Ročník 2011 electroscope Číslo III

The testing of the insulating PET material

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Anotace:
Materiál PET (polyethylentereftalát) je velmi důležitý pro průmysl a výrobu elektrických strojů. Tento článek ukazuje vliv dlouhodobého teplotního zatěžování na materiálu PET. Konkrétně se zaměřuje na objemovou rezistivitu připravených vzorků. Délka experimentu teplotního zatěžování byla šest tisíc hodin. Teplota zatěžování byla stanovena na 190 °C. Výsledky z měření objemové rezistivity byly porovnány s výsledky diferenciální skenovací kalorimetrie. Velký důraz byl kladen na opakovatelnost provedených měření.

The PET (polyethylene terephthalate) material is very important for electric machinery industry. This article explores the influence of long term thermal ageing on PET. Particularly, it is focused on the volume resistivity of studied samples. The time of thermal ageing was six thousand hours at the temperature of 190 °C. The results of volume resistivity were compared with differential scanning calorimetry. Special attention was paid to the reproducibility of measurement.

INTRODUCTION

Many laboratories in the world such as Toshiba Corporation, R&D Center in Japan, University of Essen in Germany or CNRS Toulouse, in France etc. try to find changes in electrical, mechanical, chemical and other parameters with regard to some ambient factors such as temperature, moisture, chemical pollution, applied voltage, ultraviolet radiation etc.
It is not an easy task to determine precise influence of ambient factors on important parameters of insulating materials. All ambient factors affect aforementioned parameters. The temperature of ambient is the strongest one [1].
The application of insulating organic polymers and aromatic polyesters in electrical engineering has become a commonplace today. The polymers have the advantage being used as dielectrics and insulation material in wide range of technical application. PET material is highly thermal and chemically stable. It has more preferable mechanical properties than many other types of polyesters. PEN material (polyethylene naphthalene) is a better polymer which tends to replace PET in some specific applications [2]. Nevertheless, from the economic perspective PET has become a dominant insulating material.
It is used for instance in high performance capacitors. PET is also used in food industry for wrapping goods, but the main attention in this work will focus on electric machinery industry. It is PET, that is used as an insulator in electric motors, transformers, chocking coil etc.
This work tries to find out changes of properties (the volume resistivity and the degree of crystallization) in insulator PET Semi-crystalline related to long term thermal ageing.
First of all, a proper research of scientific articles has been made. The exploration focused on long term temperature ageing. Mainly, the IEEE explorer was used to find information from 1980 to the present. Other databases of scientific articles were also used (ISI Web of Knowledge, SCOPUS, EBSCO etc.)
Many, different types of experiments were presented in these publications. One can mention e.g. measuring or evaluation of incremental loss tangent, capacitance change, partial discharge pulse count, partial discharge energy, volume resistance change. [3].
There is not enough information about ways of estimating time necessary for destruction of an insulation material. There are no models that can be used for reliable prediction [4]. There are few articles e.g. [5], [6], that deal with mathematical models.
The research in [5] presents fuzzy model of thermal ageing. The volume resistivity and other parameters have been taken into account, but it is the theoretical model which is not based on experimental results. The model has not been verified.
The study [2] deals with thermal ageing at the temperature of 120 – 210 °C during one thousand hours. The dielectric spectroscopy and DSC were used for evaluation and verification results.
It seems that the experiment was carried out only once. There are not enough experimental attempts to be able to present reproducible results. Generally, most of other publications regarding the ageing of insulator have the same demerit. There is not enough information about experiments in the above mentioned articles. That is a very important point allowing to make right conclusions. There is usually no information about the results shown to users. The best article about thermal ageing with regard to our aim can be considered [7]. The experiments are described very well there. It looks as if the authors tried to do a very complex test with a clear goal.

What was mentioned is not to be meant as criticism of the above mentioned authors. This shows that the determination of dielectric and electric parameters of polymer for mathematical models is very difficult. It is important to have relevant results of many experiments to determine properly the change in certain parameters. Afterwards, the mathematical model can be verified.

