An Improved Thermal Conductivity Polyurethane Composite for a Space Borne 20KV Power Supply¹,²

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Abstract—This effort was designed to find a way to reduce the temperature rise of critical components of a 20KV High Voltage Power Supply (HVPS) by improving the overall thermal conductivity of the encapsulated modules. Three strategies were evaluated by developing complete procedures, preparing samples, and performing tests. The three strategies were: 1. Improve the thermal conductivity of the polyurethane encapsulant through the addition of thermally conductive powder while minimizing impact on other characteristics of the encapsulant. 2. Improve the thermal conductivity of the polyurethane encapsulated assembly by the addition of a slab of thermally conductive, electrically insulating material, which is to act as a heat spreader. 3. Employ a more thermally conductive substrate (Al₂O₃) with the existing encapsulation scheme. The materials were chosen based on the following criteria: high dielectric breakdown strength; high thermal conductivity, ease of manufacturing, high compliance, and other standard space qualified materials properties (low out-gassing, etc.). An optimized cure was determined by a statistical design of experiments for both filled and unfilled materials. The materials were characterized for the desired properties and a complete process was developed and tested. The thermal performance was substantially improved and the strategies may be used for space flight.

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1. INTRODUCTION

A series of experiments were carried out to improve the thermal characteristics of a 20KV high voltage power supply (HVPS). The overall task objectives were:

1. Improve the thermal conductivity of the potting compound (Sytec EN11-EN4 Conathane)¹ while minimizing impact on other characteristics. Perform testing to validate characteristics.
2. Improve the thermal conductivity of the potted assembly by the addition of a slab of thermally conductive, electrically insulating material.
3. Improve the thermal conductivity of the potted assembly by use of more thermally conductive substrate.

The overall strategy employed was to use statistical designs of experiments for determining optima of various parameters. The general types of experiments that were performed for this task are outlined below:

1. Cure optimization of EN11-EN4 polyurethane including; filled material, presence of AlN and of A and B stages
2. Shear testing of filled and unfilled material
3. Thermal conductivity
4. Construction of mock-ups

In this paper we will focus on the first category of experiments.

2. BACKGROUND

The strategy for the first objective was to add fillers to current EN11-EN4 polyurethane to increase thermal conductivity without significantly degrading other important characteristics. The qualification criteria for objective 1 and the characteristics used for evaluating the performance of the EN11-EN4 polyurethane with fillers and are given in Table I.
Table I. Potting Compound Material Characteristics

<table>
<thead>
<tr>
<th>Characteristics Used for Evaluation</th>
<th>Important Characteristic (also to be measured)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion Strength</td>
<td>Operation above 130C</td>
</tr>
<tr>
<td>Dielectric Breakdown</td>
<td>Dielectric Constant</td>
</tr>
<tr>
<td>% Cure (DSC)</td>
<td>Bubble free</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>Dynamic Modulus (DMA)</td>
</tr>
<tr>
<td></td>
<td>CTE, Tg (TMA)</td>
</tr>
<tr>
<td></td>
<td>Particle Distribution</td>
</tr>
<tr>
<td></td>
<td>Shore hardness</td>
</tr>
</tbody>
</table>

The plan for the first objective was to evaluate several different fillers for best performance. Candidates were selected from current industry state-of-the-art are given in Table II.

Table II. Candidate Thermally Conductive Filler Materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>K (W/mK)</th>
<th>CTE</th>
<th>ε (@1MHz)</th>
<th>R (Ω-cm) @ 25°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>15</td>
<td>9</td>
<td>9</td>
<td>&gt;10²</td>
</tr>
<tr>
<td>BN</td>
<td>70</td>
<td>1.2</td>
<td>7.1</td>
<td>10⁶</td>
</tr>
<tr>
<td>AIN</td>
<td>150</td>
<td>4.8</td>
<td>8.7</td>
<td>10¹²</td>
</tr>
<tr>
<td>EN11 (current)</td>
<td>0.2</td>
<td>78</td>
<td>3</td>
<td>&gt;4.3x10¹⁷</td>
</tr>
</tbody>
</table>

Screening studies were performed on Al₂O₃ with 15, 30, 45 and 60 per cent filler additions. Optimization was performed on A stage (B stage constant) and B stage (A constant) with filler added using screening design of experiments (DoE). Response surfaces were generated based on percent cure. Optimization was verified with adhesion strength.

