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INFLUENCE OF STRUCTURE ON BRITTLENESS OF BORON NITRIDE COATINGS DEPOSITED ON CEMENTED NANOCARBIDES[1]

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Abstract

In the article is presented the brittleness study of boron nitride coatings deposited on cutting edges made of cemented nanocarbides by the pulse-plasma method (PPD). Influences of the structure (density, pores, microcracks) of coating material on the brittleness and on selected technological parameters of boron nitride formation by PPD method particularly taking into account discharge voltage on brittleness are shown. Differences between values of total average crack length ΣL, critical loads (P_{k300}, P_{k500}) and coefficients ($a_{1(300)}$ and $a_{1(500)}$) characterized susceptibility to cracking of investigated coatings manufactured using different values of production process were defined. Results of an investigations have been confirmed usefulness of Palmqvist's method for measurement of coating susceptibility to brittle cracking.

Key words: Wear-resistant coatings; PVD; Brittleness; Structure.

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1 INTRODUCTION

Coatings applied on cutting tools should be characterized among other properties by high abrasive wear resistance.[1-9] Abrasive wear resistance increases with hardness increasing. Many experiments have shown linear dependence between hardness and abrasive wear for different materials.[3,4,10-12] This relation however, according to work[9] not always is true. In this work is pointed out, that this relation is more complicated and another properties can be more important then hardness. Linear relation was confirmed in the works[4,11] for borides, oxides and silicates of transient metals, but for carbides of transient metals it was untrue. Deviation from linearity was explained by carbide brittleness leading to extremely low plasticity in cutting conditions. For example, borides possessing similar hardness exhibit higher abrasive wear resistance than carbides and silicates of transient metals because of lower brittleness.

On the base of many experiments it was observed that coatings during cutting not only underwent to abrasive wear.[4-8] Predominant role in their wear played the cracking and fragmentation of the coating.[4,5] Moreover, detaching of microflakes as the result of chip hooking on microcracks developed on the tool face as well as hooking of workpiece on tool flank is existed. Coating fragmentation is probably connected with their resistance to crack initiation and propagation.[4-9,13] Destructive influence of microcracks is especially dangerous in brittle materials where propagation rate is high. Brittle crack develops at minimal dissipation of energy due to the lack of plastic deformations. In materials of important plasticity a plastic flow is possible at the end of microcrack leading to material strengthening and in consequence to reduction of crack propagation rate.[13,14]

In this article the brittleness study of boron nitride coatings deposited by pulse-plasma method is presented. In cubic boron nitride coatings the rigid covalent bonds prevail resulting in a high hardness and unfortunately considerable brittleness. The basic problem was the influence of a coating structure on the brittleness.

2 MATERIALS AND METHODS

According to,[4,15] brittleness is defined as material property manifesting in cracking at low plastic deformation. Brittleness depends on the kind of bonding, type of lattice and density of defects as well as content of impurities.[13,16] Thus to determine the real brittleness of a material a theoretical analysis is insufficient. The experimental procedure should be employed. It is especially important in the case of coatings obtained by PVD methods due to their structural differentiation in function of manufacturing parameters and high density of defects. Coatings obtained by PVD method crystallize according to Movchan's-Demchishin's model for evaporation or Thornton's model for sputtering.[4, 8]

For a long time many investigators have tried to find a proper method for a brittleness evaluation of hard materials. A relation between brittleness and strength properties for cutting materials was not found. Even for the results of bending test of cemented carbides such relation appeared very weak. Finally it appeared that hardness measurements gave the best results. It was observed during Vickers hardness tests that cracks developed from the corners of an indent. In the papers[14,17-19] were reported that a relation should exist between brittleness and length of cracks.

