

## OPTIMALIZATION OF EVALUATION OF INITIATION OF TEMPERATURE AGEING PROCESSES OF SELECTED CABLE ISOLATION

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### Abstract

The paper is devoted of verification of possibilities of evaluation of initial changing of properties and behaviour of selected polymeric isolation of cables after defined temperature loading simulated of external operational conditions and simulated temperature loading evoked by current loading. There are evaluated initial properties and behaviour of selected polymeric cable isolations and properties and behaviour after natural ageing process and controlled accelerated ageing process during higher temperature loading and different time of exposition to the higher temperature. The method of x-ray fluorescence analysis is used for evaluation of differences of changing of properties and behaviour of polymeric isolation cable materials after natural ageing process and after accelerated controlled ageing processes. There are verificated the possibilities for analysis differences in behaviour of polymeric insulation materials after different ageing process. The paper is namely devoted by verification of the possibilities analytic methods for evaluation initial of changing of properties and behaviour by nanoindentation methods for analysis very small differences in mechanical properties and behaviour of surface layers of polymeric materials used in the application isolation of cables. There are optimized the parameters of nanoindentation measurements for analysis of differences in hardness and elastic plastic behaviour.

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

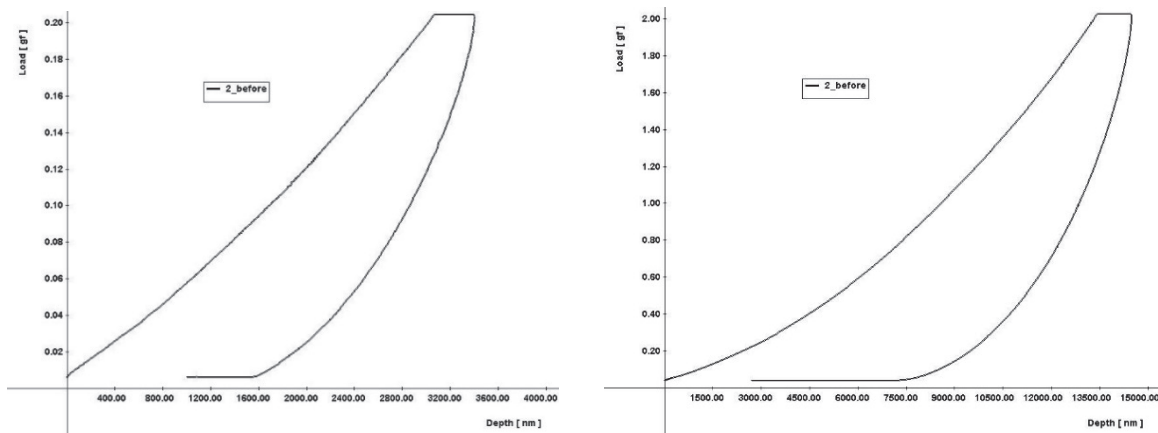
### 1. INTRODUCTION

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 [1]. The thin films are created as single, multilayers, gradient and so on [2]. The degradation processes create thin films, too, on surface or as modification to the depth of material systems from surface [3]. This kind of thin film - substrate systems are multilayer systems or gradient systems. The control of process of degradation 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 and solution of goals to optimize analytical methods for possibility evaluation of systems thin film - substrate with very different resistivity, hardness, thickness, substrate and so on. These experiences are used for optimisation analytical methods for evaluation of surfaces after corrosive and temperature degradation processes.

