VERIFICATION OF POSSIBILITIES OF EVALUATION OF CHANGING OF POLYMERIC SURFACE PROPERTIES AND BEHAVIOUR EVOKED BY SUN RADIATION AND TEMPERATURE LOADING

Lucie ŠIMEČKOVÁ, Simona DUDÁČKOVÁ, Ivo ŠTĚPÁNEK, Andrea MERTOVÁ

Institute of Thermomechanics CAS, v.v.i., Czech Republic, EU
simeckova@it.cas.cz

Abstract

The paper is devoted by simulation of conditions of sun radiation in correlation with real loading in operational conditions of selected polymeric materials. In the second the paper is devoted by simulation of temperature conditions of loading again in correlation with temperature loading in real operational conditions of selected polymeric materials. There are tested sensitivity of selected analytic methods in connection on running processes of ageing of polymeric materials for evaluation of initial of radiation ageing and temperature ageing with possibilities of prediction of evolution of running changing of properties and behaviour of evaluated materials. There are tested method x-ray fluorescence analysis and parameters of this method are optimized for increasing sensitivity for evaluation initial changing of properties and behaviour of tested materials on the base the first results. Indentation methods are used for analysis of changing of mechanical properties and behaviour namely static nanoindentation and scratch nanoindentation. These methods are optimized on the base the first results too. There are used more possibilities for control of indentation tests by the use different modes of measurements namely simple and cyclic measurement, setting different rate of loading and using different type of indentors for realization of measurements.

Keywords: Nanoindentation, scratch indentation, radiation ageing, temperature ageing, x-ray fluorescence

1. INTRODUCTION

The thin films are created by deposition process as controled process of its creation by deposition parameters. The thin films are created by modification process, too, with controled the parameters of these technology processes. The thin films are created as single, multilayers, gradient and so on. The degradation processes create thin films, too, on surface or as modification to the depth of material systems from surface. This kind of thin film - substrate systems are multilayer systems or gradient systems. The control of process of degradation [1] is possible by sensitive analytical methods as nanoindentation with different maximal load of indentation and different modes of measurement. Different analytical methods were optimized during solution goals correlation between deposition parameters and properties and behaviour of systems thin film - substrate [2] and solution of goals to optimize analytical methods for possibility evaluation of systems thin film - substrate with very different resistivity, hardness, thickness, substrate [3] and so on. These experiences are used for optimisation analytical methods for evaluation of surfaces after radiation degradation process by sun radiation and temperature degradation processes.

2. X-RAY FLUORESCENT METHOD

Surface sensitive method for evaluation of changing chemical properties is x-ray fluorescent method. This method gives the possibility to evaluate chemical composition from relatively small and relatively large depth for evaluation properties and behaviour in degraded surface layers by temperature ageing processes and by radiation ageing processes with radiation simulated sun radiation. There is very important at the first to analyse starting point of chemical composition before ageing process. The polymer materials are namely amorphous

and x-ray fluorescent spectrums are very complicated but for evaluation changing in composition it is sufficient, because we need on the basic to analys of changing of composition and not real composition. At the second here are possibilities to analyses changing of thickness of polymeric samples, because x-ray goes through the samples and analys composition of holder, too, under samples of polymer. From this point of view we changed the material composition under polymer samples and we evaluted the changing of spectrums after ageing processes as changing of transmitance through samples.

3. AGEING PROCESS OF TEMPERATURE DEGRADATION OF POLYMER MATERIALS

The samples of polymer materials were exposed to action higher temperature environment in chamber. There was realised experiments with temperature heating in long time by step by step stress and realisation temperature ageing to the 1000 and 2000 hours and heating was realise to the temperature 60 st. C. The measurement was realised on 2 different type of samples new and after real ageing process and after rfeal condition of operation.

4. AGEING PROCESS OF SUN RADIATION SIMULATED BY XE RADIATION IN CHAMBER

The samples of polymer materials were exposed to action sun radiation in sun radiation simulated chamber with three Xe lamps. The radiation was setting on 60 W/m2. In the chamber temperature was setting on 55 st. C and humidity on 50 %. The radiation was acted in the long time on surfaces of samples and realisation of radiation ageing process was to the 1000 and 2000 hours.

