

STUDY OF HIGH TEMPERATURE INSULATION MATERIALS

Václav Mentlík, Eva Kučerová, Václav Boček, Pavel Šebík

Section of Electrical Technology, Department of Technology and Measurement, Faculty of Electrical Engineering, University of West Bohemia, Univerzitní 8, 306 14 Plzeň

Summary One of current objectives of the electro insulating technology is the development of the material for extreme conditions. There is a need to operate some devices in extreme temperatures, for example the propulsion of the nuclear fuel bars. In these cases there is necessary to provide not just insulating property, but also the thermal endurance with the required durability of the insulating materials. Critical is the determination of the limit stress for the irreversible structure modification with relation to material property changes. For this purpose there is necessary to conduct lot of tests on chosen materials to determine the limits mentioned above. Content of this article is the definition of diagnostic mode, including the definition of the exposure factors, definition of the diagnostic system for data acquisition and first result of examinations.

Abstrakt Jedním ze současných úkolů elektroizolační techniky je vývoj materiálů pro extrémní podmínky, vzhledem k tomu, že je nutné provozovat některá zařízení např. při vysokých provozních teplotách jako je tomu u pohonových jednotek pro posun tyčí jaderných reaktorů. V těchto případech je nutné zajistit nejen izolační schopnost, ale také odpovídající teplotní odolnost při požadované životnosti použitých materiálů. Rozhodující je pak zejména určení oblastí, která je mezní pro vznik ireverzibilních změn ve struktuře, a tím i v chování materiálů. Pro tento záměr je nutné provést řadu ověřovacích měření na vybraných materiálech s cílem určení zmíněné hranice. Stanovení zkušebního režimu včetně expozičních činitelů a jejich hodnot i diagnostického systému pro získání potřebných informací je spolu s prvními výsledky provedených šetření náplní předloženého příspěvku.

1. INTRODUCTION

Insulating system is from the viewpoint of the reliability the key component of the electric devices. It has decisive influence to their reliability. With the increase of requirements to electric machines the demanded quality of insulation system have to increase.

There is a special group of electric devices intended for the work in extreme conditions. It means for example high temperature domains. This case should be for example nuclear reactor fuel bars position control. Mentioned devices must have insulating system suitable for the service in high temperature areas. Used materials must have different insulating properties and different mechanism of operational ageing than common materials. We presume - by virtue of the similarity of the physical effect on similar materials - that there exist also some limits of electric and thermal stress in these materials. If these limits are reached there will start irreversible changes in the material. These changes lead to degradation of the electro insulating properties and to decrease of life time. For the application of material is important the knowledge of criteria values and corresponding changes of electrical properties. Especially important is this knowledge for insulation system construction and sizing.

2. EXPERIMENT FOR MATERIAL THERMIKANIT 26.000

The knowledge of the area, where the insulating material loses its reversibility is very important for designers of the electric devices and for the users of these devices as well. This "irreversibility limit" would never be reached when the device is operating. So this limit is very important attribute for each single material. There is required to determine experimentally the limit for the representative sample of each group of insulating materials. The purpose

of this research is to determine limits for materials used for above mentioned extreme conditions.

There was chosen an insulating material for the confirmation of the method mentioned above. It is a special material for high temperature application. The material is the product of Elektroisola a.s. Thermikanit 26.000. Its composition responds to expected high operational temperature - 91 % noncalcinated mica and 9 % thermal resistant silicon resin. By compressing these components under high temperature and high pressure to the shape of compact hard boards, there is produced the material which respond to demands - stable thermal endurance up to 500 °C.

There was defined diagnostic system for conducting of required examinations. Besides the method selection all necessary point of view were respected. There were chosen both phenomenological and structural methods. The objective was to acquire full information about the material behavior. So there were selected these phenomenological methods: method of dielectric relaxation spectroscopy, measurement of the loss factor $\text{tg } \delta$, inductive capacity, record of absorbed and resorbed current. Out of the current the value of polarization index, resistivity and reduced resorbed curves (RRK) were calculated. The choice was also based on experience and following structural analyses were chosen: scanning electron microscopy, x-ray fluorescent spectroscopy and thermomechanical analysis.

