Václav Mentlík, Radek Polanský
University of West Bohemia, Pilzno

APPLICATION OF THERMOANALYTICAL DIAGNOSTIC METHODS IN ELECTROTECHNOLOGY

ZASTOSOWANIE TERMOANALITYCZNYCH METOD DIAGNOSTYCZNYCH W ELEKTROTECHNICE

Abstract: The most sensitive parts of electrical machines – insulating systems - don't get along without components based on organic substance. These components are usually very necessary by technical and technologic reasons. Operating effects cause specific processes in these components, which change their properties. It's necessary to monitor concrete changes of particular components from the aspect of equipment operation, especially in regard of reversibility and irreversibility of these processes. Irreversible processes can lead up even to destruction of materials or components. From aspect of its operating reliability, these components are key parts with their essential importance. For the monitoring of all these processes, structural analyses are (from the aspect of modern diagnostics) irreplaceable. They enable to monitor and consequently discover development of monitored properties. Generally, there is good experience with monitoring of degradation processes in materials of organic structure with a help of thermal analyses. These analyses provide us determination and monitoring of key parameters – enthalpy. This value, discoverable by Differential Thermal Analysis (DTA) is very typical for particular materials and exactly describes behaviour and application of these materials during the operation stress. The connection between the results of structural methods and classical phenomenological methods is very interesting, because structural parameters help us to complete information about monitored elements or subsystems of monitored equipment.

Streszczenie: najbardziej narażone części maszyn elektrycznych – ich układy izolacyjne – zawierają komponenty bazujące na związkach organicznych. Zastosowanie związków organicznych jest zwykle konieczne z powodów technologicznych i technicznych. Warunki w jakich pracują układy izolacyjne wywołują specyficzne procesy w tych komponentach, które przyczyniają się do zmiany ich właściwości – czyli do ich starzenia się. Konieczne jest zatem monitorowanie określonych zmian zachodzących w komponentach układów izolacyjnych ze związkami organicznymi w aspekcie zapewnienia sprawności działania urządzeń, zwłaszcza odnośnie odwracalności i nieodwracalności w/w procesów. Procesy nieodwracalne mogą prowadzić nawet do zniszczenia układu izolacyjnego. Z punktu widzenia niezawodności działania urządzenia, komponenty izolacyjne zawierające związki organiczne mają więc kluczowe znaczenie. Do monitorowania wspomnianych procesów, w nowoczesnych układach diagnostycznych wykorzystywane są analizy strukturalne. Umożliwiają one wczesne wykrywanie zmian monitorowanych parametrów układów izolacyjnych. Zasadniczo, w monitorowaniu procesów degradacji w materiałach o strukturze organicznej sprawdziły się analizy termiczne. Umożliwiają one określenie stanu i śledzenie zmian kluczowego parametru – entalpii. Wartość tego parametru określana na podstawie różnicowej analizy termicznej (ang. Differential thermal analysis – dta) jest bardzo charakterystyczna dla poszczególnych materiałów i precyzyjnie określa ich zachowanie się w czasie narażeń wynikających z pracy urządzeń. Zależność pomiędzy rezultatami metod strukturalnych i klasycznych metod fenomenologicznych jest bardzo interesująca, gdyż parametry otrzymywane z analizy strukturalnej pomagają uzupełnić informację o monitorowanych komponentach układów izolacyjnych. W artykule autorzy omawiają we wstępie podstawy teoretyczne różnicowej analizy termicznej, a następnie opisują sposób przeprowadzenia opartych na tej analizie i wykonanych przez nich badań kilku wybranych materiałów izolacyjnych. Wyniki tych badań przedstawiono w artykule i krótko je skomentowano.

1. Introduction

Modern manufacturing of electrical equipment could hardly manage without components, which (mainly for technological reasons) contain organic substances. Therefore, these components become the most sensitive parts of their serial reliability chain. Especially these components are in the centre of attention for its sensitivity to the influences effecting on equipment during its operation. These organic components are usually synthetic macromolecular substances, in which various environment influences cause a number of irreversible

reactions. Materials change their properties – they are ageing.

