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NATIONAL SCIENCE FOUNDATION FINAL PROJECT REPORT

PART I - PROJECT IDENTIFICATION INFORMATION

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|----------------------------|---|-----------|
| 1. Program Official/Org. | Daborah L. Crawford - ECS | |
| 2. Program Name | SOLID-STATE & MICROSTRUCTURES ENGIN PROG | |
| 3. Award Dates (MM/YY) | From: 09/90 | To: 01/97 |
| 4. Institution and Address | [REDACTED]
Administration Building
Atlanta GA 30332 | |
| 5. Award Number | 9058560 | |
| 6. Project Title | Residential Young Investigator Awards | |

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Part II: Summary of Completed Project

The work performed under this grant has involved a number of different aspects of the use of polymeric materials in microelectronics. A revolutionary low cost, high performance dielectric based on polynorbornene has been developed for use in electronic packaging. It is anticipated that this system will be commercialized in 1998.

Two different techniques have been developed which have potential for monitoring and controlling the polymerization reaction during chip encapsulation (as well as other polymer processing operations). One of these techniques is based on the use of dielectric microsensors and the other involves the development of micromachined sensor based on a vibrating membrane.

Techniques have also been developed to measure the through-plane modulus, the through-plane coefficient of thermal expansion and the in-plane dielectric constant of thin polymeric films (<10 microns) coated on a solid substrate. Using these techniques, the effect of spin-coating on the molecular orientation of thin polymeric films has been investigated.

Novel methods of loading polymeric films with ceramic particles to produce high dielectric constant composites for use in integrated passives has been investigated. The effect of the addition of small particles (diameter < 0.1 microns) into polymeric systems on the mechanical and rheological properties has also been explored.

Part III: Technical Information

My research supported by the NSF Presidential Young Investigator Award has focused on the use of polymeric materials in microelectronic applications. Polymers play a critical role in electronic materials processing, including packaging and interconnection of electronic components, photolithography, use as passivant overcoats and interlevel insulators. The performance of an electronic device often hinges on properties of polymer films used in these applications. My work has been directed towards the establishment of structure-property relationships, which are crucial for developing new chemical systems, formulating process models and predicting end-use performance. The research projects funded through this program are briefly described in this report. In addition, a list of publications, presentations and dissertations resulting from this award are listed.

Low Cost, High Performance Polymers for Microelectronic Packaging

Participating Students: N. Grove; T.C. Hodge; P. Chiniwalla; S. Ahmed; X. Chen

The goal of this study is to identify and characterize new polymers for use in microelectronic packaging. As the performance and speed of integrated circuits

increases, the electrical delay due to the interconnection is becoming an increasingly important part of the device and system performance. As interconnections scale, RC delay and crosstalk limits the performance, especially at high frequency. The interconnection needs can be most clearly summarized by using the RC time constant goals stated in the SIA Semiconductor Technology Workshop. If an interconnection composed of Al-SiO₂ ($\sigma = 3\mu\Omega\text{cm}$ and $\epsilon = 4.2$) meets the 1992 speed objectives, an identical structure using materials with $\sigma = 1.6\mu\Omega\text{cm}$ and $\epsilon = 2.3$ would meet the year 2004 interconnection goals.

We have been working with two polymer companies to develop new polymer dielectric for the next generation of packaging needs. Objectives in the development of these new materials include: low cost (polymers traditionally used in this application are estimated to be 40% of the MCM cost); low dielectric constants; low moisture uptake; good adhesive properties to inorganic layers found in MCMs; existing infrastructure in its use for low cost applications.

The most promising material investigated is polynorbornene, a polymeric system manufactured by BF Goodrich. Working in collaboration with BF Goodrich, the microstructure and side groups attached to the polynorbornene have been optimized for electronic packaging applications. The dielectric constant of this material ranges from 2.3 to 2.6. Alkoxy silane groups have been added to the backbone to achieve excellent adhesion to silicon, silicon dioxide copper, aluminum, silver and gold surfaces. The moisture uptake of this system is <0.1 wt%. Alkyl side groups have been added to the backbone to improve the mechanical properties (i.e. the elongation to break increased from 0% to 40% through the addition of this side group). BF Goodrich anticipates full commercialization of this new polymer by 1998.

