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Thermal Analysis of Jacketing Materials for Commercial Optical Fibers

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Environment ally induced change in the glass transition temperature (T_g) of fiber jacketing materials was investigated using a differential scanning calorimeter. Three kinds of commercial optical fibers jacketed with UV-cured acrylate, hytrel, and nylon were tested, and the UV-cured acrylate was believed to be the most suitable for the jacketing material because of its significantly stable T_g against the temperature changes. The Tg of the other jackets increased with heating beyond 40°C, suggesting that their structures changed thermally even under ordinary temperatures for the burn-in test of optical devices.

1. INTRODUCTION

Concerning jacketing materials for optical transmission fibers, various kinds of organic polymers have been developed and applied to optical communication systems [1]. For instance, ultra-violet (UV) cured acrylate, hytrel (a kind of polyester), and nylon materials are commercially applied to tightly jacket the glass fibers to provide high resistance to mechanical and chemical attacks. Further, because active devices, such as lasers and modulators, must be assured resistance over a wide temperature range between -40 and 70°C, the UV-cured acrylate and the hytrel promise to become major jacketing materials. Previous reports on the reliability issue for optical devices supported the fact that such jacketed fibers are usable enough to provide highly reliable devices [2–4].

However, characteristics of the jacketing materials are not completely known, although some requirements were standardized, such as their elastic parameters, thermal shrinkage, and adhesive strength between jacket and fiber [5]. The optical devices need to be burned-in before shipping to screen out failed products, and the influence of the burn-in test on jacketing materials demands to be investigated for predicting further device reliability. Actually, when devices with the hytrel jacketed fibers were burned-in in an oven at 80°C, the shape of the fiber placed in the oven became fixed as a result of the burn-in test. In contrast, the fiber jacketed with the UV-cured acrylate did not show such a phenomenon. These empirical results suggest that the material structure of the hytrel jacket is deformed easily by heating, compared with the UV-cured acrylate. Here in this regard, the stability of the commercial fiber jackets in a glass transition temperature (T_g) was investigated by differential scanning calorimetry (DSC). Within an ordinary temperature range for optical devices, the UV-cured acrylate was found to have a more stable structure against the temperature changes.

2. EXPERIMENTS AND RESULTS

The T_g values were measured by a DSC for three kinds of commercial fibers jacketed by UV-cured acrylate, hytrel and nylon before and after exposing them under environmental tests at: (1) 40°C for 100 h, (2) 80°C for 100 h, (3) 120°C for 100 h, (4) 40°C/95% RH for 56 days, (5) 80°C for 1000 h, (6) 20 heat-cycles between -20 and 70°C (~2 K/min ramp rate and ~15 min soaking), and (7) 500 cycles between -40 and 70°C (~18 K/min ramp rate and ~25 min soaking). A hytrel loose tube was also tested, because it is generally used to cover the UV-cured acrylate-jacketed fiber to reinforce the mechanical resistance. The DSC measurements were performed from -50 to 300°C with a heating rate of 20 K/min under a flowing N_2 atmosphere (40 ml/min). The weight of the samples was 10-15 mg including the glass fiber and the primary coating materials. The repeatability of the measurements was estimated to within 2-3 K, judging from the results of double measurements for each unannealed sample.

Figure 1 shows the DSC curves for the UV-cured acrylate jackets before and after the 100-h annealing at 40, 80, and 120°C. The measured T_g was almost constant around 35°C independent of the annealing temperatures. After the other environmental tests, the similar T_g values were also confirmed as listed in Table 1.

In contrast, T_g values of other materials were strongly influenced by the environmental tests, and the second T_g point appeared to depend on the environmental test conditions (see Table 1). Figure 2 shows the typical results observed for the hytrel jacketed fibers, in which the second T_g point (shown by a black mark)

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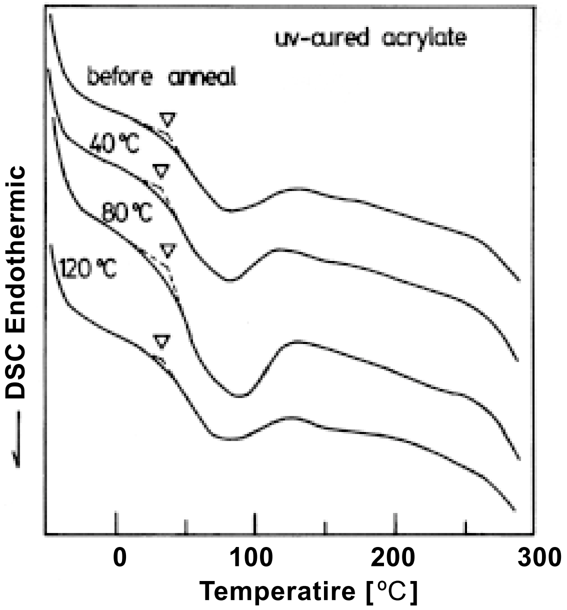

FIG.1. DSC profiles for fibers jacketed with the UV-cured acrylate before and after the 100-h annealing at 40, 80, and 120°C. Marked point corresponds to T_g.

appeared after the 100-h annealing at 40°C and increased with the annealing temperature. Two endothermic peaks in Fig. 2 correspond to the decomposition temperatures of the materials. The peak at ~ 227°C might be due to the hytrel because the hytrel loose tube showed only one peak at ~ 203°C, and the other peak at ~ 160°C was possibly due to the primary coating material of the fiber sample. Judging from the decomposition temperature (T_m) of the samples, the hytrel of the present fiber samples was thought to be prepared from a polyester-polyester system (T_m = 198-223°C) rather than a polyester-polyether system (T_m = 170-218°C). The thermal resistance of the material is known to be higher for the former system. Similarly, the nylon jacket (measured T_m = 178-179°C) was thought to be ìnylon 12î (T_m = 177°C). These decomposition temperatures of the samples were not influenced by any environmental tests, although the T_g changed greatly.

