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# Superhard pcBN tool materials with Ti3SiC2 MAX-phase binder: structure, properties, application

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## Abstract

Superhard fine-grain ceramic cutters were sintered in cBN-(Ti3SiC2-TiC) system by high pressure high temperature (HPHT) method. Short-time (60 sec) sintering was performed under the pressure 8 GPa in temperature range of 1400-2400 °C. As initial mixtures three compositions were chosen with 87, 74 and 49m% of cBN. Initial mixtures were prepared by thorough mixing of cBN (1/3µm) and Ti3SiC2-TiC (0/2µm).

Final-samples composition in all cases got different from initial mixtures composition. The reason why is Ti3SiC2 decomposition and further reaction occurs in newly made system.

Microstructure observations, phase composition estimation as well as tests of mechanical properties of ceramic cutting tools were carried out. Obtained data appeared to be sustainably affected both by varying Ti3SiC2-TiC content and sintering conditions. Was estimated the way they both affect kinetic of reactions, recrystallization, ratio between cross-reaction products and how it does reflect on their mechanical properties at the end.

**Keywords:** Superhard material, ceramic cutting tool, cBN composite, MAX-phase Ti3SiC2, high pressure high temperature, HPHT

## 1. Introduction

Often-times the future application sphere for cBN cutting tool is already determined on the design stage. Determinative parameters are binding phase, grainsize. Binding phase which can handle high temperatures and high level of chemical and abrasive wear would be the lucky finding.

First discovered at 60-th, layered ternary carbides (so-called MAX-phases[1]) came back under the spot last decades. Being built of carbide blocks they are still not the same with normal carbides. What made them behave differently from normal carbides, and thus bring the intention of scientist to them, it's their structure. Structure does determinates their abilities to withstand elevated temperatures, tolerate high amount of stresses and poses with high compaction rate (link to the max spatial properties).

The highest MAX-phases compaction [i] rate [2] reachable slightly below their decomposition temperature.

Summarizing stated above. Principal structure-dependent properties of MAX-phases determinate them to be intermediate class between metals and ceramics.

Therefore, recent years, many research groups have been working on synthesis of superhard cutting tools based on diamond/cubic boron nitride with the commercially available MAX-phases as a binder. Field of our own interests belong to cBN-Ti3SiC2 system.

For cutting tool sintering plurality of techniques might be used. Started from hot pressing [1] at ambient pressure. SPS-direct transformations, hot pressing at pressure 20-35 MPa [2] can be successfully used. Promising results obtained when going up in pressure to GPa region with HPHT synthesis [3] [4].

But in all in-used techniques, only two approaches were applied.

Within the first approach binding phase consists of MAX-phase.

One way to get there – is to sinter MAX-phase binder from row materials while cutting tool is sintering. Most common technic for that are Hot Pressing and Spark plasma sintering.

In was established, that small additions of cBN doesn't inhibit the synthesis of Ti3SiC2. Influence of significant (more than 10%) cBN content was not investigated. Research made in this direction in diamond-Ti3SiC2 system shows that increasing of diamond content significantly promote Ti3SiC2 formation [6]. At the same time small additions of diamond affect in the same way with cBN additions. Ti3SiC2 synthesis has not got inhibited [5].

But here are some difficulties when hitting this way. One of them is that superhard phases starting to be unstable under conditions required for efficient MAX-phase sintering [5] [2] [7].

Another way – use as a binder preliminary sintered MAX-phase. In this case both MAX-phase binder and superhard phase need to be preserved unchanged while cutting-tool sintering. One of possibility to make it – is go up in pressure to GPa region. This option has got a benefits. Sintering time get decreased from hours to tens of minuets. Required temperature range also will get hundreds degrees smaller [8]. What is important, because decomposition of MAX-phases started at temperatures which are lower than temperatures for their sintering[3].

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[1] Layered, hexagonal carbides and nitrides with general formula: Mn+1AXn, (MAX phases) where n = 1 to 3, M is an early transition metal, A is an A-group (mostly IIIA and IVA, or groups 13 and 14) element and X is either carbon and/or nitrogen possessing with whole set of needed properties.

