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Processing and Material Characterization of a Fiber-reinforced Ceramic Matrix Composite Used for Surge Protective Device Enclosure

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***Abstract*—Chopped quartz fiber-reinforced aluminum phosphate ceramic matrix composite was developed by reacting aluminum hydroxide ($Al(OH)_3$) with phosphoric acids at a temperature of 120° C and then curing at 250° C. The phase composition, microstructure, mechanical properties, fire resistance and electric insulation were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), 3-point bending tests, electric furnace burning and dielectric strength test, respectively. The composite matrix was found to consist of mainly phases of aluminum phosphate, gibbsite and boehmite. The composite material has a bending strength of 14.8MPa and fire-resistance up to a very high temperature (above 1100℃), with dielectric voltage strength between 2277V and 3238V, which is promising to be used for the enclosure of surge protective device (SPD) in order to improve the resistance to abnormal heat and fire of SPD.**

Keywords- ceramic matrix composite; properties; fire resistance; electric insulation; mechanical strength, SPD enclosure

I. INTRODUCTION

With the miniaturization of information communication technology (ICT) equipment, the demand for miniaturization of surge protective device (SPD) is becoming more and more prominent, while the reliability requirements of SPD is also more and more higher, especially for the fire resistance of the enclosure of SPD. The traditional material used for the enclosure of SPD is plastic. Although plastic can meet the requirements of SPD standard, such as IEC 61643-11 [1], in the practical applications, the fire accidents still occur occasionally. In comparison with plastic materials, ceramic materials are, in general, light-weight, chemically inert, refractory and hard. Most ceramics are also good electric insulators. These properties make them attractive for many applications. A major difficulty with the use of ceramics is their tendency to fracture catastrophically under various external loading conditions. To prevent ceramic materials from cracking in a brittle manner, they are often toughened by incorporation second reinforcements to form composites for crucial applications.

Among a variety of approaches to enhance their fracture toughness resistance, fiber reinforcement is effective for ceramic toughening. As demonstrated schematically in Fig. 1, extension of the cracking process in the ceramic matrix composite involves fiber-matrix interface debonding, frictional sliding, fiber bridging and subsequent fiber pullout to dissipate energy and its stress-strain behavior displays nonlinear quasi-ductility, compared to the brittle monolithic ceramic materials that break abruptly with very limited amount of deformation [2-5].

Surge protective devices are used to protect, under specified conditions, electrical systems and equipment against various overvoltages and impulse currents, such as lightning and switching surges. An SPD may fail when subjected to a surge greater than its designed maximum energy and discharge current capability. In IEC 61643-12 [6], failure modes of SPDs are divided into open-circuit and short-circuit mode. In the short-circuit mode, the system is severely influenced by the failed SPD. The short circuit current flows through the failed SPD from the power source. Energy dissipated during the conduction of short-circuit current may be excessive and cause a fire hazard, which consequently cause serious damage to properties and telecommunication equipment. Fiber-reinforced ceramic matrix composites display various excellent properties, but they have been studied extensively for high-temperature load-bearing structural materials and their potential applications for lightning protection remain less explored. In this work, chopped quartz fiber-reinforced aluminum phosphate ceramic matrix composite was produced by reacting aluminum hydroxide ($Al(OH)_3$) with phosphoric acids under benign conditions, and the material phase composition, microstructure, mechanical properties, fire resistance and dielectric strength voltages were investigated.

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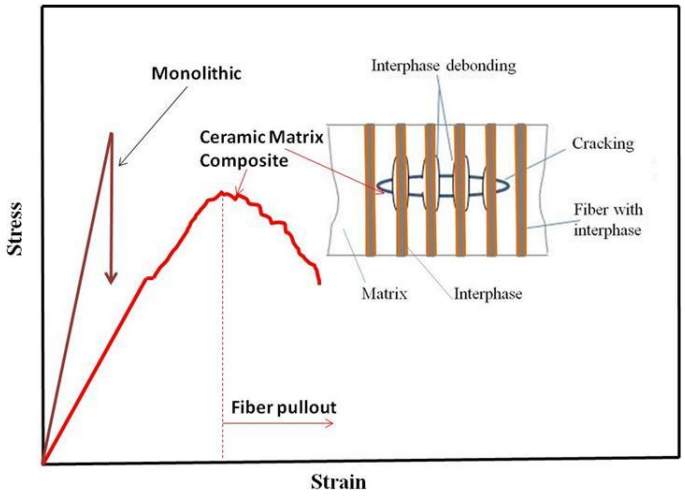

