

# Cure Matters

## Determining the Proper Cure Schedule

After selecting the appropriate adhesive, determining the proper cure schedule is a very important aspect to achieving optimal adhesive performance for your specific application.

*EPO-TEK's datasheets provide a great starting point for recommendations of cure temperatures and times. However, each application is unique and may require its own unique curing profile in order to provide optimal performance.*

## Cure Temperature Considerations



The first place to start is: Cure Temperature. While some epoxies will cure at room temperature, many will require an elevated temperature to initiate the chemical curing reaction. Suggested *minimum* temperatures and times are listed under the “minimum cure” section of the datasheet.

See EPO-TEK Tech Tip # 6 for more information on minimum bond line cure information and remember that each application/project has many variables such as: geometry, unique or substrates, thermal mass concerns, etc. that should be taken into consideration. Minimum cure temperatures stated on the datasheet should be taken as an initial recommendation only.

A *minimum* cure will result in a material that is dry to the touch and, in the case of electrically conductive products, will also have some degree of electrical conductivity. There is often a desire to cure at the lowest possible temperature or for the shortest period of time. While the performance of minimally cured materials may be acceptable for certain applications, it is important to note that the adhesive most likely will not exhibit its full mechanical, electrical or thermal properties.

**Tech Tip 6**  
**Minimum Bond Line (Cure and Thickness)**  
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**What's Minimum Bond Line?**  
Why? Minimum bond line cure and thickness play an important role in adhesive properties and performance.

**Introduction**  
Epoxy cure is a kinetically limited process known as an exothermic reaction. By adding a part B curing agent to the part A monomer, the result is a cross polymer network. Some proceed very rapidly at ambient conditions, others need to be cured with a temperature or light source, but all require a minimum amount of time for a complete cure. Epoxies also require a sufficient adhesion and performance in a specified application. This document provides recommendations on best practices for successful curing and handling.

**Minimum Bond Line Cure**  
All epoxies must be cured in order to achieve optimal mechanical, electrical, or optical properties. There are numerous methods of curing epoxies (ambient, heat oven, radiant oven, hot plate, heat gun, UV, etc.) and each can result in varying properties. For each product that is developed, a unique cure schedule is devised which includes a "minimum cure" required on each product data sheet. This "minimum cure" is the worst case scenario cure for minimal properties. Furthermore, the "minimum cure" temperature is the actual temperature of the epoxy bond line and should not be confused with the temperature setting of the heating source. It is important to note that the thermal mass of tapes, prep, acetamin, and sprays and slinging the oven door will slow down the cure rate for the epoxy to reach the minimum temperature so special care should be taken to incorporate these thermal lags when determining the optimum curing profile. Curing conditions specified on data sheets are guidelines and should not be confused with specifications. Contact techsupport@epotek.com for cure recommendations per your specific application, process, and product.

Adhesive Type	Minimum Bond Line Thickness	Comments
Die Attach	0.5 to 1.5 mils	Optimal thermal transfer through the thermal pathway is 0.5 to 1 mil. The working and handling time of resin is critical.
Thermal Paste	0.1 to 0.2 mils	The thermal interface resin is not for handling. Handling time is critical. The working and handling time of resin is critical.
Die Attach	0.5 to 1.5 mils	Optimal thermal transfer through the thermal pathway is 0.5 to 1 mil. The working and handling time of resin is critical.

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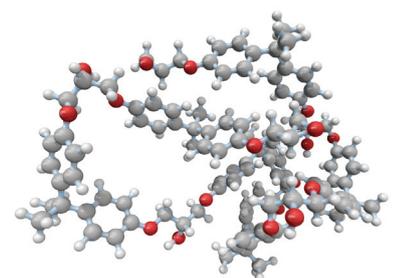
## Effect of Temperature on Cross Linked Density of an Epoxy



To reach the full performance potential of an epoxy adhesive, the user should select a cure schedule that achieves a high cross-link density. Typically, higher temperatures produce a more complete reaction with a greater degree of cross-linking than lower temperatures. Alternatively, increasing the length of time for a lower temperature cure does not always yield the same degree of crosslinking as curing at a higher temperature.

When an adhesive is cured at a lower temperature, the cross-links form slowly resulting in a more expanded network structure. As the structure forms, the mobility of any unreacted groups in the structure gradually decreases and they are impeded from migrating to the remaining open reactive sites. Once enough of this network structure has been locked in, increasing the curing time at a given temperature will not further the degree of cross-linking. On the other hand, extending the cure times for most epoxies will not harm them in any way as cure temperatures are well below the thermal degradation temperatures of the epoxy.

