NANOINDENTATION METHODS OF EVALUATION OF MECHANICAL PROPERTIES AND BEHAVIOUR AND COMPLEX APPLICATION FOR EVALUATION OF TECHNOLOGY AND DEGRADATION

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Abstract

Nanoindentation methods used for evaluation surface layers are static indentation and scratch indentation too. Static indentation methods are used namely for evaluation hardness characteristics and for evaluation elastic deformation and plastic deformation of surface layers too based on measurement indentation curves - dependence depth indentation and normal force during all indentation process during loading and unloading. Cyclic and step by step indentation modes give possibilities to evaluate much more information about resistivity of surface layers to the indentation stress. Scratch indentation methods are used for evaluation namely adhesive cohesive properties and behaviour of surface layers with very small thickness or/and thin films with very low resistivity of thin films and low hardness. The surface layering is created by technology process of creation of thin films and surface treatment process or by initiation and evolution of degradation process and ageing process. The initiation of degradation and ageing are evaluated in the starting stadium and in the next with step by step sensitive analysis of changing properties and behaviour. From this point of view static nanoindentation and scratch nanoindentation are very useful for analysis step by step changing of mechanical properties and behaviour with high sensitivity and depth resolution too. The paper presented utilization of nanoindentation static and scratch nanoindentation too for very different application in surface treatment but for evaluation of degradation and ageing process too.

Keywords: Nanoindentation, scratch nanoindentation, radiation ageing, temperature ageing, degradation of material, thin films, surface treatment

1. NANOINDENTATION ON SURFACE OF SYSTEM THIN FILM - GLASS SUBSTRATE

There was tested possibilities for analysis of properties and behaviour of thin films created by method Sol Gel [1] on glass substrate. Nanoindentation measurement was realised by setting mode 2 - indentation curve - loading, time delay 10 s and unloading. Measurement was provided by two different maximal load 25 g (**Figure 1**) and 5 g (**Figure 2**). Maximal load 25 g is for analysis modification of surface of glass by deposition process and 5 g is for evaluation differences between thin films on all systems thin film - glass substrate, because substrate is the same but thin films are different.

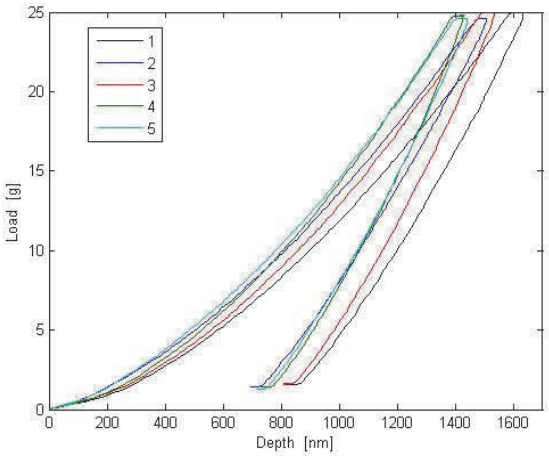

Figure 1 Indentation curves from measurement by maximal load 25 g

Indentation curves with maximal load 25 g (**Figure 1**) show the highest hardening of surface on systems 4 th and 5 th. The curves on systems 1 st show the lowest hardening of surface. Optimal hardness and toughness is on 2 nd system thin film - glass substrate.

