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OPTIMALIZATION OF CURING PROCESS DURING THE MANUFACTURING OF THREE-COMPONENT INSULATING SYSTEMS

OPTYMALIZACJA PROCESU UTWARDZANIA PODCZAS PRODUKCJI TRÓJSKŁADNIKOWYCH SYSTEMÓW IZOLACYJNYCH

Abstract: The proper set-up of manufacturing parameters (temperature and time characteristics) of the curing process of insulating system is the key presumption of the future foolproof operation of rotating machines and transformers. The curing process itself concerns the binder (epoxy resin in this study), which is the most vulnerable part of the insulating system in light of the operation. In order to obtain the curing process parameters, thermal analyses were applied. The objective of this paper is to optimise the time and temperature of curing process of three-component insulating materials (glass fabric, reconstructed mica and epoxy resin). Technique used for the purpose of this experiment is based on the measurement of residual enthalpy level, typical for differently cured material (number of samples with different times and temperatures of curing was prepared). The study was carried out on the Simultaneous Thermal Analysis, which allows a continuing record of DSC (Differential Scanning Calorimetry) and TGA (Thermogravimetry) curves. The DSC method enables (in case the enthalpy level of absolutely uncured material is known) the exact determination of percent of the curing. The two types of insulation tapes were compared. The results show expressive trends in curing progress of tested materials and demonstrate the suitability of applied technique very well.

Streszczenie: Właściwy dobór parametrów produkcyjnych (charakterystyk czasowych i technicznych) przy procesie utwardzania systemu izolacyjnego jest podstawą przyszłego bezawaryjnego działania maszyn elektrycznych wirujących i transformatorów. Proces utwardzania dotyczy spoiwa (w tym opracowaniu żywicy epoksydowej), które jest najbardziej podatnym na uszkodzenia elementem systemu izolacyjnego w czasie pracy urządzeń elektrycznych. W celu utrzymania parametrów procesu utwardzania, w ramach niniejszej pracy zastosowano analizy techniczne. Celem pracy jest optymalizacja czasu i temperatury procesu utwardzania 3-składnikowego materiału izolacyjnego (włókno szklane, mika, żywica epoksydowa). Technika wykorzystana na potrzeby eksperymentu bazuje na pomiarze poziomu entalpii szczątkowej, charakterystycznej dla różnorako utwardzonych materiałów (przygotowano próbki materiałów o różnie dobranych czasach i temperaturach utwardzania). Badania oparto na jednoczesnej analizie termicznej, która umożliwia ciągłą rejestrację krzywych DSC (ang. Differential Scaning Calorimetry) i TGA (ang. Thermogravimetry). Metoda DSC pozwala na precyzyjne określenie procentowego utwardzenia. Porównane zostały dwa typy taśm izolacyjnych. Wyniki pokazują wyraźne trendy w procesie utwardzania przetestowanych materiałów i demonstrują użyteczność zastosowanej metody.

1. Introduction

All electrical device manufacturers have to deal with curing process optimisation of three-component insulation systems. The curing process is concerned with the binder, which is the most vulnerable part of the insulating system regarding operation.

The objective of this paper is to optimise the time and temperature of curing process of three-component insulating materials. Two composites with different curing agents, composed of glass fabric, reconstructed mica and epoxy resin were examined. The final properties of the main insulating system of electrical device are much affected by epoxy resin and curing agent type.

2. Epoxy resin curing

In general, the low molecular weight, soluble and meltable epoxy monomers and oligomers are changed into unmeltable, insoluble and mainly three-dimensional polymers at chemical reactions during the curing process of epoxy resins. Epoxy resins obtain many good properties after the curing [1, 2, 3] such as mechanical strength, rubbery elasticity, dimensional stability and mainly thermal resistance and final electrical properties. All these properties together with the curing program can be influenced by good selection of monomers, binders and curing agents [4]. Cured epoxy resins are distinguished by high elastic modulus and high

tension and bending strength. They also have great stability of size; excellent electrical properties and they are medium-hygroscopic.

3. Methods of conversion degree determination

There are several possibilities how to determine a curing degree (degree of conversion) of epoxy binder. Attention is given mainly to kinetic analysis based techniques. Kinetic analysis uses specific mathematic models, which work more or less on the basis of Arrhenius law, such as mainly:

- kinetic analysis of thermal stability – analysis of curing process at dynamic temperature increase [5],
- isothermal kinetic analysis – analysis of curing process at isothermal temperature [6],
- kinetic analysis according to Borchardt and Daniels – analysis of curing process at dynamic temperature increase [7].

All presented techniques use thermal analyses for input parameters measurement (important for calculation of conversion degree). Mostly differential scanning calorimetry (DSC) or thermogravimetry (TGA) is used.