Polymerization Monitoring by Dielectric Microsensors

Participating Graduate Students: J.O. Simpson; J.E. Companik; H.J. Nanavati; M. Ratnam; M. Miller; E. Walmet

The goal of this study is to develop a dielectric cure monitoring system for controlling chip encapsulation, as well as other polymer processes. The basis of this cure monitoring system is the use of a microdielectric sensor and free volume models to correlate ion motion with changes in polymer structure and rheology which occur during the reactive process. The first project in this area has focused on epoxy encapsulants. A dielectric sensor was mounted into the bottom plate of a parallel plate rheometer. Rheological and dielectric property changes were monitored simultaneously during the isothermal polymerization of diglycidyl ether of bisphenol-A (DGEBA) epoxy resins with a tetrafunctional amine diamino-diphenyl sulfone (DDS). Free volume models were developed to correlate the changes in dielectric properties with changes in rheological properties during curing. Using these models and a

microdielectric sensor, it is possible to instantaneous follow the viscosity rise during polymerization.

The validity of these free volume models has also been tested by measuring dielectric properties of silicone polymers. Silicones currently enjoy widespread use in bipolar, metal oxide semiconductor and hybrid integrated circuitry encapsulation. In addition, silicones offer some unique characteristics which are advantageous in terms of extending our understanding of dielectric properties in polymers. As a result of the low initial level of ion impurities in many silicone polymers, we can carefully control the amount, type and size of the dominant ions through doping. To dope the polymers, a series of polydimethylsiloxane salts of various cations (i.e. sodium, potassium, tetramethylammonium, tetraethylammonium, tetrapropylammonium and tetrabutylammonium) have been synthesized. The effect of the ion type and size as well as polymer chain length on parameters in the control model has been determined.

A third project in this area involves using dielectric measurements to monitor and control a solution polymerization. Traditionally, viscosity, density and/or refractive index changes have been used to follow solution polymerizations. However, these properties are difficult to measure on-line. The use of dielectric sensors would be advantageous both in terms of their speed and ease of measurement. The systems chosen for this study are polystyrene and polymethylmethacrylate. Permittivity has been found to track the change in refractive index during polymerization, which is related to the extent of reaction. The ionic conductivity changes during the polymerization follow changes in the initiator concentration.

Molecular Orientation in Polyimide Interlevel Dielectric Films

Participating Graduate Students: L. Lin; S. Weinberg

Spin-coated polyimide films are widely used in microelectronics as passivation layers on top of devices or as dielectric interlayers between two levels of metallization. In these applications, the selection of a particular polymer system is dictated by a combination of electrical, thermal, mechanical and device fabrication requirements. In particular, since the interconnections are designed primarily to minimize signal propagation delay, resistive losses, and noise due to reflection and crosstalk, the polymer should have as low a dielectric constant as possible.

Generally the dielectric constant of potential materials to be used as interlevel dielectrics is determined by use of a parallel plate capacitor. These measurements are carried out with the electric field normal to the film plane. Since polyimide chains tend to align along the surface of the coated substrate during the imidization process, the dielectric properties are expected to be anisotropic (i.e. the in-plane dielectric constants is generally larger than the

normally measured out-of-plane values along the film thickness direction). In-plane dielectric properties have not been directly studied in any detail, despite the fact that the in-plane dielectric constants are more relevant to some important device design considerations such as the cross-talk noise between signal lines in the same wiring plane. Knowledge of in-plane dielectric constants may result in a different selection of a polyimide system for a particular application than a decision based solely on the through-plane dielectric constant.

We have fabricated comb (i.e. interdigitated) electrode structures on the surface of a silicon wafer and spin-coated a highly oriented rigid rod polyimide (DuPont PI2611) over the top surface. Two different sizes of interdigitated electrodes were fabricated: (1) with 10 micron spacings between fingers on the electrode; (2) with 6 micron spacings between fingers. Using ANSYS, a finite element package from Swanson Analysis, Inc., we have modeled the portion of electric field passing in-the-plane and through-the-plane of the wafer, and will use this information to decouple the in-plane and through-plane dielectric constants of the film. Studies on DuPont 2611 indicate that the dielectric constant in the plane of the wafer has a value that is 0.8 higher than the through-plane dielectric constant. This is in agreement with the predictions from the refractive index measurements using Maxwell's equation. We have optimized the geometry of these electrodes and then used this design to quantify the anisotropy in dielectric properties for a number of polymer systems commonly used as interlevel dielectrics.

