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Glass–ceramic bonding in alumina/CBN abrasive systems

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A glass–ceramic bond which can be applied in alumina/CBN abrasive systems was developed by the method of liquid-phase sintering of a homogenized mixture of alumina abrasives and bonding medium containing various amounts of B_2O_3. Microstructural and mechanical examinations have shown that the amount of B_2O_3 present in the starting glass frit determines the ultimate properties of the glass–ceramic bond which can be successfully used in the Al_2O_2/CBN abrasive systems.

1. Introduction

Considerable interest has been shown within the last decade in applications of the vitrified bonded abrasive materials, and in improving their strength and reliability. Abrasive grains in these materials are held together by a glass-ceramic bond.

The liquid phase between abrasive grains, formed during firing, pulls the individual grains together due to the surface tension forces [1]. During this process, some clustering of abrasive grains occurs. The glass–ceramic bond between abrasive grains offers good performances, mainly due to good mechanical and thermal properties. The glass matrix containing fine dispersed crystals causes crack branching and can decrease the probability of catastrophic fracture.

During grinding of metals and hard materials such as metal carbides, it is very important that the grinding wheel should be strong, resist mechanical shock, exhibit low wear and have good grinding efficiency. Glass–ceramic bonded cubic boron nitride, in combination with α-Al_2O_3, demonstrates superior usefulness when the grinding wheel is subjected to high mechanical and thermal shocks. These articles are formed by pressing a homogenized mixture of cubic boron nitride and alumina abrasives, and bonding medium. At elevated temperatures up to 1000 °C, the liquid-phase sintering associated with particle rearrangement and subsequent crystallization of a special glass leads to the formation of a glass–ceramic bond.

Several investigators have studied the formation of glass–ceramic bonds [2–5], and alumina abrasives of high strength containing a glass–ceramic bond have been developed.

However, in all the above cases, glass–ceramics compatible with alumina abrasives were studied. If alumina superabrasives based on CBN, which demand a lower sintering temperature, are introduced the glass composition must be changed in order to enable good wetting and flow at lower temperatures, usually below 1000 °C at which CBN is stable. For that purpose B_2O_3 is usually introduced into the glass frit in order to decrease the viscosity and to achieve good flow and wetting of both CBN and Al_2O_3 abrasive grains around 950 °C.

As the glass–ceramic bonded alumina determines the mechanical properties of the entire alumina/CBN abrasive system to a great extent, these properties must be thoroughly studied. The objective of the present study was to investigate a glass–ceramic bond which can be successfully used in alumina/CBN abrasive bodies.

2. Experimental procedure

The glass frit used during the investigations was obtained by melting mixed oxides of SiO_2, H_3BO_3, K_2CO_3, Na_2CO_3, Al_2O_3, with some alkaline earth and transition metal oxides, at 1500–1600 °C, in an alumina crucible. The quenched glass was crushed in a vibratory mill and finely milled in an agate planetary mill. Also some batches of the glass were cast and slowly cooled.

During the preparation of the glass frit, the evaporation of B_2O_3, K_2O and Na_2O could not be completely suppressed. Therefore the final composition of the glass frit was obtained from chemical analyses (Table I).

The type of abrasive grain used in this study was fused alumina oxide with a grid size of 120. Abrasive samples were made according to the volumetric grading system [6]. The constant grain, bond and pore volume percentages were 46, 11 and 43, respectively. The proportion of bond and grain was 13.9 to 86.1 by weight. The composition corresponded to the grade-structure designation of K9.

TABLE I Final composition of glass frit

Code	Composition of glass (wt %)						Linear expansion (10^{-6}/°C)	Softening temperature^a (°C)
	Al_2O_3	N_2O	K_2O	B_2O_3	SiO_2	$R^{++}O^b$		
A	16.0	4.0	1.2	19.5	58.4	0.9	4.3	603
B	16.0	4.26	1.15	24.0	53.8	0.8	4.7	582
C	15.0	4.0	1.2	32.0	46.8	1.0	5.1	560

^{a} Obtained from linear expansion.
^{b} $R^{++}O$ alkaline earth and transition metal oxides.