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Palmqvist was the first investigator who proposed a method of brittleness measurement.[20] It is necessary to underline, that disadvantage of Palmqvist's method lies in difficulty of crack length measurement using optical microscopy technique due to a low deep of focus. To overcome this problem special grinding and lapping procedures were proposed. Finally this method of brittleness determination has been almost abandoned. The come back to Palmqvist's proposal was reasonable when scanning electron microscopy (SEM) became a common tool for surface analysis. In this study we have measured accurately crack length using the SEM images formed in both secondary electron and BSE contrasts.[6,7,9]

In our study a boron nitride coatings were deposited on cutting insert edges made of S20S (P15-P25-ISO) cemented nanocarbides.[12] BN coating were produced using a pulse-plasma method elaborated at Department of Materials Science, Warsaw University of Technology.[22] In this method plasma is generated by electrical pulse discharge generating high ionized and non-isothermal plasma. The process is easy to conduct and control, a facility has a simple construction.

Raman spectroscopy analysis of boron nitride coatings has shown the presence of sp^3 bonds giving a peak of cubic c-BN as well as a peak of sp^2 bonds confirming the presence of hexagonal h-BN (Figure 1). Electron diffraction pattern confirmed the presence of both boron nitride phases in the coatings (Figure 2).[9,22]

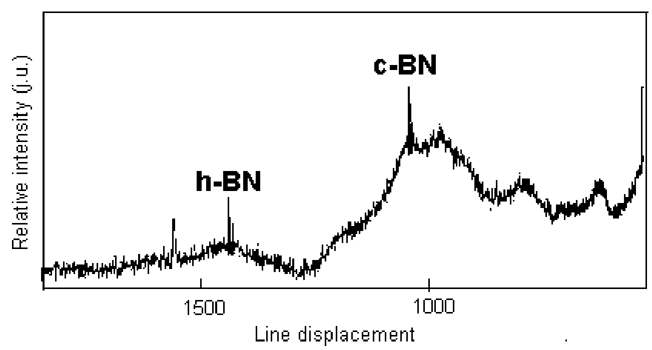

Figure 1. Raman spectra of the boron nitride (c-BN+h-BN) coating.[9]

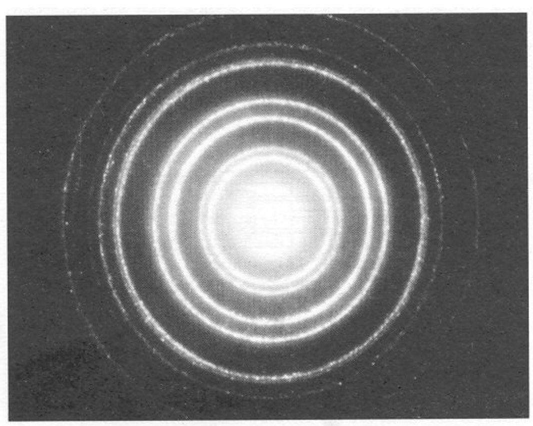

Figure 2. The electron diffraction pattern of the c-BN+h-BN coating.[9,22]

During investigations selected variable technological parameters of the boron nitride formation by the pulse-plasma method were applied:

– discharge voltage: 3 kV, 4.5 kV and 6 kV;

– process duration 3 h;
– interval between pulses: 2 s,
– boroamine (BH_3-NH_3) temperature 346 K,
– pressure of carrying gases 100 Pa (N_2, H_2),
– temperature of substrate: 1000 K.

3 RESULTS

Results of the total length of cracks measured on boron nitride coating using Palmqvist's method as a function of load is summarized in Table 1. The results have been averaged for 6 measurements.

Table 1. The set of the values of averaged total length of cracks measured on boron nitride coatings by Palmqvist's method

Variant of production process (sample designation)	Loading P [daN]					
	4,9	14,7	29,4	39,2	49,0	58,8
	Total length of cracks ΣL [□m]					
Coatings produced by using 3.0 kV of discharge voltage						
KP1 (3.0 kV)	44,6	180,7	342,4	432,1	554,2	650,6
Coatings produced by using 4.5 kV of discharge voltage						
KP5 (4.5 kV)	0,0	80,4	209,2	319,9	390,1	510,7
Coatings produced by using 6.0 kV of discharge voltage						
KP9 (6.0 kV)	0,0	22,4	122,1	209,6	278,3	390,8

Figure 3 shows the critical loads (P_{k300}, P_{k500}) determined graphically for boron nitride coatings manufactured using three different values of discharge voltage (3 kV, 4.5 kV and 6 kV).