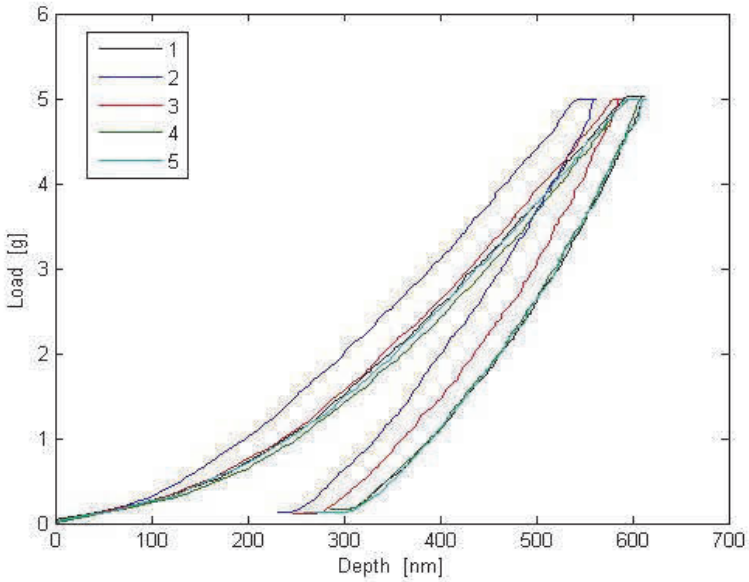

Figure 2 Indentation curves from measurement by maximal load 5 g

Measurement on **Figure 2** represent results from indentation by maximal load 5 g. The highest hardness is on 2 nd systems. The other systems are similar. The elasticity of thin films is higher then only surface of glass before deposition.

2. NANOINDENTATION ON SURFACES OF HARD THIN FILMS ON CUTTING TOOLS

The systems with different hard thin films created by different technology proces PVD [2] and PA CVD [3] with small resistivity was evaluated by nanoindentation. Measurement was realised by maximal normal load 200 g (**Figure 3**), 25 g (**Figure 4**) and 2 g (**Figure 5**) for evaluation modification of surface of substrate under thin films by deposition process and hardening of surface of substrate by thin films (200 g), evaluation properties and behaviour on interface between thin films and substrate (25 g), evaluation properties and behaviour of thin films (2 g).

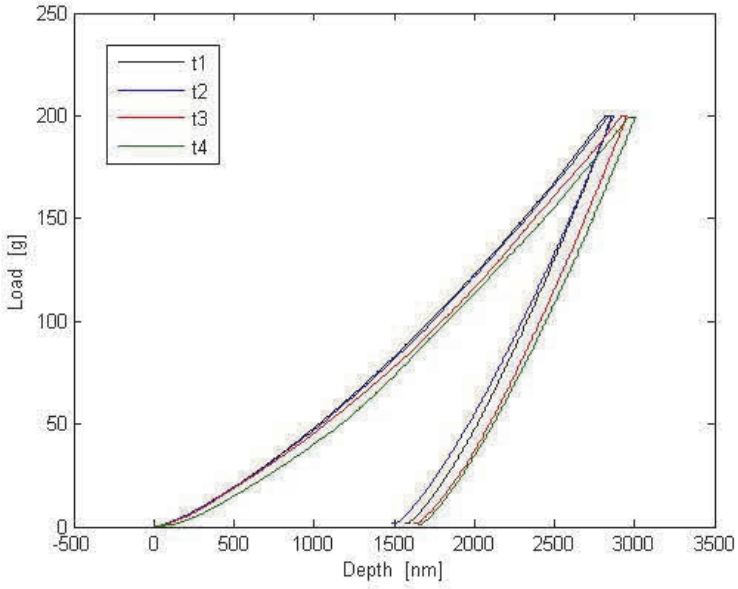

Figure 3 Indentation curves with maximal load 200 g

The indentation curves measured by mode 2 - loading, time delay 10 s, unloading with constant rate of loading show the highest hardening of surface is on systems thin films - substrate with thin films deposited by arc evaporation.

