conventionally divided into three parts: the heating zone, the active interaction or heat release zone and the final product formation or crystallization zone. In the heating zone, titanium melting and capillary flow begin in one case on boron nitride particles, in the second case on boron nitride and carbon particles, and in the third case on boron carbide particles. In the next zone, the zone of active heat release, there is intense interaction between titanium and boron nitride in one case, between titanium carbon and boron nitride in the second case, and between titanium and boron carbide in the third case. From all three components of the Ti-B-N-C system, Ti-BN, Ti-BN-C and Ti-B_4C ceramic materials with SHS compaction technology, in the production of synthetic ceramic composite materials (SCCM) and products from mechanoactivated chasm, determining the optimal technological parameters is of particular importance. These parameters are time and pressure characteristics and are determined experimentally. The study of the phase structure and microstructure of the material allows to specifically determine the areas of possible use of the material and to make conclusions about its operational properties under extreme conditions.

Keywords: SHS compaction, synthesis, ceramic materials

Published by CAUSummit and peer-reviewed under the responsibility of “World Summit: Civil Engineering-Architecture-Urban Planning Congress - CAUSummit-2024”.

INTRODUCTION

Using the original technological scheme of SHS-compaction, the scientific group of the Institute of Metallurgy and Materials Science obtained synthetic composite ceramic materials "constructed" from hard Synthetic Ceramic Composite Materials (SCCM) in Ti-B-C-N system: SCCM-1 on the basis of Ti-BN composition, SCCM-2 on the basis of Ti-BN-C composition and SCCM-3 based on Ti-B_4C composition [1].

Below are the possible chemical reactions between the initial components and the possible phase composition of the final products:

$Ti+BN=TiB_2+TiB+TiN+Ti_2N+Q$
$Ti+BN=TiB_2+TiN+Q$
$Ti+BN+C=TiB_2+TiB+TiC_XN_Y+Ti_2C_XN_Y+Q$
$Ti+B_4C= TiB_2+TiB+TiC_X+Q$

By changing the concentration of the initial components, it is possible to change the phase composition of the final product. The synthesis process can be conventionally divided into three parts: the heating zone, the active interaction or heat release zone and the final product formation or crystallization zone [2].

In the heating zone, titanium melting and capillary flow begin in the first case on boron nitride particles, in the second case on boron nitride and carbon particles, and in the third case on boron carbide particles. In the next zone, the zone of active heat release, there is intense interaction between titanium and boron nitride in the first case, between titanium carbon and boron nitride in the second case, and between titanium and boron carbide in the third case.

By considering the particles' interaction, it can be assumed that the formation of the final product formation occurs in the first case by the interaction of boron contained in boron nitride with molten titanium, resulting in the decomposition of boron nitride with the release of active nitrogen and its interaction with titanium. And, titanium borides and nitrides are obtained. In the second case, the synthesis process is carried out by the interaction of boron and carbon particles contained in boron nitride with molten titanium, and the active nitrogen separated as a result of the decomposition of boron nitride with the formed titanium carbide, as a result, Titanium borides and carbo-nitrides are obtained. In the third case, the synthesis process is carried out by the interaction of boron and carbon contained in boron carbide with molten titanium, titanium borides and carbides are obtained.

In the end zone of the reaction, practically all titanium is transferred to a finely dispersed state and is present in the form of titanium borides, nitrides, carbides and carbo-nitrides. In the crystallization zone, the structure of the final product is formed, in this zone the growth of grains and the final formation of the structure take place.

EXPERIMENTAL STUDIES, RESULTS AND DISCUSIONS

From all three components of the Ti-B-N-C system, Ti-BN, Ti-BN-C and Ti-B_4C ceramic materials, with the SHS-compaction technology, in the production of synthetic ceramic composite materials (SCCM) and products from mechanoactivated chasm, determining the optimal technological parameters is of particular importance. These parameters are time and pressure characteristics and are determined experimentally. As a result of the correct selection of these characteristics and the influence of mechanoactivation (The chasm is additionally mechanized in a high-energy planetary mill at a speed of 900 rpm for 3 hours, the ratio of the weight of the mixing balls to the weight of the chasm is 6/1) in the Ti-B-N-C system, compacted (porosity of 2% and 3.5% and 3% respectively, with a hardness of 92.3 HRA and 93.2 HRA and 93.5 HRA respectively, with density of 4.5 g /cm^3, 4.7 g/cm^3 and 4.73 g/cm^3 respectively) materials are obtained.