Some additional considerations needed to be taken into account in order to address process implementation issues. These considerations included a special mixer required for adding fillers and mixing under vacuum, and accounting for adhesion promoter interactions which have a role in the cure.

The strategy for the second objective was to add a thermally conductive substrate to current EN11-EN4 polyurethane to increase thermal conductivity without significantly degrading other important composite characteristics. The qualification criteria for objective two were the characteristics used for evaluation of the embedded substrate given in Table II.

Table III. Characteristics for Substrate Material Evaluation.

<table>
<thead>
<tr>
<th>Characteristics Used for Evaluation</th>
<th>Important Characteristic (also to be measured)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion Strength</td>
<td>Operation above 130C</td>
</tr>
<tr>
<td>Dielectric Breakdown</td>
<td>Dielectric Constant</td>
</tr>
<tr>
<td>% Cure (DSC)</td>
<td>Bubble free</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>Dynamic Modulus (DMA)</td>
</tr>
<tr>
<td></td>
<td>CTE, Tg (TMA)</td>
</tr>
<tr>
<td></td>
<td>Particle Distribution</td>
</tr>
<tr>
<td></td>
<td>Shore hardness</td>
</tr>
</tbody>
</table>

The plan for the second objective was to evaluate several different substrate materials for best performance, in light of thermal conductivity, dielectric strength, availability and ease of use. Candidates were selected from current industry state of the art including Al₂O₃, AIN BN, and BeO. Cure optimization was performed on A stage (B stage constant) and B stage (A constant) with presence of substrate. Response surfaces were generated on percent cure using a screening DoE. Optimization was verified by adhesion strength.

Within the objective is a requirement to develop a procedure for bubble free addition of substrate in close proximity to backing plate. Again an additional consideration needed to be given to the adhesion promoter interactions with the substrate materials and with the EN11-EN4 polyurethane.

The strategy for the third objective was to use a thermally conductive substrate with the current EN11-EN4 polyurethane system to increase thermal conductivity without significantly degrading other important composite characteristics. The qualification criteria for objective three and the characteristics used for evaluation of the embedded substrate are given in Table III, the same as for objective two. An additional requirement was availability as an electrical circuit within the launch timeframe of the program. This last requirement led to the selection of Al₂O₃ for the substrate material for objective three.

Materials selection was a critical part of the process. Because of the similar function of the filler and substrates, and the limited number of materials easily available, the two applications required the evaluation of nearly the same materials list (although BeO was never considered in powder form). The primary difference between the materials was their morphology. The materials considered and some of their relevant properties are given in Table IV. In addition to the materials characteristics, the selection was based on availability, delivery and the needed form factor as well as ease of use. The best properties were from BeO. Since it is extremely hazardous to machine BeO and it is very toxic in its powder form, use as a filler was not considered and substrates would have to be fired to size for the application. Determination of the correct size and having the substrates made put BeO outside the schedule needed for testing. AIN was selected for both applications as the next best material. BN was also selected for the filled applications because the powdered BN morphology was much better than the AIN due to the commercial BN powder formation process. The smooth finish of the BN particles would allow for higher loading contents. BN was going to be tested until the program was cancelled. Because Al₂O₃ powder was available in-house, some early experiments were also carried out with this material.

Table IV. Properties of Some Candidate Materials.
It should be noted that these properties are "average" properties and may very significantly depending on the supplier's process and process control.

3. Experiment

In this paper we will describe a cure optimization experiment. A number of these experiments were performed and the results from several additional experiments will be shown. Finally, we will give the results of a mock-up module that was used to demonstrate the final process.