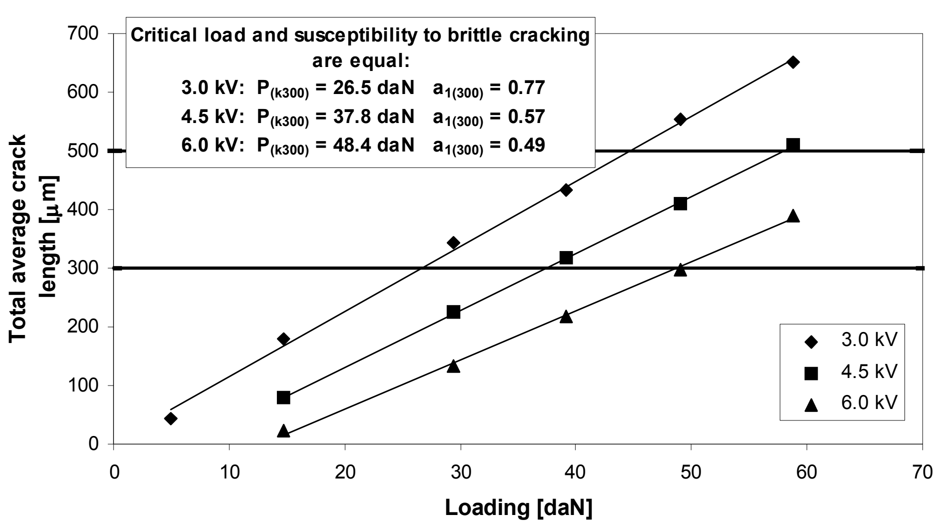

Figure 3. Determination of critical load (P_{k300}, P_{k500}) and susceptibility to brittle cracking ($a_{1(300)}$) for c-BN+h-BN coatings.

Using the results of total average crack length for boron nitride coatings deposited by three variants of the process, values of critical loads (P_{k300}, P_{k500}) and

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susceptibility to brittle cracking ($a_{1(300)}$, $a_{1(500)}$) have been calculated according to Palmqvist's formulas.[21] The results are shown in Table 2, where both P_{k300} and P_{k500} values of critical load as well as $a_{1(300)}$ and $a_{1(500)}$ values of susceptibility to brittle cracking are given, where 300 and 500 means the total crack length in μm.

The SEM pictures (Figures 4 and 5) show the Vickers indent obtained on the surface of boron nitride coating deposited on cutting tool made of S20S sintered carbides.

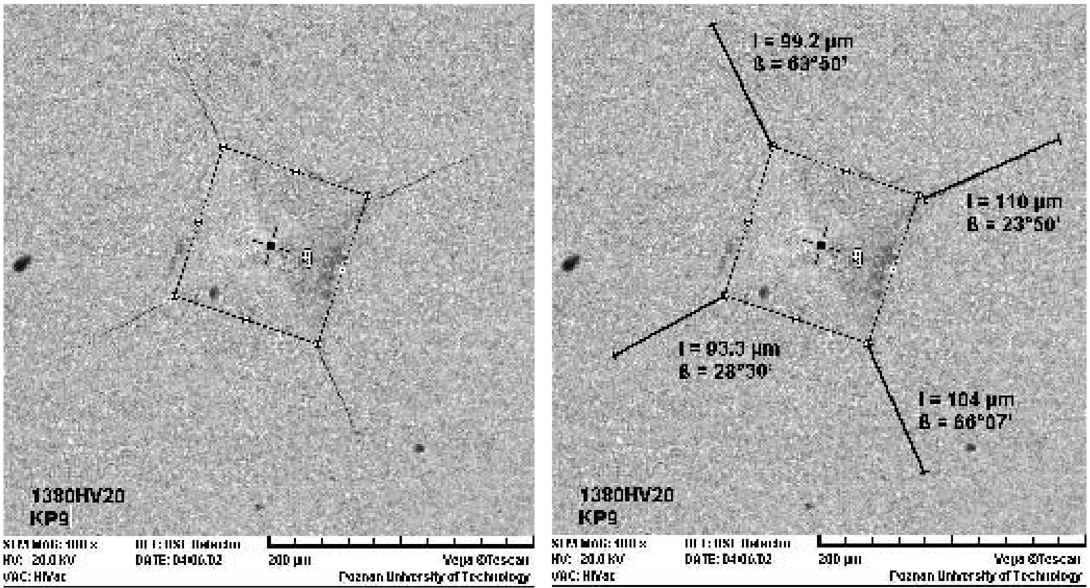

Figure 4. BSE images of Vickers indent on the surface of boron nitride coating deposited on cutting tool made of S20S sintered carbides (computer-aided measurement of cracks length by using VEGA – TS 5135 scanning electron microscope).[9]

