Abstract

The subject of this paper is to describe the practical use of micro-indentation methods, i.e. measurement of material characteristics by low loads indentation tests, for the preparation a standardized procedure of the conditional monitoring of selected system components of the safety-related systems to ensure their long-term exploitation.

The observation results presented here show, that standardized conservative methods of measurement of material properties, such as monitoring of changes of the relative elongation at break or OIT (Oxidation-Induction Time), which defines the aging rate, and can be supplemented or fully replaced by methods of micro-indentation measurements. These methods can be effectively determine parameters of the dynamic hardness significantly more accurately and consequently identify local indicators of aging with significantly higher populations of measurements.

Herewith, there is guaranteed realistically determine values of the indicators of aging and eliminate uncertainty measurement and considerably precise a prediction of service lifetime. The indisputable advantage is also non-destructive nature of the tests and demands to the size of samples.

Keywords: surface layers, mechanical properties, nanoindentation, x-ray fluorescence, ageing of polymers

1. INTRODUCTION

With regard to the requirements for long-term operation nuclear power plants that follow from legislation, it is necessary for the important equipment to demonstrate their adequacy for all design basis events and for the whole period of their service. This is one of the basic principles of nuclear safety of the defense against common cause failures. This concept is called the equipment qualification and is characterized by the systematic documentation of equipment performance capability and their components that are, so called, classified items of the plant safety-related systems. The equipment must be qualified for all postulated conditions arising from long-term operation, as well as required assessment for conditions under severe accident regime. Such conditions include effects of degradation due environmental conditions (temperature, humidity, radiation, etc.) inflict equipment parts made of non-metallic materials. As a typical example of the relevant representatives of such elements are cable systems.

Newly published a series of standards IEC / IEEE 62582 reflects the requirement of the introduction of condition monitoring, which implies of updated standards IEC 60780 (IEEE Std 323-2004) and to efficiently extend the equipment qualified life, generally limited in active equipment of safety-related systems, exactly for non-metallic materials. Condition monitoring presents a feasible method of continuous monitoring of aging indicators and subsequent extrapolation and determination of qualified life, respectively its continuous extension.
We start to use the method for evaluation of mechanical properties and behaviour of surface of polymer materials on the base experiences with evaluation of systems thin film – substrate. The thin films are created by deposition process as controlled process of its creation by deposition parameters. The thin films are created by modification process, too with controlled 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 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 [1] and solution of goals to optimize analytical methods for possibility evaluation of systems thin film – substrate with very different resistivity, hardness, thickness, substrate [2] 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 with number 1. Only nanoindentation method of indentation measurement of hardness has possibility to evaluate materials with marked elastic deformation. The limit of maximal indentation depth on this equipment is about 20 microns. This maximal depth is sufficient for our results. The maximal load must be reduced from this point of view. The properties and behaviour can be different in different depth from the point of view natural ageing of polymer materials and from the 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 of this material systems (Fig. 1 and Fig. 2).

Fig. 1: Indentation curves measured on surface of polymer material of sample number 2 with comparable elastic and plastic deformation parts.
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 of surface layers of polymer materials 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 and behaviour by nanoindentation. 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 (Fig. 3 and Fig. 4). This setting increase information from large interval of depth under surface of polymer material systems and increase sensitivity in small depth under surface of polymer materials, too.
4. EVALUATION OF CHEMICAL COMPOSITION OF POLYMER MATERIALS

Surface sensitive method for evaluation of changing chemical properties is x-ray fluorescent method. This method gives the possibility to evaluate changing of 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 (Fig. 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. ANALYSIS OF POLYMER MATERIALS AFTER AGEING PROCESS

The changing of chemical composition after temperature 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 for evaluation of polymer materials. 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 of polymer materials and basic state of polymer material under degraded layers. The analysis of changing of x -ray fluorescent spectrum gives
acceptable possibility to evaluate changing after temperature 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 Fig. 6.

![Fig. 6: X-ray fluorescent spectrums on sample 1 and sample 2 of polymer material before and after ageing process of temperature stress.](image)

### 6. Changing of Mechanical Behaviour

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 temperature ageing process for possibilities comparing mechanical behaviour before and after temperature 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 (Fig. 7 – Fig. 9). It is about 20 microns to the depth under surface of polymer materials, but measurement is influenced from higher depth.

![Fig. 7: Indentation curves measured on polymer material – sample 1 after ageing](image)
The samples of polymer materials were measured in the other step by cyclic nanoindentation after temperature 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 of polymer materials (Fig. 10 – Fig. 13).
7. 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 of rate of elastic and plastic deformation before and after temperature
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 these kind of polymer materials.

The x-ray spectrums measured before and after temperature 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 of polymer material during temperature ageing process. The polymer materials 1 not show marked changing in composition measured on surface of polymer material before and after temperature 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 temperature ageing process of degradation. The nanoindentation has presumption for control process of degradation for evaluation of changing of mechanical properties and behaviour and prediction of changing after evaluation in different steps of degradation process. The polymer materials – sample 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 – sample 1 not change properties significantly.

LITERATURE
