

EPOXY ADHESIVES IN MICROELECTRONIC HYBRID APPLICATIONS

The advantages of using epoxy adhesives in the fabrication of hybrid microelectronic devices are unfortunately offset by their tendency to release volatile materials over time into the interior of the device package, which subsequently can affect the device's long-term reliability. Our work on a model epoxy system focuses on identifying and understanding the source and production mechanism of these impurities, and aims to eliminate them.

INTRODUCTION

In many military, space, and other high-reliability applications, instrumentation packages often incorporate microelectronic hybrid devices. These devices, which perform a single function or related multiple functions, constitute a physical package that is usually hermetically sealed. The package can be considered as a building block in the structure of the overall system design. It provides physical protection and isolation of the internal elements, as well as electrical connections from these elements to the outside world, that is, integration with the other elements of the system.

Within the hybrid package, one finds a lower level of integration in which electronic components (chips), typically silicon or gallium arsenide, operate with functions ranging from those of simple resistors, capacitors, diodes, and transistors, to the more complex analog functions (e.g., amplifiers, multipliers, and mixers) and digital functions (e.g., logic gates, memory, and processors). Typically, the chips are located on a ceramic substrate that serves several functions, one of which is to provide a structural platform to position and hold the chips. The substrate also provides for the electrical interconnections between the components via patterned conductive traces. In addition, it serves as a thermal connection to the outside world to dissipate heat from the operating device, via its attachment to the hybrid package.

The methods of attaching the components to the substrate and attaching the substrate to the hybrid package entail the use of either inorganic or organic "attach" materials. Inorganic attach materials include solder, gold-silicon eutectic, and silver glasses. Organic attach materials are usually epoxy-based adhesives that contain various types of fillers such as alumina, silver, or gold to provide control over the thermal and electrical characteristics of the attachment material. It is surprising to many that the attachment process, which is seemingly straightforward and uncomplicated, is actually the source of many difficulties in the operation of the hybrid device and consequently is the focus of much research.

Most often, the attachment method of choice is the use of inorganic materials for both the attachment of the chip to the substrate and the substrate to the package, because the inorganic attach materials are chemically simple, clean, and stable. The procedure is usually straightforward: simply heat to the attach's melting temperature, hold a few seconds to allow the material to flow, then cool. For relatively simple hybrid devices, this approach is quite adequate. As the device design becomes more complex, however, either through the need for a greater number of components per device or the need to use larger chips, problems begin to arise with the use of inorganic materials. Inorganic attach materials are mechanically stiff and, when used with large chips, can impress a significant amount of stress on the chip resulting from the difference in the coefficient of thermal expansion (CTE) between the chip and attachment material. This situation is also manifested when inorganic attach materials are used to attach the substrate to the package.

The fabrication of microelectronic hybrid devices is a multiple-step process that includes many sequences involving assembly, electrical testing, environmental stressing (time, temperature, and electrical power), and then electrical retesting. Because of the significant amount of work that must be expended to ensure the reliability of the devices, the overall cost of fabrication can be quite high. Consequently, if a problem is found during testing, the strategy is often to rework the device by removing the failed parts and replacing them with new parts, rather than discarding the device entirely. When a device fabricated with inorganic attach materials is reworked, it must be heated to the melting temperature of the attach material to remove the failed part. Consequently, the attachment material used on the other components may also melt, allowing those components to move and shift their alignment on the substrate, which may introduce other problems, in addition to problems associated with the device being repeatedly taken to rather elevated temperatures.

When conditions are such that the inorganic attach materials are inappropriate for a particular fabrication process, the usual alternative is to use organic-based adhesives, which are typically epoxies. One important advantage of the organic adhesives is that the mechanical stiffness of epoxies is considerably less than that of the inorganic materials. Consequently, when an epoxy is used to attach items with largely different CTE's, the thermally induced stresses that previously were applied to the chip or substrate and threatened its integrity are absorbed by the more pliable/elastic epoxy. Another advantage that the organic adhesives have over the inorganic attach materials is that a lower temperature is required to initiate the attachment process, typically 140°C to 160°C (cure temperature) versus 350°C to 370°C (melting temperature); thus, rework can be performed at a much lower temperature. The device is brought up to a softening temperature, usually close to the cure temperature; the defective component is removed; then a new component and fresh adhesive are applied. With organic adhesives, unlike inorganic attach materials, no movement of the other components on the substrate occurs.