These materials can withstand a single dynamic impact with energy of 18000-20000 J, with a thickness of ~11.5-12 mm of the product's ceramic layer and, accordingly, at a value 68-65 kg/m^2 (6.8-6.5 g/cm^2) of the product, or a dynamic impact with an energy of 27000-30000 J, at a thickness of ~15-15.5 mm of the ceramic layer of the product and, accordingly, at a value 80-85 kg/m^2 (8.0-8.5 g/cm^2) of the product.

A thorough study of the samples obtained (synthesized) by SHS-compaction technology showed (Figure 1, Table 1) that the material obtained from the Ti-BN composition in the Ti-B-C-N system contains two grain types: columnar or needle-like and round or spherical grains. The chemical micro-analysis done by means of an electron probe proved that columnar/needle-like grains belong to the phase of the Ti-B-system, and the round or spherical grains belong to the phase of the Ti-N-system.

A thorough study of the microstructure and composition of the material obtained from the Ti-BN-C chasm in the Ti-B-N-C system showed that this material also consists of two types of columnar or needle-like and round or spherical grains. The chemical microanalysis made by means of an electron probe showed that the columnar or needle-like grains belong to the Ti-B-system phase, and the round or spherical grains belong to the Ti-C-N-system phase (Figure 2, Table 2).

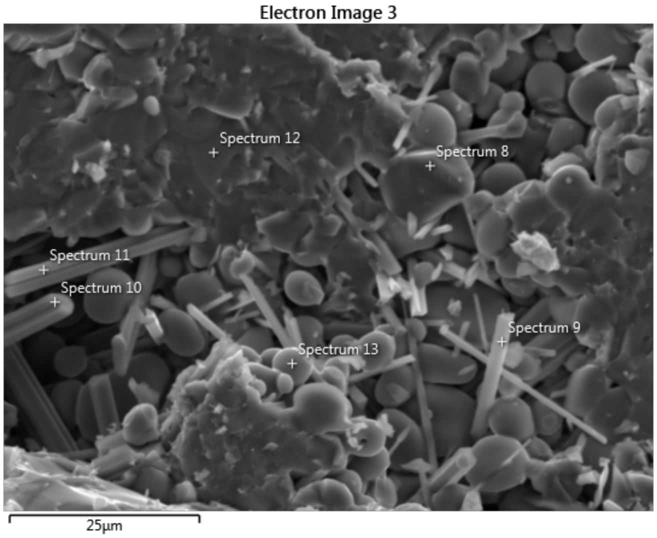

FIGURE 1. The photo from the microstructure of the material obtained in the Ti-B-N system by microanalysis determined at appropriate points.

TABLE 1. Spectral analysis results for the material obtained in the Ti-B-N system by microanalysis determined at appropriate points.

Result Type	Weight %					
Spectrum Label	Spectrum-8	Spectrum-9	Spectrum-10	Spectrum-11	Spectrum-12	Spectrum-13
B	1.96	5.24	22.37	17.01	0.95	2.30
C	4.03	2.52	3.98	3.36	5.42	6.67
N	5.26	1.88	0.00	0.00	9.20	12.10
O	0.63	2.26	0.00	0.00	1.37	2.06
Si		0.16				
Ti	88.12	87.51	73.65	79.63	83.07	76.86
Cu		0.43				
Total	100.00	100.00	100.00	100.00	100.00	100.00

The microstructure of both materials in Figure 1 and Figure 2 is quite finely dispersed, this applies especially to the columnar or needle-like grains obtained on the basis of the Ti-B system, which are several hundreds of nanometers in size. Experience shows [3,4], that the good resistance of the materials obtained by self-propagating high-temperature synthesis in the Ti-B-N and Ti-B-C-N system – SHS-compaction technology, to high-intensity dynamic loads and shocks, can be explained by the phase structure and microstructure of the material, which in turn leads to the accumulation of destructive energies and, as a result, to the increase of the material's resistance.

It should be assumed that upon penetration of identifiers with destructive energies into the samples made of the materials presented above, columnar or needle-like grains are first broken. These grains are higher ductility carriers than rounded or spherical grains based on TiN and TiCN phases, which in turn are carriers of higher viscosity and plasticity than grains based on TiB_2. During the crushing of columnar or needle-shaped grains, a certain part of the destructive energy is accumulated, however, in the first stage, the TiN and TiCN-phase is not disturbed, because it has a higher viscosity and plasticity. As a result, the propagation of the crack and its crushing in the sample is delayed, which in turn leads to an increase in the cracking resistance and life expectancy of the product.