5. ANALYSIS POLYMER MATERIALS AFTER AGEING PROCESS BY X-RAY FLUORESCENT

The changing of chemical composition after ageing was tryed to evaluate by x-ray fluorescent method. Here are three possibilities - analyse chemical composition after calibration for analysis chemical composition, analyse thickness of thin films on surface of basic material and analyse x-ray fluorescent spectrum. Evaluation after calibration for analysis of chemical composition is not possible to use, because we do not have standards for this analysis. Evaluation after calibration on analysis of thickness of thin films created by degradation process is not possible to use, too, because there is not contrast in chemical composition of degraded surface layers and basic state of substrate. The analysis of changing of x -ray fluorescent spectrum gives acceptable possibility to evaluate changing after ageing process. The standard measurement give spectrum with high noise. There was realised changing of measured parameters and setting of equipment for decreasing noise and get more precise spectrums. **Figure 1** show x-ray fluorescent spectrums before and after 1000 and 2000 hours sun radiation. There is possible to view changing in polymer material after sun radiation after decrease noise. **Figure 2** show spectrums after 1000 hours sun radiation and 1000 hours temperature heating on 60 st. C. This is measured on sample from standard of Cu. Here is possible to view differences after action sun and after action temperature, too. Better resolution for possibility evaluate changing is on **Figure 3** and **Figure 4**, where are spectrums measured on sample from standard of Ni.

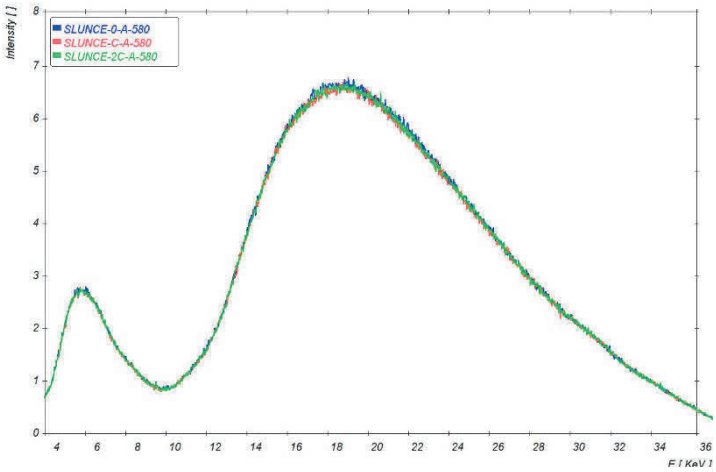

Figure 1 X-ray fluorescent spectrums before and after 1000 and 2000 hours sun radiation

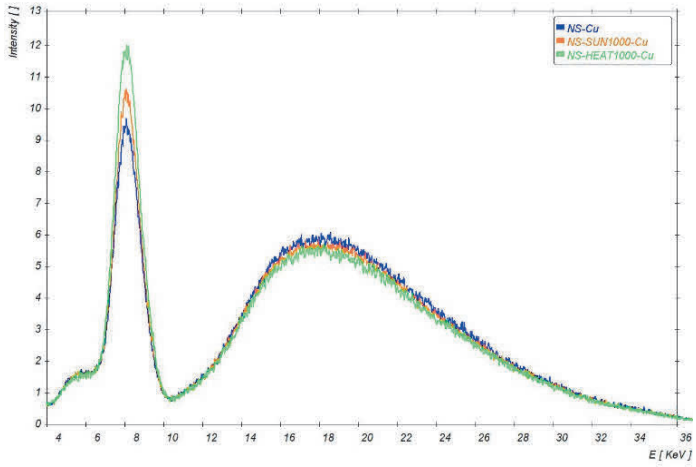

Figure 2 X-ray fluorescent spectrums before and after 1000 sun radiation and 1000 hours temeprature heating - measurement on standard sample from Cu