THE TEMPERATURE AGEING

The long term temperature ageing is a very complicated and complex problem to carry out in laboratory conditions. The main problem is the level of temperature used for ageing. Too low level does not invoke any degradation (ageing) in polymer. Too high level can change the structure of polymers differently from effects that occur in polymers at nominal temperature.

Let us take an example using a simple egg to help us explain the problem properly. When an egg is boiled in water at the temperature of 100 °C during 10 minutes, it is very good for breakfast. When the egg is warmed to a temperature of 37,5 °C for 21 days, a chicken appears. These are two different results of a structural change. The story illustrates that it is important the right level of temperature and time of experiment be taken into account. Therefore, the shortest time of experiment is necessary to reach whilst the temperature is maintained as high as to keep the processes of structural change the same as under nominal condition.

PET material has maximum temperature of utilization 180 °C (class F/H 130 – 155 °C). The standard ČSN EN 60085 shows the material can operate during 40 thousand hours by 180 °C. It is too long time for experiments. It was necessary to determine the appropriate level of aging temperature. The brief testing of PET material was made by different temperatures in order to find the maximum ageing temperature for intended experiments. The evaluation of mechanical parameters was made subjectively without any measuring instruments. The possibility of proper measuring the volume resistance was one of the most important criteria. The flat surface of tested sample was the most critical. Deformations on the surface can affect the results of measurement very strongly.

Tab. 1: The temperature level determination – results

T_a [°C]	t_a[h]	result stage of sample
240	0.5	defective, slightly deformed, high bending
230	1	defective, slightly deformed, high bending
220	2.5	slightly deformed, high bending
215	5	almost without defects, high bending, brittle
205	184	no defects, higher stiffness, brittle
180	813	no defects, higher stiffness
130	1110	no defects

The temperature of 205 °C was used for the first experiment. The results are published in [8]. The material was very brittle after 2 thousand hours. Thus aged (damaged) material cannot be used in electric machine.

This study explores the material at temperature of **190 °C**. As regards acquired results in [8], this value seemed to be right for our further research.

THE EXPERIMENT DESCRIPTION

This section is divided into three parts. The first part describes certain materials tested in experiments. The second one shows the experimental workplace, procedure for measuring and used instruments. The last section explains and describes the evaluation of measured data.

Tested material

Two insulation materials by two different producers were used. **SAMPLE A** was NEN-F 220/125 by EKO-BAL Rožnov spol. s r.o. (Czech Republic). **SAMPLE B** was Voltaflex 2598 by ISOVOLTA AG (Austria). **SAMPLE C** is virgin PET without cover layers PES by EKO-BAL Rožnov spol. s.r.o. SAMPLE C was prepared to find out influence of cover layers to volume resistance. These layers could change acquired results. Fig. 1: shows all three samples A, B and C.

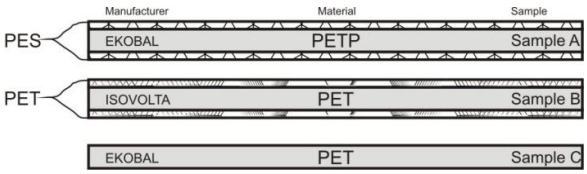

Fig. 1: Sample A, B and C layers description

SAMPLE A is a three layer insulation material. It consists of a main layer (PETP) of thickness of 125 μm and two layers on both sides made from PES. The total thickness is 22 μm. Its temperature class is H. SAMPLE B is a three layer insulation material, too. It consists of main layer PET of thickness of 125 μm and two layers on both sides made from structured PET different from SAMPLE A. The total thickness is 23 μm. Its temperature class is F/H. SAMPLE C is derived from SAMPLE A. It is only a middle layer PETP of thickness of 125 μm. SAMPLE C is prepared to recognize the influence of surface layers on results.