[2] Estimation of compaction rate via temperature was held for commercially available MAX-phases. They're Ti3SiC2 and Ti2AlC

[3] Apart from Ti3SiC2 case. It has got opposite dependency of decomposition from applied pressure [9]. Still the decomposition rate is very low.

Within the second approach binding phase consists of MAX-phase decomposition and cross-reaction products with cBN. Ti3SiC2 decomposition at ambient pressure occurs at the temperature region correspond to cBN to hBN transformation. This transformation need to be prevented in somehow. Applying high pressure (from GPa region) can stabilize cBN phase. Thus High pressure High temperature technique is commonly used with this approach.

Should be mentioned here, that in cBN-Ti3SiC2 system high pressure has got affect on both cBN and Ti3SiC2. Thermal stability of Ti3SiC2, uncovers strong inverse temperature dependence of segregation from applied pressure. With the pressure grow from ambient to P=5 GPa, temperature of Ti3SiC2 decomposition falls down from 1300 C to 800 °C [9].

This means - that already at the beginning of cutting-tool sintering (T=1400 °C) Ti3SiC2 decomposition is already running. Products of Ti3SiC2 decomposition and cross-reaction products with cBN create the binder.

Dependently of admixtures decomposition started earlier of getting the delay. TiC admixture improve temperature stability of Ti3SiC2 [10]. Dependently of sintering conditions TiB2, TiCx, $TiN_x$, SiB4 and SiC expected to be found [11, 4]. This phase composition similar to cBN-TiC and cBN-TiN commercial cutting tools. At the same time here is two important differences. Firs difference is that synthesis happens through Ti3SiC2 decomposition. What is promising in order to get cutting tool with very fine grain structure. The second difference is that cutting-toll getting multi-phase composition (5-6 phases), were all of phases are superhard.

Thus second approach seems more promising to get composite able to match better with the requirements putted forward novel cutting-tool material.

The main aim of this paper is to estimate the influence of ratios of initial components and sintering temperature on final product, cBN-Ti3SiC2-TiC based composite. Its microstructure, phase composition and phase stoichiometry as well as microhardness, fracture toughness, porosity and cutting performance of are planned to be established in current work.

## 2. Experimental procedure

### *2.1 Mixture preparation*

The ratio of initial components in the mixture determinative for a composite matrix.

We were aimed to sinter 3 types of composites. Matrix were expected to be: cBN matrix, inter-weaved cBN/Ti-Si-C matrix, Ti-Si-C matrix. To get to the aim - 3 different ratios of initial components were chosen for mixtures preparation.

***Table 1:*** *The ratios of initial components for mixtures preparation.*

| № of mixture | Initial components | Volume ratio | Mass ratio |
|---|---|---|---|
| 1. | cBN:Ti3SiC2:TiC | 90:6:4 | 87.3:7.61:5.09 |
| 2. | cBN:Ti3SiC2:TiC | 80:12:8 | 74.5:15.27:10.17 |
| 3. | cBN:Ti3SiC2:TiC | 60:24:16 | 49.1:30.54:20.34 |

The choice (Table 1) was base of the result of calculation via model of close-packing spheres. Calculation results found to be matches (cBN content) with commercial cutting tools composition.

On the role initial components of mixtures cBN (China 1-3 µm) and Ti3SiC2+TiC (commercial 0-2 µm) micro-powders were chosen. Ti3SiC2+TiC micro-powder has got Ti3SiC2 content 60%v. Also small amount of Graphite was in admixture.

Mixtures were prepared in two steps. On the first step of the mixing process dry mixtures of powders were performed in steel-metal sieves with sell ø=20 µm. Each powders mixture was triply rubbed through a sieve. On the second step wet mixing was done in “drunken mill” filled in 1/3 with $ZrO_2$ balls. Mixing was continued during 12 hours in isopropanol medium.

Purity of prepared mixtures was checked with XRD and EDX.

Mixing quality was estimated with SEM.

### *2.2. Sintering of samples*

The samples were sintered in toroidal type of high-pressure apparatus. Diameter of central hall ø=30 mm. Sintering conditions were determined by applied pressure, temperature, time and cooling and hitting modes. Alterable among them is only one. Temperature was changed during this set of HPHT experiments. The range of change 1400-2400 °C. The step 200 °C.

