

# Special glasses with submicrocrystalline sintered alumina admixture in cBN tools

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The results of physicochemical and mechanical investigations into the properties of the boron–alumina–silicate glass system, designed for cBN tools with additions of submicrocrystalline sintered alumina (cubitron), have been presented. Four glasses, obtained by the fritting method were investigated by the following experimental techniques: differential thermal analysis (DTA), infra-red spectroscopy, microhardness (3.58–4.48 GPa), bonding strength (36–41 MPa), thermal linear expansion coefficient (TLEC) ( $5.5\text{--}7.36 \times 10^{-6}$  1/deg), wettability on electrocorundum, cubitron and cBN substrates ( $\theta < 28^\circ$ ). The glasses fulfilled all the criteria of usability.

## 1. Introduction

Cubic boron nitride (cBN) was first obtained by the American scientist Wendorf in 1957 on the route of the transformation of hexagonal boron nitride at high temperature (1200–2000 °C) and high pressure (5–15 GPa) in the catalytic agent presence. Since 1980 there has been a continuous effort to develop cubic boron nitride tools and to improve their efficiency.

According to the International Institution for Production Engineering Research (CIRP) data, it has been estimated that approximately 18% of grinding tools are made of cBN and potential application has been assessed at about 60%. Much progress has been made especially in cBN tools, with vitrified binders due to the unique properties of these tools. The manufactured cBN tools with vitrified bonds may have different concentrations (75–200%), different structure, porosity and hardness, significant ability to self-sharpening, easy to sharpening and profiling. For a certain period of time, the possibilities of designing modified grinding tools made of cubic boron nitride with vitrified bonding that could work on the conventional grinding machines at standard grinding conditions have been looked for because of economic reasons. One of the directions of search is an attempt to design “mixed grinding wheel” – connecting monocrystalline grains of cubic boron nitride with submicrocrystalline sintered alumina [1]–[6].

The presence of submicrocrystalline grains helps to decrease grinding forces, which results in a decrease in the amount of heat produced during the process of abrasive machining and in the improvement of the workpiece surface finish, that is connected with an increase in the active summits number in the grinding process [7], [8].

Submicrocrystalline sintered alumina (cubitron)  $\alpha\text{-Al}_2\text{O}_3$  has got ultra-dispersive structure obtained by sol-gel transformation [1], [2], [6], [7]. Magnesia was used as a modifying agent in this process. The technology applied to corundum manufacturing makes it possible to obtain the sintered compact of specific structure containing short abrasive  $\text{Al}_2\text{O}_3$  needles separated by micro-threads of  $\text{MgAl}_2\text{O}_4$ . A significant difference of Young's elastic moduli of  $\text{Al}_2\text{O}_3$  and  $\text{MgAl}_2\text{O}_4$  (420–510 GPa and 280 GPa, respectively) is responsible for the grains self-sharpening phenomena during grinding.

Submicrocrystalline sintered alumina (corundum) is produced by the following manufacturers: American 3M concern (under the name of "Cubitron"), Norton concern ("Seeded Gel"), WMEM Corp. ("Ceramic Alumina") and Hermes ("Blue Saphir"). A proper selection of vitrified bonds for mixed abrasive wheels is an important problem [8]–[14]. These bonds should bind the cubic boron nitride and submicrocrystalline sintered corundum grains well, and, on the other hand, they should not dissolve the corundum.

As a criterion of usability the following was assumed:

- wetting angle of abrasive grain by a specific glass ( $\theta < 30^\circ$ ),
- microhardness  $> 3$  GPa,
- bending strength  $> 40$  MPa,
- thermal linear expansion coefficient (TLEC) in the range of  $4\text{--}10 \times 10^{-6} \text{ deg}^{-1}$ .

The investigation of vitrified bonds containing zinc–boron–alumina–silica glass systems were performed and described in this paper.

## 2. Experimental

The investigations object were glasses of  $\text{ZnO-B}_2\text{O}_3\text{-Al}_2\text{O}_3\text{-SiO}_2$  system with the modifying oxides  $\text{P}_2\text{O}_5$ ,  $\text{Bi}_2\text{O}_3$ ,  $\text{Li}_2\text{O}$ ,  $\text{BaO}$ ,  $\text{CaO}$ ,  $\text{B}_2\text{O}_5$ . The chemical composition of glasses was in the range of  $\text{SiO}_2$  (25–50%),  $\text{Al}_2\text{O}_3$  (10–20%),  $\text{B}_2\text{O}_3$  (6–10%),  $\text{ZnO}$  (2–9%) and admixture 0.5–8 wt%. To make a proper melt the analytically pure chemical compounds were used. The glass sets were melted in platinum crucibles in an electric furnace in the air atmosphere at approx. 1575 K. The glass sets were poured out on a steel plate and annealed at 794 K in a muffle furnace. The glass structure was determined by DTA analysis and infrared spectroscopy investigations. DTA analysis was carried out using Q1500 derivatograph unit in the range of temperatures 293–1273 K. Infrared spectroscopy investigations were performed using DIGILAB unit in the basic infrared range ( $400\text{--}2000 \text{ cm}^{-1}$ ).