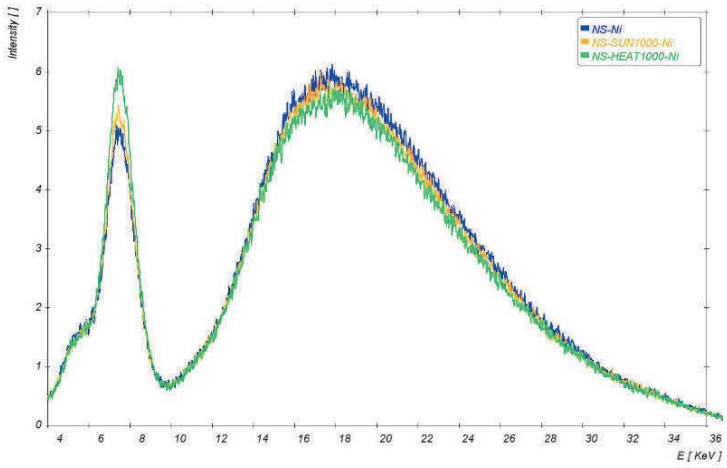

Figure 3 X-ray fluorescent spectrums before and after 1000 sun radiation and 1000 hours temeprature heating - measurement on standard sample from Ni

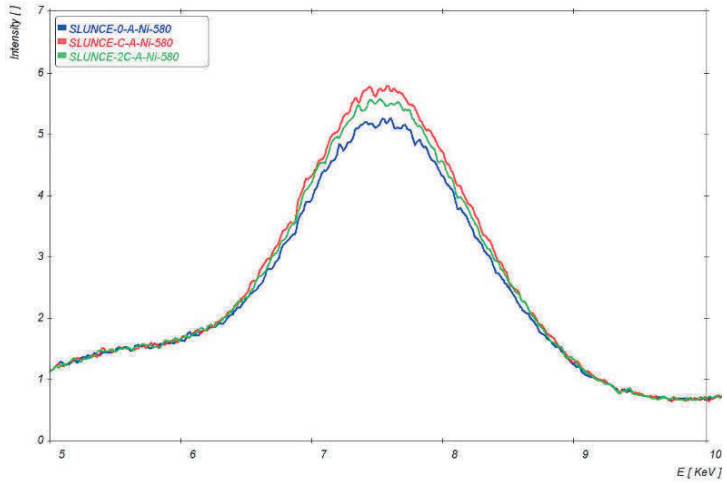

Figure 4 X-ray fluorescent spectrums before and after 1000 sun radiation and 1000 and 2000 hours sun radiation - measurement on standard sample from Ni

6. ANALYSIS POLYMER MATERIALS AFTER AGEING PROCESS BY INDENTATION

Indentation by Rockwell indentor with radius 0.01 mm

The indentation was realised by indentor Rockwell with radius 0.01 mm and maximal of load 100 mN. There was measured indetation curves during loading and in the next step was realised measurement during unloading. The measurement realised from both sides of polymer samples show differences from both sides and differences after action sun radiation, too. The sun radiation acted hardening of surfaces on both sides. The elasticity after sun radiation decrease on both sides too.

