



SHS OF FINE-GRAINED CERAMICS CONTAINING CARBIDES, NITRIDES AND BORIDES

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Introduction

Synthesized by the SHS method hard alloys and composite materials based on carbides, borides and carbon-nitrides are characterized with high values of physical and mechanical properties as hardness, compression stress, wear-resistance, resistant to oxidation on air, resistance to aggressive areas.

However, their consumption is limited due to their brittleness, which make them inefficient to resist serious impact stress

Problem

Aforementioned disadvantage of the properties of hard alloys may be eliminated by means of obtaining gradient materials and/or by granulating hard alloy structure.

Due to specific conditions of obtaining hard alloys by the SHS-compaction technology, it is possible to obtain non-equilibrium structured, oversaturated solid solutions with fine grain 1 micron structure.

Investigations carried out in this work aimed the formation of fine-grain 1 micron and partially nano structure materials possessing the properties characteristic of hard alloys together with high impact viscosity. This in its turn will allow using the developed SHS materials for manufacturing the products which will operate in the conditions of highly intensive dynamic loading

Goal

One of the main goals of this work was to avoid applying deficient, expensive, limited materials and complicated technologies for obtaining high-quality, ceramic materials. In result the produce manufactured by our company will be cheap and easy-to-manufacture in both aspects - technological and economic.

In Ti-B-N-Me and Ti-B-N-C-Me systems the study of combustion regularities was carried out during alteration of the system parameters within a wide range. The synthesis was mainly implemented in the stationary regime.

The study of the synthesis regularities allowed us to make qualitative conclusions regarding the process specifics.

Formation process During SHS

In the warming zone starts melting of copper and titanium and capillary leaking on boron nitride particles - in one case and on boron nitride and carbon particles - in the other case. In the next zone, the zone of active heat emission, an intensive interaction takes place between titanium and boron nitride - in one case and between titanium, carbon and boron nitride -in the other. According to the particles interaction may be concluded that formation of a final product occurs in the first case - in result of interaction between boron of boron nitride with molten titanium, as a result, boron nitride is decomposed due to active nitrogen isolation and in result of its interaction with molten titanium there are obtained titanium borides and titanium nitride. In the other case the synthesis process takes place, by interaction boron of boron nitride and carbon particles with molten titanium, while after decomposition of boron nitride the yielded active nitrogen interaction with the formed titanium carbide; As a result titanium carbon-nitrides are obtained. In the zone of ending of reaction, practically all titanium is transformed into fine dispersed state and is represented by titanium borides, nitrides and carbon-nitrides. Hollow places are filled up by molten binding alloy. The structure of the final produce is formed in the crystallization zone, in this zone takes place growth of grains and final formation of the structure.

Selected system and Investigations

The study of combustion regularities in Ti-B-N-Me and Ti-B-N-C-Me systems was carried out while alteration of the system parameters within a wide range. Investigation of the synthesis regularities allowed us making qualitative conclusions on the process characteristics.

Experiments & Schemes

The experiments for defining dependence of the front movement velocity to the density of initial charge were held according to two schemes

According to scheme 1, the experiment was held in the open air in atmospheric conditions. During synthesizing the front movement direction coincided with the compression direction of the specimen preliminary formation.

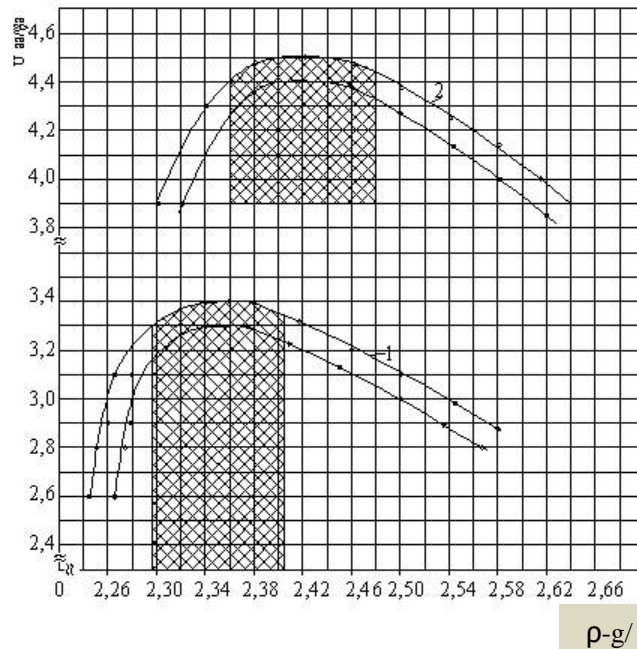
According to scheme 2, the specimen was placed in the press-form, under pressure, the value of which was corresponding to the compression value of the specimen preliminary formation. In this case, while synthesizing the materials the combustion front movement direction is perpendicular to the compression direction during its prior formation.

