APPLICATION NOTE

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# NEXTA DSC

## DSC measurements of epoxy resins

### INTRODUCTION

Epoxy resins are used as adhesives, thanks to their excellent adherence to many different types of surfaces. When cured, epoxy adhesives are extremely chemical- and wear-resistant, as well as being able to withstand a constant force over a long time. One part and two-part epoxies are available, however in this application we'll focus on the two-part systems that comprise of a resin and a hardener.

Epoxies are used as adhesives and fillers for many materials, such as metal, glass, ceramic and plastic. This means they are used in many industries where bonding or sealing is required. Individual epoxy resin systems are designed to give specific properties depending on the application. Examples of these are fast curing, ability to withstand extremes of temperatures and even flame resistance.

When the epoxy resin is mixed with the hardener, the curing process begins to take place. The epoxy group starts a polymerization reaction between the resin and hardener, creating cross-links and the mixed compounds harden. The properties of the cured epoxy are dependent on the chemistry of the system and the degree of cross-linking that occurs during the curing process. It's extremely important that curing has fully completed to ensure the final epoxy resin shows the desired properties.

Differential scanning calorimetry (DSC) can investigate various characteristics of epoxy adhesives, including glass transition temperatures before and after curing, as well as the temperature and reaction kinetics during the curing process. The NEXTA DSC differential scanning calorimeter by Hitachi High-Tech is ideal for analyzing the thermal behavior of epoxies during the curing process, thanks to its excellent sensitivity, resolution and advanced operation, such as modulated DSC.

Hitachi High-Tech Analytical Science's family of thermal analyzers have been employed in the field for more than 45 years, delivering world-class performance for precise materials and process characterization measurements, such as epoxy adhesive analysis.

# HITACHI INSTRUMENTS FOR THERMAL ANALYSIS OF EPOXY RESINS

### NEXTA DSC

The NEXTA DSC200 is a high-performance differential scanning calorimeter, designed to be easy to use and robust enough for high-volume operation within an epoxy resin production environment. High sensitivity and world-class baseline performance allow the analyzer to detect the tiniest of reactions when evaluating epoxy resins. The NEXTA DSC200 is an extremely versatile instrument, with options for automated operation and can be expanded post-installation for new applications.

For epoxy resin analysis, the unique furnace and sensor design delivers excellent baseline stability for repeatable results and ultra-low sensitivity to detect the smallest thermal event. This also gives you excellent resolution for the results, ensuring you can identify and isolate thermal evens that happen very close together.

In addition to the low noise and excellent sensitivity of the NEXTA  DSC200, the instrument is very low cost to run, with very few consumables needed. The robust design also ensures minimal downtime, supporting fast-paced epoxy production.

The auto-analysis function, together with the auto-sampler for up to 50 samples, helps to speed up analysis time and free up operators. And the unique RealView camera system allows you to watch epoxy curing in real time, which can be invaluable for troubleshooting or the development of new materials.

## ALL-INCLUSIVE SOFTWARE, INCLUDING MODULATED DSC CAPABILITY

The NEXTA DSC range comes with Hitachi's intuitive and advanced NEXTA TA software, which gives you options for how you need to operate the instrument. New users are able to get reliable and accurate results and experienced operators can use the NEXTA DSC for more advanced analysis. All modules are included with the instrument, so if you decide to expand your use into new applications, you won't have to purchase additional modules. The software includes three modes of operation:

- Guidance mode for step by step measurement and analysis including a calibration wizard.
- Simple mode for more experienced users carrying out routine analysis that requires a simple interface. All important features are available on the main screen.
- Standard mode where all modules are included (including modulated DSC) and more complex analysis can be set up.

## PERFORMANCE AND RESULTS

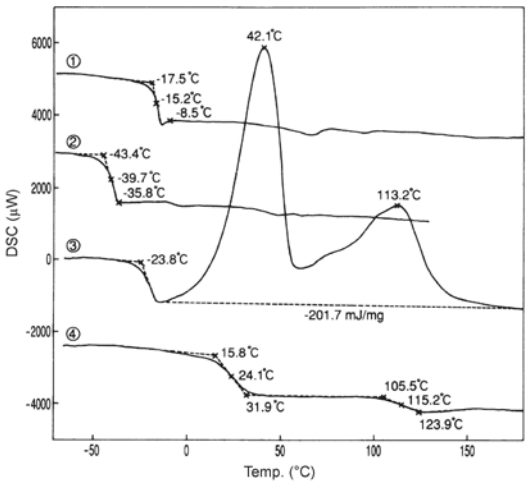
The NEXTA DSC200 was used to evaluate the thermal behavior of two different types of two-part epoxy adhesive. Sample 1 was a 5-minute epoxy adhesive, where curing begins after 5 minutes at 20ºC, and Sample 2 where curing begins after 30 minutes at 20ºC.

Experiment conditions are as follows:

- **Sample weight:** 10 mg
- **Sample pan:** Aluminum, open pan
- **Heating rate:** 10ºC/min
- **Atmosphere:** Nitrogen

### DSC curves for sample 1

From figure 1 we can see that glass transition occurred at -17.5ºC and -43.4ºC for the base compound and curing agent respectively. For the mixed sample, glass transition occurred at -23.8ºC and exothermic curing peaks are seen at 42ºC and 113ºC. These results indicate that the curing reaction started at room temperature, however the process had not completed after 5 minutes. From curve 4 we can see the behavior of the cured epoxy under further heating. No exothermic peaks are seen, however glass transitions at 18.5ºC and 105.5ºC suggests that this sample contains multiple components.

