Influence of the PVD and CVD technologies on the residual macro-stresses and functional properties of the coated tool ceramics

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ABSTRACT

Purpose: The goal of this work is to compare the macro-stresses as well as mechanical and functional properties of the PVD and CVD coatings deposited on oxide and nitride ceramics tool.

Design/methodology/approach: In the paper the residual macro-stresses of PVD and CVD coatings deposited on tool ceramics substrates, measured with the application of the grazing angle X-ray diffraction geometry are compared in relation to the fundamental mechanical properties like hardness and adhesion.

Findings: Basing on the tribological test made by the use of the „pin-on-disk" method, the influence of the residual macro-stresses to wear resistance is being investigated.

Practical implications: The process of covering tools with the thin wear resistant PVD and CVD coatings is currently the most commonly method used to extend their life. Investigations of those coatings determining their mechanical properties, chemical composition, and structure, enable to pick out the optimum coatings for given industrial applications.

Originality/value: New X-ray diffraction g-sin2ψ method based on glancing angle X-ray beam geometry (GID), macro-stresses of the examined CVD and PVD coatings have been measured.

Keywords: Tool ceramics; PVD; CVD; Residual macro-stresses, Micro-hardness; Scratch test; Tribological test

1. Introduction

Stresses that occur in hard coatings deposited on the edges of cutting tools have a significant importance for determining functional properties of these tools. Those stresses appear on almost all-vacuum metallic and non-metallic deposited coatings. Stresses that appear on coatings are determined by: dilatability factor of the coating and substrate, the structure of the coating, the relation between the deposition temperature and the melting point of the coated material, working gas pressure and its kind, glancing beam angle of the deposited material, temperature in which this coating is exploited and methods of deposition. In the coating there are thermal and internal stresses that together make up stresses crucial for determining the functional properties of cutting tools. As regards a functional use of the edges of cutting tools, it is better when there are compressive stresses in their coatings as most materials are more resistant to compression than tension. Compressive stresses in the coating are more favorable when the substrate has a bigger thermal expansion coefficient than the material of the coating. The fact that there are compressive stresses in the coating prevents arising of cracks while heating, and reduces the values of compressive stresses or even eliminates them [1-18].

The goal of this work is to compare the macro-stresses as well as mechanical and functional properties of the PVD and CVD coatings deposited on oxide and nitride ceramics tool.

2. Experimental procedure

The investigations were carried out on the inserts made of the $Al_2O_3+ZrO_2$ oxide ceramics, Al_2O_3+TiC oxide ceramics and Si_3N_4 nitride ceramics coated by the PVD and CVD process. The inserts were multilayer coated by the PVD process – Cathodic Arc Evaporation (CAE) and by the CVD process, which were compared with the commercially available inserts from various manufacturers. Specifications of the investigated materials are presented in Table 1.

X-ray diffraction g-$\sin^2\psi$ method based on glancing angle X-ray beam geometry (GID), macro-stresses of the examined CVD and PVD coatings have been measured. The Bruker D8 Advance diffractometer was applied using filtered $K\alpha$ X-ray, wavelength = 1.79021 emitted from a 40 kV cobalt tube with a current of 40mA (Fig. 1).

The g-$\sin^2\psi$ method described in detail in the paper [11,12] is based on a classical $\sin^2\psi$ method in which the σ_φ stress is calculated through measuring elastic deformation of crystal lattice $\varepsilon^L_{\varphi\psi}$ in L direction of measurement system.

$$\varepsilon^L_{\varphi\psi} = \frac{1}{2}S_2\sigma_\varphi \sin^2\psi + S_1(\sigma_1+\sigma_2) = \frac{d_{\varphi\psi}-d_0}{d_0} \quad (1)$$

$$\sigma_\varphi = \frac{\partial \varepsilon^L_{\varphi\psi}}{\partial \sin^2\psi}\left(\frac{1}{2}S_2\right)^{-1} = a \cdot \left(\frac{1}{2}S_2\right)^{-1} \quad (2)$$

where:

a – slope of a straight line,

φ – angle between the system of coordinates of main stresses and a sample,

ψ – angle between the normal to the surface and the normal to yielding planes {hkl},

S_1, $\frac{1}{2}S_2$ – diffraction elasticity constants for isotropic body: $S_1 = -\nu/E$, $S_2 = (1+\nu)/E$,

ν – Poisson ratio,

E – Young's modulus,

$d_{\varphi\psi}$ – distance between crystallographic planes {hkl} in the direction defined by φ and ψ angles,

d_0 – distance between crystallographic planes {hkl} in material without macroscopic stresses.

