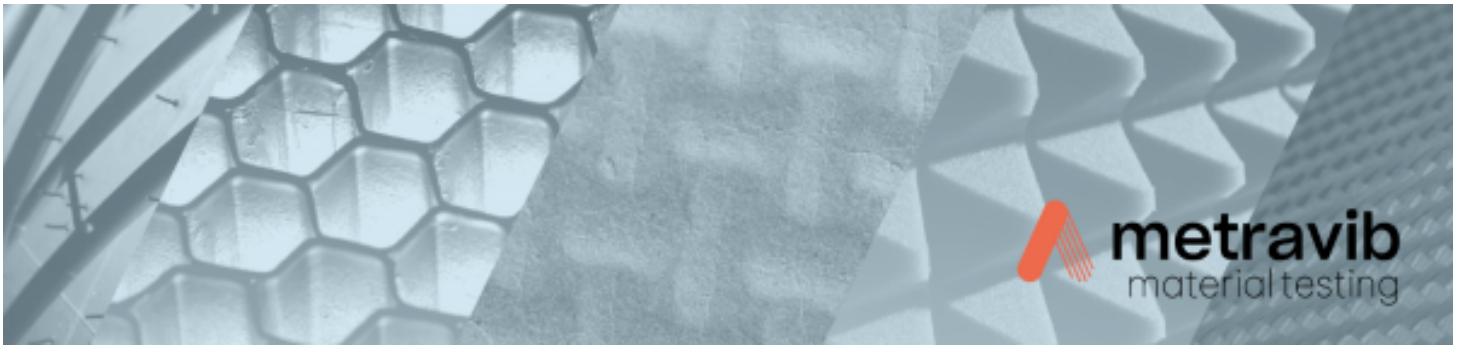
Curing Quality Control with DMA

Keywords: DMA, Temperature sweep, Glass transition, Curing effect, polymers, viscoelasticity

Figure 1. Metravib DMA+1000

Introduction

Mistakes in the conception of composite materials could lead to disappointing mechanical properties. DMA is the perfect tool to determine the properties of a large variety of materials. Among the others, DMA is the most reliable instrument for glass transition temperature (Tg) measurement. The glass transition temperature of a material is the change from a hard and relatively brittle “glassy“ state into a viscous or rubbery state. This transition is highly dependent on the chemical structure and composition of the material, and even small differences can lead to important differences in Tg values. As different operating methods could lead to various chemical structures in the material, the glass transition value can be used to spot manufacturing defects. In other words, DMA can be a reliable instrument for chemical process control, such curing. This paper illustrates how a failing curing process can be determined via DMA measurement.



Materials & methods

Specimens

Epoxy materials were studied with a DMA+1000 in tension mode. A rectangular band of epoxy with 5 x 5 x 1 mm^3 dimensions was cut.

Methods

A sinusoidal dynamic displacement is applied on the specimen from 25˚C to 250˚C (see Figure 2 and Table 1) at 2˚C/min. A small dynamic strain was applied in order to stay in the linear domain of the material, which is recommended during tests that aim to determine the glass transition of the material. A static strain was applied in order to avoid the buckling and to keep the specimen slightly stretched. This test was then repeated a second time on the same specimen in order to observe the change induced by the curing in the thermal chamber of the DMA during the first run.

Dynamic	0.1 % Strain
Frequency	1 Hz
Static	0.2% Strain
Temperature	From 25˚C to 250˚C, 2˚C/min

Table 1. The setting Parameters of the strain sweep preliminary test

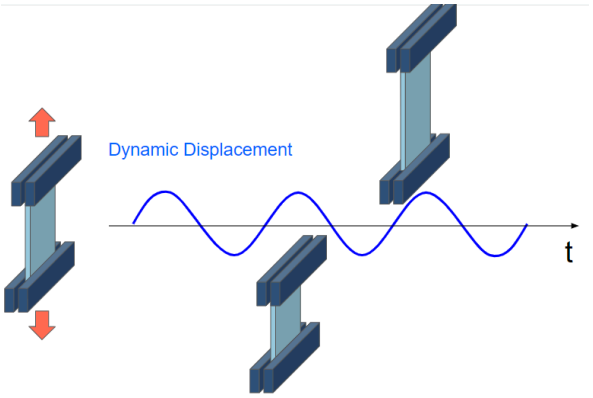


Figure 2. Graphical representation of the dynamic mechanical displacement applied to the specimen during the temperature sweep

Results

Figure 3 shows the modulus of elasticity E' and Tan δ as a function of the temperature at 1 Hz from 25˚C to 250˚C. The two successive measurements are depicted on the graph. During the first measurement, a decrease of the modulus in two steps is observed, the first one is observed above 100˚C and the second one slightly before 150˚C. Two peaks of Tan δ are observed around 100˚C and 150˚C. These results, characterized by two different glass transitions, are typical of a blend of two polymers in a material, each change in E' or Tan δ being attributed to one of the parts of the material. The second run depicts a completely different behavior: E' decrease and the Tan δ peak are not observed anymore at 100˚C, only the ones observed at 150˚C remain. In other words, the first measurement depicts an

unusual 2 peaks of Tan δ for the epoxy matrix, which is not the case during the second measurement.

Here we focus on the fact that the temperature in the thermal chamber is enough to finish the cure of the specimen. From this, it is clear that the difference observed between the two runs on the same specimen come from this curing effect and the consequence on the chemical structure. Here we assume that the Tg observed at 100°C is attributed to the "uncured part" of the material and this part is turned into a "cured part" during the first run, which is why the change at 100°C is not observed during the second measurement. We can also highlight that the height of the tan delta peak in the second measurement is higher than in the first, reflecting a distinct viscoelastic behavior.

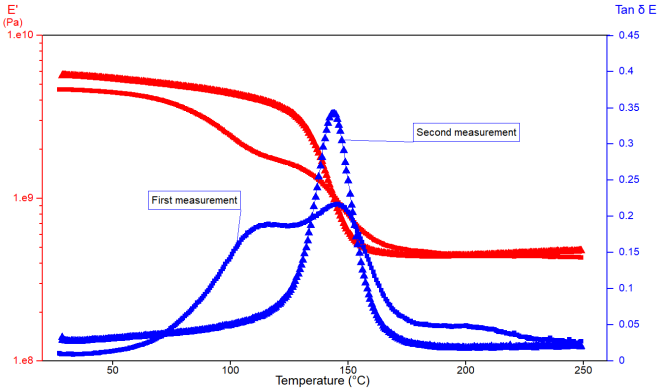


Figure 3 : E' and Tan δ as a function of the temperature. 2 successive measurements were performed on the same specimen

Conclusions

DMA is a key instrument to determine the viscoelastic properties of polymer material, and consequently can be used as a quality control tool. In this example, the DMA measurements highlight the fact that the epoxy composite was initially poorly cured with two Tan δ peaks instead of the usual one. The first temperature sweep actually finished the curing process, which explains the disappearance of the first Tan δ peak during the second temperature sweep.

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