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THE COMPARISON OF THE CURRENT AND THE ENTHALPY DURING CURE OF THE COMPOSITIONS

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Abstract

The curing of composition containing epoxy bond is complicated chemical and technological process where under temperature and pressure conditions a change of its structure occurs. The structure changes are possible to know by thermal methods as DSC, DTA, etc. and also by measurement of dielectrical response under the low frequency electrical field.

Keywords: composition, curing level, enthalpy, epoxi bond

Introduction

The hardening of the bonds of composites is complicated technological process where at the increased temperature the chemical reaction (network reaction) and the change of its structure occurs. The temperature and the time of hardening are given by the used bond. At the described compositions containing epoxy bond the hardening passes at the temperature from 160 to 170°C for 6–8 h.

From the physical and chemical view of the material structure the partial bond hardening occurs already at the production of epoxy composite – the so called prehardening of the composite. The prehardening level of the resinoid bond of the composition depending on temperature and time of prehardening process is possible to be identificated form the enthalpy change of resinoid bond and to be registered by DTA. For evaluation of DTA curves of compositions containing the epoxy bond the area of the first DTA peak is especially important because of the peak corresponding to structural changes and the change of the epoxy bond enthalpy.

Experimental

The possibility of continual measuring of the change of enthalpy and indication of the curing level provides the new method of measuring by low frequency alternating current (Fig. 1). The base of that method is the peak measuring of

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current during the cure process. Alternating voltage with amplitude $U_{max}=15$ V is supplied to the sample from the generator "G". It was found experimentally that the method is most sensitive at the frequencies of cca 0.1 Hz. For the measurements from the point of view of spurious signals, the frequency $f=1$ Hz was used (Fig. 2).

The value of the current flowing through the measured dielectric placed in the measuring condenser Tettex 2904 is proportional to the voltage of the resistor $R_1=5.6$ MΩ. The top voltage value on the resistor R_1 is scanned oscilloscopistically; the input resistor of the oscilloscope is $R_{OSC}=1$ MΩ.

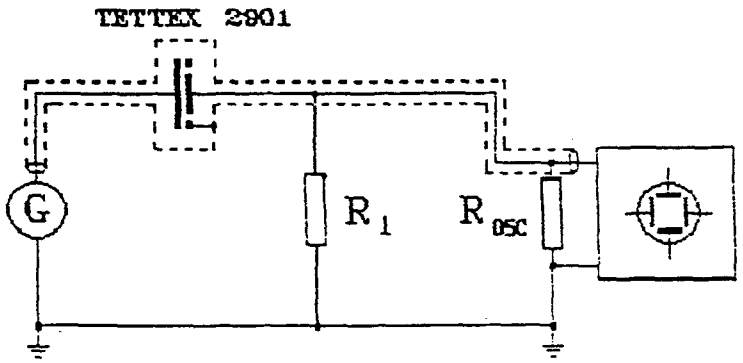

Fig. 1 Connection diagram

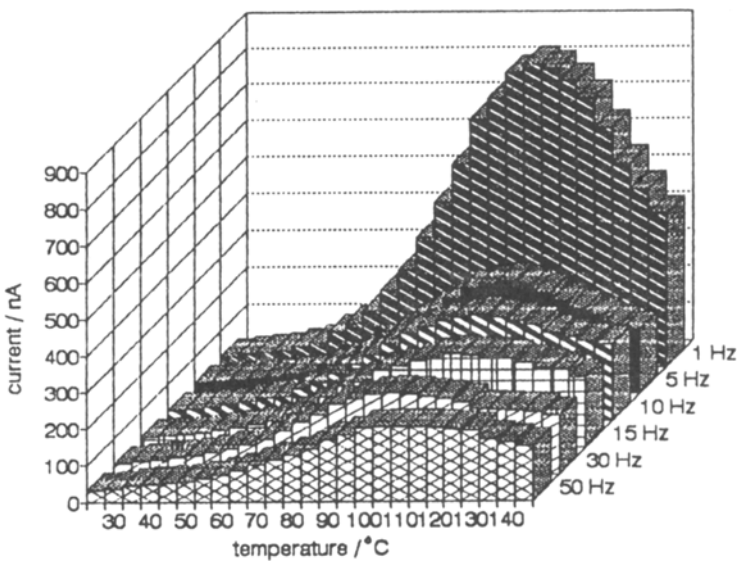

Fig. 2 Dependence of the current flowing through epoxy composition on the frequency

The method is based on the tracing of the magnitude of the top current value passing through the dielectric during the prehardening process (Fig. 3). The initial increase of the current is caused by the increasing of the apparent bond conductance during nonisothermal heating. In the initial phase of the bond heating (in the temperature range from 25 to 110°C) the increase of the bond viscosity occurs and the bond properties during the prehardening process can be considered as the liquid with the conductivity.