Following physical, chemical and mechanical properties of glasses were investigated:

- density (by helium method),
- microhardness (Vickers method),

- bending strength (by three-point bending test),
- TLEC coefficient (by dilatometric method),
- glass-substrate wettability (by sessile drop method).

Investigations concerning phenomena at the abrasive grain and vitrified bond phase boundary were run using scanning electron microscopy (SEM) and X-ray microanalysis.

### 3. Results and discussion

#### 3.1. Differential thermal analysis of glass systems

From the DTA curves the following thermal effects were read:

- Endothermic effect derived from the glass transformation as the beginning of the deflection of the curve from the fundamental line for the glass sets 1–4.
- Endothermic effect derived from the dilatometric soft point at the temperatures of 853, 813, 818, 833 K.
- The beginning of exothermic effect from the beginning of the crystallisation temperature (glass 1 at 915 K, glass 2 at 943 K).
- The maximum of exothermic effect related to the crystallisation temperature (glass 1 at 993 K, glass 2 at 1043 K).
- Endothermic effect connected with the melting point at the temperatures of 1123, 1108, 1126, 1116 K.

All four investigated glasses belonged to the medium-fusible glasses. Among them two sets have shown tendency to the crystallisation. The two others have not exhibited any exothermic effects connected with the crystallisation phenomena.

#### 3.2. Infrared spectroscopy

The spectroscopy investigations were done in the near infrared ( $400\text{--}2000\text{ cm}^{-1}$ ) region. The samples were prepared as the tablets with potassium bromide. All the spectra were very similar, with the comparable values of the wave numbers and also the intensity of spectra. The distinctly defined bands with a large bandwidth were observed as well as broadened bands characteristic of the amorphous substances (Fig. 1a, b). The following bands were identified in the spectra:  $460\text{ cm}^{-1}$  derived from O–Si–O oscillations,  $\sim 680\text{ cm}^{-1}$  derived from bending oscillations in co-ordination number three,  $1100\text{ cm}^{-1}$  derived from asymmetric stretching oscillations Si–O–Si. For the last band the influence from the borosilicate Si–O–B stretching oscillations was found:  $\sim 1400\text{ cm}^{-1}$  obtained from B–O–B oscillations with B in co-ordination number three, and  $\sim 1620\text{ cm}^{-1}$  derived from O–H bonds oscillation in water molecules.

The band of  $580\text{ cm}^{-1}$  and  $600\text{ cm}^{-1}$  appeared in the glasses 1 and 2 additionally. It was connected probably with the liquation or crystallisation of glass.

#### 3.3. Investigations of chosen properties of the glasses

The results of the investigation are presented in Tab. 1 (average value from 3 to 10 measurements, depending on glasses properties).

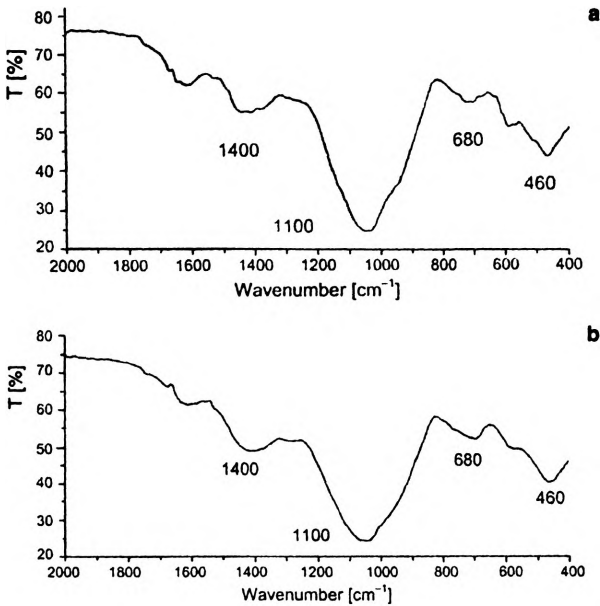


Fig. 1. Transmittance  $T$  of glass 1 (a) and 2 (b)

T a b l e 1. Physicochemical properties of glasses.