These advantages are accompanied by some disadvantages, however. Organic adhesives are chemically complex systems and much less stable than the inorganic attach materials. The complexity arises because organic adhesives consist of several materials with different responsibilities and functions, and they react with one another as the material is cured. Two essential ingredients of the adhesive include the epoxy resin itself, which forms the backbone of the adhesive, and a curing agent, which is responsible for joining the epoxy resin subunits into a long extended ensemble. In all epoxy formulations, other materials are also present to modify the properties of the resin/curing agent mix. Generally, some type of filler is used; silver or gold can be added to make the epoxy mix electrically conductive, and an insulating filler such as alumina or silica can be added to provide better thermal conductivity and CTE. Other additives can include an accelerator, which is a compound designed to speed the action of the curing agent. An activator is sometimes present to activate the curing agent at the elevated curing temperature, so a less active curing agent can be used, allowing for a longer shelf life. Coupling agents, which are small bifunctional molecules that provide a chemical bridge between the epoxy, the filler, and the attachment surface, are usually present. Finally, solvents or reactive diluents are added to adjust the viscosity of the mixture.

During the cure process, many simultaneous and sequential chemical reactions occur at different rates that depend on how fast the adhesive was brought to its cure temperature, the cure temperature itself, and the viscosity of the mix, which varies throughout the curing process. In addition, some secondary parameters can modify the effects of the primary parameters, such as the way the adhesive was deposited. For example, a thin film of epoxy sandwiched between two big surfaces will cure differently than a blob of epoxy at the edge of a chip (known as a fillet). The variations in these systems are obviously quite great. Given the large number of constituents in the adhesive mix and the number of different reaction path-

ways possible (intentional and unintentional), it is not surprising that at the end of cure, a single, well-defined, homogeneous material is not produced. Rather, what one obtains is a heterogeneous collection of amorphous polymerized resin, randomly cross-linked by the curing agent, with voids containing trapped solvents or diluents, unreacted or partially reacted curing agents, accelerators, coupling agents, and condensation/degradation products. Also, the polymerized resin is not entirely inert, as it too can still possess reactive or partially reacted epoxy groups, hydroxyl groups, and other groups, which may undergo rearrangements over time to produce other by-products.

One of the more serious problems encountered with the use of epoxy adhesives is the release of materials from the bulk, usually occurring during the curing process, but also possible after cure. Material can escape from the bulk as a gaseous product, a process known as outgassing, or it can escape by diffusion along surfaces, a process known as bleed. Whether released by outgassing or bleed, the material will often find its way to other areas of the hybrid and may cause problems. Typical problems include wire-bond failures, where the impurity causes the metal-to-metal interface of the wire bond to be weak or more susceptible to other failure mechanisms.¹ The deposition of polar chemical species onto and in between metal lines under power has been implicated in electromigration processes and subsequent electrical short formation.^{2,3} Outgassing or bleed impurities have also been implicated in problems where conductive epoxies have developed electrically insulating interfaces.⁴ Although these and other problems have often been attributed to epoxy adhesives, they are still being used, primarily because no good alternative exists. Since the Laboratory uses epoxy adhesives in many high-reliability applications, the Materials Science Group, in collaboration with the Microelectronics Group, has been investigating their properties. In this article, we will limit the discussion to our work in the area of outgassing studies of epoxy systems.