The material was analyzed by above mentioned methods. The material was in original condition, before the exposure.

Samples of Thermikanit 26.000 have the size 200 × 200 mm and thickness 0,25 mm. The samples for all following examinations were exposed to 320 °C during 500 hours as an application of accelerated thermal ageing. This approach was chosen because

of there was not possible to expose the samples to temperature and electrical stress in same time – that would substitute the real operational stress of the device much more realistic, of course. In this stage of experiment the material properties were measured again.

Then the electrical exposure followed. For this purpose the electrodes, made of brass plate was provided. The electrode has the size $115 \times 115 \text{ mm}^2$, thickness 5 mm, corner fillet with radius 15 mm. Chamfer of electrodes edges was 1 mm. Values of exposure voltage were set base on experience of authors. In first stage of experiment the voltage levels 3, 4, 5 kV. For 3 kV were of exposed during 80, 140, 200, 280 hours. The upper limit of time of exposure were set of value, when the quick rise of samples breakdowns start – it means, when the level of withstand voltage was close to level of breakdown voltage. For the level of 4 kV time values set was 20, 50, and 80 hours and for 5 kV to 7 and 14 hours, respectively.

so, that during electric exposure the silicon ascends to the surface and there it is scanned. From another chemical elements the similar behavior has zinc.

Loss factor have been measured in the frequency range from 26 Hz to 100 kHz. In this range there were no extremes of the characteristic of individual samples. The same characteristic we would get by evaluation of measurement by any frequency of bridge's voltage. But we confirm the higher sensitivity of loss factor at lower frequencies to material changes. The difference between the limit values of frequency range is 100 %. For the purpose of this article the measurement of loss factor at low frequency has been conducted. The decrease of sensitivity at 50 Hz comparing to 26 Hz is just about 5 %. The loss factor characterizes very well the level of exposure. It distinguishes the intensity of electric stress, as we can see in the graphs illustrated in the Fig. 1. As we compare the characteristic, we can

