# *Differential Scanning Calorimetry (DSC) for the Characterization of Curing Behavior of Epoxy Thermosetting Adhesives*

## 1. Background

Epoxy-based thermosetting adhesives are essential materials in modern manufacturing.(1) Their combination of high mechanical strength, chemical resistance, dimensional stability, and strong adhesion to a wide range of substrates has made them indispensable in electronic packaging (EMC), automotive structural lightweighting, fiber-reinforced composites, high-performance coatings, and general industrial bonding applications.(2)

The final properties of an epoxy system, such as glass transition temperature ($T_g$), modulus, strength, thermal resistance, and long-term durability, are not dictated solely by the raw resin and curing agent. Instead, they depend almost entirely on the curing process, in which epoxy groups react with curing agents (e.g., amines, anhydrides) to form a three-dimensional cross-linked network.(3) The rate and degree of curing determine the integrity of this network, which in turn governs the reliability and performance of the adhesive. As a result, accurate characterization, monitoring, and optimization of the curing process are critical steps in ensuring product quality.

Thermal analysis is central to this effort. Among available techniques, differential scanning calorimetry (DSC) is one of the most powerful tools for evaluating epoxy curing behavior.(4) DSC directly measures the reaction enthalpy (ΔH) associated with the curing reaction, enabling calculation of the degree of cure. It also identifies the onset, peak, and end temperatures of the curing process—parameters that guide the design of curing schedules (heating rates, hold temperatures, dwell times). In addition, DSC provides quantitative $T_g$ measurements for uncured, partially cured, and fully cured states, allowing users to correlate curing progression with final material performance.(5)

In this *AMI Note*, we demonstrate how DSC can be used to comprehensively characterize the curing behavior of epoxy thermosetting adhesives using both dynamic-temperature and isothermal-curing modes, and we discuss the relationship between degree of cure and glass transition temperature ($T_g$) as a practical basis for process optimization.

## 2. Experiment

The curing behavior of the two-component epoxy resin and curing-agent system was evaluated using the **AMI DSC 600** differential scanning calorimeter. The procedure was as follows: at room temperature, the epoxy resin and curing agent were thoroughly mixed at a mass ratio of 10:3. After allowing entrapped air to dissipate, a 5–10 mg portion of the mixture was transferred into a sealed aluminum crucible. All measurements were conducted under a nitrogen atmosphere. For dynamic-temperature curing, samples were heated at different heating rates over a temperature range of –40 °C to 350 °C to study the evolution of curing reactions. For isothermal curing experiments, the DSC instrument was first stabilized at 100 °C; the sample crucible was then quickly placed onto the sensor, and data acquisition was initiated immediately.

## 3. Results

### 3.1 Dynamic heating and curing analysis

Dynamic DSC curing curves provide two major benefits for industrial applications:

✓ Enables rapid screening and quality control of epoxy formulations. By comparing peak temperature ($T_p$) and reaction enthalpy ($\Delta H$) across batches, differences in reactivity or functional-group availability can be quickly identified.

✓ Defines the appropriate curing process window. The onset temperature ($T_0$) and peak temperature ($T_p$) provide essential reference points for designing heating schedules and selecting curing temperatures.

Dynamic DSC scans were performed at heating rates of 10, 20, 40, and 60 °C/min, as shown in Figure 1. As the heating rate increased, the exothermic peak shifted to higher temperatures. For example, $T_p$ increased from 142.71 °C at 10 °C/min to 175.95 °C at 60 °C/min. This shift reflects a typical kinetic effect: at higher heating rates, the system absorbs heat more rapidly, causing the reaction to lag and resulting in higher characteristic temperatures.