EXPERIMENTAL INVESTIGATIONS

Our early work on epoxy outgassing mainly involved commercial formulations that were state-of-the-art products being used in hybrid manufacturing.⁵ It quickly became clear that the commercial formulations were very complex and difficult systems to study. Not only did the formulations contain several ingredients—many of which were proprietary and, therefore, frequently difficult to identify—but we also discovered that the principal components of the formulation (resin, curing agent, solvents, etc.) appeared to be contaminated with many impurities. Some of the impurities were tentatively identified as solvents used in the synthesis of the principal components. Other impurities were chemically similar to one or more of the principal components and may have been by-products of their synthesis. Presumably, they were not removed during any subsequent purification steps and consequently were carried along when the formulation was mixed. Alternatively, the chemically similar impurities may have arisen from some degradation of the epoxy

formulation, either during storage, the cure process, or after cure. The distinction between these two alternatives is obviously important in terms of understanding the origin of a material that outgasses from an epoxy adhesive. If the impurities are a consequence of the initial synthesis, then the responsibility lies with the manufacturer to clean the product. If the impurities arise through some degradation mechanism, then the user must understand the generic mechanism and how it might be eliminated or minimized, such as by control of the adhesive's exposure to incompatible materials and/or deleterious time and temperature profiles, either before, during, or after cure throughout its service life.

To achieve more control over the material, we collaborated with a commercial epoxy adhesive manufacturer (Hysol Corp.) to investigate a model epoxy system that was similar to current commercial formulations. This model system was selected to gain a better understanding of outgassing from microelectronic adhesives; it was not intended to have all of the chemical and physical properties necessary for adhesion or shear strength. Its primary advantage was that a detailed investigation of outgassing could be conducted since all of the components were known and could be analyzed individually. The model system included five components: the epoxy resin, an imidazole curing agent, silver flake filler, an epoxy silane coupling agent, and a reactive diluent. The reactive diluent we examined was 1,4-butanediol diglycidyl ether (BDE). It is reactive in the sense that it contains the same epoxy groups as the base resin (the glycidyl ether group is alternatively known as an epoxy group in the sometimes arcane world of organic chemistry nomenclature). Consequently, it not only provides the necessary solvent function of viscosity modification, but, by virtue of the epoxy groups, it can enter into the various reactions that the resin undergoes to develop the multimolecular three-dimensional structure that typifies organic polymers of this type. The purpose of providing the diluent molecule with this reactive ability is to offer a mechanism by which any trapped diluent contained within the cured polymer matrix can be chemically bound to the final product, thereby eliminating or reducing the often serious problem of diluent or solvent material outgassing from the cured epoxy.

Analytical Techniques

The investigation of the model system involved the use of several analytical techniques, including differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), thermal gravimetric analysis (TGA), and gas chromatography coupled with a mass spectrometer detector (GC/MS). The DSC technique measures the heat flow into or out of a sample as a function of temperature. Consequently, it is useful in determining the temperature at which an endo- or exothermic reaction is taking place, as well as in providing information about the kinetics of that reaction. In our study, DSC was used to determine the temperature and time at which the model system should be cured. As mentioned earlier, when an epoxy is cured, the final state of the cured material is not a uniquely defined state, but rather is strongly dependent on the

conditions, such as solvent concentrations and temperature profiles, that are used to achieve the cured state. The DSC technique was also used to examine the thermodynamics of the cure reactions under a variety of curing conditions.

Once a material has cured, the material's glass transition temperature (T_g) is often measured. The T_g is similar in some respects to the melting point of a crystalline material, in that it indicates what temperature is required to overcome the intermolecular forces (primarily van der Waals) between adjacent nonbonded resin segments in the three-dimensional structure, allowing them to move relative to one another. Since the T_g depends partially on how the neighboring resin segments interact with one another, it therefore depends on how the segments were initially constructed in the curing process. Consequently, the T_g is used as an indicator of how the material was cured. Another characteristic of the T_g is that it corresponds to the temperature at which one can expect to observe significantly higher levels of outgassing from the material, because the mobility of trapped volatiles is enhanced for $T > T_g$, allowing them to escape at a significantly higher rate. Typically, in actual applications, the T_g is engineered to be higher than the device's operating temperature. Many techniques can be used to measure the T_g . We used TMA, which measures a material's CTE. At the T_g , because of increased internal movement, the CTE typically increases by a factor of 4 or more. This effect makes the TMA a very convenient and straightforward way of measuring the T_g .

When volatile material is released by an epoxy, the loss is manifested in the reduced mass of the remaining material, which can be monitored with a suitably sensitive balance. When mass loss is recorded as a function of time and temperature, the technique is known as TGA. This method is quite handy in determining not only how much material is released by an epoxy, but also at what temperature and at what time. This information allows one to speculate about the identity of the material; that is, is it a solvent, is it occurring at the cure or after cure, or is it a degradation product?

