

# Effect of polycrystalline mullite fibers on the properties of vitrified bond and vitrified CBN composites

Baohui Zhao, Zhihong Li\*, Yumei Zhu

*Key Laboratory for Advanced Ceramics and Machining Technology of Ministry of Education, School of Materials Science and Engineering, Tianjin University, Tianjin 300072, People's Republic of China*

Received 6 July 2012; received in revised form 20 August 2012; accepted 17 September 2012

Available online 23 September 2012

## Abstract

The effect of polycrystalline mullite fibers (PMFs) on the properties of vitreous bonds and vitrified CBN composites was investigated. The results show that the addition of PMFs can increase the porosity of composites and reduce the fluidity of binders. The vitrified composites incorporating 6.4 wt% PMFs display excellent mechanical strength, which is enhanced by 21.2% compared with that of composites without PMFs sintered at the optimal sintering temperature. Meanwhile the thermal expansion coefficient of vitrified bond reduces from  $6.256 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$  to  $4.805 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$  with increasing fraction of PMFs. The improvement of mechanical strength is associated with the change of cracking mechanisms of the composites with fibrous crystals and the existence of several observed mechanisms, including fiber pull-out, fiber bridging and rupture.

© 2012 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

**Keywords:** Vitrified bond; Polycrystalline mullite fibers; Mechanical strength; Microstructure

## 1. Introduction

Due to its high hardness, good chemical inertness, and good thermal conductivity, cubic boron nitride (CBN) grinding wheels are becoming one of the most potential grinding tools in manufacturing of modern engineering materials, especially ferrous materials [1,2]. Compared with resin and metal bond CBN grinding wheels, recent years vitrified CBN grinding wheels have been increasingly applied in high speed, high efficiency and high precision grinding because of their outstanding properties, including relatively high bond strength, high elastic modulus, low fracture toughness, controlled porosity and good self-dressing capability [3,4]. In the manufacturing process of CBN grinding tools, to avoid the oxidation of diamond/cubic boron carbide (CBN) abrasive grains and maximize the super-abrasive grinding capacity, vitrified bond should meet characteristics such as low-melting point and high strength [5]. However, in certain grinding applications these low temperature binders have demonstrated insufficient

mechanical strength to meet commercial objectives, thus promoting the development of stronger binders. Previous studies have investigated the effects of vitrified bond compositions [6–8], interfacial microstructure [3,9] and microcrystalline structure [10–12] on the strength of vitrified bond alumina/CBN grinding tools whereas, there are very few reports on fiber reinforced vitrified bond except from the company Winterthur up to now. Fibers are generally used to overcome the mechanical deficiencies of materials, so it would be interesting to develop fiber reinforced vitrified bond.

In this study, we choose polycrystalline mullite fibers (PMFs) as reinforced materials to develop high strength vitrified bond with low melting point. It is well known that PMFs have good chemical stability, high melting temperature, excellent mechanical properties and low thermal expansion coefficient [13,14]. In addition, there is good interface cohesion between PMFs and  $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--B}_2\text{O}_3\text{--R}_2\text{O} + \text{RO}$  vitrified bond due to their similar chemical composition.

Accordingly, the primary aim of this paper is to investigate the effect of different amounts of PMFs on the properties of vitrified bond and vitrified CBN composites,

\*Corresponding author. Tel./ fax: +86 22 27404260.

E-mail address: [lizhihong@tju.edu.cn](mailto:lizhihong@tju.edu.cn) (Z. Li).

Table 1  
Composition of the basic glass powder used in this study (mass fraction).

Bond component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	Li <sub>2</sub> O	MgO	ZnO
wt%	60	10	15	7	3	2	3

Table 2  
Content of PMFs and refractoriness of different vitrified bonds.

Denomination	PMF/ (wt%)	Refractoriness /(°C)
F <sub>0</sub>	0	710
F <sub>1</sub>	1.6	714
F <sub>2</sub>	3.2	721
F <sub>3</sub>	6.4	730

especially the mechanical strength, and then reveal the strengthening mechanisms of PMFs reinforced vitrified bond.

