Eftect of Heating on Jacketed Optical Fibers

Hirotoshi Nagata

Optoelectronics Research Division, New Technology Research Laboratories, Sumitomo Osaka Cement Co., Ltd., 585 Toyotomi-cho, Funabashi-shi, Chiba 274-8601, Japan E-mail: hinagata@sits.soc.co.jp

Received November 8, 1999

Optical fibers installed in optoelectronic devices are frequently exposed to heat during assembly and inspection. In order to examine the effect of heating the fibers, the thermomechanical performance of a commercial fiber jacketed with a UV-cured acrylate was measured and partial delamination of the jacket materials between the primary and secondary coats was found after the test. Further, it is possible that delamination may occur even at temperatures less than the 80°C required for burn-in of the devices. © 2000 Academic Press

1. INTRODUCTION

In the fabrication of optoelectronic devices, installed optical fibers are heated together with the devices for their burn-in-test after assembly. Temperatures of up to 85°C are commonly applied to devices as an aging and/or heat cycles test [1, 2]. Similar tests for system components are performed at lower temperatures, e.g., 50 to 65°C (expected operation temperature). However, the effects of burn-in on the commercial jacketed fibers, which are designed mainly for long-haul usage, are not well known. For instance, because a fiber jacket is not designed to strongly bond to the fiber in order to reduce a jacket-stripping force, it is possible that the jacket may debond from the fiber at elevated temperatures [3]. Concerning the abrasion resistance of jacketed fibers, Wissuchek *et al.* reported that debonding of the primary coat from the glass fiber was caused by a physical impression onto the jacket surface [4]. The thermal resistance of fiber jackets has been investigated by Gebizlioglu *et al.* [5, 6]. They found that the glass transition temperature (T_g) of the jackets changed depending on the history of the fibers such as fabrication lot and



environment, through their original dynamic mechanical thermal analysis (DMTA) on commercial fibers.

In this report, the thermomechanical performance of jacketed fibers is similarly examined, but from the viewpoint of whether or not the device burn-in temperatures cause a problem in jacketed fibers. It is a fact that delamination of jackets is sometimes found in installed fibers after burn-in tests. The points of delamination can be detected as many surface reflections appearing sporadically along the fiber. In order to monitor the occurrence of delamination, a dynamic mechanical thermal analysis method was applied to commercial UV-cured acrylate jacketed fibers and the jacket materials were found to debond even at temperatures that are typical for device burn-in tests. However, because most of the delamination was found between the primary and the secondary jackets, and not at the interface with a glass fiber, the fiber itself was thought to remain intact.

2. EXPERIMENTS

A 400-, μ m-diameter jacketed fiber was used as a sample for thermal and thermomechanical analyses of its jacket materials. Figure 1 shows a cross-sectional optical micrograph of the fiber, in which the outer diameters of the primary and secondary jackets were measured to be 240 and 400 μ m, respectively. The material for the secondary jacket was expected to be UV-cured acrylate.

In order to examine the thermal performance of the fiber jackets, samples including a glass part were set in a differential scanning calorimeter (DSO and a thermogravimetry-differential thermal analyzer (TG-DTA). A DSC measurement was carried out on the 10-mg sample under flowing N_2 from - 150 to 300°C with a heating rate of 40°C/min. The TG-DTA was done on another 5-mg sample under flowing N_2 from 30 to 600°C with a heating rate of 10°C/min. Thermomechanical



FIG, l. Optical micrograph of a cross section of the 400- μ m-diameter jacketed fiber used for the experiments. The glass fiber was double coated with a 240- μ m-diameter primary jacket and a 400- μ m-diameter secondary jacket.

characteristics of the samples were measured with a dynamic mechanical analyzer (DMA). In the DMA measurement, a cyclic tensile stress (0.05% at I Hz) was applied to the approximately 22-mm-long jacketed fiber sample under flowing N_2 , while the ambient temperature was changed from -50 to 300°C (150°C for some samples) by 3°C steps, each with 0.5 min of soaking.

