Curing of Epoxy Adhesive Studied by TAM Air

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INTRODUCTION

Two-component epoxy adhesives consist of epoxy-resin and a hardener that react to form a solid when they are mixed. The rate of the curing process is very different for different epoxies. Rapid epoxies cure in a few minutes whereas a "standard" epoxy reaches its full strength after more than 24 hours. Isothermal calorimetry is an excellent technique to follow such a process.

MATERIALS AND METHOD

An experiment was performed where a large batch of epoxy adhesive (Araldit Professional, Ciba, Switzerland) was mixed according to the manufacturers' guidelines. Eight samples of 7.1 -11.2 g were loaded into glass ampoules. Hardening was then recorded for 45 hours at 22°C.

RESULTS AND DISCUSSION

Figure 1 shows the heat production rate as a function of time. It can be seen that the spread of results is low and that after an initial reaction period of five hours the heat production rate decrease is similar to an exponential decay.

After 45 h the thermal power is approx. 0.06 mW/g, i.e. 600 μW for a 10g sample. As the detection limit for TAM Air is better than 3 μW it would still be possible to follow the reaction for an even longer time than was done here.

In figure 2, the same data as in figure 1 is plotted with logarithmic axes. The logarithmic time starts at 1 hour and is extended to 100

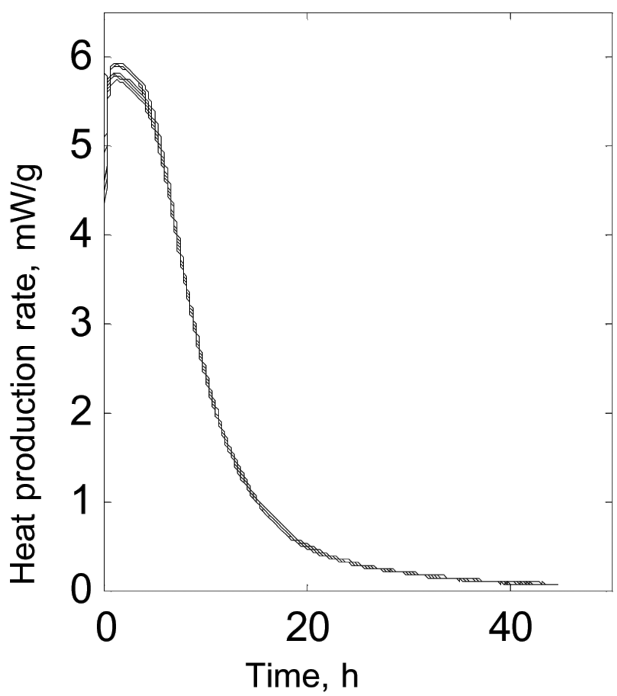

Fig 1. The thermal power from the reaction of eight samples of epoxy adhesive.

hours.

An interesting application of this type of measurement is the assessment of the fraction of unhardened epoxy as a function of time. However, to do this a model for the hardening process is required. Such a model should describe the complete hardening process, e.g. movement and diffusion in the stiffening polymer network. To show the principle we assume that the linear rate dependence seen in the logarithmic scales in figure 2 between 10 and 45 hours is also valid after 45 hours. It is thus possible to extrapolate the rate development into the future.

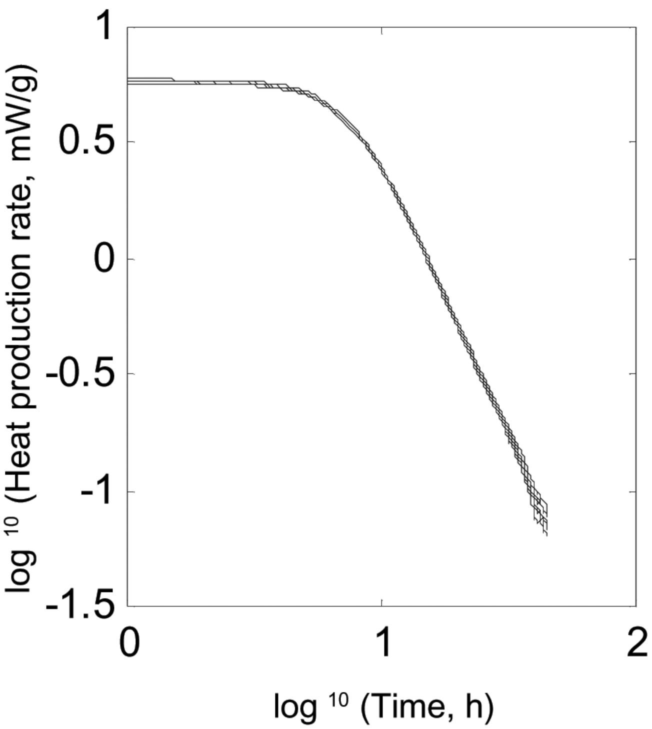

Fig 2. The logarithm of thermal power from the reaction of eight samples of epoxy

The heat production rate is proportional to the rate of the reaction and the total heat produced is proportional to the extent of the reaction. This may be used to test reaction models and calculate the parameters of such models [1]. The total heat produced during the 45 hour experiment was approx. 600 J/g. If we assume that most of the reaction has taken place at this time and that we have the same chemical reaction during the whole hardening process, we may calculate the relative rate of the reaction at any time. The initial and final heat production rates measured were 6 and 0.06 mW/g. Dividing by a total heat of 600 J/g gives relative reaction rates of 4 and 0.04 percent reaction per hour at the start and end of the measurement. The measurements described above were made with TAM Air. However, for the study of the hardening process over a longer time the more sensitive microcalorimeter 2277 TAM is recommended.

FURTHER EXPERIMENTS

Another interesting application of this instrument is to study the influence of other materials and substances on the hardening process. For example, different types of surfaces or contaminants with which an epoxy adhesive might come into contact can be studied. It is also possible to assess the influence of other factors such as temperature, mixing ratios, mixing intensity etc. on the hardening process.

REFERENCE

1. Willson R.J., Beezer, A. E., Mitchell. J.C., and Loh, W. **(1995)** "Determination of thermodynamic and kinetic parameters from isothermal heat conduction microcalorimetry: applications to long-tern-reaction studies" J. Phys. Chem. 99 7108-7113