It was found that when obtaining materials by the second scheme the synthesis wave movement rate 2,5 times exceeds the synthesis rate of obtaining materials by the first scheme for the system Ti-B-N-Me and about 4 times - for the system Ti-B-N-C-Me.

Scheme 1

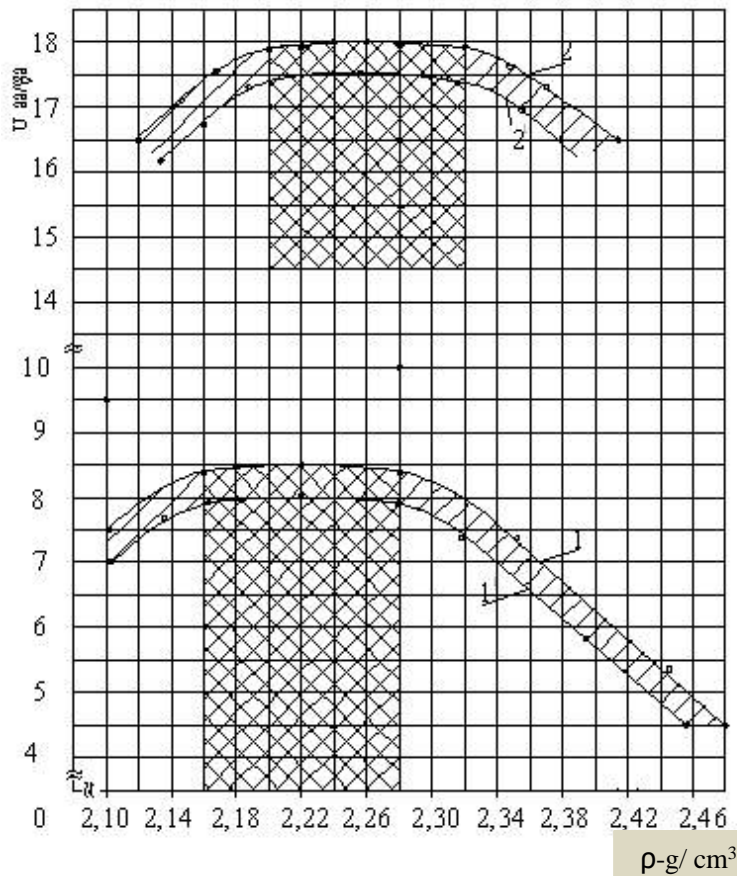
It should also be noted, that while synthesizing materials by the second scheme, maximums of dependences of the materials synthesis rates to the billet density are shifted towards lower values.

Scheme 1 and scheme 2 Ti-BN-Me curve 1 and Ti-BN-C-Me curve 2 show in the systems the dependence of the synthesis rate upon specimen density.



Scheme 1. Specimen diameter 20mm, height 20-30mm, Me quantity 0-10%

Scheme 2



Scheme 2 Ti-BN-Me system curve-1, Ti-BN-C-Me system curve -2 the model sizes 70X70 and the height 20-30mm

In Ti-BN-Me and Ti-BN-C-Me systems dependence of synthesis rate to initial billet density when in the charge inert component is within 0-10%.

