

MECHANICAL PROPERTIES AND MICROSTRUCTURES OF SYSTEMS THIN FILM - SUBSTRATE

OLGA BLÁHOVÁ, IVO ŠTĚPÁNEK, ANTONÍN KŘÍŽ,
JAROSLAVA BRŮNOVÁ, PETR HORA

This paper is devoted to study of mechanical properties and microstructures of the systems thin film - substrate. The nanohardness and adhesive - cohesive behaviour are investigated by nanoindenter and scratch - tester. The microstructures are studied by scanning electron microscopy with microprobe.

1. Introduction

There is a considerable need to characterise, understand and optimise the properties of coated systems for particular applications. This is particularly critical for systems with thin (10 μm or less), hard coatings where property enhancements are often out of all proportion to the volume of coating material. Our department prepares systems thin film - substrate by reactive cathodic arc evaporation in vacuum and is concentrated on complex study of their properties. This paper includes the measurements of the systems thin film TiN-high speed steel (HSS) substrate deposited with different negative bias voltage.

1. Nanohardness of coating - substrate systems

Conventional techniques for measurement of microhardness of thin films yield results that are difficult to evaluate, because it is difficult to separate the influence of the substrate. To obtain intrinsic hardness of the thin coatings ($\sim\mu\text{m}$), it is required to use very low loads. This is an area ideally suited to application ultralow load indentation systems (nanoindenters). These instruments provide continuous records of load and displacement as an indenter is pushed into a surface (Fig. 1a). Exploitation of these systems enables providing the more complete assessment of elastic, plastic and fracture properties of coated systems [1,2].

The measurements are realized by nanoindenter SHIMADZU DUH-202 with the Vickers indenter. Load range of this instrument is 0.1 mN – 1961 mN (0.01 gf – 200 gf). Depth of indent as small as 0.2 nm can be measured.

Ing. Olga Bláhová, RNDr. Ivo Štěpánek, Ing. Antonín Kříž, Jaroslava Brůnová, Department of Materials Science and Metallurgy, University of West Bohemia, Veveřská 11, 301 14 Plzeň, Czech Republic,

Ing. Petr Hora, CSc., Institute of Thermomechanics, Academy of Science of the Czech Republic, Veveřská 11, 301 14 Plzeň, Czech Republic.

Fig. 1b shows measured values of the nanohardness $DHV1 = L / A_{\max} = 0,0378 L / h_{\max}^2$ (L - load [gf], A_{\max} - area of the indent under load [μm^2], h_{\max} - maximum depth [μm]) of the systems thin film TiN - HSS substrate.

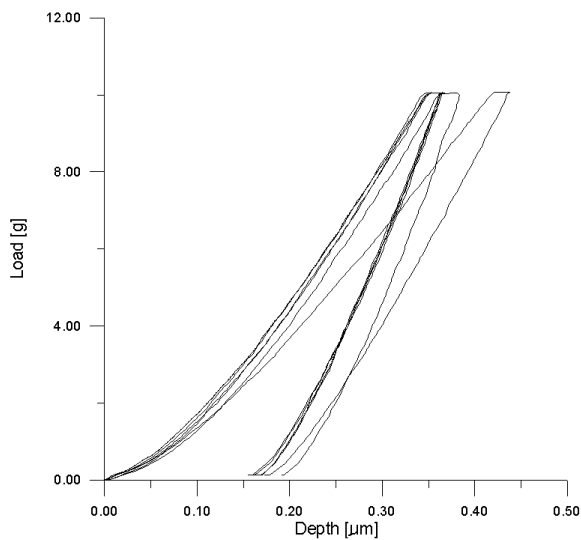


Fig. 1a. Indentation curves of the systems TiN - HSS substrate prepared with different bias voltage on the substrate.

