IS-5157

AMES LABORATORY

On-Line Sensor Systems for Monitoring the Cure of Coatings on Glass Optical Fibers and Assemblies

Final Report
April 1, 1999, to September 30, 2000

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PREPARED FOR THE UNITED STATES DEPARTMENT OF ENERGY
OFFICE OF ENERGY EFFICIENCY AND RENEWABLE ENERGY

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Executive Summary

Glass fiber technology, which enables light transmission and communication over great distances, was developed in the U.S. and has evolved into a major industry in this country. Continuous innovation is required to maintain the leadership position that the U.S. enjoys in glass optical fiber technology. This project addressed a key quality assurance aspect of glass optical fiber and cable assembly production—namely, assuring full cure of the polymer coating that protects the fiber from physical damage and moisture degradation. Properly cured coatings are imperative for the cable-assembly industry because they provide protection from the environment and assure both mechanical strength and long-term performance of the cable assembly. The objective of this project was the development of a sensor for continuous, on-line monitoring of coating cure. Two candidate technologies were examined in the project for application as the sensor, a noncontact spectroscopic approach called transient infrared spectroscopy and a tack-sensing probe, dubbed a tackmeter. Both proved capable of sensing the surface-tack or cure level of fiber-optic cables on line.

Introduction

Glass fiber technology, which enables light transmission and communication over great distances, was developed in the U.S. and has evolved into a major industry in this country. Continuous innovation is required to maintain the leadership position that the U.S. enjoys in glass optical fiber technology. This project addressed a key quality assurance aspect of glass optical fiber and cable assembly production—namely, assuring full cure of the polymer coating that protects the fiber from physical damage and moisture degradation. Properly cured coatings are imperative for the cable-assembly industry because they provide protection from the environment and assure both mechanical strength and long-term performance of the cable assembly. The industry loses approximately 5% of its production due to under-cured fiber. This loss amounts to roughly $70,000,000 per year. The standard procedure in the industry is to spot check cables after production because no on-line method has been available to continuously monitor cure during either the glass-fiber drawing process, which operates at high speeds of roughly 1 kilometer per minute, or the cable-assembly process. Many kilometers of out-of-specification material can therefore be produced before tests catch the flaw. This project strove to eliminate this delay.

The project was a collaboration between Ames Laboratory and Siecor Corporation (which became Corning Cable Systems LLC near the end of the project) under CRADA No. AL-C-99-04. The principal investigator at Ames Laboratory was John McClelland and the technical leader at Siecor was Naren Patel. The objective of this project was the development of a sensor for continuous, on-line monitoring of coating cure. The concept is illustrated in Figure 1. Ultimately, the sensor output could be linked to process control either directly, via automatic feedback control, or by alerting the human operators through a visual display or an alarm. Two candidate technologies were examined in the project for application as the sensor, a noncontact spectroscopic approach called transient infrared spectroscopy and a tack-sensing probe, dubbed a tackmeter.

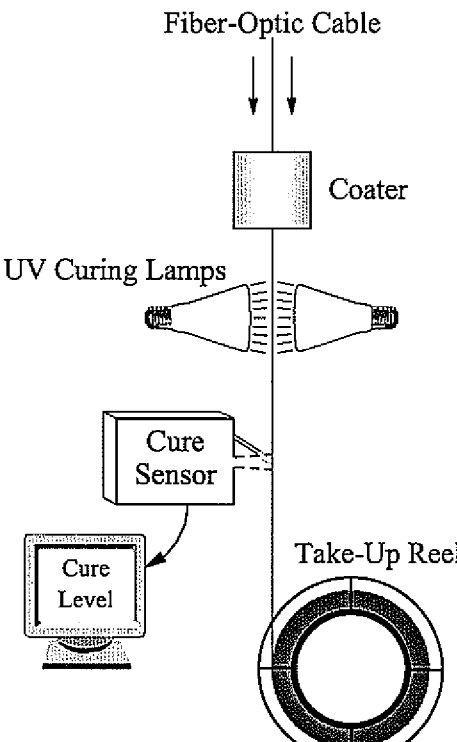

Figure 1. Concept of an on-line cure sensor.