Polyurethane Cure Optimization

Experimental Objective

The objective of the first experiment was to optimize the cure of the EN11-EN4 polyurethane system. This polyurethane system has a two stage cure recommended by the manufacturer of 60°C for 24 hours and a demold time of 60°C for 8-10 hours. The traditional cure for space-qualified processes at JPL was a two-stage process 24 hours@ 35°C, 16 hours @ 60°C. Because the eventual presence of the filler would change the optimum cure, it was thought best to check the optimization of the existing cure. Generally, this type of polymer cures in two stages, A. chain addition and B. cross-linking. With this thought in mind, the optimization of the chain addition was the first step in this process. The objective was to find a minimum in residual uncured polymer as determined by digital scanning calorimeter (DSC) while retaining the other properties of the material including: adhesion strength, (determined by lap shear strength), compliance (determined by Shore hardness) and dielectric breakdown.

Design of Experiment

The experimental design was based on a central composite design generated by a statistical design of experiments software, JMP[2]. The ranges were centered on existing schedules and bounded by an estimate of the lower end and then the formation of an orthogonal experimental design. The design is given below in Table V. Some replicates were also run.

<table>
<thead>
<tr>
<th>Material</th>
<th>$K$ (W/mK)</th>
<th>CTE</th>
<th>$\varepsilon$ (@1MHz)</th>
<th>$R$ (Ω-cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$</td>
<td>15</td>
<td>9</td>
<td>9</td>
<td>$&gt;10^{12}$</td>
</tr>
<tr>
<td>BN</td>
<td>70</td>
<td>1.2</td>
<td>7.1</td>
<td>$&gt;10^8$</td>
</tr>
<tr>
<td>AlN</td>
<td>150</td>
<td>4.8</td>
<td>8.7</td>
<td>$&gt;10^{12}$</td>
</tr>
<tr>
<td>BeO</td>
<td>235</td>
<td>8</td>
<td>6.6</td>
<td>$10^{16}$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Pattern</th>
<th>Time (hrs)</th>
<th>Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>00</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>++</td>
<td>42</td>
<td>30</td>
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<td>0</td>
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<td>40</td>
</tr>
<tr>
<td>a0</td>
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<td>35</td>
</tr>
<tr>
<td>a0</td>
<td>30</td>
<td>40</td>
</tr>
</tbody>
</table>

Experimental Procedure

The EN11-EN4 were mixed according to the procedure developed. With a ratio of 1 to 0.55 as recommended by the manufacturer with degassing during mixing and again after mixing was complete. The mixture was poured into 10 identical aluminum trays approximately 7cm in diameter and 1cm deep. The trays were placed in calibrated, monitored ovens at the appropriate temperatures and removed at the appropriate times for the experiment.

The experimental procedure for powder and block addition was similar to the previous experiment, but with the following additions: 1. The addition of 45 % (wt) of AlN powder to the EN11-EN4 mixture; 2. The placement of an Al block 0.050 inches from the AlN block (similar to the ones used in the previous experiment). This was to simulate the precise geometry of the final product which would have an AlN block 0.050 inches away from an Al mounting plate. A procedure for mixing the AlN powder into the EN11-EN4 was developed. This turned out to be somewhat of a challenge because the AlN catalyzed the reaction. Additionally, the morphology of the AlN was that of a frit with a long needle-like structure and rough edges. This morphology is less conducive to mixing than some of the synthetic additives such as BN and fused silica, which tend to be more spherical. The blocks were spaced 0.050 inches apart using a pre-formed spacer made of EN11-EN4.

Calculating an optimal particle size for the powder was also a challenge. Smaller particles have higher surface areas making creating more of a catalyst surface. Larger particles do not mix well and cannot fit into complex geometries (including the 0.050 inch gap between the Al and AlN). After a survey of vendors and the current literature, a mesh size of 100-120 was determined to be optimal. The other question was whether to add an adhesion promoter or not with the powder. In the previous use of the blocks and based on general practice of the industry, an adhesion promoter was used. The amount of adhesion promoter to be added was enough to coat the surface of each particle with about 2μm of material based on Puddleman[3]. A particle size –surface area worksheet was developed by P. Willis[4] to determine the precise amount of adhesion promoter to use. Initial results were found both using the adhesion promoter previously used with the EN11-EN4 system, Dow Corning Z-6020[5], an alkoxysilane compound designed to adhere selectively to the ceramic, and without using the adhesion promoter.
4. Results