The experimental workplace and procedure for measuring

The experimental workplace consists of multimeter Keithley 6517A, testing cell Keithley Model 8009, software 6517 Hi-R Test, hot-air sterilizer SteriCell 22 and a personal computer connected via GPIB bus to a multimeter (see Fig. 2:).

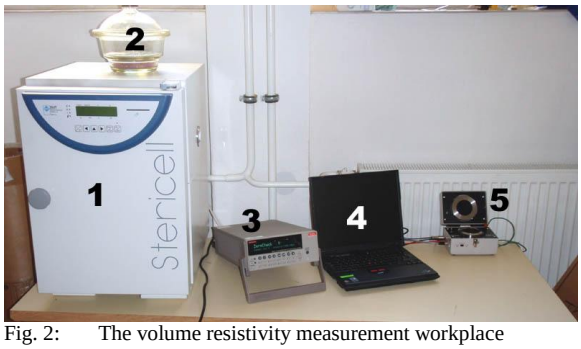

Fig. 2: The volume resistivity measurement workplace **1** hot air sterilizer, **2** valve desiccator, **3** multimeter HEITHLEY 6517A , **4** software Hi-R Test on PC, **5** testing cell Keithley Model 8009.

Fig. 3: The DSC Perkin Elmer – 1 gas, 2 DSC, 3 software PYRIS, 4 weighing machine.

For the purpose of determining the changes in the volume resistivity the Perkin Elmer DSC 6 was used (see Fig. 3:).
The procedure of measuring volume resistivity consists of these steps:

a) sample preparation size of 8 x 8 cm
b) hot-air sterilizer preheating to 60 °C
c) placing samples in hot-air sterilizer
d) conditioning for 30 minutes
e) thermal heating to 190 °C for 6 thousand hours
f) the consecutive retirement and conditioning
g) the volume resistivity measurement after 48 and 336 h

The most important matter was the reproducibility of results. It is well known the currents are about some pA by measuring volume resistivity of polymers. The testing of reproducibility was carried out in regard to measuring voltage (range 50 – 1000 V). Tab. 2. shows results. The S is standard deviation of volume resistivity.

Tab. 2: The selection of level voltage - results

U [V]	*50*	*200*	*400*	*800*
S [%]	10	6,6	1,9	0,6

Our results in Tab. 2. correspond to S according KEITHLEY's equation (1). The best reproducibility is carried out at 800 V. The applied voltage of 1 kV (maximum of 6517A) was not used because the maximum electric strength of polymer in application is about 3,6 kV/mm.

$$S = \Delta I_{BG} \frac{R}{U_{ALT}} + 0{,}1\% \ [\%] \qquad (1)$$

The alternating voltage regime which is implemented in multimeter KEITHLEY was used. Eleven pulses were applied during one measurement for one sample. Eight of them were used for the final evaluation. The first three pulses were to avoid transient effects at the beginning of measurement in a material. The time and count of pulses affect the accuracy and reproducibility, too. In our case 11 pulses and pulse time of 35 s were selected. It means the time measuring of one sample is 6 min and 25 s.
The effect of humidity was tested, too. Humidity 4 and 60 % were applied in three samples. The testing was done on non aged samples. Each sample was measured three times. The volume resistivity is independent on humidity of ambient.

Tab. 3: SAMPLE A volume resistivity for 4% humidity

4 %	*ρ_v[PΩcm]*		
SAMPLE A-1	2,23	2,32	2,28
SAMPLE A-2	2,35	2,33	2,38
SAMPLE A-3	2,21	2,20	2,22

Tab. 4: SAMPLE A volume resistivity for 60% humidity

60 %	*ρ_v[PΩcm]*		
SAMPLE A-1	2,36	2,37	2,28
SAMPLE A-2	2,36	2,27	2,23
SAMPLE A-3	2,23	2,23	2,24

Tab. 3: and Tab. 4: show the similar values of volume resistivity for both levels of humidity. Which means the volume resistivity is **independent** on humidity.

