

# Chemical Properties of Fused Fiber Coupler Surface

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The fused fiber coupler is examined for its glass properties. The process of fusion by an H<sub>2</sub> flame causes not only an increase of H<sub>2</sub>O combined with SiO<sub>2</sub> but also a loose SiO<sub>2</sub> Structure and a rough surface near the center of the coupler. Such a change in glass properties is inevitable for commonly produced fiber couplers, although its effect on the long-term stability of the couplers is not known at this time. © 2000 Academic Press

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## 1. INTRODUCTION

With respect to materials reliability of fused fiber couplers, their glass properties are investigated here. Arc-fused fiber splices have been studied from a similar viewpoint, and the occurrence of anomalies in the glass property near the spliced point was reported by Volotinen *et al.* [1]. Reportedly, Fourier transform infrared (FTIR) spectroscopy and atomic force microscopy (AFM) are reliable methods for microanalysis of glass fibers used here for the examination of fused couplers. They confirm a change in their glass properties, similar to a fused splices. In particular, the glass structure of the regions away from the center of the coupler is slightly deteriorated, possibly leading to lower stability of the coupler under humid conditions.

## 2. EXPERIMENTAL RESULTS

The fiber couplers for evaluation were fabricated by a common fusion process consisting of placing a pair of bare glass fibers in contact with each other, tensing the fibers, heating the center of the fibers with an H<sub>2</sub> flame, and pulling the fibers to make a coupling region. Approximately 5 mm of the fibers was heated during the process, and the fibers fused together and became elongated.

At first, in order to examine the chemical state of hydrogen in the fiber coupler, thermal desorption spectrometry (TDS) was carried out. Three pieces of coupler including the fused region (approximately 15 mm) were cut from the fibers and placed in the chamber of the TDS analyzer. The sample was heated to 1000°C at a rate of 60°C/min under vacuum ( $4 \times 10^{-7}$  Pa), with gases desorbing from the sample being detected by a mass analyzer. For comparison, the same measurement was carried out using standard bare fibers, i.e., before the fusion process: this was the reference fiber. Figure I shows the TDS results with respect to H<sub>2</sub>O (mass number 18), in which desorption profiles for the coupler and the reference fiber are shown by a black line and a gray line, respectively. A sharp peak at 150°C and a broad peak from 200 to 300°C are observed for both samples. Judging from the temperatures, these peaks were due to the H<sub>2</sub>O molecules that had adsorbed on the fiber surface. For the coupler sample, an additional peak appeared at 500°C, indicating H<sub>2</sub>O combining with SiO<sub>2</sub> more strongly. The most plausible origin for this H<sub>2</sub>O peak was the desorption of the Si-OH in the glass structure.

The glass structure of the coupler was examined by the attenuated total reflection (ATR) method of FTIR, in which the SiO<sub>2</sub> structure near the fiber surface could be detected. Figure 2 shows the peak position of an asymmetric Si-O-Si stretching band that was measured at various positions along the fiber (marked by black circles). The white circle denotes the result on the reference fiber without any coating. Only the peak positions measured 4 and 6 mm from the center of the coupler were slightly higher (by about 10 cm<sup>-1</sup>) than those measured near the coupler center and on the reference fiber. A similar tendency was observed in the measurements on the symmetric Si-O-Si stretching band around 800 cm<sup>-1</sup>, as shown in Fig. 3. The shift of the Si-O-Si stretching peak position, i.e., the vibration energy of stretching mode, is caused by changes in the Si-O bond distance, the Si-O-Si bond angle, etc. [2-5]. This shift toward a higher position suggests a decrease in the fictive temperature of the glass and an increase in the Si-O-Si bond angles, plausibly due to an increase of tensile stress.

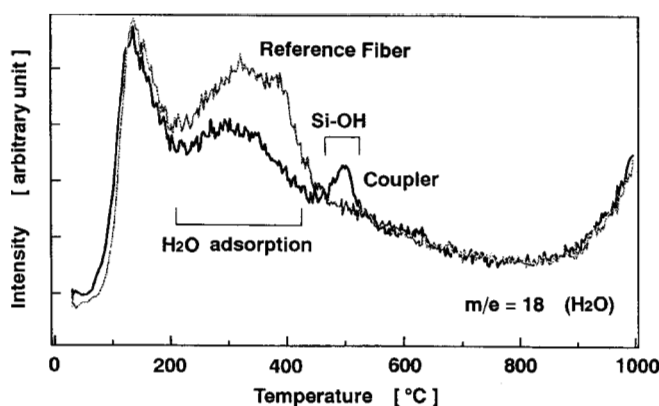


FIG. 1. TDS profiles with respect to H<sub>2</sub>O (mass number 18): a black line for the coupler and a gray line for the reference fiber.