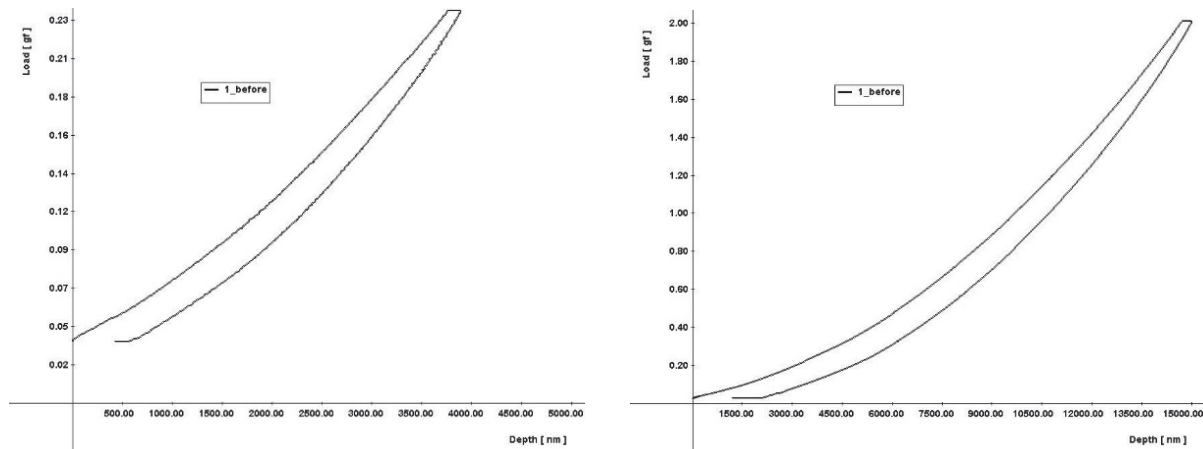
### 2. NANOINDENTATION MEASUREMENT OF POLYMER MATERIALS

At the first there are realised the basic evaluation of properties and behaviour of different type of polymer materials by nanoindentation. The standard measured mode of nanoindentation was used with setting different maximal value of load. The indentation curves measured during loading, time delay in maximal load and during unloading show much more elastic deformation then plastic deformation on sample 1. Only nanoindentation

has possibility to evaluate materials with marked elastic deformation. The limit of maximal indentation depth is about 20 microns. The maximal load must be reduced from this point of view. The properties and behaviour can be different in different depth from point of view natural ageing of polymer materials and from point of view producing of polymer materials, too. Indentation with different value of load brings information about properties and behaviour from different depth under surface (**Figure 1** and **Figure 2**).



**Figure 1** Indentation curves measured on surface of polymer material sample 1



**Figure 2** Indentation curves measured on surface of polymer material sample 2

### 3. CYCLIC NANOINDENTATION MEASUREMENT OF POLYMER MATERIALS

The cyclic nanoindentation bring much more information about behaviour of surface of polymer materials because during indentation step by step increase maximal load during several indentation tests with evaluation depth during loading, time delay in maximal load and during unloading. Step by step increasing depth in maximal load bring information from different depth from surface about changing of elastic and plastic deformation, changing of hardness in different depth and hardening during indentation process of stress. Here is possible to evaluate natural ageing created from time of production polymer material to the time of evaluation properties. These measurement give a start point for evaluation of changing after non-natural ageing created by temperature heating in long time. The measurement was created with using different value of final maximal load in the end of all cyclic measurement (**Figure 3** and **Figure 4**). This setting increase information from large interval of depth under surface and increase sensitivity in small depth under surface, too.

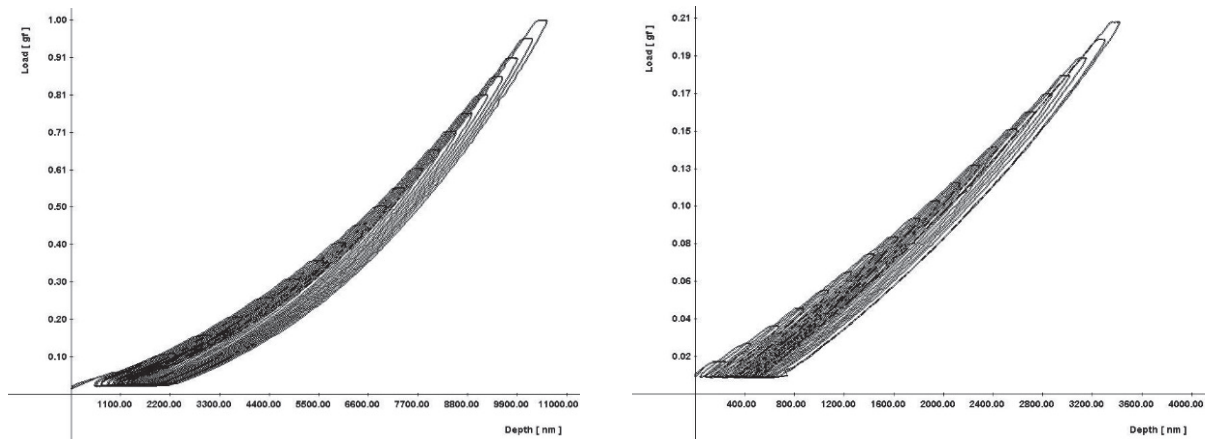


Figure 3 Cyclic indentation curves measured on surface of polymer material sample 1