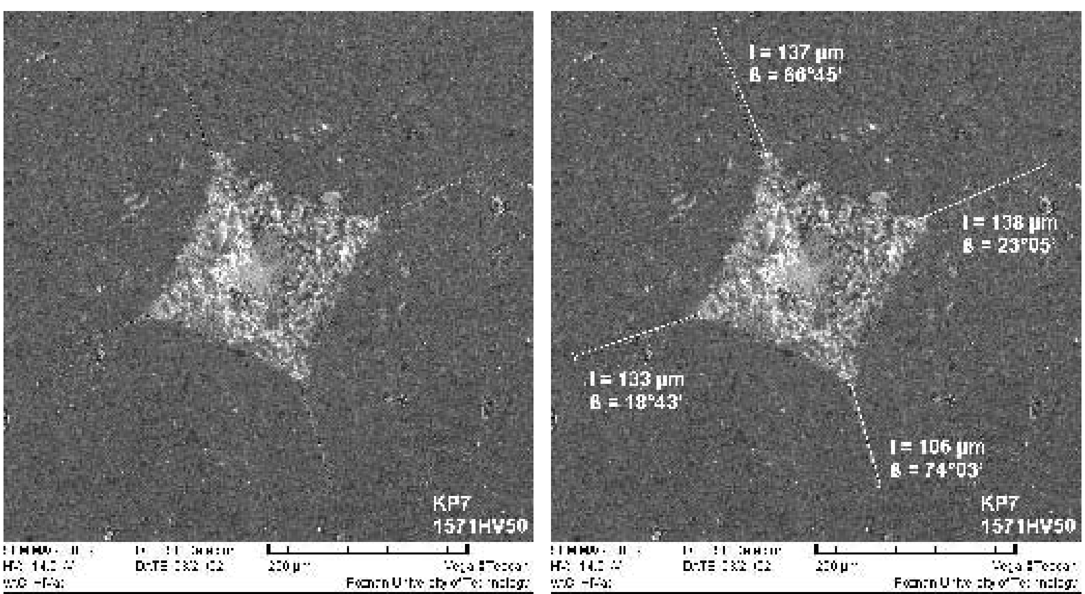

Figure 5. SE images of Vickers indent on the surface of boron nitride coating deposited on cutting tool made of S20S sintered carbides (computer-aided measurement of cracks length by using VEGA – TS 5135 scanning electron microscope).[9]

Table 2. Results of the critical load and susceptibility to brittle cracking for boron nitride coatings (KP1 – 3 kV, KP5 – 4.5 kV, KP9 – 6kV)

Variant of production process (sample designation)	Critical load P_k [daN]		Susceptibility to brittle cracking	
	P_{k300}	P_{k500}	$a_{1(300)}$	$a_{1(500)}$
KP1	26.5	43.0	0.77	0.52
KP5	37.8	57.9	0.57	0.38
KP9	48.4	–	0.49	–

In Figures 6-8 are presented SE images of structures of boron nitride coatings (c-BN+h-BN) obtained using 3 kV, 4.5 kV and 6 kV of discharge voltage.