The response measurements for this optimization were percentage cure based on residual exotherms from DSC measurements. Table VI gives the responses based on these DSC measurements. Figure 1 shows a sample DSC with the exotherm indicated. The area under the curve corresponds to the total energy released in the temperature range in this case, between about 115°C and 225°C. The heating rate for the sample was 10°C/min. Thermomechanical analysis (TMA) was used to determine glass transition temperatures. DSC data was also used to determine the glass transition, in this case at about -74°C. The two measurement techniques are roughly correlated, although the DSC detected the transition of the onset several degrees earlier as would be expected. Figure 2 shows the complementary TMA measurement with its corresponding glass transition. A secondary transition is also evident at about 50°C.

![DSC Measurement](image1)

**Figure 1. Typical DSC measurement for EN11-EN4 polyurethane cure.**

![TMA Measurement](image2)

**Figure 2. Typical TMA measurement for EN11-EN4 polyurethane cure.**

<table>
<thead>
<tr>
<th>TIME (HRS)</th>
<th>Temp. (°C)</th>
<th>Residual Exotherm (J/g)</th>
<th>Tg (°C) DSC</th>
<th>Tg (°C) TMA</th>
<th>Shore A</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>30</td>
<td>10.92</td>
<td>-73.79</td>
<td>-77.77</td>
<td>65</td>
</tr>
<tr>
<td>42</td>
<td>30</td>
<td>26.01</td>
<td>-72.85</td>
<td>-78.34</td>
<td>65</td>
</tr>
<tr>
<td>18</td>
<td>40</td>
<td>19.65</td>
<td>-73.00</td>
<td>-78.34</td>
<td>65</td>
</tr>
<tr>
<td>18</td>
<td>35</td>
<td>25.77</td>
<td>-73.49</td>
<td>-76.39</td>
<td>65</td>
</tr>
<tr>
<td>42</td>
<td>35</td>
<td>33.53</td>
<td>-72.89</td>
<td>-76.79</td>
<td>67</td>
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<td>30</td>
<td>35</td>
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<td>-72.97</td>
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<td>42</td>
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<td>67</td>
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<td>18</td>
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<td>-73.02</td>
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<td>66</td>
</tr>
<tr>
<td>30</td>
<td>40</td>
<td>32.85</td>
<td>-72.6</td>
<td>-79.4</td>
<td>65</td>
</tr>
</tbody>
</table>

5. Analysis and Discussion

Polyurethanes such as EN11-EN4 are typically a statistical block co-polymer consisting of hard and soft regions of the molecule. In addition to the chain linking of the two major functional groups, these chains may also cross-link. Reaction 1 shows a polyurethane block co-polymerization for EN11-EN4 with the soft and hard regions identified on the products side of the reaction.

The catalysis/cure reactions of this type of reactions have a number of possible outcomes, listed below:

1. Chain addition of di-isocyanate
2. Chain addition of prepolymer diol
3. Chain addition of the hexane diol
4. Cross-link at the isocyanate
5. Cross-link at the double bonded oxygens

In addition, it is common for manufacturers to put blocking molecules on the di-isocyanate such as amids and oximes to slow the reaction or to keep contamination from interfering with the polymerization. It is also possible for the isocyanates to block themselves. This is a special case with the particular starting material for EN4. The particular dimeric isocyanate, 2,4-tolulene di-isocyanate does react with itself as a blocker as shown in Reaction 2. These cyanate blockers and the EN4 dimer give two more possible reactions to consider as discussed in Potter, Rosthauser and Schmelzer. [6]

1. Di-isocyanate self reaction
2. Blocker disassociation reaction
2,4 toluene di-isocyanate polybutadiene-diol pre-poloymer 2-ethyl-1,3hexane-diol

The objective is to balance these reactions with the following criteria:

1. No residual uncured material (which could outgas and contaminate the spacecraft)
2. Compliance of the final polymer (rigidity of the final polymer will stress the components during thermal excursions)
3. Sufficient adhesion strength

Additional response surfaces of these experiments provide a view of the residual reactions after the cure (criterion 1) the Shore A hardness of the material after cure (criterion 2) and the adhesion strength measured with lap-shear testing (criterion 3). Note that the last two are not shown due to length constraints, and did not add significantly to the understanding.