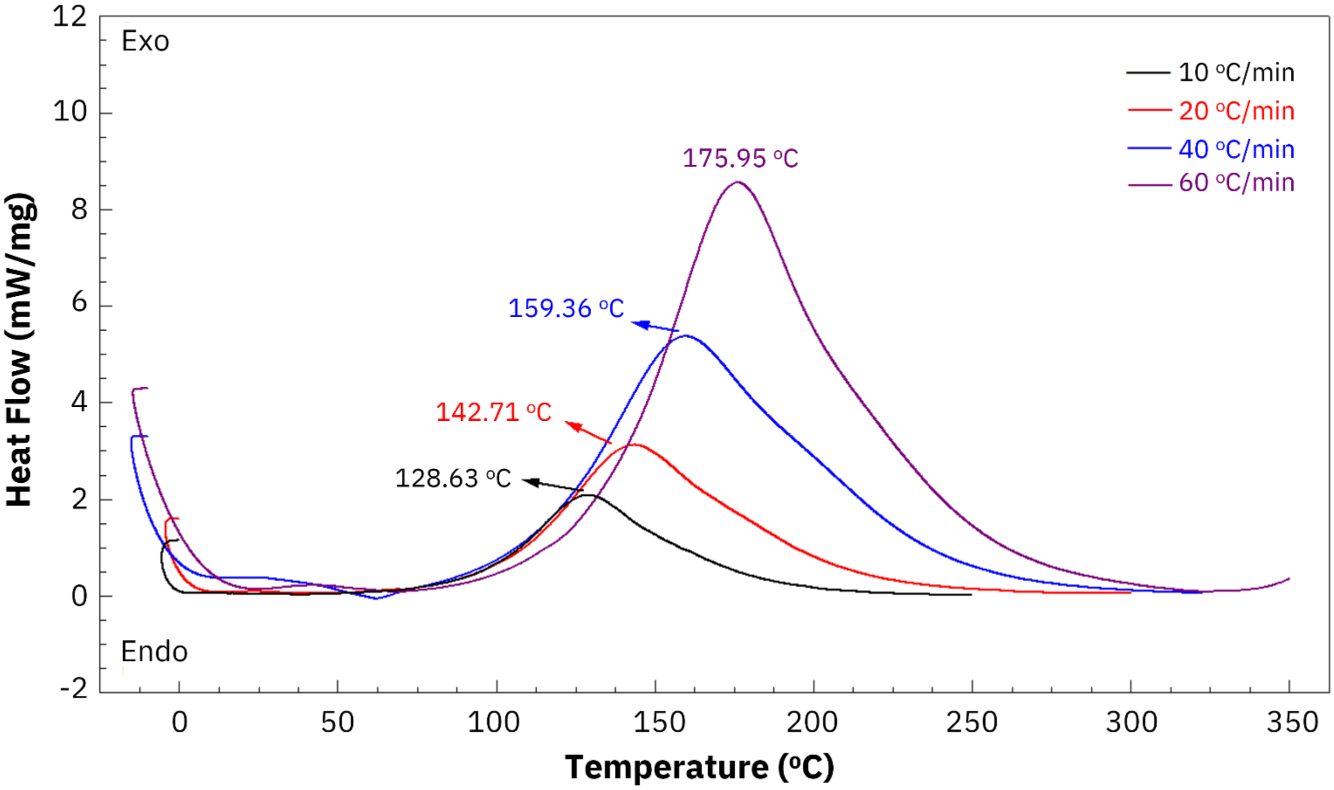

*Figure 1: Change in curing peak at different heating rates*

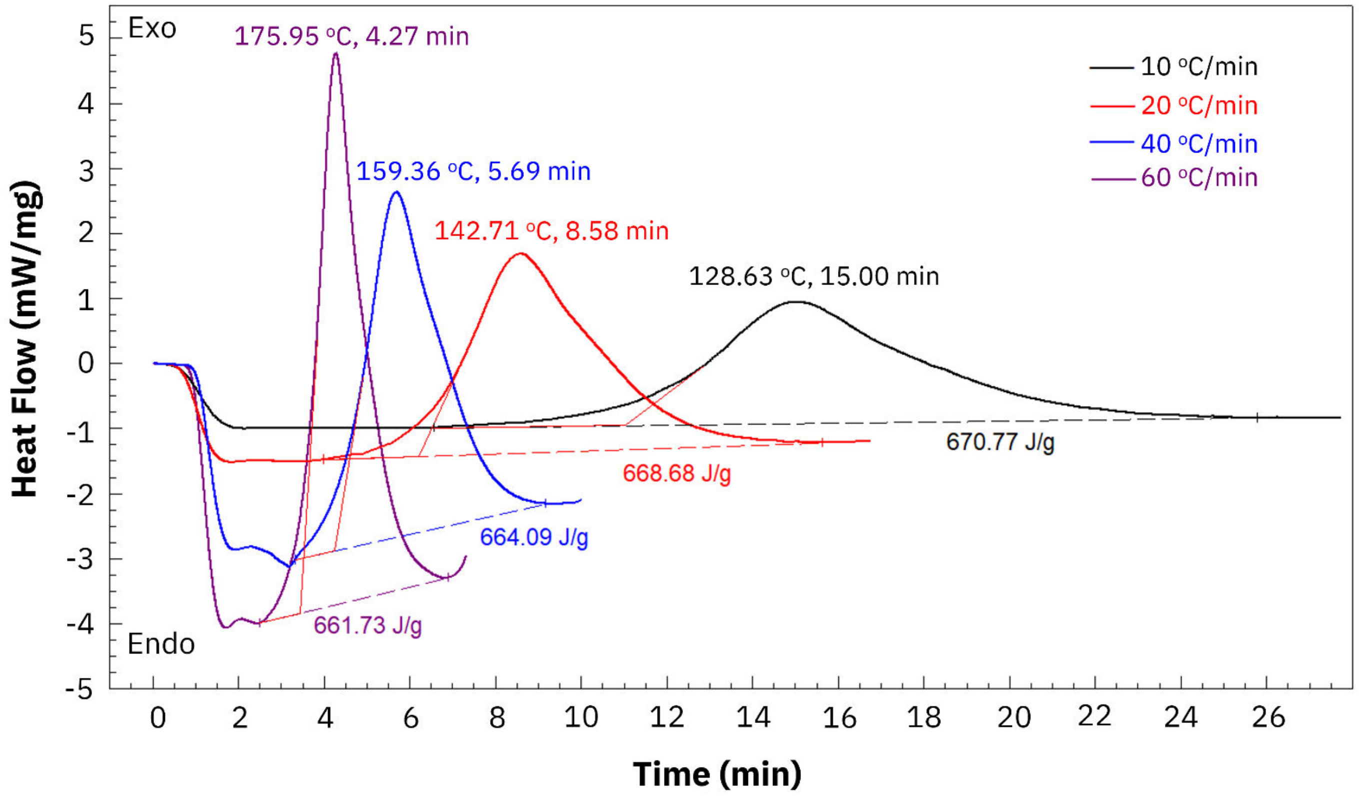

*Figure 2: Curing peaks vs time at increasing heating rates;* ***DSC 600*** *analysis software was used to calculate the curing exothermic enthalpy value of epoxy resin adhesive at different heating rates*

Using time as the horizontal axis, the **DSC 600** analysis software was used to calculate the reaction enthalpy at each heating rate. Despite the different heating rates, the total reaction enthalpy remained nearly constant (660–670 J/g), indicating that the rate of temperature increase does not significantly influence the total heat released. This confirms that dynamic DSC is reliable for evaluating the degree of cure. For subsequent analysis, the enthalpy measured at 10 °C/min is defined as 100% curing, and isothermal curing degrees are calculated relative to this value.

### 3.2 Isothermal curing analysis

Isothermal curing curves also provide valuable information for practical adhesive formulation:

- ✓ Determine the minimum temperature required for effective curing and whether the resin system can react adequately at a target temperature.
- ✓ For two-component adhesives, low-temperature isothermal data simulate the reaction behavior immediately after mixing at room temperature; longer times to reach the exothermic peak correspond to longer workable pot life.
- ✓ Provide quantitative guidance for selecting appropriate curing temperatures and hold times, helping to avoid issues such as under-curing or over-curing.

