COPPER-PHOSPHATE BONDED HIGH STRENGTH FIBER COMPOSITE MATERIAL

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SUMMARY

Copper-phosphate bonding can offer a low cost process to fabricate high strength fiber composites. When the volume friction is 25 — 30% its flexure strength can reach 382-430 MPa below 350°C, 113-280 MPa at 700°C and the specific gravity is 2.8-3.0 g/cm³.

Chemical copper plating on the fibers can separate the fibers from each other and improved the infiltration process.

KEYWORDS: phosphate, inorganic adhesive, fiber composite

INTRODUCTION

CuO/H₃PO₄ reaction has been developed in China for about 40 years[1] and it is widely used as a kind of high temperature inorganic adhesive. This reaction takes place in situ during processing at a low temperature of about 100°C, so it can also be used as a binder to produce fiber composite materials. Its product is copper phosphate (CuHPO₄•HO₂) and has been proved with good water stability, heat insulation and aging stability. The main physical properties of this adhesive are shown in table 1. By using it this paper developed a kind of high strength composite material and its flexural strength from room temperature to 700°C have been tested. The test shows it is suitable for military applications, such as radomes of tactical missile to resist the air-friction heating. All the processes are easy to carry out to fabricate application structure and are of much lower processing costs than other ceramic material processes.

Chemical bonding techniques are based on the formation of strong bonds as the result of room temperature chemical reaction. This reaction is based on the formation of metal phosphates. In 1995 Prof.A.Kojima and S.Hoshii of Gunma National College of Technology developed a kind of fiber composite bounded by Al_2O_3/H_3PO_4 reaction, Its flexural strength has reached 516 Map[2].

Material	Density (g/cm ³)	Specific heat (J/Kg•K)	Thermal conductivity (watt/M•K)	Expansion coefficient (K ⁻¹)	Melting point (°C)	Specific resistance (Ω•cm)
copper phosphate adhesive	3.1-3.5	1400	0.93	12×10 ⁻⁶	950	6×10 ⁶
Titanium	4.5	580	15	8×10 ⁻⁶	1600	42×10 ⁻⁶
Aluminum	2.7	920	209	23.1×10 ⁻⁶	690	2.8×10 ⁻⁶

Table 1. The physical properties of copper-phosphate inorganic adhesive

EXPERIMENTAL MATERIALS

The fiber used here is M40j(6K) carbon fiber and Nicalon 202 SiC fiber made in Japan TORAY Inc. The properties of the fibers are shown in table 2. The phosphor acid solution has been concentrated at about 130° C and a few of Al(HO)₃ added in it at first. The CuO powder was been treated at different temperature and the size is less than 5 µm.

Table 2. The properties of the fiber

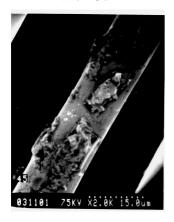
fiber	Tensile strength (MPa)	K	Average diameter(µm)
Nicalon 202	2500	500	10
M40J	4400	6000	5

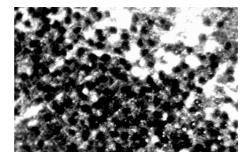
FABRICATION OF THE COMPOSITE

Heated fibers at 700°C under the protection of N₂ for 10 seconds the original adhesive on surface of the fibers can be removed, then a chemical copper-plating process was proceeded. Only two minutes were needed to plate Cu onto the fibers at 25 °C. Figure 1 is the plated fiber. After that the fibers were cut into 15 cm length and arranged on a polyester film in the same direction for use.

In experiment the technical for fabricating the specimen was carried out as follows: Firstly, a kind of inorganic adhesive paper was prepared. It was prepared by coating the inorganic adhesive on the surface of another polyester film. By control the diameter of the CuO powder and the surface activity we can control the solidify velocity of the CuO/H₃PO₄ reaction. Secondly, covering the adhesive paper on the arranged fiber and rowing on the top side with a round bar we can infiltrate the adhesive into the fiber gaps and well wetted with it.

Figure 1. The SEM observation of C_f coated Figure 2 . The micrograph of C_f CuHPO₄•HO₂ with Cu





After infiltration the paper with fiber would be cut into a tape (15×150 mm²). Put the tape on a die then tear the polyester film away, do above process again and again till the thick of the specimen in the die was 2 mm. Closed the die and put it in a oven then heated to 60 °C for 2 hours and to 100 °C for 2 hours the specimen was prepared. All the processes demonstrated low

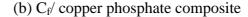
cost and easy to fabricate various structures. Infiltration effect is shown on figure 2. It was found that the Cu player on the fibbers is very useful to improve the infiltration process. The small Cu powder on a fiber can separate the fibers from each other and improve the infiltration ability. The volume fraction of the specimen can reach 25 - 30% and the density is about $2.8 - 3.0 \, \text{g/cm}^3$.

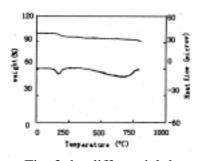
RESULTS AND DISCUSSION

To find the temperature stability of these composite materials, the differential thermal analysis and the thermo gravimetric analysis were measured. Fig 3 is the curves. Their total weight lost are less than 6.2% and it happened mainly during room temperature to 400°C. There is a heat absorption peak at 164°C for the copper- phosphate matrix and the heat absorption peak of the composite is higher about 50°C.

The flexure strength of the specimens were tested at room temperature, 350°C and 700°C. Their results are shown in table 3. Before testing the specimen has been keep at the high temperature for 10 minutes. The size of the specimens is $50 \times 7 \times 2$ mm³. The testing machine is WDW-10KN produced by Doli-CCTM. The effect of fiber type can by seen in table 3. For the carbon fiber reinforced composite can by used blow 350°C and the Nicalon fiber can stand higher temperature.







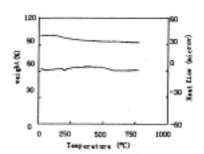


Fig. 3 the differential thermal analysis and the thermo gravimetric analysis

Table 3. The bending strength of copper-phosphate bonding fiber composite materials

Material \ temperature	25°C (MPa)	350°C (MPa)	700°C (MPa)
carbon fiber /Copper-phosphate	382	408	113
SiC fiber/ Copper-phosphate	387	430	280

REFERENCES

- 1. Xiao-xian He, "Inorgnic Adhesive and Application", edited by Jing-tai Cuei, published by National Defense Industry Publish Company, 1993, in Chinese
- 2. A.Kojima and S.Hoshii, "High-Strength Composite Carbon Fiber Using Phosphate Parent Material", JETRO, November 1995, pp. 18-19
- 3. I.G.Talmy, C.A.Martin, D.A.Haught, "Development of Phosphate-Bonded Silicon Nitride Ceramics and Ceramic-Matrix Composites", Technical Digest, September 1993, pp. 9-15