Dielectric Based Measurement of Mechanical and Thermal Properties for Use In Electronic Packaging

Participating Graduate Student: T.C. Hodge; K. Patel; B. Sinno

This study focuses on the development of a new method for in-situ determination of the modulus and thermal expansion coefficient in the direction normal to the surface of the thin film for evaluating polymers for multi-chip modules and other packaging applications. A number of in-situ techniques have been developed for measuring thin film properties; however, each of these methods are restricted to the measurement of in-plane mechanical properties. The spin coat processing of polymer films lead to molecules which are oriented in the plane of the film; this molecular anisotropy will cause the mechanical and thermal properties in the plane of the film to be different from those properties through the plane of the film. This work involves the use of microfabricated electrodes to determine small changes in film thickness that result when the temperature or pressure in the film is varied.

Work has concentrated on refining the electrode structures and measurements techniques for determining the through-plane coefficient of thermal expansion of thin films for two polymer systems. Differences have been detected between the in and through-plane CTE measurements, even on systems which are believed to be electrically and optically isotropic. Part of this effect has been attributed to the

fact that a film constrained on a substrate surface will have a different coefficient of thermal expansion than a free film (i.e. Poisson's effect). The study is being extended to evaluate a wider range of materials.

This technique has been adapted for measuring the through plane modulus of a thin polymeric film (<10 microns) spin-coated on a polymeric surface. We have obtained preliminary results for the through-plane modulus of benzocyclobutene (BCB) using this technique and have measured the same value that was obtained on bulk samples of BCB using standard techniques.

Micromachined Viscosity Sensors

Participating Post-doctoral Associate: O. Brand

This work deals with the use of microfabrication technology to realize *microfluidic devices*, and the application of these devices to a specific problem: *the measurement of the viscosity of reacting polymer systems in real time*. Such real-time measurement allows assessment not only of the degree of reaction but also the ability to determine whether desired mechanical properties of the polymers are being achieved during the reaction. The microfluidic devices are based on the concept of a vibrating mechanical member in contact with a fluid. The fluid 'loads' the vibrating member to various degrees depending on fluid viscosity, changing its resonance frequency and quality factor (Q-factor); these changes can be used to assess viscosity. We have successfully fabricated and tested prototype devices which have been shown to react to standard polymeric systems with viscosities ranging from 1-600,000 cSt.

The results of the work can be summarized as follows: (A) It was confirmed using commercially available piezoelectric resonators that this approach would be feasible for viscosity measurement using single standard PDMS solutions; (B) micromachined membrane resonators were fabricated and tested; and (C) It was confirmed that micromachined membrane resonators show substantially the same behavior as the commercial transducers for single standard PDMS solutions, thus confirming the feasibility of using micromachined membrane resonators for viscosity measurement.

Both commercially available piezoelectric transducers and micromachined membrane resonators were investigated for viscosity sensing. The piezoelectric transducers had a diameter of 25 mm and consisted of a circular brass membrane with a piezoceramic glued to it (see Fig 2). Two separate electrodes (having a common counter electrode) are available for piezoelectric excitation and detection of transverse vibrations. The silicon based membrane resonators have a sidelength between 1 and 3 mm and feature p-doped silicon resistors for electrothermal excitation and piezoresistive detection of transverse membrane vibrations. The membrane is comprised of a monocrystalline silicon layer and several dielectric layers on top (Figure 1).

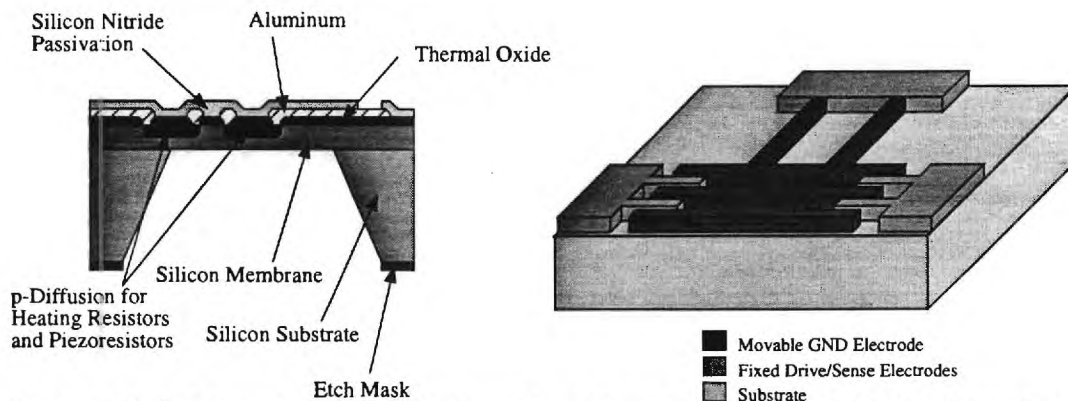


Figure 1. (Left) Schematic side view of micromechanical membrane resonator. (Right) Perspective view of comb drive lateral shear resonator.