As shown in Figure 2, isothermal DSC measurements were performed on the same epoxy resin system at 100 °C, 130 °C, and 180 °C. The results clearly demonstrate the strong influence of temperature on curing kinetics. At 180 °C, the curing reaction proceeded extremely rapidly, with the heat-flow curve reaching its maximum exothermic peak within only 2–3 minutes, reflecting very high reactivity and an exceptionally short curing cycle at this elevated temperature. At 130 °C, the reaction rate slowed noticeably, the time to reach the exothermic peak increased, and the peak intensity decreased. At 100 °C, the curing process was the slowest, characterized by a broad and low exothermic peak and the longest time to reach maximum heat release.

The cure-rate-versus-time curves (shown in the upper-right inset of Figure 3) make this trend especially clear: higher temperatures result in steeper slopes and significantly shorter times to achieve a given degree of cure. For example, reaching 60 percent conversion requires only a few minutes at 180 °C, but can require tens of minutes or more at 100 °C.

### 3.3 Relationship between degree of cure and $T_g$

Industry applications of cure-$T_g$ analysis include the following:

- ✓ Determining the endpoint of the curing process. By verifying whether the $T_g$ of the cured material reaches the expected value, manufacturers can assess whether curing is complete and whether the product meets specification.

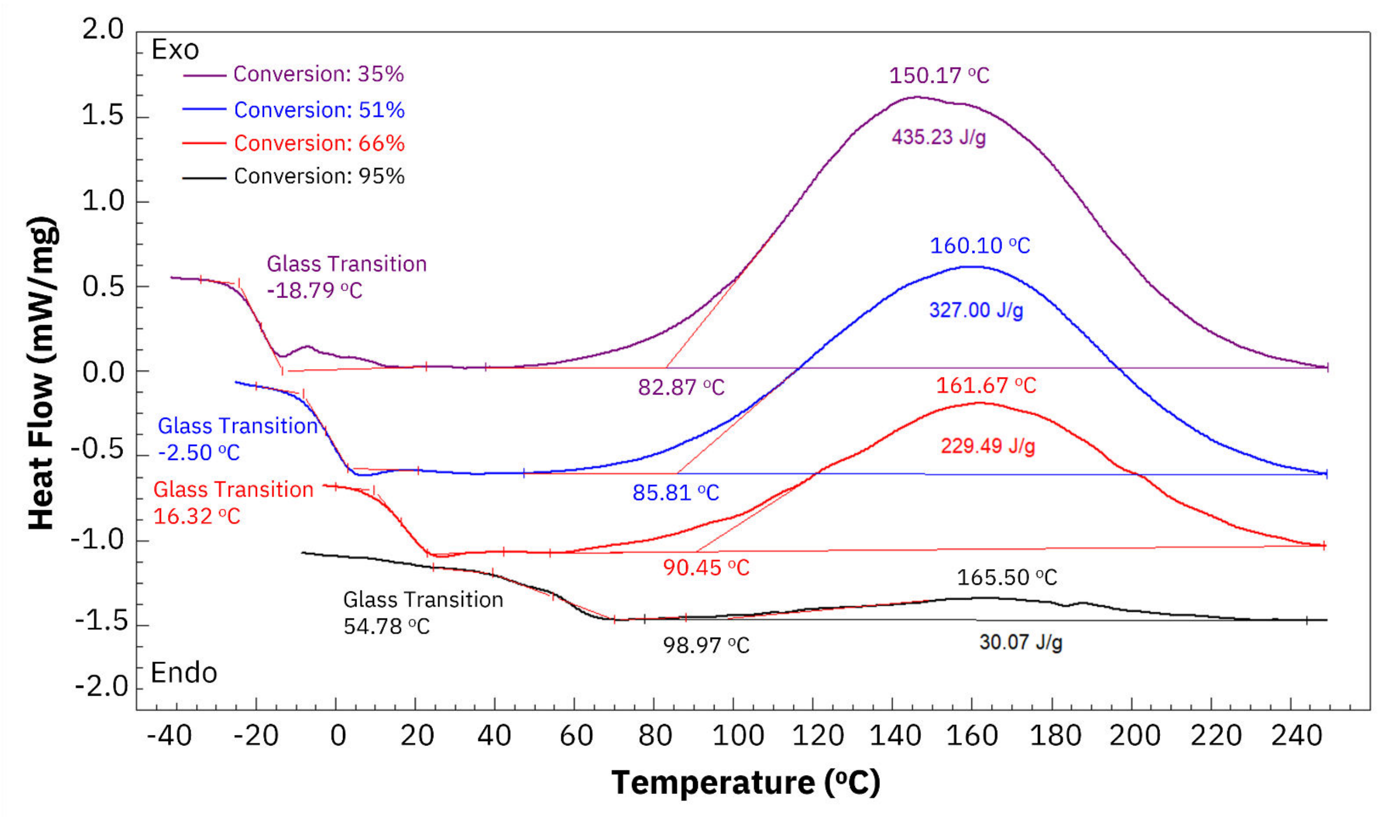

*Figure 3: Isothermal curing DSC curves at different temperatures; Inset: Degree of curing vs curing time*