Table 1.
Specifications of the PVD and CVD coatings deposited on the tool ceramics

| Substrate	Coating type	Coating composition	Coating thickness, μm	Process type
$Al_2O_3+ZrO_2$ oxide ceramics	multilayer	TiN+multiTiAlSiN+TiN	2.3	PVD
	gradient	TiN+TiAlSiN+AlSiTiN	2.2	PVD
Al_2O_3+TiC oxide ceramics	multilayer	TiN+multiTiAlSiN+TiN	1.5	PVD
	gradient	TiN+TiAlSiN+AlSiTiN	2.0	PVD
Si_3N_4 nitride ceramics	double layer	TiC+TiN	5.4	CVD
	double layer	TiN+Al_2O_3	10.0	CVD
Si_3N_4 nitride ceramics (commercial inserts)	multilayer	TiN+Al_2O_3+TiN	3.8	CVD (commercial)
	double layer	Al_2O_3+TiN	2.6	CVD (commercial)
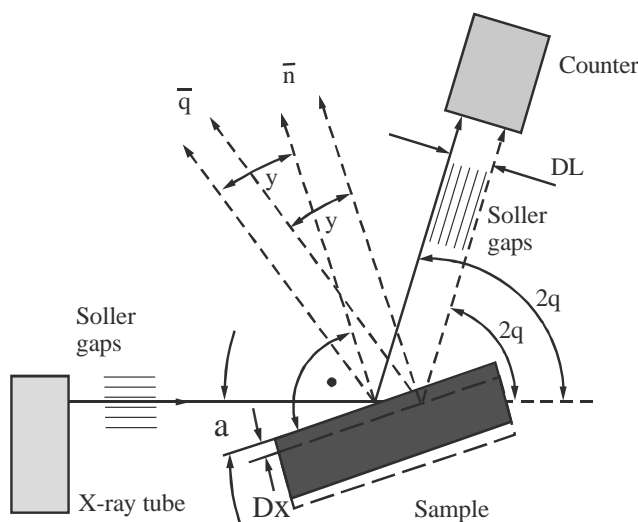
Fig. 1. Using glancing angle X-ray beam diffraction geometry a minor plane shift from the goniometer axis does not influence on the value of the error of determining angle positions of diffraction lines [12]

A linear interrelation between elastic deformation of crystal lattice and components of stress tensor refers to an isotropic body in a plane and homogenous state of stress [12].

The glancing angle of the X-ray beam was small 2. For all the samples, diffraction lines that come from the external TiN coating have been analyzed, from planes: (220), (311), (222), (400), (331), (420), while in case of TiN+Al_2O_3 coating – for the Al_2O_3 coating-from planes: (012), (104), (110), (113), (024), (116), (214), (300), (220), (223), (0210), (226), (042), (2110), (318), (229), (324).

The microhardness tests of coatings were curried out on the SHIMADZU DUH 202 ultra microhardness tester. Test conditions were selected so that the required and comparable test results should be obtained for all analyzed coatings. Measurements were curried out under at 0.07 N loading, eliminating influence of the substrate on the measurement results.

Adhesion evaluation of the coatings on the investigated inserts was measured using the scratch test on the CSEM REVETEST device, by moving the diamond intender along the examined specimen's surface with the gradually increasing load. The tests were made with the following parameters: load range 0-100 N, load increase rate (dL/dt) 100 N/min, intender's speed (dx/dt) 10 mm/min and acoustic emission detector. The critical load L_C, at which coatings' adhesion is lost, was determined based on the registered values of the acoustic emission AE.

Tribological tests were carried out on the CSEM „pin-on-disk" tester in the following conditions: counter-specimen – ball made of the WC and titanium carbide with 6 mm in diameter, counter-specimen load – 5 N, friction radius – 5 mm, linear velocity – 0.1 m/sec, ambient temperature – 20°C. The character of the developed failure was evaluated by observations on the light microscope and on the scanning electron microscope.

3. Discussion of investigation results

3.1. Residual macro-stresses of the investigated coatings

The measurement of macro-stresses of the investigated PVD and CVD coatings in the external layer, carried out using the X-ray diffraction method due to glancing angle diffraction geometry (Table 2).