Pre-weighed abrasive grains were blended with 1.5 wt % of a temporary binder (a solution of dextrin) in a planetary mixer. Then pre-weighed fritted bonds with compositions A, B and C (Table I) were added to the prepared moistened mixture and thoroughly homogenized in an agate mortar until a homogeneous mix was obtained. From these homogenized mixtures, bars were pressed of the dimensions 24 × 5 × 10 mm. The forming pressure and the mould charge during the preparation of green bars were obtained from the desired green density and die dimensions. Samples were fired in an electric furnace using a heating schedule with a 6-h soak at temperatures of 800, 900, 1000 and 1100 °C.

The bars were broken in three point bending on an Instron Universal Testing Machine (Instron 1362) at a rate of 1.7 mm min^{-1}. The breaking loads were recorded and used to calculate the strength of each bar. The cast glasses were cut into sections with a low-speed diamond saw. Thermal expansion measurements were performed on 25-mm sectioned samples with a heating rate of 2 °C min^{-1}.

The microstructure of sintered samples was examined by scanning electron microscopy (SEM) on polished and on as-fired surfaces. In some cases the samples which were thoroughly powdered were inspected with transmission electron microscopy (TEM). The glass posts between two alumina grains, in sample A where the glass phase contains about 19.5 wt % of B_2O_3, and sample B with a glass phase containing about 24.0 wt % B_2O_3, were analysed with an electron microanalyser (Cambridge Instruments M9). The glass-phase region between alumina grains was analysed in a 10 μm section (see Figs 7 and 8 below).

The density of samples was measured using the Archimed method and mercury, and the porosity was measured with a mercury porosimeter (Micrometrics Pore Sizer 9310).

3. Results and discussion

In Fig. 1, the dependence of the normalized bending strength of alumina abrasives with various glass–ceramic bonds A, B and C sintered at 800, 900, 1000 and 1100 °C in air is shown. The firing cycle was identical for all samples. As seen from Fig. 1 there is a remarkable difference in the course of the bending strength as against the sintering temperature between samples studied. Samples containing the glass–ceramic of composition A exhibit a gradual increase in the normalized bending strength from 8 MPa cm^3 g^{-1} to ~ 23 MPa cm^3 g^{-1}. Particularly in the temperature region between 900 and 1000 °C, a pronounced increase in bending strength can be noted.

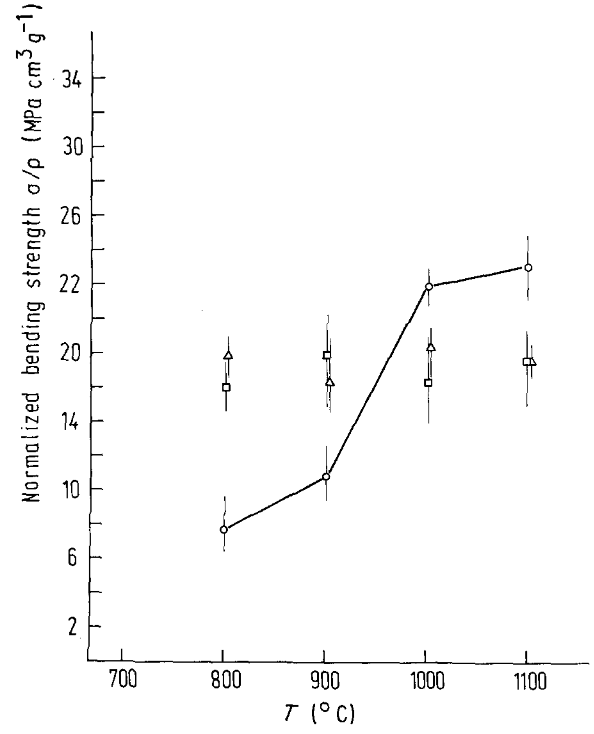

Figure 1 Normalized bending strength of glass–ceramic bonded alumina abrasives with various glasses, with compositions (○) A; (□) B and (△) C (see Table I), sintered at 800, 900, 1000 and 1100 °C.

Figs 2 and 3 show SEM micrographs of the as-fired surface of sample A, fired at 800 and 900 °C, respectively. It can be seen that at 800 °C the wetting and flow of glass A is poor and the posts between abrasive grains in sample A are in a state of formation (Fig. 2a). However, at 900 °C the wetting of abrasive grains is nearly completed (Fig. 3).

