New Methods for Evaluating Optical Fiber Coating Materials

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ABSTRACT UV-curable resins are used as the coating material for optical fibers, and their physical properties change with the degree of curing, thereby influencing the transmission characteristics of the fiber. In order to maintain stable quality of the finished product it is necessary to evaluate the degree of curing of the coating resin while it is on the fiber. The authors have measured the logarithmic damping ratio for optical fibers double-coated with UV resin using a rigid-body pendulum-type viscoelasticity testing apparatus, and, from the results, have clarified the relationship between the dose of UV irradiation and the degree of curing of the coating resin on the optical fiber.

It is also necessary, as a means of improving the efficiency of manufacturing optical fibers, to develop a rapidly curable UV coating material, but since the reaction proceeds in such a short time, it has been difficult to establish an effective method of evaluating the degree of UV curing. In this work it has been possible, using a rheometer incorporating a fast oscillation mode program, to evaluate changes over time in the modulus of elasticity of UV-curable resins under irradiation.

1. INTRODUCTION

Optical fibers are crucial in the field of telecommunications, offering the advantages of low loss. broad bandwidth, fine diameter, light weight, freedom from electromagnetic induction, freedom from crosstalk, and efficiency in resource consumption. In comparison to the copper conductors used in conventional telecommunications cables, however, they are at a disadvantage in terms of their low mechanical strength, which makes them brittle. From the processes of fiber drawing, coating and cabling to the laying of the cables, they are subjected to tensile force, and subsequently, under a variety of environments, they must have superior static and dynamic fatigue characteristics. For this reason optical fibers, immediately after drawing, are covered by a coating of resin to improve their mechanical strength. This paper describes methods for evaluating the degree of curing of optical fiber coating materials, and then introduces a method for evaluating the degree of resin curing using a rigid-body pendulum-type viscoelasticity testing apparatus.

2. EVALUATING DEGREE OF CURING OF COATING MATERIALS

Optical fibers are so designed that their transmission characteristics will not change despite fluctuations in the temperature and humidity of their environment or the passage of time, and in this the material used as the coating plays an important role. The most generally used fibers are silica-based, and they have superior transmission characteristics, but silica glass is easily fractured by external forces. The fibers are therefore covered with a coating of resin which is usually of a 2-layered structure. The primary layer--the layer directly in contact with the glass--has the purpose of dispersing external force, and is made of a resin having a low modulus of elasticity. It must also adhere closely and form a stable interface. Outside this primary layer is the secondary layer, which, to protect it and compensate for its mechanical strength, is made of a resin having a comparatively large modulus of elasticity (see Figure 1). Also, in multifiber cables, there may be a pigmented layer on the outside for color coding.

In recent years, to improve manufacturing efficiency, the trend has been to use UV resins that cure in an extremely short time. It has therefore become extremely important, from the standpoints of assuring the quality of the finished product and achieving stable production, to obtain an understanding of the rheological properties of the resin--coating characteristics, curing behavior, curing conditions, and so on (see Figure 2).

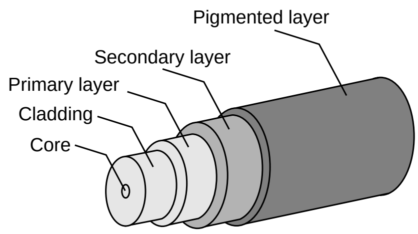

Figure 1 Structure of coated optical fiber.

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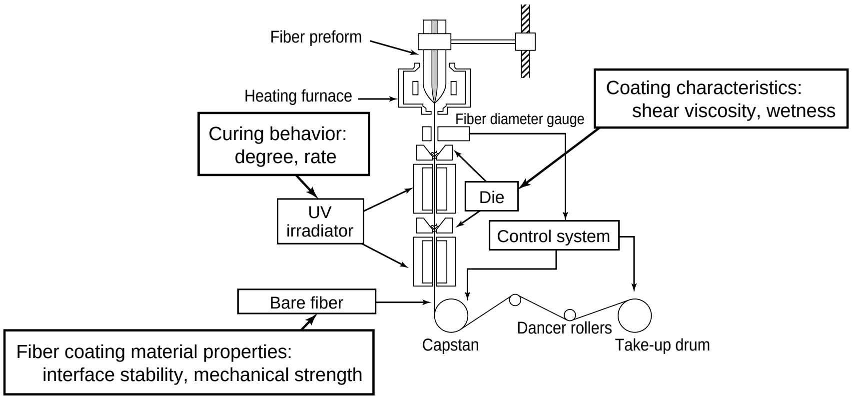