In case of PVD coatings deposited on oxide tool ceramics substrate the occurrence of compressing stresses has been stated while in case of CVD coatings deposited on nitride ceramics substrate the occurrence of tension stresses have been noted. The value of the internal stresses of the investigated coatings may be assumed to be small for the level up to 500 MPa, and medium, for values about 1000 MPa, compared to the data obtained in other research project reports [9,12,13], in which the coating stresses are in the range from 1000 to 6000 MPa. The biggest absolute value of compressive stress (σ_φ = -216 MPa) has been noted in case of TiN+multiTiAlSiN+TiN, coating deposited on Al_2O_3+TiC substrate while the smallest absolute value of tension stress (σ_φ = +590 MPa) has been stated in TiN+Al_2O_3 coating deposited on Si_3N_4 substrate. Moreover, it has been noted that the minimal absolute value of tension stress as well as maximal absolute value of compressive stress influenced both the increase of micro-hardness and the improvement of adhesion and resistance to abrasive wear of examined coatings (Figs. 2,3). Results of internal macro-stresses of the investigated coatings' indicate to the qualitative correlation between the stress and hardness and adhesion as well.

3.2. Hardness and adhesion of the investigated coatings

The microhardness tests revealed that the uncoated oxide ceramics has hardness from 18.5 GPa in case of Al_2O_3+ZrO_2 substrate to 19.7 GPa in case of Al_2O_3+TiC substrate. Deposition of the PVD and CVD coatings onto the Al_2O_3+ZrO_2 based specimens causes the surface layer hardness increase reaching from 21.0 to 40.9 GPa that is up to 100% more compared to the uncoated substrate hardness. The highest hardness i.e. 40.9 GPa was observed in case of the TiN+multiTiAlSiN+TiN coating. It was found out, basing on microhardness tests of the Al_2O_3+TiC based inserts, that the oxide ceramics with also TiN+multiTiAlSiN+TiN coating is the hardest one, with hardness of 40.3 GPa. No dependence was revealed between the substrate hardness and hardness of the deposited surface layer (Table 2).