For optimal cross-link density, curing at a high enough temperature will provide sufficient kinetic energy to quickly initiate chemical reactions at even the most hindered locations and give the molecules enough mobility to fully network. High cross-link density formation will enable your cured material to exhibit the best possible mechanical and physical properties for that formulation. Properties such as Tg, hardness, modulus, electrical conductivity, thermal conductivity, strength and degradation temperature will be maximized and properties such as CTE, outgassing, and moisture uptake will be minimized.



Crosslinked Epoxy



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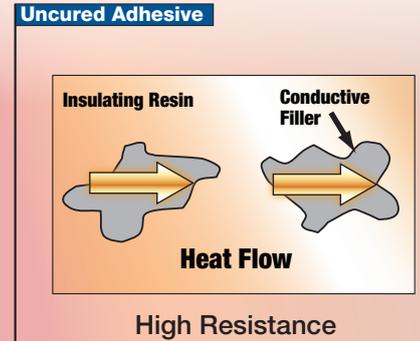
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# Cure Speed Considerations

## General Considerations

The process of transitioning from an uncured liquid state to a cured cross-linked benefit. For instance, Thermal Conductivity and Electrical Conductivity are often

Drawing illustrates the effects of proper cure (shrinkage) for a conductive filler system

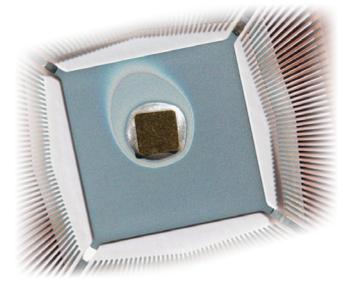


## Snap/Fast/High Temperature Cure



Faster, high temperature cures enable chemical cross-links to form faster and more completely. This generally results in higher shrinkage than lower temperatures. In many cases, this shrinkage can provide benefit. For instance, thermal conductivity and electrical conductivity can be enhanced as explained above.

Snap cures can also provide an added benefit by reducing resin bleed in some situations. Resin bleed is usually a substrate dependent phenomenon and is generally solved through modification of the substrate surface (see *EPO-TEK Tech Tip# 16*), but it may also be impacted, to some degree, by the cure. The surface tension or surface morphology of the substrate may actually attract the resin component of the adhesive to migrate along the substrate surface. However, this capillary-type migration occurs more readily with lower viscosity resins or if the resin is in contact with the substrate for long periods of time before curing. If an adhesive is heated slowly, it will drop in viscosity with increasing temperature until it reaches a high enough temperature for cross-linking to begin and for the adhesive to start building its structure. Higher temperature cures can initiate this cross-linking on a short enough time scale to prevent this initial viscosity drop and deny the resin the time needed to migrate.



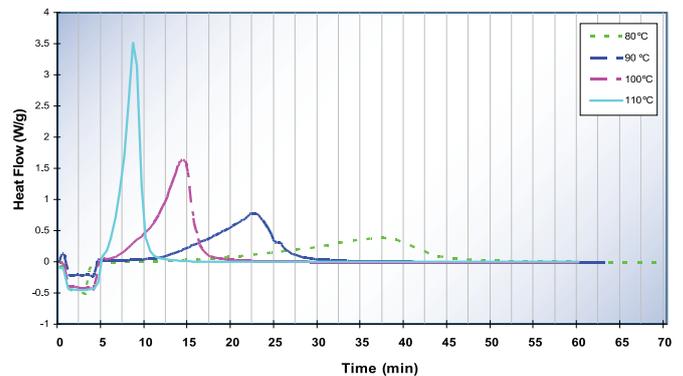
Example of Resin Bleed

## Low Temperature Cure



Many applications are very sensitive to the stress that higher cure temperatures and resulting higher degrees of shrinkage can produce. In those cases, a lower temperature cure should be used. While a low stress state is mostly determined by the chemistry of the adhesive selected, it can also be influenced by the cure. The drawback of lower temperature cures is that they may not yield the desired optimal performance of a highly cross-linked system.