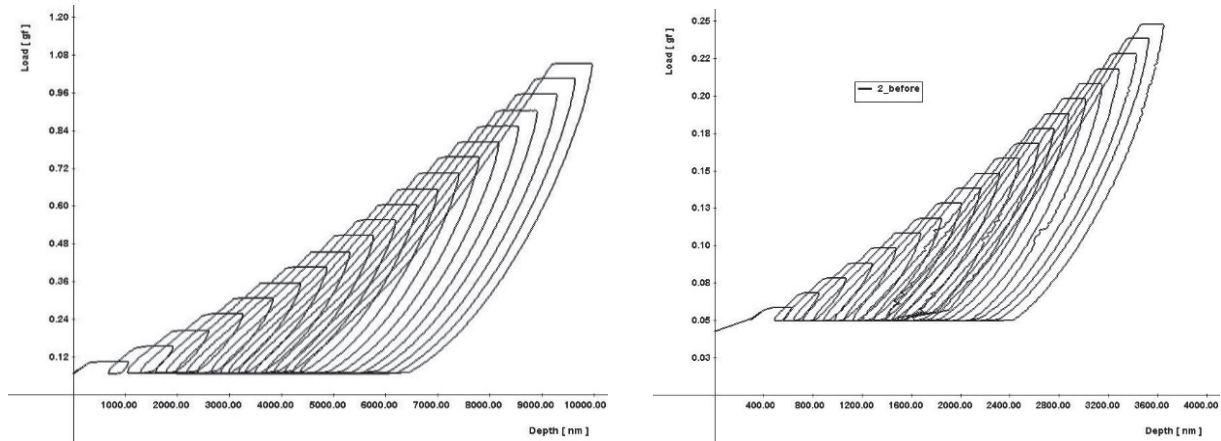


Figure 4 Cyclic indentation curves measured on surface of polymer material sample 2

#### 4. EVALUATION OF CHEMICAL COMPOSITION OF POLYMER MATERIALS

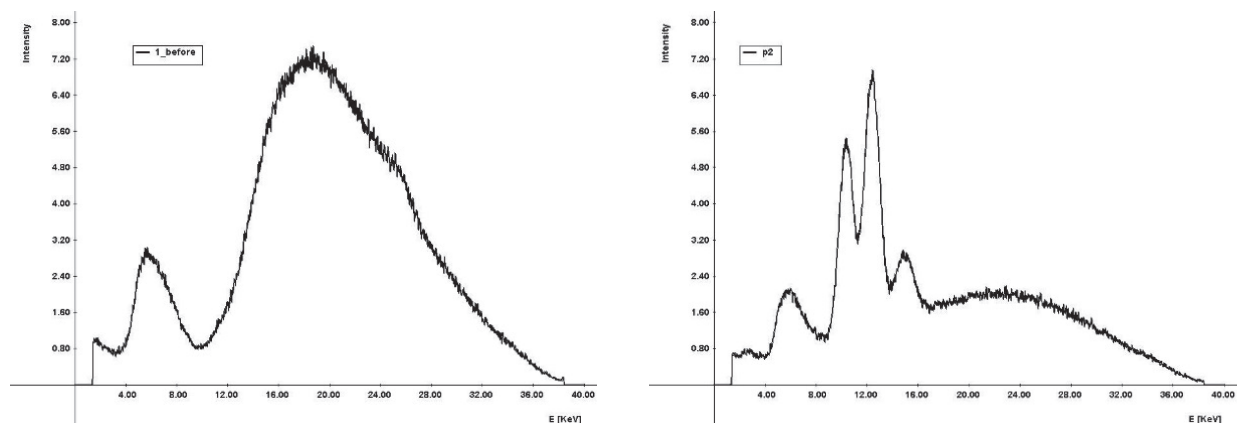


Figure 5 X-ray fluorescent spectrum on sample 1 and sample 2 of polymer material

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 corrosive stress, temperature stress therefore ageing process during temperature heating in long time, too. There is very important at the first to