Structural Investigations

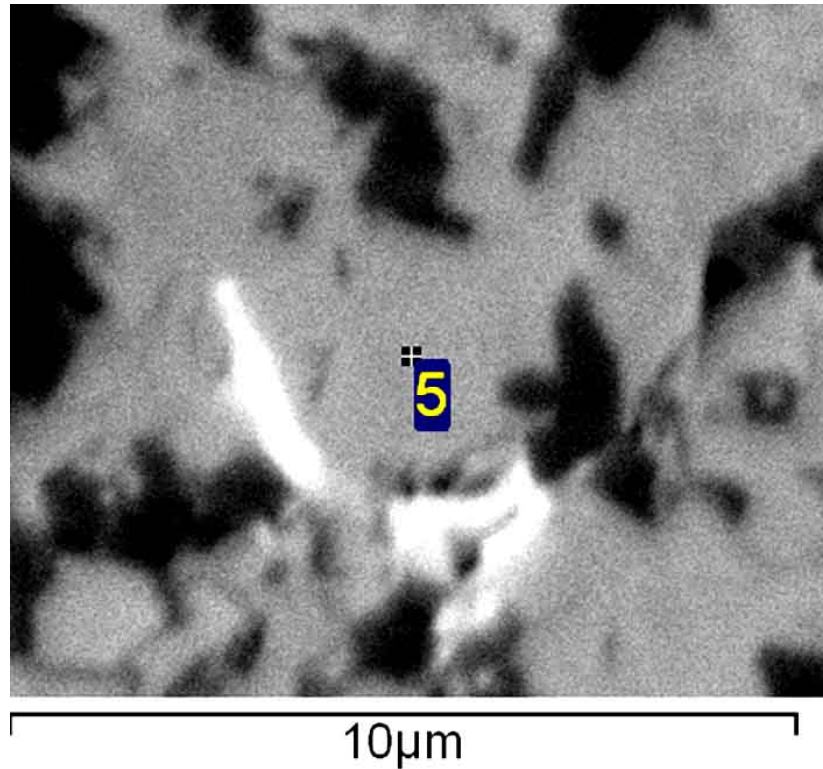


Fig.1 Ti-BN system microstructure. The image is obtained by means of reflected electrons “bse-topo”, so-called topographic contrast. ~90% of the material represents a hard ceramic nano structure (120-130nm) component upon the Ti-N (light color) and Ti-B (dark color) systems bases.

Structural Investigations

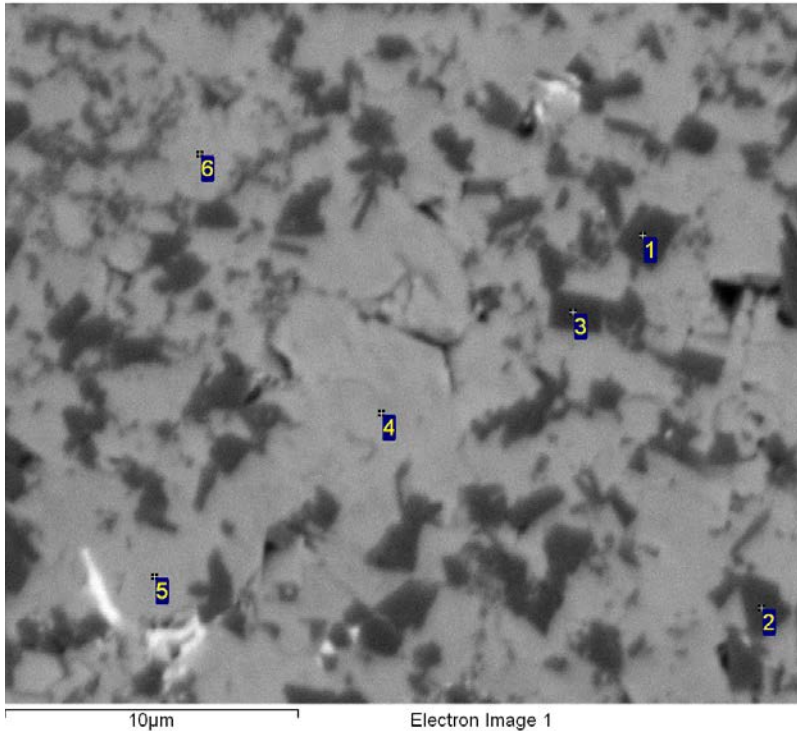


Fig.2 represents Ti-BN-Me microstructure. In the points on this microstructure, which are corresponding to various phases based on Ti-N and Ti-B systems and to fourth period d – element metal and/or the alloys upon their basis, the phases microanalysis are implemented by means of electronic probe and in each point there is defined the phases composition and the elements concentration, Table #1.

