

Sintering of new SiC-Phosphate composite materials for grinding wheels fabrication

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The effect of several variables such as molar ratio P_2O_5/B_2O_3 (X), temperature and heating cycle on the strength of chemically bonded SiC materials for grinding wheels fabrication was studied. It was shown that the highest compressive strength (52.4 MPa) could be obtained using an optimal molar ratio $X = 4.5$ and a multiple step heating cycle up to a sintering temperature of 800°C. DTA, XRD, SEM and dilatometrical analysis were performed to follow the different stages of the structural development.

Keywords: phosphate bonding, strength, grinding tools, SiC, chemical bonding.

Sinterización de nuevos materiales compuestos de sic aglomerados con fosfatos para la fabricación de muelas abrasivas

Se estudia el efecto de diferentes variables tales como la razón molar P_2O_5/B_2O_3 (X), temperatura y ciclo de calentamiento sobre la resistencia de materiales de SiC aglomerados químicamente para la fabricación de muelas abrasivas. Se ha comprobado que la más alta resistencia a la compresión (52,4MPa) pueda obtenerse usando una razón molar óptima $x= 4,5$ y un ciclo de calentamiento en múltiples etapas hasta la temperatura de 800° C. Estudios mediante ATD, DRX, MEB y análisis dilatométricos fueron llevados a cabo para seguir las diferentes etapas del desarrollo microestructura.

Palabras clave: Aglomeración química, resistencia mecánica, muelas abrasivas de SiC

1. INTRODUCTION

The chemistry of different kind of materials bonded by phosphate binders was reviewed and classified by Kingery (1) and Cassidy (2). The good adhesive properties, the high refractoriness and thermal resistance, the superior mechanical resistance and the ability of phosphate binders to hardening by polycondensation, gives an opportunity for their industrial application as special kinds of glues, refractories, cements, ceramics and metal coatings production (3).

Usually, these binding materials are phosphate solutions, namely hydrophosphates, yielded by dissolving oxides or hydroxides in water and neutralization with phosphoric acid. Such binders are saturated molecular solutions. Through the hardening process the binder turns to a disperse system due to the hard phase separation.

The physical and the mechanical properties of aluminum phosphate binders are improved by B_2O_3 modification, leading to formation of condensed aluminum phosphates, at lower temperatures than in the case of pure aluminum phosphate binders, or to BPO_4 formation (4,5). For example, the strength of porcelain is known to considerably increase by addition of BPO_4 (6).

The insertion of inert additives allows varying the mechanical, chemical, thermal and electrical properties in a wide range. Various kinds of materials based on aluminum phosphate binders with SiC additions were developed for refractory production (7,8). In spite of this, the use of phosphate binders in abrasive grinding tools hasn't been researched yet. Hence, we are focusing our work on the replacing of conventional binders by boron phosphate matrices in such articles, which most important characteristics are: i) Self-sharpening – the ability of the grinding tool to loose by bonding strength lack, the abrasive grains that reached a critical radius of roundness of their cutting edge, and to replace them by new sharp grains at the surface; ii) Structure – the abrasive material, binder and pores amount and volume; iii) Hardness – the capacity of the binder to hold the abrasive grains (9).

2. EXPERIMENTAL PROCEDURE

2.1. Starting materials

The starting materials were α -SiC (ZZAM–Ukraine; main fraction 200 – 250 μ k), H_3PO_4 (Merck; 85%), B_2O_3 (Merck), kaolin.

2.2. Specimen preparation

The binders were prepared from 65% water solution of H_3PO_4 by step-by-step small additions of B_2O_3 , during heating and continuously mixing for about 20 min. After few days, the obtained colloidal solutions decompose into crystalline and liquid phases. Solutions with molar ratio P_2O_5/B_2O_3 (X) = 3, 3.5, 4 and 4.5 were prepared. The final abrasive grades were processed by mixing 70wt.% of SiC (60/70 mesh), 11.83wt.% phosphate binder and 18.17wt.% kaolin. Bars (5x5x50mm) and cylinders (\varnothing 20x20mm) were produced by uniaxial pressing at 50MPa for mechanical characterization. The green samples were dried at 60°C for 24h. All samples were heated at 4°C/min up to 550°C, excepting those from compositions with molar ratio $X = 3$ and 4.5 that were sintered at 550°C or 800°C under special heating cycles (Fig. 1) based on preliminary dilatometrical and DTA experiments (Fig. 2 and Fig. 3).

3. Results and Discussion

The colloidal solutions were separated into crystalline and liquid phases. X-ray Diffraction Analysis (XRD) proved that the crystals, after drying at 60°C, were consisted of boron orthophosphate. Also, XRD indirectly demonstrated that the liquid phase is pure orthophosphoric

acid as AlPO_4 was formed through its reaction with Al_2O_3 from kaolin additions. The boron orthophosphate crystals were then separated from the solutions and heated at 120 and 1000°C during 4h, losing 45 % of its initial mass, which proves that the obtained phase contained crystalline water, accordingly with the results reported from Kmecel et al. (10).