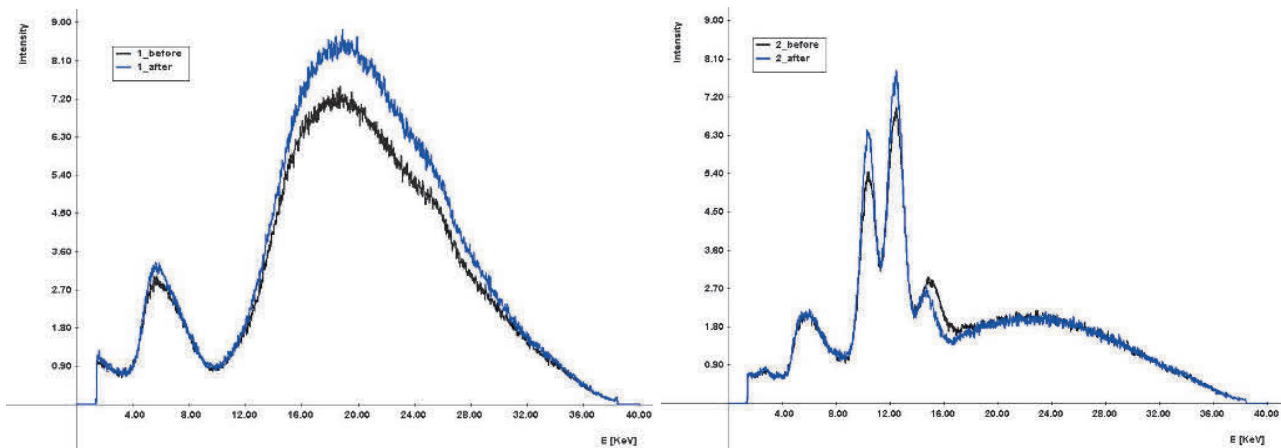
analyse starting point of chemical composition before ageing process (**Figure 5**). The polymer materials are namely amorphous and x-ray fluorescent spectrums are very complicated but for evaluation changing in composition it is sufficient. The polymer materials have different additives and from evaluation of amount of these additives give good possibility for evaluation its changing.

## 5. AGEING PROCESS OF TEMPERATURE DEGRADATION OF POLYMER MATERIALS

The samples of polymer materials were exposed to action high temperature environment in furnace. There was realised experiments with temperature heating in long time by step by step stress and realisation temperature ageing to the 800 hours and heating was realise to about 100 C. The measurement was realised on 2 different samples from 2 different kinds of polymer materials. The sample 2 of polymer materials has as marked additive Pb.

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

The changing of chemical composition after ageing was tried 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 6**.

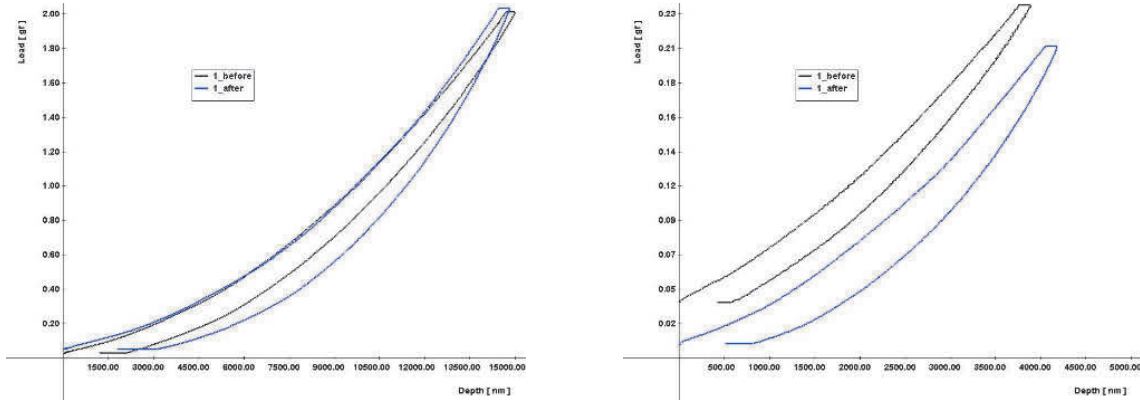


**Figure 6** X-ray fluorescent spectrums on sample 1 and sample 2 of polymer material before and after ageing process of temperature stress