The procedure of DSC experiment consists of the following steps:

a) sample preparation, diameter of 5 mm, weight of 10-12 mg, material was pressed in to aluminous cell
b) sample weighing on Tchniproton weigh
c) the normalization of parameters 1 min, 25 °C
d) heating up to 275 °C, 15 °C/min
e) cooling down to 25 °C, 15 °C/min
f) the evaluation of measured enthalpy

The reproducibility of the acquired results was tested in this case, too. The test was only done for two testing samples with regard to the long term measuring cycle. Repeated measuring (e.g. ten times) was not possible to perform. The time of one temperature cycle was roughly 30 min. The measured curves by DSC were the same in all cases. The result allowed to perform measurement only once for each one sample.

The measured data evaluation

Three samples for each period of ageing t_a were prepared. It is necessary to avoid improper results by material nonuniformity. Each sample was measured ten times, thus thirty values of volume resistivity for each t_a were acquired. Due to number of results normal distribution can be used.

Tab. 5: SAMPLE A volume resistivity T_a = 2160 hours, t_a = 190 °C – all measured data for one T_a.

order num.	*SAMPLE A-1* ρ_v[PΩcm]	*SAMPLE A-2* ρ_v[PΩcm]	*SAMPLE A-3* ρ_v[PΩcm]
1	79,2	79,4	78,6
2	78,8	78,5	79,1
3	78,5	77,3	79,6
4	79,1	75,1	79,9
5	80,8	75,6	80,2
6	80,4	74,6	80,1
7	77,5	74,5	80,6
8	76,3	78,7	78,5
9	75,6	79,6	77,4
10	75,9	78,8	77,3
ρ_v = 78,2 ± 0,3 PΩcm			

The computing of expected value was done according to equation (2). The error of expected value was computed according to (3).

$$\overline{\rho}_v = \frac{\sum_{i=1}^{N} \rho_{vi}}{N} \qquad (2)$$

$$\hat{s} = \sqrt{\frac{\sum_{i=1}^{N} (\overline{\rho}_v - \rho_{vi})^2}{N(N-1)}} \qquad (3)$$

The same measuring and evaluation was used for all samples and times of ageing. The measurement of volume resistivity was carried out 27 times on SAMPLE A and B. SAMPLE C was measured 14 times because SAMPLE C arrived later during the experiment.

The enthalpy and start temperature of melting point were evaluated with software PYRIS. The value of enthalpy ΔHm was determined for each sample. The start temperature T_m of melting point was determined too. The crystallinity was evaluated according to (4). The enthalpy ΔH_{m100} has theoretical value of 140 J/g.

$$X_c = \frac{H_m}{H_{m100}} \cdot 100 \qquad (4)$$

EXPERIMENTAL RESULTS

The results are summarized in tables and graphs below. As well as in the Tab. 5 it was impossible to present all measured data due to extent of this article. The expected value of volume resistivity is presented for all t_a. The first volume resistivity results are presented. Furthermore, the rate of crystallinity is showed by DSC.

The volume resistivity results

The volume resistivity was measured after 48 h and 336 h of conditioning after temperature ageing. It could reveal whether the change is permanent or not. Tables Tab. 6:, Tab. 7:, Tab. 8: show all results measured and computed during experiment. The error of expected value is higher towards longer ageing time. The accuracy and reproducibility of measuring is very high. The measured data of volume resistivity can be considered as valid for further evaluation.

Tab. 6: SAMPLE A volume resistivity *Ta* = 190 °C – measured after 48 h and 336 h