Fig. 3 exhibits the DTA traces of specimens with $X = 3$ and 4.5 under a rate of heating of 3°C/min. The large endothermic peak that appears at 900°C is associated with the evolution of water from BPO_4 and the formation of AlPO_4 as a result of the reaction between H_3PO_4 and Al_2O_3 from kaolin.

Fig. 4 exhibits the XRD spectra for the compositions with $X = 3, 3.5, 4$ and 4.5 heated at 4°C/min up to 550°C. The missing of the peaks typical for AlPO_4 is due to the endothermic processes for dehydration of BPO_4 and the phase transformations in kaolin, which prevent the formation of aluminum phosphate phase. The XRD spectra of samples sintered at 550°C and 800°C under the special heating cycle shown on Fig. 2, are given on Fig. 5 and Fig. 6, respectively. Obviously, in samples with molar ratio $X = 3$ sintered at 550°C, AlPO_4 formation started along with kaolin and BPO_4 . Also, by increasing the solutions acidity ($X =$

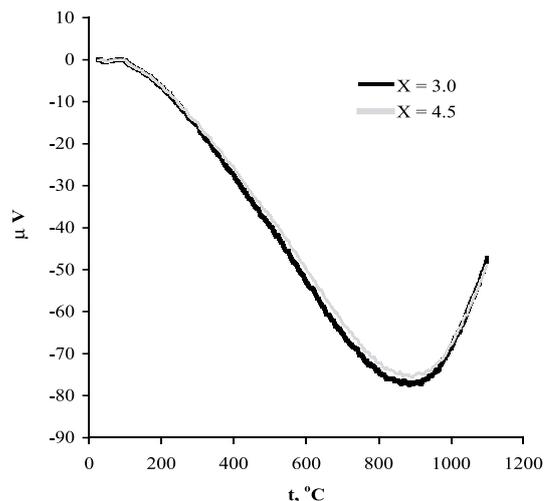


Figure 3 – DTA analysis of samples with $\text{P}_2\text{O}_5/\text{B}_2\text{O}_3$ molar ratio (X) = 3 and 4.5.

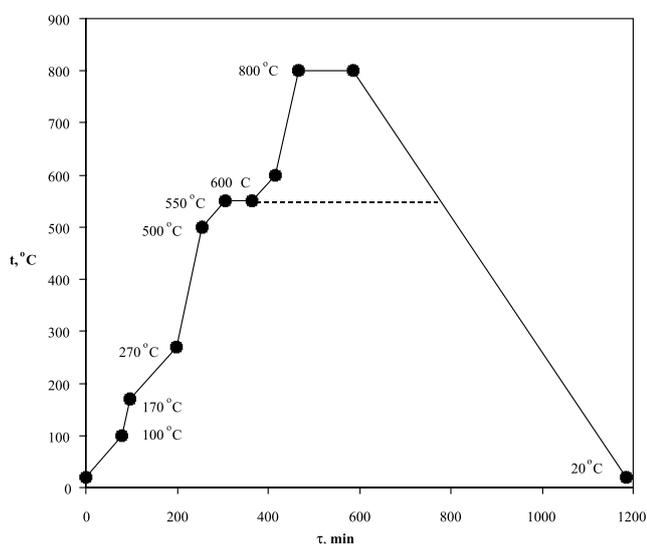


Figure 1 – Profile of the special sintering cycle with multiple step heating used for samples with $\text{P}_2\text{O}_5/\text{B}_2\text{O}_3$ molar ratio (X) = 3 and 4.5.

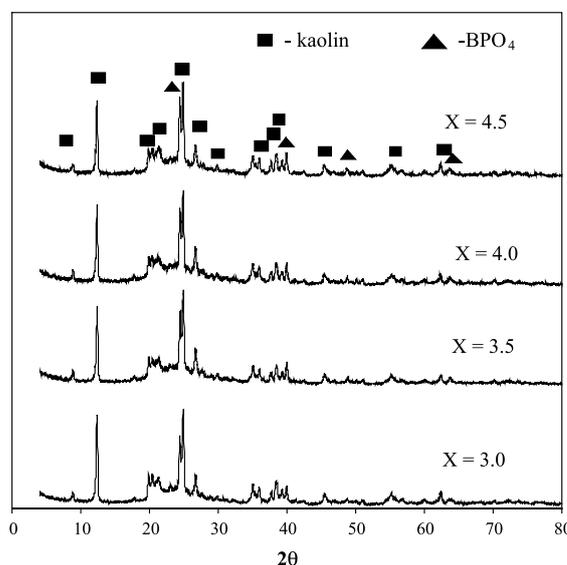


Figure 4 – XRD spectra of samples with $\text{P}_2\text{O}_5/\text{B}_2\text{O}_3$ molar ratio (X) = 3, 3.5, 4 and 4.5, heated at 4°C/min up to 550°C.