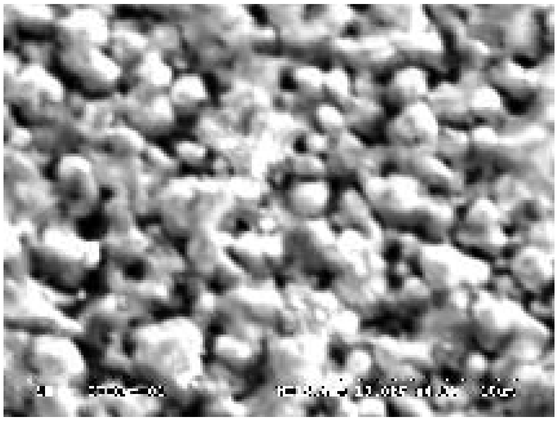

Figure 6. Structures of boron nitride coating obtained by pulse-plasma method using 3 kV of discharge voltage.[9]

In Figure 6 is visible that coating has a granular structure. Structure of coatings produced by using 3 kV possesses a lot of pores.

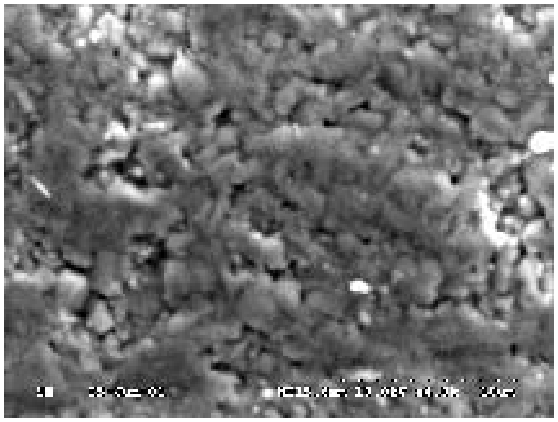

Figure 7. Structures of boron nitride coating obtained by pulse-plasma method using 4.5 kV of discharge voltage.[9]

In Figure 7 is shown SE image of structure of boron nitride coating (c-BN+h-BN) obtained using 4.5 kV of discharge voltage. In this figure is visible boron nitride coating with more dense structure and smaller pores then coating in Figure 6.

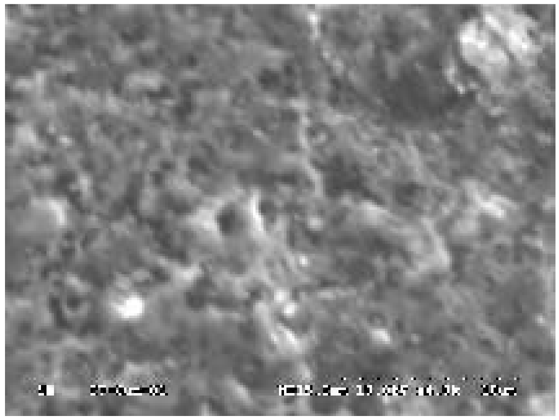

Figure 8. Structures of boron nitride coating obtained by pulse-plasma method using 6 kV of discharge voltage.[9]

In Figure 8 is presented SE image of structure of boron nitride coating (c-BN+h-BN) obtained using 6 kV of discharge voltage. In this figure is visible coating with dense structure and almost without pores.

On the base of the investigations results it is worth to underline an influence of microcracks and pores (according to Griffith's theory)[23] on the rate of crack propagation and on the material strength. This is a problem of proper choice of technological parameters (discharge voltage) during the deposition process.[9, 24]

4 CONCLUSIONS

On the base of investigations the following conclusions result from the studies of brittleness of boron nitride coatings obtained by the pulse-plasma method:

- an influence of coating structure on brittleness (dense coatings without pores have lower brittleness) was observed;
- significant differences of critical load have been found between samples KP1-KP5, KP1-KP9 and KP5-KP9 (the significance of the difference between the values of average critical load P_k, for the total crack length, number of degrees of freedom f = 10 and the significance level α = 0,05 has been determined using *t* Student's test);
- significant differences between values of $a_{1(300)}$ and $a_{1(500)}$ coefficients characterized susceptibility to coating cracking of investigated samples were existed;
- a lowest susceptibility to brittle cracking revealed for the coating obtained in KP9 process, characterized by the highest discharge voltage (6 kV);
- the Palmqvist's method is usefulness for measurement of coating susceptibility to brittle cracking has been confirmed;
- the measurements of crack length using a scanning electron microscope are of sufficient accuracy giving linear dependence of total crack length as a function of a load;
- a comparison of the brittleness can be perform for coatings deposited on the same substrate.[4, 9, 24]

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