2. Theory

The Guldberg-Waag´s Law determines a time variation of the products of reactions proceeding in materials:

$$\frac{d}{dt}(m_0 - m) = k \cdot m. \qquad (1)$$

It describes the fact, that whenever $\mathbf{m_0}$ of starting molecules enters the reaction, their number **m** does not change in time **t** during the reaction. Velocity coefficient of appropriate chemical reaction k is given by the well known Arrhenius´s relation. Varying concentration of active molecules is closely connected with the change of material physical properties. Time variation of monitored physical property **P** is possible to describe by equation:

$$\frac{dP}{dt} = -A \cdot e^{-\frac{E}{RT}} \cdot f(P), \qquad (2)$$

where

- **A** preexponential factor [s^{-1}] setting the frequency of meeting molecules,
- **E** activation energy [$kJ.mol^{-1}$] setting the level of energetic barrier, which must be overcome by the molecules entering the reaction,
- **R** universal gas constant = 8,315 $J.K^{-1}mol^{-1}$,
- **T** absolute temperature [K],
- **f (P)** function containing order of proceeding reaction, it respects connection between concentration of reactive elements and monitored material property.

Basic relation of differential thermal analysis (DTA) supports sequence and usability of this method that records changing processes in materials. The main term of this equation is above mentioned activation energy describing state of reactive elements.

$$\frac{d(\Delta T)}{dt} + A \cdot (\Delta T - \Delta T_{ust}) = \frac{E}{C_v} \cdot \frac{dm_r}{dt}, \qquad (3)$$

where

- **ΔT** the difference of the actual temperatures of the sample and the inert normal,
- **ΔT_{ust}** the difference of temperatures indicated by the differential thermocouple – in the case where the change of enthalpy in the sample does not occur – it corresponds with the zero line of the thermogram,
- **C_v** thermal capacity of the sample and the container of DTA cell,
- **m_r** relative concentration of active elements:

$$m_r = \frac{(m_0 - m)}{m_0}, \qquad (4)$$

m_0 is number of molecules entering to monitored reaction, **m** means number of elements, which hasn't been changed during the reaction.

3. Experimental

DTA result applied on composite material containing epoxy resin (functional relation of thermo-voltage difference ΔU of the sample and thermal inert standard on the testing time) indicates the local extremes – peaks, corresponding to monitored reactions. Further described experiment demonstrates a correspondence between DTA results and results of other methods, exploring a development of monitored material properties. In our case, the experimental material is composite (glass fabric 110 $g.m^{-3}$ ceramized, modified by aminosilane and bromine epoxy resin). Hardening treatment of this material runs in two stages (after setting to hot presser for 5 min at 170°C and pressure of 0,2MPa, follows temperature of 170°C and pressure of 2 MPa). Decisive factor for final quality of material is the time length of the second stage. Hardening was realized in five different versions – 36, 40, 44, 48, 52 min. The samples have been tested by DTA (composite had been crushed to the powder - granularity of 160 mesh, analyses have been proceeded in dynamic oxygen atmosphere - flow rate 50ml/min, heating rate 5°C/min). In DTA curves in Fig. 1, two exothermic peaks are evident, which corresponds to isomerization in interval of lower temperatures and also to thermal-oxidative reactions during the higher temperatures. For example, it is given thermogram of the sample with hardening time of 52 min in Fig. 1. The relation of peaks surface on hardening time is shown in Fig. 2. There were consequently realized measurements of other properties with the same sample, such as dissipation factor and permittivity, ultimate bending strength, absorbability, determination of unreactive elements quantity by extraction.

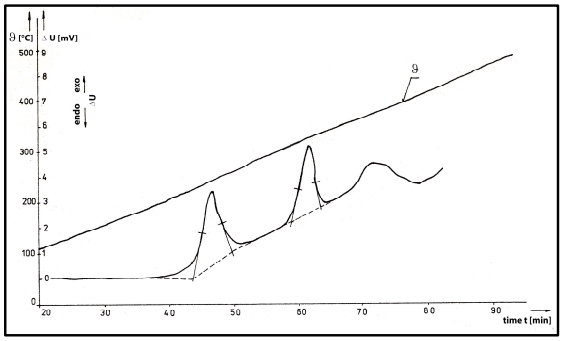

Fig. 1. DTA thermogram of the sample with hardening time of 52 min

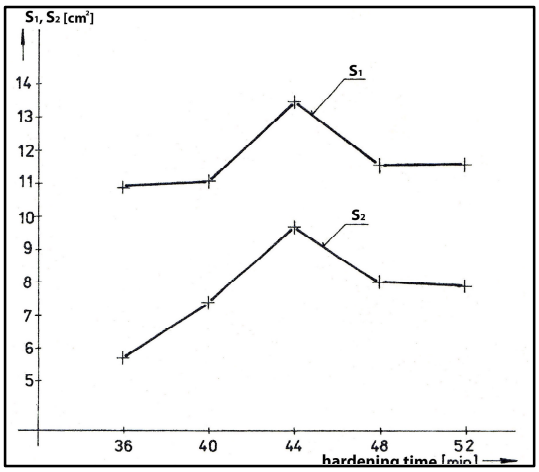

Fig. 2. Relations of peaks surface on hardening time

Graph in Fig. 3 indicates dependence of mentioned properties on hardening time together with data obtained by method DTA – values corresponding to both monitored effects. There is well-evident result correspondence of all methods here. This fact can also confirm correlative coefficients **r (x,y)** introduced in Tab. 1.