It should be noted that the composition $Ti\text{-}B_4C$ was calculated in such a way that the presence of only two phases titanium boride TiB and titanium carbide TiC was expected, however, as thermodynamic calculations and also the analysis of the results of the research of the structure and construction of the samples obtained as a result of the experiments showed us that the material consists of: titanium from boride TiB, titanium diborideTiB_2, titanium carbide TiC and their solid solutions. This inconsistency is explained by the fact that, firstly, titanium and boron are thermodynamically more attracted to each other than titanium and carbon, that is, the probability of formation of titanium borides (TiB, TiB_2) is higher than that of titanium carbide - TiC. However, in the Ti-C system, titanium and carbon have a large margin of homogeneity. Presumably, TiC is present in a non-stoichiometric form, the lattice does not change, but the lattice parameters change, which in turn is confirmed by a slight shift of the peaks of titanium carbide on the X-ray image (Figure 3), which may also be caused by the partial dissolution of boron atoms in titanium carbide and the formation of solid solutions.

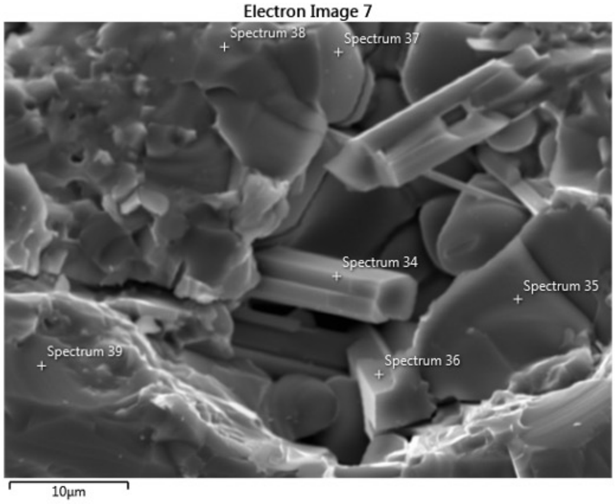

FIGURE 2. The photo from the microstructure of the material obtained in the Ti-B-N-C system by microanalysis determined at the appropriate points.

TABLE 2. The spectral analysis results for the microstructure of the material obtained in the Ti-B-N-C system by microanalysis determined at the appropriate points.

Result Type	Weight %					
Spectrum Label	Spectrum-39	Spectrum-34	Spectrum-35	Spectrum-36	Spectrum-37	Spectrum-38
B		25.10		22.65		
C	9.26	11.16	9.20	11.42	7.08	7.91
N	13.08		12.64		10.75	14.46
Ti	77.66	63.26	78.16	65.92	82.17	77.63
Cu		0.48				
Total	100.00	100.00	100.00	100.00	100.00	100.00

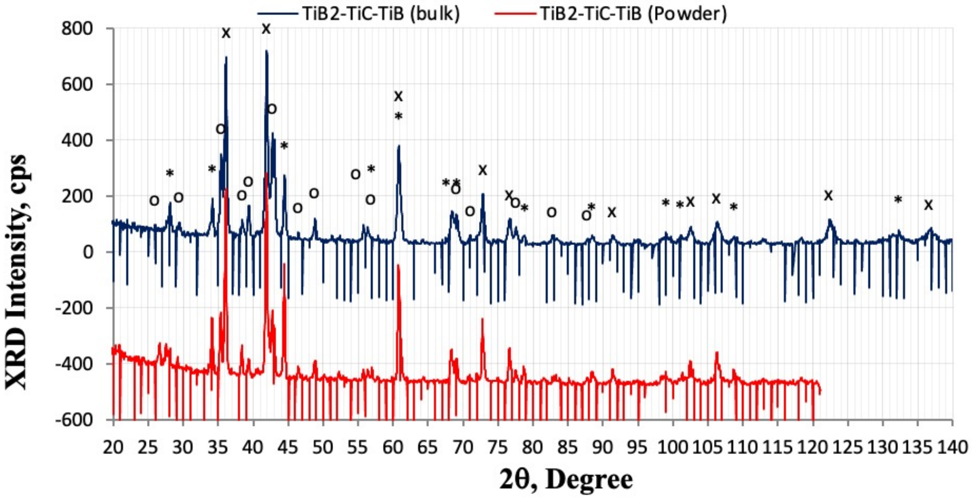

FIGURE 3. X-ray of Ti-B_4C composition 1.-Ti-82%(wt); 2.B_4C-18%(wt) + Ti 5% (wt) (X – TiC, o – TiB, * - TiB_2)

In addition, the most important thing to note is that the hardness of this material (which actually consists of TiB, TiB2 and TiC phases) (93-93.5 HRA) is higher than the hardness of the material (~88-90 HRA) estimated by the initial calculations (from TiB and TiC phases). This is probably because of the presence of the TiB2 phase in the material, which is characterized by a higher viscosity than TiB and TiC, as well as the presence of solid solutions in the material, which leads to an increase in both viscosity and strength indicators, which in turn leads to an increase in the resistance of the material to shocks and high intensity dynamic loads.