The procedure of sintering was the next. After pressure P=8 GPa was reached (about 5 seconds), heat got started immediately. Temperature rise up in the way to make from 20 °C up to sintering temperature in 2 sec. Than it held-on during 45 sec. Sintering is completed. Heat shutting down immediately. Decompression takes 5 sec. Sintered modules culled down to room temperature at the open air.

### *2.3. Samples characterization*

In current work samples characterization includes investigation of microstructure, phase composition and phase stoichiometry as well as estimation of microhardness, fracture toughness, density and cutting performance. Preparation for characterization got started from surface preparation accordingly to RNGN090300T. Next step was polishing with diamond suspension of 9 µm and 1 µm, followed by super-finishing with 40 nm SiO2 colloidal solution.

SEM, EDX, XRD (Cu irradiation), were used for microstructure, phase composition and phase stoichiometry investigation. Micro-hardness by Vickers was estimated using 500 gram-load. Fracture toughness was estimated using 30 kg-load. Hydrostatic weighing was used for porosity measurements.

### *2.4. Cutting performance*

Cutting performance was estimated through finishing/hard turning of cold work tool steel Vanadis 4E (HRC 59). Turning conditions were the next: speed – 150m/min; feed – 0.1 mm/revolution; depth of cutting – 0.2 mm; no cooling.

## 3. Results

### *3.1 Quantification of prepared mixtures*

Prepared mixtures appeared to have different mixing quality. Thus in the mixture with 87m% cBN content small amount (10%) of Ti3SiC2-TiC content got agglomerated. Size of agglomerates belongs to the range 5-15 µm. No Ti3SiC2-TiC agglomerates were found in mixtures with 74m% and 49m%

cBN content. For both mixtures with and without agglomeration the same grinding and fljaking of MAX phase was detected.

All 3 prepared mixtures appeared to be polluted. EDX detected presence of very small (≈ 0.2µm) ZrO2 inclusions. We believe grinding media became the source of ZrO2. No reactions between ZrO2 inclusions and surrounding material were observer. ZrO2 amount, size and dispersion of inclusions is about the same for all prepared mixtures. The amount of ZrO2 was not enough to be detected with XRD (***Fig. 1***). Considering everything mentioned above we wouldn't take ZrO2 presence into account further-on.

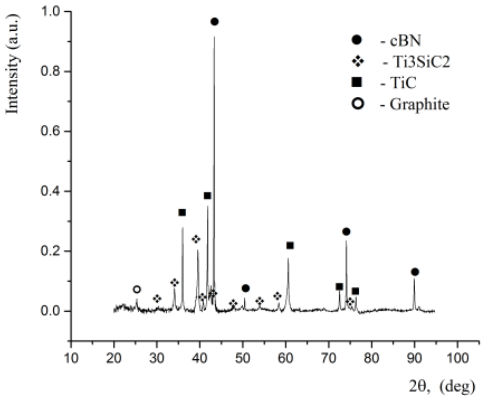

***Fig. 1:*** *XRD pattern of the starting powder mixture №3. Apart of expected phases in the mixture also small amount of graphite was detected.*

Already on the polishing stage samples show difference in properties. For some of them increased wear of non-cBN phase was detected. First time increased wear was found on the sample with 49m% cBN sintered at T=2000 °C. On the samples with higher cBN content increased wear became noticeable after sintering temperature raised up to T=2200 °C.

### *3.1 Phase composition of the samples*

Phase composition of sintered samples was determined by XRD. Phase composition of all of them is different from initial mixture. Firs difference is that one of initial components - Ti3SiC2, gone from all patterns. The second difference - couple new phases where detected (***Fig. 2***).

Some of new phases could be determined uniformly. Like TiSi2, SiC and TiB2. In $TiC_xN_y$ case stecxiometry changes with the temperature. Precisely it can be estimated only with EDX.

Tracing Si-containing phases while temperature grow up leads to interesting observation. After TiSi2 decomposition[4] at 1400 °C no Si-containing phases were found until 2000 °C. At 2000 °C SiC have been detected with XRD, but only in a small amount and only on 49m% cBN samples patterns. EDX in opposite to XRD-results shows presence of significant Si accumulations already at 1400 °C (***Fig. 3***). Their preferable location is the centre of the region formed by Ti3SiC2 decomposition-, reaction-products.