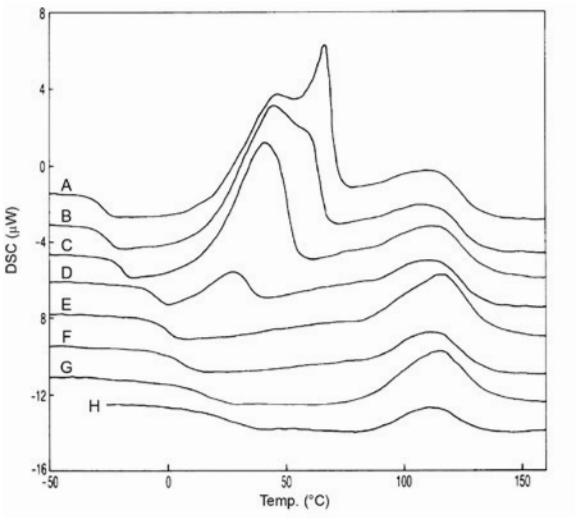



(1) Base compound
(2) Curing agent
(3) Base compound and curing agent mixed and left to stand for 5 minutes
(4) 2nd heating for (3) sample

**Figure 1** DSC curves for the 5-minute type adhesive

**DSC curves for sample 1 at various intervals after mixing**

In this experiment, the epoxy was mixed and left to cure at room temperature for different amounts of time before measuring. The results in figure 2 show that the longer the curing time, the higher the glass transition temperature. Exothermic curing peaks were also seen, suggesting that the sample had not completely hardened even after being left for 1 day. The difference in exothermic peak shape and temperature could be due to the degree of polymerization in each sample.




A : 2min,
B : 3min,
C : 5min,
D : 10min,
E : 15min,
F : 30min,
G : 4hr,
H : 1day

**Figure 2** DSC curves for the 5-minute type adhesive after mixing

**Glass transition temperature as a function of curing time for sample 1**

Figure 3 shows the effect of the glass transition temperature from curing time. This type of analysis allows you to evaluate the effectiveness of the curing time.

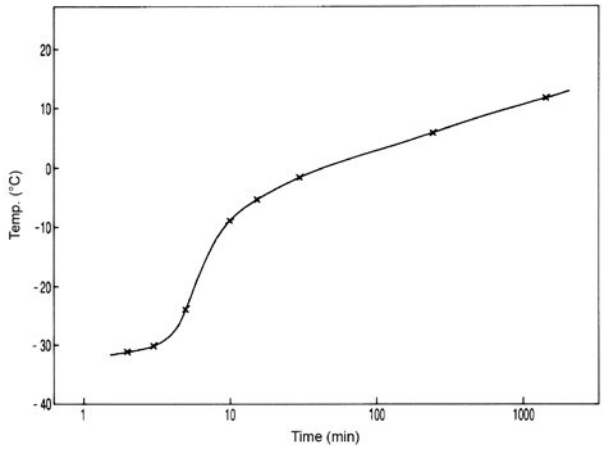
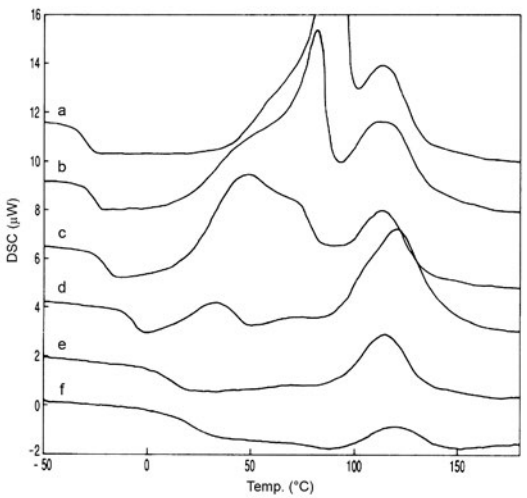



**Figure 3** Curing time related to glass transition temperature for the 5-minute type adhesive

**DSC curves for sample 2 at various intervals after mixing**

This is the same experiment as figure 2 above, but for sample 2. As with sample 1, different curing times produced different glass transition temperatures and exothermic curing peaks. Comparing figure 4 with figure 2, we can see that the 30-minute adhesive needed more time to harden at room temperature than the 5-minute type. Again, this type of measurement determines whether the material has cured completely.




a : 5min,
b : 15min,
c : 20min,
d : 30min,
e : 1hr,
f : 1day

**Figure 4** DSC curves for the 30-minute type adhesive after mixing

## SUMMARY

The **NEXTA DSC200** reliably delivers curing analysis of epoxy resins. Excellent baseline performance and high sensitivity makes it easy to determine whether curing has completed and evaluate hardened material for glass transition temperature. Simple to use and robust, the NEXTA DSC includes advanced features, such as modulated DSC, for comprehensive thermal analysis.

Many applications have been optimized for Hitachi High-Tech Analytical Science's thermal analyzers. For more information on other applications, please contact our experts at **contact@hitachi-hightech.com**.

## NEXTA DSC SERIES: HIGH ACCURACY MATERIALS CHARACTERIZATION

Designed for accurate determination melting point, glass transition and crystallization temperatures, our range of differential scanning calorimeters deliver excellent sensitivity and baseline flatness.

The NEXTA DSC range offers:

- High sensitivity and baseline performance, with unique furnace design for accuracy Real View camera system that allows you to watch material behavior on screen
- Intuitive, easy-to-use software, with advanced functionality for specific applications
- Reliable auto-sampler testing and auto analysis function for faster testing
- High degree of flexibility, allowing for addition of options after installation

Visit **www.hitachi-hightech.com/hha** for more information.