## 2. Experiment procedure

In this study, we study the effect of PMFs on the properties of vitrified bond and vitrified CBN composites. The compositions of the basic vitrified bond used in this study are given in Table 1, and they were directly introduced in the form of commercial silica, alumina, boric acid, sodium carbonate, lithium carbonate, magnesium carbonate, and zinc oxide. Then polycrystalline mullite fibers (the average diameter is about 10  $\mu\text{m}$ , the length is about 20–40 mm, Zhejiang Hongda crystal Fiber Co. Ltd, China) were added to the basic vitrified bond powder, and the content of polycrystalline mullite fibers and refractoriness of the corresponding vitrified bonds are listed in Table 2. In a typical experimental procedure, firstly, PMFs were dispersed into ethanol by ultrasonic stirring for 30 min, and then the obtained suspension was dried in an electric drying oven. Secondly, the powders of the basic vitrified bond were wet milled for 6 h in a ball mill (ball: absolute ethyl alcohol: powders = 2:1:1); then the treated PMFs were mixed together with the as-prepared powders for further milling for about 1 h. Eventually, the uniform powders were dried and sieved through 120-mesh sieve.

Vitrified CBN composites were prepared with CBN abrasive (grit size 80#, ca. 180  $\mu\text{m}$ ), the prepared vitrified bonds and temporary binder. The proportion of CBN, vitrified bond and temporary binder (dextrin) was 75:20:5. Green samples were dry-pressed into rectified parallelepiped bars with dimensions of 30 mm  $\times$  6 mm  $\times$  4 mm. Then green samples were sintered in an electric furnace using predetermined firing schedule with 2 h holding at the optimal sintering temperature of 820 °C.

The porosity and bulk density of samples were measured by the Archimedes drainage method. The plane flowing method (after the bond powders are molded into cylinders with certain dimensions, the specimens are put on the smooth ceramic plates and sintered in the furnace using

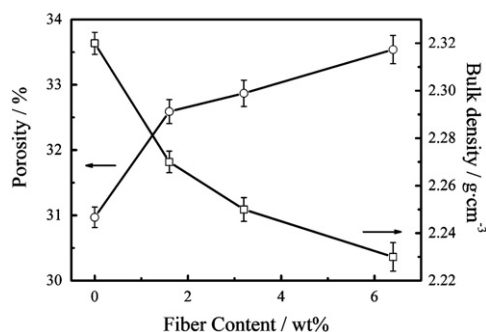


Fig. 1. Effect of fiber content on the porosity and bulk density of vitrified CBN composites sintered at 820 °C.

predetermined heating schedule; then fluidity can be calculated by measuring the change of cylinders diameters) was used to measure fluidity of vitrified bond that was applied to evaluate the bonding state between vitrified bond system and abrasive grains. The mean bending strength of samples was measured with a universal tester using a three-point test method with a span length of 20 mm and a cross-head speed of 0.5 mm min<sup>-1</sup>. Microstructures were observed by field emission scanning electron microscopy (FESEM); Model TDCLS-48000). Thermal expansion coefficient measurement was performed by a NETZSCH DIL402C apparatus (Germany).

## 3. Result and discussion

### 3.1. Effect of PMFs on porosity and bulk density of vitrified CBN composites

Fig. 1 shows the variation of the porosity and bulk density of vitrified CBN composites with increasing fraction of PMFs. As a whole, with the increase of PMFs content, it can be found that the porosity of composites gradually increases and the variation of bulk density is opposite to that of the porosity. As shown in Fig. 1, the porosity of vitrified composites without PMFs is 30.97%, and the bulk density is 2.32 g/cm<sup>3</sup> accordingly. By comparison, the porosity of composites containing 6.4 wt% PMFs reaches 33.54%, and the bulk density is 2.23 g/cm<sup>3</sup>. The increase in porosity of composites with the increasing fraction of PMFs may be explained as follows. Firstly, the viscosity of the binder without PMFs is low at the sintering temperature, which will benefit the exhaustion of gases to form dense bond bridges. Meanwhile, the distance among abrasive grits will be reduced due to the liquid formation in the basic vitrified bond. By contrast, the viscosity of the

binders is improved with the increasing fraction of PMFs, and gases produced in sintering process cannot be fully exhausted. Small pores, which are left in bond bridges after discharging gases, do not easily grow up and densify due to the high viscosity. Furthermore, the decrease of liquid phase with the increasing fraction of PMFs in binders is also adverse to the densification of composites. From the perspective of grinding, pores in the grinding are conducive to wheel cooling, chip pocket and chip removal, so the raised porosity may have excellent grinding performance for vitrified bond grinding tools.