	SAMPLE A – 48 h	*SAMPLE A – 336 h*
t_a[h]	ρ_v[PΩcm]	ρ_v[PΩcm]
0	2,32±0,01	2,32±0,01
1	2,79±0,01	2,80±0,01
5	3,87±0,01	3,87±0,01
24	7,34±0,01	7,33±0,01
48	12,3±0,01	12,3±0,01
192	45,6±0,1	44,8±0,1
216	57,2±0,1	54,6±0,1
384	59,4±0,1	58,3±0,1
552	61,1±0,1	59,4±0,1
720	63,2±0,2	62,7±0,1
816	74,2±0,2	71,5±0,2
1152	81,2±0,2	78,1±0,2
1488	85,5±0,3	83,7±0,3
1824	88,4±0,3	85,4±0,3
2160	78,2±0,3	75,3±0,3
2496	83,6±0,3	75,3±0,3
3168	81,4±0,3	76,4±0,3
3504	83,8±0,3	76,8±0,3
3840	84,6±0,3	77,6±0,3
4176	85,9±0,3	79,9±0,3
4512	83,5±0,3	79,5±0,3
4848	81,1±0,4	78,1±0,4
5184	80,4±0,4	77,4±0,4
5352	80,1±0,4	77,1±0,4
5592	88,2±0,4	79,2±0,4
5928	86,8±0,4	80,8±0,4
6096	76,3±0,4	78,3±0,4

The ageing times are different for SAMPLEs A, B and C. The delivery time of materials was different.

Tab. 7: SAMPLE B volume resistivity *Ta* = 190 °C – measured after 48 h and 336 h

	SAMPLE B – 48 h	*SAMPLE B – 336 h*
t_a[h]	ρ_V[PΩcm]	ρ_V[PΩcm]
0	3,54±0,01	3,54±0,01
1	4,42±0,01	4,38±0,01
5	5,13±0,01	5,07±0,01
24	7,49±0,01	7,41±0,01
48	8,28±0,01	7,28±0,01
192	10,3±0,1	9,6±0,1
312	35,7±0,2	33,7±0,1
648	70,3±0,1	68,3±0,1
984	82,6±0,2	80,2±0,2
1320	64,9±0,2	63,1±0,2
1656	57,9±0,2	56,4±0,2
1992	52,6±0,3	51,1±0,2
2328	55,5±0,3	53,8±0,2
3000	59,3±0,2	59,9±0,2
3336	65,8±0,2	66,2±0,3
3628	69,3±0,3	67,7±0,3
4008	77,1±0,3	75,1±0,3
4344	82,5±0,4	80,2±0,3
4680	81,1±0,4	79,1±0,4
5016	82,5±0,4	80,5±0,4
5230	81,8±0,4	79,8±0,4
5352	81,2±0,4	79,2±0,4

Tab. 8: SAMPLE C volume resistivity *Ta* = 190 °C – measured after 48 h and 336 h

	SAMPLE C – 48 h	*SAMPLE C – 336 h*
t_a[h]	ρ_V[PΩcm]	ρ_V[PΩcm]
0	3,55±0,01	3,55±0,01
1	4,95±0,01	4,92±0,01
5	6,53±0,01	6,51±0,01
24	11,2±0,1	11,9±0,1
48	19,5±0,1	17,5±0,1
192	58,3±0,2	56,3±0,1
300	63,5±0,2	62,5±0,2
408	75,2±0,2	73,2±0,2
816	85,9±0,2	84,6±0,2
1152	85,6±0,2	84,1±0,2
1488	81,9±0,2	80,1±0,3
1840	82,5±0,3	80,2±0,3
2160	82,7±0,3	80,3±0,3
2496	82,2±0,3	80,1±0,3
2800	83,1±0,3	81,5±0,4
3168	83,9±0,3	82,4±0,4
3504	84,8±0,3	82,9±0,4

Fig. 4:, Fig. 5: and Fig. 6: show graphical interpretation of measured results. Each graph shows the results of measuring after 48 and 336 hours. The squared red curve is lower for all SAMPLEs. It can indicate the volume resistivity change as not permanent. Fig. 4: and Fig. 6: are very similar. These show the side layers of sample are not important with respect to measurement accuracy. To conclude, the side layers do not affect the results.

There is a very steep volume resistivity increase in the first thousand hours. Afterwards, the volume resistivity has roughly the same value about of 80 PΩcm. The material seems to be saturated. The question is which physical phenomenon affects the volume resistivity.

