

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 20, 1997	3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97	
4. TITLE AND SUBTITLE Photophysics of Polycyanate Resin and Triazine Compounds			5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145	
6. AUTHOR(S) Y.E. Xu and C.S.P. Sung				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136			8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 54	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219			10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES published in ACS Polymer Preprints, 37(2), 208, 1996				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Dicyanate ester resins have a strong fluorescence and phosphorescence emission. In this work, we report fluorescence and UV spectroscopic characterization of triazine compounds, and identify that the strong fluorescence emission comes from substituted triazine rings which are formed during the cure reaction of polycyanate resins. A series of model compounds were investigated. The results show that substituted triazine rings have similar fluorescence emission to triazine vapor. The emission at 350nm corresponds to sharp emission (335nm) in triazine vapor, and that at 420nm to broad emission (395nm). The large aromatic substituent enhances broad emission which corresponds to the singlet-triplet coupling, and solvation decreases it. When samples are photo or thermally excited, the coupling states start to interact with environment and split to the original states. The split depends on excitation time, concentration and polarity of media.				
14. SUBJECT TERMS dicyanate ester, polycyanate, triazine, fluorescence and phosphorescence			15. NUMBER OF PAGES 2	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	

19970714 071

REPORT DOCUMENTATION PAGE

Form Approved
OME No. 0704-0168

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0168), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 20, 1997		3. REPORT TYPE AND DATES COVERED Technical 06/95-05/97	
4. TITLE AND SUBTITLE Spectroscopic and Model Compound studies in the cure characterization of Bismaleimide/Diallylbisphenol-A Thermoset Resin				5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145	
6. AUTHOR(S) J. Phelan and C.S.P. Sung					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136				8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 55	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219				10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Published in ACS Polymer Preprints, 38(1), 227, 1997					
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Fluorescence, FT-IR and UV-reflectance changes which accompany bismaleimide/diallylbisphenol-A resin cure are reported for three cure schedules. The formation of succinimide moieties is attributed to Ene, Diels-Alder, and alternating copolymerization reactions involving phenylmaleimide and allylphenol groups. The formation of 2-propenylphenol vinyl groups is attributed to Ene reaction between allylphenol groups and maleimide groups, as well as by direct isomerization of allylphenol. The destruction of 2-propenylphenol vinyl groups is provided by UV-reflection spectroscopy. Evidence for Diels-Alder reactions followed by re-aromatization processes is provided by UV-reflectance spectroscopy. Fluorescence signals are initially quenched, but increase and then level off as the resin cures. Model compound studies indicate that emissions that occur at 356nm when the resin is excited at 280nm are from the phenolic portion of the resin, while emissions that occur at 440nm when the resin is excited at 380nm are from phenyl-succinimide. Structure arising from Diels-Alder-Ene and alternating copolymerization reaction sequences have also been confirmed by model studies. Support that the final stages of cure involve re-aromatization of phenolic groups and crosslinking reactions of 2-propenylphenol vinyl groups is presented.					
14. SUBJECT TERMS Fluorescence, FT-IR, UV reflectance, bismaleimide, diallylbisphenol-A, Ene, Diels-Alder and Alternating copolymerization Reactions.				15. NUMBER OF PAGES 2	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT		

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE June 20, 1997	3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97		
4. TITLE AND SUBTITLE Imidization study by Charge Transfer Fluorescence		5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145		
6. AUTHOR(S) J.W. Yu and C.S.P. Sung				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136		8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 56		
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219		10. SPONSORING / MONITORING AGENCY REPORT NUMBER		
11. SUPPLEMENTARY NOTES Published in Macromolecules, 30, 1845, 1997				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The extent of imidization for several polyamic acids was investigated by using charge transfer(CT) fluorescence at a longer wavelength. The fluorescence excitation near 460nm showed red shifts and their intensities decreased as the imidization proceeded. Fluorescence solution study confirmed the origin of CT complex formation. These spectral changes were correlated with the extent of imidization. The fluorescence of the CT complex is a sensitive indicator of the extent of imidization of aromatic polyamic acids.				
14. SUBJECT TERMS Imidization, polyamic acid, polyimide, charge transfer complex fluorescence			15. NUMBER OF PAGES 2	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	

REPORT DOCUMENTATION PAGE

Form Approved
OME No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 20, 1997		3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97	
4. TITLE AND SUBTITLE Interphase Cure Characterization in Epoxy composites by Fluorescence Technique				5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145	
6. AUTHOR(S) J.W. Yu and C.S.P. Sung					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136				8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 57	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219				10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Published in J. Appl. Polym. Sci., 63, 1769, 1997					
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Monitoring the reaction of an aromatic diamine cure agent with epoxy by fluorescence technique was used for cure characterization of the interphase in epoxy/glass and epoxy/carbon composites. The effect of the various surface treatments was first studied by the model interphase obtained by using a quartz plate for glass or a modified quartz plate for carbon surface. Aminosilane treated quartz cured faster and showed increased cure extent, while water aging and air oxidation showed almost no effect on the cure kinetics in comparison to the untreated quartz surface. For a model carbon surface, air oxidation showed a faster reaction only at the early stage of cure. The effects of the various surface treatments on glass or carbon fiber were also studied with the actual composites made by a thin coating of epoxy-diamine melt on the model interphase system. In the case of epoxy-carbon fiber composite, both air oxidation and water aging treatment showed a faster cure reaction at the early stage of cure. Furthermore, air oxidation treatment for the epoxy/carbon fiber composite showed somewhat increased cure extent. The reasons for these trends have been discussed.					
14. SUBJECT TERMS Composite interphase, cure characterization, diamine cure agent, fluorescence technique, surface treatment, glass fiber and carbon fiber composites.				15. NUMBER OF PAGES 7	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT		