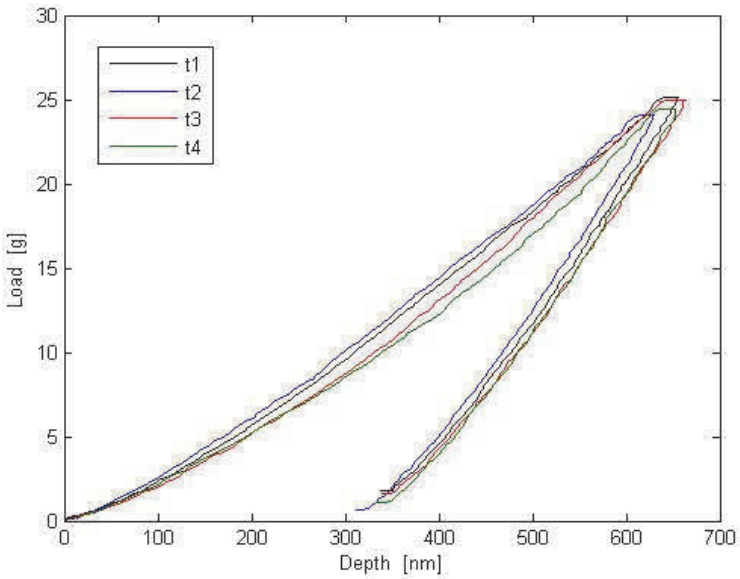

Figure 4 Indentation curves with maximal load 25 g

Indentation curves on **Figure 4** measured by 25 g maximal load show the highest hardening of surface on systems with thin film from arc evaporation too. From this measurement is possible to view, what thin films has higher elasticity then substrate on all systems thin film - substrate.

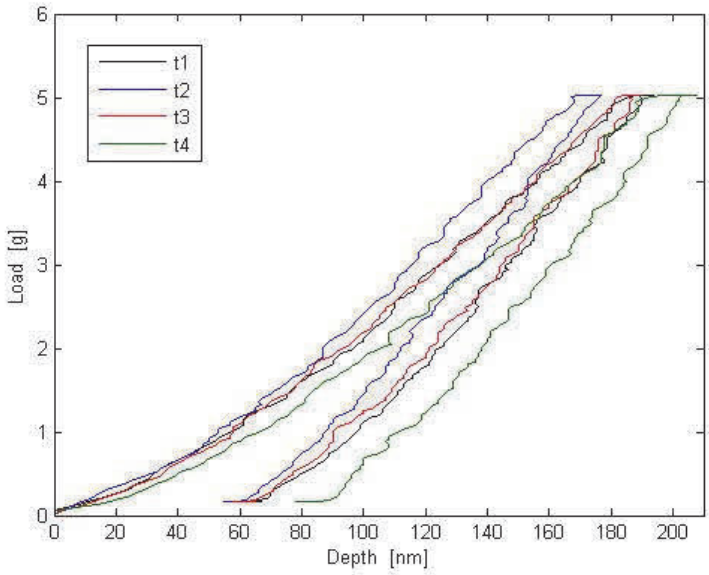

Figure 5 Indentation curves with maximal load 2 g

Indentation curves measured by maximal load 2 g (**Figure 5**) give evaluation namely properties and behaviour thin films. The highest hardness has thin films deposited by arc evaporation. Similar hardness has thin film from electron beem evaporation. The elasticity of thin films is higher then substrate.

3. NANOINDENTATION ON SURFACES AFTER TEMPERATURE DEGRADATION PROCESS

Indentation curves during loading and unloading with using different normal load 5 g (**Figure 7**), 25 g and 200 g (**Figure 6**) bring information about changing of mechanical properties and behaviour in different depth of thin film system. Changing of indentation curves with maximal load 200 g start at temperature loading 600 C. The indentation curves has lower angle and increase depth of indentation. From this temperature the hardness of substrate decrease because the temperature of treatment this HSS was crossed. The decreasing of hardness increase more rapidly, increase ratio plastic deformation towards elastic deformation. Indentation curves with lower load has similar changing of curves, only hardening of surface is better evaluated and changing is possible to evaluate from temperature 650 C. Similar changing is for loading 5 g too. Surface brittle failures is possible to evaluate from 600 C temperature loading.