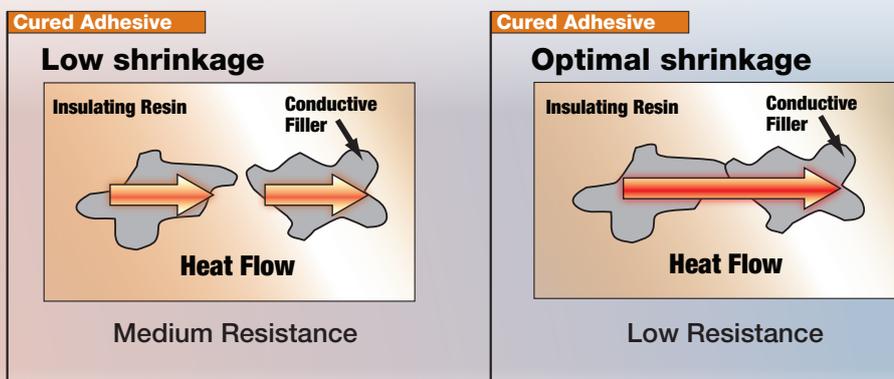
## Isothermal Cure Profiles



Different exotherm peaks for a typical epoxy at 80°C, 90°C, 100°C and 110°C.

# - Snap/Low/Ramped/Staged

solid state will impart some degree of shrinkage to the cured adhesive. In many cases, this shrinkage can provide a n enhanced by increasing the degree of shrinkage during cure as this brings the conductive filler particles closer together.



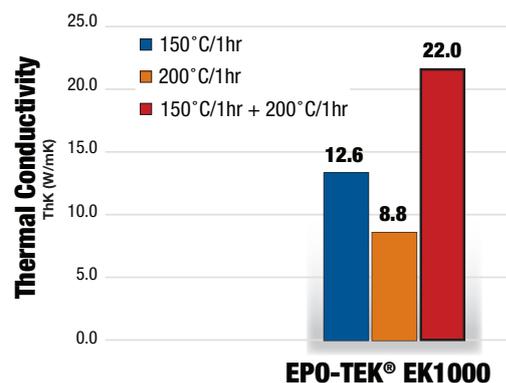
## Impact of cure conditions:

- **Too low** of a cure temperature will result in slow cure and low crosslink density.
- **Too high** of a cure temperature can cause high exotherms that may actually cause the system to expand rather than shrink.
- **Proper** cure conditions are dependent on the chemistry of the adhesives.

## Ramped/Stepped Cure

One good compromise between a high and a low temperature cure is the ramped or stepped cure. In this case, the cure temperature is slowly ramped up to the final desired curing temperature. This often provides a good balance between lower stress as the cross-linked structure is locked in more slowly and a higher overall level of cross-linking as higher temperatures are ultimately achieved. For example, a step cure for EPO-TEK 301-2 would be: 40°C/1 hour followed by 80°C/3 hours. This yields similar performance to EPO-TEK 301-2 cured directly at 80°C, but with lower stress.

In some cases, a significant increase in thermal conductivity can be achieved through a proper stepped cure.



## Staged Cure

An extreme version of a ramped or stepped cure is a staged cure. The adhesive is left for several hours at room temperature to begin to gel and build-up some structure before it is placed into the oven for a higher temperature post cure. This generally works only for adhesives capable of a low temperature cure. This can yield a very low stress final cured state. However, the process needs to be managed carefully as small changes in ambient temperature and humidity during the staging time may have a significant impact on the resulting cured material (see EPO-TEK Tech Tip# 15). As so much of the network forms during the long Room Temperature staging, the final curing density will not be as high as for a direct cured material or even a ramped product. The resulting material may have lower Tg, hardness, modulus, conductivity, and cohesive strength than a higher temperature direct cure product, but it would have enhanced mechanical properties over a product cured only at a lower temperature.



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### Tech 15 Tip

#### The Effect of Relative Humidity and Temperature on VR for Room Temperature Curing ECAs

**What >** The Effect of Relative Humidity and Temperature on Volume Resistivity for Room Temperature Curing ECAs

**Why >** It could be your environment.

Room Temperature Curing Electrically Conductive Adhesives (RT ECA) are an attractive option for use with substrates and components that cannot withstand typical EPO-TEK® 1226's cure conditions. Although room temperature curing ECAs can provide similar performance to heat curing ECAs, there are guidelines that need to be taken into consideration when using these adhesives.

**Here are the guidelines:**

1. Resin and hardener proportions (into serial) should always adhere to the data sheet values. For all types of epoxy, hardener and resin must be mixed exactly as prescribed. Even a slight difference may cause the epoxy preparation to change. (See Tech Tip #11 - Concerning Mix Ratio by Weight or Mix Ratio by Volume if needed)
2. Room temperature conditions can vary depending on your geographical location and laboratory and/or manufacturing conditions. It is important to maintain a temperature between 23°C and 27°C (73°F - 81°F). Significant temperature fluctuations can affect the cure rate, potentially changing the rate of cross-linking as well as the cross-linking density.
3. Relative Humidity (RH) conditions are especially critical for curing room temperature ECAs to ensure optimal electrical conductivity. This is achieved by maintaining an RH between 40% and 60%.