A further increase of the firing temperature from 900 to 1000 °C increases the bending stress appreciably. However, an examination of the microstructure, particularly of the co-ordination of grains and of the habits of posts, show that there is no significant change in the microstructure during sintering when the firing temperature is raised. Besides, the porosity measurements show that the pore size and distribution is nearly identical in both samples. Neither the porosity nor the density can account for such a drastic change in the normalized bending strength. Therefore the bulk properties of the posts, rather than the microstructure, must have been changed during the rising of

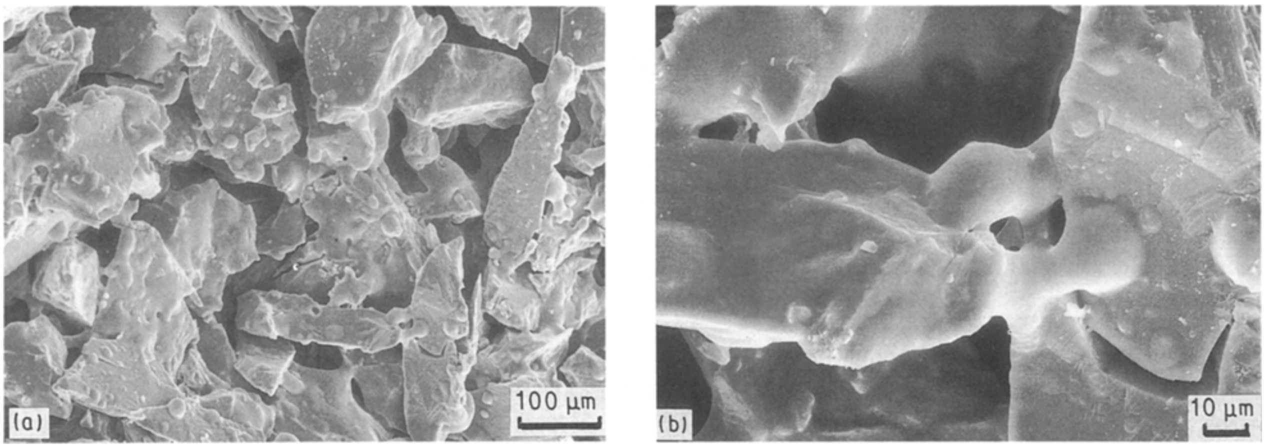

Figure 2 (a) SEM micrographs of the as-fired surface of sample A sintered at 800 °C; (b) detail of the microstructure showing a bond post between two abrasive grains in the state of formation.

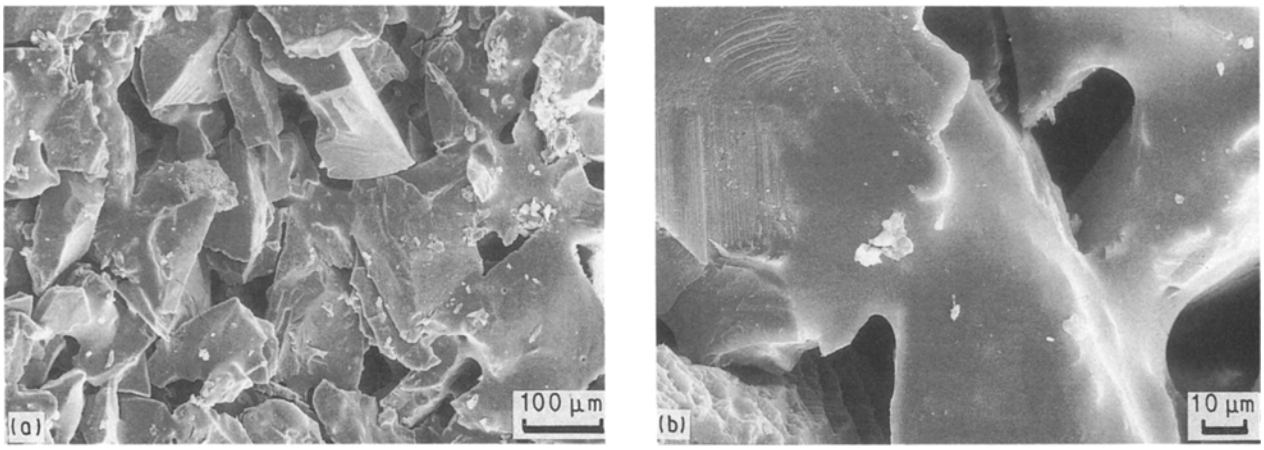

Figure 3 (a) SEM micrographs of the as-fired surface of sample A sintered at 900 °C; (b) detail of the microstructure showing the bond post between abrasive grains which is nearly completed.