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 20, 1997		3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97	
4. TITLE AND SUBTITLE In-Situ Cure Monitoring of Polyimide Via UV/visible reflectance spectroscopy				5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145 n	
6. AUTHOR(S) D.K. Hestermann and N.H. Sung					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136				8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 58	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219				10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Published in ACS PMSE proceedings, 75, 385, 1996.					
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) UV/Visible reflection spectroscopy coupled with a bifurcated fiberoptic probe has been developed as an in-situ monitoring technique for polymer reactions. Thermal imidization of 4,4'-(Hexafluoroisopropylidene) diphthalic anhydride(6FDA) and p-phenylene diamine(PDA) was monitored in-situ by measuring UV reflectance of the sample during its cure. Deconvolution of the reflectance spectra allowed quantitative assay of the amic acid to imide conversion in 6FDA/PDA. A new class of high temperature resin based on phenylethynyl endcapped polyimide p repolymers has also been investigated by UV/Visible, fluorescence, and IR spectroscopy. Cure reaction, involving only the endgroups, was found to be measurable only by IR, and to some extent by fluorescence, but not by UV reflection. The paper also discusses the issues involved in the use of a bifurcated optical probe in obtaining high sensitivity in reflectance measurements.					
14. SUBJECT TERMS UV/Visible reflection spectroscopy, bifurcated fiberoptic probe, thermal imidization, phenylethynyl endcapped polyimide, fluorescence				15. NUMBER OF PAGES 2	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT		

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0168	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0168), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE June 20, 1997	3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97		
4. TITLE AND SUBTITLE In-Situ characterization of BMI/DABPA via Fiberoptic Fluorescence		5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145		
6. AUTHOR(S) H.J. Paik and N.H. Sung				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136		8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 59		
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219		10. SPONSORING / MONITORING AGENCY REPORT NUMBER		
11. SUPPLEMENTARY NOTES Published in ACS PMSE proceedings, 75, 385, 1996				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any prucpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Cure Reactions in 4,4'-bismaleimido-diphenylmethane (BMI) / 0,0'-diallyl bisphenol-A (DABPA) system has been investigated with bifurcated fiberoptic fluorimeter. BMI, the key monomer itself, was observed to show a fluorescence emission at ~590nm, an unusually longer wavelength region when compared to other common aromatic molecules. The spectra was not subject to photobleaching, and was very stable even at those elevated cure temperatures. According to UV-Vis analysis, a very weak shoulder peak was observed at around 550nm, which led us to believe that a charge transfer complex on BMI might be responsible for this long wavelength emission. During the cure process, there was a systematic blue shift of the emission peak upto 60nm. An attempt was made to follow the reaction of BMI/DABPA by monitoring this characteristic fluorescence peak position shift. Using a custom-built, bifurcated fiberoptic fluorimeter, the spectral shift was monitored in-situ or on-line at the cure temperature through out the cure cycle.				
14. SUBJECT TERMS Cure characterization, bismaleimide, diallyl bisphenol-A, bisfurcated fiber optic fluorimeter, a longwavelength emission			15. NUMBER OF PAGES 2	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	

REPORT DOCUMENTATION PAGE			Form Approved OME No 0704-0188	
Public reporting burden for this question is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 20, 1997	3. REPORT TYPE AND DATES COVERED Technical 06/96-05/97	
4. TITLE AND SUBTITLE Reaction Monitoring Epoxy/Fiber Interphase using Evanescent wave Fluorimetry-Instrumentation and Methods			5. FUNDING NUMBERS NR 413 m 011 N00014-89-J-1145	
6. AUTHOR(S) A. Fuchs and N.H. Sung				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Institute of Materials Science 97 N. Eagleville Rd. University of Connecticut Storrs, CT 06269-3136			8. PERFORMING ORGANIZATION REPORT NUMBER Technical Report No. 60	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) ONR 800 N. Quincy Ave. Arlington, VA 22219			10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Published in a book "Polymer Surfaces and Interface" 345, Ed. K.W. Lee and L.K. Mittal, USP. Netherland, 1997				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Reproduction in whole or in parts is permitted for any purpose of the United States Government, this document has been approved for public release and sale; its distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Abstract—Cure characteristics of epoxy in the epoxy/fiber interphase in composites can be studied using an evanescent wave fluorescence technique. This paper describes the methodology and instrumentation which allows <i>in situ</i> monitoring of epoxy reactions at the interface. The reaction of a stoichiometric mixture of diepoxide, diglycidyl ether of bisphenol-A (DGEBA), and the curing agent 4,4'-diaminodiphenyl sulfone (DDS) is followed by measuring the fluorescence intensity change of the externally added reactive dye, diaminoazobenzene (DAA), as it is converted from a primary amine to a tertiary amine state during cure. By using a sapphire optical fiber as an evanescent wave probe, the fluorescence intensity can be measured within an approximately 150 nm thin layer of epoxy at the fiber surface. A high refractive index value and the good UV-visible transmission character of the sapphire make it the only fiber acceptable as an evanescent probe for DGEBA/DDS epoxy. A number of optical innovations are introduced to maximize the evanescent wave intensity, which include a polymer cladding for the sapphire fiber and metallization of the fiber tip. Cure reaction in the interphase region is compared with that of bulk epoxy.				
14. SUBJECT TERMS Epoxy; cure monitoring; interphase; evanescent fluorescence; fiberoptic, sapphire optical fiber, optical innovations, methodology, instrumentation			15. NUMBER OF PAGES 15	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT	