

EPOXY ADHESIVE

# TEST MEASUREMENT GUIDE



## ABOUT THIS GUIDE

This guide is an educational tool designed to aid adhesive users in gaining a more thorough understanding of adhesive properties and testing methods. It was compiled through the combined efforts of multiple departments at Epoxy Technology, including Quality Control, Research & Development, and Technical Service.

Although we have based our information on the most up-to-date data, advances in test methods and materials are constantly evolving. Please use this reference booklet as a guide only, and follow any recommendations listed on the data sheets, along with any technical information that may be included with your adhesive product shipment.

We hope that the information in this guide will be helpful and valuable to you in choosing the best adhesive for your specific application.

## ABOUT EPOXY TECHNOLOGY

Epoxy Technology, A Meridian Adhesives Group Company, offers an extensive line of specialty adhesives including Electrically Conductive, Thermally Conductive, Optical Grade Adhesives, UV Epoxy and UV Hybrid Adhesives.

All of our products are tested thoroughly and consistently in our state-of-the-art laboratories to ensure product reliability. Epoxy Technology is very proud of its recognized quality program, including comprehensive ISO 9001 and MIL-STD 883/5011 certifications, as well as RoHS Compliance and Green Partnerships.

Our EPO-TEK® brand adhesives have been recognized worldwide for quality and reliability since 1966.



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## 1.0 Viscosity/Rheology

Viscosity is a term and value used to describe the internal friction of a fluid. The degree of friction is observed when a force is applied to the material. The more friction detected, the more force required to move the material or to "shear" it. Shearing occurs when the fluid is actually moved. A low viscosity product like water requires less force to flow than a high viscosity material, like peanut butter. The force required to shear a material can be measured and reported using a Brookfield® Viscometer. One type is a cone and plate viscometer shown in figure 1.

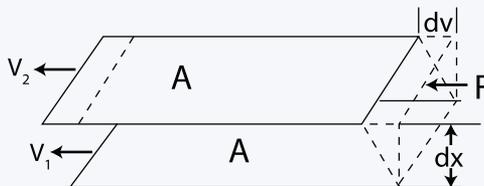
The cone and plate viscometer precisely measures the torque established by the resistance in a fluid, sandwiched between a rotating cone and stationary plate. As torque is directly proportional to the shear stress in the material, it can easily be converted to absolute centipoise (cPs) units through known geometric constants of the cone, the rate of rotation, and the stress related torque (or from pre-calculated range charts). The plate portion of the assembly is the sample cup (shown in figure 1) which holds the fluid and remains stationary. It is jacketed so that a constant temperature is maintained throughout the test. The cup is locked onto the viscometer so that the material comes into contact with the cone.

figure 1



Newton defined viscosity by the model in figure 2 below. Two parallel planes of fluid of equal area "A" are separated by a distance "dx", moving in the same direction, but at different velocities ( $V_1$  and  $V_2$ ). He assumed that the force to maintain this difference in speed was proportional to the difference in speed throughout the liquid. Also called the velocity gradient. To express this, Newton said that " $\eta$ " is a constant for a given material and is called its "viscosity." The velocity gradient,  $dv/dx$ , is a measure of the change in speed at which the layers move with respect to one another. This describes the shearing a liquid goes through and is referred to as the "shear rate (S)" which is reported in "reciprocal second" (sec<sup>-1</sup>). The term  $F/A$  indicates the force per unit area required to produce the shearing action. It is referred to as "shear stress" and will be symbolized by "F". Its unit of measurement is "dynes per square centimeter" (dynes/cm<sup>2</sup>).

figure 2

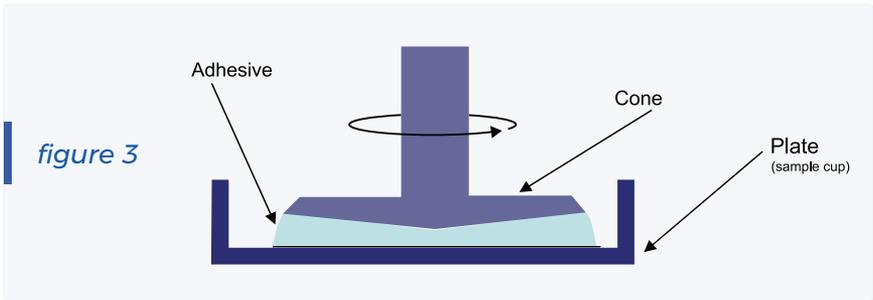


Using these simplified terms, viscosity may be defined mathematically by this formula:

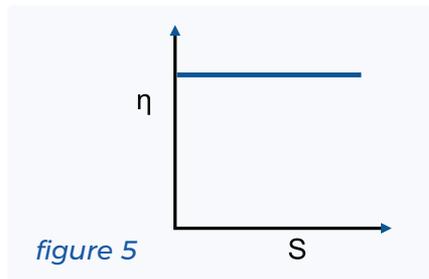
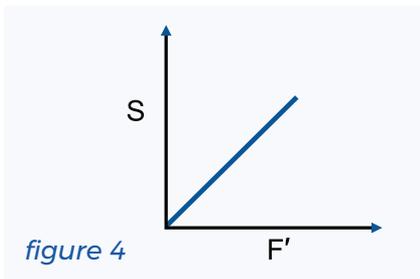
$$\eta = \text{Viscosity} = F/S = \text{shear stress/shear rate}$$

ASTM Test Method D2196, "*Standard Test Method for High-Shear Viscosity Using a Cone and Plate Viscometer*", is based on the above definition and is followed for viscosity measurements.

The fundamental unit of viscosity is the "poise." If a material requires a shear stress of one dyne per square centimeter to produce a shear rate of one reciprocal second, it would produce a viscosity of one poise, or 100 centipoise. Viscosity can also be expressed in "milli-Pascal-seconds" (mPa·s). One milli-Pascal-second is equal to one centipoise. Newton assumed that all materials have, at a given temperature, a viscosity that is independent of the shear rate. In other words, twice the force would move the fluid twice as fast. This holds true for Newtonian fluids only. The following is a schematic (figure 3) of a cone and plate viscometer in action:

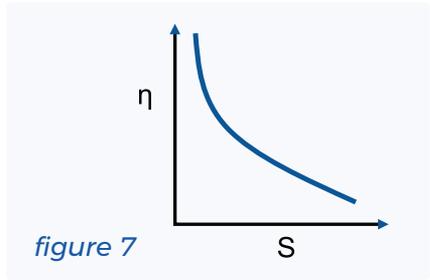
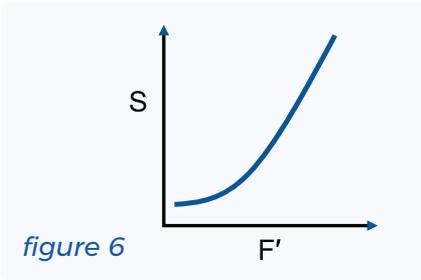


The type of flow described above is true for Newtonian fluids only. The following graphs show how this type of product behaves. Figure 4 shows the straight line relationship between shear rate ( $F'$ ) and shear stress ( $S$ ). Figure 5 shows how the viscosity remains constant (assuming temperature is held constant) with varying shear rates.

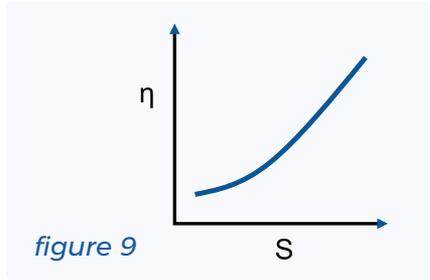
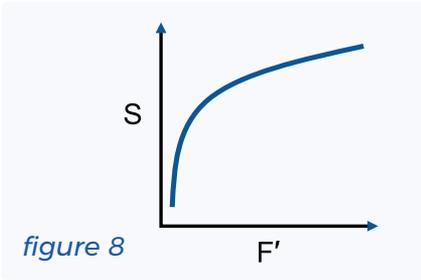


But not all fluids are Newtonian in their flow characteristics. In fact, there are several types of flow behavior. These products are classified as Non-Newtonian. There are three subgroups within the Non-Newtonian classification: Pseudoplastic, Dilatent and Plastic.

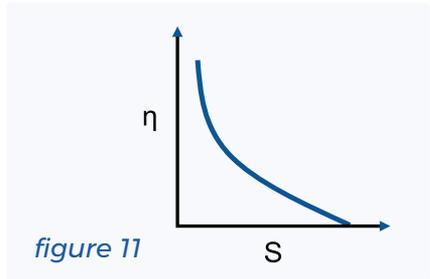
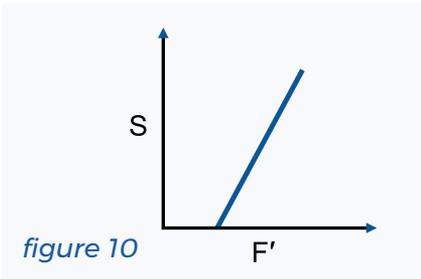
**PSEUDOPLASTIC** materials will decrease in viscosity with an increase in shear rate. This is sometimes referred to as shear thinning. Figures 6 & 7 depict this:



**A DILATENT** type of behavior as seen in figures 8 and 9 below, causes products to increase in viscosity with an increasing shear rate. This is also called shear thickening and is commonly seen with deflocculated solids such as clay.



The third and final type of flow is **PLASTIC**. When a material exhibits Plastic behavior similar to figures 10 and 11 below, it will remain in a "solid" state until a certain amount of force (yield value) is applied to it before it will flow. A great example of this is ketchup.



Epoxies can exhibit any or all of these flow behaviors which is important to consider when choosing an adhesive for a specific application. Thixotropy is another parameter that is related to viscosity and can be measured using a viscometer. A thixotropic fluid decreases in viscosity with time, while it is subjected to constant shearing as seen in figure 12. This type of behavior can occur in combination with any of the above flow types.

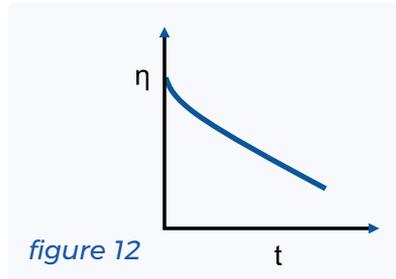


figure 12

When talking thixotropy, often we refer to Thixotropic Index (TI). This is a value reported by taking the ratio of two separate readings at different speeds on a viscometer. For example, if a material produces a reading of 15,000 cPs at 1 RPM and 10,000cPs at 10 RPM, the thixotropic index is equal to 15,000/10,000 or 1.5. Usually, TI is calculated from viscosities measured at RPMs that are a decade apart (i.e between 1 and 10 RPMs or between 10 and 100 RPMs). This number is also key in choosing the proper type of material for a particular dispensing technique.

All of the preceding terminology and definitions are also "rheological parameters". Rheology is defined as the science of the deformation and flow behavior of materials. Flow behavior is important in many industrial processes and the success or failure of an application is widely based on these properties for a specific adhesive. Coatings, molded plastics, adhesives, personal care items, cosmetics, inks, cement, solder pastes and medicines are examples of the range of commercially available products whose viability depends on having the proper rheology. For all of the above materials, the necessary rheological properties must be examined before processing and application.

The following graph (figure 13) shows how a user may want to balance viscosity and thixotropy in order to determine the best application method for a given adhesive:

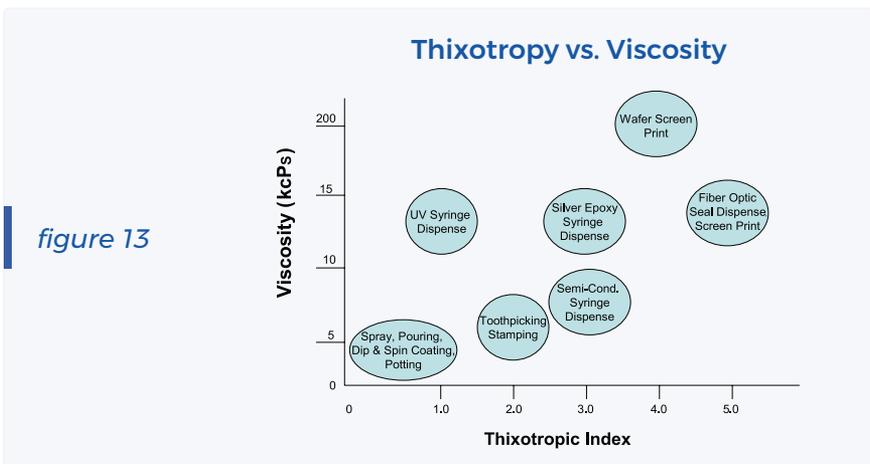


figure 13

## 2.0 Pot Life and Working Life

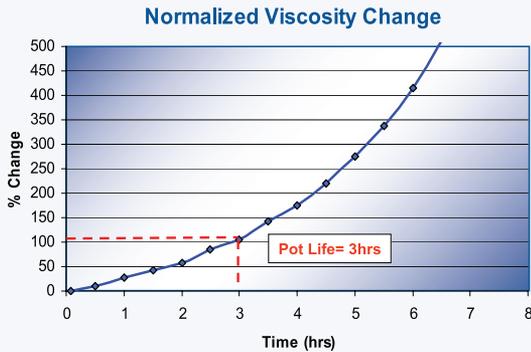
Pot life is a metric used to define the length of time that a material can be handled before its viscosity changes substantially. Generally, pot life is defined as the amount of time it takes for the initial viscosity to double. Figure 1 below shows the increase in viscosity over time for a typical low temperature cure epoxy system. You will notice that the viscosity builds slowly at first as the chemical reaction begins. However, the reaction itself gives off heat which, in turn, increases the rate of reaction. This process causes the viscosity to increase in an exponential manner.

figure 1



As shown in figure 2, this material has about 3 hours of pot life, i.e. it takes three hours before the viscosity doubles (100% change).

figure 2



Often, knowing the pot life alone is not sufficient to determine how long a given material can be used for a given manufacturing process. For example, figure 3 shows the normalized (% change) viscosity versus time for two different products. Both the blue and green products exhibit the same percent change in viscosity versus time. Since both formulations double their viscosity after 3 hours (shown as a 100% change), they both would have 3 hours of pot life.

However, as figure 4 shows, the two materials have very different starting viscosity values. The starting viscosity of the green material is ten times greater than that of the blue material. As a result, the magnitude of viscosity change over a given period of time for the green product is much higher than the blue product. In these cases, the process limits for a specific application should be used to define the working time for a given material in a given process.

figure 3

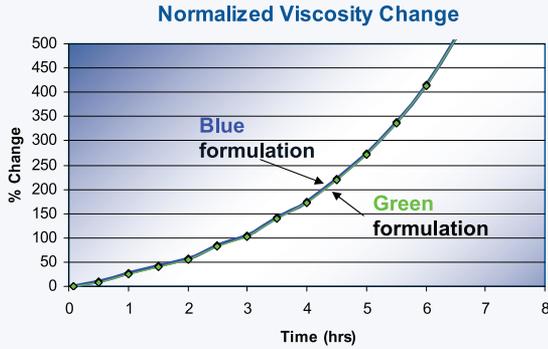
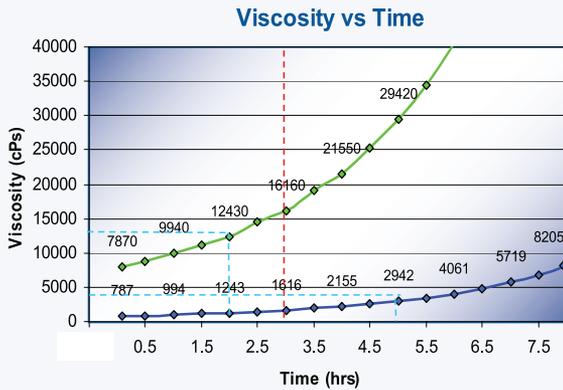


figure 4

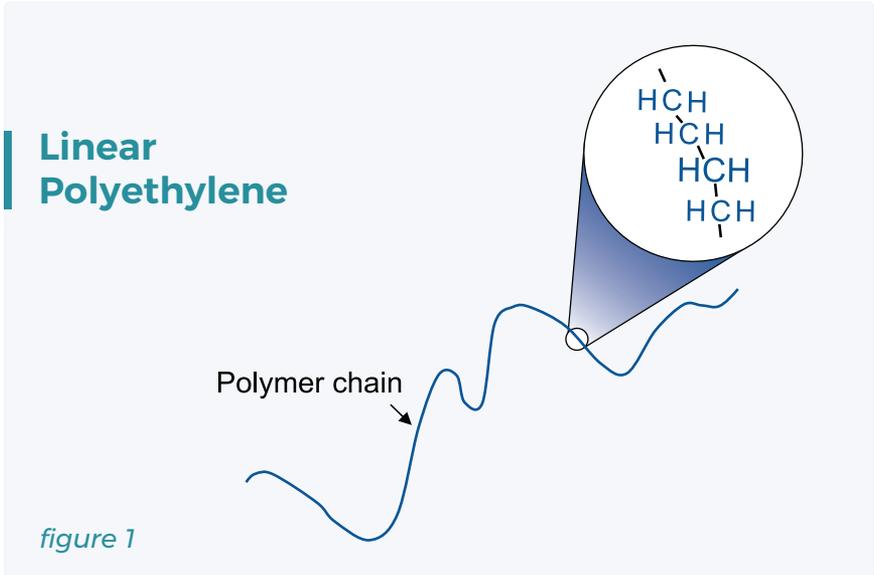


In the example above, customer A may be able to work with the blue material as long as the viscosity is lower than 3,000 cPs. This would give customer A, 5 hours of working life (2 hours longer than the pot life). On the other hand, customer B may only be able to work with the green material if the viscosity is less than 12,500 cPs. In this case, the material would have a working life of only 2 hours in customer B's process.

Thus, pot life is a data sheet value designed to give a first order description of the speed of viscosity increase over time. However, the actual working time of the product will be application specific and should be determined and defined based on the individual process.

## 3.0 Adhesive Curing

The word polymer comes from the Greek words poly (meaning many) and meros (meaning parts). As such, polymers are long molecule chains composed of many thousands of small units (monomers) joined end to end. An example of linear polyethylene, the simplest hydrocarbon polymer, is shown in figure 1:



Polymers fall into two main categories: thermoplastics and thermosets. Thermoplastic materials are generally composed of linear polymer chains with little or no branching or side groups similar to the thermoplastic polyethylene polymer in the diagram above. As a result, these chains are able to easily slide past each other when heated. In this way, thermoplastics can be heated and formed, then re-heated and re-formed repeatedly without damage.

On the other hand, thermosets undergo a chemical reaction when initially heated and cured to form a three-dimensional cross-linked network. Once these materials are cured, this structure is locked in place and the material cannot be reformed or reprocessed. An example of the chemical crosslink structure for a common type of thermosetting polymer, an epoxy, is shown in figure 2.

# Example of Thermoset Cross-Linking Reaction

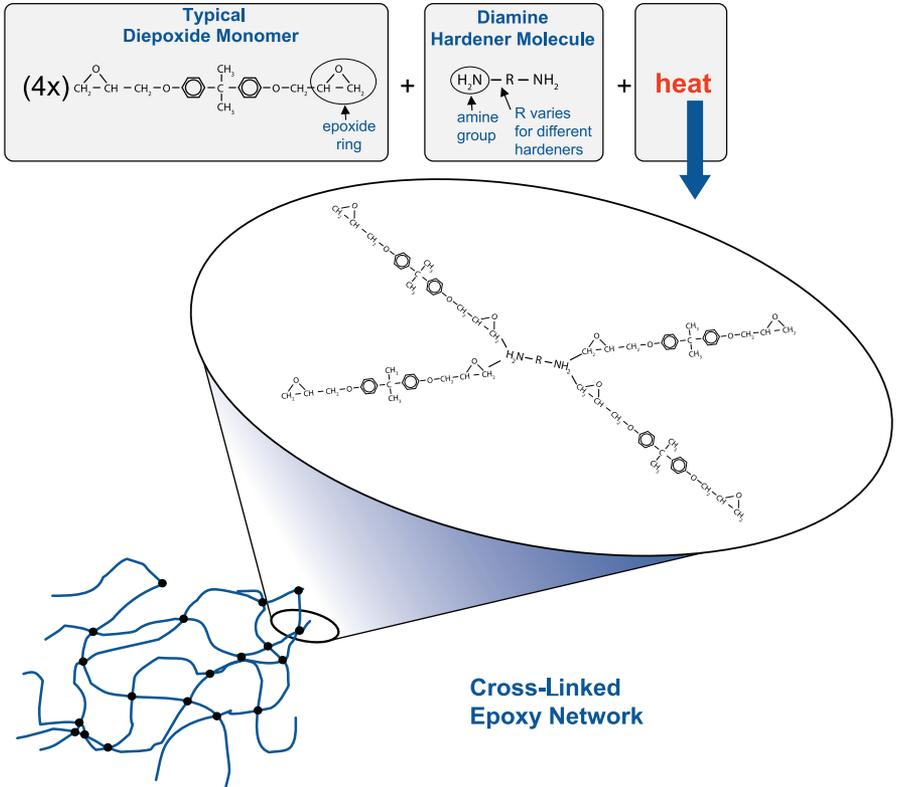


figure 2

Epoxies get their name from the three membered "epoxide" rings located on the starting epoxide monomer. In the case above, the starting monomer is actually a diepoxide, which contains two epoxide groups one at each end of the monomer. The hardener in the above system is a diamine, which contains a reactive amine group at either end of the molecule. Each of the epoxide rings can be opened by an active hydrogen on the end of the diamine to produce chemical bonds between the epoxide monomer and the hardener.

This particular diamine hardener provides four active hydrogens that are capable of reacting with four different epoxide monomers to chemically cross-link them together. Typically, the higher the functionality (number of reactive sites) of the hardener, the more tightly cross-linked the resulting cured thermoset will be. In general, highly cross-linked systems will have higher hardness and more thermal, chemical and moisture resistance.

## 3.1 Kinetics of Cure

The cure kinetics rate for thermosetting adhesives can be monitored using Differential Scanning Calorimetry (DSC). DSC measures the energy absorption of a sample as it is subjected to a specified thermal profile. The amount of heat contained in a material at any given time is known as its enthalpy. As additional thermal energy is supplied to the material, its enthalpy will increase. The level of the resulting temperature change in this material will be dictated by its specific heat. At a given temperature, specific heat is the amount of thermal energy needed to change the temperature of that material by one unit.

Figure 3 below shows the cross-section of a typical DSC sample holder. A small amount of uncured sample (~5-10mg of resin content) is placed in a small aluminum sample pan with lid. The sample pan is placed on top of an individual chromel heating disc. An identical empty aluminum pan is placed on a second chromel heating disc to serve as a reference for the experiment. Energy is supplied separately to the sample pan and the reference pan in order for each of them to exactly match the heating rate of a predetermined temperature profile. The rate of energy supplied to the sample to heat it at a controlled rate is proportional to the specific heat of the sample.

The specific heat of a material will change only slightly with temperature as long as that material remains in the same physical state. However, when the thermal profile imposed on a material causes a transition to a new state, (i.e. melting, decomposition, curing, etc.) this will cause a significant change in the specific heat. The rapid change in specific heat at a transition, then causes a change in the amount of energy needed to be delivered to the sample in order to maintain the desired heating or cooling profile.

### Standard DSC Cell\*

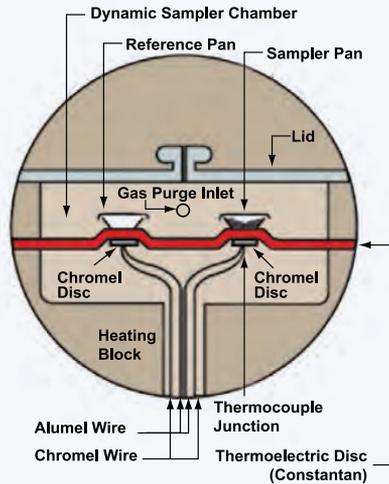
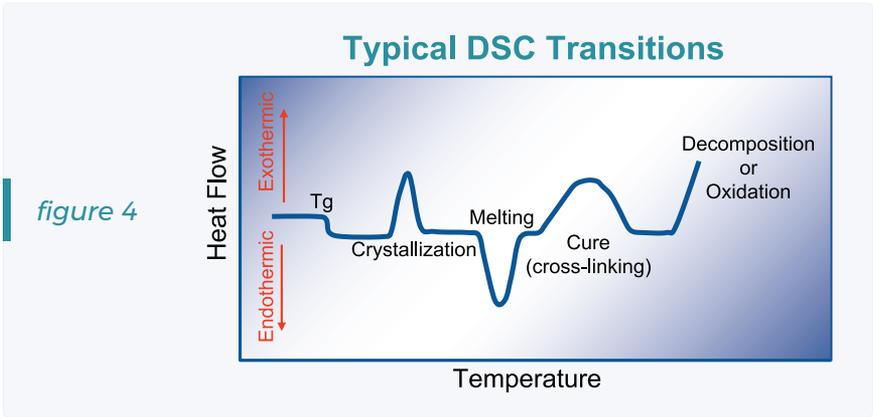


figure 3

\*Courtesy of TA Instruments

Changes in state that absorb energy are known as endothermic changes. Melting is an example of an endothermic state change. Heat is put into the system to allow molecules the mobility to begin to move. Eventually, enough heat is put into the system to cause overall flow of the melted material. At this point, the heat energy becomes converted to the kinetic energy of the liquid. On the other hand, changes in state that actually evolve energy are exothermic transitions. Heat of reaction (curing) and crystallization are examples of exothermic transitions. Figure 4 shows some typical endothermic and exothermic transitions as they would appear in a DSC scan.



As mentioned earlier in this section, DSC can be used to monitor the cure of a thermosetting material. This is accomplished by subjecting the uncured thermoset to a controlled temperature ramp and analyzing the location, size and shape of the resulting exothermic cure peak.

Figure 5 shows a DSC kinetic curing profile for a typical epoxy system. The peak in the sample's heat flow response is located above the baseline, representing an exothermic event. The peak of this curing exotherm is located at approximately 150 °C, indicating that the fastest cure for this product will occur at this temperature. The onset of the cure peak for this product is 140 °C.

However, this does not mean that the product cannot cure below 140 °C. As the enlarged portion of the kinetic curve reveals, there is actually a small amount of exotherm starting as low as 80 °C. This particular product will cure at 80 °C, but it will take around four hours. On the other hand, when this product is cured at 150 °C, the peak cure temperature, it will only take about 10 minutes.

## DSC Kinetic Cure

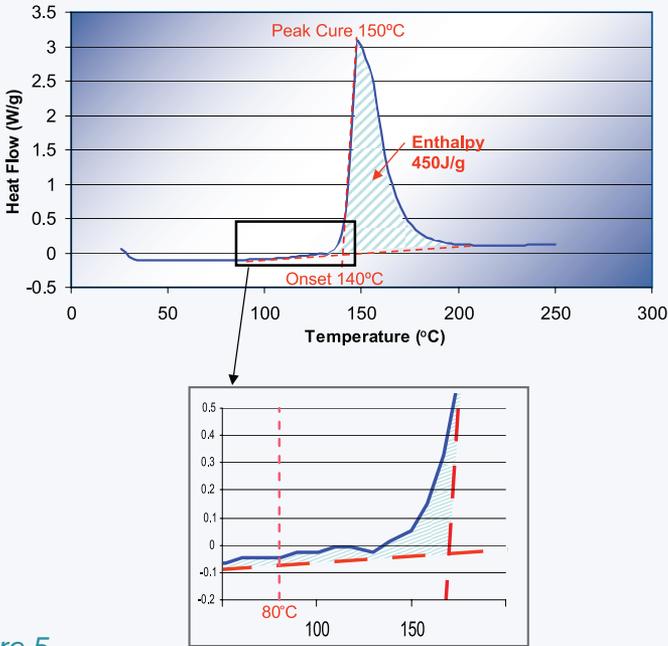


figure 5

The total change in enthalpy that the sample undergoes during cure can be calculated by integrating the area under the exotherm peak. For the material in the diagram above, the total heat of reaction is 450J/g. As specified in ASTM D3418, "Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry", this total heat of reaction can then be used to determine the extent of reaction of the material after it is cured according to a desired cure condition. The cured material is analyzed in the DSC using the same thermal profile used to measure the uncured material. Figure 6 shows the original exotherm of the uncured material above, along with that of the same material cured at 150 °C for 10 minutes.

## DSC to Calculate Residual Cure

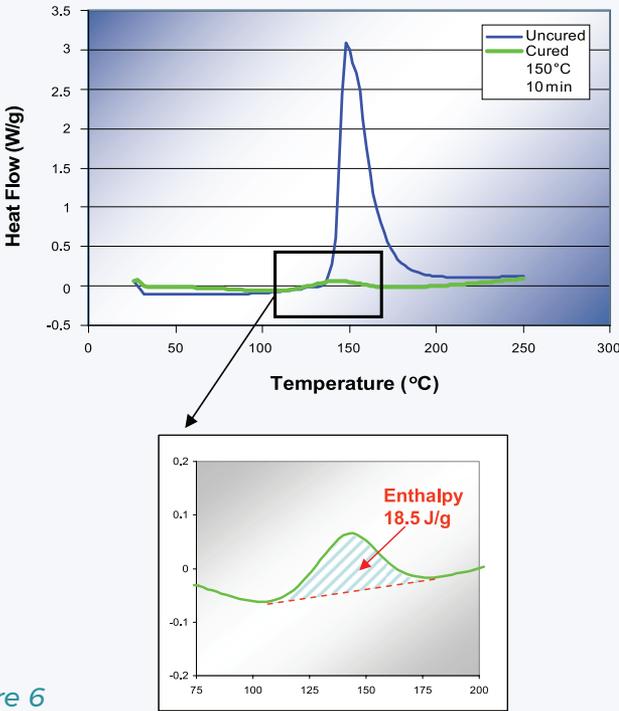
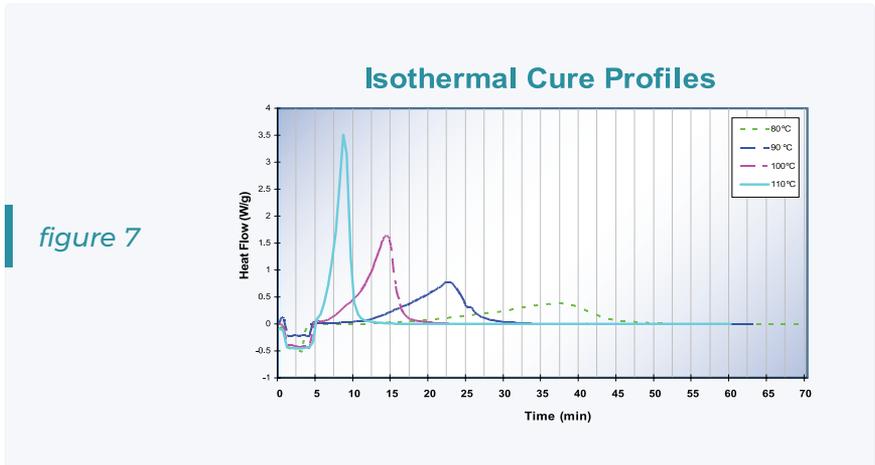


figure 6

The material cured at 150 °C for 10 minutes exhibits only a very small residual exotherm peak. This indicates that most of the theoretical cross-linking reactions were completed during the cure. The enlarged view of the cured sample exotherm shows that only 18.5 J/g of reactivity remains of the original 450 J/g heat of reaction for this material. Thus, only 4.1% of the material is left unreacted by the imposed cure. Epoxies do not need to achieve a full 100% reaction in order to perform well as adhesives. Generally, systems that have been cured to at least 90% conversion will exhibit mechanical and physical properties that do not differ significantly from their fully reacted state. In addition, even lower degrees of conversion may produce properties that are perfectly sufficient for a given application.

DSC can also be used to determine how long a particular material will need to be cured at a given temperature in order to reach the maximum degree of conversion at that temperature. This is achieved by measuring the exotherm curing peak of a material exposed to a constant temperature over time or an isothermal scan.

Figure 7 shows the different exotherm peaks for a typical epoxy cured at 80 °C, 90 °C, 100 °C and 110 °C. If the material below is cured at 80 °C, it requires close to an hour to reach the full conversion level at this temperature. However, if it is possible to increase the cure temperature to 110 °C, the material will need less than 15 minutes to reach final conversion.



One important note is that the faster a thermoset is cured, the more stress is imparted to it. As a result, it may not always be desirable to cure quickly at the highest temperatures. For some applications, it may be necessary to ramp the temperature to the desired cure condition, or start with a lower temperature precure to yield lower stress bond lines.

## 3.2 Glass Transition Temperature (T<sub>g</sub>)

As discussed in the beginning of this section, the chemical cross-links that form during the cure of thermosetting materials do not allow the cured material to melt or flow when re-heated. However, cross-linked materials do undergo a very slight softening at elevated temperatures at a point known as the Glass Transition Temperature (T<sub>g</sub>).

The T<sub>g</sub> is not a discrete thermodynamic transition, but is actually a temperature range over which the mobility of the polymer chains increases significantly and the bulk material changes from a rigid/glassy state to more of a rubbery/ compliant state. The temperature range over which this transition takes place is very dependent on the level of chemical cross-links and physical chain entanglements in the system, as well as the degree of flexibility of the polymer chains themselves.

Polymers with rigid backbone segments, bulky side groups and a high crosslink density will require very large amounts of thermal energy to provide the system with enough mobility to transition to a rubbery state. Such systems, like that shown in figure 8b below, will have a high Tg. On the other hand, systems like that shown in figure 8a, which have long sections of flexible polymer chains between cross-links, will shift to a rubbery state at significantly lower temperatures, resulting in a lower Tg.

Tg is an endothermic state change where heat energy must be supplied to the material in order to transition to a rubbery state. This transition can also be characterized using DSC in accordance with ASTM E1356, "Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry". Samples are prepared and tested in the same manner used for measuring curing kinetics, with the exception that the sample is already cured. The sample can either be cured outside the DSC using an application specific cure profile (ie. box oven, tunnel oven, heated tooling, etc.), or can be cured in the DSC during a previous kinetic or isothermal scan.

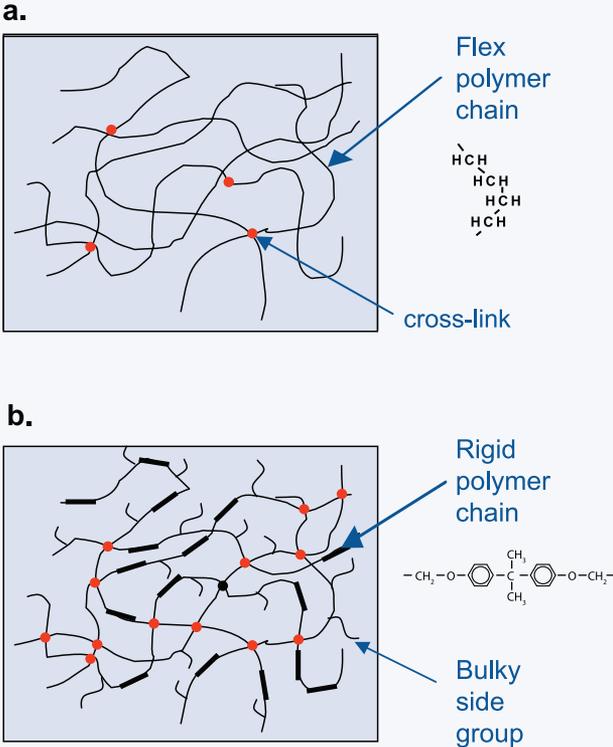
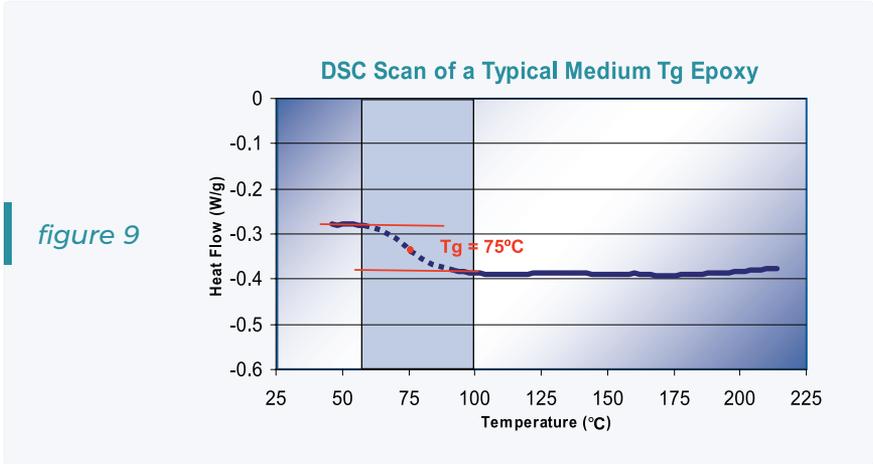


figure 8

The format of a Tg scan is very similar to that of the kinetic scan. Temperature is plotted on the X axis and the heat flow response on the Y axis. However, unlike the large exothermic peaks evolved during the kinetic cure, Tg is exhibited as a small endothermic step change in the heat flow. Figure 9 below shows a typical Tg for an epoxy. The Tg is actually a transition region, rather than a specific temperature. In this case, the transition begins around 60 °C and is complete by about 100 °C. However, the convention is to report a single temperature defined as midpoint of the transition region as it is bounded by tangents to the two flat portions of the heat flow curve. For this material, the Tg would be reported as 75 °C.



The reason that the Tg spans a temperature range rather than occurring at a specific temperature, is that the cross-linked polymer chains take time to increase their mobility in response to applied thermal energy. With just a little bit of applied energy, some of the more flexible chain segments and side groups can begin to move a little bit. This small-scale movement allows an increase in free volume that in turn allows the freedom for some of the larger and more rigid segments to begin to move once higher levels of energy are applied.

Thus, even at constant temperature, the transition to the rubber-like state will take some time to evolve. However, a material's Tg is measured by subjecting it to some sort of temperature ramp. The rate of this scan will play a large role in determining the temperature location of the Tg. Slower scans will allow more time for the polymer chains to orient in response to the applied energy and will result in a lower temperature measurement of the Tg.

On the other hand, a very quick scan will cause the temperature to increase more quickly than the chains are able to respond to the supplied energy, and the resulting Tg response region will be pushed to higher temperatures. Thus, it is very important to check the test conditions under which Tg has been reported. Ramp rates of 10 °C/min and 20 °C/min are typical testing conditions used in the industry. The 20 °C/min condition is frequently used as it cuts testing time in half. However, it will result in a higher reported value for Tg.

The Tg of a material will also depend on its degree of cure. Generally, the reported Tg for a material will be based on 100% cure/conversion. However, depending on the cure schedule used for a given application, a lower degree of conversion may be attained. This may have less cross-linking, and therefore, will result in a lower Tg.

A high Tg is not necessarily required for good performance of an adhesive. In many cases, it is actually desirable for a material to operate above its Tg. Since the system is chemically cross-linked, it will not degrade in performance above the Tg, it will just be in a softer state. Softer/tougher materials are often desirable for applications in which resistance to thermal or mechanical shock is required.

High Tg materials are generally desirable for applications which require low movement in a given temperature range or very high temperature resistance.

Figure 10 shows Tg scans for some typical low, medium and high Tg epoxies.

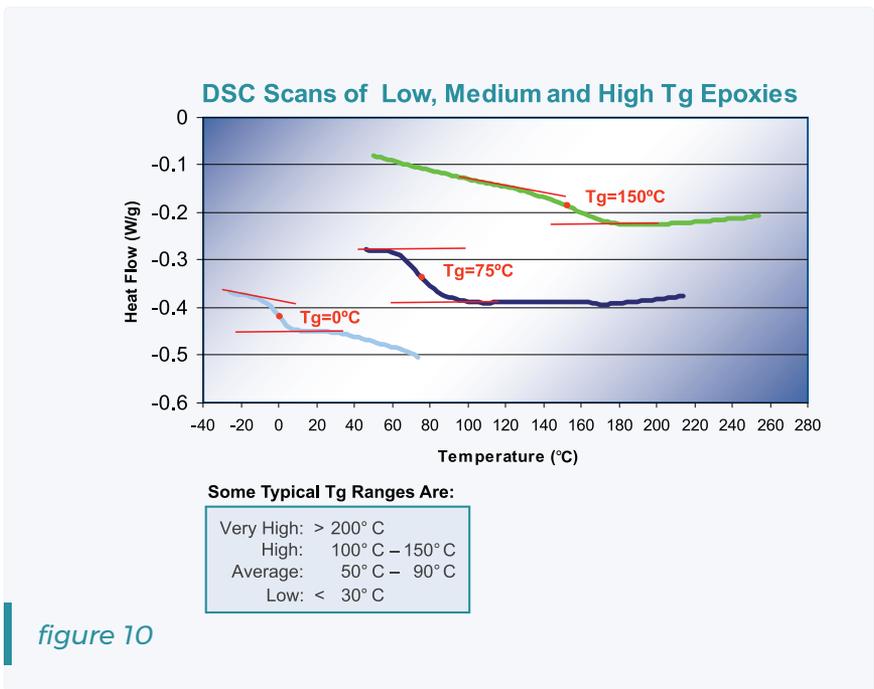


figure 10

## 4.0 Thermogravimetric Analysis (TGA)

TGA is a method for measuring very small weight changes in a material as a function of temperature. This technique is used to determine thermal degradation temperature. It can also be used to measure moisture content and level of outgassing. Additionally, it is used as a method to check the content of inorganic fillers (such as silver flake) in formulations for QC lot acceptance. ASTM D3850, "Standard Test Method for Rapid Thermal Degradation of Solid Electrical Insulating Materials by Thermogravimetric Method (TGA)", governs this testing.

As shown in the schematic in figure 1, a TGA is essentially a very high precision micro-balance in a controlled atmosphere. A sample basket is suspended from one arm of the balance while a weighted tare pan balances the other side. The system is designed to compensate for any changes in sample weight to maintain a balanced position.

As a sample is heated, it will normally begin to lose mass. At low temperatures, this is generally due to either evaporation of moisture or low molecular weight species; while at higher temperatures, larger weight losses due to thermal degradation will eventually occur. The mass loss will cause the sample arm of the balance to tip up slightly. An infrared LED is mounted in the middle of the balance arm. When the balance tips, the light from the LED moves off center and a photodetector notes a difference in the measured intensity. Current is then supplied to a meter which rotates the balance until the light from the LED is again centered on the photodetector. The amount of current required to return the balance to its zero position is directly proportional to the displacement of the sample balance arm and, hence, the mass change of the sample.

### TGA 2950 Schematic\*

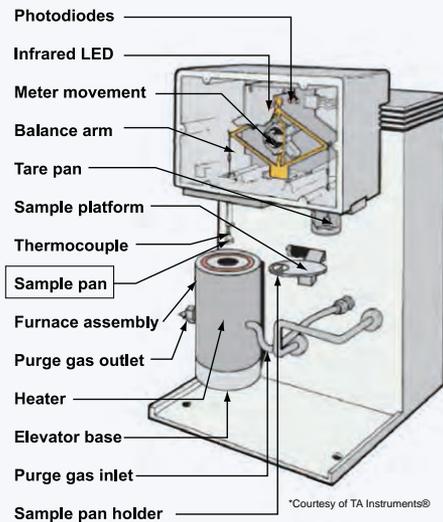
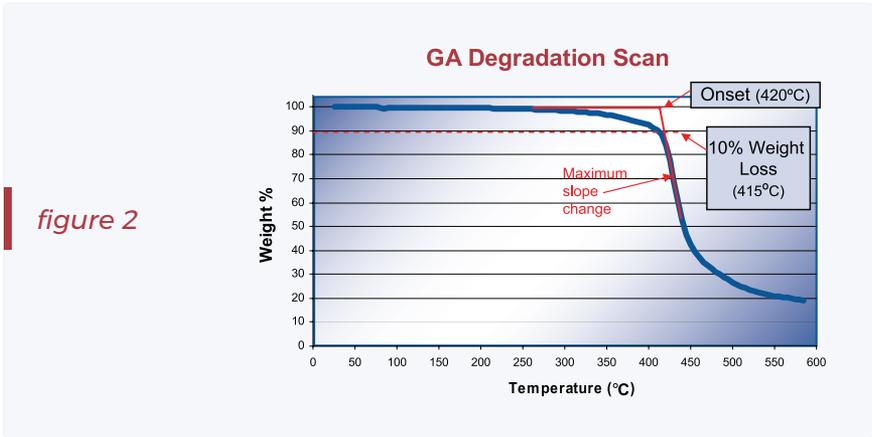


figure 1

## 4.1 Degradation Temperature

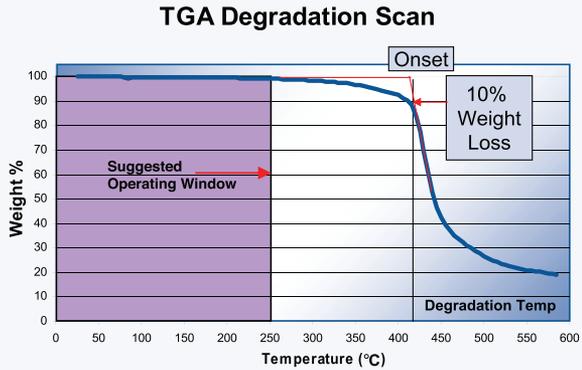
The output of a TGA scan is generally a plot of percent sample weight versus temperature (as shown figure 2). The degradation temperature of a cured material can be calculated in two ways. The first approach is the onset degradation temperature. This temperature is calculated based on the intersection of the tangent to the maximum change in slope of the weight loss curve with the tangent to the baseline.

The second approach is the 10% weight loss temperature. This calculation is very straight forward for unfilled systems. In this case, it is simply the temperature at which the material has lost 10% of its starting weight. For filled systems, this weight loss is determined based on a loss of 10% of the polymer component of the formulation. For example, if a conductive material is 70% filled with silver flake, only 30% of the system is polymer. A loss of 10% of the weight of the polymer component will be a 3% loss in weight of the overall system. Generally, as seen in the system below, the two methods result in very similar values for degradation temperature.



As generally reported on data sheets, the suggested operating temperature windows is calculated on the degradation temperature. The upper end of the continuous operating window is calculated by subtracting 150 °C from the degradation temperature.

figure 3



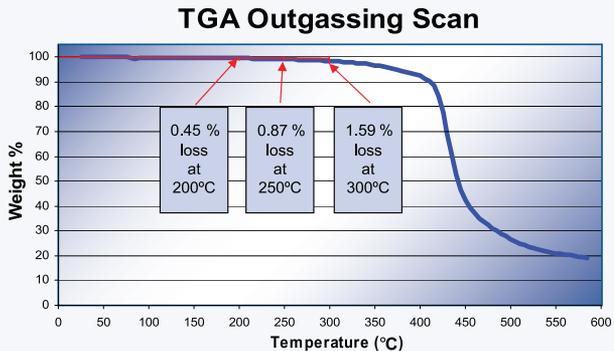
For the example material in figure 3, the degradation temperature is approximately 420 °C. Thus, 270 °C would be calculated for the suggested operating temperature window. Generally, we would add a safety factor so that the datasheet would actually list 250 °C as the continuous operating windows.

## 4.2 Outgassing (Thermal Stability)

The percent weight versus temperature curve is also used to determine weight loss at specified temperatures. This weight loss is often referred to as outgassing or thermal stability. Datasheets generally report outgassing levels at 200 °C, 250 °C and 300 °C. <1.0% @ 200 °C is required by MIL-STD 883 Method 5011 (low outgas)

Figure 4 below shows an example of outgassing calculations.

figure 4



One important industry specification is the NASA outgassing requirement. Products that meet this specification must exhibit less than 1.0% Total Mass Loss (TML) after being exposed to 125 °C for 24 hours in a vacuum. They must also contribute less than 0.1% Collected Volatile Condensable Materials (CVCM) during this exposure.

# EPO-TEK Materials Meeting NASA Outgassing Requirements

Product	TML* (<1.0%)	CVCM** (<0.1%)	Cure Time	Cure Temp	Description
301	0.98	0.01	1 Hour	65° C	Optical
301-2	0.70	0.01	3 Hours	80° C	Optical
302-3M	0.70	0.01	7 Days	25° C	Optical
314	0.77	0.00	2 Hours	120° C	Optical
320	0.99	0.00	2 Hours	65° C	Black
353ND	0.81	0.01	1 Hours	120° C	Optical
354	0.31	0.00	3 Hours Hours	80° C +100° C	Optical
377	0.54	0.02	1 Hours	150° C	Optical
390	0.43	0.01	30 Min.	25° C	Polyimide
920	0.65	0.01	45 Min.	80° C	Alumina-Filled
930	0.49	0.00	45 Min.	80° C	Boron Nitride-Filled
930-4	0.82	0.01	1 Hours	150° C	Boron Nitride-Filled
E2101	0.32	0.01	1 Hours	150° C	Silver-Filled
H20E	0.62	0.01	1 Hours	150° C	Silver-Filled
H20E-D	0.54	0.01	1 Hours	150° C	Silver-Filled
H20E-PFC	0.76	0.01	1 Hours	150° C	Silver-Filled
H21D	0.39	0.00	1 Hours	150° C	Silver-Filled
H22	0.99	0.01	20 Min.	100° C	Silver-Filled
H27D	0.52	0.09	1 Hours	150° C	Silver-Filled
H31	0.54	0.01	1 Hours	150° C	Silver-Filled
H31D-LV	0.47	0.02	1 Hours	125° C	Silver-Filled
H35-175MP	0.33	0.02	1.5 Hours	150° C	Silver-Filled
H37-MP	0.27	0.01	1 Hours	150° C	Silver-Filled
H44	0.27	0.00	1 Hours	150° C	Gold-Filled
H63	0.19	0.01	1 Hours	120° C	Alumina-Filled
H67-MP	0.37	0.00	1.5 Hours	150° C	Alumina-Filled
H70E	0.99	0.03	12 Hours	60° C	Alumina-Filled
H72	0.31	0.00	30 Min.	100° C	Alumina-Filled
H73	0.43	0.01	20 Min.	100° C	Alumina-Filled
H74	0.27	0.01	24 Hours	50° C	Alumina-Filled
H77	0.22	0.00	1 Hours	125° C	Alumina-Filled
H81	0.62	0.00	16 Hours	60° C	Gold-Filled
MA-5	0.40	0.04	1 Hour	150° C	B-Stage
OG198-54	0.53	0.00	UV Cure + 1 Hour	150° C	Cationic UV
T7109	0.80	0.13	1 Hours	150° C	Boron Nitride-Filled
T7110-38	0.60	0.01	7 Hours	70° C	Alumina-Filled
U300-2	0.97	0.01	30 Min.	150° C	Underfill

\* Total Mass Lost \*\* Collected Volatile Condensable Materials

## EPO-TEK Products Meeting MIL-STD 883/5011 Standards:

Electrically & Thermally Conductive Adhesives

EPO-TEK®	Cure	Outgassing ( $\leq 1.0\%$ )
EK1000-MP	200°C - 60 min	0.11%
EK1000-1MP	200°C - 60 min	0.04%
H20E-MP	150°C - 60 min	0.25%
H35-175MP	150°C - 90 min	0.33%
H35-175MP-LV	180°C - 60 min	0.79%
H35-175MPT	180°C - 60 min	0.51%
H37-MP	150°C - 60 min	0.69%
H37MP-2	150°C - 60 min	0.30%
H37-MPT	150°C - 60 min	0.31%

### Thermally Conductive Adhesives

EPO-TEK®	Cure	Outgassing ( $\leq 1.0\%$ )
H65-175MP	165°C - 90 min	1.00%
H67-MP	150°C - 60 min	0.37%
H67MP-GB	150°C - 60 min	0.05%
H67-MP-T	150°C - 60 min	0.54%

The Fiber Optics industry also has a set of outgassing requirements. These are known as Telcordia GR-1221. The cured adhesive is heated from 50°C to 150°C at 5°C/min in the TGA. A 0.1% weight loss or less is considered evidence of a properly cured thermal cure system. While, 0.25% weight loss or less is considered evidence of a properly cured UV system.

## EPO-TEK Materials Meeting Telcordia Standard:

Telcordia GR-1221

EPO-TEK®	Cure	Weight Loss
323LP	150°C - 60 min.	0.070%
353ND	150°C - 30 min.	0.037%
375	150°C - 30 min.	0.041%
383ND	150°C - 30 min.	0.085%
OE184	150°C - 30 min.	0.027%
OG116-31	UV - 2 min. at 320 - 500 nm	0.087%
OG198-54*	UV - 2 min. at 320 - 500 nm	0.084%

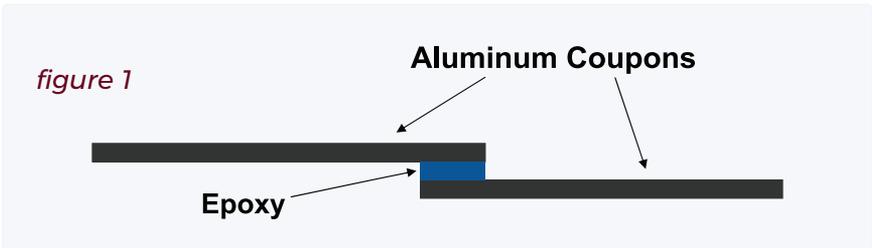
\* Material was post cured for 1 hour @ 150°C

## 5.0 Lap Shear and Die Shear

Lap shear strength and die shear strength tests are commonly used in the adhesive world to characterize the bond strength of epoxies. Each measures the strength of an adhesive on a specific substrate.

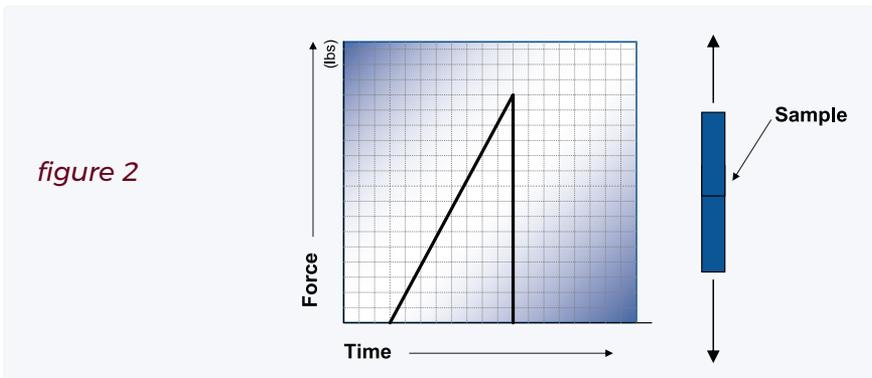
Lap shear strength is assessed using ASTM D1002, "Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal to Metal Bonding)". This test allows not only the determination of shear strength of an epoxy when bonded to aluminum, but also the examination of its mode of failure which is equally as important.

Each lap shear specimen is created using two 1 inch wide acid etched aluminum coupons with a 1/2 inch overlap. A side view of this assembly is shown in figure 1.



Once the specimens have been assembled and cured according to the recommended cure schedule on the data sheet, they are cooled to room temperature and placed one at a time into the Instron® Test System for analysis. The two coupons are clamped vertically between "grips" and pulled 180°, at a constant rate set by the Operator. The pulling force is increased on the coupons, until the adhesive joint fails resulting in a curve similar to figure 2. The tensile shear strength is then calculated from the curve using the following formula:

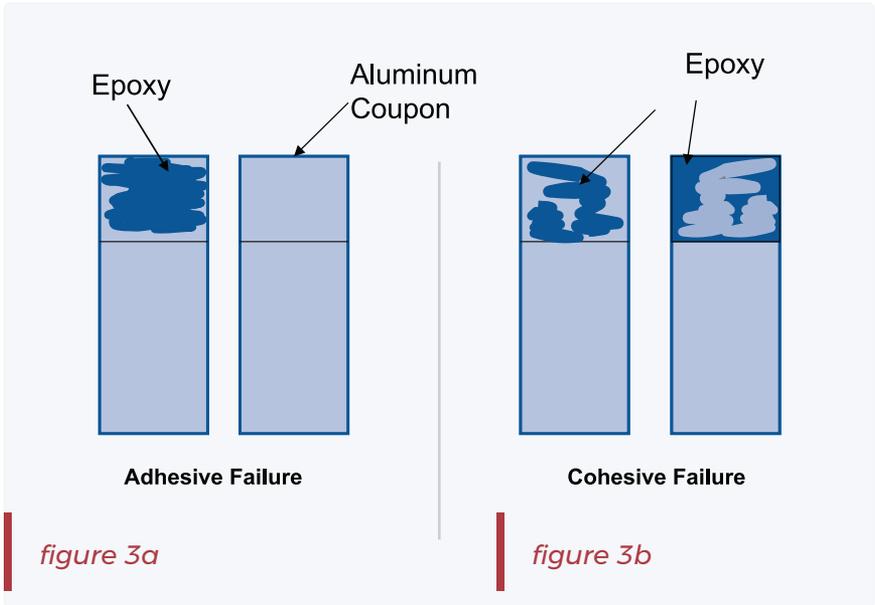
**Lap shear strength (psi) = maximum load force (lbs.) / bond area (in<sup>2</sup>)**



Typical values for lap shear strength range from 500 - 3000 psi. However, the strength reading is not the only important parameter of the test. The mode of failure is equally, if not more important to consider.

There are two types of failures that can occur adhesive and cohesive. When a material fails adhesively, the adhesive completely loses its bond to the substrate. This occurs when the adhesive strength of a bonding material is lower than its cohesive strength. With a cohesive failure, the strength of the adhesive itself is not as great as the forces applied to it and the adhesive pulls apart leaving portions of the adhesive bonded to both substrates.

Figure 3a & 3b show an example of both failure modes.

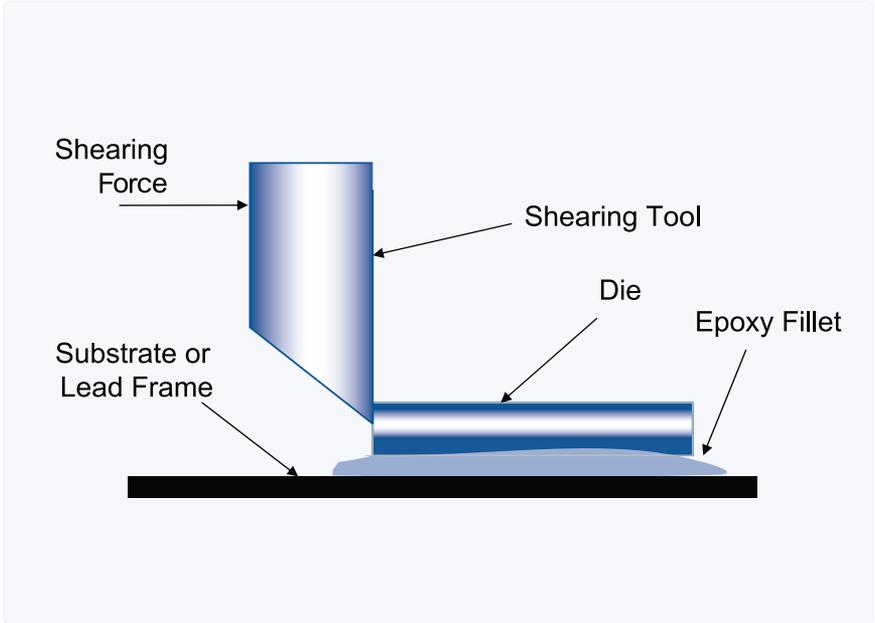


In applications where a very high constant load is present, a high tensile strength adhesive with good mechanical hold to a chosen substrate is recommended.

High:	2500 – 4000	psi
Average:	1200 – 1900	psi
Low:	< 800	psi

Die shear strength is a test common to the semiconductor industry. It is used to determine the strength of a die bonded to a package substrate using a die attach adhesive. By subjecting the die to a stress that is parallel to the substrate, the result is a shearing stress at the interface between the die and die attach material. The purpose of die shear testing is to gauge the overall quality of the bond to both die and substrate.

The following is an enlarged schematic of the test:



Die shear strength is typically reported in kilograms and can vary significantly due to die and substrate composition as well as die size. For larger dies, a lower stress material is recommended because of the greater surface area. Typical die shear testing is done on 2mm x 2mm gold dice on gold plated Kovar® substrates. The higher the value, the stronger the bond.

High:	> 10 kg / 3400	psi
Med:	5 – 10 kg / 1700 – 3400	psi
Low:	<2 – 5 kg / <1000	psi

## 6.0 Dynamic Mechanical Analysis (DMA)

DMA measures a material's response to an imposed oscillating stress or strain. Either the imposed stress is controlled and the resulting strain in the sample is recorded, or the strain is controlled and the resulting stress level is measured.

When a stress is imposed on a polymeric system, this stress will be dissipated throughout the material and generally results in some level of deformation. Polymers are viscoelastic in nature. As a result, only a portion of the deformation will be reversible when the stress is released. This is the elastic portion of the response.

**Recall Hooke's Law for an ideal elastic solid:**

$$\sigma = \kappa * \epsilon$$

Where:

$\sigma$  = stress

$\epsilon$  = strain

$\kappa$  = spring constant

The above equation shows that the amount a spring will be stretched is proportional to the amount of stress imposed on it. The proportionality is described by the spring constant,  $k$ . The spring constant may be thought of as the stiffness of the spring. Another term for the stiffness of a material is the modulus,  $E$ .

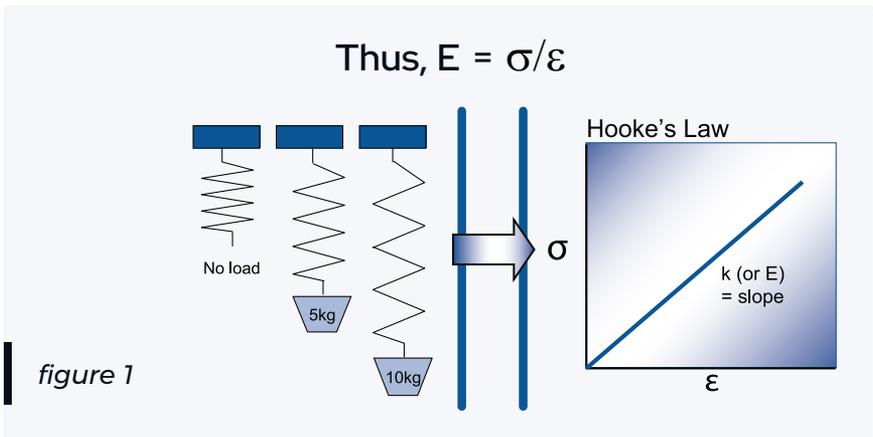
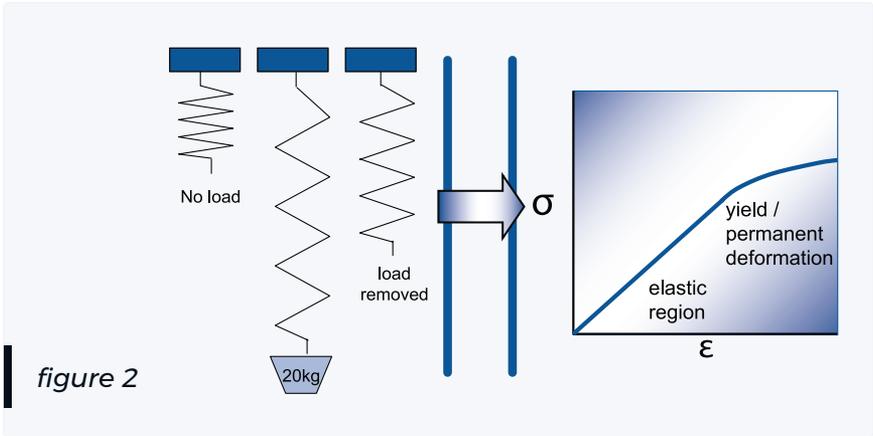


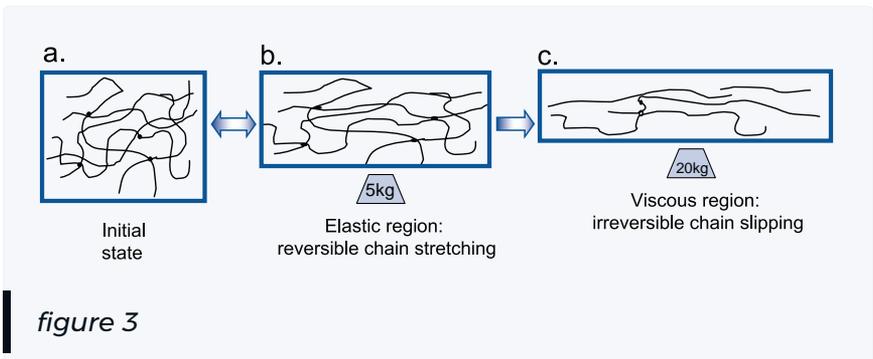
Figure 1 above shows **HOOKE'S LAW** for the linear relationship between applied load and elongation of an elastic spring.

However, as shown below (figure 2), if the load becomes too large, the spring will actually undergo some permanent deformation and will no longer return to its original length when the load is released.



As shown in figures 3a and 3b below, when very low levels of stress are applied to a polymer system, the coiled polymer chains will begin to stretch and straighten out slightly. This results in a low level of deformation. However, like the spring, entropy will drive the majority of chains to recoil back into their original preferred orientation as soon as the load is released.

At higher loads, as shown in figure 3c, the chains will not only stretch and uncoil, but will actually start to untangle and slide past each other. This deformation is not reversible. At the extreme, the slipping of chains becomes similar to a viscous flow. If the system is not cross-linked, the material will eventually pull apart. However, if the system is cross-linked, it will stretch and flow until the cross-links become the only forces holding it together. Eventually, the chains will break and the sample will fail.



While **HOOKE'S LAW** describes the elastic component of a polymer's stress/strain behavior, Newton's law of ideal fluids is often used to describe the other extreme of a polymer's response to stress viscous flow.

$$\sigma = \eta(dy/dt)$$

Where:  $\sigma$  = stress  
 $\eta$  = viscosity  
 $dy/dt$  = strain rate

Newton's equation shows that the amount an ideal fluid will flow in a given period of time will be proportional to the amount of stress imposed upon it. The proportionality is described by the fluid's viscosity.

Often, car shock absorbers or dashpots are used to illustrate this viscous flow behavior. As figure 4 depicts, the rate at which the liquid is forced through the holes in the cylinder is dependant on the stress/load placed on the dashpot. Unlike the reversible deformation of the spring, the displacement of the fluid in the dashpot is completely irreversible. Thus, all work that has been put into the system is dissipated into the fluid.

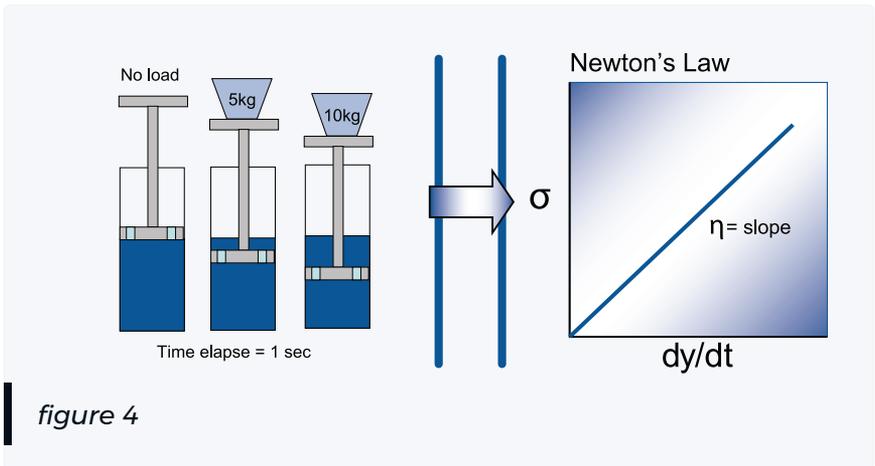
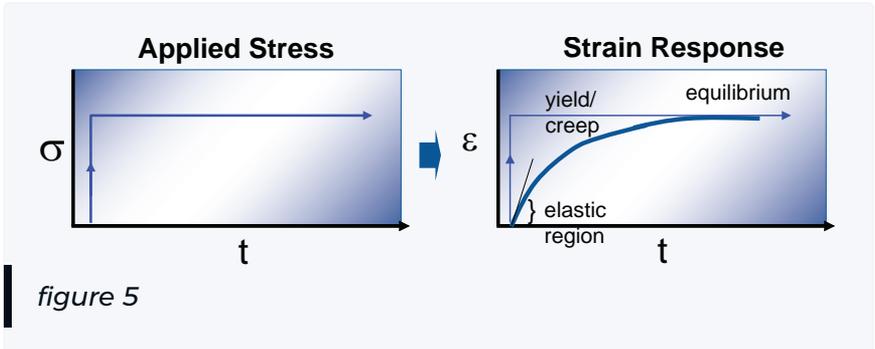


figure 4

You will note that the viscous component of the deformation introduces time as a key variable. As shown earlier, a polymer will respond to an applied stress first, by stretching and uncoiling of the chains followed by disentanglement and finally slipping of the chains past each other. These polymer rearrangements require time to occur.

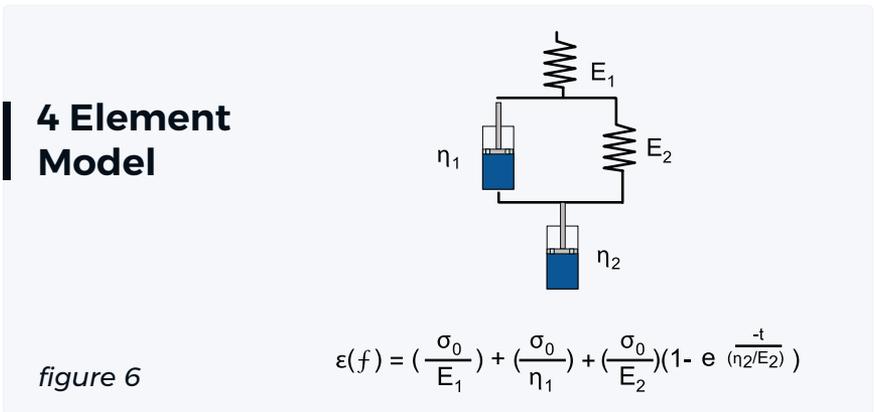
As a result, over time, a polymer's actual response to an applied stress looks more like figure 5 below:



As the figure above shows, when a constant stress or load is applied to a polymer system, the first response is elastic chain stretching/uncoiling resulting in elastic deformation. If the polymer were a true spring, the deformation would quickly reach an equilibrium point and no additional deformation would occur (like a weight hanging on the end of a spring).

However, with a visco-elastic system, the sustained load will cause the polymer chains to start to untangle and slide a bit. The untangling process will also allow the polymer coils to stretch a bit more. This slipping and additional stretching results in additional deformation over time, called creep. Eventually, the chains will have stretched as far as they can and further deformation will come from pure viscous chain slippage of any non-crosslinked chains.

Many mechanical models have been developed to simulate visco-elastic behavior. One of the most popular is the 4 element model shown in figure 6 below. The first spring governs the initial elastic response, followed by a mixed region of spring and dashpot elements in parallel and then, finally the purely viscous dashpot element in series with all of the above.



When the stress/load is released, entropy will slowly cause the stretched polymer chains to coil back to their original state. However, only the elastic portion of the deformation will be recovered and the sample will remain somewhat deformed relative to its initial state (figure 7).

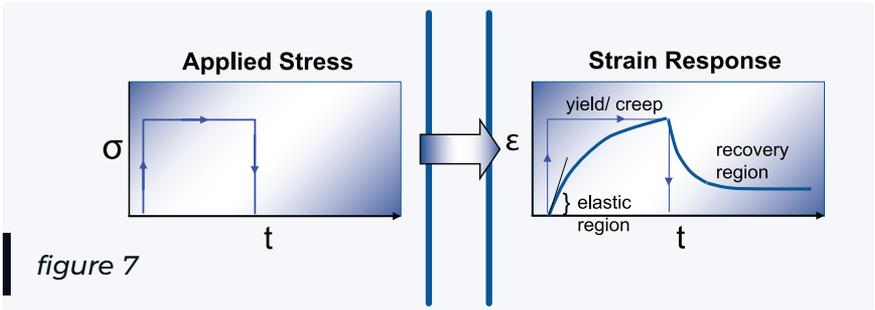


figure 7

The final key element that can have an impact on the stress/strain behavior of a material is temperature. As shown in figure 8, higher temperatures give increased mobility to polymer chains. This results in faster and larger deformations at a given load than if the same material was tested at lower temperatures.

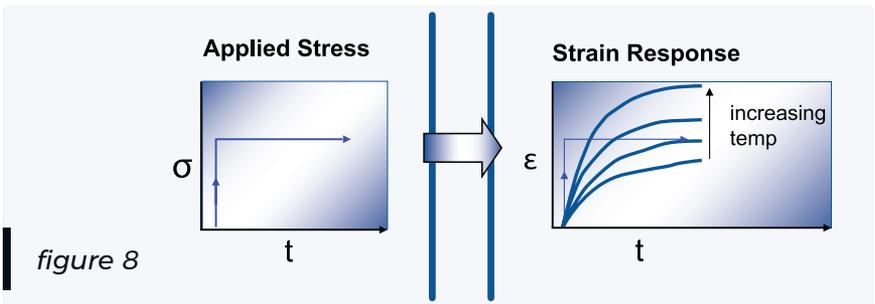


figure 8

**DYNAMIC TESTING** As mentioned at the very beginning of this section, Dynamic Mechanical Analysis generally measures the strain response of a material to an oscillating applied stress. In some cases the applied strain is controlled, and the resulting stress level in the material is measured, but this is less common. Figure 9 shows some of the many possible test geometries for DMA.

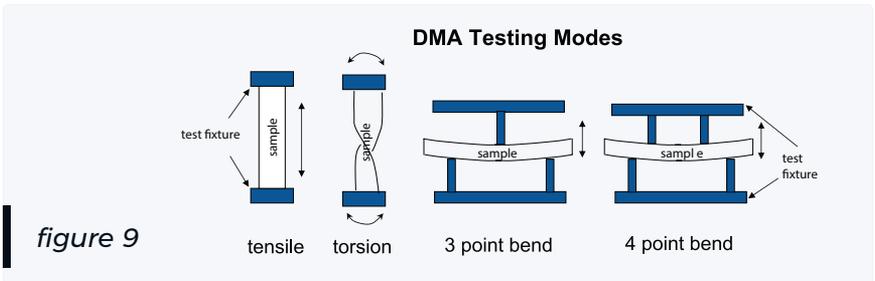


figure 9

Regardless of which form of stress is imparted to the sample (tensile, torsion or bending), all DMA impart either a sinusoidal stress or strain to the given sample and measure the resulting material response.

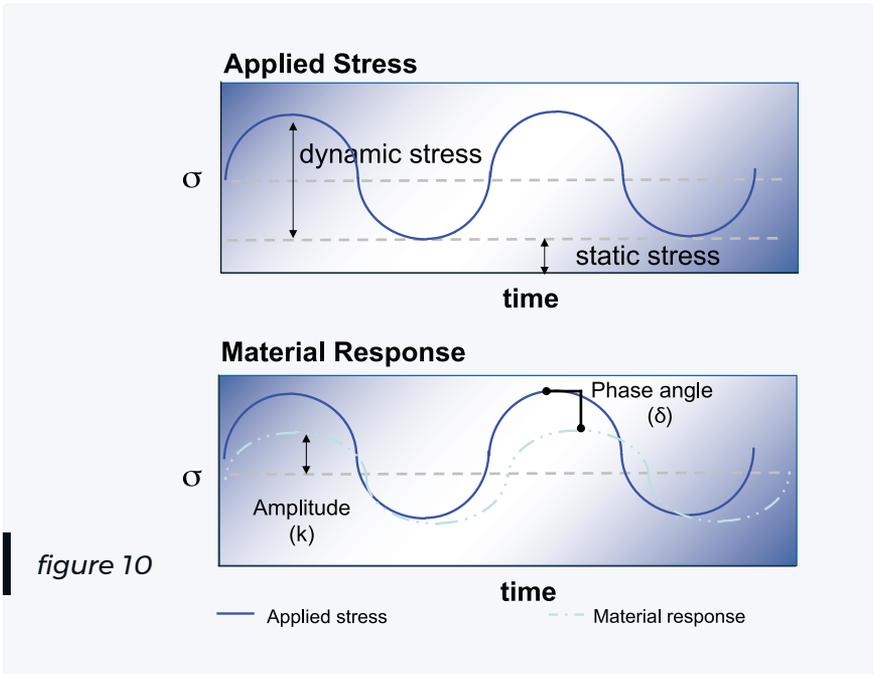


figure 10

Therefore, if the equation for the stress applied to material shown above is given by:

$$\sigma = \sigma_0 \sin(\omega t)$$

- Where:
- $\sigma$  = stress at a given time
  - $\sigma_0$  = the maximum applied stress
  - $\omega$  = the frequency of the oscillation

The material's response will depend on its relative degree of elastic versus viscous character. The more elastic the material, the closer the response will track with the applied stress profile. However, the more viscous the material, the more time it will take to flow and respond to the applied load and the deformation will be less reversible.

Therefore, the resulting strain response for the material shown in figure 10 will be out of phase with the applied stress by an amount  $\delta$ :

$$\varepsilon = \varepsilon \sin(\omega t + \delta)$$

The strain can be expanded to the form:

$$\varepsilon = \varepsilon \sin(\omega t) \cos \delta + \varepsilon \cos(\omega t) \sin \delta$$

This expression shows that the strain is actually composed of two terms that are  $90^\circ$  out of phase with each other. The above equation can then be written in terms of the contribution of real and imaginary components:

$$\varepsilon = \varepsilon' \cos(\omega t) + i \varepsilon'' \sin(\omega t)$$

The first term contains the real component. This represents the portion of the strain response that is due to the elastic nature of the material. The second term is the out of phase portion of the response, which reflects the ability of the material to dissipate energy through viscous mechanisms. The complex modulus of the material can be calculated by dividing the equation for stress by that for strain:

$$E^* = \sigma / \varepsilon' + \sigma \varepsilon''$$

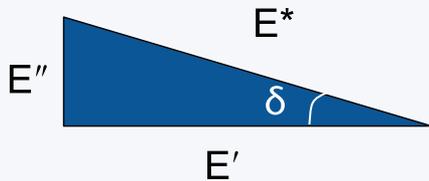
$$E^* = E' + E'' \quad \text{Where:}$$

$E'$  = the real or "storage" modulus

$E''$  = the imaginary or "loss" modulus

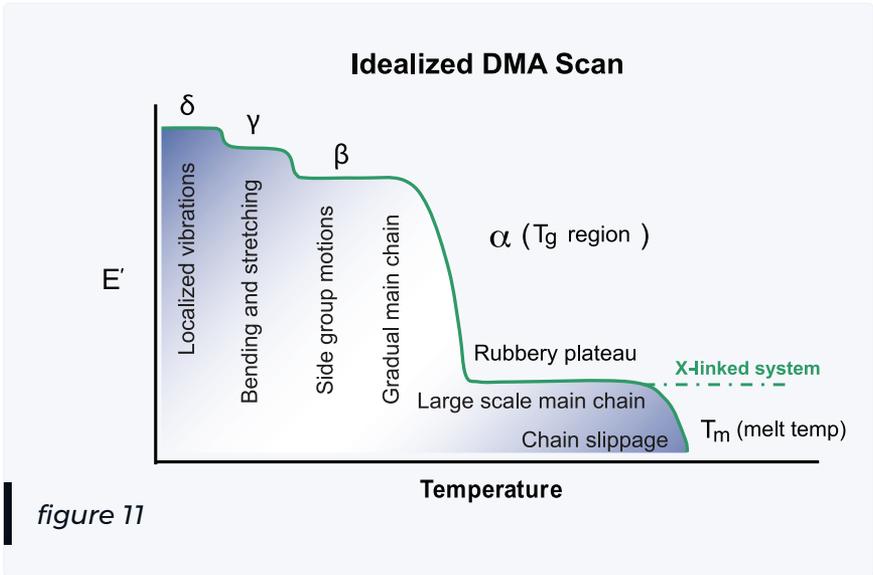
The ratio of the loss modulus to the storage modulus is known as the  $\tan(\delta)$ :

$$\tan(\delta) = E'' / E'$$



The  $\tan(\delta)$  is a meaningful property, as it represents the ratio of the viscous versus elastic contributions of the material at a given condition.

While both the absolute and relative values of the storage and loss moduli at room temperature are valuable, generally DMA is run as a function of temperature. Measuring a material's response to oscillating stress over a broad temperature range gives a wealth of information about thermal transitions. For instance, DMA provides one of the highest resolution techniques for measuring a material's  $T_g$ .



The idealized DMA scan in figure 11 shows how changes in a material's storage modulus with temperature may be able to reveal a great deal about its thermal transitions. The most commonly reported thermal transition for a thermosetting polymer is its Glass Transition Temperature ( $T_g$ ). As discussed previously, the  $T_g$  of a material is not actually a discrete thermodynamic transition, but is actually a temperature region over which the mobility of the polymer chains increases significantly and the bulk material changes from a rigid, glassy state to a more rubbery/compliant state. This change is indicated by a significant drop in the storage modulus as the material response changes from primarily elastic/ reversible deformation to more and more viscous/irreversible chain motion. This large transition, the alpha transition ( $\alpha$ ), is generally where the  $T_g$  is originated from.