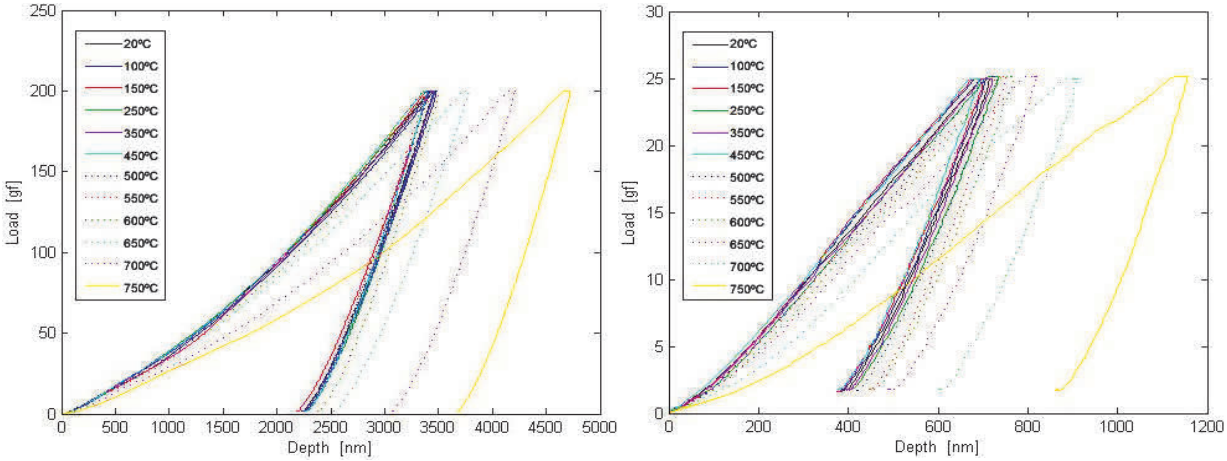

Figure 6 Comparison of indentation curves with maximal load 200g and 25g on sample after temperature loading from 20 C to 750 C

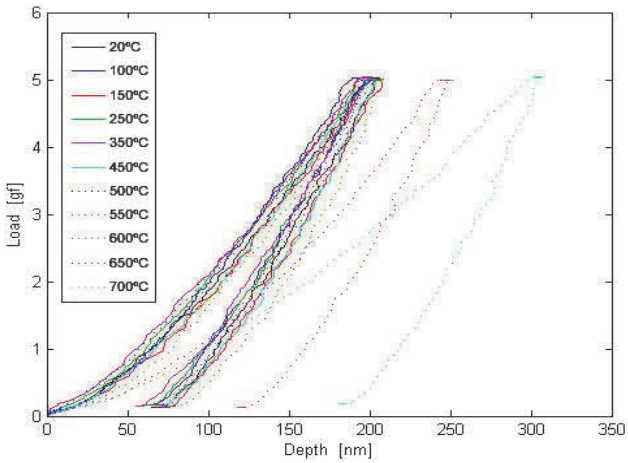

Figure 7 Comparison of indentation curves with maximal load 5g on sample after temperature loading from 20 C to 750 C

4. NANOINDENTATION ON SURFACES OF POLYMER MATERIALS AFTER DIFFERENT AGEING PROCESSES

Indentation curves with maximal load 1000 mN was measured by Berkovich indentor on surfaces of samples after action sun radiation 1000 hours and 2000 hours. The results show (**Figure 8**) what after radiation the

large hardening was created after 1000 hours action of sun radiation. Other 1000 hours influence of sun radiation create smaller hardening.

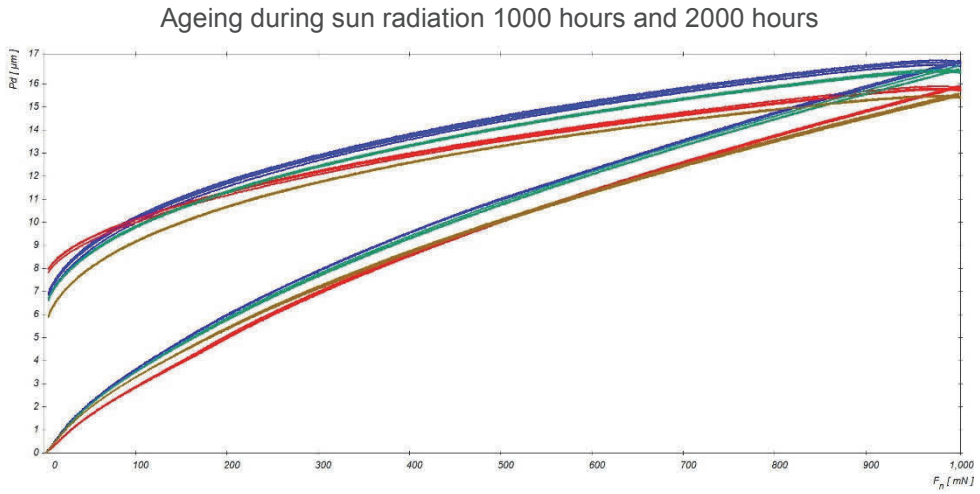

Figure 8 Indentation curves measured before and after action sun radiation on surface of sample of polymer material