The response surface was developed by plotting the residual exotherms (measured as in Figure 1) against time and temperature for the A stage cure. The cure optimization of the EN11-EN4 system alone is shown in Figure 3. What can be seen in this plot is that there is a local minimum at 30°C, 30 hours and a secondary minimum at 40°C, 18 hours, which is less clear in the surface plot.

Figure 3. Residual exotherm (DSC) as a function of time and temperature for the EN11-EN4 system
This result is somewhat curious. It means that longer times and higher temperatures the cure is worse. This can be explained by the competing reactions such as Reaction 2. If one of the reactions is initiated by a higher temperature and does not complete, residual chain ends will react upon further heating at higher temperatures (in the DSC). Note that this is a two stage process with chain lengthening being one process and cross-linking happening at a higher temperature, hence the two stage cure process.

*Cure optimization with AlN block and AlN powder*

The resulting contour plot with the added AlN substrate is shown in Figure 4. If this is compared to Figure 3, it is clear that there has been a shift in the optimization. The optimum has shifted to a shorter time (18 hours). The secondary minimum has also shifted, but in the opposite direction. This minimum has moved from 18 to 30 hours. The first is easily explained by an increase in the rate of reaction due to the presence of the AlN block. The AlN block appears to provide a surface that assists the breaking of the cyanate bonds. The reaction temperatures were substantially higher with the presence of the block, and the reaction proceeded faster as witnessed by a shorter working life witnessed by the operators of about 15 minutes (the standard is about 45 minutes). Based on our previous analysis, these all indicate a de-blocking of the isocyanate (in this case probably with itself) which would occur at higher temperatures leaving some reaction ends open for further polymerization of any residual material. This data appears to be consistent with the previously provided explanation of blocked isocyanate groups. Additionally, this data also supports using the minimization of the DSC residual exotherm as a measure of residual un-reacted material.

Figure 5 shows the cure optimization of 45% AlN filled EN11-EN4 with an Al and an AlN blocks present. Note that this optimization, in absolute terms, contains the best cured samples in the entire set of experiments. This is most likely due to the AlN solid surfaces actually participating in the reaction. The AlN surfaces do provide a catalysis site as well as a source of N, which participates in the urethane reaction. Adhesion promoter was also determined to participate in the reaction and had the effect of greatly accelerating the reaction rate. As a result, adhesion promoter was not used on the AlN powder, but was used on the substrates.

The optimization becomes more complicated. The 30 hour -30°C optimum is still present, however, the optima at 30 hours and 40°C deepens and the one at 18 hours appears to shift towards 35°C.
Thermal Conductivity of Filled Systems

The thermal conductivity as a function of filler is plotted in Figure 6. The results were initially somewhat disappointing. A simple mixing model can be developed to explain the results. The data behaves as though there is no connectivity between particles. If connectivity were attained the plot would show a curve upward between 60 and 100% filler addition.

Significant limitations were encountered due to processing. Two effects worked against increasing the amount of filler. First, the viscosity significantly increased with the amount of filler. Since the AlN particles are less than sphere-like, the mixture became extremely thixotropic, preventing it from pouring well into the confined area of the molds. A second effect was that the AlN catalyzed the reaction. Reaction 3 will actually take place faster with the large number of surface sites for the di-isocyanate material to adhere. This effect was clear from the rapid heating of the mixing bowl due to the exothermic reaction. With the added cooling jacket for the bowl, the problem was reduced. Also, the addition of the alkoxy silane primer (adhesion promoter) accelerated the reaction, reducing the working life to about 5 minutes. With the cooled mixing bowl and 45% addition of AlN, a working life of about 25 minutes was achieved, which was at the extreme limit required for the process.

Ceramic Fillers Added to EN11-EN4 Polyurethane

Figure 6. Thermal Conductivity as a function of % added filler.