Figure 1. Schematic of monolithic ceramic and fiber-reinforced ceramic matrix composites.

II. MATERIALS PROCESSING AND CHACTERIZATION

A. Processing

Many preliminary investigations on processing conditions led to the following formula. 15.00 grams of phosphoric acid with concentration no less than 85% (Sinopharm Chemical Reagent), 12.00 grams of aluminium hydroxide ($Al(OH)_3$) powder of purity 97% (Shanghai Chaoqiang Chemical), 3.00 grams of kaolinite (Shanghai Fengxian Fengcheng Reagent) and 0.70 gram of chopped quartz fibers (Hubei Feilihua Quartz Glass Co.) were mixed vigorously in a plastic beaker. The beaker was then kept in an oven pre-heated at 120 ℃ in ambient air for 20 minutes, which was taken out for occasional stirring. The products were moved into a mold, consolidated and hot-pressed under a pressure of 30MPa at 120 ℃ in ambient air for about 2 hours before complete curing at 250 ℃ for 1 hour. The final products obtained were thin plates 100mm long, 50mm wide and 1 mm thick (also see ahead to a specimen photograph of Fig. 3).

B. Chacterization

1) XRD and scanning electron microscope Anlysis

The composite sample was ground into fine powders. Phase analyses were performed using a BRUKER-AXS D8 Advance X-Ray diffractometer system (Germany), operated with Cu Kα（λ= 0.1541nm） incident radiation at 35mA and 40 kV. Data acquisition was carried out in continuous scanning mode over the range 10 to 90 °(2θ) with a step width of 0.02 °and a scan speed time of 0.30s/step.

Scanning electron microscopy (FEI SIRION 200 Field Emission SEM) was employed to investigate the composite morphology.

2) Bending test

The force/deflection behavior of the composite was measured in a 3-point bending test on a universal testing machine (CMT 4503, MTS). The span length was 30mm. Test pieces were cut into samples 50mm long, 10mm wide and 1mm thick. The loading was applied at a cross-head speed of 1-5 mm/min until fracture.

3) Dielectric Strength Test

Dielectric Withstand Tester (ES2/5KV) was used to measure the voltage and leakage current by direct current mode. The voltage was gradually increased and the current was measured. The maximum voltage was applied within 10 seconds and the duration of the test is 60 seconds. The leakage current limit was set to 10 mA. The test was performed in a controlled environmental temperature 20 ℃ and relative humidity 80%. Three respective measurements were carried out on three different samples.

4) Fireproofing test

An electric furnace was preheated to 1100 ℃ and a piece of the composite specimen was then placed into the furnace to detect whether the composite material burns.

III. RESULTS AND DISCUSSION

Fig. 2 shows the X-ray powder diffraction patterns of the composite, along with standard XRD peak positions of aluminium phosphate (pdf # 20-0045), kaolinite (pdf # 14-0164), boehmite or böhmite (pdf # 21-1307) and gibbsite (pdf # 33-0018). With all diffraction peaks assigned properly, it is clear that the main crystalline phases of the composite materials are aluminium phosphate, kaolinite, boehmite and gibbsite. Small amount of quartz fibers from the raw material is present, but is of a typical amorphous nature so that it does not show up in Fig. 2.