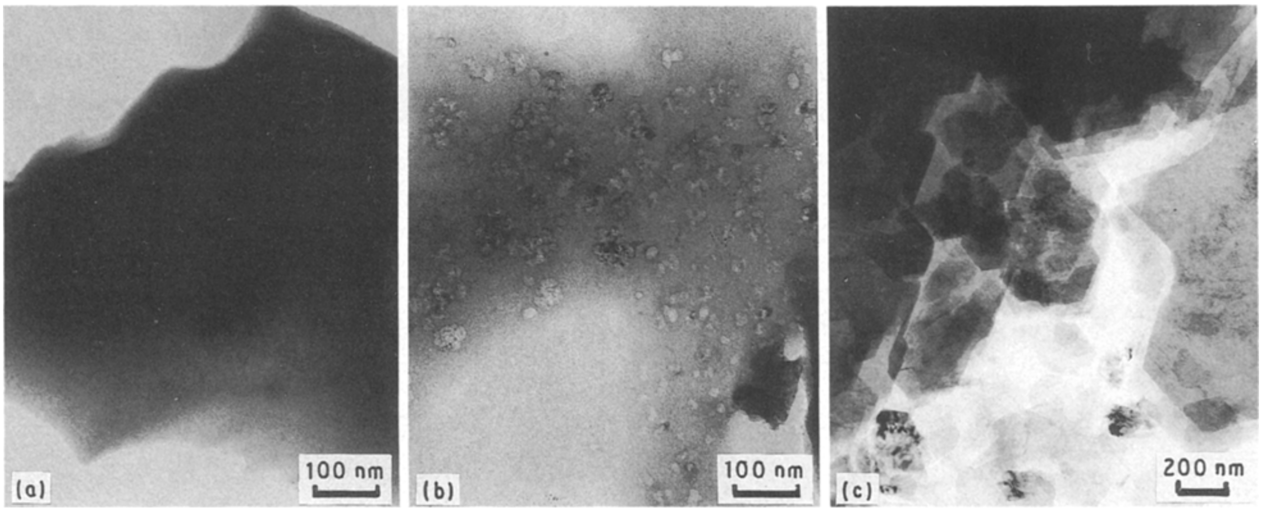

Figure 4 TEM images of sample A sintered at (a) 800; (b) 900; (c) 1000 °C.

sintering temperatures and caused the increase of normalized bending strength σ/ρ. The TEM investigation of samples confirms that prediction.

In Fig. 4 the TEM images of glass-post regions in sample A sintered at 800, 900 and 1000 °C are shown. In sample A sintered at 800 °C, no crystallized glass regions could be identified. However, in samples sintered at 900 °C and higher temperatures, crystallized glass regions can be identified. The post regions between grains show small crystals which become larger at higher sintering temperatures. An analysis of the diffraction patterns of crystals showed that the phase which crystallizes out of glass is most probably albite ($NaAlSi_3O_8$).

Since the crystallization of the glass posts between abrasive grains in sample A between 900 and 1000 °C is accompanied by a remarkable increase in bending strength, it is justifiable to assume that the improvement in the mechanical properties of sample A in dependence on the sintering temperature comes primarily from the formation of a glass–ceramic matrix. Here, the crystallization is retarded and starts to be effective when the posts between grains have already formed.

Samples B and C exhibit a completely different course of normalized bending strength against sintering temperature. In these samples, if sintered at 800 °C, the measured normalized bending strength is nearly three times higher than that of sample A. If the sintering temperature of samples B and C is further increased, the normalized bending strength stays more or less constant within the limits of the σ/ρ standard deviation.