### 3.2. Effect of PMFs on fluidity of vitrified bond

The variation of fluidity for vitrified bonds incorporating different contents of PMFs is given in Fig. 2. It can be seen that the addition of PMFs has large effect on the fluidity of vitrified bond. As shown in Fig. 2, the fluidity of vitrified bonds with PMFs is lower than that without PMFs at the optimal sintering temperature. Moreover, it is clear that higher amount of PMFs results in lower fluidity of vitrified bonds.

The decline of the fluidity might result from the increase of high temperature viscosity of vitrified bonds with different fractions of PMFs. The variation tendency of the high temperature viscosity is closely related to the refractoriness of vitrified bonds at a certain sintering temperature, and as a general rule, the higher the refractoriness, the higher the viscosity of the binder. As shown in Table 2, when different contents of PMFs are introduced into the basic vitrified bond, the refractoriness is gradually raised, which reflects the increase in the high temperature viscosity of vitrified bonds to some extent. The enhanced high temperature viscosity ultimately leads to the descent of the fluidity of vitrified bonds.

### 3.3. Effect of PMFs on bending strength of vitrified CBN composite

Fig. 3 shows the bending strength of composites incorporating different amounts of PMFs. It is obvious that the

bending strength is improved remarkably by the addition of PMFs. The result displays that the maximum bending strength of the composites with 6.4 wt% PMFs is 60.83 MPa, after sintering at 820 °C, while it is 50.2 MPa for the composites without PMFs. This is about 21.2% increase in bending strength for the composites compared with that of the composites without PMFs. It is concluded that the remarkable increase of the bending strength is attributed to the strengthening effect of PMFs on the basic bond matrix. However, it is also possible that the bending strength of samples would decrease with the continuously increasing PMFs content, which results from the increment of flaw caused by increasing porosity. Of course, this forecast needs to be verified by further experiment.

In a general way, the increase of pores improves the chance of crack initiation in the bond, which will lead to decrease in bond strength. However, the experiment presents the opposite trend. As a result, it is inferred that the addition of PMFs has remarkable influence on the special strength of vitrified bond composites within an appropriate range. Compared with composites without PMFs, the increase in strength for composites with the increasing fraction of PMFs might be attributed to the load transmission and the change of cracking mechanisms after adding fibers. On one hand, the broken fibers and pullout of PMFs can effectively transfer loads from bond matrix to fibers; on the other hand, the inclusion of fibers changes the cracking mechanisms of composites. For the vitrified CBN composites without PMFs, the fracture mode is mainly brittle fracture, because cracks in binders with amorphous structure are not easy to be furcated due to the lack of inter-granular boundaries where these cracks could be stopped. Once cracks begin to spread in binders of this type, their further increase requires lower loads and catastrophic fracture will take place. However, for the composites with PMFs, cracks are locally blocked to form crack deflection, bridge and pinning when cracks spread ahead of PMFs, which is conducive to increasing the energy of cracking. Meanwhile the main crack spreading can be postponed because of the formation of a great amount of energy consumption structures, such as crack

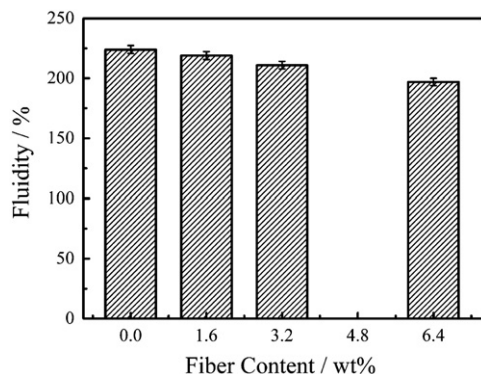


Fig. 2. Effect of fiber content on the fluidity of vitrified bond sintered at 820 °C.