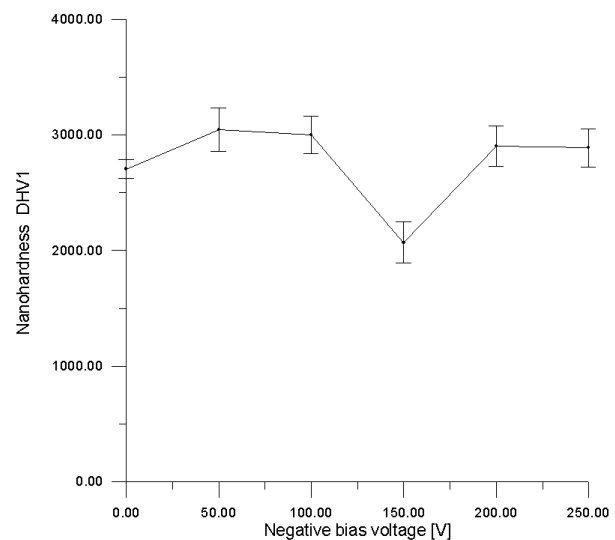


Fig. 1b. Nanohardness DHV1 thin films versus negative bias voltage on the substrate.

2. Adhesive - cohesive behaviour of coating - substrate systems

The most commonly used methods for the testing of the adhesive-cohesive behaviour of hard coating - substrate systems are the indentation methods. They are based on measuring stresses necessary for breaking the bond between the coating and the substrate during static pressing of the indenter or during a longitudinal scratch. Different failure modes (cohesive and adhesive) occur as a result of the compressive stress field. They are observed by optical and/or scanning electron microscopy and by the electron microanalyses. The coating is separated at a critical load, which serves as a quantity for the comparison of the adhesion.

The scratch test method is the most frequently used method for testing of the adhesive-cohesive behaviour of hard coatings, however, its results should be used cautiously, because the measurement is influenced of different parameters (properties of system substrate-coating, wear of indenter, machine factors,...) [3,4].

The testing is provided by the scratch-tester CSEM REVETEST connected with computer. The scratch-tester outputs are normal and tangential forces acting on a test diamond indenter, coefficient of friction and acoustic

emission signal sensed on a indenter holder. These acoustic emission signal characteristics differ for each of thin film microstructure and composition.

The images of the scratches are digitalized by image processing system consisting of optical microscope NIKON OPTIPHOT 100S, RGB camera, frame grabber card in PC and software Image-Pro Plus 1.3. The images are analysed by image processing software Image-Pro Plus 1.3 and Ip De luxe.

The computational programs for treatment results was created. The program was written in MATLAB. The main program window consists of the scratch image, the tangential force plot (F_t), the friction coefficient (μ) and the acoustic emission (AE) signal plot (Fig.2). The program provides the coincident movement of the scratch image as well as the plots. Therefore, it is easy to find the places, that correlated with the acoustic emission peaks.

Fig.3 shows the mechanisms of the failures on the part of the scratches where load affects in the range from 40 N to 50 N.