Figure 2 Process model of optical fiber drawing/coating apparatus and required rheological properties.

Table 1 Methods of evaluating degree of curing for optical fiber coatings.

Mechanical methods	Spectroscopic methods	Thermoanalytical methods	Qualitative / quantitative analysis
Dynamic viscoelasticity TMA Push-in modulus Dynamic cure meter Rigid-body pendulum-type viscoelasticity testing apparatus	ATR-FTIR Microscopic FTIR Time-resolved FTIR	Photo DSC Photochemical reaction calorimetry	GC-MS Gel fraction measurement

Methods of evaluating the degree of curing may be divided broadly into spectroscopic methods, thermoanalytical methods, mechanical methods and qualitative/quantitative analysis. Table 1 lists the most important methods. The degree of curing of a UV-curable resin varies depending on the intensity of irradiation, the dose of irradiating energy, the temperature during curing, and the curing atmosphere. It is therefore commonly seen that evaluations differ when based on a bench test of the resin alone or on the cured coating of a finished product. The authors have given particular emphasis to evaluating the degree of curing of resin as a finished product--when it forms the coating on a silica-glass fiber.

3. EVALUATING DEGREE OF CURING OF COATING MATERIAL

Using an A&D Model RPT-3000 rigid-body pendulum type viscoelasticity testing apparatus (Figure 3), the authors measured the logarithmic damping ratio Δ of the UV-curable coating resin on an optical fiber and the period T of the pendulum, end evaluated the degree of curing. The fulcrum of the pendulum was placed on the surface of the sample and the pendulum was made to oscillate, and by analyzing the mechanism of the damping of the amplitude of oscillation and the period of oscillation it is possible to evaluate the degree of curing and other mechanical properties of the sample. At the temperature at which the sample undergoes a phase change from a vitreous state to become a rubbery elastic body, the loss in pendulum motion increases and its amplitude is rapidly damped. The waveform of the pendulum damping curve is modeled in Figure 4. The logarithmic damping ratio Δ can be calculated by Equation (1)

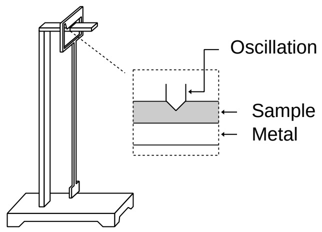

Figure 3 Rigid-body pendulum type viscoelasticity testing apparatus.

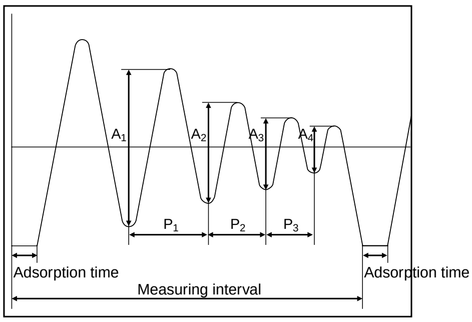

Figure 4 Model of pendulum damping curve.