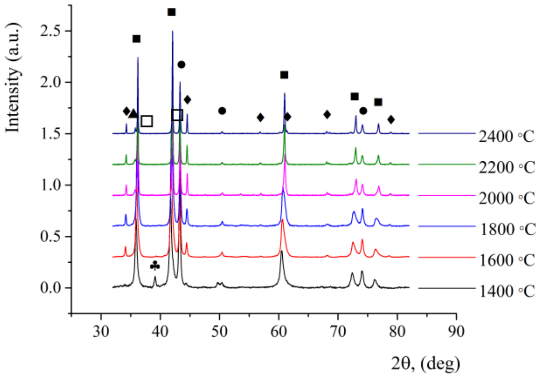

***Fig. 2:*** *Typical phase composition for samples sintered under different HPHT conditions. 49m% cBN samples patterns. List of the phases appeared to be found on the pattern: ■ – TiC; ▲ – SiC; □ – $TiC_xN_y$; ● – cBN; ♦ - TiB2; ♣ - TiSi2.*

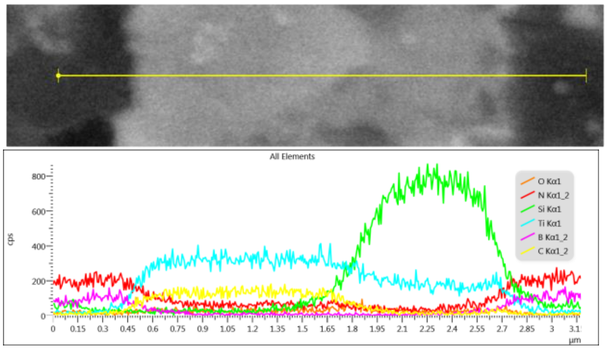

***Fig. 3:*** *EDX image of the sample with 74% cBN content. Sample was sintered under P = 8 GPa and T=1400 °C. In the central part of line scan is the region formed by Ti3SiC2 decomposition-, reaction-products.*

As it is seen from ***Fig. 4***, the situation doesn't get changed even at highest sintering temperature. Si location preferences are also not dependent of cBN content in initial mixture. This features are independent on starting mixture composition.

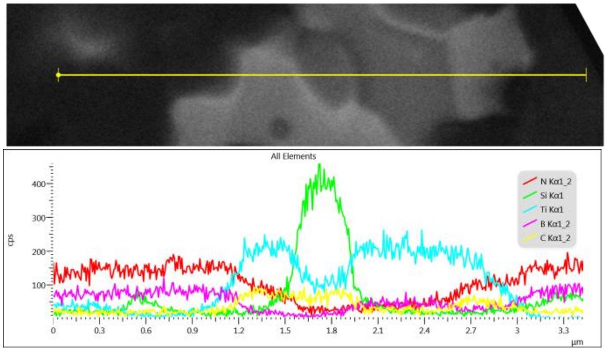

***Fig. 4:*** *EDX image of the sample with 87% cBN content in initial mixture. Sample was sintered under P=8 GPa and T=2400 °C. In the central part of line scan is the region formed by Ti3SiC2 decomposition-, reaction-products.*

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[4] It would be pertinently to mention here that in the case of sintering only TiC and TiSi2 containing mixture the maximum corresponding to TiSi2 could be observed up to 2200 0C.