SAMPLE B has between one thousand and four thousand hours significant drawdown curve (see Fig. 5:). It was confirmed for both 24 and 336 hour measurements. It cannot be a measurement error. The question is what changes occurred in the material during the thermal ageing.

Both questions are not easy to answer now. It is necessary to perform more experiments to be able to answer questions induced by these results.

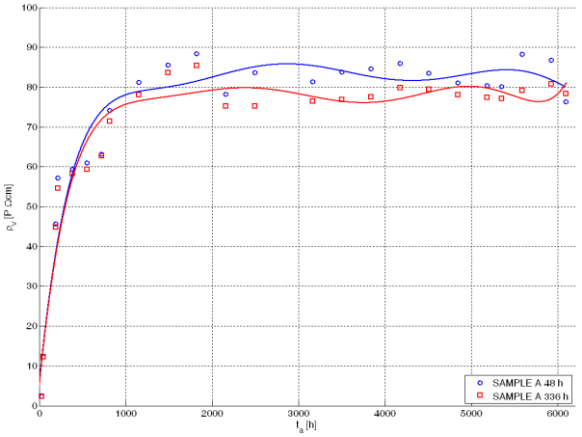

Fig. 4: SAMPLE A volume resistivity after 48 and 336 h

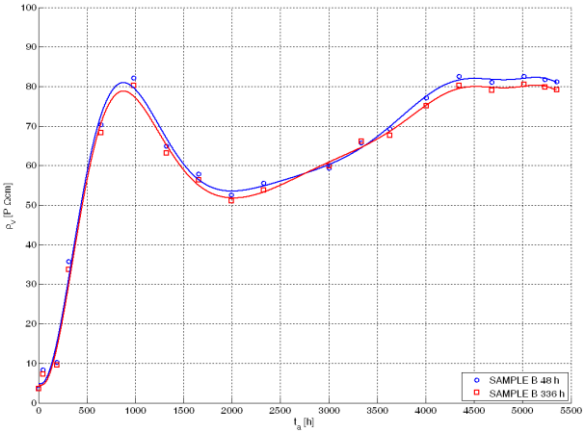

Fig. 5: SAMPLE B volume resistivity after 48 and 336 h

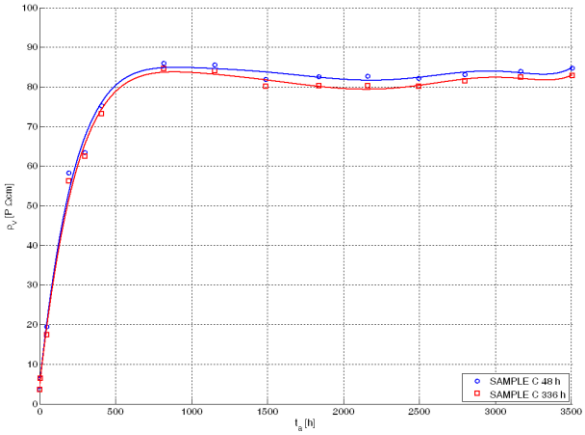

Fig. 6: SAMPLE C volume resistivity after 48 and 336 h

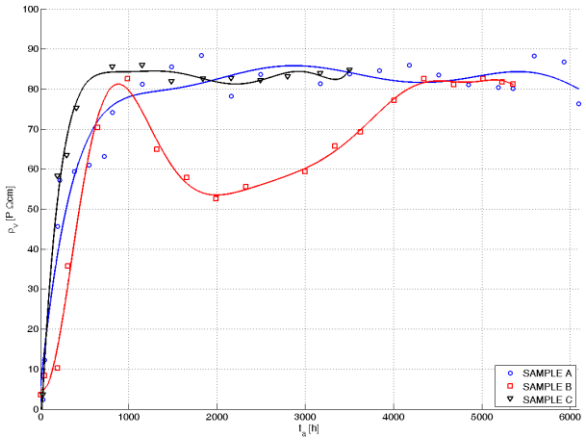

Fig. 7: SAMPLE A, B and C volume resistivity after 48 h

The last Fig. 7: shows all results in one graph. The results can be compared easily.