3. RESULTS AND DISCUSSION

Figure 2 shows a typical DSC result and the main glass transition temperature (T_g) of the jacket materials at 23.6°C. The T_g of similar jacketed materials from a different fiber lot was previously measured to be 36.9°C by the same DSC method [7]. Gebizlioglu also reported that the T_g of the jacketed materials measured by the DMTA method varied between -10 and + 70°C, depending on the fiber manufacturer [5, 6]. On the other hand, the starting temperature for thermal decomposition of jacket materials was measured to be 274 and 390°C from the TG-DTA results of Fig. 3. The observed two-step decomposition was considered to be consistent with the double-jacketing structure shown in Fig. 1.

Next the thermomechanical performance of jacketed fiber samples whose jacket materials had the above characteristic temperatures were examined by DMA. The measurements were made several times on different fiber pieces cut from the same manufacturing lot. Figure 4 shows an example of the DMA result measured from – 50 to 300°C, in which E' and E'' denote a storage modulus and a loss modulus, respectively. In a rough expression, E' indicates elasticity while E'' indicates viscosity of the material. The ratio E''/E' gives a mechanical loss tangent (tan) in the graph. The peak temperature appearing in the tan 8 curve commonly reveals a characteristic temperature of the sample, such as T_g [5, 6]. The peak detected below 0°C was considered to be the T_g of one of the jacketing materials, which could not be detected by the DSC method. Judging by the large decrease in E'',



FIG. 2. Result of DSC measurement of the jacketed fiber.



FIG. 3. Result of TG measurement of the jacketed fiber. The jacket material was burned out completely at 450°C.

the jacket materials soften above 120°C. However, E'' was found to increase again beyond 200°C, suggesting a recovery of hardness. Such rehardening was thought to be caused by thermal decomposition of jacket materials (see Fig. 3).

Another specific feature of Fig. 4 is a slipping of the data at 75°C. Similar DMA measurements were carried out several times using different samples; this specific temperature was found to depend on the sample. In order to check the effect of sample cramping on measurement results (e.g., mechanical slipping), measurement conditions such as tensile strain were also changed in the tests. As a result, I guess that the observed slip was caused by the sample itself rather than by a measurement scheme. As a possible origin of the slip of E values, delamination of jacket



FIG. 4. Result of DMA measurement of the jacketed fiber from -50 to 300°C.

materials due to application of cyclic stresses was considered, because the slip was observed at elevated temperatures at which the jackets soften.

Figure 5 exhibits other DMA results from a different sample, in which the measurement was iterated three times on the same sample between –50 and 150°C. Because the heating was stopped at 150°C, sufficiently lower than the thermal decomposition temperature of the jackets, the jackets showed their intrinsic colorless transparency even after the measurement, while the sample of Fig. 4, which had been heated up to 300°C, changed to an amber color. As seen in Fig. 5, data slipping was observed only in the first heating. The additional two temperature cycles did not cause any anomaly in the measurement results. The observed phenomenon might reveal a generation of irreversible change in jacket materials after the heating process.

In order to check the mechanical change possibly induced in the jackets by the test, the sample of Fig. 5 was observed by the naked eye and with an optical microscope. After the DMA test, sections showing a different surface reflection (seen to be whitish) were found along the fiber, while the virgin fiber was colorless transparent. A similar anomaly in surface reflection was sometimes observed in fibers assembled in the devices after burn-in. Figure 6 presents optical micrographs of the region exhibiting the reflection anomaly, in which images from two different angles were shown. The observed surface reflection was considered to be caused by a void-like defect in the jacket, as appears in Fig. 6. Because of the size, this void-like defect was found to exist between the primary and secondary jacket materials, suggesting delamination at the jacket interface. This result is different from the generally reported expectation that the strength of bonding between the glass fiber and the primary jacket is weaker than that of bonding between the primary and secondary jackets [3, 8]. In my experiments, although debonding of the primary jacket from the glass fiber was not confirmed by optical microscopy, the delamination observed in the jackets was considered to be consistent with DMA



FIG. 5. Other results of DMA measurement of the jacketed fiber. The temperature was cycled three times between –50 and 150°C.