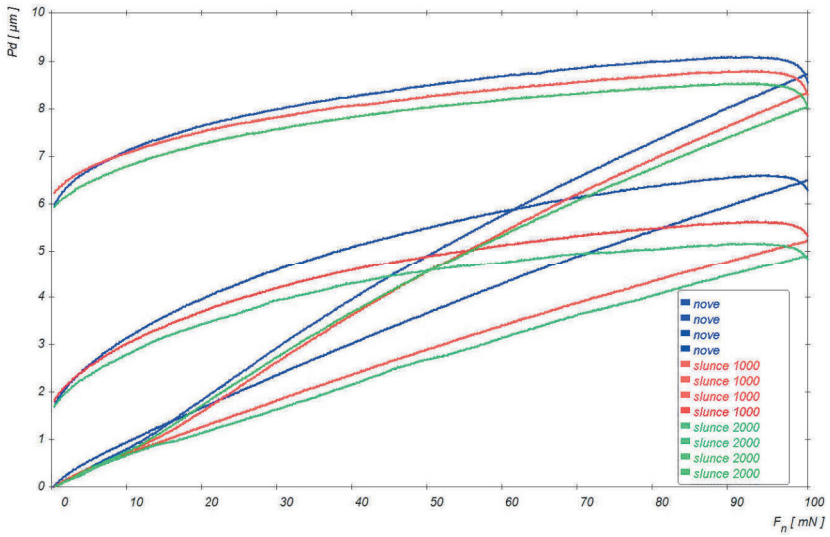

Figure 5 Indentation curves measured during loading and unloading with maximal load 100 mN

By influence sun radiation increase surface hardness on the both sides and there is changed rate of plastic and elastic deformation (**Figure 5**). With increasing action of sun decrease of elasticity of surface on both sides of polymer samples.

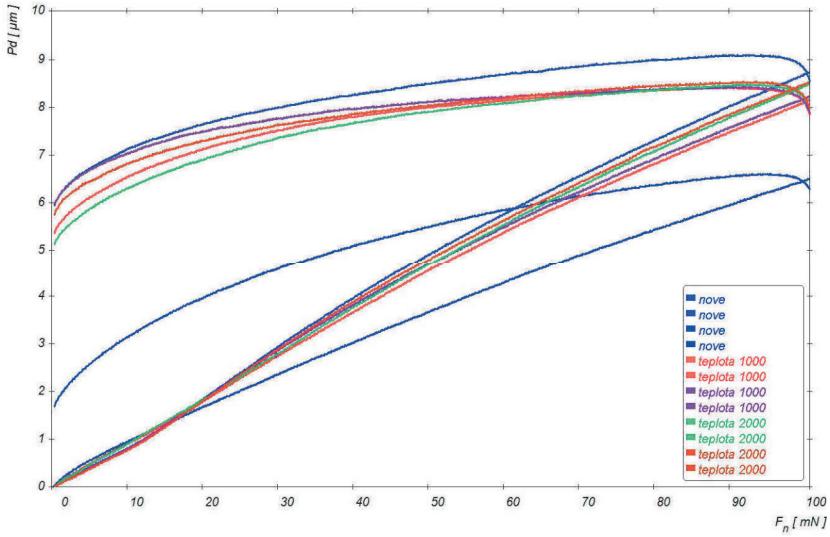

Figure 6 Indentation curves after action of temperature

Figure 6 show influence of long term acted of temperature on changing mechanical properties and behaviour of surfaces on both parts of samples from polymer material. There is possible to view what surface hardness on one side which was in started point with higher hardness decrease but hardness on side, where was

hardness lower, hardness increase. Probably the surface tretment of one side of polymer sample lost its propreties during long time acted temperature.

Scratch indentation on nanoscratch tester

Scratch indentation was realised with indentor Rockwell with diameter 0.01 mm and maximal normal force 100 mN. The rate of scratches was 1 mm per minutes. Depth of indentation was measured during scratch measurement and friction force was measured inf dependence on acted normal force during all measurement. The measurement was realised on both sides of polymer samples after sun radiation and after temperatue heating which introduces in above mentioned.

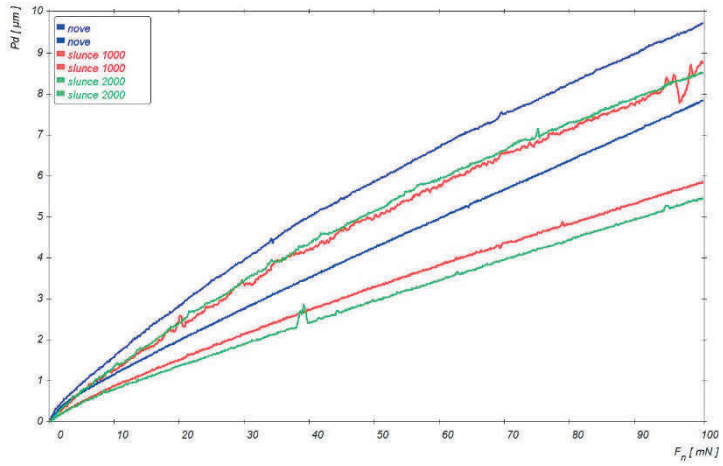

Figure 7 Dependence depth of indentation on acted normal force during scratch measurement on both sides of polymer samples before and after different time of action of sun radiation

