

A STUDY OF A FAILURE OF THIN PVD COATINGS ON PM TOOL STEEL

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Abstract

The behaviour and failure of the system thin layer – substrate after laboratory “Pin-on-disc” test was investigated. The wear was studied by optical emission spectroscopy – GDOES and related to the chemical composition of the coated system. The basic experimental material (substrate) was steel produced by powder metallurgy and used for making tools intended for cold machining. An AlTiN thin layer was deposited onto the substrate surface by reactive arc evaporation method. Tribological tests and depth concentration profile allowed us to obtain a specific picture of the behaviour of the thin layer – substrate system. Evaluation of tribological properties of the thin layer – substrate system is difficult and one must consider a number of additional factors – roughness, hardness, deposition parameters, type of coating and subsequent processing of tribological data, including mechanical and physical properties of tribological pairs, temperature of specimen surface, ambient environment, time of wear, magnitude of load, etc. These data were supplemented with EDX analysis which enabled us to obtain qualitative and quantitative description and thus provide more complex information about processes taking place at the contact area of tribological pairs.

Keywords: PVD coating, thin layer, failure, tribology

INTRODUCTION

The tools and parts that are stressed during their use are subject to wear, changes in temperature and aggressive environment. Consequently the composition and structure of material surface do not correspond to the state of the internal parts of this material. Therefore, the modification of a surface layer of the respective part can intervene with its response to ambient environment and affect essentially the use of such a component [1,2]. Recently we could observe considerable development in the field of new physical technologies enabling the production of thin surface layers and thus improving considerably the utility properties of tools and components [3]. Results of the research of thin layers deposition are reflected in machine industry, particularly in the field of cutting and mechanical working tools and greatly stressed machine parts. The present literature dealing with the issues of thin layers and their deposition on tools or parts fails to provide information necessary for complex assessment of the system thin layer – substrate, particularly in those cases in which the substrate was produced by powder metallurgy technology [4]. The current technological methods of surface treatment allow one to resolve the problems of short-term service life and reliability of tools and mechanically stressed

parts. The system thin layer – surface has been investigated by testing at normal temperatures, but the tools are heated to high temperatures during the working process. Surface treatment and combination of a number of technological processes produce a composite zone on the material surface and its properties are affected by mutual diffusion of individual components into the system. Deposition of a suitable coating onto the surface of parts can decrease significantly the coefficient of friction or heat transfer. PVD technology enables us to obtain coatings noted for their high hardness, wear resistance and chemical stability [5]. With tribologically well designed contact pairs the stable state persists for a very long time, which contributes to long life-time. However, even at low wear, the coating becomes thinner as it gradually thins away until such a state is reached when the coat can no longer protect the substrate against wear [6]. Tribology, as a study of the principles of friction and wear, is suitable for evaluation of the system thin layer - substrate. One must, however, take into consideration that the natural role of tribology does not involve epochal technical or technological changes, but is concerned with development of existing techniques and technologies. Therefore it holds that any progress in technologies must be associated with progressive development in tribology [7,8,9].

MATERIAL AND METHODS

The experimental programme was run on commercially produced PM steel, marked Vanadis 4 Super Clean, heat treated and coated with an AlTiN layer produced by PVD technology. It is a Cr-Mo-V alloyed steel and the designation „super clean“ denotes the highest quality grade of presently produced high-alloyed PM steels, which offers remarkably good combination of wear resistance and toughness, not attainable by conventional technology. The thin AlTiN surface layer was deposited onto the substrate surface using PVD method of reactive arc evaporation – Linear-Arc procedure. The method is rapid, effective and attractive for deposition of thin functional films with good mechanical properties. Important is also the preparation of the substrate surface before deposition of the coatings re cleanliness, microgeometry and hardness. The appropriate management of the deposition process and uniform thickness and suitable quality of the coating are parameters which are reflected eventually in mechanical properties of the entire system. Complex results can be obtained using common laboratory methods (SEM, EDX, GDOES), but also by tribological „Pin-on-disc“ test. This test represents one of the most frequent methods for determination of tribological properties of the thin layer-substrate system. Wear and friction are the products of the same tribological process which occurs between two moving surfaces. However, the relationship between friction and wear has not yet been investigated fully. It is a common belief that low friction corresponds to low wear and high friction to high wear. However, this rule does not hold generally. There are in fact several examples of completely opposite behaviour [10]. Friction between two surfaces is accompanied by the process of wear, whether abrasive or adhesive. Specially prepared specimens were subjected to a „Pin-on-disc“ test on a tribometer, Fig.1. The principle of a tribotest is that a non-rotating pin - ball produces a track on a surface of the specimen at a certain selected distance r from the specimen centre and the magnitude and extent of wear, margins, depth and eventually also wear of the ball are evaluated at the defined load F .