Table 1 further indicates that in the hytrel and nylon jackets, the T_g values were affected strongly by the annealing time (see results of (2) 80°C for 100 h and (5) 80°C for 1000 h in the table). The influence of humidity was not clear (see (1) and (4) in the table). In the case of heat cycles, there was a possibility that the heating (cooling) rate affected the degree of change in the T_g Value (see (6) and (7)). Here, the slow heating rate seemed to enhance the Tg change.

3. DISCUSSION AND CONCLUSION

The T_g point of organic polymers changed due to the chemical and/or crystal structure of the materials. Generally, polyester such as ìhytrelî and nylon are crystallized partially (not completely !) depending on the fabrication process. Therefore, their structure transforms inevitably toward a more stable state, resulting in the change of the T_g value and the mechanical deformation. Further, because the intrinsic T_g of the materials was less than 40°C, the transformation of the materials structure oc-curred rapidly within ordinary burn-in test temperatures and operating temperatures. In the nylon jacket, the transformation caused a large shrinkage of the material along the fiber and a consequent protrusion of the glass fiber from the fiber terminal. This phenomenon lead to a break in the fiber connection at the device, and therefore was a reason for excluding the nylon jacketed fibers from devices [2, 6].

The hytrel jacket seemed not to be accompanied by shrinkage, although the T_g changes were the same as the nylon. The 500 heat cycle test results between -40 and 70°C for optical modulator devices with the hytrel jacketed fibers also showed no fiber break and no deterioration in the optical characteristics. A minor problem in the hytrel jacket was that during the

TABLE 1

T_g of the Jacketing Materials before and after the Environmental Tests

Test conditions	UV-cured acrylate [℃]	Hytrel [℃]		Hytrel (loose-tube) [℃]		Nylon [℃]	
Before test	36.9	46.4		47.8		38.1	
1) 40℃,100h	31.7	37.9	59.4	34.2	58.6	34.8	58.0
2) 80℃,100h	35.9	36.8	77.5	33.3	77.4	31.9	68.8
3) 120℃,100h	32.0	38.8	130.6	34.2	127.7	35.3	126.6
4) 40℃,95%RH 56 days	33.9	35.5	62.9	34.4	61.6	33.3	63.5
5) 80℃,100h	33.6	37.0	94.6	35.7	95.0	35.1	88.5
6) –20/70℃, slow 20 cycles	35.1	38.0	92.7	33.4	92.9	33.0	89.0
7) –40/70℃, fast 500 cycles	34.7	36.6	83.5	33.9	82.8	35.5	78.3

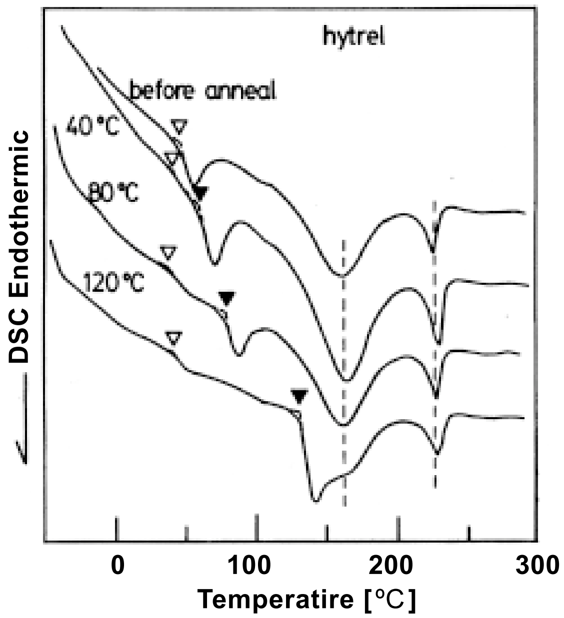

FIG. 2. DSC profiles for fibers jacketed with the hytrel before and after the 100-h annealing at 40, 80, and 120°C. Marked point corresponds to T_g.

burn-in test of the devices, for instance, a curved (angled) shape of the fiber in the oven was fixed due to a structural transformation (T_g change) in the jacket material. Such angled fiber could be straightened by heating it higher than the burn-in test temperature, as predicted from the T_g change in Fig. 2: it is noted here that the lower decomposition temperature of the jacket materials is only 160°C. A preannealing of the jacketed fibers before assembly at temperatures higher than the test temperature (~80°C) was also expected to effectively prevent the problem.

The reason for the observed stable T_g in the UV-cured acrylate jacket was that the structure of this material was probably amorphous-like through a wide temperature range. The decomposition temperature was measured to be ~ 325°C, suggesting that the chemical structure of the material was stable enough, too. As a result, among the tested commercial fiber jackets, the UV-cured acrylate was believed to be the most stable material from the view point of the absence of the chemical and mechanical transformations within ordinary temperatures.

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