Properties	Glass set			
	1	2	3	4
Density [ $\text{g}/\text{cm}^3$ ]	2.851	2.853	3.010	2.831
Microhardness [GPa]	3.58	3.72	3.92	4.48
Bending strength [MPa]	36	39	39.5	41
TLEC [ $\text{deg}^{-1}$ ] heating cooling	$7.36 \times 10^{-6}$	$5.05 \times 10^{-6}$	$7.58 \times 10^{-6}$	$5.50 \times 10^{-6}$
	—	$5.72 \times 10^{-6}$	$6.91 \times 10^{-6}$	$5.65 \times 10^{-6}$

Investigated glasses may be reckoned with the group of light glasses (their density 2.83–3.01  $\text{g}/\text{cm}^3$ ) of medium microhardness (3.58–4.48 GPa), medium bending strength (36–41 MPa) and of very similar values of good  $\alpha$ -coefficients of the submicrocrystalline sintered alumina (cubitron).

### 3.4. Investigations of wettability

It has been shown, in many research works [4], [9], [10] that the bonds containing inorganic glasses give strong abrasive tools. It is a result of very good wetting of abrasive grains by vitrified bonds due to strong bridges between individual grains. The lower value of the extreme wetting angle, the better wettability; preferably the value of angle  $\theta$  should be lower than  $30^\circ$ . That would provide better adhesion of the bond to the grain surface, enhancing thus the binding power of the bridges. The

measurements of the wetting angle were carried out using a high temperature microscope of the Leitz–Wetzlar type, by means of the sessile drop method in air atmosphere. The electrocorundum, cubitron and cBN substrates were used for investigations. The results of the present measurements of wetting angle are shown in Tab. 2.

Table 2. Wetting angles of selected glasses at corresponding temperatures.

Substrate	Value of the angle [°]/temperature			
	Glass 1	Glass 2	Glass 3	Glass 4
Electro-corundum	20/1178 K	18/1183 K	16/1190 K	20/1180 K
Cubitron	27/1150 K	28/1158 K	24/1165 K	25/1152 K
cBN	25/1143 K	22/1150 K	23/1157 K	24/1170 K

All the glass systems have very good wettability on electrocorundum, cubitron and cBN substrates ( $\theta < 28^\circ$ ), so they meet requirements for the bonds of the super abrasive tools.

### 3.5. Investigations of the grain boundary phase

The range of chemical reactions between the vitrified bond and super hard grain was the more accurate evaluation criterion for the choice of vitrified bond during heat treatment [11], [12]. The cohesive bond strength of the vitrified bond and the strength of the grain-bond interface decided about the bond strength of the abrasive sinter [13], [14]. For the investigations of the grain boundary phase phenomena the scanning microscope with EDS microanalysis of the fresh fracture of vitrified

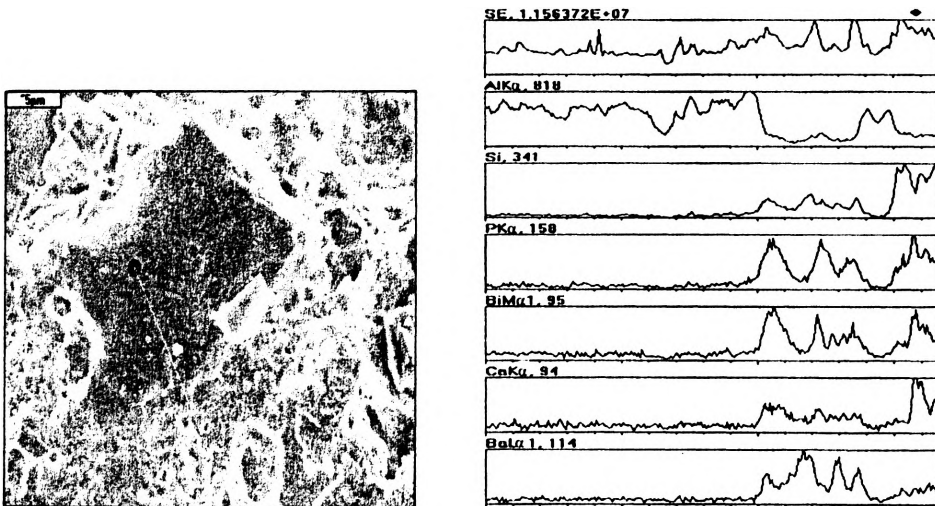


Fig. 2. SEM of the sintered sample (glass 4 + cubitron), linear distribution of chemical elements.