The reauslt show what with increasing of time of action of sun radiation decrease depth of indentaion on both sides of polymer sample. The higher differences are measured on side which was before measurement treated to the influnce of sun. After both time steps of radiation influence increase hardness of surfaces on both sides of polymer sample. There was changed rate of elastic and plastic deformation, too. This is analysed from rate of depth during scratch measured and measured after scratc test.

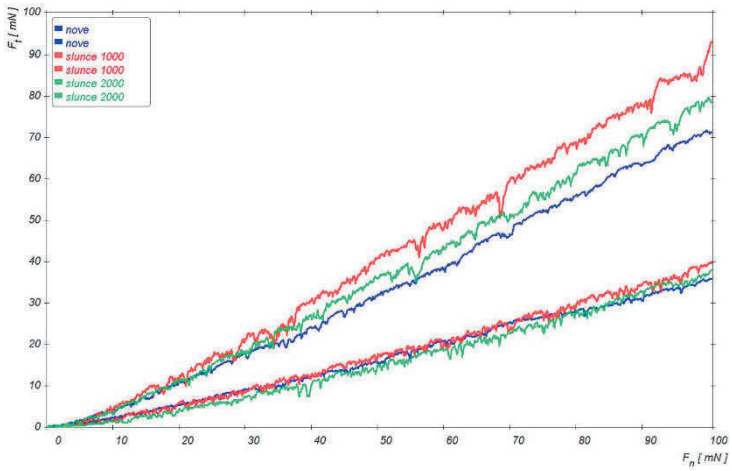

Figure 8 Dependence of friction force on acted normal force measured during scratch measurement

The friction force not changed on the side with treatment after sun radiation 1000 and 2000 hours, too. On the side without treatment surface friction force increase after sun radiation. This show what increase brittle properties of surface of polymer material.

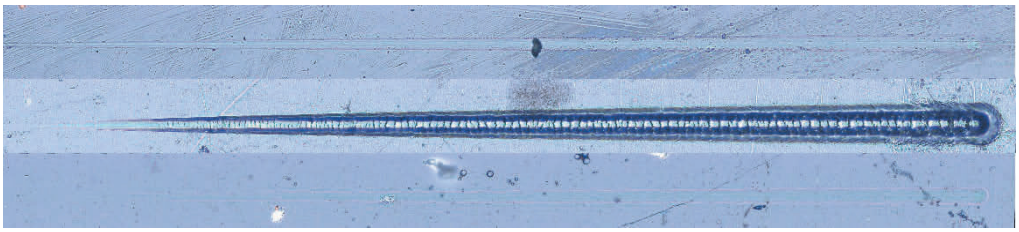

Figure 9 Morphology of failures aroung and in the scratches during indentation created

Figure 9 show the morphology of failures after realised scratch measurement after 1000 and 2000 hours of action sun radiation on side with treatment surface. The results show what at the start the surface is hardened by surface treatment. There was increased failuring after 1000 hours sun radiation becasuse the surface treatment layers lost their properties. In the other step of loading of sun radiation the surface layer is changed by radiation and lost brittle failure.