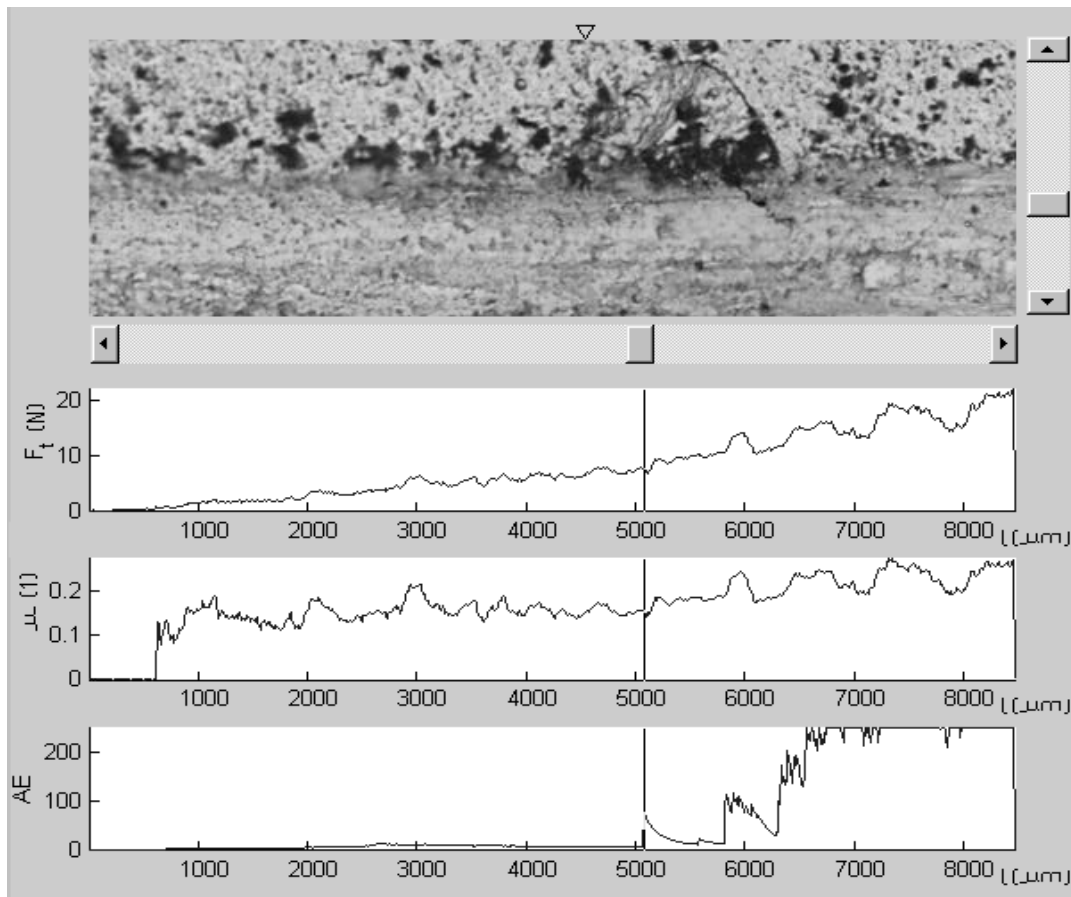


Fig. 2. The main window of the program for treatment of results of the scratch test.