On the other hand, the microstructural examinations of samples B and C show that the mutual dependence of processes which govern the microstructure development and modify the mechanical properties in these samples is different. The SEM images of samples B and C sintered at 800 °C in Figs 5 and 6 show that the formation of bond posts between abrasive grains in these samples has proceeded to a noticeably greater extent in comparison to sample A.

The diagrams in Figs 7 and 9 show the concentration profiles of K_2O, Na_2O, Al_2O_3 and SiO_2 and B_2O_3 across the glass post between two alumina grains in samples A and B, respectively. The concentration profiles indicate that the glass phase in sample B increases more its alumina content in comparison to the glass phase A during sintering.

Glasses B and C have lower softening points (Table I) and start to flow and wet alumina grains earlier, in comparison with glass A containing a lower amount of B_2O_3. The higher reactivity of glasses B and C, which have originally a higher wetting ability, increases their viscosity due to the solution of alumina [7]. At higher sintering temperatures, glasses B and C dissolve a larger amount of alumina in comparison to glass A (Figs 7 and 8) which increases their viscosity and hinders the further flow of the glass phase. Besides, the liquid phase near the phase boundary becomes saturated on Al_2O_3 which results in a growth of needle-like crystals, most probably mullite (Fig. 9),

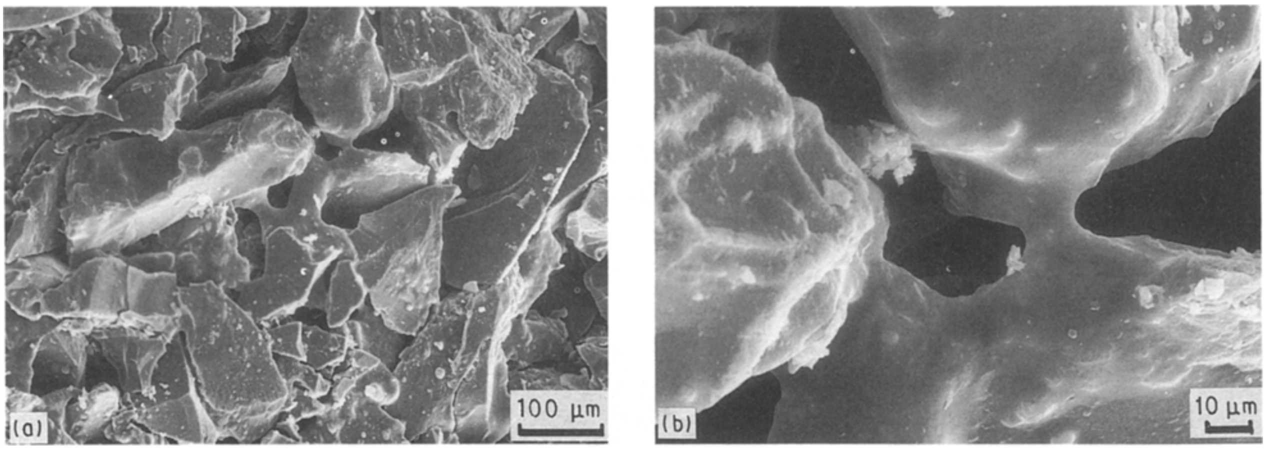

Figure 5 (a) SEM micrographs of the as-fired surface of sample B at 800 °C; (b) detail of the microstructure showing the bond posts between abrasive grains.

