

NOTE

Determination of Adhesion of UV-Curable Protective Coatings of Optical Fibers

Adhesion of UV-curable polymer protective coatings of optical fibers to the fused silica surface was investigated. The method of adhesion determination of UV-cured polymers to cylindrical objects is presented.

INTRODUCTION

Most optical fibers are produced from high-silica glasses or fused silica and their diameter is on the order of 0.1–1 mm.¹ Such a fiber, placed in an atmosphere containing water vapor, is subject to water corrosion and its mechanical strength decreases dramatically. Therefore, from a technical point of view, unprotected optical fiber is not capable of performing its task because it is not possible to incorporate it into the energy transmission network.

The optical fiber resistance to the influence of the environment may be considerably improved if the fiber is covered by a protective polymer layer immediately after forming. The polymer layer protects the fiber primarily from abrasion that causes defects and flaws on its surface. Small defects may be a source of greater cracks that lead to the destruction of optical fiber during its exploitation.^{2–4} The influence of the polymer protective layer on the mechanical strength of optical fibers was widely studied^{2,4–6} but the mechanism of stress corrosion as well as the dependence of mechanical strength of the fused silica fibers upon the properties of protective coatings are not clear. Certainly both strength and time-to-failure of optical fibers are good if the polymer protective coating adheres well to the glass fiber surface and the diffusion of water vapor through this polymer is small.⁴

The influence of water vapor diffusion through the polymer protective layers on the mechanical properties of optical fibers was widely investigated. There are also many methods of quantitative determination of the water diffusion coefficient.^{3,7} Unfortunately, the determination of adhesion of UV-cured polymers to the fused silica optical fibers is more troublesome. First, the shape and dimensions of optical fiber make the direct measurement of adhesion on the fiber rather impossible. Second, the majority of the methods applied in determination of adhesion of varnish coatings (cross-cut test, scratch bundless knife, etc.^{8,9}) may

be used if the investigated surface is flat. The application of other methods like direct pulloff⁸ or torque spanner⁸ is very difficult in the case of UV-cured polymers because they require free access of UV radiation to liquid composition placed between two solid specimens.

It seems also that the mechanical strength of optical fiber is decided not only by “thermodynamic adhesion” interpreted as equilibrium interfacial forces, but also by energies associated with the reversible process such as ideal adhesive strength, work of adhesion, etc.¹⁰ The optical fiber should be treated as the composite material composed of a hard core (glass or fused silica) and a polymer protective coating. The viscoelastic and rheological properties of both, glass and organic polymer, materials differs greatly. In such a case the most important factor determining the mechanical strength of an optical fiber will be the energy of adherence of a polymer layer to the fused silica fiber. The value of this energy is determined by such factors as viscoelastic properties of the polymer, shrinkage of the polymer taking place during the curing process and, of course, adhesion in its exact thermodynamic sense. Thus, the term “adhesion” used here should be understood in this broader sense. We suppose that determination of adhesion in such a sense may be of a greater importance for designers and users of optical fibers than thermodynamic adhesion.

Investigations in the field of the protective coatings of optical fibers, carried out in our laboratory, led us to develop a method of adhesion determination of UV-curable polymers on cylindrical objects.

EXPERIMENTAL

Materials

As the fused silica specimens (Carl Zeiss Jena, Germany), 3-mm diameter rods, freshly drawn in the optical fiber oven, were used.

The proposed method of adhesion determination was tested with the four polymer formulations applied in our laboratory as the protective coatings of optical fibers:

1. epoxyacrylate formulation¹¹;
2. urethaneacrylate formulation¹²;

3. formulation composed of epoxy- and urethaneacrylate mixed in the weight ratio 4 : 1;
4. formulation delivered by DeSolite (DSM Resins International BV, The Netherlands) as 950-0.75 lacquer.

Formulations B and D are usually used as the primary coatings of optical fibers; formulation A as the secondary coating; and formulation C, the properties of which (hardness and relative elongation) are intermediate between those of A (very hard) and B (soft), is used as a single coating on optical fibers.

Methods and Apparatus

The specimens for adhesion measurements were prepared as follows. The fused silica rods (3-mm diameter) were placed vertically in a polytetrafluoroethylene (PTFE) matrix (Fig. 1) and the cavity around the rod was filled with a liquid formulation. Next, the formulation was cured with UV radiation, using a 400-W mercury lamp, always maintaining the same distance between the lamp and the specimen, equal to 30 cm. The irradiation process was carried out in an argon atmosphere. The optimum time of curing was found for each formulation in the separate experiments.

After curing, the fused silica rod with a polymer ring adjacent to it was taken out of the PTFE matrix and the thickness (about 1.2 mm) of the polymer ring was measured. Next one end of the fused silica rod was placed in the stationary grip of a tensile tester TIRATEST 2200 (Berlin, Germany). The moving grip of the tester was connected by the elastic steel cord with the cone-shape ring (Fig. 2). This ring tears off the polymer ring from the surface of the fused silica rod. All measurements were performed at a speed of the moving grip of the tester equal to $1 \text{ mm} \cdot \text{min}^{-1}$. It was assumed that adhesion of the in-

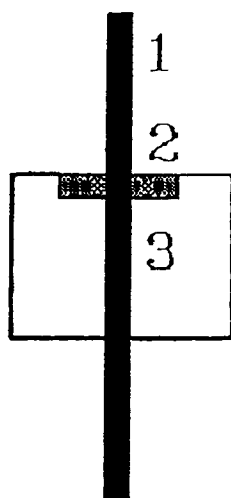


Figure 1 Cross section of PTFE matrix used for sample preparation. (1) fused silica rod; (2) polymer ring; (3) PTFE.