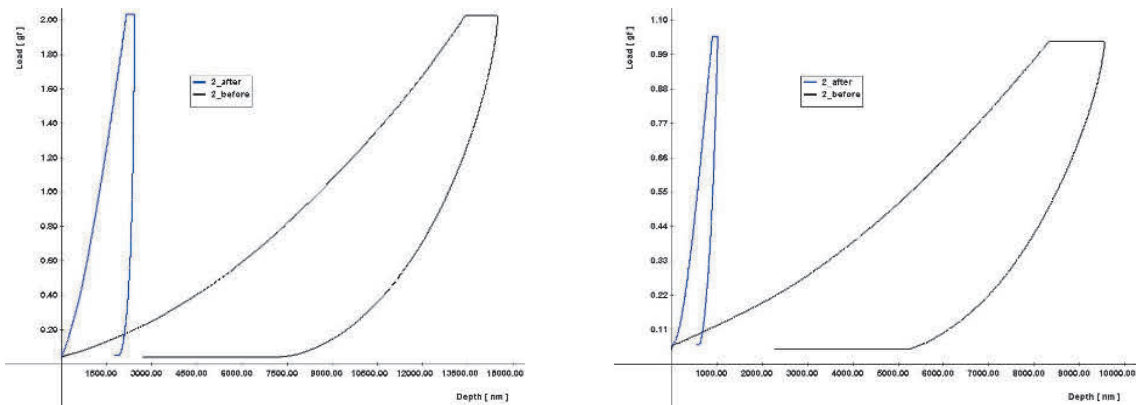
## 7. ANALYSIS OF CHANGING MECHANICAL BEHAVIOUR BY NANOINDENTATION

The samples of polymer materials were measured by nanoindentation after ageing process. At the first the same value of maximal load was used for measurement samples of polymer materials after ageing process for possibilities comparing mechanical behaviour before and after ageing process of degradation. At the second large value of maximal load was used for evaluation changing properties to maximal depth under

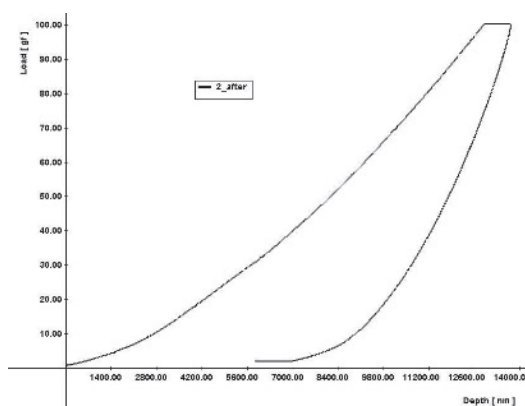
surface, which enable nanoindentation measurement (Figure 7 - Figure 9). It is about 20 microns to the depth under surface, but measurement is influenced from higher depth.