To provide more information on the identity of volatile materials, the GC/MS technique is employed. In this method, a sample of the volatile material is passed through a chromatographic column that causes the various components of the sample to be separated in time. The separated components are then sequentially detected by the mass spectrometer, which provides information that often leads to the identification of the compound. Sometimes, the precise identification may not be achievable, but one can often infer that the compound belongs to a particular family of compounds, or possibly that it contains a characteristic chemical group. In either circumstance, the GC/MS provides a great deal of valuable information about what is coming out of an epoxy adhesive and under what conditions.

Results

An example of how these techniques are employed will be presented using the model system described previously: resin, curing agent, coupling agent, silver flake filler,

and the reactive diluent BDE, in a ratio of 18.5:4.0:0.25:70.0:7.25, respectively. For the sake of brevity, we will present part of a data set in an attempt to highlight the problem and point out the utility of some of the techniques mentioned earlier.

In the study,⁶ all five components were examined individually with TGA and GC/MS. Once the characteristics of the components were determined, epoxy formulations of different complexity were similarly examined, starting with the simplest model system containing just the epoxy resin and curing agent. This binary formulation was then followed by the ternary formulation where the BDE reactive diluent was added to the binary mixture. Finally, the five-component formulation (resin, curing agent, reactive diluent, silver flake filler, and coupling agent) was examined.

The three different epoxy formulations (binary, ternary, and five-component) were all subjected to the same thermal and temporal treatment to simulate the processes that the material would undergo in an actual device fabrication procedure. The individual formulations were placed in a sealed, stainless steel, temperature-controlled chamber, to collect the outgassing materials released during the various processing stages of cure, pre-seal bake, and burn-in. The curing step was initiated by bringing the chamber temperature to 160°C and maintaining it at that temperature for two hours. At the end of this period, the gas in the chamber was sampled by the GC/MS, yielding the identification of the outgassing products resulting from cure. The chamber was then evacuated and lowered to 125°C for twenty-four hours, simulating the standard pre-seal bake. At the end of this period, the gas was again sampled and measured. After evacuating the chamber, the time/temperature sequence was repeated again, simulating the burn-in process.

The intent of this experiment was to determine the identity and the amounts of material released by the sequential addition of the different ingredients. In this way, the increased or decreased presence of a particular volatile compound could be associated with just one of the ingredients and thus enable a determination of the probable mechanism or cause of the concentration change.

In Figure 1, we show the TGA analysis of the individual (uncured) epoxy resin. In this experiment, the temperature of the sample was increased from 50°C to 160°C at 20°C/min, and then maintained at 160°C for the remaining time. The uncured resin exhibited a nearly linear decrease in mass with time, indicating either a very slow diffusion of volatile impurities out of the bulk material, or a general degradation of the bulk material over that period of time. The belief is that the loss of mass was primarily due to the latter. In a similar experiment, the curing agent lost about 14% of its mass over the same period. The reactive diluent BDE and the epoxy silane coupling agent lost 100% of their masses within moments of reaching 160°C, because they are both very volatile materials. The silver flake, on the other hand, lost a minimal 0.15% of its mass in the same experiment.

When the epoxy resin and curing agent were mixed, the TGA of this combination, presented in Figure 2, showed that a 2.3% mass loss occurred almost immedi-

ately after the sample reached 160°C. This mass loss was significantly higher, by a factor of 4, than that expected from the individual components over the same short period of time. This result suggests that a fair amount of material, about 1.75% of the total epoxy mass, was released to the environs as a consequence of the cure reaction(s). After cure, although not apparent from Figure 2, a much smaller mass loss of 0.05% occurred. It is unclear at this point whether this additional loss was due to either degradation or simply continued, but much slower, diffusion of the volatiles formed initially during the cure. When the same experiment was performed with the ternary formulation (resin/curing agent/reactive diluent), the results were essentially the same in that initially a rapid decrease in mass of 3.25% occurred, followed by a very slow decline over the remaining period. In the ternary formulation, however, the volatile reactive diluent was about 25% of the formulation's mass. Thus, the small