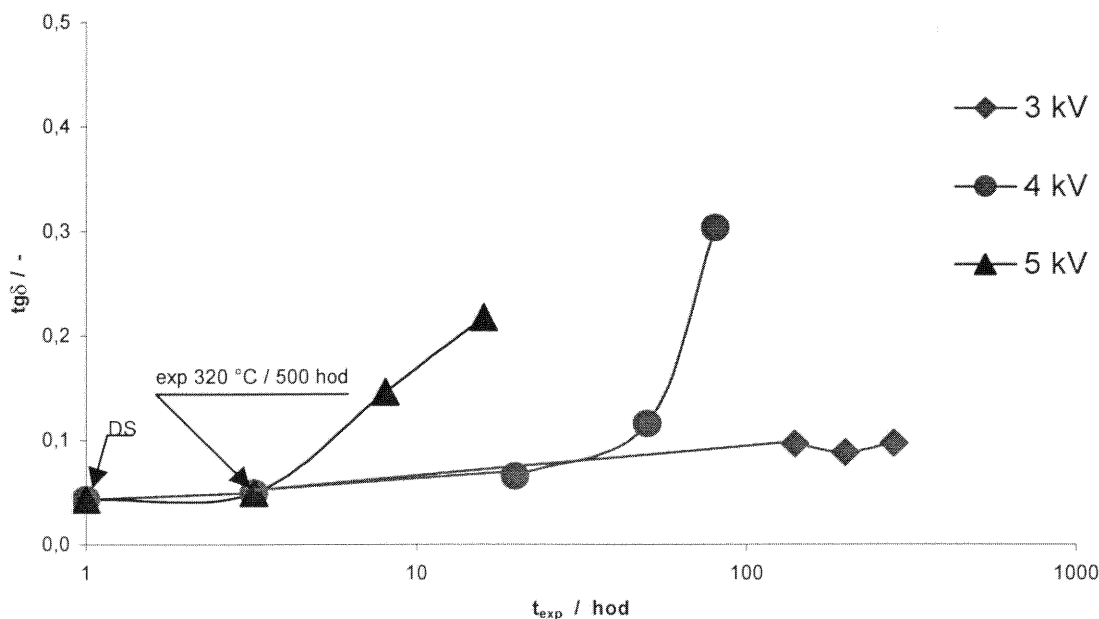


Fig.1. The relation between loss factor $tg \delta$ and the exposure.

3. THE RESULTS OF EXPERIMENT FOR MATERIAL THERMIKANIT 26.000

First obtained results were very interesting. There is necessary to notice, that it is large scale project, which is far from over and it will require many more experiments.

Very interesting for example is to watch first polarizing spectrum of x-ray fluorescent spectroscopy. We can find out that electric exposure leads to increase of the attendance of silicon on the surface of samples. We could interpret it

find out that the 3 kV exposure causes slight increase of loss factor. Another two exposures causes significant increase, so we should expect the overgrowing of the irreversibility limit. This phenomenon should be documented at 4 kV characteristic, which shows after first two collections (20 and 50 hours) just slight increase of loss factor. Not until 80 hours, when significant changes appears.

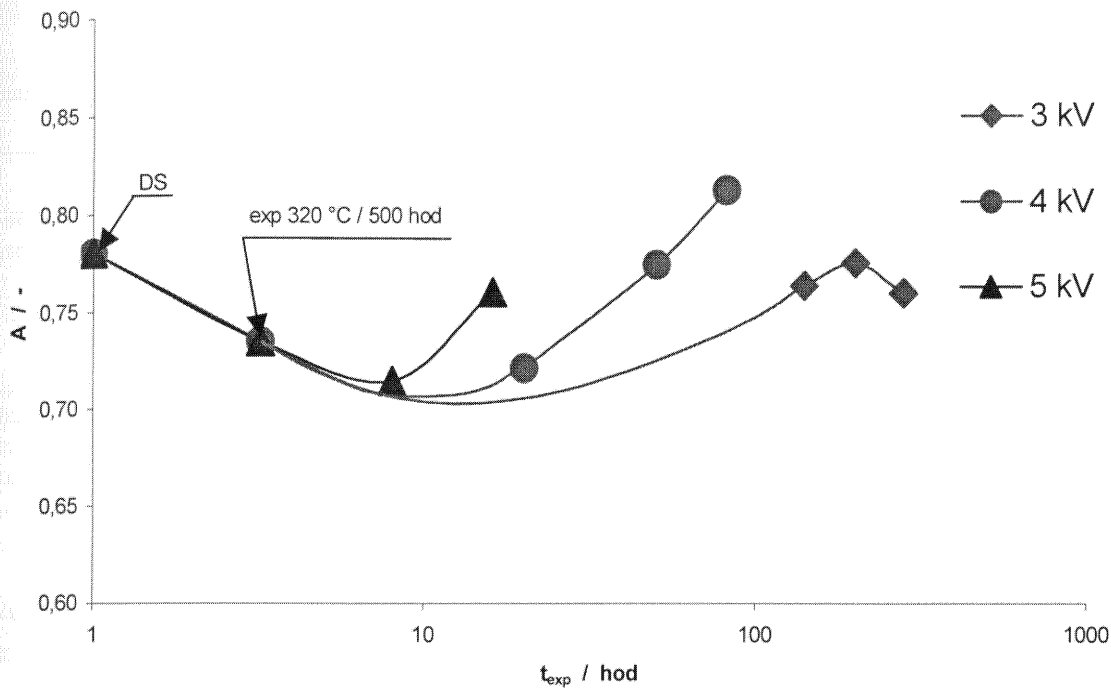


Fig. 2. The relation between values of substitutions of reduced resorbed curves slopes and the exposure time.