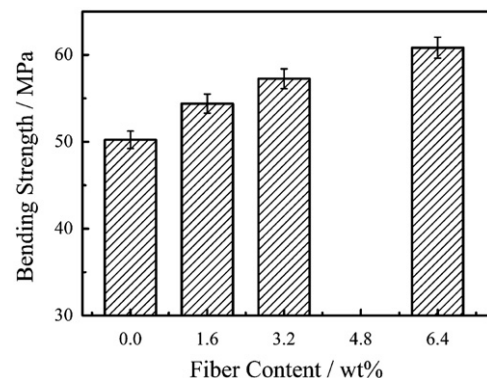


Fig. 3. Effect of fiber content on the bending strength of vitrified CBN composites sintered at 820 °C.

deflection and pinning. The higher fracture energy of composites with PMFs shows a progressive fracture behavior rather than a catastrophic fracture behavior, and the strength of composites is improved. With the increasing fraction of PMFs, the benefit of PMFs is so obvious that the strength is increased accordingly.

### 3.4. Effect of PMFs on the thermal expansion coefficient of vitrified bond

The coefficient of thermal expansion (CTE) of vitrified bond has important influence on the performance of vitrified grinding wheels. To obtain good adhesion and avoid thermal stress forming at the interface, it is better to achieve thermal matching as high as possible between vitrified bond and abrasive grains [15]. The thermal expansion coefficients of the binders in this study have been given in Fig. 4. The thermal expansion coefficients of vitrified bonds  $F_0$ ,  $F_1$ ,  $F_2$  and  $F_3$  sintered at 820 °C are  $6.256 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ ,  $5.923 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ ,  $5.756 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  and  $4.805 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ , respectively. From the above result, it is indicated that the thermal expansion coefficient of vitrified bonds can be reduced by adding PMFs. The result may owe to the mechanical restraints imposed by PMFs with low thermal expansion coefficient. That is, the rigid fibers mostly prevent expansion of the matrix in the longitudinal direction; thus the matrix is forced to expand less than normal in the longitudinal direction. Meanwhile, because the fiber orientation is random in the matrix, the vitrified bonds incorporating PMFs exhibit isotropy in the thermal expansion coefficient. Furthermore, with increasing fraction of PMFs, the restraint effect of PMFs is more notable and the thermal expansion coefficient further reduces. The coefficient of thermal expansion of CBN in the temperature range 20–1000 °C is reported to be  $2\text{--}5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ , while the average thermal expansion coefficient of vitreous bond  $F_3$  in the temperature range 40–500 °C is  $4.805 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  which is very close to the thermal expansion coefficient of CBN grains. So we can conclude that one reason to achieve optimal strength for vitrified composites containing vitrified bond  $F_3$  is the better thermal matching between vitrified bond  $F_3$  and CBN grains.

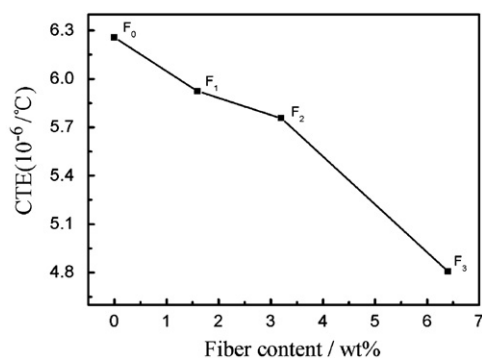


Fig. 4. Effect of fiber content on the thermal expansion coefficient of vitrified bond sintered at 820 °C.

### 3.5. Micro-structural analysis

Fig. 5 shows the fracture morphology of vitrified CBN composites. We can see that abrasive grains are almost absolutely wrapped by vitreous bonds as a result of the good fluidity of vitrified bonds. In CBN grinding wheels, the mechanical strength of vitrified grinding wheels mainly depends on phase boundary strength and the strength of bond bridges [9]. Fig. 5 shows that there are no distinct interfaces between abrasive grains and binders and the fracture behavior of composites mainly occurs on the bond bridge, which indicates that the phase boundary strength is higher than the strength of bond bridges. In other words, the strength of composites in this study is mainly governed by the strength of bond bridges. Comparing the fracture morphology of composites with and without PMFs, some differences can be observed from Fig. 6. Cracks exist on the fracture planes of composites with PMFs, especially around PMFs, while the fracture planes of composites without PMFs are relatively integrated and appear as a “mirror” zone as shown in Fig. 6(a). The fracture mode of vitrified composites without PMFs is ascribed to the brittle fracture of vitrified bond, and the appearance of the “mirror” zone is the main characteristic of brittle fracture. However, for the vitrified composites with PMFs, cracks are locally blocked when they try to spread ahead of PMFs and the furcation of cracks is formed around PMFs as shown in Fig. 6(b), which will consume the energy of main crack growth. This effect is also called crack bridging [16].