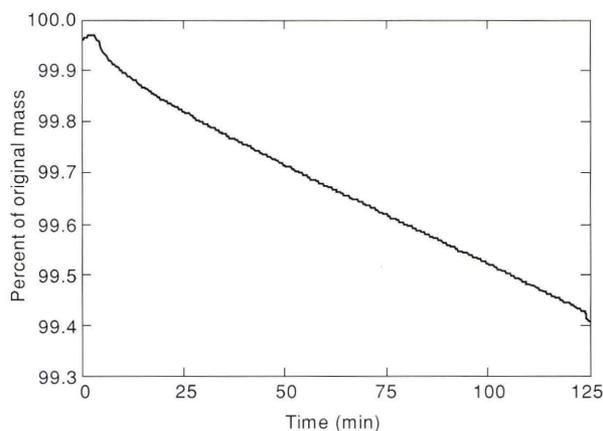


Figure 1. Thermal gravimetric analysis of the epoxy (bisphenol-F) resin measured with a temperature profile; the sample was held at 50°C for 3 min, heated to 160°C at 20°C/min, and then maintained at 160°C for the remaining time.

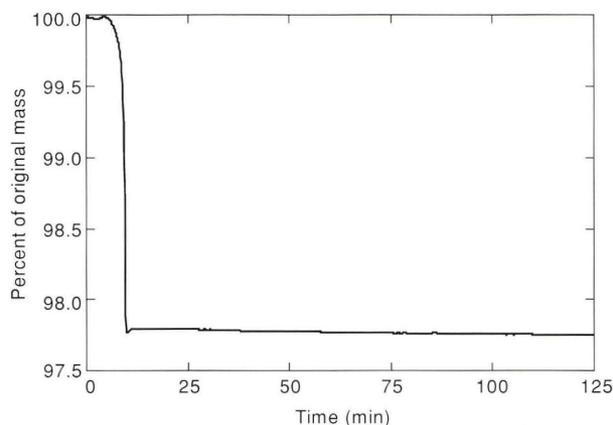


Figure 2. Thermal gravimetric analysis of the binary (resin/curing agent) mixture measured with a temperature profile; the sample was held at 50°C for 3 min, heated to 160°C at 20°C/min, and then maintained at 160°C for the remaining time.

3.25% decrease indicated that a majority of the BDE reactive diluent remained in the epoxy, as it was designed to do. Similar experiments were performed with a simple nonreactive solvent, in which nearly 100% of the solvent was eventually released from the epoxy over twenty-four hours.⁷

When silver was added, the situation changed even more dramatically. The TGA of the five-component formulation is shown in Figure 3. Here, the mass loss was still abrupt, but not nearly as abrupt as for the binary or ternary samples, suggesting that mass loss continued for some time after the cure temperature was obtained. Also, the magnitude of the mass loss was much higher, indicating that the silver had a pronounced effect on the production of volatiles at cure. This effect was recently investigated in more detail, where the nature of the ambient gas present during the cure process was shown to play an additional role in the magnitude of mass loss.⁸

These are examples of how the TGA technique is used to determine how much material is lost and when the loss occurs. This information is needed to try to piece together the puzzle of what different mechanisms and materials are responsible for the production of volatiles. Now, the volatiles must be identified.

The GC/MS technique was used for identification. As was done in the TGA work, the characteristics of the individual components were examined. In Figure 4, the GC/MS of the imidazole curing agent shows the type of information obtained from GC/MS, as well as the typical level of impurities observed in many of the ingredients used in these formulations. This plot represents the time-dependent signal obtained from the mass spectrometer detector that monitored the gaseous components released from the GC column. At time $t = 0$, the volatile materials that outgassed from the sample, in this example the curing agent, were collected and loaded onto the column, and then passed through the column along with an inert carrier gas. The time it took for a particular material to

exit the column depended on how strongly the material interacted with the column's interior coating; the stronger the interaction, the longer it took to pass through the column. The signal level in Figure 4 represents the total number of ions that the MS detected per unit time. The peaks simply indicate that something eluted from the column. Identifying the material required an examination of the mass spectrum corresponding to the individual peaks, as was done for all the peaks in Figure 4. Of the eight major peaks, we were able to identify six. Obviously, one would like to identify everything if possible, but even if a peak (compound) is not identified, the information is still useful later because the peak's time and mass spectrum, although not interpretable, are unique enough to associate it with a particular starting ingredient.