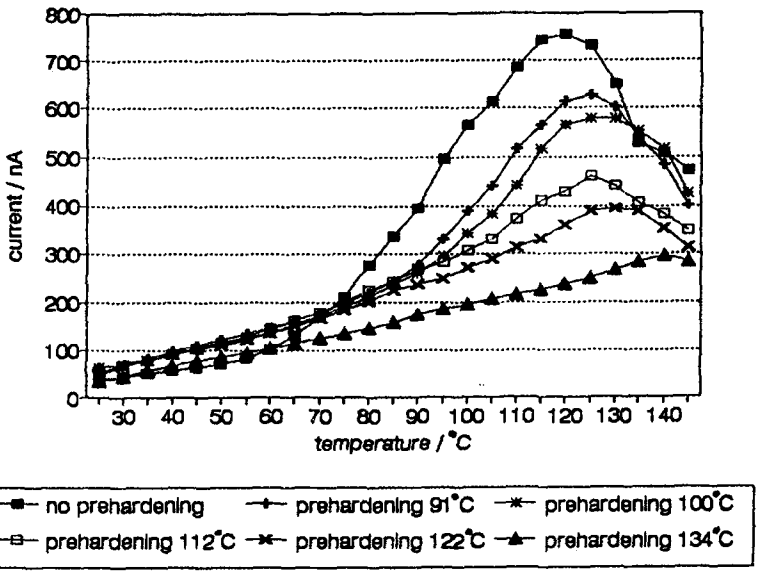

Fig. 3 Dependence of the current flowing through epoxy composition on the prehardening temperature

At the increasing temperature with the expansion of the apparent conductivity of the material the network reaction takes place. The forming of the three-dimensional structure and thus the gradual prevention of the motion of the current carriers arises in the epoxy bond what manifests in the whole current fall flowing through the dielectric (Fig. 3). The magnitude of the current maximum indicates the state of the bond prehardening i.e. the concentration of the reaction capable particles.

The sample of the measured composition was cured in precision dielectric capacitor Tettex during nonisothermal heating 3 deg·min^{-1}. The current was recorded depending on the curing temperature. The advantage of using alternating current is the measuring of current without contact.

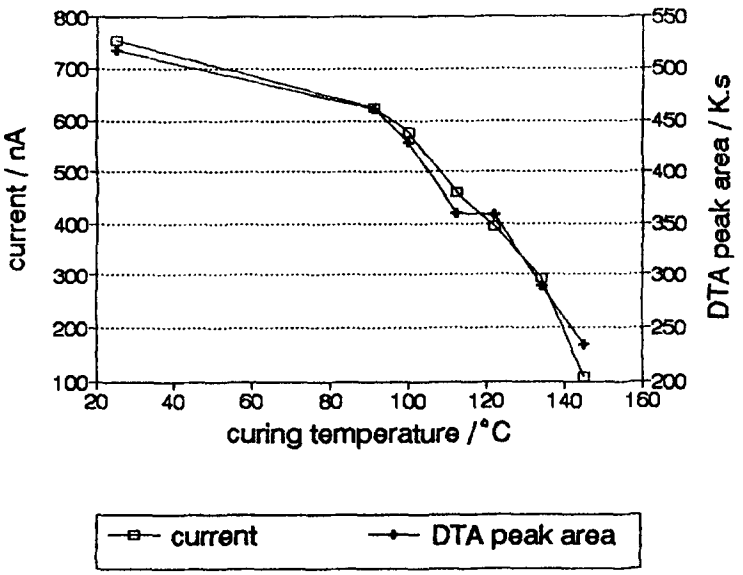

Fig. 4 Comparison of the changes of the enthalpy (DTA) and the current passing through the epoxy composition

Conclusions

It was confirmed experimentally that the character of the change of DTA peak size by influence of different time and temperature cure during nonisothermal heating is equal to changes of composition current (Fig. 4). Thanks to these facts you can consider DTA and the method of measuring current from the point of curing influence as comparisonable.

Reference

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Zusammenfassung — Das Aushärten von epoxybindungshaltigen Mischungen ist ein komplizierter chemischer und verfahrenstechnischer Prozeß, bei dem unter Einwirkung von Temperatur und Druck eine Strukturänderung erfolgt. Eintretende Strukturänderungen können mittels thermischer Verfahren, wie z.B. mit Hilfe von DSC, DTA usw. sowie durch Dielektrizitätsmessungen in einem niederfrequenten electrischen Feld ermittelt werden.