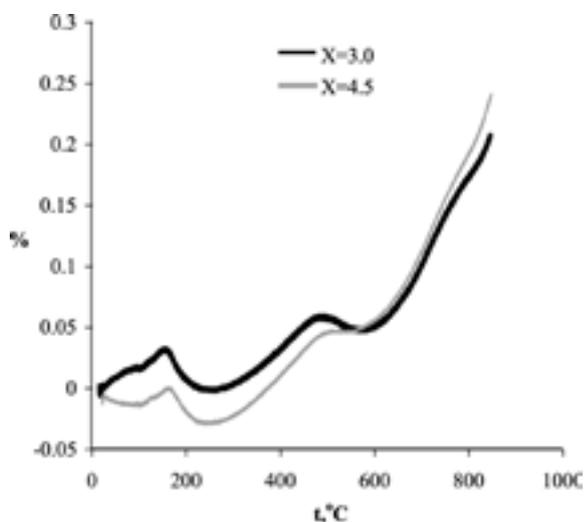


Figure 2 - Dilatometrical analysis of samples with $\text{P}_2\text{O}_5/\text{B}_2\text{O}_3$ molar ratio (X) = 3 and 4.5.

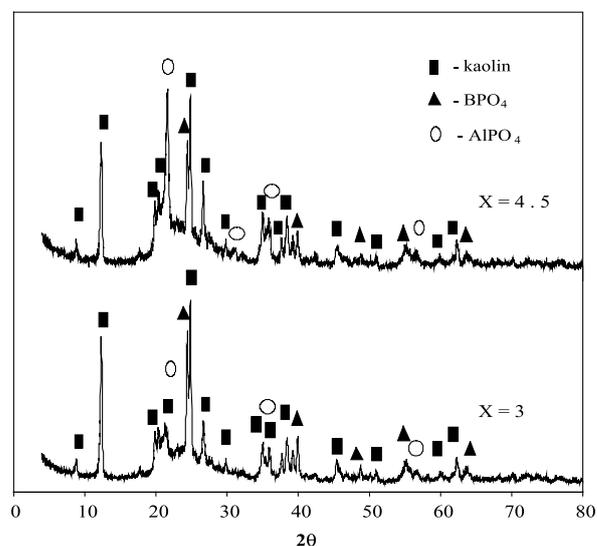


Figure 5 – XRD spectra of samples heated up to 550°C with multiple steps heating.

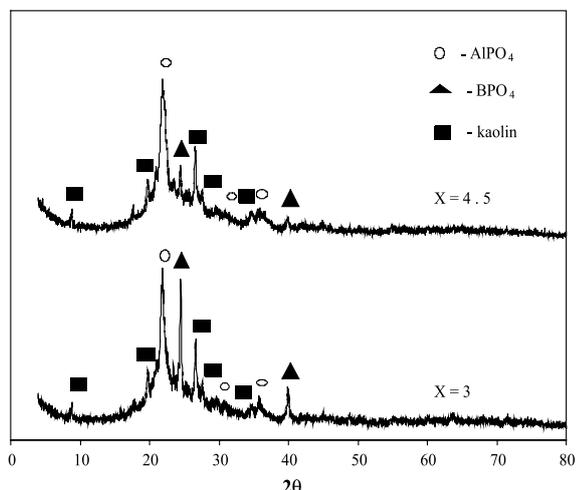


Figure 6 – XRD spectra of samples heated up to 800°C with multiple steps heating.

4.5), the amount of aluminum phosphate phase rises (Fig. 5). The XRD analysis of samples sintered at 800°C (Fig. 6) shows the presence of the same phases but the amount of BPO₄ decreases to compensate the amorphous phase formation, which provides a higher strength. All the referred XRD spectra were taken for samples corresponding to the binder matrix and not the final SiC-phosphate composite because the SiC peak masks all the others due to the high amount of this phase.

Samples of all compositions were finally mechanical characterized by bending and compressive tests. The corresponding results are listed in Tables 1 to 3. It can be seen that, by increasing the dwell temperature and the heating duration, the mechanical strength raises, since the needed time and energy for particles/matrix consolidation, densification and amorphous phase formation is provided. Fig. 7 show typical fracture microstructures of samples with X = 3 and 4.5 sintered at constant heating rate or multiple step heating, revealing a SiC transgranular failure mode with no net differences between the tested grades.