The crystallinity results

The crystallinity measurement was carried out to determine changes in the structure. The crystallinity of all samples was measured. Fig. 8: shows results of DSC measurement. The crystallinity is roughly similar to all three samples. All measured and computed data are shown in Tab. 9: and Tab. 10: The crystallinity rose very steeply during the first thousand hours. After that the crystallinity gently grew up to 62 %. The shape of the crystallinity curve is similar to the volume resistivity curve.

Tab. 9: Tab. 9. The SAMPLE A and B crystallinity T_a = 190 °C

SAMPLE A			*SAMPLE B*		
t_a[h]	ΔH_m[J/g]	X_c[%]	t_a[h]	ΔH_m[J/g]	X_c[%]
0	43,8	31,2	0	42,1	30,1
1	43,9	31,3	1	42,2	30,1
5	44,0	31,4	5	43,3	30,9
24	45,4	32,4	24	48,5	34,6
48	45,8	32,7	48	57,8	41,3
192	69,0	49,3	192	66,9	47,8
216	70,2	50,2	312	75,9	54,2
384	69,6	49,7	648	78,3	55,9
552	76,5	54,6	984	75,7	54,1
720	77,5	55,4	1320	79,9	57,1
816	78,5	56,1	1656	79,6	56,8
1152	79,7	56,9	1992	78,8	56,3
1488	79,3	56,6	2328	82,3	58,8
1824	83,3	59,5	3000	81,6	58,3
2160	82,7	59,1	3336	78,7	56,2
2496	85,5	58,9	3628	84,9	60,6
3168	86,3	61,7	4008	82,3	58,8
3504	87,3	61,3	4344	79,8	57,1
3840	85,6	61,2	4680	79,8	57,1
4176	86,0	61,4	5016	84,1	60,1
4512	85,5	61,1	5352	85,8	61,3
4848	88,1	62,9			
5184	85,2	60,8			
5352	89,0	63,5			
5592	86,1	61,5			
5928	84,4	60,3			
6096	86,9	62,1			

Tab. 10: The SAMPLE C crystallinity T_a = 190 °C

SAMPLE C			*SAMPLE C*		
t_a[h]	ΔH_m[J/g]	X_c[%]	t_a[h]	ΔH_m[J/g]	X_c[%]
0	46,5	33,2	816	81,7	58,4
1	49,7	35,5	1152	82,6	59,1
5	52,4	37,4	1488	84,2	60,1
24	64,7	46,3	2160	86,2	61,6
48	64,2	45,9	2496	85,9	61,4
192	69,6	49,7	3168	87,3	62,4
408	77,0	55,1	3504	88,6	63,3

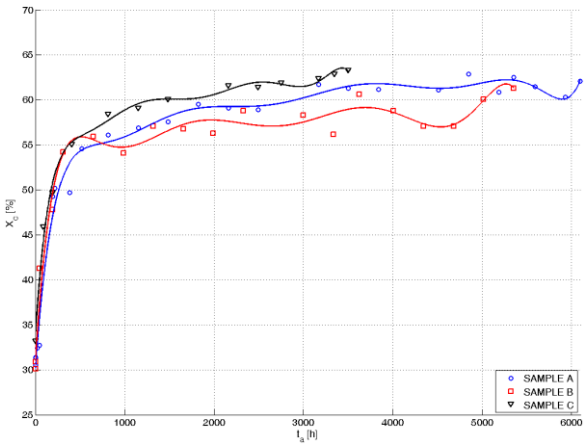

Fig. 8: The SAMPLE A, B and C crystallinity during experiment

The results of volume resistivity and DSC do not correspond to SAMPLE B. The crystallinity rose and after that was roughly the same, whilst the volume resistivity rose, then went down and rose again. The question remains why SAMPLE A and SAMPLE C correspond and SAMPLE B does not (compare Fig. 7:, and Fig. 8:). Perhaps it shows that crystallinity has no effect on value of volume resistivity. The similarity can be done only by chance.