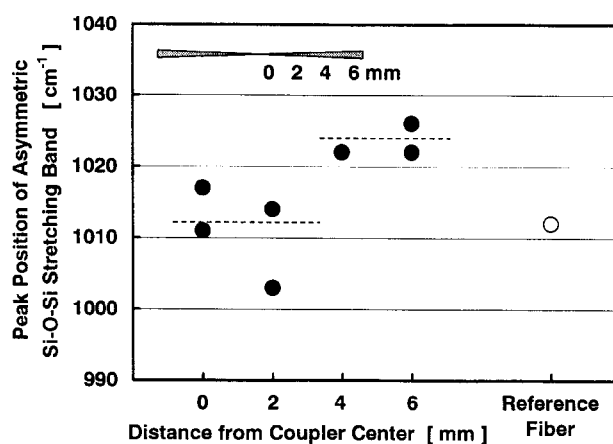


FIG. 2. , FTIR peak position of an asymmetric Si-O-Si stretching band measured at various positions along the fiber coupler. , a similar measurement result on the reference fiber without any coating.

For a final evaluation, the surface roughness was measured by AFM along the fiber, and typical images of observed surface morphology are shown in Fig. 4: (a) is the center of the coupler, (b) is the position 2 mm from the center, (c) is the position 4 mm from the center, (d) is the position 6 mm from the center, and (e) is the reference bare fiber. All of the images of Fig. 4 are shown for a  $500 \times 500$  nm area with the same height magnification; one division of the vertical axes denotes 1 nm. The surface morphologies of all observed positions were similar, but the surface of the positions 2 to 6 mm from the coupler center seemed to be slightly rougher.

Figure 5 shows the position dependency of the surface roughness,  $R_a$ , measured for the  $1 \times 1$   $\mu\text{m}$  area. The surface roughness of the positions 2, 4, and 6 mm from

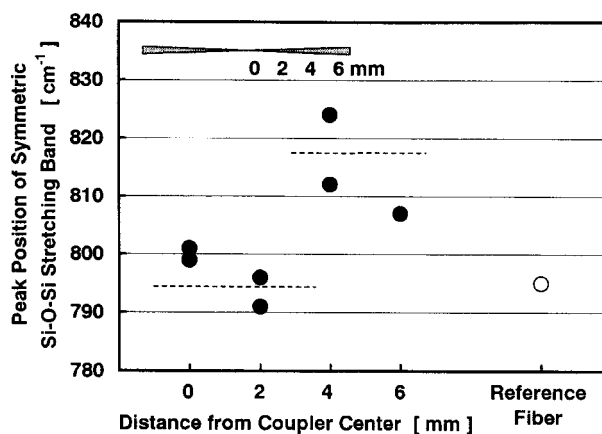


FIG. 3. , FTIR peak position of a symmetric Si-O-Si stretching band measured at various positions along the fiber coupler. , a similar measurement result on the reference fiber without any coating.