The microhardness tests revealed that the uncoated nitride ceramics has hardness equal to 18.5 GPa. Deposition of the PVD and CVD coatings onto the specimens causes the surface layer hardness increase reaching from 19.8 to 32.6 GPa that is almost 100% more compared to the substrate hardness. The highest hardness of 32.6 GPa was observed in case of the TiN+Al_2O_3 coating.
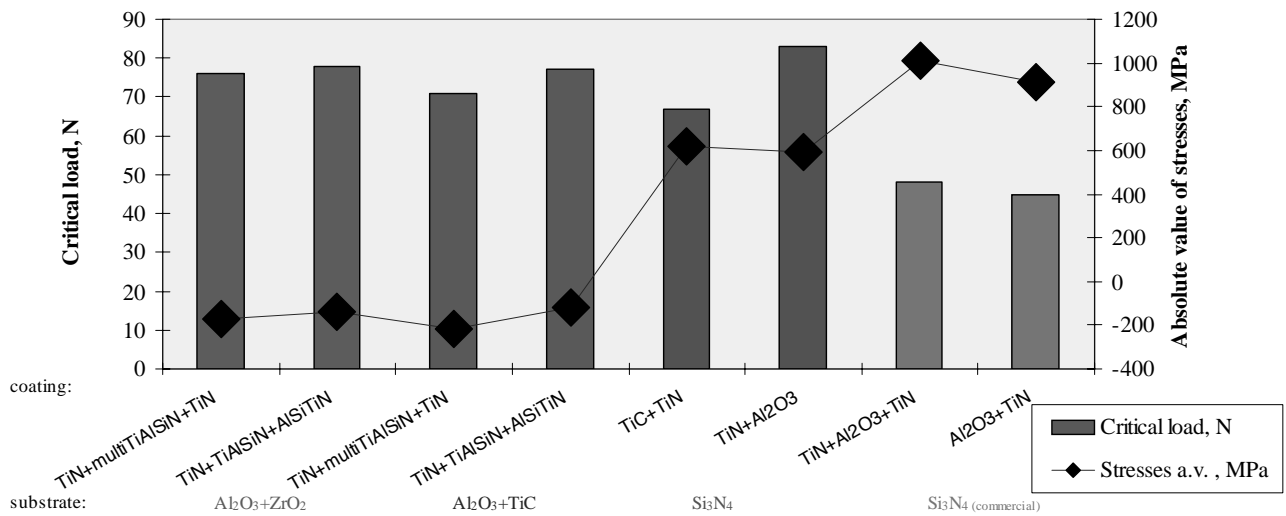
Fig. 2. Comparison of residual stresses and critical load of investigated coatings deposited on different substrates
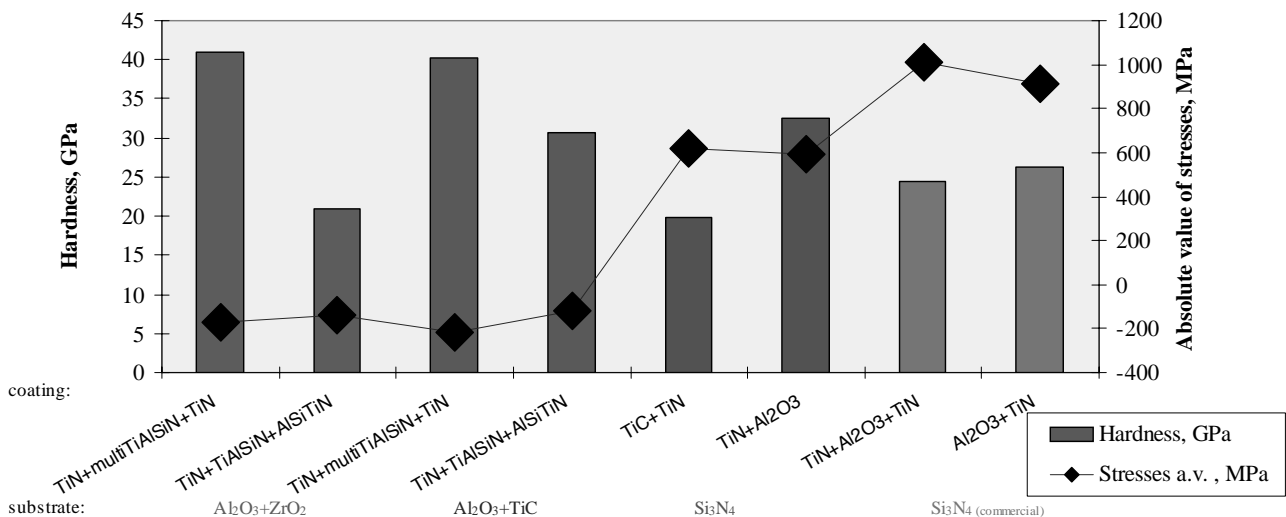

Fig. 3. Comparison of residual stresses and microhardness of investigated coatings deposited on different substrates

It was found out, basing on microhardness tests of the commercially available CVD coated inserts, that the nitride ceramics with the Al_2O_3+TiN coating is the hardest one, with hardness of 26.3 GPa. No dependence was revealed between the substrate hardness and hardness of the deposited coatings (Table 2). Three samples with compressive residual stresses have also largest micro hardness of coatings, whereas tensile residual stresses appear in samples with smaller micro hardness (Fig. 3).

The critical load values L_c (AE) were determined using the scratch method with the linearly increasing load („scratch test"), characterizing adherence of the investigated PVD and CVD coatings to the oxide and nitride ceramics.

The critical load was determined as the one corresponding to the acoustic emission (AE) increase signaling beginning of

spelling of the coating. The character of the defect responsible for the acoustic emission increase due to scratch development during the test is scanned with simultanously light microscope coupled, determining the critical load L_c value basing on the metallographic examinations (Fig. 4).

In case of Al_2O_3 based inserts, it was found out that the highest critical load of L_c = 77-78 N reveals the TiN+TiAlSiN+AlSiTiN PVD coating deposited on booth oxide substrates (Al_2O_3+ZrO_2 and Al_2O_3+TiC); whereas the lowest one of L_c = 71 N the TiN+multiTiAlSiN+TiN PVD coating deposited onto Al_2O_3+TiC substrate (Table 2).