Mock-up Module

A bubble-free process was developed for embedding AlN substrates with filled or unfilled EN11-EN4 urethane in a module the size of one of the HVPS modules (using the actual molds). The AlN plate was placed 0.050 in from the top of the module, the same configuration as in the actual modules. The modules were successfully potted according to the procedure. X-ray images of the interior of a module are shown in Figure 7-9.

Figure 7. Mock-up module X-ray top left corner.
temperatures. The addition of AIN powder shows three distinct minima, the original at 30 hours and 30°C, the deepening of the secondary at 30 hours and 40°C and the movement of the third at 18 hours to 35°C. Because of increasing rigidity the higher temperature cure is not recommended.

Processes were developed for both the mixing of AIN powder into EN11-EN4 and for pouring void free assemblies with a narrow (0.050 inch) gap between AIN and Al blocks. It was determined that adhesion promoter should not be used when mixing powders for this system.

The thermal conductivity of the EN11-EN4 material nearly doubled by the addition of 45% AIN. Further addition of AIN to the EN 11-EN4 shortened the working life to make the mixture unusable. The loading limit of Al₂O₃ was the same as for the AIN, but with only slightly lower thermal conductivity.

The process followed was successfully implemented on a full-size mock-up of an HVPS module. The accurately simulated spacing of components and the AIN block used in this procedure show that this process is ready for use on flight hardware.

6. CONCLUSIONS

The cure of this urethane system is complex. It involves competing reactions making optimization difficult. The optimum cure appears to be at 30°C for 30 hours for the first cure stage. Longer times or higher temperatures increase the rigidity, the primary and secondary glass transitions as well as the uncured material. Lower times and temperatures tend to leave more residual uncured material.

At 30°C and 18 hours, a similar type of optimum was found for the EN11-EN4 system with an AIN block as was found for the pure system (minimum at 30°C and 30 hours). The original optimum of 30 hours and 30°C was still evident. The presence of the AIN block increases the reaction rate approximately 3-fold. Again, longer times and higher temperatures increase the rigidity and the amount of residual uncured material, however, a secondary minimum (not as deep as the primary) is apparent at 30 hours at higher temperatures. The addition of AIN powder shows three distinct minima, the original at 30 hours and 30°C, the deepening of the secondary at 30 hours and 40°C and the movement of the third at 18 hours to 35°C. Because of increasing rigidity the higher temperature cure is not recommended.

Processes were developed for both the mixing of AIN powder into EN11-EN4 and for pouring void free assemblies with a narrow (0.050 inch) gap between AIN and Al blocks. It was determined that adhesion promoter should not be used when mixing powders for this system.

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Glen Anderson - responsible for much of the work performed, materials ordering and logistics and final assembly aids, shore testing etc., etc. etc.

Johnny Duong - responsible for many of the original concepts, processes and instructions

Tran Nguyen - EN11-EN4 polyurethane mixing and experiment execution

Gary Plett - DSC, TGA, DMA

Walt Solek - Vacuum mixing machine set-up, installation and process instructions

Paul Willis - significant insight into the polymeric process as well as the powder/size mesh worksheet and adhesion promoter calculations

REFERENCES


**Biographies**

Andrew A. Shapiro has been working in microelectronic interconnects for twenty years. He has worked as a member of the technical staff at Rockwell International and Hughes Aircraft, where he was responsible for the packaging of a number of phased array radars and ran their high density interconnect line. He was a Principal Scientist at Newport Communications/Broadcom, where made the first commercial polymer 10GHz Si packages, and he has designed and packaged 10 and 40GHz optoelectronic modules. He has also been Project Manager at California Institute of Technology's Jet Propulsion Laboratory and is currently a Principal Engineer in the Electronic Packaging and Fabrication Section (349) at JPL where he is implementing new electronic, RF and optical technologies onto space missions. He earned his BS in chemical engineering at U.C. Berkeley, his MS in Materials Science at UCLA and his Ph.D. in Materials Science at U.C. Irvine. He is on several national committees including NEMI optoelectronics roadmap, ECTC optoelectronics and, IMAPS education. Dr. Shapiro is also currently Assistant Adjunct Professor in Materials Science and Engineering at U.C. Irvine and is performing research in environmentally friendly manufacturing of electronics and optical and high frequency packaging.