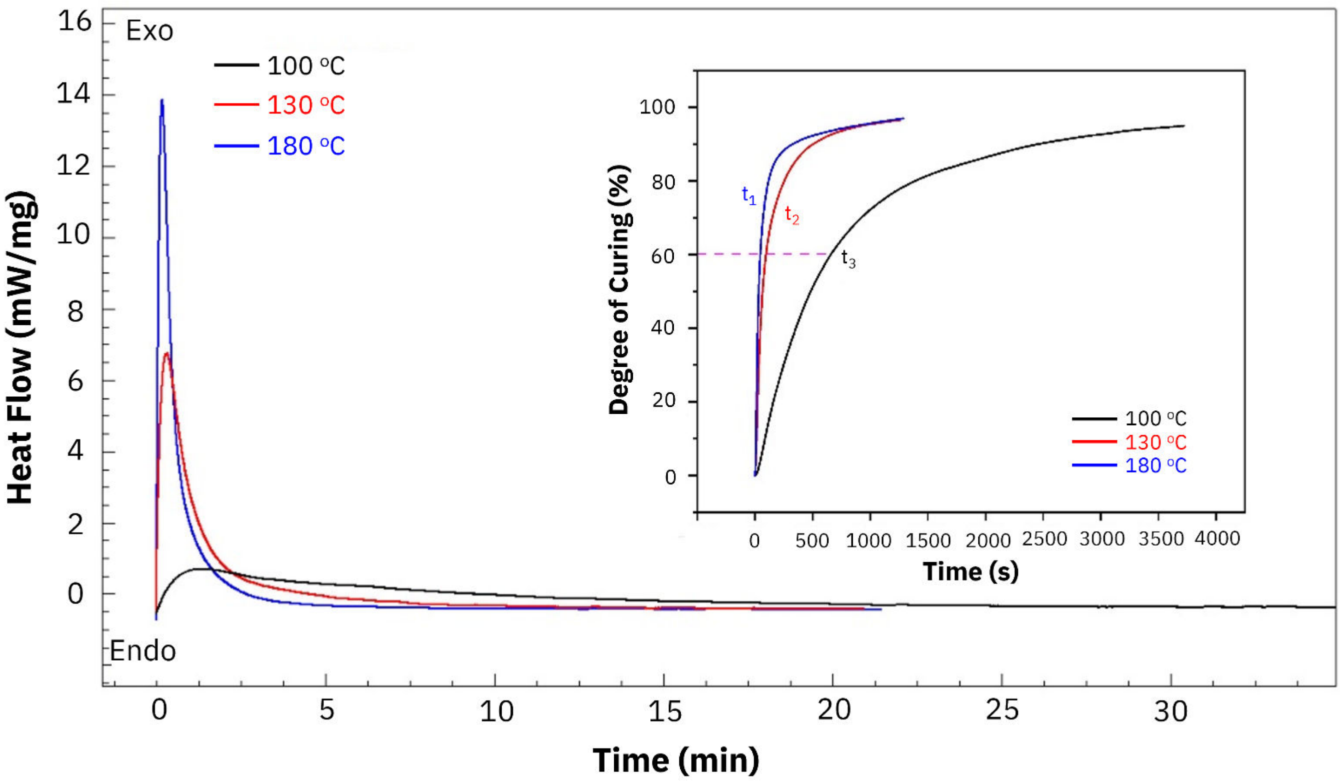

*Figure 4: DSC curves for samples cured to different degrees, heating rate 20 °C/min*

- ✓ Establishing a predictive cure–$T_g$ model. Measuring $T_g$ in partially cured samples allows the current degree of cure to be inferred, providing a non-destructive tool for process monitoring and in-line quality control.

- ✓ Predicting final performance. $T_g$ strongly correlates with mechanical properties such as modulus and strength; therefore, $T_g$ measurements offer a practical way to estimate the final service performance of the adhesive.

As shown in Figure 4, DSC measurements were performed on epoxy samples cured to different conversion levels (35%, 51%, 66%, and 95%) to evaluate their corresponding glass transition temperatures ($T_g$). At low degrees of cure, the network contains few cross-link points and the polymer chains retain significant mobility, resulting in the lowest $T_g$ (approximately −18.79 °C). As curing progresses and the three-dimensional network begins to form, chain mobility becomes increasingly restricted, and $T_g$ rises accordingly; samples cured to 51% and 66% showed $T_g$ values of −2.5 °C and 16.32 °C, respectively. At high conversion (≈95%), the network approaches full cross-link density, free volume decreases substantially, and chain-segment motion becomes highly constrained, raising $T_g$ to 54.78 °C. At this stage, only a small residual exotherm ($\Delta H \approx 30$ J/g) remains on the DSC curve, indicating that the reaction is nearly complete.

This behavior reflects the fundamental definition of $T_g$: the temperature at which polymer chain segments transition from a frozen to a mobile state. As the degree of cure increases, the growing cross-linked network effectively “locks” the chains in place, requiring higher thermal energy to initiate motion; thus $T_g$ increases. The fully cured $T_g$ is an intrinsic property of the epoxy formulation and serves as a key reference point for assessing cure completeness and final performance.