Tab. 1.
Correlative coefficients of relations between DTA results and chosen methods

Correlated property	Value of correlative coefficient related to DTA
Permittivity	0,614
Dissipation factor	0,948
Determination of unreactive elements quantity by extraction	0,839
Ultimate bending strength	0,720
Absorbability	0,658

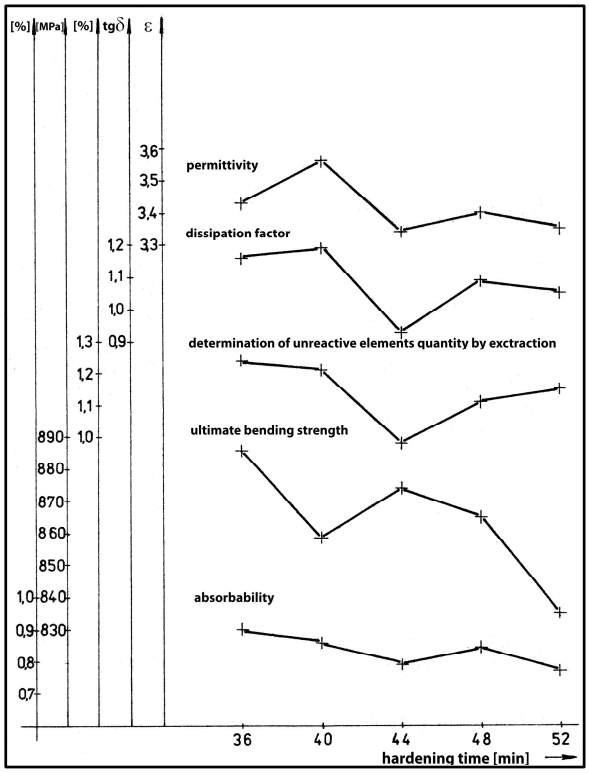

Fig. 3. Relations of monitored properties and DTA results on hardening time

4. Discussion

These introduced results clearly prove by evidence suitability of using DTA at the study of properties of composite materials containing organic substances. Analyses monitor energy of elements respective to isomerization and thermal-oxidative reactions and document in this manner development of material properties by definition of actual concentration of elements, able to participate in particular reactions. This above described case is concerned to finding of optimal hardening time corresponding to processed material. It was confirmed by correlation with other methods, that DTA results and the results of these other methods reflect very good correspondence. Optimums of all measured properties correspond to optimums of DTA results. All methods specify hardening time of 44 minutes as the optimal hardening time.

5. Conclusion

For that reason DTA method has been applied for study of hardening processes in organic resins, for definition of degradation degree of these materials (with degradation degree falling also the number of reactive elements decreases) and also for distinction of particular kinds of resins. DTA, as a prompt, operational and fast

method, helps to solve many projects in electrotechnology.

This article was carried out with support of Ministry of Education, Youth and Sports of Czech Republic, Project No. MSM 4977751310 - Diagnostic of Interactive Processes in Electrical Engineering.

6. Bibliography

[1]. Mentlik V.: *New Application of the DTA in Heavy-Current Electrotechnology,* Thermochimica Acta, Proceedings of ICTA ´85, 93/1985, pp. 353.
[2]. Mentlik V.: *Experience with the Application of Thermal Analysis in High-Voltage Insulation*, Journal of Thermal Analysis, vol. 39/1993, pp. 1355.
[3]. Mentlik V.: *The Study of Curing Reaction of the Selected Composition by the DTA Method*, Calorimetry and Experimental Thermodynamics, Praha 1993, pp. 156.

Authors

Prof. Ing. Václav Mentlík, CSc.
Ing. Radek Polanský

University of West Bohemia in Pilsen
Faculty of Electrical Engineering
Department of Electrotechnology
Univerzitní 26, 306 14 Pilsen
Czech Republic

Tel.: (+420) 377 634 506
Fax: (+420) 377 634 502
E-Mail: mentlik@ket.zcu.cz
rpolansk@ket.zcu.cz