$$\Delta = \{\ln(A_1/A_2)+\ln(A_2/A_3)+\cdots\cdots+\ln(A_n/A_{n+1})\}/n \qquad (1)$$

and oscillation period T (in seconds) by

$$T = (P_1+P_2+\cdots\cdots+P_n)/n \qquad (2)$$

where: n is the number of oscillations

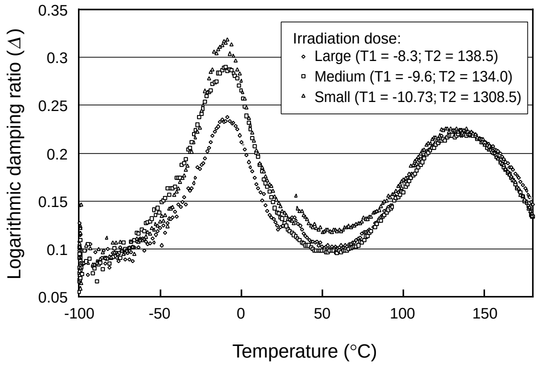

Figure 5 Temperature dependence of logarithmic damping ratio Δ of a prototype UV-resin coated optical fiber.

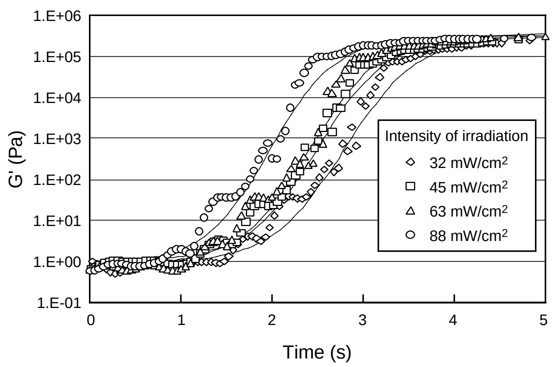

Figure 6 Change in modulus of elasticity G' under UV irradiation for UV-curable resin.

Figure 5 shows the temperature dependence of the logarithmic damping ratio Δ of a prototype UV-resin coated optical fiber. The peaks corresponding to the glass transformation temperature T_g of the materials of the primary coating and secondary coating were observed in the vicinity of -10°C and 130°C respectively. Correlations between these peaks and the glass transformation temperatures measured using other viscoelasticity testing equipment have been confirmed. When the dose of UV irradiation was increased the peak temperatures using the present evaluation method shifted higher. The shape of the peaks also became broader, indicating that it is the peaks in logarithmic damping ratio Δ that shows the condition of curing. Since the present method makes it possible for the condition of curing of the materials of both the primary and secondary coatings on the fiber to be evaluated directly, it promises to be applicable to on-site quality control during manufacture.

4. EVALUATING CURING BEHAVIOR OF RESINS UNDER UV IRRADIATION

Understanding the behavior that takes place in the process of UV curing is of major concern with respect to optimizing the conditions under which optical fibers are manufactured and improving product reliability. Changes in the modulus of elasticity were measured during the course of the resin curing process using Rheologica's DAR-100 rheometer, which incorporates a fast oscillation mode program enabling evaluation to be accomplished with extremely short sampling times. It can therefore be applied to the behavior of optical fiber coating materials that exhibit very quick UV-curing.

This program samples each sinusoidal wave cycle during dynamic measurement of viscoelasticity at 512 points, enabling the calculation/detection of the torque at each of these points. Thus if dynamic viscoelasticity is measured at a frequency of 1 Hz, data can be obtained for 512 points per second. Figure 6 shows changes in the stored modulus of elasticity G' during the curing of coating resin under constant UV irradiation. It was observed that in a period of approximately 1 second, UV irradiation produced an increase in G' of some five orders of magnitude--from 1 Pa to 10^5 Pa. And by measuring the change over time in the modulus of elasticity while changing the intensity of irradiation, it was also possible to confirm that an increase in irradiation intensity produced faster curing of the resin. These results mean that this system clarified the changes in G' during the curing process, and has proved to be highly effective for evaluating coating resins that are cured very quickly under UV irradiation.

5. CONCLUSION

This paper has touched briefly on methods used to evaluate the degree of curing of optical fiber coating materials. It then describes an evaluation method advanced by the authors that uses a rigid-body pendulum-type apparatus for testing viscoelasticity and presents the results of the use of a stress-control rheometer in evaluating the curing behavior of a fiber-optic coating material cured under UV irradiation.

It is believed that evaluating viscoelasticity with respect to optical fiber coating materials will contribute significantly to developing new products and maintaining product reliability.