## 4. Conclusions

Differential scanning calorimetry (DSC) is an essential technique in the development, optimization, and quality control of epoxy adhesives. Isothermal curing analysis provides direct insight into how a formulation reacts at a specific temperature, supplying the data needed to determine workable pot life and define practical curing cycles. Dynamic DSC measurements offer complementary information, including characteristic temperatures, total reaction enthalpy, and activation-energy trends, forming the basis for kinetic modeling and establishing a reliable curing process window. Tracking the evolution of the glass transition temperature ($T_g$) throughout the cure links the degree of cure to final mechanical and thermal performance, enabling accurate assessment of product quality.

- ✓ High-sensitivity heat flow sensor
- ✓ Temperature control accuracy ± 0.01 °C
- ✓ Intuitive software interface

**DSC 600**

*Figure 5: Highlighted features of* ***DSC 600*** *from* ***AMI***

The **AMI DSC 600** is designed for this type of analysis. Its stable baseline, high sensitivity, and precise temperature-control capability allow curing exotherms to be captured cleanly across both dynamic-heating and isothermal modes. The wide operational temperature range accommodates low-temperature pot-life evaluation as well as high-temperature cure-schedule design, while the instrument’s fast response enables accurate detection of onset, peak, and end temperatures even for rapid reactions. These features make the **DSC 600** an ideal tool for formulating, validating, and monitoring epoxy adhesive systems where curing behavior directly determines final product performance.

## 5. References

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