Also other results of phenomenological methods give us significant information about influence of the above mentioned kind of exposure to material. It is evident from Fig. 2. There are shown the characteristics of slopes of reduced resorbed curves. Because of higher nonlinearity of substitutions of reduced resorbed curves for Thermikanit 26.000 is more advantageous to calculate the slopes substitutions of reduced resorbed curves out of the beginning of resorbed currents. So we use because of technical limitation the time range $15 \div 45$ s. After certain decrease of slopes of RRC, which characterize the material degradation, the slopes increases. Most evident is it in endpoint at the voltage level 4 kV. It should be the possible overrun of the irreversibility limits.

Figures 3 and 4 show changes of polarization index and volume resistance. The value of polarization index after exposure decreased to critical value, which is close to 1. There is again evident that 3 kV exposure cause lesser decrease of both polarization indexes. The resistance characteristic matches to characteristic of loss factor. Also here is evident at 4 kV level of exposure the decrease toward to demanded irreversibility limit.

At all characteristics are first two points shared. These two points are values of original condition (DS) and values after exposure at 320°C for 500 hours.

4 CONCLUSION

At first appearance there is evident the exposure influence to mentioned parameters. The most transparent is the exposure influence to loss factor. There is possible to

assume the character of degradation from quite different characteristic of other parameters. The characteristic of loss factor and volume resistivity indicate the irreversible changes of the resin. The increase of reduced resorbed curves slopes indicates the disintegration of material.

REFERENCES

- [1] Mentlík V.: Posouzení expozičních změn skloslídových materiálů sledováním jejich entalpie. In: Diagnostika '97 ISBN 80-7082-342-9, Plzeň 1997, s. 306.
- [2] Mentlík, V.: Vztah entalpie a stavu vn izolačního systému. přednáška na Kolloquiu s mezinárodní účastí, FEI TU Košice, 28. 11. 1997.
- [3] Mentlík V., Lang J.: Studium polarizačních jevů v nehomogenním dielektriku. [Závěrečná výzkumná zpráva Fondu rozvoje MŠMT 1997 - č. 0434.] Plzeň, ZČU 1997.
- [4] Mentlík V.: Posouzení pojiv pro izolační systémy. In: Nove smery v diagnostike a opravách elektrických strojov a zariadení. ISBN 80-7100-489-8, Žilina 1998, s. 45.
- [5] Šíkula J., Koptavy B., Hruska P., Vasina P., Koptavy P., Hajek K.: Partial Discharges as Reliability Indicators in Foil Capacitors. In: 11th Annual European Passive Components Conference CARTS-EUROPE '97, October

[6] Šikula J., Lokajiček V., Kořenská M., Pavelka J., Koptavý P., Sedláková V.: Electromagnetic and Acoustic Emission from Solids under Ramp Increasing Pressure. In: 24th European Conference on Acoustic Emission Testing EWGAE 2000, May 24 ÷ 26, 2000, Senlis (Paris), France.

[7] Mentlík V., Kučerová E., Boček V., Matějka F.: Aspekty ověření vlastností izolantů pro vysokoteplotní expozice. In: Diagnostika '03, ISBN 80-7082-952-4, Plzeň: ZČU 2003, s. 243

This article was created by help the Grant Agency of the Czech Republic (grant 102/03/0621) Irreversible Processes in Insulating Materials for High Temperature.