Transient infrared spectroscopy (TIRS) is based on emission spectroscopy. All materials spontaneously emit infrared light by virtue of their temperature. The hotter they are, the stronger they emit. In conventional emission spectroscopy, a sample is warmed until it emits sufficient infrared light, then the emission spectrum is recorded. Thin, warm materials preferentially emit the same wavelengths they preferentially absorb when infrared light is passed through them, so the analysis is identical to conventional transmission spectroscopy, which has been a mainstay in analytical laboratories for decades. Fortunately, materials need be only a few tens of degrees above ambient temperature to emit sufficiently in the infrared. Unfortunately, if the warm sample is too thick, it emits at all wavelengths, producing a featureless, blackbody spectrum. TIRS is innovative in that it avoids this blackbody problem without the sample being physically thinned.

TIRS uses a temperature gradient to isolate spectroscopically a thin surface layer of the moving solid material, thereby circumventing the blackbody problem. Figure 2 shows schematically how TIRS works. The moving process stream passes through the field of view of an infrared spectrometer. As it does so, a small jet of hot air strikes it, warming the surface. This heated surface layer acts as a thin emission source separate from the rest of the process stream. Because it is thin, it produces a structured, analytically useful spectrum, which the spectrometer records. Because the material is moving, the heated layer is carried out of the spectrometer field of view before it can thicken and cool appreciably. The spectrometer therefore observes the infrared emission from a heated layer that seems to stay perpetually thin. The acquired spectra are analogous to conventional spectra, so the same methods and tools developed for laboratory spectroscopy can be applied to TIRS spectra for chemical analysis. The technical aspects of TIRS are described in more detail elsewhere.[1,2] TIRS is effectively a noncontact probe because only a stream of heated air needs to contact the material on the process line. Other applications of TIRS have been documented elsewhere.[2-4] Patents on TIRS are held by the Iowa State University Research Foundation.[5-7]

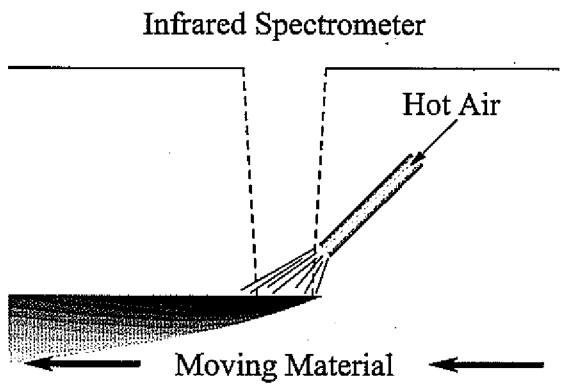

Figure 2. Schematic of the operation of an on-line TIRS sensor.

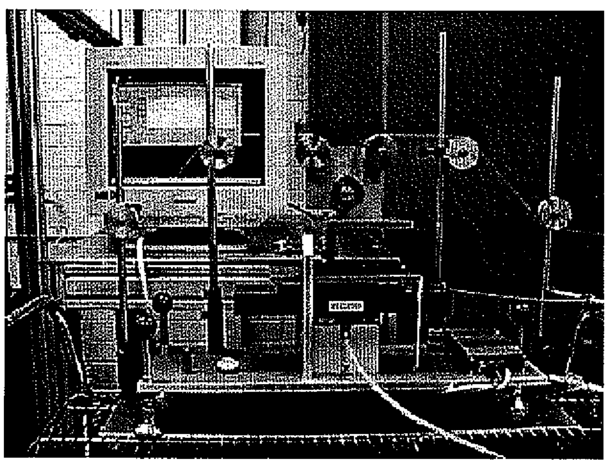

Figure 3. Tackmeter on line at Siecor. The fiber-optic cable passes through the rollers from right to left.

The tackmeter is a direct contact device that determines the amount of tack on the surface of the process line material by sensing the force the material applies to rollers as it passes over them. As implemented in this project, the rollers were mounted on an air-bearing-supported slide on a track. The device is shown Figure 3. The slide was held in place against the pull of the passing cable by either a coiled spring or an air cushion. Whenever the tackiness of the cable material increased, the pull of the cable increased, and the slide moved downstream from its equilibrium position. The distance of the displacement was related to the amount of tack. This approach was developed specifically for this project, so it has not been used in other applications.