Fig. 7 shows several morphology characteristics of PMFs in the fracture plane of vitrified CBN composites: (1) PMFs pull-out (compared with the PMFs with smooth surface, the surface of the PMFs pulled out from the fracture plane was sags and crests due to the residual glass phase on the surface of PMFs as shown in Fig. 7(a)); (2) PMFs rupture; (3) to serve as a part of bond bridge by wrapping fibers in bond bridge; (4) fiber bridging between bond bridges and forming a network structure. These

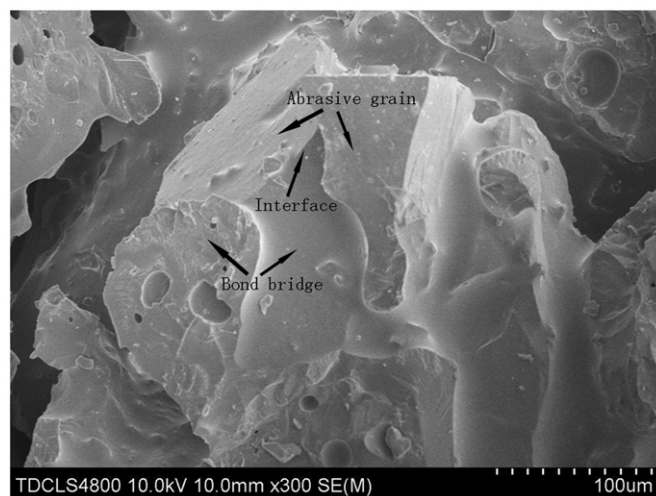


Fig. 5. FESEM observation of the sintered CBN composites with vitrified bond  $F_0$ .



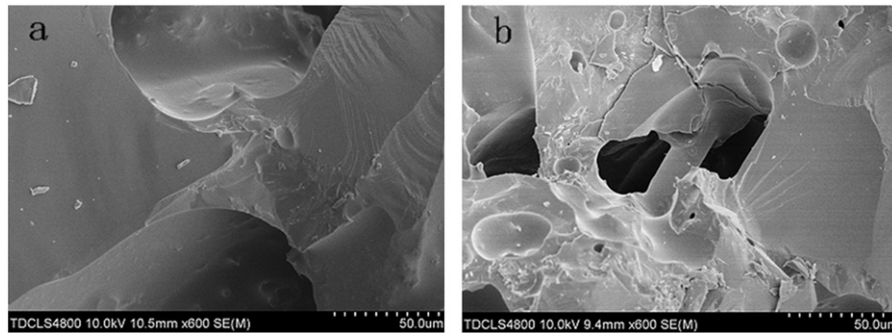


Fig. 6. Comparison of fracture surfaces of the vitrified CBN composites: (a) the fracture surface of the vitrified composites without PMFs; and (b) the fracture surface of the vitrified composites with PMFs.