Table #1. Data in atom percentage

Spectrum	B	Ti	Spectrum	N	Ti
1	66.9	33.1	4	49.7	50.3
2	65.8	34.2	5	50.7	49.3
3	66.2	33.8	6	51.1	48.9
Medium	66.3	33.7	Medium	50.5	49.5
error	0.6	0.6	error	0.7	0.7

Fig. 2 Microstructure of the material obtained in Ti-B-N-Me system

Structural Investigations

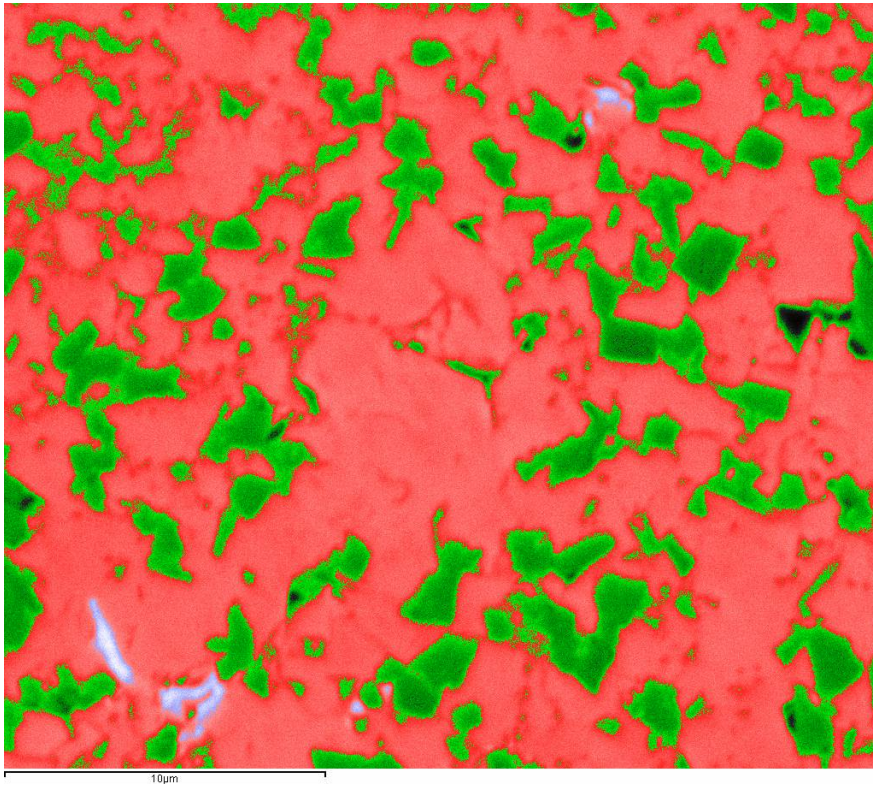


Fig. 3

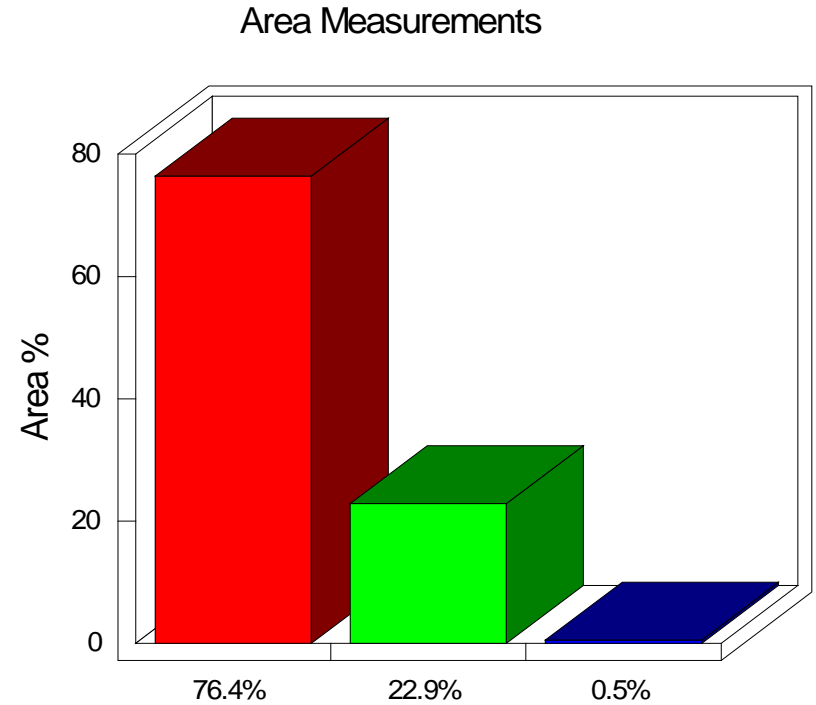


Fig. 4

In figures 3 and 4 there are shown as evidence different phases in various colors. In red - phase Ti_2N , in green - TiB_2 and in blue - copper.