The project succeeded in showing that both technologies are capable of sensing the degree of cure/tackiness of the passing cable. The TIRS system had somewhat shorter response time and higher sensitivity, but there were still potential improvements that could have been made to the tackmeter system.

References:

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5. J. F. McClelland and R. W. Jones, "Apparatus and Method for Transient Thermal Infrared Spectrometry of Flowable Enclosed Materials," U. S. Patent 5 191 215, 1993.

6. J. F. McClelland and R. W. Jones, "Apparatus and Method for Transient Thermal Infrared Emission Spectrometry," U. S. Patent 5 075 552, 1991.

7. J. F. McClelland and R. W. Jones, "Apparatus and Method for Transient Thermal Infrared Spectrometry," U. S. Patent 5 070 242, 1991.

Project Activities

The performance period for the project was April 1, 1999, through September 30, 2000.

Project activities centered on three series of on-line tests of both monitor technologies. These on-line tests took place at the Hickory, North Carolina facility of Siecor Corporation (now Corning Cable Systems). These tests took place July 11-14, 1999; December 13-15, 1999; and September 13-15, 2000. A cycle of activities surrounded each series of on-line tests. Before each on-line series, the monitor equipment was prepared for the tests and then shipped to Siecor. During each on-line series of tests, the tackmeter and TIRS spectrometer were mounted on a pilot optical-fiber-cable assembly line and data were recorded while the line was operated under a variety of conditions so as to produce cable having a variety of cure levels in its protective jacket. Ames Laboratory staff operated the monitors while Siecor staff controlled the process line. After each series, Siecor staff provided Ames Laboratory with off-line analyses of the cure level of the cable jacket produced during the tests. These analyses were then used at Ames Laboratory to calibrate the data from both monitors and to determine their performance characteristics. These results were then used to identify needed improvements in the equipment and in the data reduction. The improvements were instituted and the equipment was tested off line (at Ames Laboratory) before the next on-line test series was scheduled.

The TIRS spectrometer is shown in Figure 4 during the first (July) series of on-line tests. After the July tests, a larger hot-air tool was incorporated into the TIRS monitor, and a mirror with a tighter focus was substituted for the one that had been used. These produced and gathered, respectively, a greater amount of infrared radiation from the fiber cable, improving the quality of the spectra observed. Different, repositioned idler wheels were also incorporated to hold the optical-fiber cable more precisely and with less potential to stress the cable. The tackmeter was improved after the July tests by the incorporation of both a laser-based position sensor and a less massive roller mount with lower-resistance roller bearings. These changes were made to increase the sensitivity of the tack monitor, and the position sensor also provided a digital record of the tackmeter monitor measurements, which had been manually recorded during the July tests.

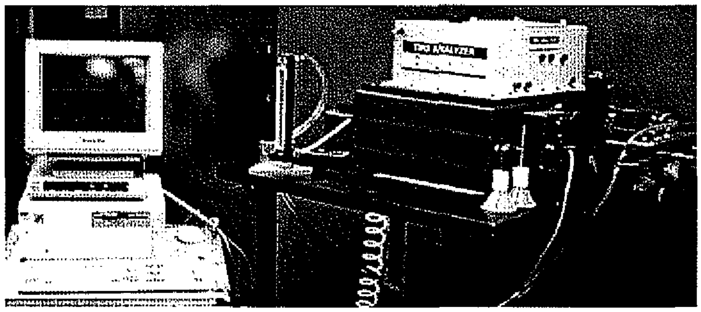

Figure 4. TIRS monitor on line at Siecor.

At the second (December) series of tests, the Siecor staff was not able to produce much variation in cure level, so fewer needed improvements could be identified. The Siecor staff later determined that a change by a supplier in the formulation of the cable-jacket material prevented their manipulation of cure level. Despite the modest outcome of the December tests, the TIRS monitor was modified after the tests by the addition of software that provided a real-time readout of its analysis while on line, and the tackmeter was improved by the addition of a spring-based roller-mount stabilizer to shorten its recovery time. This last version of the tackmeter is pictured in Figure 3 during the final (September) on-line tests.