Figure 5 shows the GC/MS of the reactive diluent BDE, where the largest peak is that of BDE itself (peak 3). This is an example of a moderately clean starting material. To see the difficulty in identifying the sources and mechanisms of the various volatile materials, one can compare Figure 5 with Figure 6, which is the GC/MS of the same material, BDE, but which was held at 125°C to 160°C for seventy-two hours, under conditions similar to the combined cure, pre-seal bake, and burn-in processes. After such treatment, the BDE contained a host of species (labeled "x") that were identified as being related to BDE, as well as some other unidentified species. Here is an example of a compound reacting entirely with itself, producing a collection of other compounds that can then react in the formulation with all the other compounds present. Clearly, the number of potential products can quickly become intractable.

A GC/MS of the ternary formulation at the end of a two-hour cure at 160°C is shown in Figure 7; most of the peaks were identified. Of these, only nitrogen, oxygen, carbon dioxide, water, and acrylonitrile were original ingredients. All others are new and a consequence of the cure. During the pre-seal bake and burn-in periods, the

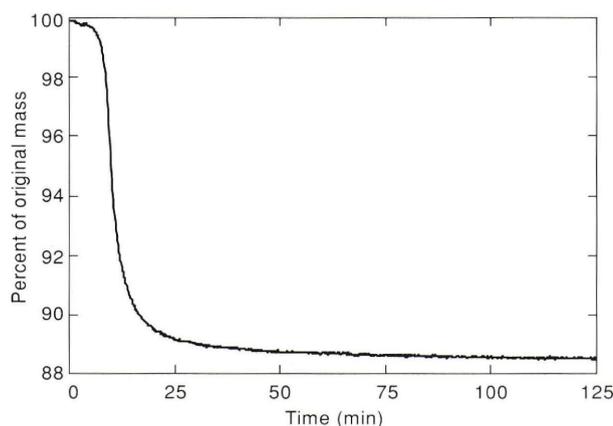


Figure 3. Thermal gravimetric analysis of the five-component model adhesive formulation measured with a temperature profile; the sample was held at 50°C for 3 min, heated to 160°C at 20°C/min, and then maintained at 160°C for the remaining time. The percent of original mass refers to the organic (epoxy resin, curing agent, and reactive diluent) fraction of the formulation, so the mass loss can be made comparable to the previous figures.

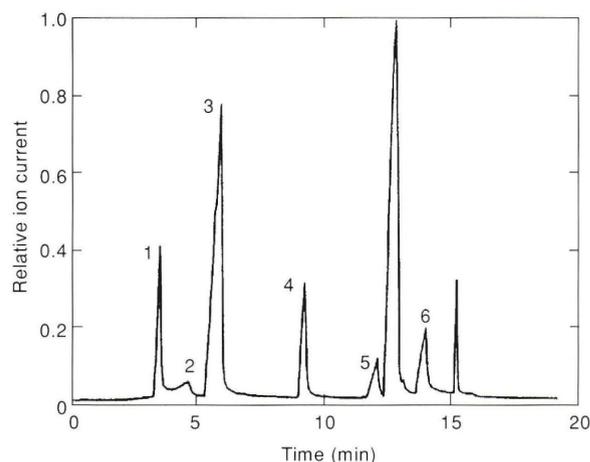


Figure 4. Gas chromatography/mass spectrometry total ion chromatogram (HP-1 column) of species outgassed from the imidazole curing agent (Curezol 2PHZ-CN) at 80°C. 1 = acrylonitrile, 2 = water, 3 = *tert*-butyl alcohol, 4 = 3-methoxypropanenitrile, 5 = 3-hydroxypropanenitrile, 6 = 3-hexenedinitrile.

