Cure Matters

Determining the **Proper** Cure Schedule

EPOXY TECHNOLOGY
A Meridian Adhesives Group Company

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

Epoxy Technology'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.

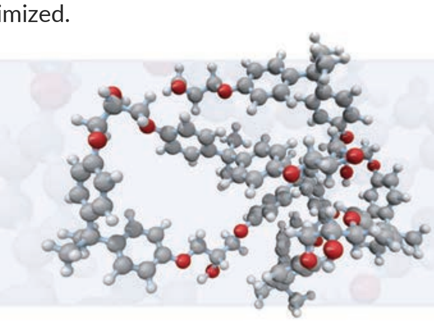
Effect of Temperature on Cross-Linked Density of an Epoxy

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

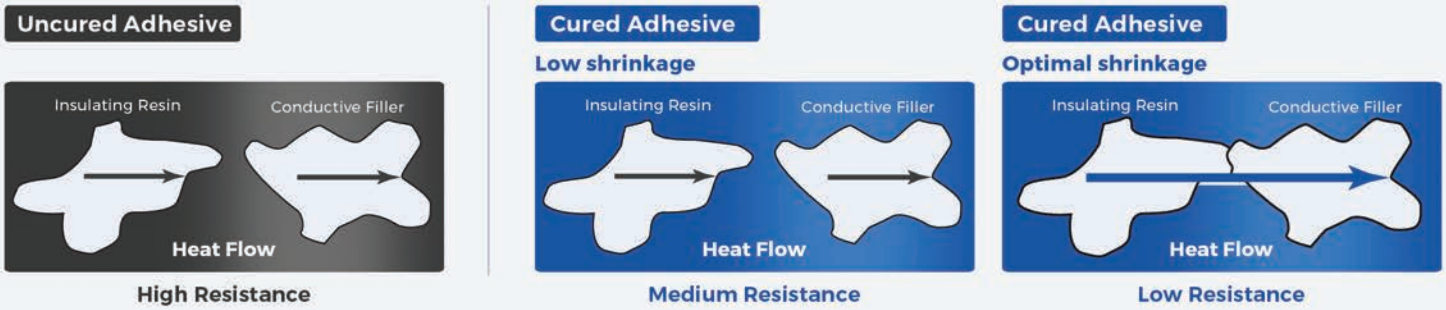


Cure Speed Considerations
Snap/Low/Ramped/Staged

General Considerations

The process of transitioning from an uncured liquid state to a cured cross-linked solid state will impart some degree of shrinkage to the cured adhesive. In many cases, this shrinkage can provide a benefit. For instance, Thermal Conductivity and Electrical Conductivity are often enhanced by increasing the degree of shrinkage during cure as this brings the conductive filler particles closer together.

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



Impact of cure conditions:

- **Too low** of a cure temperature will result in slow cure and low cross-link 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.

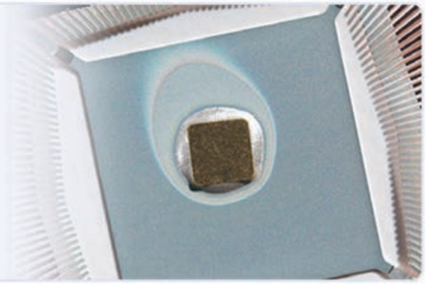
Snap/Fast/High Temperature Cure

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.

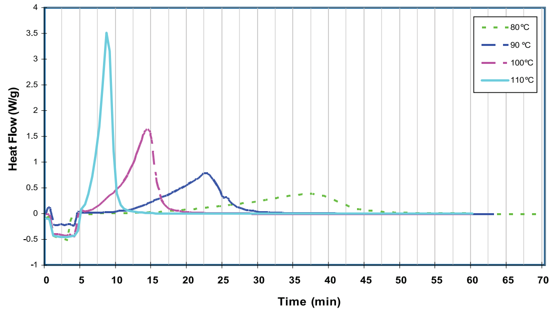
Example of Resin Bleed



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



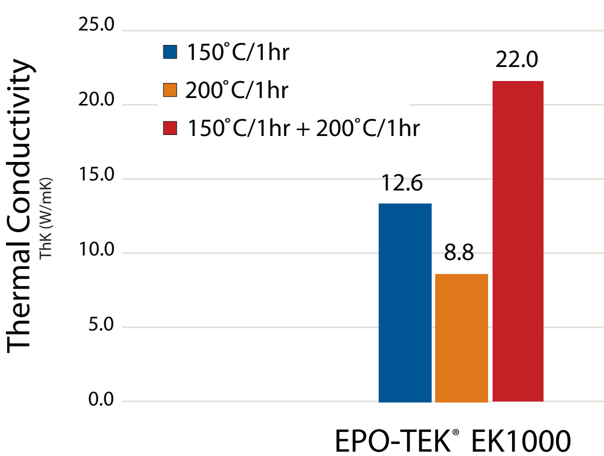
Different exotherm peaks for a typical epoxy at 80°C, 90°C, 100°C and 110°C.

Cure Speed Considerations
Ramped/Stepped/Staged

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.

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.

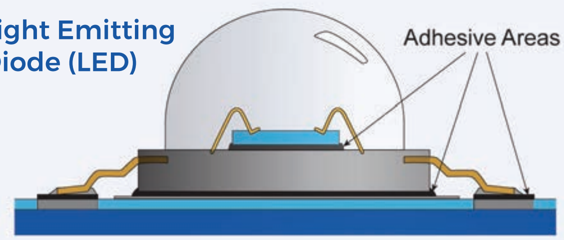
Very small volumes of material should be cured at higher than usual temperatures and most likely for longer times in order to ensure proper curing. As there is no risk of a runaway reaction with the small volume (unless temperatures used are excessive) it is unlikely that high stress is involved at the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

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

Light Emitting Diode (LED)



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:

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