It has been known that aluminum phosphate forms different polymorphous crystallized phases [7]. Depending the type of aluminium oxides, P/Al ratios, acidic, neutral and basic conditions, and reaction or heat treatment temperatures, possible products may be aluminum metaphosphate $Al(PO_3)_3$, aluminum cyclohexaphosphate $Al_2P_6O_{18}$, berlinite or aluminum orthophosphate $AlPO_4$ and aluminum dihydrogen tripolyphosphate $H_2AlP_3O_{10}$·H_2O [7-13].

In this work, dissolution of various forms of alumina in an acidic condition is given by the following reactions [14].

$$AlO_{3/2}(3/2)H_2O + 3H^+ \rightarrow Al^{3+}(aq) + 3H_2O \quad \text{for gibbsite.} \quad (1)$$

In the pH range equal to 2–8, dissolved phosphate species such as $H_2PO_4^-$ and HPO_4^{2-} are abundant The cationic concentration Al^{3+} in the acidic range is high, but rapidly decreases as the pH is increased. The neutralization of the acid in this range occurs due to the following reaction [7, 14]

$$Al^{3+} + H_2PO_4^- + HPO_4^{2-} + H_2O = AlH_3(PO4)_2 \cdot H_2O. \quad (2)$$

Once $AlH_3(PO4)_2$·H_2O is formed, it subsequently reacts with the remaining oxide components on the surface of their

grains to form berlinite and binds them. The reaction may be written as [7]

$$Al(OH)_3 + AlH_3(PO4)_2 \cdot H_2O \rightarrow 2AlPO_4 + 4H_2O. \quad (3)$$

$AlPO_4$ (berlinite) is the bonding phase that binds individual particles and forms the ceramic [7]. The current work confirmed early studies [10-13] in phosphate bonding that involved room temperature bonding of oxide-phosphoric acid mixtures and formation of monoaluminum phosphate ($AlH_3(PO_4)_2 \cdot H_2O$) at 100–150 ℃ and its conversion to berlinite above 150 ℃ . [15] studied the isothermal decomposition of gibbsite (aluminum hydroxide) over the temperature range from 458 to 508 K and confirmed its partial conversion to boehmite (AlOOH), which is coincident with the presence of the boehmite phase in Fig. 2.

Shown in Fig. 3 are a photograph of the composite specimen with the dimension stated above and a micrograph of its magnified SEM structure. The shape and distribution of fiber reinforcement can be clearly identified. Small defects in the form of pores appeared in the fiber-rich region. The fiber diameter is roughly 5μm with an average length of 30mm (full length is not shown in this figure).

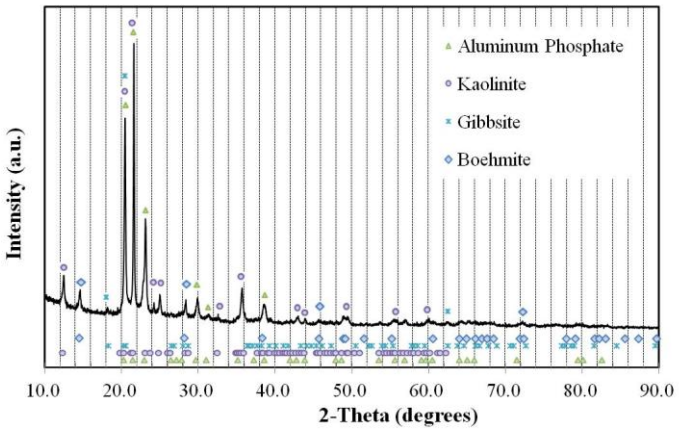

Figure 2. Typical XRD patterns of the composite materials.

Fig. 4 presents a typical bending stress-crosshead displacement curve obtained from the 3-point bending tests. The stress-displacement relationship shows an initial linear stage corresponding to elastic deformation, but deviates to nonlinear deformation before reaching the maximum stress. During the quasi-ductile stage, crack deflection, fiber-matrix debonding, fiber pullout, and fiber bridging were recognized as crack growth retarding mechanisms. These toughening mechanisms in the fiber-reinforced composites impart the materials with mechanical toughness, which is essential to avoiding catastrophic failure. The ultimate bending strength is 14.8MPa.

