EVALUATION OF THIN PVD COATINGS BY ADHESIVE - COHESIVE TEST

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Received 10.08.2010
Accepted 20.11.2010

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Abstract
The paper deals with experimental evaluation of properties of PVD coatings. TiAlN, TiAlN/CrN and CrN coatings were deposited by ARC technology onto steel substrates, types S600, K190 Microclean and Vanadis 4 – Super Clean. The (TiAl)N/a-Si_3N_4 (thereinafter nACo) coating was deposited by advanced PVD procedure - LARC (LAteral Rotating ARC-Cathodes). Nanocomposite coatings (nACo) prepared by PVD technology based on low voltage arc and using two cylindrical central cathodes, which were made from Ti and AlSi. The surface of nanocomposite layer contained lower proportion of macro-particles compared to the Ti-Al -Cr-N layers which provided a guarantee of lower roughness and better quality of the respective layer. The coated systems were evaluated by a scratch test. Results of the test were documented by the graphical record of acoustic emission AE, friction coefficient and metallographic analysis on specimen surface produced by the test.

Keywords: PVD, ARC, LARC, nanocomposite coatings, scratch test

1 Introduction

A number of technologies and methods are available for application of thin coatings onto the surface of stressed machine parts. In applications involving high mechanical deformation stress, friction, wear, and similar, physical vapour deposition (PVD) has been used successfully. The process generates thin hard coatings exhibiting high hardness, good wear resistance and chemical - thermal stability and corrosion resistance [1-5]. The coated systems have been used extensively in many fields of industry, medicine and also in consumer sphere. Due to increasing demand of the market and high competitiveness in this area, new modifications of PVD techniques and new types of coatings have been developed and introduced into practice. ARC technology of application of thin coatings is one of the processes commonly used to apply thin hard PVD coatings [6,7,8]. It is based on evaporation of coating material by means of a low-voltage arc. The cathodic arc burns only at cathode spot, several tens of millimetres in diameter. With the current of approx. 100 A, the temperature at the cathode spot reaches approx. 10^4 °C which ensures evaporation of practically any material. Motion of the cathodic arc can be controlled by a magnetic field and thus controlled vaporization of material can be ensured. At the cathode, the place of emission of electrons, ions and macroparticles, the proportion of ions reaches approx. 80 % [9]. Increasing proportion of high-energy ions increases effectiveness of the deposition process. The energy of positive ions can be increased by adding negative bias voltage to the surface of conductive substrate. The products emitted from the cathode spot contain macroparticles, 0.1 to 100 μm in diameter, which are an unwanted product of deposition of thin layers [10]. They limit the use of thin coatings in the fields where high compactness and minimum surface roughness are required. The LARC coating technology (Lateral Rotating Arc-Cathodes) is used predominantly for depositing nanocomposite and nanostructural layers. With this technology two rotating electrodes, located closely next to each other, are used as a source of evaporated material. Magnetic field is produced by a combination of a permanent magnet and a magnetic coil. Rotation of electrodes in the coating machine ensures uniform vaporization of the material from entire surface of electrodes and thus it is possible to evaporate materials even in strong magnetic field. This procedure ensures high ionization of plasma and low number of macroparticles imbedded in the coatings. The so-called virtual shutter enables cleaning of electrodes before deposition process. The most important advantages of the LARC technology are derived from rotating electrodes and their closeness. Low microgeometry of coating surface with minimum number of macroparticles is achieved by rapid motion of the cathode spot due to strong magnetic field and rotating electrodes. Owing to optimum utilization of the working space, with cylindrical rotating electrodes located at the wall of the coating machine, one can deposit multi- or nano-layer coatings in one machine by only one process [11,12]. The increasing demands on quality of thin PVD coatings necessitate development of suitable methods for evaluation of their properties. Deposition of coating on material surface produces a coated system and evaluation of its characteristics must consider both adhesion behaviour of coatings and cohesive properties of the coated substrate.

2 Experimental materials

Evaluation of properties of the system thin coating – substrate was carried out on specimens prepared from S 600 steel, K190 Microclean (DIN-X 220 CrVMo 13 4) and Vanadis 4 Super Clean. Chemical composition of the steels is presented in **Table 1**. After thermal heat treatment to hardness 63 to 65 HRC the structure of steels was isotropic, fine-grained, martensitic – carbidic [13,14].