CHEMICAL PROPERTIES OF FUSED FIBER COUPLER SURFACE

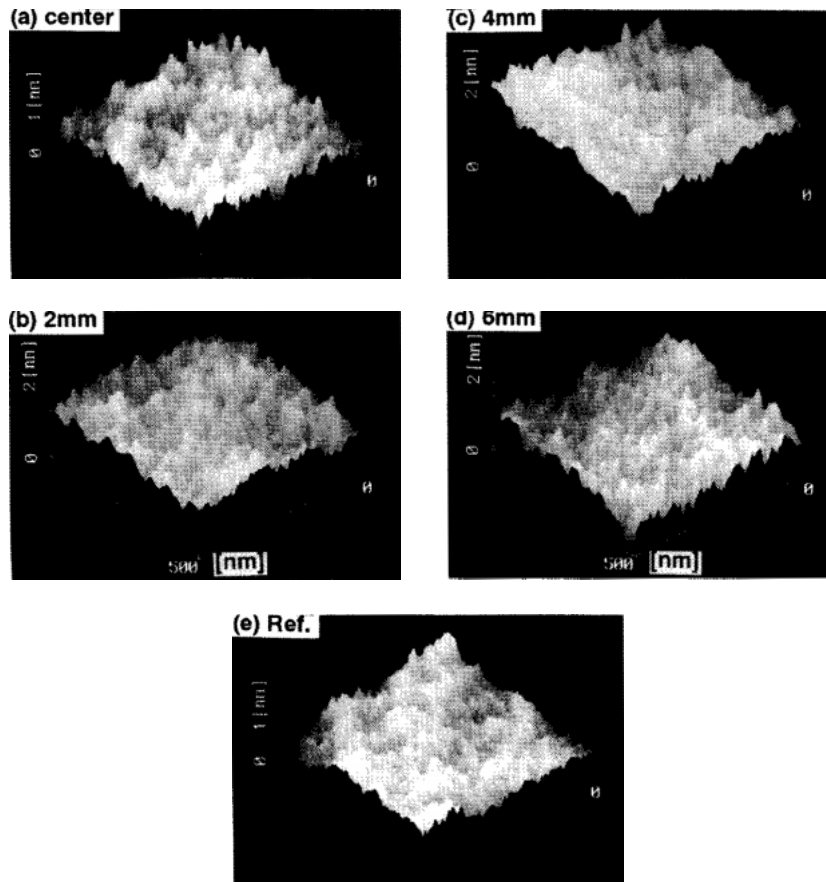


FIG. 4. AFM images: (a) center of the fiber coupler, (b) position 2 mm apart from the center, (c) 4 mm from the center, (d) 6 mm from the center, and (e) the reference bare fiber.

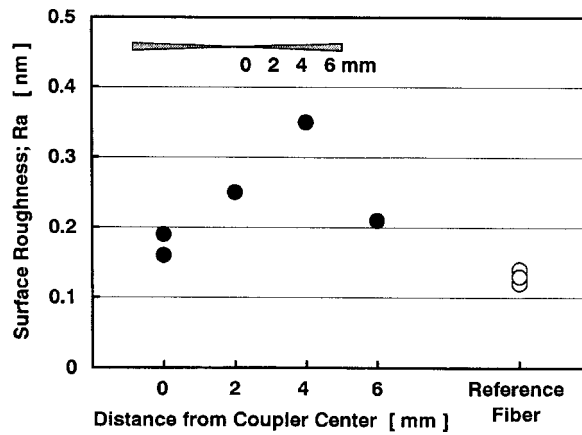


FIG. 5. , position dependency of surface roughness,  $R_a$ , of the fiber coupler measured by AFM for the  $1 \times 1 \mu\text{m}$  area. , a similar measurement result on the reference fiber without any coating.

the coupler center had deteriorated. Even at the center, their surfaces were slightly rougher than the surface of the reference fiber.

### 3. DISCUSSION AND CONCLUSION

The facts determined for the fused fiber couplers are:

- (1) The presence of H<sub>2</sub>O combining strongly with SiO<sub>2</sub> as in Si-OH,
- (2) Distortion of the SiO<sub>2</sub> structure in the fiber surface 4 to 6 mm from the center of the coupler, and
- (3) Slight coarsening of the fiber surface especially in the region away from the coupler center.

The incorporation of H<sub>2</sub>O into the glass structure was probably caused by use of the H<sub>2</sub> flame in the fusion process. Here, we consider that the resultant loose SiO<sub>2</sub> structure and rougher fiber surface are disadvantageous for the fiber couplers to be stable against a H<sub>2</sub>O attack from ambient high humidity. However, a complete elimination of such a structural deterioration in the glass fiber is difficult, because the deterioration is inevitably caused by the fusion process.

In a common fabrication process of fiber couplers, only a certain short length of the fibers corresponding to the center region of the coupler is heated with an H<sub>2</sub> flame, and the fibers are pulled outward from the flame. In this procedure, the cooling rate of the pulled-out fibers may not be rapid because of the small distance from the flame, resulting in a decrease of their fictive temperature. Further, because the fibers are tensed during the fusion process and cooled, the SiO<sub>2</sub> network structure is loosened. The structural distortion in the glass fiber expected from this assumption was consistent with the FTIR measurement results. On the other hand, the center region of the coupler is heated throughout the process, becomes narrower in its diameter, and is then abruptly cooled by turning off the torch. Thus, the center region is expected to keep the higher fictive temperature and the smooth surface.

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