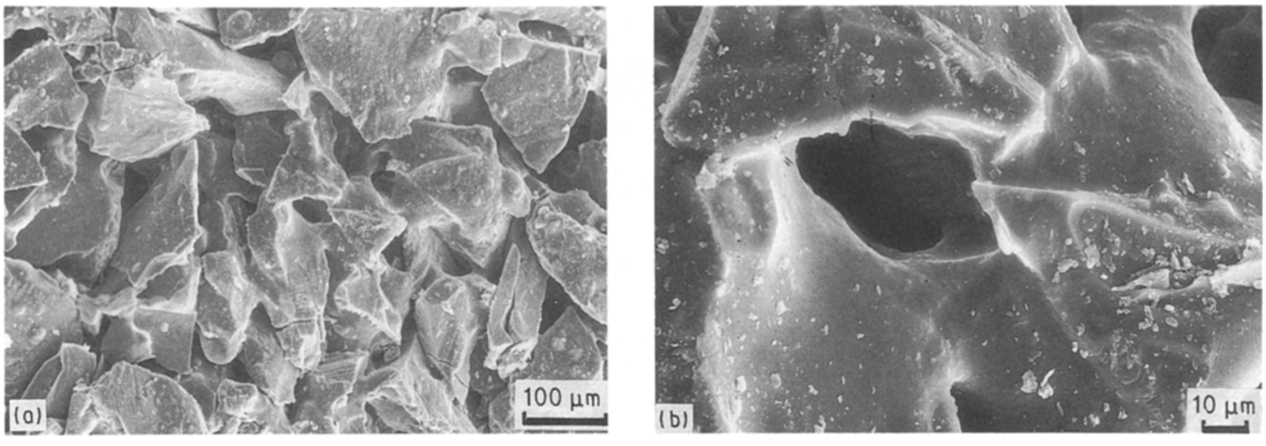

Figure 6 (a) SEM micrographs of the as-fired surface of sample C at 800 °C; (b) detail of the microstructure showing the bond posts between abrasive grains.

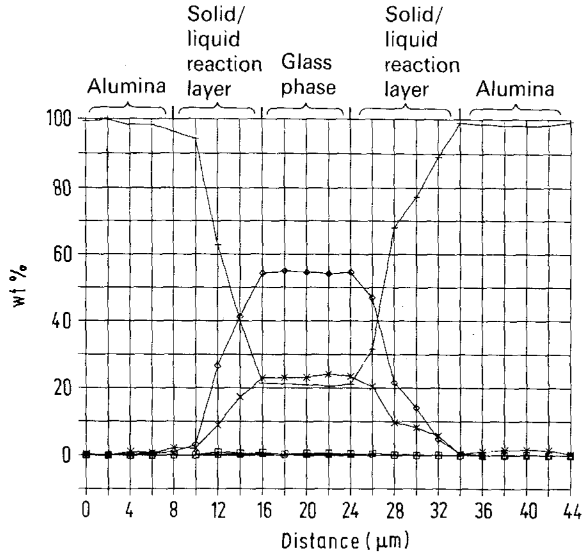

Figure 7 Concentration profiles of (△) K_2O; (□)Na_2O; (+) Al_2O_3; (◇) SiO_2; and (×) B_2O_3 in sample A across a glass post between two alumina grains on 70-µm sections, taken with an electron microanalyser. The concentration profile of B_2O_3 was estimated by taking into account that wt % (B_2O_3) = 100% − Σwt %.

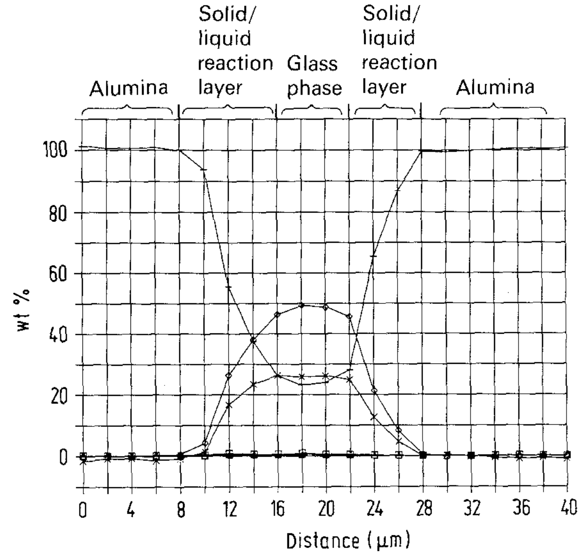

Figure 8 Concentration profiles of (△) K_2O; (□) Na_2O; (◇) SiO_2; (+) Al_2O_3; and (×) B_2O_3 in sample B across a glass post between two alumina grains on 10-µm sections. The concentration profile for B_2O_3 was estimated by taking into account that wt % (B_2O_3) = 100% − Σwt %.

which hinder the formation of post bonds between alumina grains.

