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Improved Fiber Optics Final Report CRADA No. TSB-957-94

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Improved Fiber Optics

Project Accomplishments Summary
CRADA No. TSB-957-94

Date: August 31, 2000 Revision: 1

A. Parties

The project was a relationship between the Lawrence Livermore National Laboratory (LLNL) and Lumenyte International Corporation (LIC).

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B. Background

The existing chemistry of Lumenyte® (an illumination fiber optic developed by LIC) was such that the component monomers inherently polymerized to a very hard mass if exposed to environmental IR, UV, or a combination of these frequencies. Lumenyte optic also would cure to a hard mass by exposure to the UV & IR generated by the illuminating lamps—although this could occur at a much slower rate, and the hardening could occur even when the adverse frequencies were filtered. The resultant product did not have the flexibility for the required applications.

C. Description

LIC's objective was to include other monomeric components in the formulation to impart permanent flexibility. LIC sought the expertise and the use of the facilities in the Polymeric Materials Section at LLNL to achieve this objective.

The work conducted under this CRADA is described with reference to the Tasks outlined in Appendix A of the Joint Work Statement.

Task 2: LIC provided the processing equipment and processing knowledge necessary to fabricate the Lumenyte ® fiber optic.

Task 1: LLNL reviewed the chemistry and processing steps that LIC used to produce its Lumenyte® fiber optic. Several potential problems were identified as possibly contributing to the lack or the loss of flexibility in the plastic optical fiber (POF). These were:

1. possible impurities introduced during the filtering of CR-39
2. separation between physical components of the fiber, and
3. phase separation within the polymer.

Several methods were selected to evaluate samples provided by LIC. It was established that transmission tests in ultraviolet and visible wavelengths and Fourier transform infrared (FTIR) analysis would be conducted. In addition, scanning electron microscope (SEM) high resolution morphological studies with accompanying energy dispersive spectral (EDS) elemental analysis would be conducted to determine the presence of impurities. There is no record that mechanical tests were required by the evaluation or that they were undertaken.

With respect to the range of chemicals to be considered for new formulations, it was concluded that only methacrylates would be considered.

Task 3: Three production samples were evaluated: EL 500, 300, and 100. The PI for this work was Peter Elliker. The results, summarized below, indicate nothing that would be responsible for the loss of flexibility of interest to LIC.

UV/Vis spectra of EL 500, 300, and 100 indicated little absorbance across the spectrum (300-850 nm). What few differences were observed between specimens were attributed to sample diameter. Both EL 100 and 300 produced a broad absorbance in the red range. In the UV range, EL 100 produced little absorbance; EL 300 slightly greater, and EL 500 exhibited a large absorbance. Characteristics common to all samples include absorbances in the red/near IR and one or two peaks in the 450-500 nm region. Large carbonyl absorbance at 1740 cm^{-1}, ester C-O at 1250 cm^{-1}, and alkyl C-H at 2990 and 1450 cm^{-1} were evident in preliminary FTIR spectra of thin slices of EL 500. The spectrum mirrors that of polymethyl methylacrylate (PMMA). However, a vinyl stretching absorbance at 1650 cm^{-1}, indicated unreacted monomer. Further indication of residual reactants were two bands around 750 and 791 cm^{-1} which are usually indicative of aromatic groups. They may also be due to CF_3/CF_2 groups; in either case, it would not have been expected to see anything in this region. Similar evidence of residual monomer was observed in preliminary spectra of EL 300.

SEM observations suggested little structure in the teflon cladding, which is smooth except for some wavy structure visible at the 10 and 3 _m scales. The corresponding EDS detected mostly fluorine, as expected. The surface of the polymer fiber was also

smooth, even down to the 3 _m scale. EDS analysis of dark spots arranged irregularly on the surface indicated the presence of fluorine suggesting that the spots are probably teflon from the cladding which adhered to the exposed polymer fiber. EDS of the polymer surface shows carbon and oxygen, as expected. No metal impurities were observed by EDS in any sample, at least to a depth of approx. 1 _m which is the sampling depth. However, EDS is not especially sensitive to trace amounts of metals in an organic environment, therefore metals may be present in concentrations below approx. 0.1-0.5 % which is still more than enough to contribute to losses.

Several formulations were tested using the basic preparation method described by LIC but varying the relative proportions of the compounds: methyl methacrylate (MMA), lauryl methacrylate (LMA), stearyl methacrylate (SMA), and CR-39 organic glass. Preliminary results of these trials are provided in Table 1.

Table 1. Results of fiber optic formulation trials.

Trial	Relative amount (by volume) of monomer				Comments
	MMA	LMA	SMA	CR-39	
1	-	0.5	-	0.5	Phase separation
2	-	0.33	-	0.67	Phase separation
3	0.33	0.17	-	0.5	No results
4	0.29	0.14	-	0.57	Phase separation
5	0.5	0.17	-	0.33	Slightly yellow
6	0.42	0.33	-	0.25	Slightly yellow
7a	0.42	0.16	-	0.42	Slightly yellow
7b	0.42	0.16	-	0.42	Phase separation
7c	0.42	0.16	-	0.42	Phase separation
7d	0.42	0.16	-	0.42	Slightly yellow. No phase separation. Possible presence of a crystallite
8	0.5	-	-	0.5	Good. No yellowing. No phase separation.

D. Expected Economic Impact

The proposed project would provide a mechanism for the development of a new fiber optic product line at LIC.

E. Benefits to DOE

This project would benefit DOE/LLNL by enhancing its core competency in advanced materials and leading to new intellectual program.

F. Industry Area

Chemistry

G. Project Status

This project is completed.

H. LLNL Point of Contact for Project Information

University of California
Lawrence Livermore National Laboratory
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I. Company Size and Point(s) of Contact

Lumenyte International Corporation is privately held company with annual sales of less than $10 million. At the time of the CRADA, the company employed less than 50 people.

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J. Project Examples

There are no project examples.

K. Subject Inventions

This small value contractual mechanism did not anticipate any generation of Intellectual Property (IP) including subject inventions. The LLNL contributors and the company participants both indicate that no new intellectual property was generated.

L. Release of Information

I certify that all information contained in this report is accurate and releasable to the best of my knowledge.

Karena McKinley, Director
Industrial Partnerships
and Commercialization

9/29/00
Date

Release of Information

I have reviewed the attached Project Accomplishment Summary prepared by Lawrence Livermore National Laboratory and agree that the information about our CRADA may be released for external distribution.

Sandy Wilford, Corporate Secretary
Lumenyte International Corporation

9/22/00
Date