

## Weathering of $\text{H}_3\text{BO}_3$ -ZnO Bond for Forming Glass Fiber Reinforced Composite

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**Synopsis.**  $\text{H}_3\text{BO}_3$ -ZnO bond stored in humidity above 75% causes the bending strength of a fiber reinforced composite to decrease. The lowering behavior corresponds to formation of  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  and to a decrease of  $\text{H}_3\text{BO}_3$ . The weathering of the bond is attributed to the decreasing of  $\text{H}_3\text{BO}_3$ , which becomes liquid during hot pressing.

A powder mixture of  $\text{H}_3\text{BO}_3$  and ZnO can be used as a bond for forming a glass fiber reinforced composite with hot pressing and subsequent heat treatment.<sup>1)</sup> Hot pressing at 170 °C makes the powder mixture melt and makes it penetrate into the spaces of the reinforcing elements. Heat treatment advances the reaction in the bond and makes the composite durable. However, long term storage of the mixture sometimes decreases its bonding power. The decreasing behavior resembles the weathering of portland cement.

This study deals with the cause of weathering in this  $\text{H}_3\text{BO}_3$ -ZnO bond.

### Experimental

The bond was prepared with a powder mixture in 3 : 1 mole ratio of  $\text{H}_3\text{BO}_3$  and ZnO. It was stored in various humidity atmospheres at 30 °C for 7 d. Humidities were kept constant with water vapor of saturated aqueous solutions of salts, which were  $\text{KNO}_3$ , KCl, NaCl, and  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  for 92, 84, 75, and 52%, and with drying silica gel for 0%. A composite was prepared from the stored powder and glass fiber mats with the composition of 40 wt% and 60 wt%. To form a composite, the powder layer and the glass layer were laminated one after the other and were pressed under 100 kg  $\text{cm}^{-2}$  at 170 °C. The pressure was sometimes taken away to eliminate excess water produced by dehydration of  $\text{H}_3\text{BO}_3$ . The composites were used for testing the bending strength. On the other hand, the crystal phases and their behavior in the stored bond were examined by X-ray diffraction and differential thermal analysis.

### Results and Discussion

In forming a composite, the bonds which were stored in low humidity penetrated fully into the spaces of glass fiber mats, whereas those which were stored in high humidity penetrated only partly into the spaces. The test pieces for the bending strength were taken from a part of a composite into which the bond had penetrated. Figure 1 shows the relationship between bending strength of composites and storing humidity of bond. As shown in Fig. 1, there was no significant difference between the bending strength of a composite which was formed by the bond stored in 0% humidity and the bending strength of one which was formed by the bond immediately after mixing. Moreover, the bending strength was independent of storing humidity up to 75%. However, the storage of the bond in humidity above

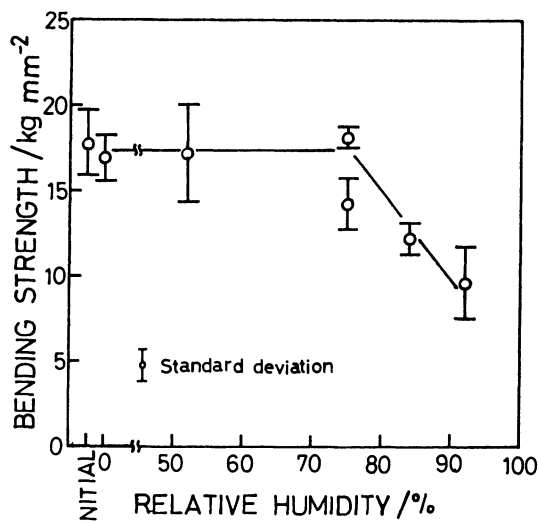


Fig. 1. Bending strength of composite formed with bond stored in various humidity.