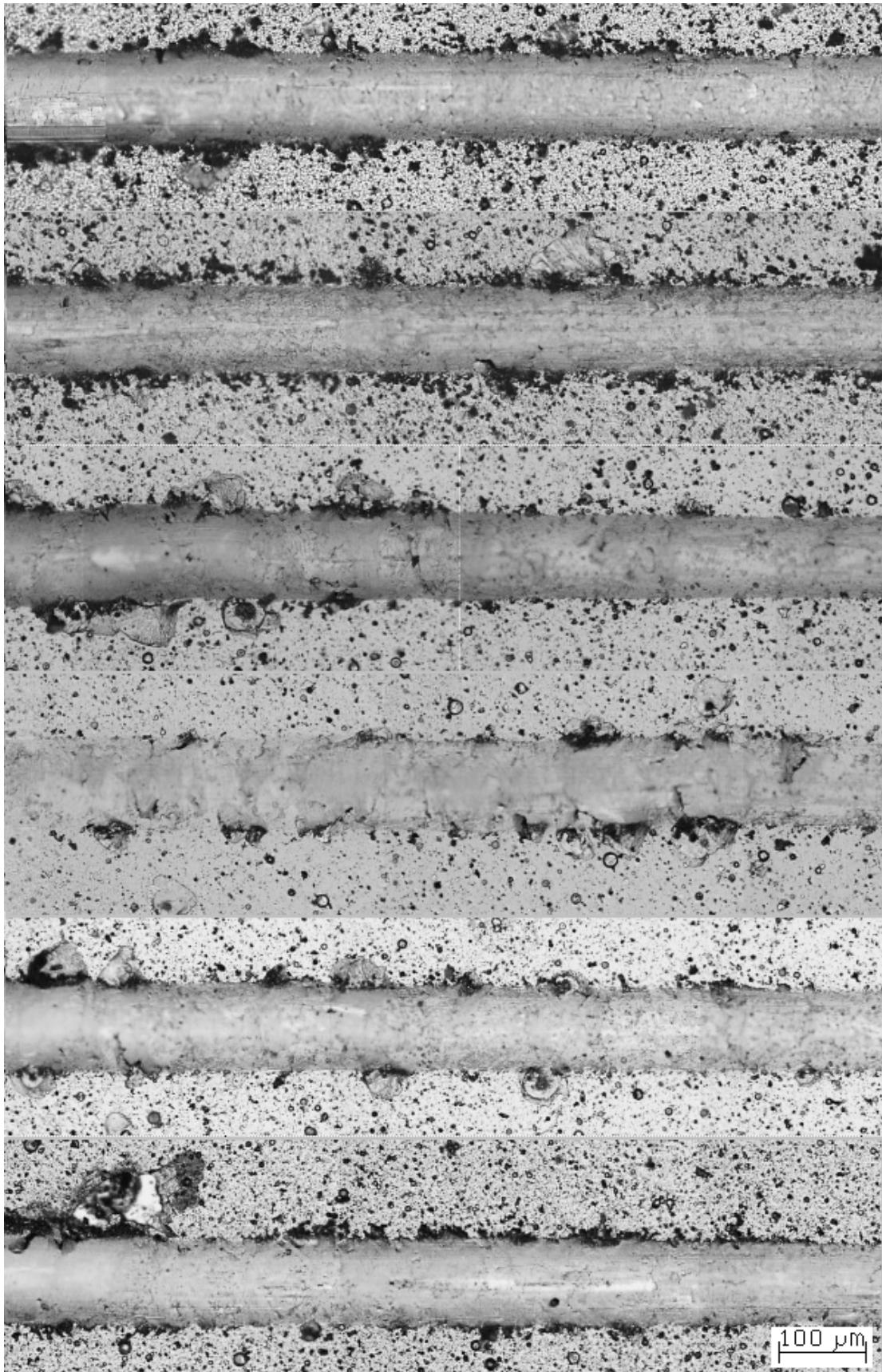


Fig. 3. The part of the scratches on the thin films deposited with different bias voltage from -250 V to 0 V on the substrates.

3. Microstructure

The microstructures are studied by scanning electron microscopy. Fig. 4 shows fracture cross-section of systems thin film TiN - HSS substrate deposited with negative bias voltage on the substrate. Microstructures are very dense.

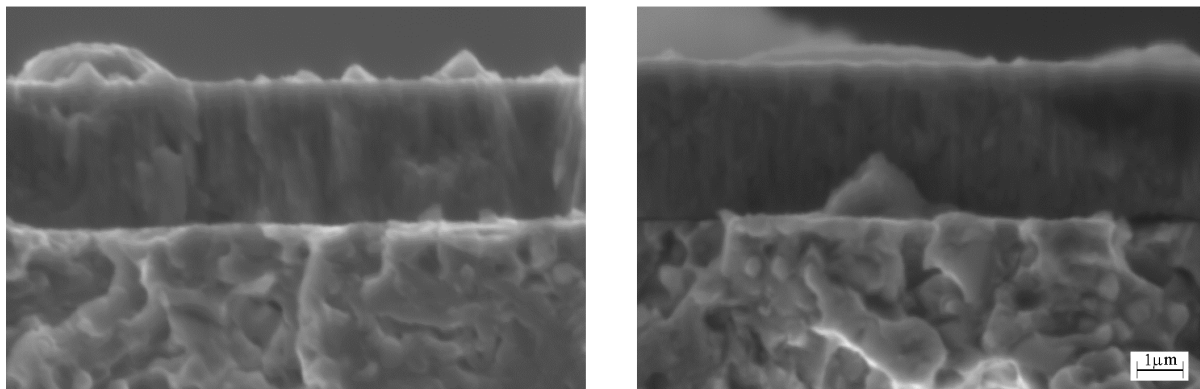


Fig. 4. Fracture cross-section of systems thin film TiN - HSS substrate deposited with negative bias voltage 0 V and 250 V.

4. Conclusion

This paper includes evaluation of systems thin film TiN - HSS substrate deposited with different negative bias voltage on the substrate. The measurements are provided by created method for complex analysis of mechanical properties and behaviour of systems thin film - substrate [5]. This paper includes measurement by nanoindenter and scratch-tester. The results show the high values of nanohardness and good adhesive - cohesive of systems thin film TiN - HSS substrate. Minimum value of the nanohardness on Fig. 1b was probably brought about by higher value of the internal stress. Presented methods of evaluation of mechanical properties and behaviour of these systems above mentioned contribute to complex optimisation of the deposition processes.

Financial support of these investigation by Grant n. 106/96/0127 GA ČR.

LITERATURE

- [1] DOERNER, M.F. - NIX, W.D.: Journal of Materials Research, 1, 1986, p. 601
- [2] PAGE, T.F. - HAINSWORTH, S.V.: Surface and Coating Technology, 61, 1993, p. 201
- [3] PERRY, A.J.: Thin Solid Films, 78, 1981, p. 77
- [4] STEINMANN, P.A. et al.: Thin Solid Films, 154, 1987, p. 333
- [5] BLÁHOVÁ, O. - ŠTĚPÁNEK, I. - ŠIMŮNKOVÁ, Š. - VÁVROVÁ, B.: In: International symposium Innovation '97 Praha, p. 4