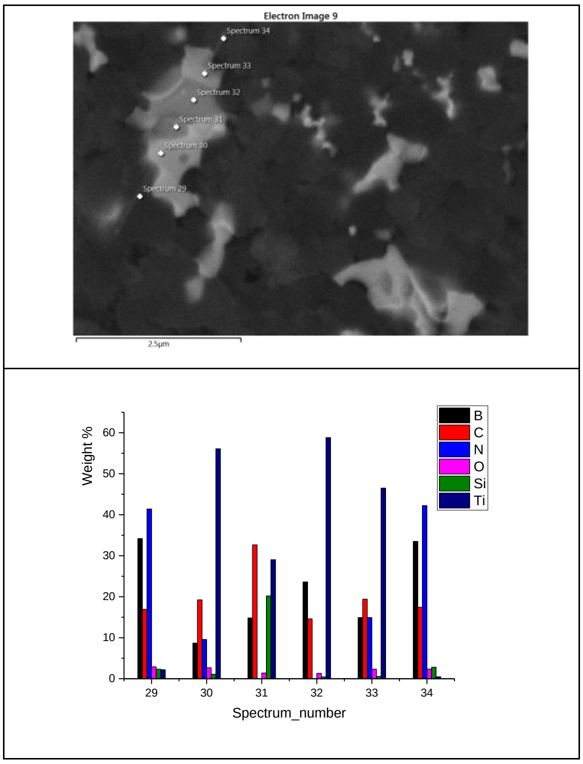

***Fig. 5:*** *EDX line scan from the region represented on* ***Fig. 4****.*

At the same time intensity of their occurrence increasing with decreasing of cBN content.

Similarly to Si, O in structure is visible only with EDX. While temperature rise from T=1400 to 1800 °C, O amount is staying almost at the same level. After 1800 °C O content drops down almost twice and stay on the same level further on.

### *3.2. Possible reactions*

Possible reactions (1), (2) in-between initial components under given sintering conditions were reported [11, 4]. In our case list of initial components slightly wider than the one from (2) reaction. We have graphite in addition (3). To prove that in our experiment reaction was running the same way with (2) We need to show that phase composition of our sample completely matches with (2).

$$TiC+Ti3SiC2 \rightarrow TiC+Ti_xC_y+TiSi2 \quad (1)$$

$$cBN+TiC+Ti_xC_y+TiSi2 \rightarrow$$

$$\rightarrow cBN+TiC+Ti_xC_y+SiC+Ti_zC_v(N,O)+TiB2+Si_{amorf} \qquad (2)$$

$$cBN+TiC+C_{grafite}+Ti_xC_y+TiSi2 \rightarrow$$

$$\rightarrow cBN+TiC+Ti_xC_y+SiC+Ti_zC_v(N,O)+TiB2+Si_{amorf} \qquad (3)$$

$Ti_xC_y$ - obtained after firs stage Ti3SiC2 decomposition. Expected stehiometry is about TiC0.67.

$Ti_zC_v(N,O)$ - created under HPHT from basic elements

No $C_{grafite}$ was found in any of sintered samples, therefore we believe that it became a part of one of C-containing phases. Non-stechiometric carbides $Ti_xC_y$ were found in region of Ti3SiC2 decomposition-, reaction-products. These carbides have got different values of x and y indexes, dependently of sintering temperature. Their size, morphology and location in the structure makes as able to distinguish $Ti_xC_y$ formed under high pressure from micron-sized TiC[5] sourced by initial mixture. Only very low reflection of SiC[6] phase were found on XRD ***(Fig. 2)***. Allot of Si appear on EDX line scan (***Fig. 5***). This point on amorphous nature of the most of silicon. TiB2 presence was confirmed via EDX. Elements-mapping ***(Fig. 6)*** uniquely determinate it's when on XRD its maximums overlope and got shadowed with another phases.