Table 1 Chemical composition of steel types

Steel	Chemical composition [wt. %]						
	C	Mn	Si	Cr	Mo	V	W
S600	0.88	0.35	0.22	4.12	4.97	1.77	6.50
K190	2.30	0.33	0.40	12.5	1.10	3.94	-
Vanadis 4	1.28	-	-	4.20	5.00	3.10	6.40

The steel surface was polished and cleaned and the method of reactive arc was used to deposit the following thin PVD coatings: CrN, TiAlN, TiAlN/CrN, and (TiAl)N/a-Si_3N_4 (nACo). The deposition process allowed us to prepare coated systems intended for determination of the investigated properties. Technological conditions of the process are summarised in **Table 2**.

Table 2 Technological parameters of the PVD process

Coating	Substrate	Technological parameters			
		Coating temperature(°C)	Current (A)	Voltage (V)	Pressure (MPa)
TiAlN	**S600**	430	AlTi/180	40	1.0
			Ti/210	160	
CrN		430	Cr/130	120	2.0
nACo		450	AlSi/82	75	1,4
			Ti/100		
AlTiCrN	**Vanadis 4**	430	Al/150	50	2.0
			Ti/110		
			Cr/110		
			Al/150		
nACo	**K190**	450	Al/Si/82	75	1.4
			Cr/100		

3 Scratch test and its evaluation potential

The scratch test was carried out using a scratch tester CSEM REVETEST in the mode of changing normal force F_n, which was increased at a constant rate over the range of 0 - 100 N. During the test the holder with a specimen travelled at a constant speed beneath the indenter. We used a standard Rockwell diamond indenter with tip radius of 0.2 mm. During the measurement we recorded relationship between acoustic emission AE and coefficient of friction μ as a ratio of tangential and normal forces F_t/F_n in dependence on normal force F_n. After the test we documented morphology of the coated specimen surface failure under a light microscope. The force resulting in the first marked failure of the thin coating is called critical force F_c and quantifies the adhesion of the thin coating to the substrate. The morphology was accentuated by means of polarized light and Nomarski differential interference contrast [15]. In order to ensure lucidity of evaluation, the specimens were divided to two groups: Ist group included coatings based on TiAlN and CrN on steel S600 and the IInd group comprised nanocomposite coating (TiAl)N/nc-Si_3N_4 deposited on steel S600, Vanadis 4 and K190. **Fig. 1** shows a graphical illustration of the relationship between the signal of acoustic emission and the load acting on the indenter on specimen surface for the coated systems TiAlN-S600, CrN-S600 and TiAlN/CrN-S600.

The course of the AE signal in dependence on normal force F_n served as a signal of failure reflected in scratch morphology. Adhesive – cohesive failure of the coated system was indicated by the first marked increase in the signal of acoustic emission AE, i.e. at normal force F_n ~ 80 N for CrN coating, F_n ~ 55 N for TiAlN coating and F_n ~ 76 N for TiAlN/CrN coating.

Graphical relationship between coefficient of friction and normal force, shown in **Fig. 2**, reflected morphology of failure resulting from the scratch test. Higher coefficient of friction *μ* was measured for specimen with TiAlN coating. Starting from the medium level of normal force F_n, approx. 56 N, one could observe higher “undulation” which corresponded to specimen surface failure due to scratch indentation. Failure of the system TiAlN/CrN showed more pronounced manifestation of coefficient of friction with regard to the different behaviour of both thin coatings, showing a more marked increase in μ at normal force F_n ~ 75 N.

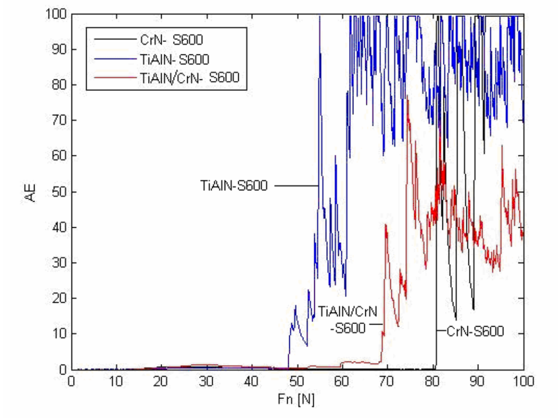

Fig.1 Graphical recording of acoustic emission AE for the respective coated systems

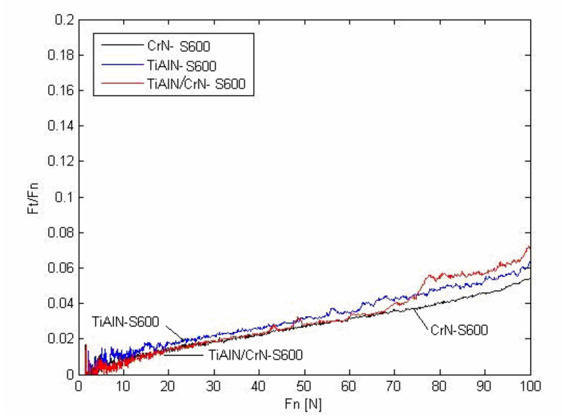

Fig.2 Graphical recording of friction coefficient as a ratio of forces F_t/F_n in dependence on the normal F_n