Electrical connections are made with an aluminum layer. The fabrication sequence is compatible with standard CMOS or bipolar IC technology. The membrane resonators are released in a post-processing anisotropic etching step using a 30 % potassium hydroxide solution. Control of fabrication induced in-plane stresses allow the fabrication of membrane resonators with axial loads close to the buckling load. These resonators show minimal fundamental resonance frequency and maximum vibration amplitudes.

Both devices were appropriately packaged and tested as viscosity sensors. The fundamental resonance frequency and the resonance quality factor Q was monitored as a function of the viscosity. Polydimethylsiloxanes (PDMS) with viscosities ranging from unity to 600,000 cSt (corresponding to $8 \cdot 10^{-4}$ to 600 Pa·s) were used to investigate the properties of the resonators in viscous polymer solutions. All measurements were performed at room temperature and pressure without stirring the polymer solutions.

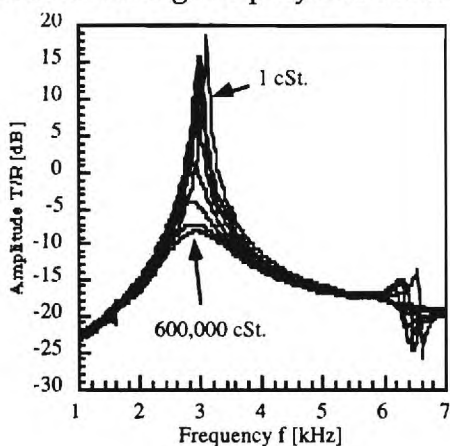


Figure 2

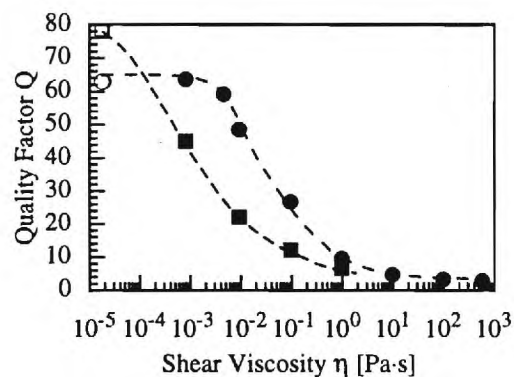


Figure 3

Figure 2 shows a typical resonator amplitude plot as a function of frequency as parameterized by the ambient fluid viscosity. A clear decay of Q -factor is seen as the viscosity is increased. Figure 3 is extracted from these data taken on both resonator types and shows the Q -factor of the micromachined membrane resonator (squares) and the piezoelectric transducer (circles) as a function of the

shear viscosity of the surrounding media. The quality factor of the micromachined membrane resonator drops from 78 in air to about 6 in a 1000 cSt PDMS solution, demonstrating the feasibility of this technique.

Rheological Studies of Epoxy Molding Compounds

Participating Post-Doctoral Associate: T. Wang

In order for reactive flow simulations to yield an accurate representation of the resin transfer molding process, knowledge of the kinetics and rheology of each specific molding compound is required. Although extensive work has been done on chemorheological relations for epoxy systems, the majority of these studies have focused on slower reacting, model systems rather than the fast reacting (< 1 minute) commercial encapsulation systems. This study has focused on measurement of the cure kinetics and viscosity as a function of reaction time, reaction temperature and shear rate for designated commercial molding compounds. A variety of chemorheological models have been examined to determine which yields the best description of the rheological changes during the molding process. This information combined with flow simulation programs can effectively reduced the mold design-to-implementation cycle time. For a given polymeric system, various mold layouts can be evaluated, alterations studied and problems pinpointed before any actual metal is actually machined. Process conditions can also be optimized a priori to produce defect-free molded parts. These reactive flow simulations can yield large cost savings in the elimination of unnecessary prototype building and molding trials.

The goal of this project is to develop constitutive equations for selected commercial epoxy molding compounds, incorporating kinetics and rheological behavior. For this study, we have selected Sumikon EME 6300 HN and Sumikon EME 6531 epoxy molding compounds. Kinetics were modeled using an autocatalytic kinetic equation. Substantial differences in kinetics were observed between the dynamic and the isothermal cure. We believe this is due to diffusion controlled kinetics dominating the dynamic experiment. The viscosity rise was modeled using a modified Castro-Macosko equation.