In Fig.5 there is shown Ti-BN-C-Me microstructure. In the points on this microstructure, which are corresponding to different phases on the Ti-C-N and Ti-B systems basis and to the fourth period d-element metal and/or the alloys on their basis. The phases chemical microanalysis is made by means of electron probe and in each corresponding point there is defined the composition of these phases and the elements concentration. Table #2

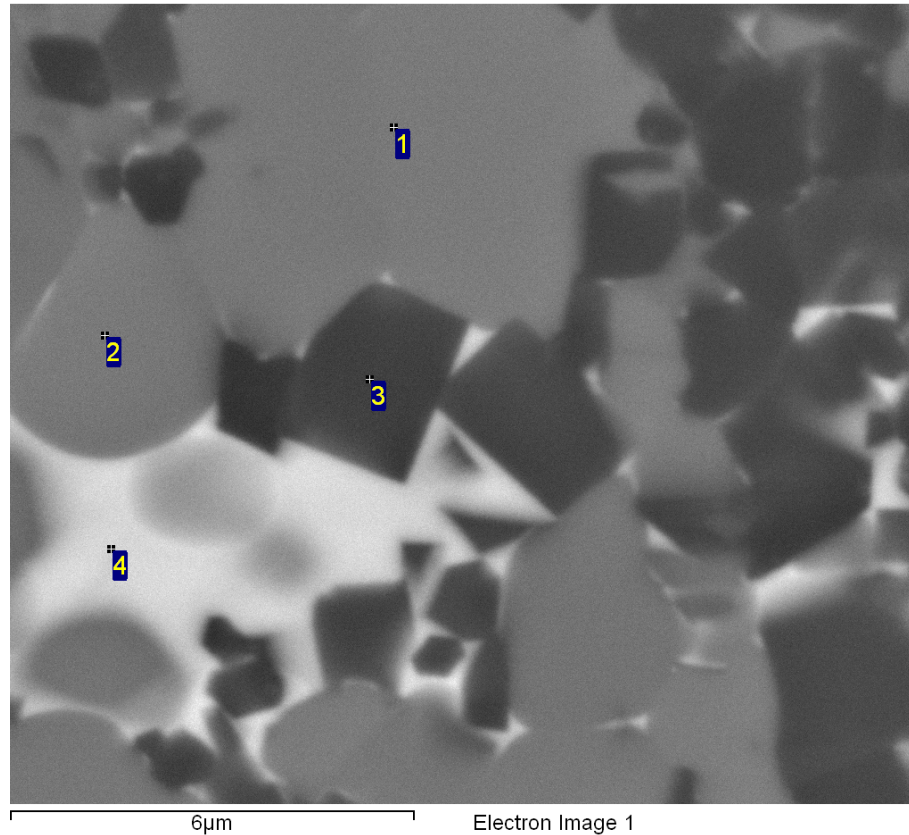


Fig. 5 Ti-BN-C-Me microstructure.

Table #2

Point 1

Element	Weight	Atom %
C K	7.1	21.3
N K	5.1	13.0
Ti K	87.8	65.7

Point 2

Element	Weight	Atom %
C K	6.6	20.1
N K	4.6	12.0
Ti K	88.8	67.9

Point 3

Element	Weight	Atom %
B K	27.7	62.9
Ti K	72.3	37.1

Fig 6 represents microstructure of Ti-B-N-C-Me system. The image is obtained by means of reflected electrons “bse-topo”, so-called topographic contrast. ~95% of the material represents a solid ceramic nano-structure (90-100nm) component based upon Ti-C-N (light color) and Ti-B (dark color) systems.