These techniques have their disadvantages, since the curing itself proceeds right in DSC apparatus and it is performed with very small size of samples (approx. 5 mg). Such small samples are able to absorb the heat very quickly, but their shape, structure and weight don't correspond to facts. Moreover these techniques are based on mathematical models, which are not always reliable. When we concern in these models in more details, we find out they have besides advantages also disadvantages and they are not suitable in all cases. That's why it is necessary to perform the large study of applicability of these models for each particular material. Hence these techniques are suitable e.g. for approximate estimation of curing program, for repeated comparison of the same insulating materials within one group or for better understanding of processes proceeding in the binder. The measurement of residual enthalpy level right on the samples is the other way how to determine the conversion degree of epoxy binder. This enthalpy notifies of the curing degree. The samples had been firstly prepared to correspond to facts, they were partly cured (e.g. in laboratory oven) at different temperatures and times then and in the next step the sample for thermal analysis was prepared. This method is more time-intensive compared to kinetic analysis; as it requires very accurate sample preparation. This techniques corresponds more to facts, but it enables to determine only the curing degree (compared to kinetic analysis), thus this method doesn't notify of activation energy, pre-exponential factor or order of curing reaction

4. Experimental

4.1 Materials

The samples for measurement (of rectangular shape and size of 155×110 mm) were prepared from insulation tapes of both materials.

The curing process was carried out on three-electrode system Tettex 2914 YY with heated electrodes. Condenser paper was used as separative agent, which prevents the binder leakage on electrodes during the curing process.

Prepared sample in uncured state was placed to pre-heated electrode system and then curing process at constant temperature for different chosen times followed.

4.2 Simultaneous Thermal Analysis

Simultaneous Thermal Analysis (STA) allows the simultaneous measuring by differential scanning calorimetry (DSC) and thermogravimetry (TGA) in the course of one sample heating. Thermal analysis data was collected on a TA Instruments SDT Q600 analysis. Quite small samples (cca 12,5 mg) were cut out from precured material because of quick heat transfer. Samples were tested from ambient temperature to 330 °C, at a temperature scanning rate of 10 °C/min under air atmosphere (100 ml/min). This temperature program provided complete record of the first peak corresponding to residual enthalpy level. Calorimeter was calibrated for temperature and heat flow by using sapphire and zinc.

5. Results and Discussion

DSC curves of both experimental materials were analysed to assess the level of residual enthalpy. The first exothermic peak conforms to curing reaction and the area of this peak then conforms to residual enthalpy necessary for curing. That's why enthalpy was used as the main structural parameter of analysis.

Figure 1 shows DSC curves of material no. 1 for the curing time of 30 minutes (as an example of obtained data).

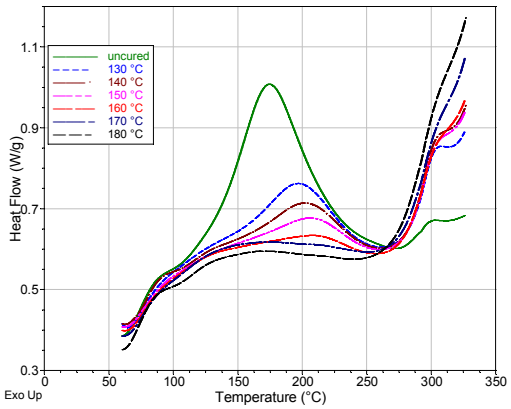

Fig. 1. Material no. 1 – DSC curves for curing time of 30 min

Enthalpy value of purely uncured material no. 1 was 175,9 J/g. Comparing a peak area (which corresponds to uncured state) with area of thermal program of 30 min at 130 °C, it can be seen that just very short curing program causes relatively expressive decrease of peak area.
Also DSC curves of material no. 2 for the same curing program are illustrated in Fig. 2 for comparison.

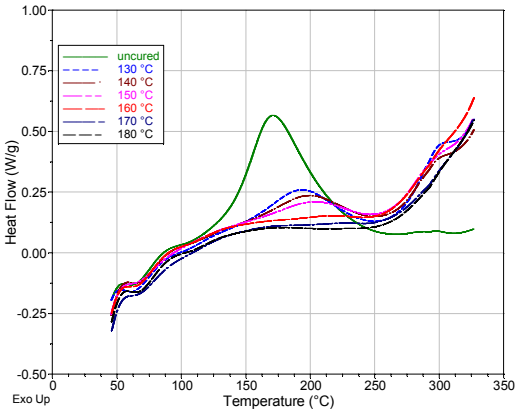

Fig. 2. Material no. 2 – DSC curves for curing time of 30 min

The enthalpy value of purely uncured material no. 2 was 185,9 J/g. As obvious from both presented graphs, essentially higher curing degree was obtained in case of material no. 2 then in material no. 1 (by using the same curing program). This fact can be attributed to the application of more suitable curing agent.
The following graphs indicate degree of curing and enthalpy level in dependence on curing time and temperature.
Figure 3 presents dependence of conversion degree on curing temperature and time of material no. 1. As can be seen, the maximum conversion degree is 83,5% at curing temperature of 150 °C for 200 min.