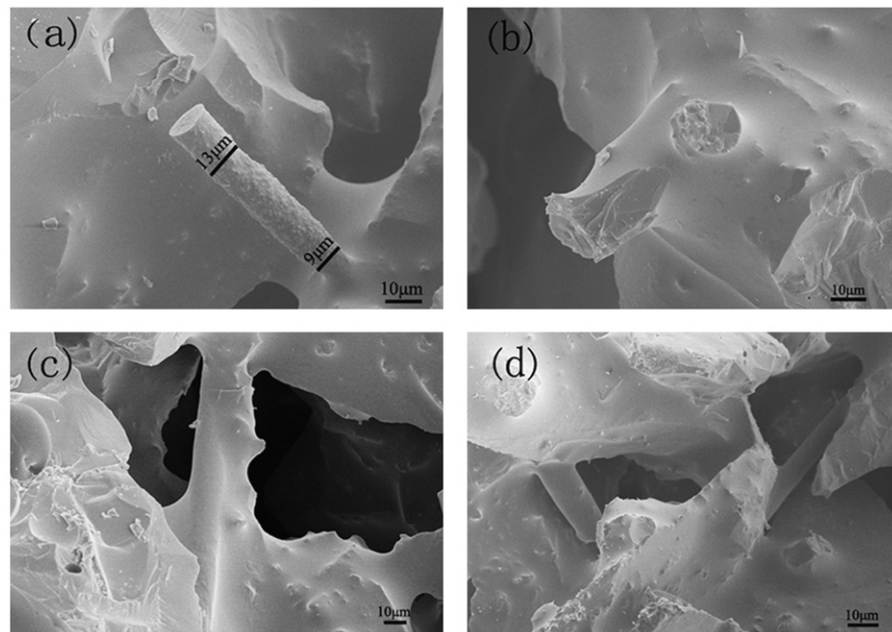


Fig. 7. Different states of PMFs in fracture surface of the vitrified CBN composites: (a) pull-out of fiber; (b) breakage of fiber; (c) as bonding bridge wrapped in vitrified bond; (d) fiber bridging between bond bridges.

characteristics of PMFs in vitrified CBN composites may be the determinants of the improvement in the bending strength of vitrified composites incorporating PMFs.

Fiber pull-out is one of the reinforcement mechanisms in fiber reinforced composite materials [17–19]. Fiber pull-out requires pulling the embedded broken fibers out against any residual frictional stresses at the fiber/matrix interface [20]. The good bonding interface between fibers and bond matrix results in the consumption of more energy when PMFs are pulled out from bond matrix, which allows the vitrified bond composites to sustain greater loads. Fiber rupture is another strengthening mechanism in composites. In FESEM of the fracture plane of composites, some broken or split-off fibers can be observed. The rupture of PMFs will effectively transfer the load from bond matrix to fibers, because the strength of PMFs is higher than that of bond matrix. Moreover, PMFs are wrapped in bonding bridges to serve as a part of bonding bridges, which vastly improves the strength of bonding bridges so as to make bonding bridges

sustain greater applied load. In addition, the overlap joint of fibers among bonding bridges forms a network structure. This structure can share the load imposed on a single bond bridge, thus improving the mechanical property of vitrified bond composites. No matter what the strengthening mechanisms are, the addition of PMFs is helpful to obtain higher work of fracture value.

#### 4. Conclusion

PMFs reinforced vitrified bond for CBN grinding wheels was prepared in this study. The properties of vitrified bond, including porosity, fluidity and thermal expansion coefficient, were measured. The bending strength and microstructure of vitrified CBN composites were investigated. The results are obtained as follows:

- (1) With the increase of polycrystalline mullite fibers, the porosity of vitrified CBN composites has a slight

increase, and the bulk density of vitrified composites decreases. The fluidity of vitrified bonds decreases with increasing fraction of PMFs, which results from the augment of high temperature viscosity of vitrified bonds. The thermal expansion coefficient of vitrified bonds reduces with the increase of PMFs, and the coefficient of thermal expansion ( $4.805 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ ) of vitrified bond containing 6.4 wt% PMFs is very close to the thermal expansion coefficient of CBN grains.

- (2) The addition of PMFs has a remarkable effect on the mechanical strength of composites; when the additive content of PMFs is 6.4 wt%, there is about 21.2% increase in bending strength of vitrified bond composites sintered at the optimal sintering temperature of 820 °C.
- (3) According to the analysis of microstructure, the existence of PMFs can locally block the spreading of cracks and makes cracks furcated. In addition, the strengthening mechanisms of composites containing PMFs also include fiber pull-out, rupture, bridging and the strengthening of bond bridges by PMFs.

## Acknowledgment

The authors would like to thank Professor Li Jinyou for providing a research fellowship.