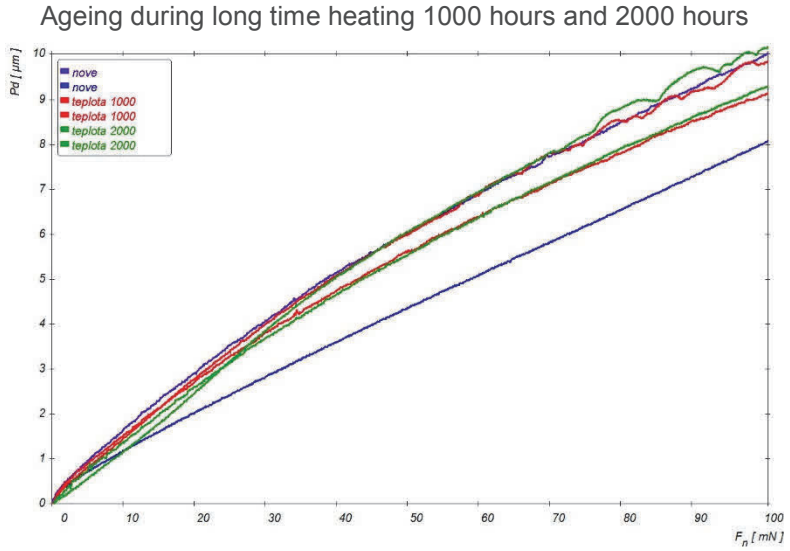

Figure 9 Indentation curves measured before and after action higher temperature on surface of sample of polymer material

Scratch indentation measurement was realised with increasing normal force with constant rate of loading on surfaces of polymer material after 1000 and 2000 action higher temperature 60 st. C. The results show (**Figure 9**) what the hardness of surface of polymer material increase after 1000 hours action of temperature and after 2000 hours action temperature, too, on both sides of measured samples.

5. CONCLUSION 1

On the start the results give information what is not possible to evaluate these specific systems by our method for analysis. The problem is in very small thickness, small resistivity thin films, small resistivity surface of glass substrate - brittle fracturing, transparency of thin films and here are not differences in color in contrast with glass. The first results show what it is very important refinement of measurement by changing parameters of measurement - changing rate of samples under indentor, maximal load is important decrease and geometry

of diamond Rockwell indentor was changed. There was evaluated adhesive cohesive behaviour, hardness and elastic plastic deformation by indentation and thickness by calotest and x-ray fluorescent method. The result give us differences. The highest resistivity is on systems 2 nd, then on systems 1 st and 5 th and the lowest resistivity is on systems 3 rd and 4 th. Thickness the highest on systems 3 rd and here is problem with resistivity. Optimal thickness is about 400 nm.

6. CONCLUSION 2

There was evaluated different systems of thin film - substrate with the same substrate from HSS and the same kind of thin films but prepared by different technology process of deposition - PVD - arc evaporation, elektron beem evaporation and magnetron sputtering. The best resistence to scratch indentation test has system with thin films prepared by elektron beem evaporation method deposition and arc evaporation. The lowest resistence has systems with thin films deposited by magnetron sputtering method of deposition. The highest hardening surface of systems is on systems with thin films prepared by arc evaporation and then by elektron beem evaporation.

7. CONCLUSION 3

The results in this paper show changing of mechanical properties and behaviour after temperature stress with different temperature. Here is possible to view step by step changing during different temperature. At the first is decreasing small cohesive failures because decrease stress in thin film created by deposition process. In other step slowly change TiN thin film from surface to the oxide film. It is possible to view from calotest measurement. Changing TiN to oxide film is resulted in increasing surface hardness but increasing brittle fracture in surface layer. From 600 C temperature hardness of substrate under thin film decrease and plastic deformation around indents and scratches increase and influence on expansion brittle fractures around indents.

8. CONCLUSION 4

The reasult show what changing of properties after sun radiation and action higher temperature in the long time on surface of sample of selected polymer material is measurable by sensitive nanoindentation measurement and scratch nanoindentation measurement, too.

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