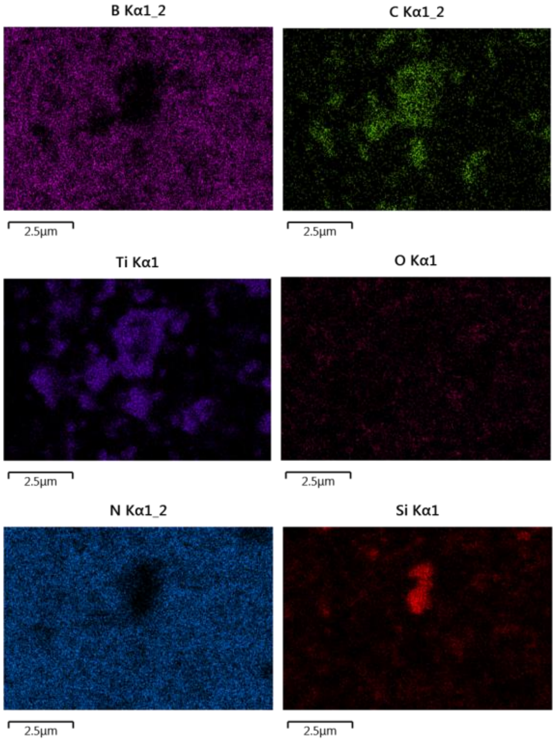

***Fig. 6:*** *EDX mapping of the sample with 74m% cBN in initial mixture. Sample sintered under P=8 GPa and T=2200 °C.*

From the same (***Fig. 5***) and (***Fig. 6***) we can see pretty high O content in the structure. At the same time any kind of oxides were found on XRD. It points on O presence in structure as admixture. EDX shows that O amount changes together with sintering temperature. It goes down almost twice when the temperature rises from 1400 up to 2400 °C. Such a behaviour is in nice correlation with ΔG (Ogas) change. It increasing 1.8 times while temperature grow from 1400 to 2400 °C.

### *3.2 Microstructure*

Matrix phase of sintered samples is determined by initial mixture composition. 87m% cBN content lead to formation of cBN-matrix. 74m% cBN lead to formation of mingled type of

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[5] $Ti_xC_y$ grains left after Ti3SiC2 decomposition are not carcass from were Si was leaked out. They get recrystallized as well (SEM)

[6] SiC maximum is present on XRD of the samples with low cBN content. EDX shows, that amorphous silicon has worked as a pump, pumping out carbon from the surrounding $Ti_xC_y$. After some level of SiC got created. In case, if Ti3SiC2 amount was lacking to create the agglomerates, carbon efflux occurred preferably toward cBN grains. As a result, SiC formation was slowed down.

matrix. 49m% of cBN containing initial mixture end up with formation of matrix which consists of Ti3SiC2 decomposition-reaction-products.

Microstructure of the samples mostly affected by sintering temperature. Both cBN phase and Ti3SiC2 decomposition- and cross-reaction products has got affected, but in a different way. Thus initial mixture composition also make its influence.

Temperature affect on cBN component in the next way.

Affect of temperature changes can be seen from *eatening-out* of the cBN grains at T=1400-1600 °C. At 1800 °C some grain boundaries became perfect. At 2000 °C boundaries became uncontentious. On the grain boundaries nano and micro-pores appears. At 2200-2400 °C pores grow and have tendency to move to triple grains junction. cBN grains gets rounded. No total recrystallization detected even at 2400 °C.

Mixture composition make influence on cBN behaviour in the next way. Process of *eatening-out* of cBN grains gets more prominent with cBN content grow. In the samples sintered from mixtures with 74% and 87m% cBN content at 2400 °C local cBN recrystallization is observed[7] (***Fig. 7***).

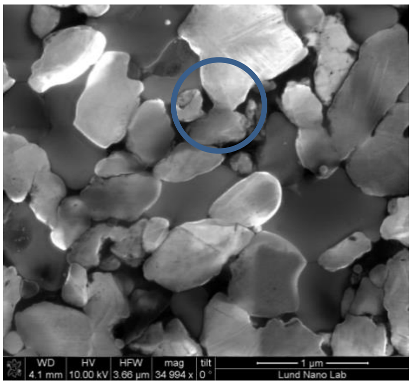

***Fig. 7:*** *SEM image of the sample with 74m% cBN in initial mixture. Sample sintered under P=8 GPa and T=2400 °C. With the blue circle recrystallized cBN grains are marked.*

This can be explained with decreasing of contacts amount, stresses and their equal-axial distribution inside the sample. On the decreasing of stresses twinning and micro-twinning reduction in cBN grains points on. Hexagonal shape of $Si_{amorf}$ agglomerates corresponds to equal-axial distribution of stresses.

Temperature affect on Ti3SiC2 decomposition-, reaction-products component is much more prominent then on cBN component. Morphology of this component changes in the next way.

At T=1400-1600 °C is the region with unclear submicron grain-structure (***Fig. 8***) and accumulation of amorphous Si in the centre (in most cases). At T=1600-1800 °C crystallization process begins from the region boundaries.