The tribological properties were supplemented with another important characteristic affecting property of the coated system, namely changes in chemical composition of the material depending on depth. GDOES (*Glow Discharge Optical Emission Spectroscopy*) analysis enables us to obtain a graphical record of depth concentration profiles of respective layers in weight or atom per cent. An optical spectrometer uses an excitation source (the so-called Grimm lamp) in the process of glow

discharge for excitation of atoms in the specimen. This way radiation is produced of wavelength typical of the respective element [11].

RESULTS

In our case the tribological tests focused mostly on determination of the coefficient of friction and its changes in the course of the experiment and provided information on adhesion - cohesion behaviour of the respective material at room temperature. More realistic results can be obtained at elevated temperatures simulating the working process of respective tools [11].

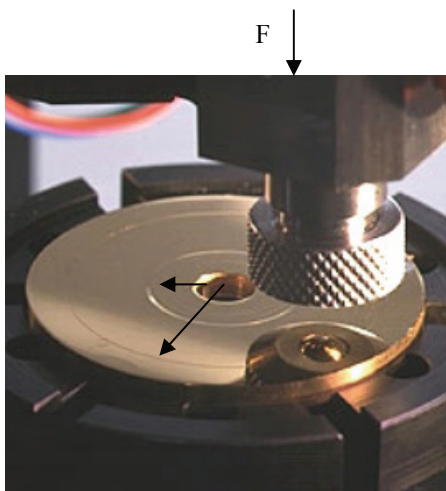


Fig.1. „Pin-on-disc“ test.

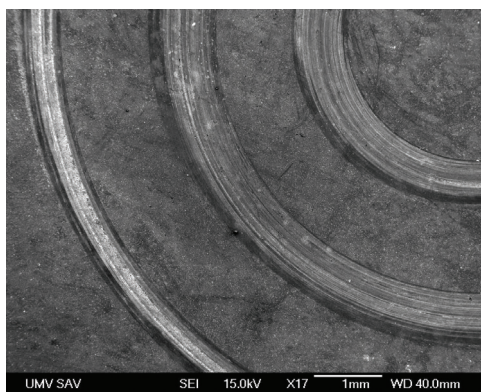


Fig.2. Tribological wear with a track radius $r = 2.4$ and 6 mm.

The character of wear after the „pin-on-disc“ test was evaluated by electron microscopy and EDX analysis. Figure 2 shows the tribological wear tracks of a rigid pin - ball with a pathway radius $r = 2, 4$ and 6 mm obtained by electron microscopy. The friction coefficient on test pathways at determined process conditions (Table 1) was sufficiently high $\mu_{str} = 0.829 - 0.862$. The satisfactory coefficient of friction can be ascribed to hardness of the surface which reached 2443.5 ± 310 HV0.05 and the roughness 0.09 ± 0.02 μm . The graphical recording of the course of coefficient of friction is presented in Fig.3, in which the oscillations at approximately 10000 cycles indicate impairment of the layer. Figure 4 shows a more detailed picture of the structure of tribological wear track in individual locations. The wear behaviour of the coat was standard, the coat was thinning away and gradually exposed the substrate. Results of EDX analysis in selected locations are presented in Table 2 in which the proportion of iron indicates a initial exposure of the substrate.

Tab.1 Tribological test parameters [12].

Specimen	r [mm]	v [cm.s ⁻¹]	F [N]	μ_{str}
Va4 -AlTiN	2	5	5	0.829 ± 0.039
	4	10		0.853 ± 0.065
	6	15		0.862 ± 0.055

* Pin testing pin - ball ($\varnothing 6$ mm), track radius r, rate v, load F, $\mu_{str} = \mu_{poč} - \mu_{max}$.

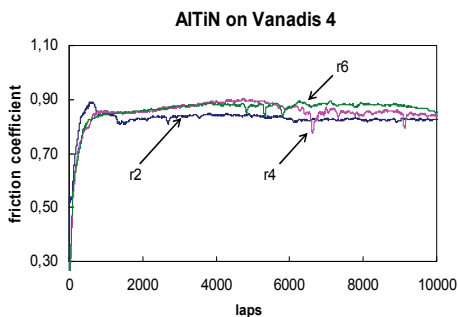


Fig.3. The graphical recording of the friction coefficient.