**Figure 7** Indentation curves measured on polymer material - sample 1 after ageing



**Figure 8** Indentation curves measured on polymer material - sample 2 after ageing



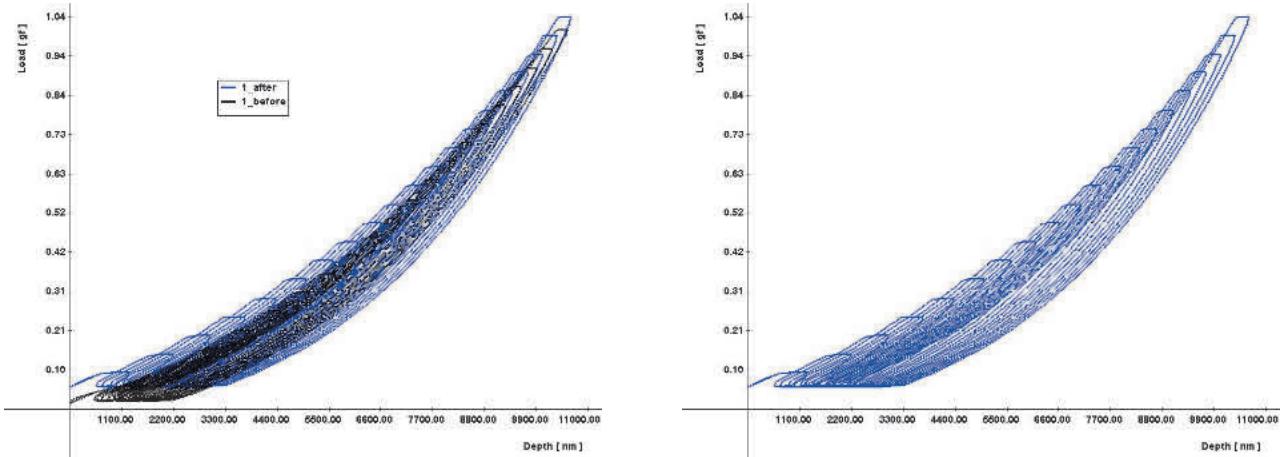
**Figure 9** Indentation curve measured on polymer material - sample 2 after ageing

## 8. ANALYSIS OF CHANGING MECHANICAL BEHAVIOUR BY CYCLIC INDENTATION

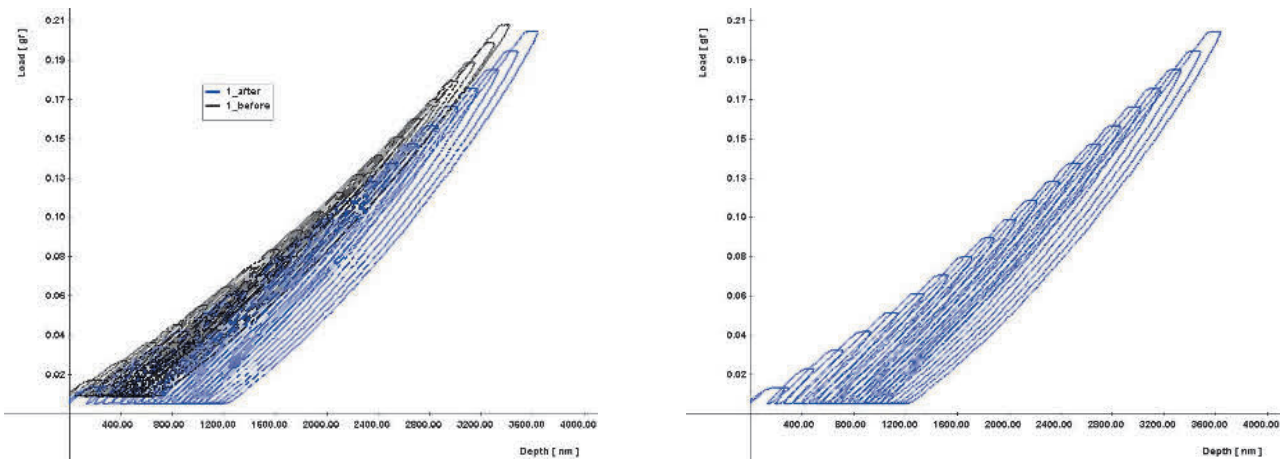
The samples of polymer materials were measured in the other step by cyclic nanoindentation after ageing process of degradation. At the first the measurement from point of view good comparison between before and after degradation process was realised with the same value of maximal load. At the second the measurement