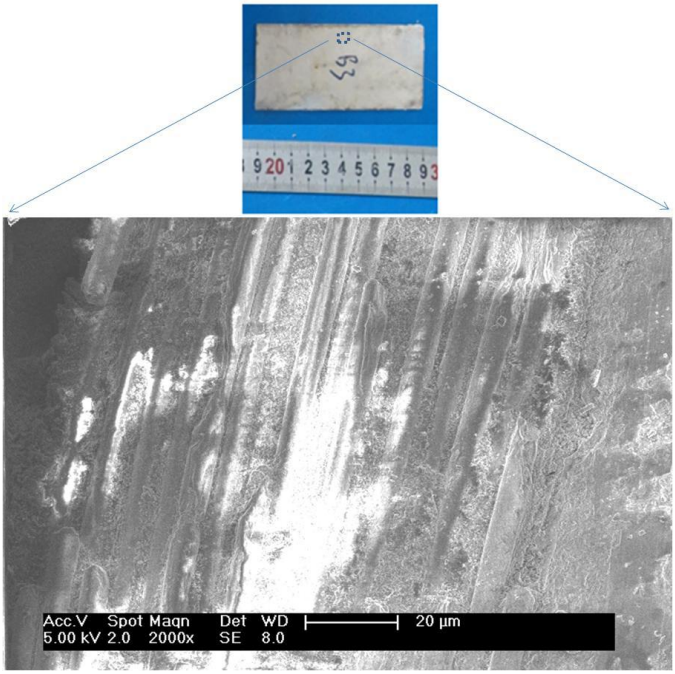

Figure 3. A sample photo (above) and its typical SEM micrograph (below) of fiber-reinforced ceramic matrix composites.

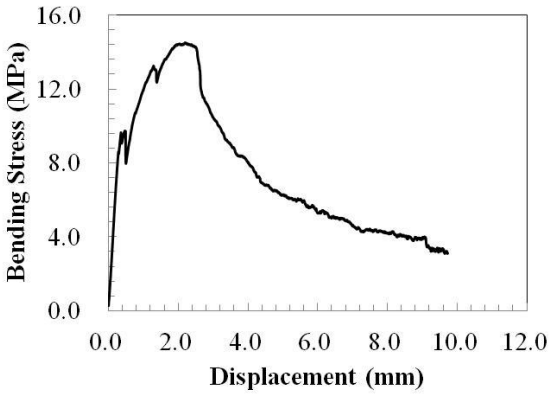

Figure 4. Bending stress versus crosshead displacement.

Breakdown voltages for three composite samples are $2702V_{rms}$, $2277V_{rms}$ and $3238V_{rms}$, as shown in table I, all exceeding $2000V_{rms}$, similar to the data reported in [16] for measuring under dry condition the dielectric strength of an aluminium phosphate sealing on plasma-sprayed alumina coatings. Further enhancement of the breakdown voltage may be possible by increasing heat treatment temperatures.

TABLE I. TEST RESULTS OF BREAKDOWN VOLTAGE TEST

Breakdown voltage (V_{rms})	**Test reults**		
	Sample 1	***Sample 2***	***Sample 2***
1500	Pass	Pass	Pass
2000	Pass	Pass	Pass
Maximum	2702	2277	3238

Fireproofing testing indicates the composite materials do not burn, when put suddenly to a high-temperature furnace of 1100℃. This has demonstrated the excellent fire resistance of the material.

IV. CONCLUSION

Chopped quartz fiber-reinforced matrix composites were prepared through phosphoric acid reacting with aluminium hydroxide ($Al(OH)_3$) with addition of kaolinite and the main matrix phase composition were aluminium phosphate, kaolinite, boehmite, gibbsite and kaolinite. The composite material showed a nonlinear quasi-ductile deformation behavior with a bending strength of 14.8MPa and its dielectric voltage strength varied between 2277V and 3238V. The fiber-reinforced composite is stable and fire-resistant at very high-temperature. The materials developed in this work are promising for lightning-related fire prevention and modest high-voltage protection.

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