Therefore, in samples B and C a relative high normalized bending strength is already obtained at 800 °C due to the lower softening point and better flowing of these glasses. However, when the sintering temperature increases further, dramatic changes in the microstructure development occur. Instead of hardening of bond posts at higher sintering temperatures, a growth of needle-like crystals on the phase boundary occurs, which hinder the wetting and flow of the glass phase and therefore the formation of post bonds between abrasive grains. Such a course of microstructure development in samples B and C results in a more or less constant bending strength against sintering temperature.

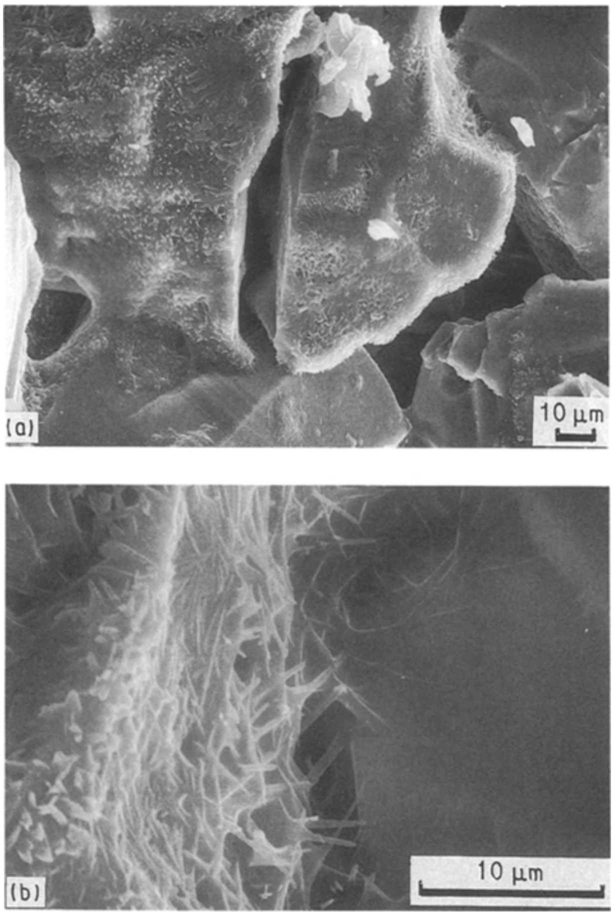

Figure 9 (a) SEM images of the as-fired surface of sample B sintered at 1000 °C; (b) detail of the microstructure showing the needle-like crystals between abrasive grains.

We see that the amount of B_2O_3 is of crucial importance for modifying the mechanical properties of the abrasive systems studied. A greater amount of B_2O_3 gives rise to the formation of glass bonds already at 800 °C. On the other hand, the abrasive sample A with glass containing a smaller amount of B_2O_3 exhibit a lower bending strength at 800 °C, which increases with the rise of the sintering temperature. As in Al_2O_3/CBN abrasive systems the CBN grains are sensitive to temperatures above 950 °C, a compromise must be found between the sintering temperature and the amount of B_2O_3 present in the glass used to prepare abrasive systems.

4. Conclusions

An alumina abrasive of high strength containing a glass–ceramic bond which can be used in Al_2O_3/CBN abrasive systems was developed. The optimal amount of B_2O_3 present in the glass–ceramic matrix was found to be around 20 wt %. A greater amount of B_2O_3 (20 wt %) present in the glass phases B and C causes good wetting and flow below 900 °C. A lower amount of B_2O_3 in sample A decreases the solubility of Al_2O_3 component in flowing glass and delays the onset of crystallization.

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References

1. W. D. KINGERY, A. P. SIDHWA and A. WAUGH, *Amer. Ceram. Soc. Bull.* **42** (1963) 297.
2. T. J. CLARK and J. S. REED, *ibid.* **65** (1988) 1506.
3. A. BAKON, *Szklo Ceram.* **28** (1977) 303.
4. N. POPIVANOVA, *Epitoanyag* **32** (1980) 423.
5. A. JAGER, *Rud. Metal. Zb.* **27** (1980) 77.
6. US Patent. No. 1 983 082, December 1934.
7. W. SCHILLER, J. WIEGMANN, M. NEIBUHR, D. HAUPT and P. BLANK, *Silikattechnik* **35** (1984) 108.

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