Filled Polymers for Use as Integrated Passives

Participating Students: K. Farnsworth; R. Manepalli

The integration of components into electronic packages adds functionality and performance. If these components (resistors, inductors, and capacitors) can be formed in-mass from polymer composites, they can also be produced at a lower cost than individual components. In this project, opportunities for developing filled-polymer composites with special electrical (high K) properties will be explored. Our approach has been to attach high K ceramic particles to the polymer backbone which may result in minimizing the amount of polymeric material and maximizing the amount of high K material. Silane groups have been attached to BaTiO₃ particles and then reacted to the polymeric backbone

(benzocyclobutene). Using this technique, we have increased the dielectric constant of benzocyclobutene from 2.7 to 50. In addition, improved mechanical properties have been observed using silane treated particles versus simple loading of untreated particles into the polymeric system. Work is now in progress to investigate treated particle loading in epoxy systems.

Understanding the relationship between particle loading and rheological properties is critical for processing these polymeric composites for use in integrated passives. The second portion of this study involves determining the relationship between the percent filler, the size and type of filler on the rheological properties. This study will aid both in the processing of loading polymers for integrated passives as well as provide a fundamental understanding of the effect of adding small particles (diameter < 0.1 microns) on the rheological properties of polymeric composites.

Books, Journal and Proceedings Publications

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H. J. Nanavati, "Study of Imidization of Polyamic Acid Using Microdielectrometry" M. S. Thesis, School of Chemical Engineering, Georgia Tech, Winter 1992.

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J. E. Companik, "Segment and Ion Mobility in Polydimethylsiloxanes", Ph.D. Thesis, School of Chemical Engineering, Georgia Tech, Summer 1993.

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PART IV - FINAL PROJECT REPORT -- SUMMARY DATA ON PROJECT PERSONNEL
(To be submitted to cognizant Program Officer upon completion of project)

The data requested below are important for the development of a statistical profile on the personnel supported by grants. The information on this part is solicited in response to public Law 99-383 and 42 USC 1885C. All provided will be treated as confidential and will be safeguarded in accordance with the provisions of the Privacy 1974. You should submit a single copy of this part with each final project report. However, submission of the information is not mandatory and is not a precondition of future award(s). Check the "Decline to Provide below if you do not wish to provide the information.

Please enter the numbers of individuals supported under this grant.

Do not enter information for individuals working less than 40 hours in any calendar year.

	Senior Staff		Post Doctorals		Graduate Students		Under-Graduates		Other Participants ¹	
	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.
A. Total, U.S. Citizens					4	6	21	9		
B. Total, Permanent Residents			1							
U.S. Citizens or Permanent Residents: ²										
American Indian or Alaskan Native....										
Asian.....						2	3	1		
Black, Not of Hispanic Origin.....						1	2	2		
Hispanic.....										
Pacific Islander.....										
White, Not of Hispanic Origin.....					4	3	16	6		
C. Total, Other Non-U.S. Citizens			1		6	1				
Specify Country										
1. Germany			1							
2. India					5					
3. China					1	1				
D. Total, All participants (A + B + C)			2		10	7	21	9		
Disabled ³										

Decline to Provide Information: Check box if you do not wish to provide this information (you are still required to return this along with parts I-III).

¹Category includes, for example, college and precollege teachers, conference and workshop participants.

²Use the category that best describes the ethnic/racial status to all U.S. Citizens and Non-citizens with Permanent Residency. (If more than one category applies, use the one category that most closely reflects the person's recognition in the community.)

³A person having a physical or mental impairment that substantially limits one or more major life activities; who has a record of impairment; or who is regarded as having such impairment. (Disabled individuals also should be counted under the appropriate ethnic/racial group unless they are classified as "Other Non-U.S. Citizens.")

AMERICAN INDIAN OR ALASKAN NATIVE: A person having origins in any of the original peoples of North America and who maintains cultural identification through tribal affiliation or community recognition.

ASIAN: A person having origins in any of the original peoples of East Asia, Southeast Asia or the Indian subcontinent. This area includes, for example, China, India, Indonesia, Japan, Korea and Vietnam.

BLACK, NOT OF HISPANIC ORIGIN: A person having origins in any of the black racial groups of Africa.

HISPANIC: A person of Mexican, Puerto Rican, Cuban, Central or South American or other Spanish culture or origin, regardless of race.

PACIFIC ISLANDER: A person having origins in any of the original peoples of Hawaii, the U.S. Pacific territories of Guam, Samoa, and the Northern Marianas; the U.S. Trust Territory of Palau; the islands of Micronesia and Melanesia; or the Philippines.

WHITE, NOT OF HISPANIC ORIGIN: A person having origins in any of the original peoples of Europe, North Africa, or the Middle East.