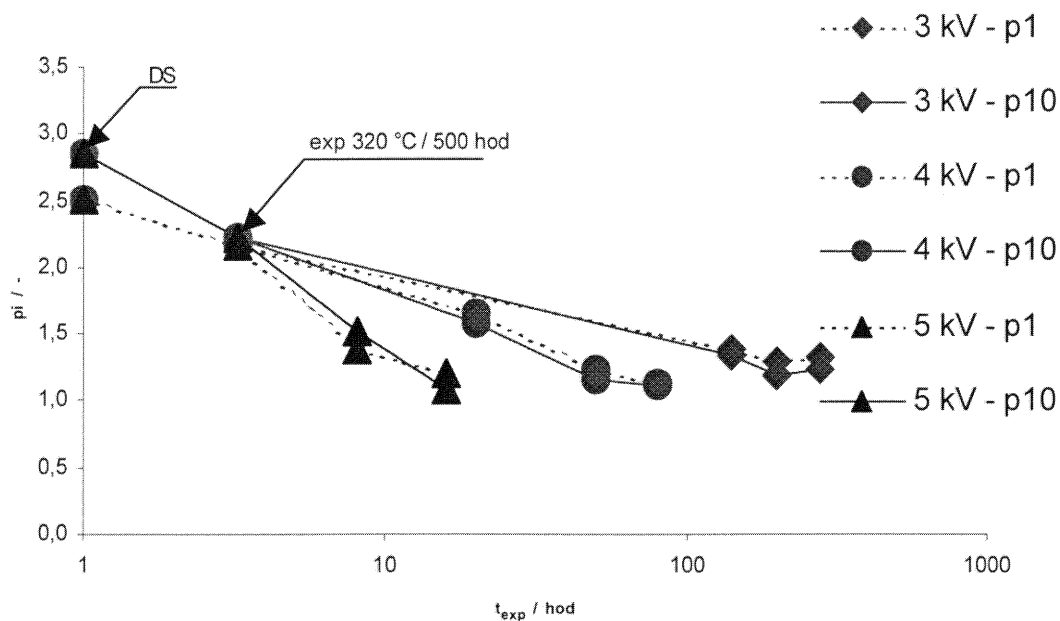


Fig. 3. The relation between polarization index and the exposure.

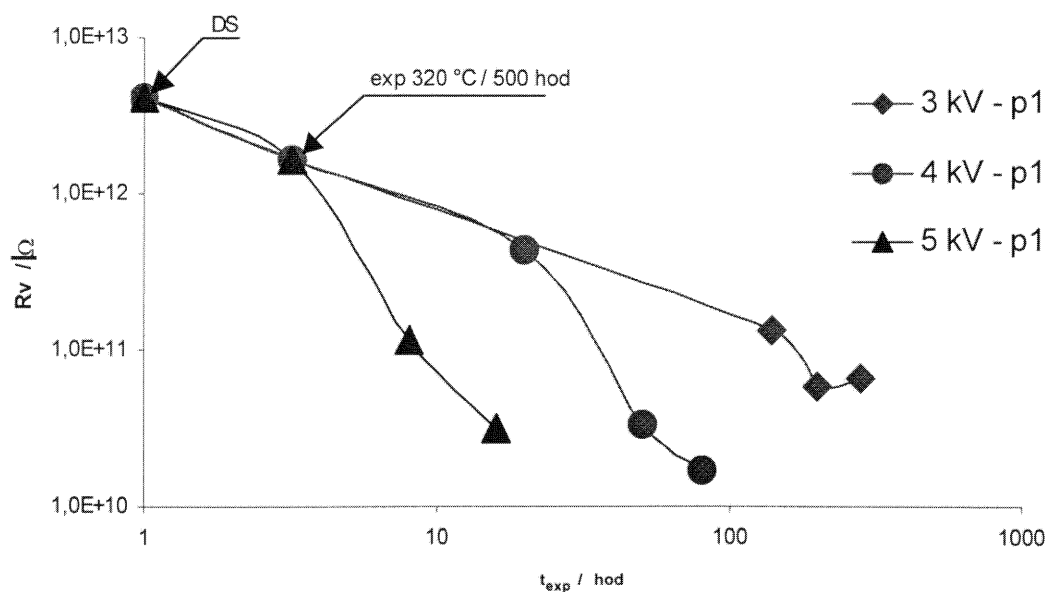


Fig. 4. The relation between volume resistance and the exposure.