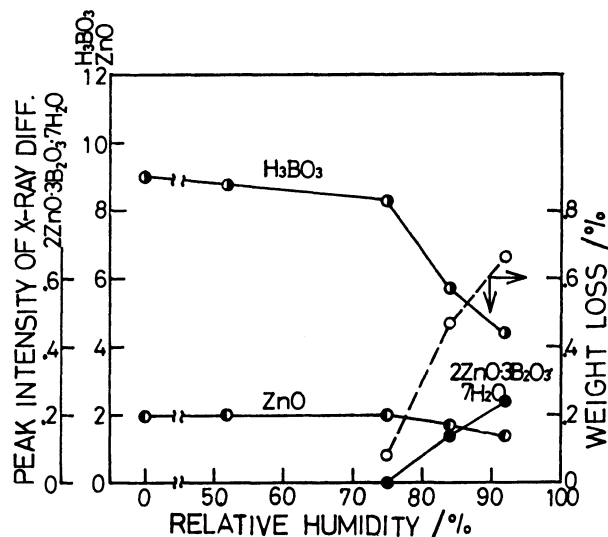


Fig. 2. Peak intensity of X-ray diffraction of crystal phases in bond stored in various humidity.

75% caused the bending strength to decrease.

The X-ray diffraction pattern of the bond immediately after mixing showed peaks of  $\text{H}_3\text{BO}_3$  and ZnO crystals. However, the patterns showed that the bond stored in humid atmosphere contained not only  $\text{H}_3\text{BO}_3$  and ZnO but also  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  crystal.<sup>2,3)</sup>

The variation in each one of the diffraction peaks of  $\text{H}_3\text{BO}_3$ , ZnO, and  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  crystals is plotted as a function of storing humidity in Fig. 2. In humidity above 75%,  $\text{H}_3\text{BO}_3$  was decreased in the bond and a new crystal of  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  was produced instead of  $\text{H}_3\text{BO}_3$ . The starting humidity of the decrease

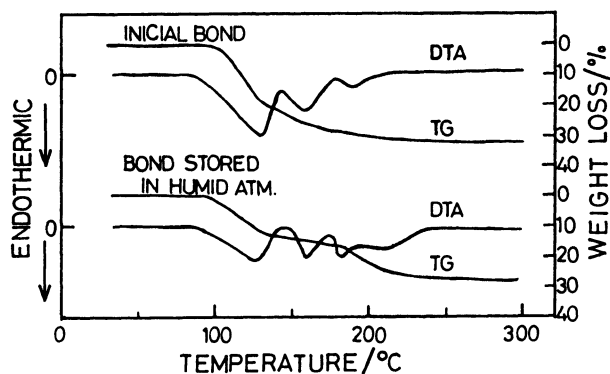


Fig. 3. DTA and TG of initial bond and of bond stored in 84% humidity.

or the production agrees with that of bending strength. Figure 2 also shows weight loss from the initial bond. The crystal  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  contains less water than the composition of initial bond, that is,  $2\text{ZnO} \cdot 6\text{H}_3\text{BO}_3 = 2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 9\text{H}_2\text{O}$ . The weight loss in Fig. 2 seems mainly to be water loss caused by production of the new crystal and partly to be  $\text{H}_3\text{BO}_3$  loss due to its sublimation in humid atmosphere.<sup>4)</sup>

Differential thermal analysis of the bond is shown in Fig. 3. A broad peak appears from about 170 °C to 240 °C, omitting the three peaks of  $\text{H}_3\text{BO}_3$  dehydration; this peak corresponds to dehydration of  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  crystal. The crystal became amorphous as a result of its

dehydration. It seems to behave as a filler in a composite, because its dehydration temperature is rather higher than the hot pressing temperature. However, its production may somewhat obstruct the flow of the bonding liquid under hot pressing by bridging between  $\text{ZnO}$  particles. On the other hand, the decrease of  $\text{H}_3\text{BO}_3$  causes the bonding liquid to decrease under hot pressing.  $\text{H}_3\text{BO}_3$  considerably decreased as humidity increased above 75%, as shown in Fig. 2. Therefore the decreasing behavior of bending strength is attributed to  $\text{H}_3\text{BO}_3$  decreasing in the bond due to the production of  $2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  crystals.

It is concluded that the decrease of  $\text{H}_3\text{BO}_3$  in humid air gives rise to the weathering of the bond in the system  $\text{H}_3\text{BO}_3 \cdot \text{ZnO}$ .

#### References

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