Beyond the glass transition region is the "rubbery plateau" region where the ratio of elastic to viscous contributions reaches an equilibrium. For a crosslinked thermosetting material, this plateau is maintained until the degradation temperature is reached. For non-cross-linked systems the melt temperature will eventually be reached and cause liquid-like flow.

Figure 12 shows an example of a typical DMA plot for an epoxy resin. This plot shows both storage and loss modulus as well as  $\tan(\delta)$ . From this plot, the  $T_g$  region can be seen in several ways. The first method for calculating the  $T_g$  is from the onset of the drop in the storage modulus ( $E'$ ) curve. This indicates the point where viscous motions begin to kick in. The second method is the peak of the loss modulus ( $E''$ ) curve. This indicates the point of maximum chain slippage in a cross-linked system. Finally, the peak of the  $\tan(\delta)$  curve can be used to determine  $T_g$ . This indicates the point where the ratio of viscous response to elastic response is the highest.

## Typical DMA Result

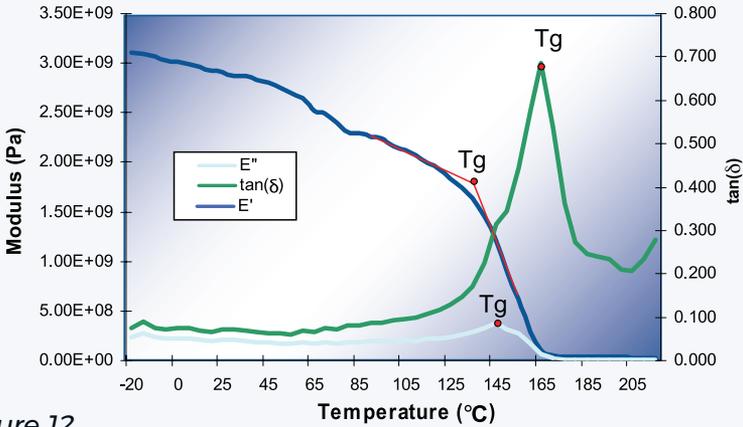


figure 12

The peak of the  $\tan(\delta)$  curve is a popular measurement point for the  $T_g$  as it is usually easier to isolate than to determine the onset of the drop in the storage modulus. However, this case again highlights the fact that it is important to identify how a  $T_g$  was measured as each of these three methods of determining  $T_g$  are accepted in the industry, but will give different absolute values.  $T_g$ 's calculated from the  $\tan(\delta)$  peak will always be higher than those calculated from the storage modulus. Generally,  $T_g$ 's calculated from the loss modulus will fall somewhere in between.

The  $\tan(\delta)$  versus temperature curve can be used to determine much more about a system than just its  $T_g$ . The width of the  $\tan(\delta)$  peak can indicate how homogeneous a system is. Systems with very broad peaks are generally composed of a blend of different polymer chain lengths or structures which gives rise to a broader temperature range, to initiate significant viscous chain motions for the various components. On the other hand, systems with narrow peaks generally have more narrow distribution of chain types and molecular weights.

The height or amplitude of the  $\tan(\delta)$  curve is directly related to a material's ability to dissipate energy through segmental motion. Systems with tall  $\tan(\delta)$  peaks have higher ratios of energy absorbing viscous motions and are, therefore, generally tougher systems than those with low  $\tan(\delta)$  amplitudes.

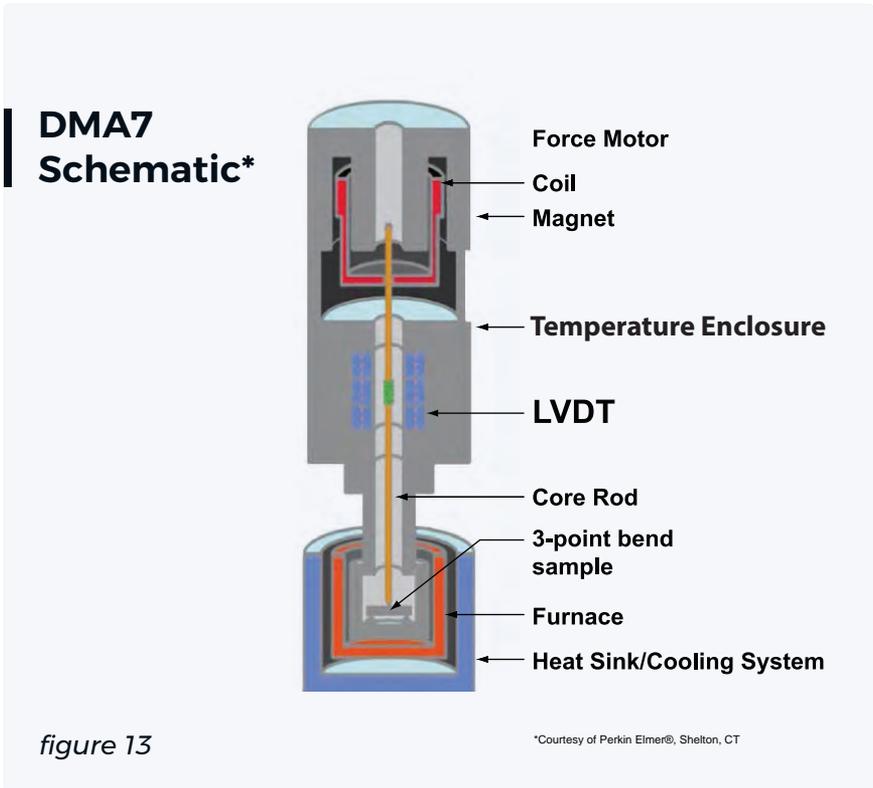
The final common use of the data from a DMA curve is the most basic—to determine the modulus of a material at a given temperature for use in design calculations. Since the modulus measures the stiffness of a material, this is often used in combination with knowledge of the material's CTE (which will be discussed in the next section) to determine levels of stress that may occur during thermal stressing of bonded substrates.

The best way to reduce bond stresses during thermal cycling is to match the thermal expansion character of the adhesive to that of the substrate. Unfortunately this is not always possible. The next best choice may be to select a material with a lower modulus or higher toughness that can absorb any stresses incurred by thermal expansion mismatch. For these calculations, we generally use the storage modulus as we assume that the deformations in the bond line will most likely be small enough to keep the adhesive within its elastic limit.

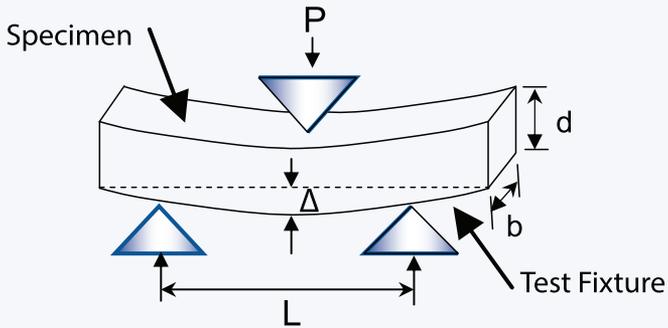
Some typical ranges for Elastic Modulus are:

High:	>900,000	psi	(generally high Tg)
Med:	400,000 – 900,000	psi	(generally med Tg)
Low:	<150,000	psi	(soft/flex/resilient)

Figure 13 shows a schematic of the sample chamber of a typical DMA.



## 3 Point Bend Set-up



$$E = \frac{L^3 P}{4bd^3 \Delta}$$

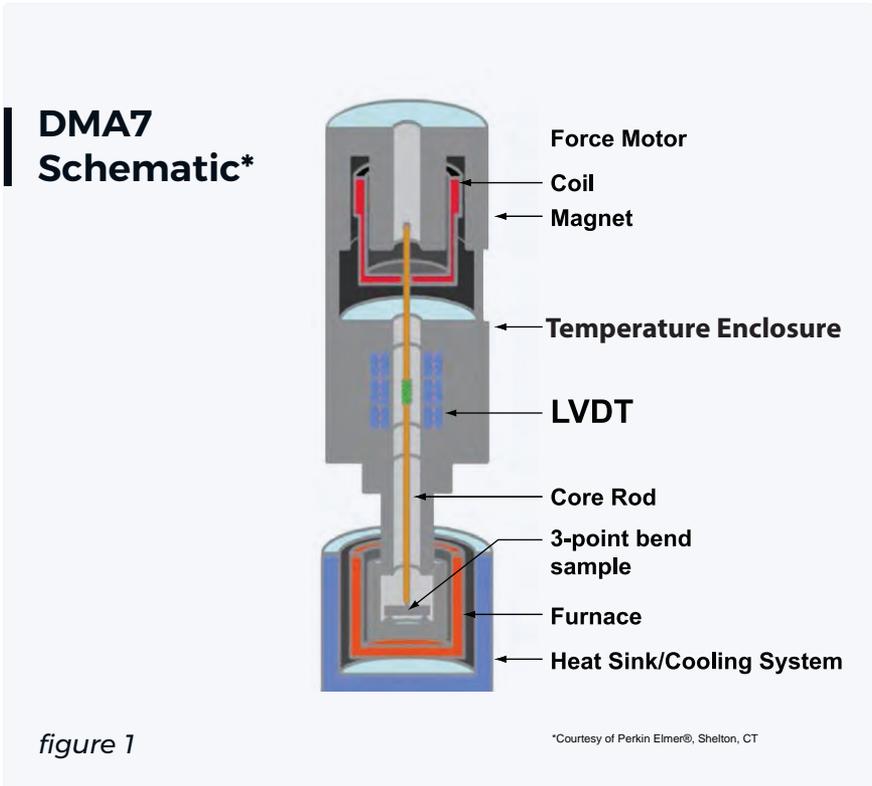
figure 14

Figure 14 shows the sample geometry used to measure modulus at EPO-TEK. A small, 3.0mm x 1.0mm x 18.0mm bar of material is tested in three point bending.

## 7.0 Thermomechanical Analysis (TMA)

TMA is a technique for measuring very small expansions or contractions in a material as it is heated or cooled. The linear expansion of a material in response to temperature is known as the linear Coefficient of Thermal Expansion (CTE).

CTE can be measured using the same equipment as Dynamic Mechanical Analysis (DMA). However, for CTE, the knife edge probe and three point bend fixture are replaced with a flat bottom cylindrical probe and flat sample holder (as shown in figure 1 below).



Typically, TMA measures the linear expansion (or contraction) of a material based on the change in height ( $\Delta z$ ) of a small bulk sample. The sample is usually a small cylinder or cube with very flat, parallel top and bottom surfaces. The sample is placed on a flat sample platform with very low thermal expansion—usually quartz. A flat bottom probe is lowered until it just touches the surface of the sample. A very small load is then applied to make sure that the probe contacts the sample in a controlled manner. This load remains constant throughout the scan. The position of the probe is sensed using a Linear Variable Differential Transducer (LVDT).

As the sample experiences a thermal ramp, it will expand (if heated) or contract (if cooled) under the probe. The LVDT will track this probe position versus temperature (figure 2).

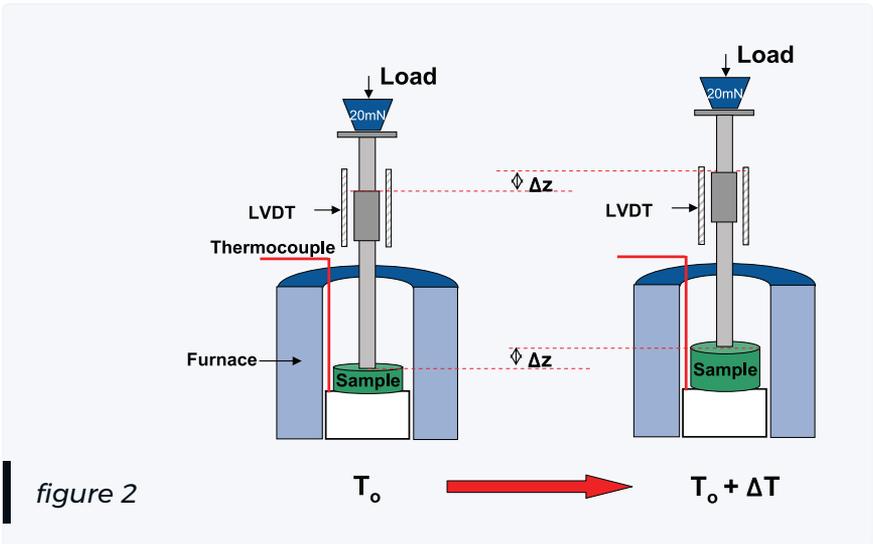


figure 2

CTE testing is performed in accordance with ASTM E831, "Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis". The figure below shows a typical TMA plot of probe position versus temperature. CTE is calculated as the relative change in height of the sample per one degree change in temperature:

$$\text{CTE } (\alpha) = \text{ppm}/^\circ\text{C}$$

$$\alpha = \frac{1}{h_0} * \frac{dh}{dT}$$

Thus, the CTE can be calculated by dividing the slope of the probe position ( $dh/dT$ ) versus temperature curve, by the initial height ( $h_0$ ) of the sample.

## Sample TMA Curve

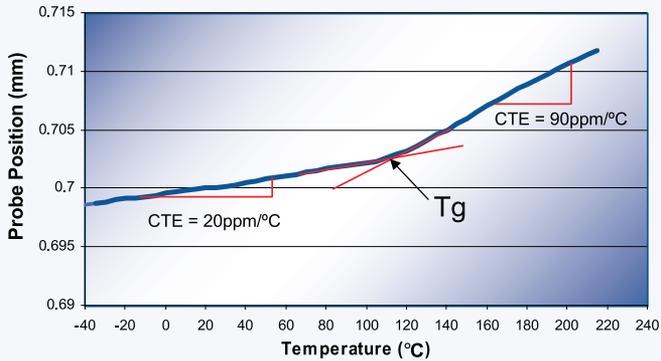


figure 9

A typical TMA plot is shown in figure 3. It will have two different slopes; a shallow slope at lower temperatures, and a steeper slope at higher temperatures.