Results

Both technologies proved capable of monitoring cure/tack level of the fiber-optic cable on line by the end of the project. For determining cure level off line, Siecor used attenuated total reflectance (ATR) spectroscopy. The ratio of the strengths of two bands in the off-line ATR spectrum was a direct measure of coating cure.

The tackmeter data consisted of measurements of how far the roller slide was carried downstream by the force of the moving cable. Figure 5 shows the correlation between data from the tackmeter at the final (September 2000) series of on-line tests and post-test, off-line measurements by ATR. After the elimination of two points, which were suspect because of measurement difficulties, the remaining tackmeter data correlates well with the ATR data.

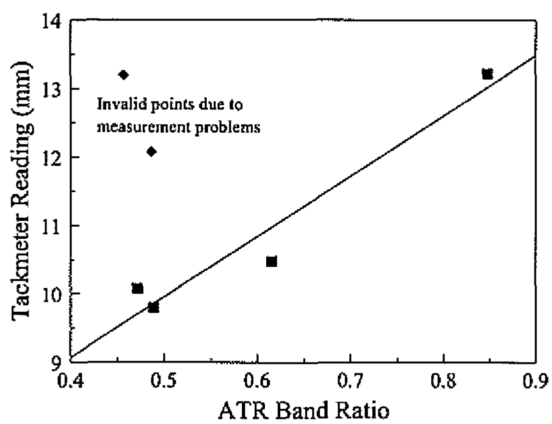

Figure 5. Correlation between on-line tackmeter data and off-line sample analysis.

The root-mean-square error for the linear fit between the tackmeter and ATR measurements is only 0.33 mm (or 0.037 in the band ratio).

The TIRS data were correlated with the off-line ATR data using partial least square (PLS). PLS is a statistical technique that builds a calibration by matching up the variations among a "training set" of spectra with the known differences among the samples that produced the spectra. In this case, the known differences were the variations in the ATR band ratios that represent cure levels. The calibration then allows the instrument to "predict" (i.e., determine) the calibrated property for any future sample from its spectrum. Figure 6 shows a PLS-based cross validation of the on-line TIRS spectra from the September 2000 tests. Again, the correlation is good, with a standard error for the cross validation of 0.040 band-ratio units.

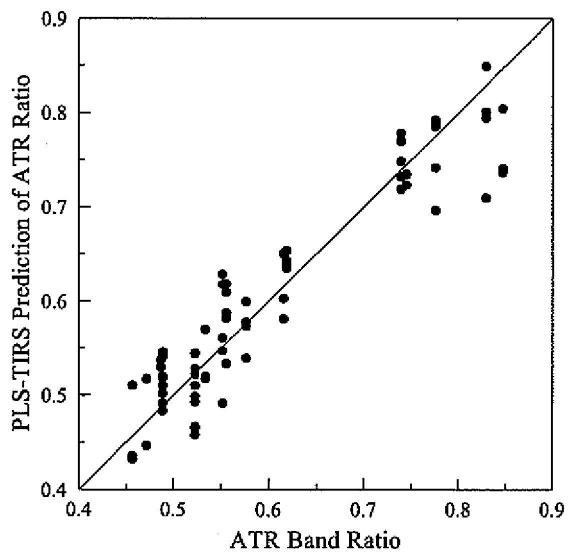

Figure 6. Correlation between on-line TIRS spectra and off-line (ATR) sample analysis.

Siecor Corporation was jointly owned by Corning, Inc., and Siemens AG. Near the end of the project, Corning bought out Siemens share of the company and Siecor became Corning Cable Systems LLC. The management change that accompanied this, combined with supplier-provided improvements in the coating resins during the project, resulted in a substantial reduction in the perceived importance of the cure-level problem by Siecor/Corning Cable management. For this reason, no TIRS or tackmeter unit were permanently installed on a fiber-cable production line.