EFFECT OF HEATING ON OPTICAL FIBERS



FIG. 6. Optical micrograph of the jacketed fiber after DMA measurement between –50 and 150°C. A void-like defect in the jackets was not observed before the DMA measurement.

measurements. Because the interjacket bonding was strong [3], the process of separation appeared as an increase of the viscosity, i.e., the loss modulus E'', of the sample, and then after complete separation the E'' value returned to the intrinsic profile. Further, due to a possible distribution of bonding strength along the fiber, the increase in E'' was considered to be abrupt as shown in Fig. 4 or gradual as in Fig. 5.

Concerning the failure in the jackets, Wissuchek *et al.* reported that an intentional indentation (load application) on the jacket surface caused crack growth in the jacket materials and debonding at the primary/glass interface [4]. However, the present test situation was different from their experiments, and the cyclic force was applied along the fiber at elevated temperatures. In assembly and inspection processes of optical devices, mishandling of fibers may induce unforseen force application normal to the fiber and cause cracks in the jackets and debonding at the primary/glass interface. The cyclic force along the fiber is considered to be applied during temperature cycling processes such as a screening test of devices, via a difference in thermal expansion of materials. In this regard, it is possible that quality assurance tests of devices generate anomalies in the fiber jackets.

4. CONCLUSION

The application of cyclic force along optical fibers at elevated temperatures was found to cause separation of the secondary jacket from the primary jacket. This indicated that a similar phenomenon might occur in fibers installed in devices during temperature cycling processes. However, because the separation occurred at the interface of jacket materials rather than at the glass surface, the glass fibers were considered to be intact and to have retained their intrinsic mechanical reliability.

HIROTOSHI NAGATA

ACKNOWLEDGMENT

I thank Dr. Yamaguchi of UBE Scientific Analysis Laboratory Co. for help with the measurements.

REFERENCES

- H. Nagata, N. Mitsugi, J. Ichikawa, and J. Minowa, "Materials reliability for high-speed lithium niobate modulators," SPIE, vol. 3006, 301 (1997).
- [2] R. S. Moyer, R. Grencavich, R. W. Smith, and W. J. Minford, "Design and qualification of hermetically packaged lithium niobate optical modulator," in *Proceedings*, 1997 Electronic Component and Technology Conference, May 18-21, 1999, San Jose, pp. 425-429.
- [3] B. Overton, G. Camilo, M. Purvis, and R. Greer, "Interface integrity-How material interface can affect the performance of dual coated optical fibers," in *Proceedings*, NFOEC'99, September 26-30, 1999, Chicago (CD-ROM Proc.).
- [4] D. J. Wissuchek, D. J. Walter, D. A. Clark, and G. S. Glaesemann, "Fracture and abrasion resistance tests for optical fiber coatings," *Mat. Res. Soc. Symp. Proc.*, vol. 531, 309 (1998).
- [5] O. S. Gebizlioglu and I. M. Plitz, "In-situ characterization of fiber coatings for faialure analysis and reliability investigation," in *Proceedings*, NFOEC'96, vol. 2, 113 (1996).
- [6] O. S. Gebizlioglu and C. R. Kurkjian, "Optical fiber strength and its relationship to dynamic mechanica] properties of fiber coatings by direct measurements of fibers," *Mat. Res. Soc. Symp. Proc.*, vol. 531, 301 (1998).
- [7] H. Nagata, "Thermal analysis of jacketing materials for commercial optical fibers," Opt. Fiber Technol., vol. 3, 87 (1997).
- [8] Private communications from fiber manufacturers.