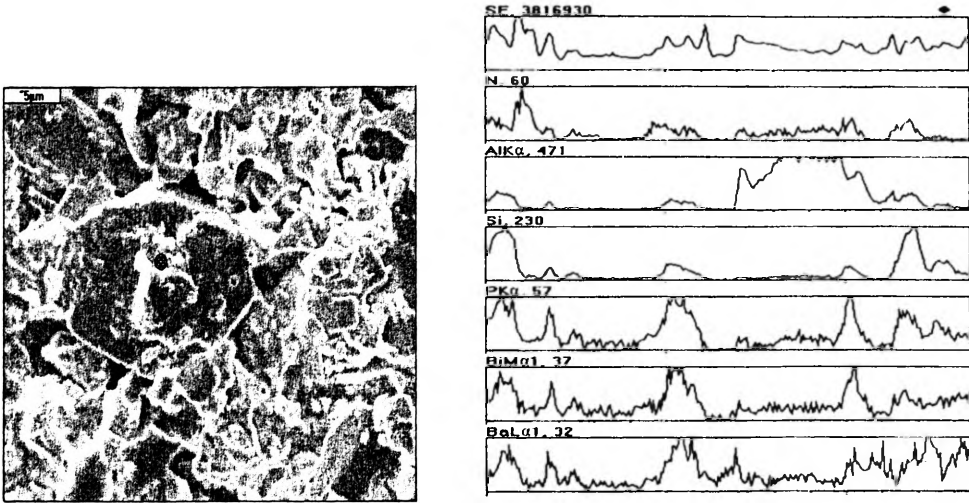


Fig. 3. SEM of the sintered sample (glass 4 + cBN), linear distribution of chemical elements.

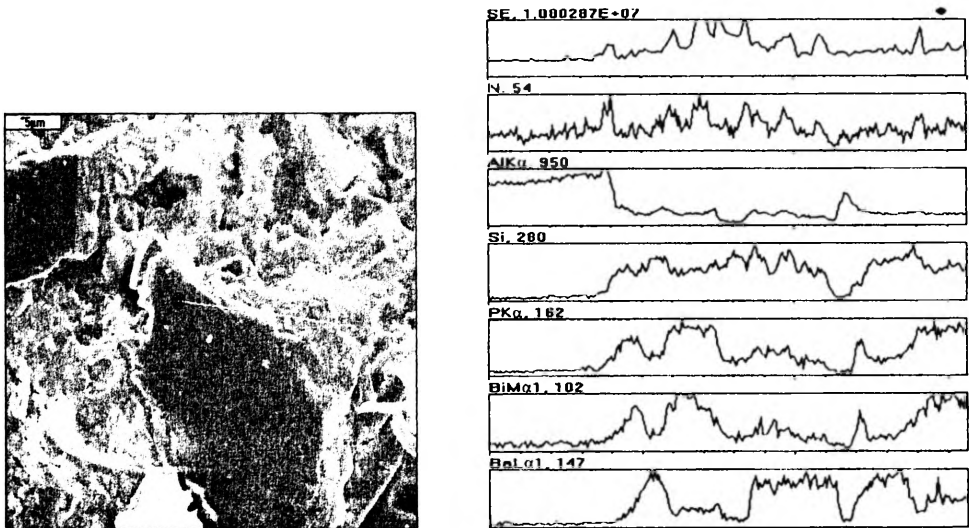


Fig. 4. SEM of the sintered sample (glass 4 + cubitron + cBN), linear distribution of chemical elements.

bond-substrate was applied. In the case of electrocorundum substrate the presence of oxide on the both parts of the sample, and diffusion of bismuth, calcium and bar without the intermediate layer could be seen. For the cBN substrates the small intermediate layer forming on the phase boundary was observed. Due to problems with the cubitron substrate, the samples of the sintered glass with cubitron, cubitron and cBN, and cBN were done (Figs. 2–4). The linear distribution of elements in the

sintered samples showed that the diffusion of glass components and abrasive grains occurred.

## 4. Conclusions

Basing on the investigation results the following conclusions can be drawn:

1. Investigated glasses belong to the light group of glasses ( $\rho = 2.8\text{--}3.1$  g/cm) of medium microhardness (3.58–4.48 GPa) and of medium bending strength (36–41 MPa).

2. The infrared spectroscopy showed the possibility of crystallisation in the glasses 1 and 2 (smaller width of bands) confirmed by DTA.

3. The values of TLEC coefficient were in the range of  $5.5\text{--}7.58 \times 10^{-6}$  deg<sup>-1</sup> during the heating processes. They were near to the submicrocrystalline sintered alumina (cubitron)  $\alpha$ -coefficient .

4. The electrocorundum, cubitron and cBN substrates were well wetted ( $\theta < 26^\circ$ ) by all the glasses.

5. In the samples consisting of glass 4 and cubitron, cubitron with cBN and cBN not quite sharp grain boundaries were observed, probably due to the chemical reaction that occurred at the interphase boundary. It was confirmed by the linear distribution of elements and the chemical elements mapping for the sintered samples.

6. All the glass systems fulfilled the criterion of usability as the vitrified bonds assigned for cBN tools with submicrocrystalline sintered alumina (cubitron) addition.

*Acknowledgments* – This work was supported by Polish State Committee for Scientific Research (KBN), Research Project No. 7 T08D 027 20.

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*Received September 26, 2002*