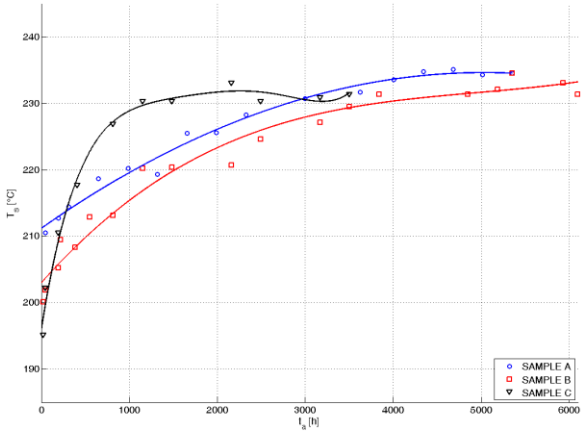

Fig. 9: SAMPLE A, B and C melting temperature during experiment

The temperature of melting point is shown in Fig. 9:. The graph shows the temperature of melting point as higher for longer periods of thermal ageing which means the material needs more energy to decompose

the crystalline structure. It corresponds with the degree of crystallinity in Fig. 8:.

CONCLUSION

This work presents the results of thermal ageing experiments in PET material. Two experiments were carried out to find some changes in volume resistivity and crystallinity of the material.

There were found some significant changes in volume resistivity during thermal ageing. The volume resistivity rose forty times compared to its original state. It is not certain whether the change has a permanent or temporary character. It looks as if the volume resistivity went down after 336 hours of thermal ageing. It must be verified by the next experiment in the near future.

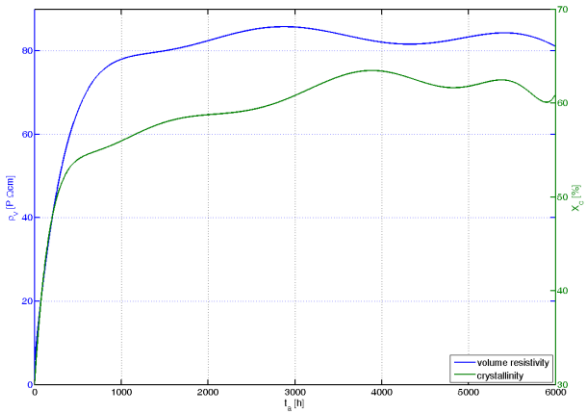

Fig. 10: SAMPLE A comparison of volume resistivity and crystallinity

The crystallinity has changed significantly too. It corresponds with the volume resistivity as shown Fig. 10:. There are some results that must be verified by the help of further experiments as shown in Fig. 11:.

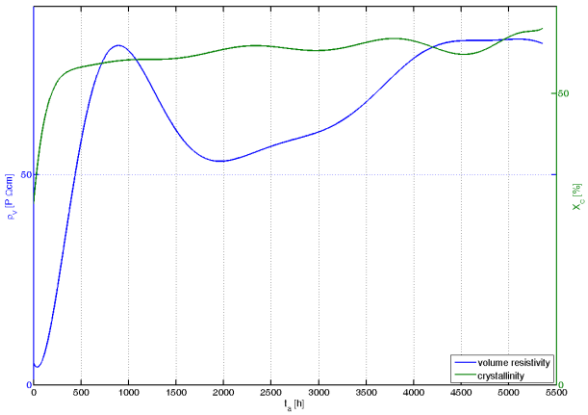

Fig. 11: SAMPLE B comparison of volume resistivity and crystallinity

At the end of this paper, it must be honestly announced that the dependence between the volume resistivity and crystallinity was not validated. There is a high probability that the volume resistivity growth has a different cause than expected. It is the main task for further studies and experiments to find out more.

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Acknowledgments: The authors would like to thank the MEYS for funding the project 1M0553 Research centre textile II.