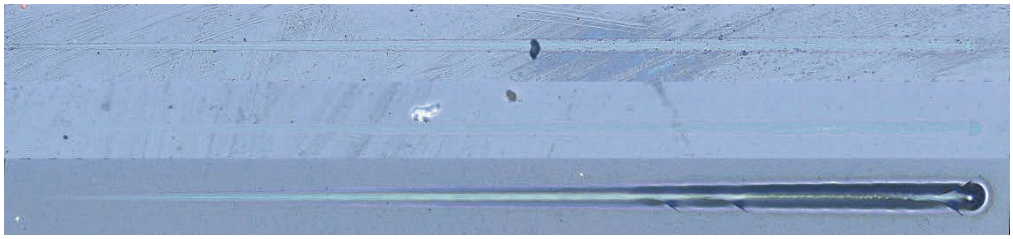

Figure 10 Morphology of failures aroung and in the scratches during indentation created

The morphology of failures around and in the scratches after scratch indentation with increased normal force during measurement show what brittle failure was changed after temperature acted in the long term. In the next step o temperature heating increase depth of indentation during scratch test and in the scratches increase and show, what chacter of failure in the scratches in the end of scratch identation in large force chaged to brittle failure.

Cyclic indentation measurement

In the next step there was realised cyclic indentation measurement. Number of cycles was 20 and maximal load was 1000 mN. The measurement was with using Berkovich indentor. The measurement was realised before and after sun radiation on sample of polymer material. There is possible to view what after 1000 and 2000 hours sun radiation increase surface hardness, but after cyclic indentation the final depth was on samle after 2000 hours sun radiation, becasuse increace brittle failuring of surface with increasing nimber of cycles.

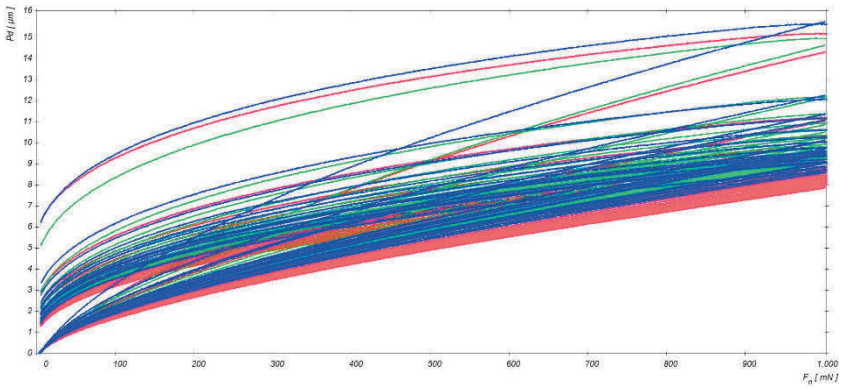

Figure 11 Cyclic indentation curves on samples before and after 1000 and 2000 hours sun radiation

7. CONCLUSION

There was tested and optimized the possibilities of using analytic method for surface analysis for application on evaluation of surfaces of polymer material after different ageing proces. In the second there was tested sensitivity for evaluation of initiation of changing of properties after degradation process. The results show what sensitivity of x-ray fluorescent method was increased for possibilities to evaluate small changing in properties after ageing process temperature, which was realized in temperature chamber Elastocon 1000 and 2000 hours, and sun radiation ageing process, which was realised in chamber with simulation sun radiation Q-Sun 1000 and 2000 hours. In the third there was tested sensitivity of nanoindentation method static and scratch indentation method and its increasing by optimization of parameters of measurement and preparing new method for evaluation polymer materials after different ageing processes.

ACKNOWLEDGEMENTS

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REFERENCES

[1] FECHINE, G.J.M., RABELLO, M.S., SOUTO MAIOR, R.M., CATALANI, L.H. Surface characterization of photodegraded poly(ethylene terephthalate). the effect of ultraviolet absorbers. Polymer, 45 (7). 2004. pp. 2303-2308.

[2] ČERNÝ, F., ŠTEFEC, R., GUROVIČ, J., HOREJS, S., FAJKUS, M. Ti(C,N) Films Deposited from Organometallics Using LT PACVD. Fizika plazmy. 2006. 49(0), pp. 494-496. ISSN 0367-2921.

[3] JAKUBÉCZYOVÁ, D. , KOČÍK, M., HVIZDOŠ, P. Multicomponent thin films deposited by PVD ARC and LARC technology. In Materials Science (Medžiagotyra). 2014. vol. 20, no. 1, pp. 36-41. ISSN 1392-1320.