## References

- [1] M.J. Jackson, C.J. Davis, M.P. Hitchiner, High-speed grinding with CBN grinding wheels—applications and future technology, *Journal of Materials Processing Technology* 110 (2001) 78–88.
- [2] D. Herman, J. Krzos, Influence of vitrified bond structure on radial wear of CBN grinding wheels, *Journal of Materials Processing Technology* 209 (2009) 5377–5386.
- [3] P.F. Wang, Z.H. Li, J. Li, Y.M. Zhu, Effect of ZnO on the interfacial bonding between  $\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{SiO}_2$  vitrified bond and diamond, *Solid State Sciences* 11 (2009) 1427–1432.
- [4] K.H. Lin, S.F. Peng, S.T. Lin, Sintering parameters and wear performances of vitrified bond diamond grinding wheels, *International Journal of Refractory Metals and Hard Materials* 25 (2007) 25–31.
- [5] P.F. Wang, Z.H. Li, Y.M. Zhu, Effect of CaO on the surface morphology and strength of water soaked  $\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{Al}_2\text{O}_3-\text{SiO}_2$  vitrified bond, *Journal of Non-Crystalline Solids* 354 (2008) 3019–3024.
- [6] M.J. Jackson, N. Barlow, B. Mills, The effect of bond composition on the strength of partially-bonded vitrified ceramic abrasives, *Journal of Materials Science. Letters* 13 (1994) 1287–1289.
- [7] Y. Shang, Y.G. Hou, G.Y. Qiao, W.J. Zou, F.R. Xiao, B. Liao, Effects of nano-AlN on phase transformation of low temperature vitrified bond during sintering process, *Transactions of Nonferrous Metals Society of China* 19 (2009) 706–710.
- [8] M.J. Jackson, B. Mills, Materials selection applied to vitrified alumina and CBN grinding wheels, *Journal of Materials Processing Technology* 108 (2000) 114–124.
- [9] M.J. Jackson, Sintering and vitrification heat treatment of CBN grinding wheels, *Journal of Materials Processing Technology* 91 (2007) 232–234.
- [10] D. Herman, J. Plichta, T. Karpinski, Effect of glass-crystalline and amorphous binder application to abrasive tools made of microcrystalline alumina grains type SG, *Wear* 209 (1997) 213–218.
- [11] A.M. Hu, M. Li, D.L. Mao, Growth behavior, morphology and properties of lithium aluminosilicate glass ceramics with different amounts of CaO, MgO and  $\text{TiO}_2$  additives, *Ceramics International* 34 (2008) 1393–1397.
- [12] D.U. Tulyaganov, S. Agathopoulos, Synthesis of glass-ceramics in the  $\text{CaO}-\text{MgO}-\text{SiO}_2$  system with  $\text{B}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$ ,  $\text{Na}_2\text{O}$  and  $\text{CaF}_2$  additives, *Journal of the European Ceramic Society* 26 (2006) 1463–1471.
- [13] H. Tan, Y. Ding, J. Yang, Mullite fibers preparation by aqueous sol-gel process and activation energy of mullitization, *Journal of Alloys and Compounds* 492 (2010) 396–401.
- [14] W.G. Wang, H.W. Li, K.R. Lai, Preparation and characterization of mullite whiskers from silica fume by using a low temperature molten salt method, *Journal of Alloys and Compounds* 510 (2012) 92–96.
- [15] M.J. Jackson, Thermal expansion of alumino-alkalisilicate and alumino-borosilicate glasses—comparison of empirical models, *Journal of Materials Science Letters* 16 (1997) 1264–1266.
- [16] Z. Xia, L. Riester, W.A. Curtin, Direct observation of toughening mechanisms in carbon nanotube ceramic matrix composites, *Acta materialia* 52 (2004) 931–944.
- [17] T.S. Lin, D.C. Jia, P.G. He, Effects of fiber length on mechanical properties and fracture behavior of short carbon fiber reinforced geopolymer matrix composites, *Materials Science and Engineering A* 497 (2008) 181–185.
- [18] M. Du, J.Q. Bi, W.L. Wang, Microstructure and properties of  $\text{SiO}_2$  matrix reinforced by BN nanotubes and nanoparticles, *Journal of Alloys and Compounds* 509 (2011) 9996–10002.
- [19] J. Wang, H.M. Kou, X.J. Liu, Reinforcement of mullite matrix with multiwalled carbon nanotubes, *Ceramics International* 33 (2007) 719–722.
- [20] J. Yuan, D.W. Wang, H.B. Lin, Effect of ZnO whisker content on sinterability and fracture behaviour of PZT piezoelectric composites, *Journal of Alloys and Compounds* 504 (2010) 123–128.