TABLE I. "MECHANICAL PROPERTIES OF COMPOSITIONS HEATED AT 4°C/min UP TO 550°C"

Molar ratio, P ₂ O ₅ /B ₂ O ₃ (X)	Bending strength, MPa	Compressive strength, MPa
3	12.1	28.8
3.5	12.8	39.1
4	17.4	49.3
4.5	11.1	37.3

TABLE II. "MECHANICAL PROPERTIES OF SAMPLES SINTERED AT 550°C WITH MULTIPLE STEPS HEATING"

Molar ratio, P ₂ O ₅ /B ₂ O ₃ (X)	Bending strength, MPa	Compressive strength, MPa
3	11.5	36.7
4.5	13.2	40.9

TABLE III "Mechanical properties of samples sintered at 800°C with multiple steps heating"

Molar ratio, P ₂ O ₅ /B ₂ O ₃ (X)	Bending strength, MPa	Compressive strength, MPa
3	13.2	44.2
4.5	15.7	52.4

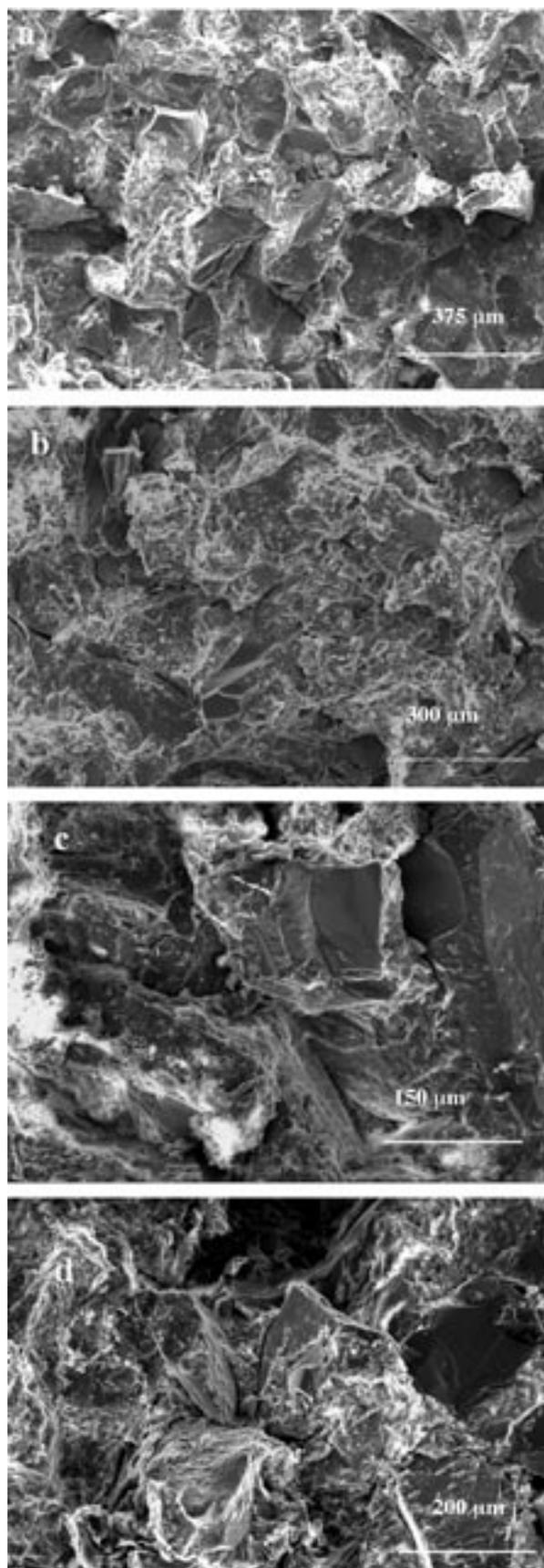


Figure 7 – Representative SEM micrographs of (X) P₂O₅/B₂O₃ molar ratio (X) = 3 compositions heated at 4°C/min up to 550°C; (b) X = 4.5 compositions heated at 4°C/min up to 550°C; (c) X= 3 samples sintered at 550°C with multiple steps heating; (d) X= 4.5 samples sintered at 800°C with multiple steps heating.

4. CONCLUSIONS

The use of colloidal solutions of B_2O_3 into H_3PO_4 are a very promising alternative route for binder preparation for SiC abrasive tools, as relatively low processing temperatures are used and final thermal and mechanical properties are kept. The presence of BPO_4 in the phosphate matrix compositions lowers the temperature needed for amorphous phase formation. The amount of the amorphous phase increases the final mechanical strength of the SiC-phosphate composite materials. Also, by increasing the sintering temperature and the heating step time, the mechanical strength rises as the time and energy for the composite consolidation and amorphous phase formation is guaranteed.

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