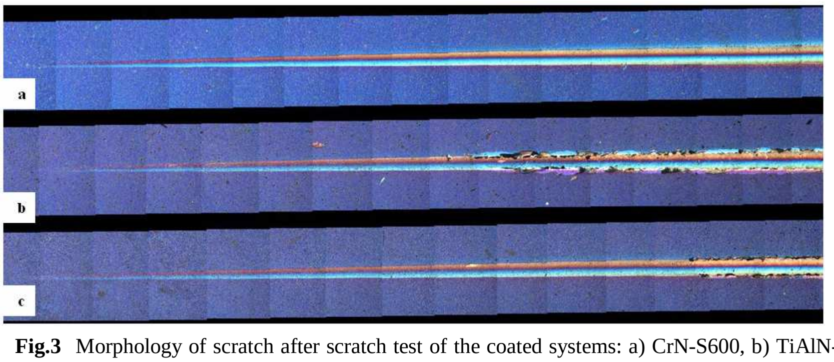

Fig.3 Morphology of scratch after scratch test of the coated systems: a) CrN-S600, b) TiAlN- S600, c) TiAlN /CrN-S600

Morphology of scratch for the investigated types of coatings is illustrated in **Fig. 3**. Considering the total extent of the acting load force 0 - 100 N (corresponding to the width of figures), it was possible to determine that the failure occurred at Fn ~ 80 N with CrN, at Fn ~ 56 N with

TiAlN and at Fn ~ 78 N with TiAlN/CrN. These values show the degree of adhesion of the investigated types of coatings.
Graphical illustration of the acoustic emission AE for specimens with nanocomposite coating deposited on the respective steel substrates in **Fig. 4** is shown.

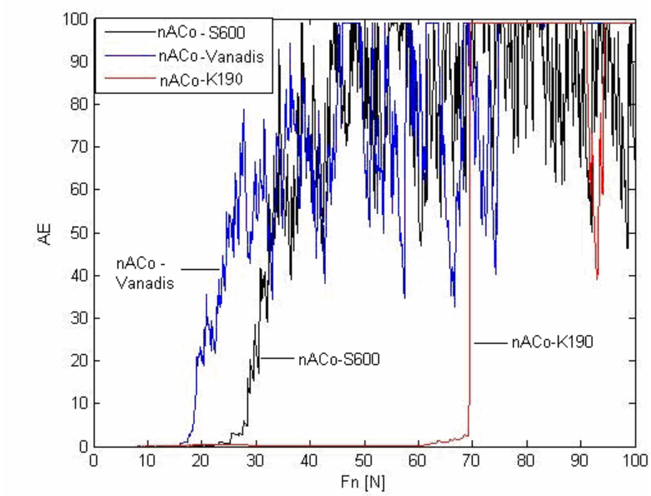

Fig.4 Graphical recording of acoustic emission AE for the respective coated systems

Adhesion – cohesion failure of the systems with deposited nanocomposite coating (TiAl)N/a-Si_3N_4 (nACo) was determined on the basis of graphical increase in the signal of acoustic emission AE. The first marked failure of the coating nACo was observed at normal force $F_n \sim 25$ N when deposited on steel Vanadis 4, at $F_n \sim 45$ N when deposited on steel S600 and the first marked increase in AE occurred at $F_n = 70$ N when nACo was deposited on steel K 190. Graphical illustration of the relationship between coefficient of friction and the normal force for the nanocomposite coated systems is presented in **Fig. 5**.