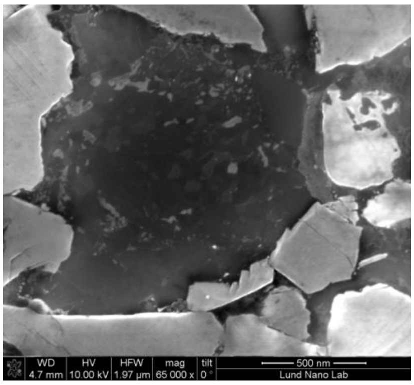

***Fig. 8:*** *SEM image of the sample with 40m% cBN in initial mixture. Sample sintered under P=8 GPa and T=1400 °C.*

Crystallized phases have dendritic nanoscale structure. Starting from 1800 °C spheroidal formations with size 5-20 nm appeared to be found. (***Fig. 9***). Phase composition of dendritic and spherical formation is not established. But EDX spectrum-scanning point on $Ti_xC_y$, TiB2, SiC, $TiC_xN_y$ formation. Further temperature rises up to 1800-2200 °C make dendritic structure disappear. It also makes grains-grow started and run local aggregative recrystallization. All those process continued at 2400 °C with local areas of perfect structure formation accompanied with pores grow at the triple grain junctions. Should be mentioned here, that this process was not completed. Small amount of spheroid regions inside normal grains are remained. Microstructure, which is formed by Ti3SiC2 decomposition-, reaction-products, demonstrate start of recrystallization about 200-300 °C earlier than cBN component.

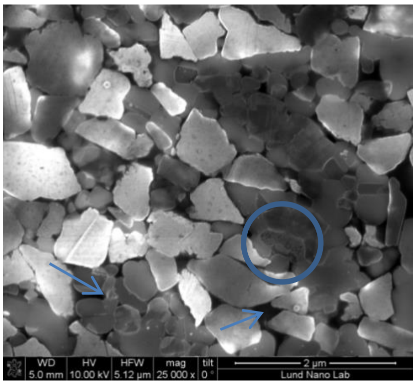

***Fig. 9:*** *SEM image of the sample with 40m% cBN in initial mixture. Sample sintered under P=8 GPa and T=2000 °C. With the blue circle spheroidal formations are marked. Arrows point on porous.*

Apart of light effect on phase transformation rate, which going to be discussed later, initial mixture composition has not got significant influence on second component behavior.

### 3.3 Mechanical properties

Densities of the sintered samples differs. Density depends of sintering conditions and initial mixture composition. The last is determinative for *ρ(T)* dependency behavior. This

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[7] at the same time very particular cBN grains kept their borders developed

dependency demonstrate nearly the same behavior for the samples with 87 and 74m% cBN-content in initial mixtures. But when it gets to 49m% cBN-content, *ρ(T)* dependency behave is completely different way (***Fig. 10***). Such density grow of the samples with high Ti3SiC2 content in the line with information about thermal conductivity grows with increasing temperature of sintering [12]

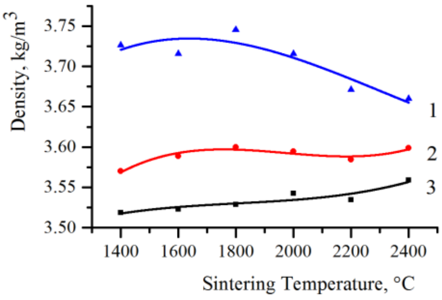

***Fig. 10:*** *Average density change with the sintering temperature change for the samples with:* ***1*** *- 87m% cBN,* ***2*** *- 74m% cBN,* ***3*** *- 49m% cBN.*

Microhardness and fracture-toughness changes follow the density change. They have got the similar trends. Only one but significant difference is that the samples with highest hardness posses with the lowest fracture-toughness, and another way around (***Fig. 11, Fig. 12***).

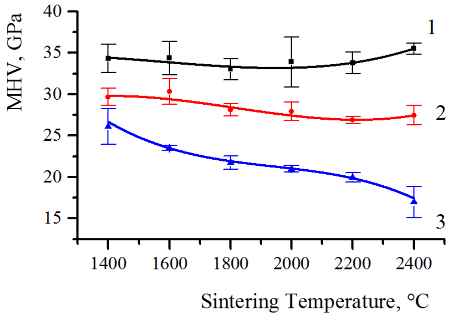

***Fig. 11:*** *Microhardness change dependently of cBN content in the sample and sintering temperature.* ***1*** *- 87m% of cBN;* ***2*** *- 74 m% of cBN;* ***3*** *- 49 m% of cBN.*