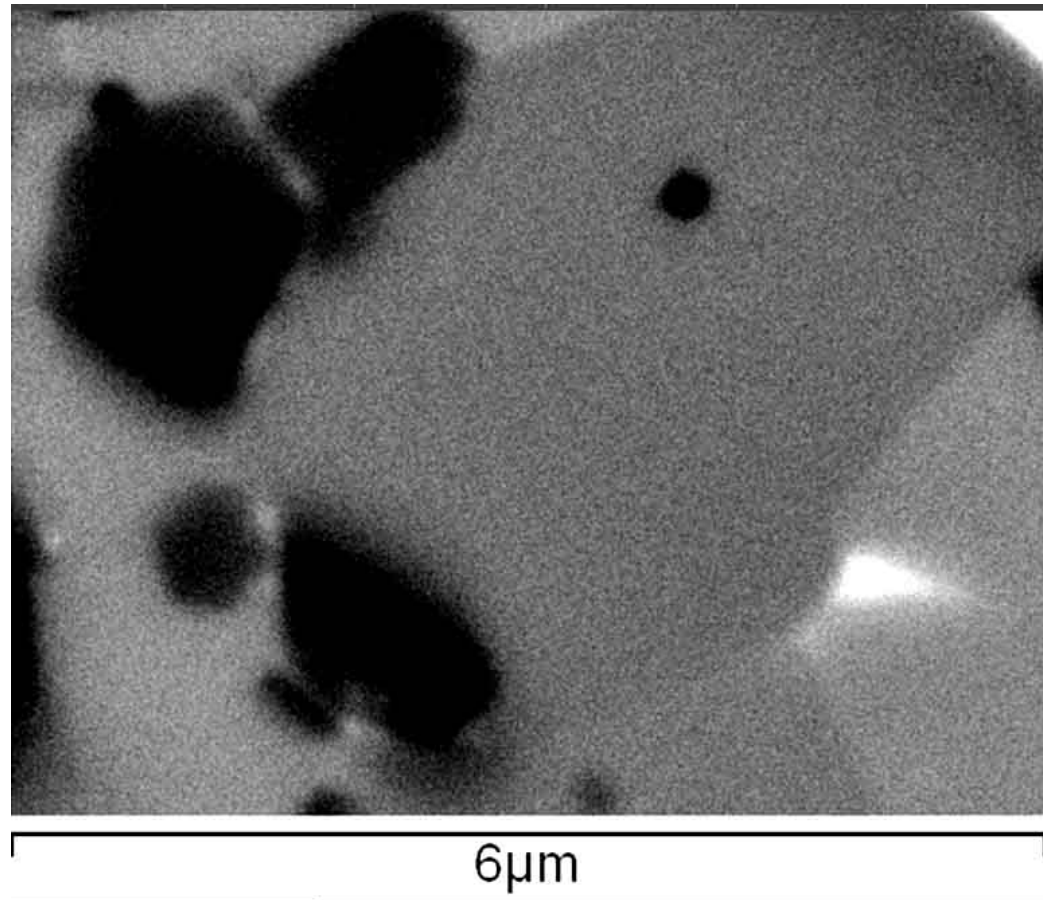


Fig. 6 Microstructure of the material obtained in Ti-B-C-N-Me system.

Conclusions

The results of these experiments clearly confirm the consideration aforementioned in this article as regards the ongoing processes taking place while the materials synthesizing, particularly, despite the fact that initially each component of the charge is solid, the character of the synthesis processing is not characteristic of the systems – solid-solid, because BN is decomposed during synthesizing, isolating/yielding gaseous active nitrogen. Therefore, when the experiments are held in atmospheric conditions by scheme 1 there occurs leakage of nitrogen from the reaction zone, causing lowering of the synthesis parameters approximately 2,5 times for the system Ti-B-N-Me and approx. 4 times for the system Ti-B-N-C-Me. As a result, while obtaining the material by scheme 1, there occurs reduction of the synthesis rate and temperature and lowering of nitrogen content in the material.

An analysis of the research results allowed drawing the following conclusion. While obtaining synthetic compact composite ceramic materials (SCCCM) by means of compression of synthesized hot SHS product, the most significant is to select optimal technology parameters. These parameters are time and pressure.

For investigation of phase and phase elemental composition of specimens there were carried out the SEM (Scanning Electronic Microscope) and ERMA (x-ray microanalyzer) analyses.

The investigation results are presented in the corresponding microstructures and X-ray spectrum diagrams.

While carrying out chemical microanalysis (EDS) and phase distribution qualitative analysis, for defining correctly light elements (B, N, C), electron diffraction was applied right in the microscope (Electron Back Scatter Diffraction-EBSD).

Thank You for Attention