The shift in the slope of the probe position versus temperature plot will occur at the material's glass transition temperature (Tg). In the Tg region, the polymer chains will have enough thermal energy to undergo larger scale motions which will enable a higher degree of expansion with temperature. Thus, the TMA plot is a convenient method to determine a material's Tg. This is calculated by finding the point at which the tangents to the two different slopes of the TMA plot intersect. Typically, the CTE above Tg is 3 to 4 times that of the CTE below Tg.

From an engineering design standpoint, it is often important to match the CTE of an adhesive as closely as possible to the substrates being bonded in order to minimize stress at the interfaces. Thus, it is important to keep in mind that the adhesive will actually have two different expansion rates over a broad temperature range. The temperature at which this transition occurs will also be an important factor. If the Tg is sufficiently high relative to the operating range of the part, only the lower CTE will come into play. On the other hand, it is also important to keep in mind that the modulus of the adhesive will drop significantly at temperatures above the Tg. A lower modulus or higher toughness material will be able to better absorb and lessen the stresses incurred by thermal expansion mismatch.

Examples of some CTE values of epoxy:

Silica Filled:	Lowest	( 5-15 ppm / °C )
Alumina Filled:	Good	( 25-35 ppm / °C )
Silver Filled:	Average	( 40-55 ppm / °C )
Optical (unfilled):	Typical	( 55-100 ppm / °C )

## 8.0 Electrical Conductivity (Volume Resistivity-VR)

Semiconductor and SMD applications require electrical contacts for the circuit to work properly. Thus, VR is very critical to the performance of the circuit. Polymers, however, are inherently electrically insulating materials. They can be made to conduct electricity by adding electrically conductive filler materials. Silver and gold are the most common conductive fillers used as gold does not oxidize and while silver does oxidize, silver oxide remains conductive.

The most common type of electrically conductive adhesive is an Isotropic Conductive Adhesive (ICA). In this case, the distribution of the conductive filler particles is homogeneous throughout the adhesive, resulting in a material that is equally conductive in all directions.

A less-common type of electrically conductive adhesive is an Anisotropic Conductive Adhesive (ACA). As shown in figure 1 below, the amount of conductive filler particles used in an ACA is much lower than the levels in an ICA. In addition, the particle size in an ACA is designed to be the exact thickness of the bond line. As a result, these materials are electrically conductive in only one direction (through the bond line), and are actually electrically insulating in the remaining directions.

The majority of current applications are designed around ICAs.

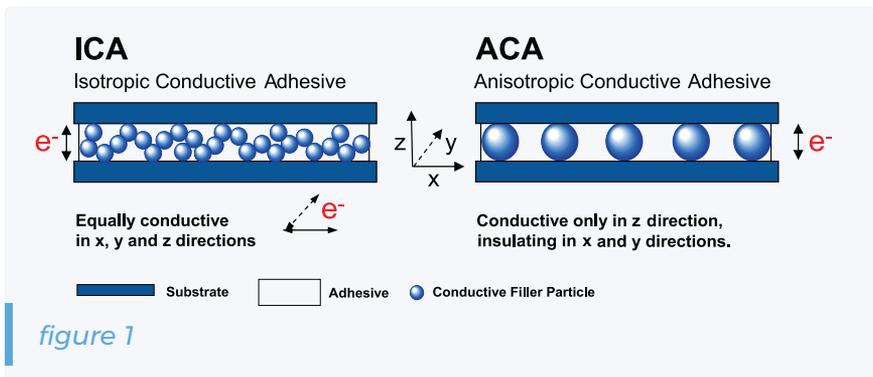


figure 1

In order to establish electrical conductivity in an otherwise insulating resin system, enough conductive filler must be added to create a three-dimensional network of the filler particles for the electrical current to flow across. The size and shape of these filler particles often dictate the loading level required. As shown in figure 2, a flake type filler geometry (diagram 2b) will generally form a continuous conductive network at considerably lower volumetric loading levels than spherical particles of the same material (diagram 2a).

Large flakes (diagram 2c) will typically form a conductive network at lower volumetric loadings than smaller flakes of the same material. However, there are some cases where a combination of large and small flakes (diagram 2d) may produce the optimum conductivity at a given loading due to the ability for the small flakes to pack well between the interstices of the larger flakes.

## Conductive Filler Geometries

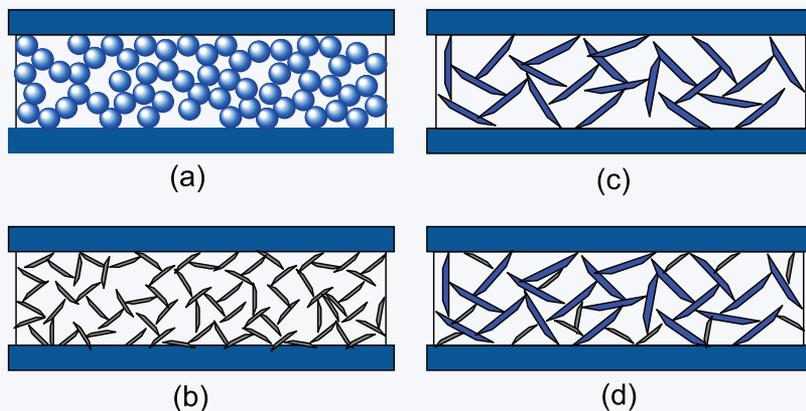


figure 2

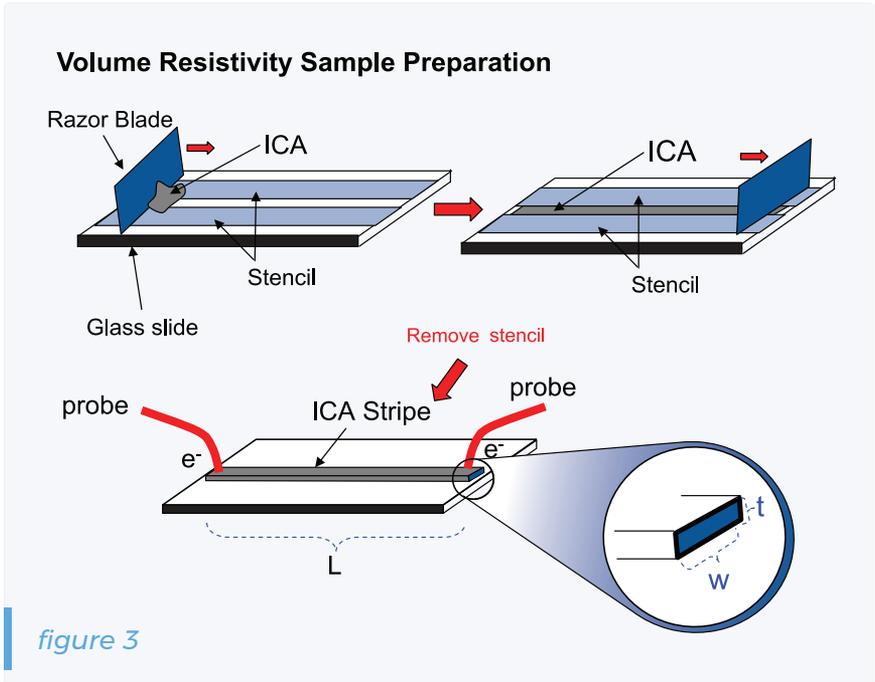
The choice of flake size to impart conductivity to a resin system is also driven by the ICA's intended application. If the ICA is to be dispensed through a fine gage syringe needle, or screen-printed to very tight tolerances, there may be a limit to the maximum size of flake allowed. The required rheology of the ICA may also play a large role in flake size selection. Smaller flakes have much higher surface area per unit volume than larger flakes. As a result, adding smaller flakes will increase the viscosity of the ICA much more than the addition of the same loading content of larger flakes.

The chemical compatibility of the resin system with the surface of the flake can also play a role in determining the degree of conductivity obtained. Many conductive metal flakes are coated with a very thin layer of organic lubricant material. This lubricant is necessary for the manufacturing of the flake. The lubricant layer prevents the flakes from irreversibly welding together during the mechanical process required to flatten them from the particulate starting material to the flake geometry. The compatibility of the resin system with this coating will determine how well the adhesive wets the flake. When there is good compatibility between the lubricant and the resin system, the flakes will disperse well and the resin will wet out the surface of the flake more completely.

While an uncured ICA paste may have some degree of conductivity, the optimum electrical conductivity for a given system is not obtained until the material is fully cured. When a thermosetting resin system cures, the chemical cross-links that form between polymer chains bring the chains closer together and cause an overall shrinkage of the system. When the resin shrinks, it pulls the conductive flakes closer together. This reduces the thickness of insulating resin between the flakes and improves the electrical flow throughout the ICA. Thus, it is very important to fully cure ICA's in order to achieve optimum electrical properties.

Volume resistivity is measured according to ASTM D2739, "Standard Test Method for Volume Resistivity of Conductive Adhesives" and MIL-STD 883/5011. A thin stripe of the ICA is painted onto a glass slide. Typical coating are .150 mm thick, 2.55 mm width, and the length equals the distance between the ohmmeter probes (typically 7.5 cm). The width and thickness of the stripe is usually controlled by using a stencil template.

Figure 3 below shows a typical process for preparing a VR sample.



Once the sample has been prepared and cured, the two probes of an ohmmeter are applied to the ends of the stripe to measure the resistance across the sample. Volume resistivity is then calculated according to the following equation:

$$\text{Volume Resistivity (ohm}\cdot\text{cm)} = \frac{R * w * t}{L}$$

R = resistance (ohms)  
w = width (cm)  
t = thickness (cm)  
L = length (cm)

## 9.0 Thermal Conductivity

In the continued quest for miniaturization, thermal conductivity is playing an even more important role in today's products. More and more adhesives are relied upon to dissipate generated heat away from active components. Thermal conductivity is a fundamental material property that is essential for characterizing this heat transfer.

Thermal conductivity, by definition, is equal to the quantity of heat that is transferred in a specific period of time through a known sample area when the sample's opposite faces are subject to an applied temperature gradient. Typical units of thermal conductivity are:

$$\frac{\text{Watts}}{\text{meter} \cdot \text{Kelvin}} \quad \left( \frac{\text{W}}{\text{m} \cdot \text{K}} \right)$$

One method, ASTM E1461, "Standard Test Method for Thermal Diffusivity by the Flash Method", for measuring thermal conductivity is Laser Flash Diffusivity. This test method has evolved into one of the most widely used techniques for the measurement of the thermal diffusivity and thermal conductivity of polymeric materials in the adhesive industry. Using this technique, the front side of a small, usually disc-shaped sample, is placed into a horizontal fixture and heated by a short energy (laser) pulse. The resulting temperature rise on the alternate surface is measured versus time using an IR detector. Although this is a fairly quick test, the sample preparation and thickness are critical to the end result.

An advantage of this test is that it is non-contact and, is therefore nondestructive to the sample.

Here is a schematic of the test apparatus:

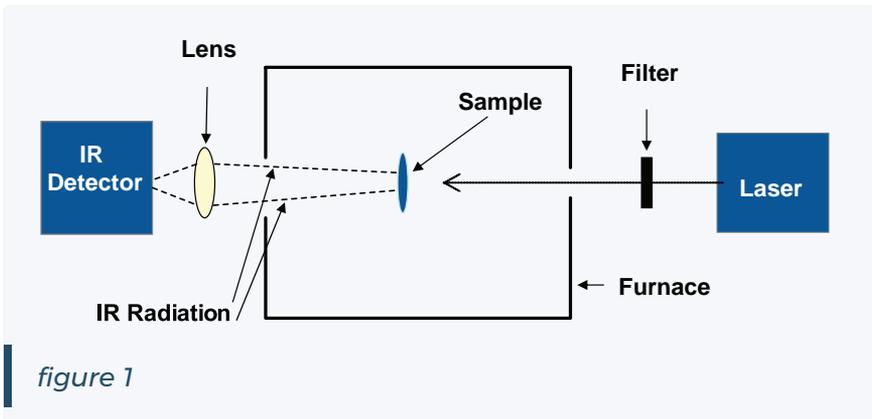


figure 1

Thermal conductivity (ThK) = Heat flow rate / (Area × Temperature gradient)

There are several filler types that exhibit exceptional thermal conductivity: silver, boron nitride (BN) and alumina. Any of the three will greatly aid the thermal conductivity of epoxies which, by nature, are thermally insulative materials. Silver provides the best thermal conductivity, but also provides electrical conductivity. Some applications require a material to be electrically insulating so as not to affect the performance of an electronic package. Alumina is electrically insulating and allows for good thermal conductivity. It is commonly used in potting applications where abrasion is not critical. To reduce abrasion, boron nitride is often used. This provides excellent thermal properties as well as reduced abrasion for critical applications.

In general, higher particle size and higher loadings will result in a higher thermal conductivity value for the epoxy system. Some ranges of performance for thermally filled epoxies are:

Very High:	> 4.0 W/m*K (new technologies)
High:	> 2.0 W/m*K (generally silver filled)
Average:	0.7 – 2.0 W/m*K (Alumina and BN filled)
Low:	< 0.7 W/m*K (optical and unfilled materials)
Poor:	0.1 – 0.3 W/m*K

Filler Type	Thermal Conductivity of Filler Only (W/m*K)
Alumina	36
Boron Nitride	60
Silver	429

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