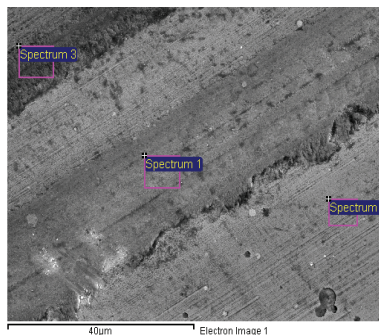


Fig.4. Structure of tribological wear track in individual locations.

Tab.2. EDX analysis in selected locations of the track from Fig.4 (% by weight).

Spectrum	In stats.	C	N	O	Al	Ti	Cr	Fe
S_1	Yes	-	23.97	35.18	5.46	30.06	-	5.32
S_2	Yes	-	49.09	-	27.41	23.50	-	-
S_3	Yes	6.43	18.36	35.99	15.16	19.75	0.54	3.76

The analyses performed indicated suitability of application of a thin layer of AlTiN to the respective PM steel substrate and the tribological test confirmed suitable adhesive-cohesive properties of the investigated system. It is obvious that the microstructure of the thin layer and parameters of its deposition affect the respective tribological properties. Adhesive failure is determined by two factors – porosity of microstructure and diffusion of heat at the sliding contact, but wear due to oxidation can predominate in these layers [6,11].

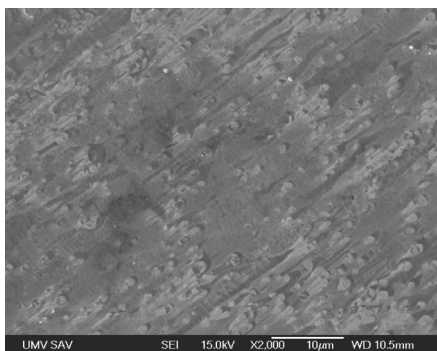


Fig.5. Abrasive wear results from the effect of hard particles.

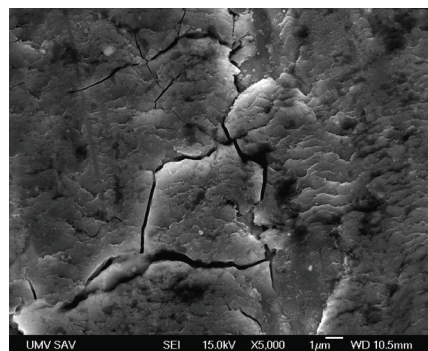


Fig.6. Stage of abrasive wear or chipping.

In the initial stage of wear, adhesion impairment occurs at micro junctions which develop in points of contact with surface unevenness. Abrasive wear results from the effect of hard particles or unevenness of the hard surface of one body coming into contact with the surface of another body, i.e. when two surfaces slide against each other, as it is

mentioned in [13], and this phenomenon is documented by Fig.5. The destruction regimen is responsible for development of cracks in the thin coat and subsequent formation of larger particles leading to another stage of abrasive wear or chipping, Fig.6. Additional wear results in corrugation of the surface or even to the exposure of the substrate at the track bottom, as is illustrated in Fig.7a. EDX analysis of such location is shown in Fig.7b, in which an increased proportion of iron and chromium were identified.

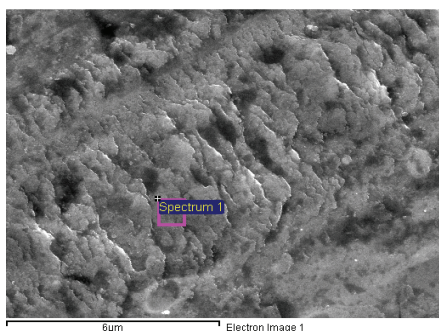


Fig.7a. Exposure of the substrate at the track bottom.

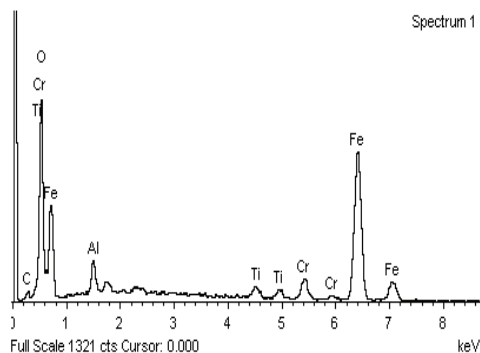


Fig.7b. EDX analysis of selected location.