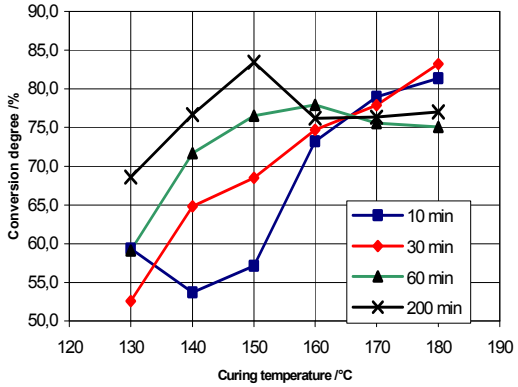

Fig. 3. Material no. 1 – conversion degree vs. curing temperature

Such maximum curing can be realized only due to relatively long-time curing program. A better way seems to be an examination of point of intersection of all curves, which corresponds to the optimum temperature (165 °C). The curing at this temperature for all chosen times leads to the same curing degree and it is planed to focus on this interesting effect in our further studies.
Figure 4 summarizes values of residual enthalpy in dependence on curing temperature and time of material no. 1.

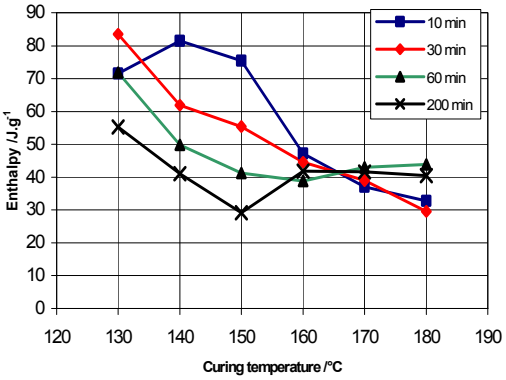

Fig. 4. Material no. 1 – residual enthalpy level vs. curing temperature

As said before, enthalpy of uncured material no. 1 (175,9 J/g) was decreased at curing process to approx. 30 – 45 J/g, to 1/6 of original value, respectively.
Maximum conversion degree of material no. 2 is higher (88,2%) than of material no. 1 (83,5%), as shown in Fig. 5. Such maximum curing is also possible only due to long-time curing program at high temperature (180 °C for 200 min).
Contrary to material no. 1, two optimum curing temperatures – 160 and 170 °C (curves more or less cross each other at these temperatures) were recorded on material no. 2. May be added,

the same curing degree can be obtained at the same temperature for different chosen curing times again.

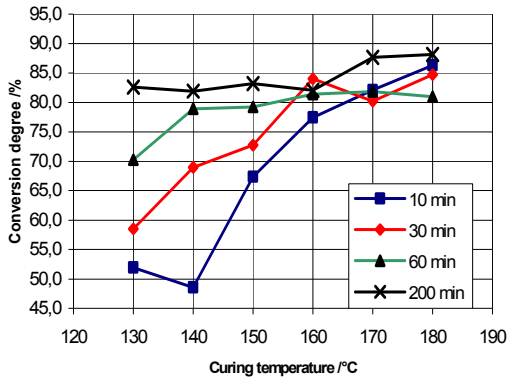

Fig. 5. Material no. 2 – conversion degree vs. curing temperature

Fig. 6 illustrates the residual enthalpy dependence on curing temperature and time of material no. 2.

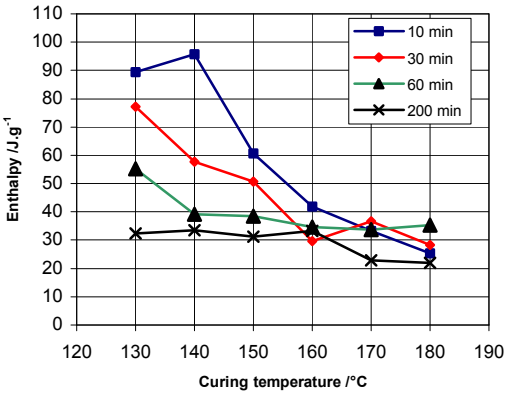

Fig. 6. Material no. 2 – residual enthalpy level vs. curing temperature

Enthalpy value of uncured material no. 2 (185,9 J/g) was higher than in case of material no. 1. This value was decreased at curing process to approx. 22 – 35 J/g, i.e. to 1/8 of original value.

6. Conclusion

Obtained data indicates, that material no. 2 enables to reach higher curing degree, even at lower temperatures and for shorter times. The curing process has a better dynamics then. This dynamics can be determined also on the basis of rate constant of Arrhenius law (obtained from kinetics analysis). The comparison of these approaches to conversion degree determination could produce very interesting results and it will be the main objective of our coming study.

Also a study dealing with comparison of electrical parameters during the curing process (such as loss factor and permitivity) was previously performed on presented materials [3, 8] and provided the same final results.

7. Acknowledgment

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