**Why Proper Mix Ratio and Temperature are Required**

In order for an ECA to achieve optimal electrical conductivity, it must be well cross-linked. This requires that the material be mixed at the proper ratio of A and B and that this cross-linking occurs fairly rapidly and generally means that all of the components stay homogeneously mixed throughout the cure.

Room temp ECAs cross-link much more slowly, allowing more time for the cure to be affected if the correct environmental controls are not in place. As the RT ECA is curing, the lower surface tension components of the adhesive migrate to the higher surface tension interface, the higher being the open air. What this happens in excess, the adhesive cannot properly crosslink. It may eventually harden, but it may not have good electrical conductivity and may even increase in resistance if you cure at a higher rate will be too slow to allow sufficient cure.

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## Mass Consideration on Curing

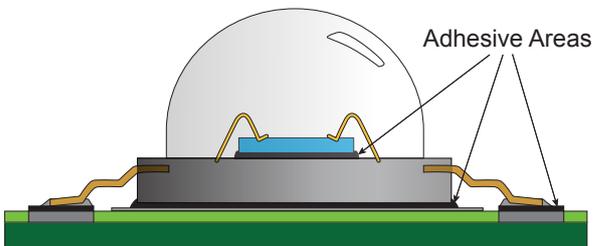
Epoxies cure through an auto-catalytic process. This means that heat is generated during bond formation and the initial cross-linking process. This generated heat speeds up the curing reaction, which then generates additional heat, speeds up the reaction further and so on. The larger the volume of reacting material, the faster it will cure and the more heat is built up during the cure.

Large masses of materials may build up so much heat during curing that a runaway reaction occurs and they actually expand during the cure. This is referred to as an excessive exotherm. In extreme cases, highly exothermed materials may appear to have “foamed” due to the large amounts of air present in the expanded state. Severely exothermed materials may exhibit weakened mechanical properties as they have essentially become a foamed system. Loss in cohesive strength, decreased modulus and hardness, and increased CTE can occur. Both electrical and thermal conductivities may also suffer.

Ramped or stepped cures are a good way to avoid runaway reactions for curing large volumes of materials. This slows down the reaction rate, but still enables the material to see the high ultimate curing temperatures needed for full cross-linking.

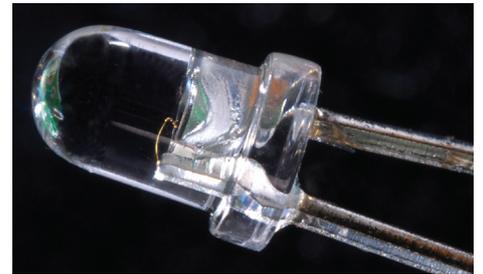
Very small volumes of material should be cured at higher than usual temperatures and most likely for longer times in order to ensure proper curing. As there is no risk of a runaway reaction with the small volume (unless temperatures used are excessive) it is unlikely that high stress is involved at the small size scales. These applications should generally employ a direct, high temperature cure.

An example of this would be a 9mil x 9mil die for die attach in high brightness LEDs. Here the adhesive has a much higher ratio of surface area to volume. The result is that any extra heat generated by the epoxy curing process is quickly wicked away through the high surface area.



Light Emitting Diode (LED)

Another factor to consider is whether there are any large fixtures or thermal masses that are part of the curing process such as a heat sink. Any such masses will absorb heat during the cure and it may take longer for the heat to reach the adhesive being cured. As a result, the temperature profile seen by the adhesive during a direct cure may effectively look more like a ramped cure as it takes more time for the entire mass to heat up to the oven temperature. Cure temperatures and/ or times should be increased for processes involving large thermal masses.



Epoxy Exotherm

### Special Standards / Testing Notes:

For products tested to meet specific standards for example; USP Class VI or NASA low outgassing, it is important to note how the product was cured for these tests as alternate cure schedules may not deliver the same performance.

Please consult our Technical Services Experts at Epoxy Technology for any questions or assistance you may need in working with our EPO-TEK® adhesives.

Contact Information: [techserv@epotek.com](mailto:techserv@epotek.com) or 978-667-3805.

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