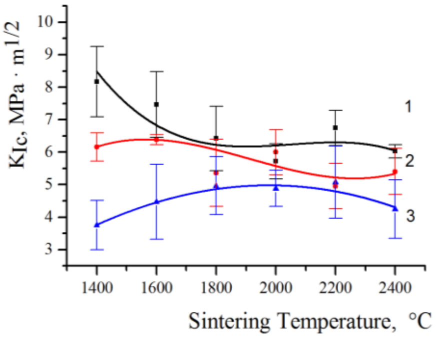

***Fig. 12:*** *Fracture toughness of the samples with different cBN content dependently of sintering temperature.* ***1*** *- 87m% of cBN;* ***2*** *- 74 m% of cBN;* ***3*** *- 49 m% of cBN.*

*3.4 Cutting performance*

Samples with the best mechanical properties were chosen for the estimation of working performance. Results of the cutting test are listed in ***Table 2***.

***Table 2:*** *Results of the cutting test for samples best mechanical properties. Their content, microhardness, fracture toughness*

| **cBN, m% and sintering T, °C** | **MHV, GPa** | **K1C, MPa·m$^{1/2}$** | **Depth of the crater, µm** | **Wear, µm** |
|---|---|---|---|---|
| 74m% cBN<br>T = 1600 °C | 30.3 | 6.4 | 55 | 157 |
| 87m% cBN<br>T = 2000 °C | 33.8 | 5.7 | 58 | 190 |
| 74m% cBN<br>T = 2000 °C | 30 | 6 | 83 | 269 |
| 49m% cBN<br>T = 2000 °C | 21 | 4.9 | 70 | 183 |
| 87m% cBN<br>T =2400 °C | 35.5 | 6. | 44 | 167 |
| 87m% cBN<br>T =2200 °C | 33.8 | 6.7 | - | - |

Accordingly to the measured values of fracture toughness and microhardness, three samples with 49, 74, 87 m% cBN were chosen for test-cutting. All those samples are sintered under T=2000 °C. Results of the cutting test are shown at the ***Fig. 13:***

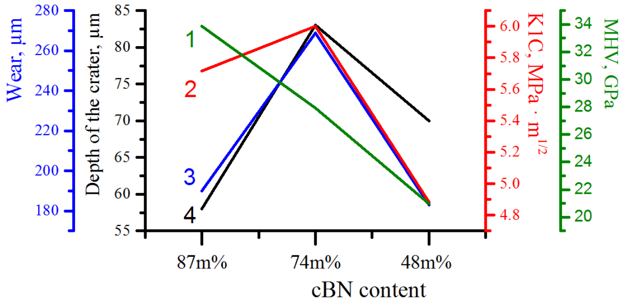

***Fig. 13:*** *Results of the cutting test for samples with 49, 74, 87 m% cBN. Also corresponding values of microhardness and fracture toughness are plotted. 1 – microhardness, 2 – fracture toughness, 3 – wear, 4 – depth of crater.*

## Conclusions

The phase composition of all composites sintered in temperature range of T=1800-2400 °C for all mixtures is the same. Only percentage ratio of phases and their stoichiometry has been changed.

Decreasing of cBN content in initial mixture results in:

- pore cluster destruction at lower temperatures.
- decreasing of contact stress and their equal-axial distribution inside the sample.
- inhibitory effect on the process of Ti3SiC2 decomposition, recrystallization and grain growth of decomposition- and reaction-products.

No massive cBN recrystallization has been observed. The sizes of cBN grains in the sintered sample smaller that initial cBN powder grain-size due to their crushing and due *eating-out* while reaction. Ti3SiC2 got totally decomposed.

Recrystallization and grain grows of its decomposition- and reaction-products results in sub-micron structure creation.

As the result sintered cutting tool material has got finer grain-size than initial mixtures had.

Hardening mechanism connected to process of twinning and micro twinning is the most prominent for samples with cBN matrix. To some extend it lead to increasing in hardness, but at the same time significantly reduces fracture toughness.

The best combination of mechanical properties cBN-Ti3SiC2–TiC cutting tools gains being sintered in temperature ranges T=1800-2200 °C. The value of temperature preferable for sintering grows up together with grows of cBN content.

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