GDOES analysis performed on the investigated material provided concentration profile related to the depth of dedusting, Fig.8. The relationship indicated a decrease of N content in the coating towards the interface thin layer - substrate. There was also a visible layer of Al and Ti with increasing proportion of Ti towards the interface. The gradual decrease in concentration of elements may be related to the presence of a very thin interlayer. Thickness of the layer reached 2.7 to 3 μm , but this value depends on varying rates of dedusting of individual elements. Method of Kalotest was realized for specification of layer thickness value that confirms the layers thickness of 2.7 - 3 μm . A disadvantage of the GDOES analysis is that it fails to provide information on phase composition of the layers. On the other hand, it provides exact information on chemical composition of layers and it appears advantageous in the case of routine control of the deposition process or involving changes in the process itself [6].

The results of GDOES analysis allow one to obtain better picture about the structure of the prepared systems thin layer – substrate and the method occupies an irreplaceable place in the study of chemical properties of new types of layers.

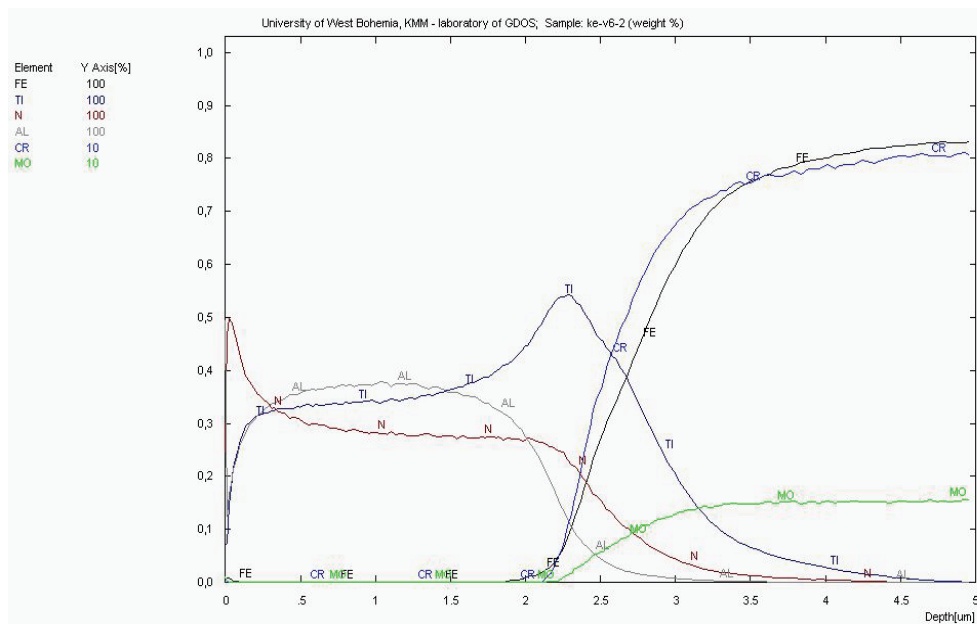


Fig.8. GDOES analysis of AlTiN layer – substrate system.

CONCLUSIONS

On the basis of this set of experiments the following results were obtained:

- The coefficient of friction of tested pathways at determined process conditions was relatively high $\mu_{str.} = 0.829 - 0.862$. This fact can be assigned to the surface hardness that reached 2443.5 ± 310 HV0.05 and to the surface roughness $Ra = 0.09 \pm 0.02$ μm.
- The concentration profile determined by GDOES analysis indicated a decrease of N content in the coating towards the interface of thin layer - substrate. Thickness of the layer reached 2.7 to 3 μm, but this value depends on varying rates of dedusting of individual elements. The results are not only data but also depth concentration profiles which allow a graphical presentation of the changes in volume up to the depth of 0.1 mm. All these values provide reliable information for identification of material and surface actions.
- Method of Kalotest was realized for specification of layer thickness value that confirms the layers' thickness of 2.7 - 3 μm.

The evaluation of tribological properties of the system thin layer – substrate is a difficult process and it is necessary to take into account many additional factors. It involves not only high quality preparation of the surface before deposition, its roughness and hardness, but also deposition parameters, type of the coating and subsequent processing of tribological data, including mechanical and physical properties of tribological pairs, temperature of specimen surface, ambient environment, number of cycles (cycle duration), magnitude of load, etc.

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