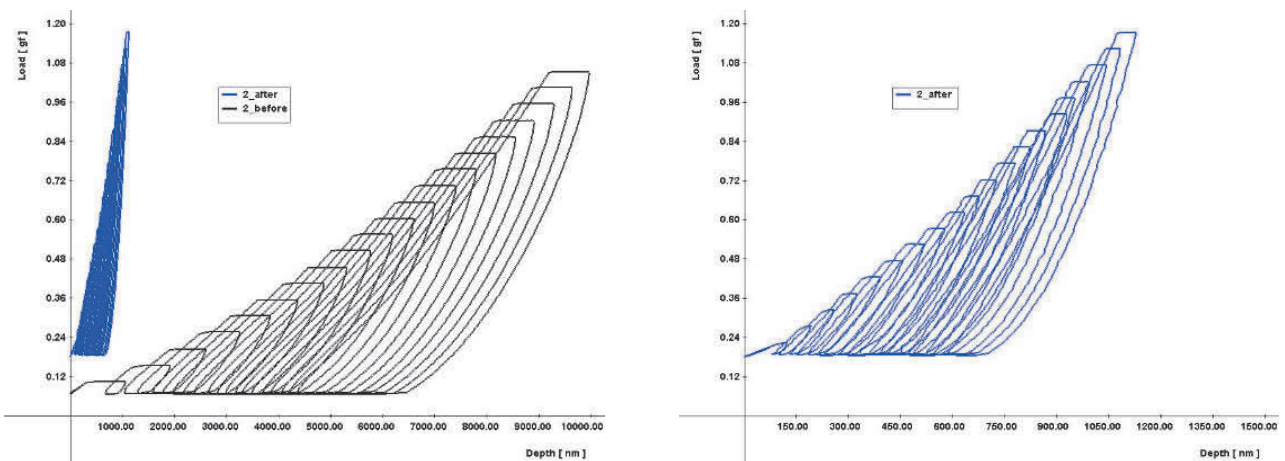
was realised with value of maximal load from point of view to get information about changing mechanical behaviour in the large depth under surface (**Figure 10 - Figure 13**).



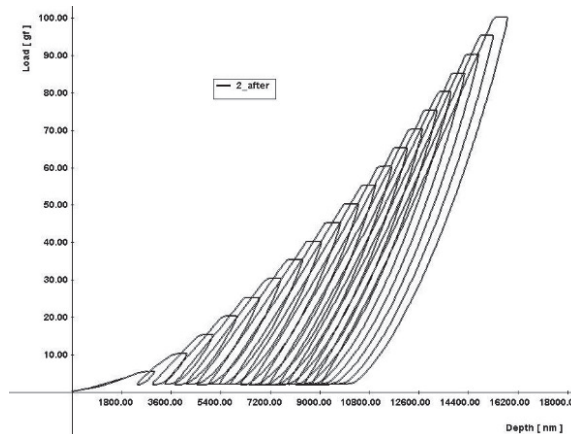
**Figure 10** Cyclic indentation curve measured on polymer material - sample 1 after ageing



**Figure 11** Cyclic indentation curve measured on polymer material - sample 1 after ageing



**Figure 12** Cyclic indentation curve measured on polymer material - sample 2 after ageing



**Figure 13** Cyclic indentation curve measured on polymer material - sample 2 after ageing

## 9. CONCLUSION

The optimisation of measurement of mechanical properties and behaviour of polymer materials from point of view its large elastic deformation was realised by nanoindentation in mode for measurement indentation curves during all indentation process loading to maximal load, time delay in maximal load and unloading. Here is possible to analyse changing rate of elastic and plastic deformation before and after ageing process of degradation. The much more information is given by measurement not only simple indentation curves but by using mode for measurement by cyclic nanoindentation method with step by step increasing maximal load in each cycle of measurement. There was optimised measurement by x-ray fluorescent method for possibility to measure polymer materials.

The x-ray spectrums measured before and after ageing process give possibility evaluate changing of chemical composition created by ageing process of temperature degradation. The results show, what the polymer materials 2 change rapidly chemical composition on the surface during ageing process. The polymer materials 1 not show marked changing in composition measured on surface before and after ageing process.

The nanoindentation curves single and cyclic measured before and after ageing process give possibility evaluate changing of mechanical properties and behaviour more sensitive which are created by ageing process of degradation. The nanoindentation has presumption for control process of degradation for evaluation changing mechanical properties and behaviour and prediction of changing after evaluation in different steps of degradation process. The polymer materials 2 has marked changing of behaviour during indentation process. The elastic deformation is decreased and hardness of polymer materials on surface increase. The polymer materials 1 not change properties significantly.

## ACKNOWLEDGEMENTS

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