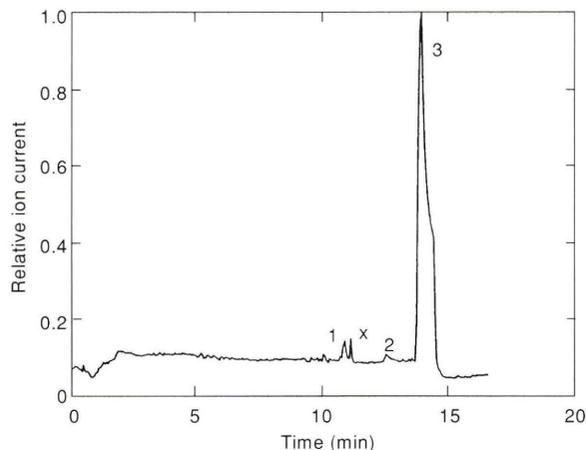


Figure 5. Gas chromatography/mass spectrometry total ion chromatogram (HP-1 column) of species resulting from an injection of 1,4-butanediol diglycidyl ether (BDE). 1 = butoxymethyl oxirane, 2 = water (from the system), 3 = BDE, x = related to glycidyl ether moiety.

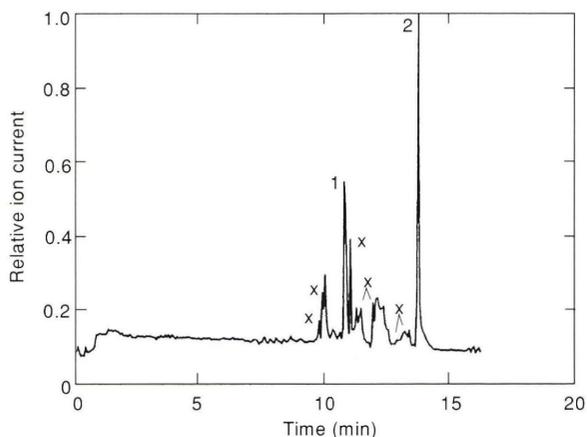


Figure 6. Gas chromatography/mass spectrometry total ion chromatogram (HP-1 column) of species outgassed from 1,4-butanediol diglycidyl ether (BDE) after extended heating (see text). 1 = butoxymethyl oxirane, 2 = BDE, x = related to BDE.

same general collection of volatile compounds was observed with some variations in their relative intensities. The binary formulation was not quite as complicated as the ternary, so the differences can be attributed to the reactive diluent BDE.

The results obtained from the five-component formulation cure, pre-seal bake, and burn-in processes were somewhat similar to the ternary, but they showed some very interesting differences. When silver was present (Fig. 8), numerous alcohols and aldehydes were also present, which appeared to have their origin in the nitriles found in the ternary formulation. It is tempting to suggest that the silver, possibly in concert with the water, affected either the reactions that formed the nitriles initially or, alternatively, the transformation of the nitriles to their reduced oxygen analogs. Either way, the potential effect involving the commonly used silver filler material is quite surprising and at the same time unsettling.

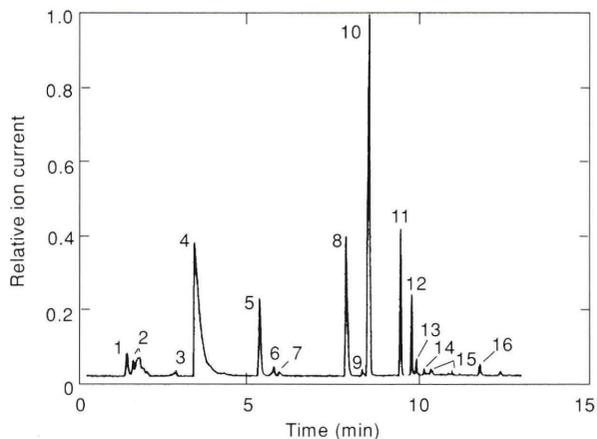


Figure 7. Gas chromatography/mass spectrometry total ion chromatogram (Chrompack column) of species outgassed from the ternary mixture at the end of a two-hour cure at 160°C. 1 = air (nitrogen, oxygen, and carbon dioxide), 2 = carbon dioxide, 3 = propene, 4 = water, 5 = butene, 6 = butadiene, 7 = methanol, 8 = acetonitrile, 9 = acetone, 10 = acrylonitrile, 11 = propanenitrile, 12 = butenenitrile, 13 = tetrahydrofuran, 14 = benzene, 15 = various nitriles, 16 = toluene.