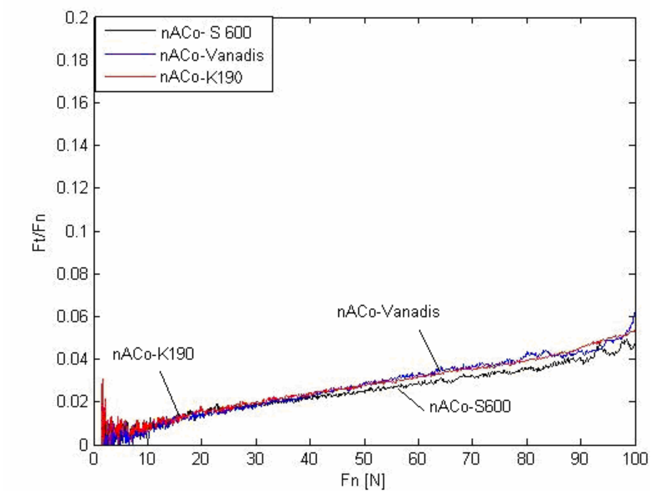

Fig.5 Graphical recording of the coefficient of friction μ in relation to the normal force F_n

The coefficient of friction as a function of the normal force showed the lowest values with nACo coating on steel S600. After the normal force F_n of approx. 60 N we observed an "undulation" in the course of μ which corresponded to adhesion – cohesion failure of the system. Close to the maximum force, at approx. 90 N the oscillations were bigger and corresponded to a more marked failure caused by the indenter. Similar course of μ was recorded for the system Vanadis 4-nACo, with the first more marked „undulation" in the course of coefficient of friction observed close to $F_n \sim 27$ N. A steady growth of μ was recorded for the specimen nACo-K 190. At the normal force $F_n \sim 85$ N this specimen exhibited a change in the slope of the recording of the coefficient of friction signal which could reflect deeper penetration of the indenter into specimen surface and lower hardening of the surface layer.
Morphology of the scratch of the investigated coated systems types is presented in Fig. 6. Considering that the acting normal load force ranged between 0 and 100 N (corresponding to the width of figures), it was possible to determine that the failure occurred at $F_n \sim 45$ N for nACo on steel S600. For nACo coating on steel Vanadis 4 the failure was recorded at $F_n \sim 45$ N. Contrary to the study of morphology of coating failure by scratch indentation it is obvious that a more marked increase in acoustic emission and coefficient of friction occurred somewhat earlier (see **Figs. 4** and **5**) which could result from fine brittle cracking of surface layers. With the system nACo-K190 a marked coating failure was observed only at $F_n \sim 70$ N. The values reported showed the degree of adhesion of coating consisting of nanocomposite-nACo to individual types of steel substrate.

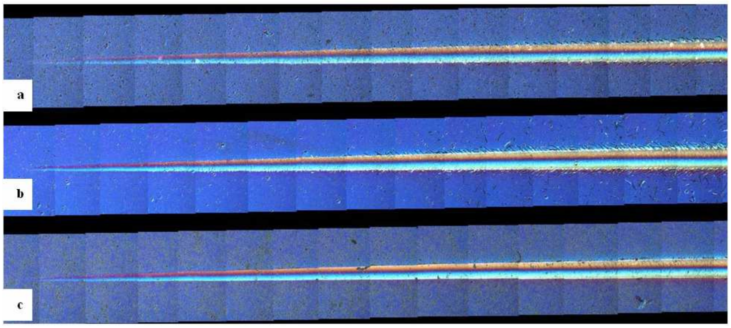

Fig.6 Morphology of scratch track after the scratch test of the investigated coated systems: a) nACo-S600, b) nACo-Vanadis 4, c) nACo-K190

4 Conclusions

Scratch test and its potential evaluation power present ways of complex evaluation of adhesion – cohesion behaviour of materials with deposited thin PVD coatings. On the basis of determination of the course of acoustic emission and coefficient of friction in dependence on the normal force of Rockwell indenter acting on the surface of coated specimens and also on the basis of evaluation of morphology of the scratch after the scratch test it is possible to draw the following conclusions:

- The origin and development of failure indicated that the highest resistance to adhesion failure was detected for the system CrN-S600, with critical force of failure $F_c \sim 80$ N; TiAlN/CrN-S600 showed degree of adhesion characterised by $F_c \sim 76$ N, and for nACo-K190 the first marked failure occurred at normal force $F_n \sim 70$ N. This confirmed very good adhesion properties of the coatings CrN (internal layer within the multilayer coating TiAlN/CrN) and nACo which were frequently reported in specialised literature [16,17];

- Good adhesive – cohesive properties were observed also with coating TiAlN on steel S600, with critical force F_c , reflecting degree of adhesion, reaching approx. 55 N; Difference between the values obtained from graphical recordings of acoustic emission AE and coefficient of friction μ for nACo coating on steels Vanadis 4 and S600 were ascribed to fine brittle failure which did not result in the loss of adhesion and was reflected only in the increase in the signal AE and μ. Because of that the first marked failure observed at investigation of scratch morphology on specimen surface appears to be more accurate way of determination.

Acknowledgements

Authors are grateful for the support of experimental works by national project VEGA 2/0109/08.

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