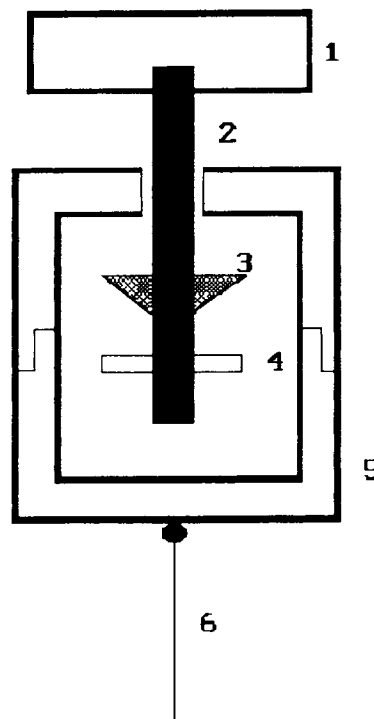


Figure 2 Schema of the set-up for adhesion measurements. (1) stationary grip of testing machine; (2) fused silica rod; (3) cone-shaped ring; (4) polymer ring, thickness about 1.2 mm and diameter 10 mm; (5) steel ring, connected trough and elastic cord; (6) with a moving grip of the testing machine.

vestigated polymer to the fused silica surface was equal to the largest strain force registered by the tester, divided by the surface of the polymer ring adjacent to the fused silica rod.

RESULTS AND DISCUSSION

The results of the measurements testing the proposed method of adhesion determination are shown in Table I. These results indicate the clear dependence of adhesion on the chemical nature of the investigated polymers. The highest values of adhesion were determined in the case of the epoxyacrylate formulation and the formulation containing epoxyacrylic oligomers (formulations A and C). The urethaneacrylate coating (B) shows the lowest adhesion to the fused silica surface.

The obtained results show also that the method of adhesion determination of UV-cured polymer coatings to the cylindrical objects has been attained. This is evidenced by good reproducibility of the results in a rather wide range of adhesion energy. The differences in the results obtained for individual polymers are probably caused by fused silica surface contamination by water and organic substances present in the laboratory atmosphere.

Table I Adhesion ($N \cdot mm^{-2}$) of UV-Cured Protective Coatings of Optical Fibers to Fused Silica Rod Surface

Measurement Number	Formulation			
	Epoxyacrylate	Urethaneacrylate	80% Epoxy-20% Urethaneacrylate	950-0.75 DeSolite
1	13.74	1.10	11.93	1.87
2	11.74	1.08	11.97	2.03
3	12.41	1.09	11.97	1.87
4	11.11	1.14	10.29	2.08
5	10.97	1.18	11.41	2.05
6	—	1.05	10.77	1.81
7	—	—	12.30	—
Average	11.99	1.11	11.63	1.95
SD, δ	1.01	0.04	0.81	0.06

Further investigation on the influence of fused silica hydroxylation on the energy of polymer coatings adhesion will be carried out.

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References

- W. A. Gambling, in *Principles of Modern Optical Systems*, I. Andonovic and D. Uttamchadani, Ed., Artech House Inc., Norwood, MA, 1989, p. 129.
- F. V. DiMarcello, A. C. Hart Jr, J. C. Williams, and C. R. Kurkijan, in *Physics of Fiber Optics. Advances in Research and Development*, B. Bendow and S. S. Mitra, Ed., Plenum, New York, 1979, pp. 125–135.
- H. N. Vazirani, H. Schornhorn, and H. M. Zupko, *J. Appl. Polym. Sci.*, **23**, 887 (1979).
- Ta-Sheng Wei, *Adv. Ceramic Mater.*, **1**, 237 (1986).
- F. V. DiMarcello, D. L. Branlow, and D. S. Shenk, *Tech. Dig. Int. Conf. IOOC*, **26–27**, OSA (1981).
- T. T. Wang and H. M. Zupko, *J. Mater. Sci.*, **13**, 2241 (1978).
- J. Rayss, W. Podkościelny, and A. Walewski, *Proceedings of the SPIE 1085*, 1989, p. 288.
- L. J. Calbo, Ed., *Handbook of Coating Additives*, Marcel Dekker, Inc., New York and Basel, 1986, p. 285.
- ISO 2409-(1972(E)); NFT 30-038-(1991); NFT X41-022 (1984).
- S. Wu, *Polymer Interface and Adhesion*, Marcel Dekker Inc., New York and Basel, 1982, p. 337.
- J. Rayss, W. M. Podkościelny, and J. Widomski, *J. Appl. Polym. Sci.*, **49**, 835 (1993).
- W. Podkościelny, B. Tarasiuk, and W. M. Podkościelny, *Polimery*, **38**, 432 (1993).

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