Figure 4 presents -Ti-82%wt. B4C-18%wt + Ti -5%wt; Spectral analysis of microstructure marked areas and distribution of elements on these areas, which are presented in different and different colors for visibility (Table 3).

The above assumption is well confirmed by the results of the spectral analysis (Table 3) and most importantly by the increase of the material's resistance to shocks and high intensity dynamic loads.

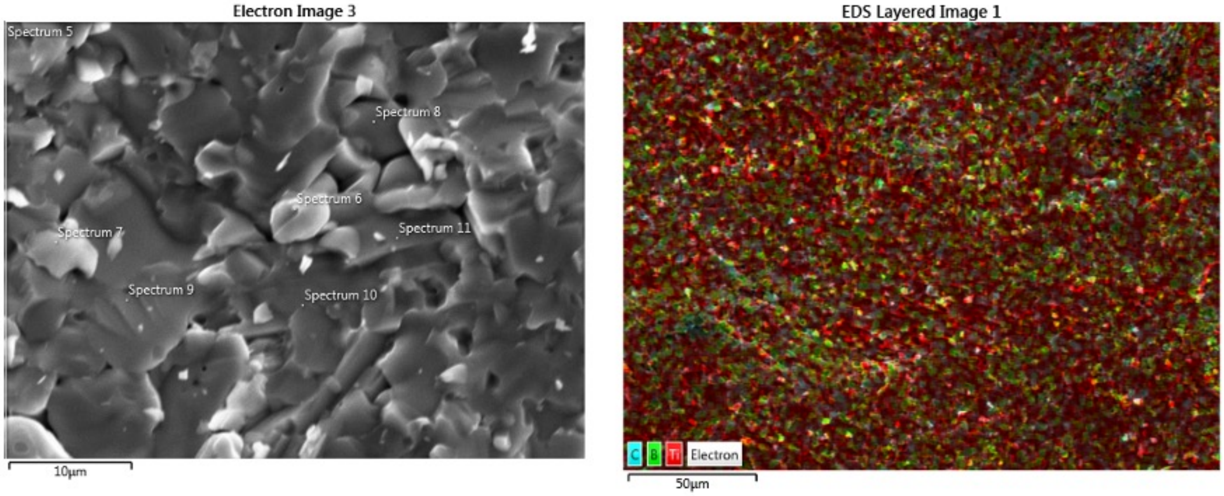

FIGURE 4. Spectrum analysis of Ti-82%wt. B_4C-18%wt + Ti -5%wt and distribution of elements

TABLE 3. The results of spectrum analysis of Ti-82%wt. B_4C-18%wt + Ti -5%wt.

Result Type	Weight %						
Spectrum Label	Spectrum-11	Spectrum-5	Spectrum-6	Spectrum-7	Spectrum-8	Spectrum-9	Spectrum-10
B	29.19	17.90	35.28		33.72		13.62
C		24.23	12.50	29.57	22.09	32.47	28.80
Al							0.20
Si		0.11					0.31
Ti	70.81	57.76	52.22	70.43	44.20	67.53	54.18
Fe							2.42
Ni							0.48
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Statistics	B	C	Al	Si	Ti	Fe	Ni
Max	35.28	32.47	0.20	0.31	70.81	2.42	0.48
Min	13.62	12.50	0.20	0.11	44.20	2.42	0.48
Average					59.59		
Standard Deviation					10.25		

Grain sizes are 2-3 µm, although there are areas where their sizes are ~0.5 µm. Electron probe chemical microanalysis showed that the grains mainly contain TiB, TiB2 and TiC phases, although it is likely that TiB and TiB2 grains contain different concentrations of carbon in the form of a solid solution, and TiC grains also contain boron in the form of a solid solution.

CONCLUSION

In accordance to the conducted experiments and investigation results, it can be concluded, that the study of the morphology, phase structure and microstructure of the material allows to specifically determine the possible areas of use of the material and to make positive conclusions about its operational properties under extreme conditions.

ACKNOWLEDGMENTS

This work was supported by Shota Rustaveli National Science Foundation of Georgia (SRNSFG) [Grant Number AR-22-955, Project Title: “Development of consolidated material containing of ceramic nanostructure and preparation of pilot samples based on Ti-B-N system”]

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