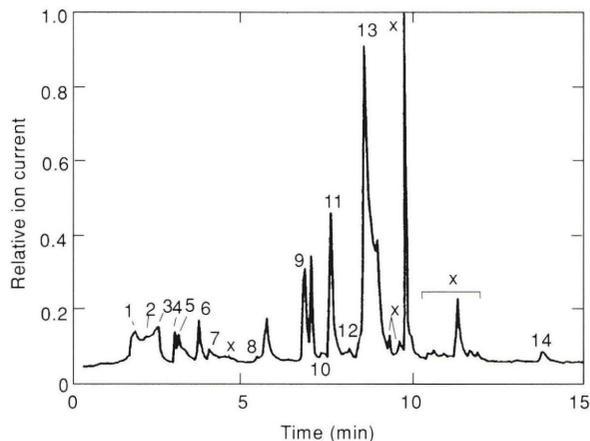


Figure 8. Gas chromatography/mass spectrometry total ion chromatogram (HP-1 column) of species outgassed from the five-component model adhesive formulation at the end of the burn-in period at 125°C. 1 = water and propanal, 2 = acetone, 3 = acrylonitrile, 4 = butenal, 5 = propanenitrile and butenenitrile, 6 = tetrahydrofuran, 7 = benzene, 8 = propanal, 9 = γ -butyrolactone, 10 = benzaldehyde, 11 = benzonitrile, 12 = 1,4-butanediol and phenol, 13 = hexanedinitrile and methyl benzonitrile, 14 = related to methylene 1,1'-bisphenol, x = related to 1,4-butanediol diglycidyl ether.

CONCLUSION

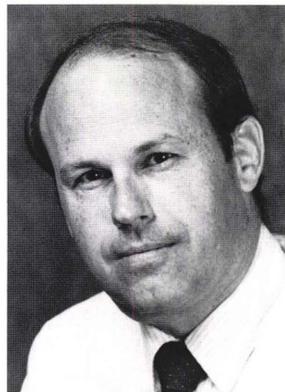
Because of the considerable advantage afforded by epoxy adhesives, they will continue to be used in the fabrication of hybrid microelectronic devices for the foreseeable future, despite the problems associated with them. This article highlighted some of the techniques used to investigate outgassing from epoxy adhesives, which is the most serious problem associated with their use. Results from our studies and similar studies of the outgassing problem will enhance our understanding of the chemical processes that occur throughout the life of

an epoxy adhesive. With this knowledge, the chemistry of the adhesive formulation can be improved to minimize its problems and thereby improve the reliability of electronic devices fabricated with epoxy adhesives.

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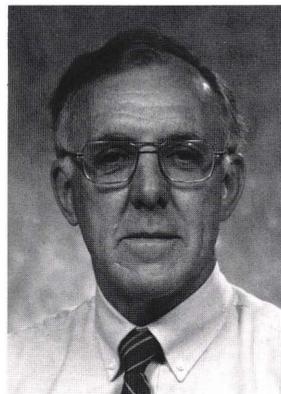
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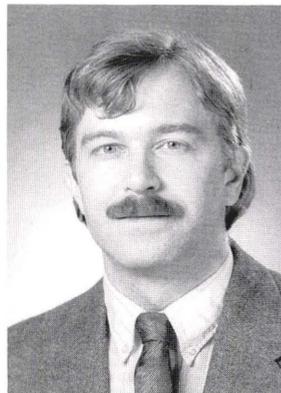


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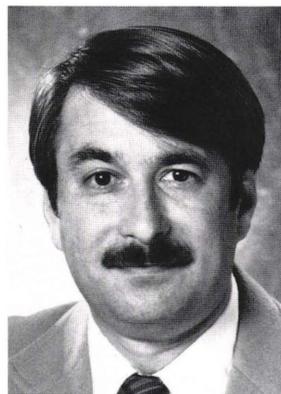
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