In case of Si_3N_4 based inserts (Fig. 4), it was found out that the highest critical load of L_c = 83. N reveals the TiN+Al_2O_3 CVD coating deposited onto Si_3N_4 substrate; whereas the lowest one of

Table 2.
Mechanical and functional properties of uncoated and coated ceramic tools are compared

Substrate	Coating composition	Residual Stresses, MPa	Hardness, GPa	Critical load, Lc
$Al_2O_3+ZrO_2$ oxide ceramics	uncoated	-	18.5	-
	TiN+multiTiAlSiN+TiN	-170	40.9	76
	TiN+TiAlSiN+AlSiTiN	-141	21.0	78 (opt.)
Al_2O_3+TiC oxide ceramics	uncoated	-	19.7	-
	TiN+multiTiAlSiN+TiN	-216	40.3	71
	TiN+TiAlSiN+AlSiTiN	-120	30.7	77 (opt.)
Si_3N_4 nitride ceramics	uncoated	-	18.5	-
	TiC+TiN	616	19.8	67
	TiN+Al_2O_3	590	32.6	83
Si_3N_4 nitride ceramics (commercial inserts)	uncoated	-	18.5	-
	TiN+Al_2O_3+TiN	1008	24.4	48
	Al_2O_3+TiN	915	26.3	45

a)

b)
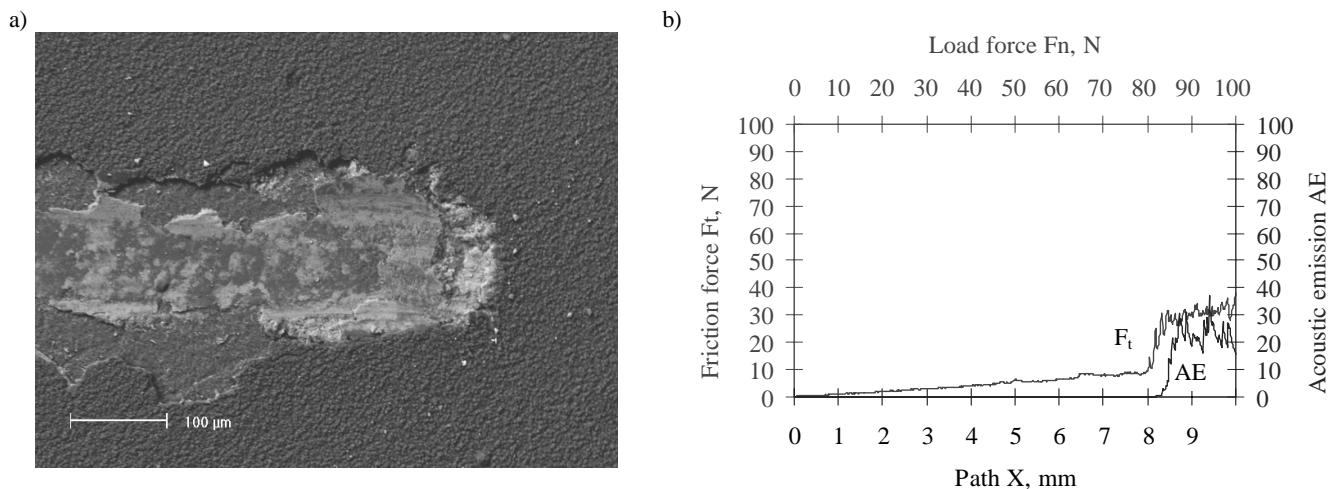
Fig. 4. a) Indenter trace with the L_c=100 N load, b) scratch test results of the TiN+Al_2O_3 coating surface deposited on Si_3N_4 substrate

L_c = 45 N the Al_2O_3+TiN commercial coating deposited onto Si_3N_4 substrate (Table 2). The lawest critical load was found in samples with highest tensile residual stresses, whereas highest adhesion load appeared in samples with compressive residual stresses (Fig. 2).

3.3. Tribological properties of the PVD and CVD coatings

The extensive adherence coating defects were revealed at the contact zone with the counter-specimen in the tribological examinations of the PVD coatings deposited onto the oxide and nitride ceramics substrate. On the basis of preliminary analyses and tests done, a constant n-30000 cycles for all examined coatings deposited on booth oxide ceramics ($Al_2O_3+ZrO_2$ and Al_2O_3+TiC) has been accepted, that allows for carrying out metallographical comparative analysis of the band of a coating damage. For the majority of coatings deposited on oxide ceramic tools substrate there has been no full damage of the coating noted after less than n=30000 number of cycles. In case of TiN+TiAlSiN+AlSiTiN coating deposited on $Al_2O_3+ZrO_2$ substrate, the number of cycles was smaller because of the noticed damage of the coating, yet the damage of the coating after less than n=5.500 number of cycles has not been noticed, what also shows very good tribological properties of the coating (Fig. 5).
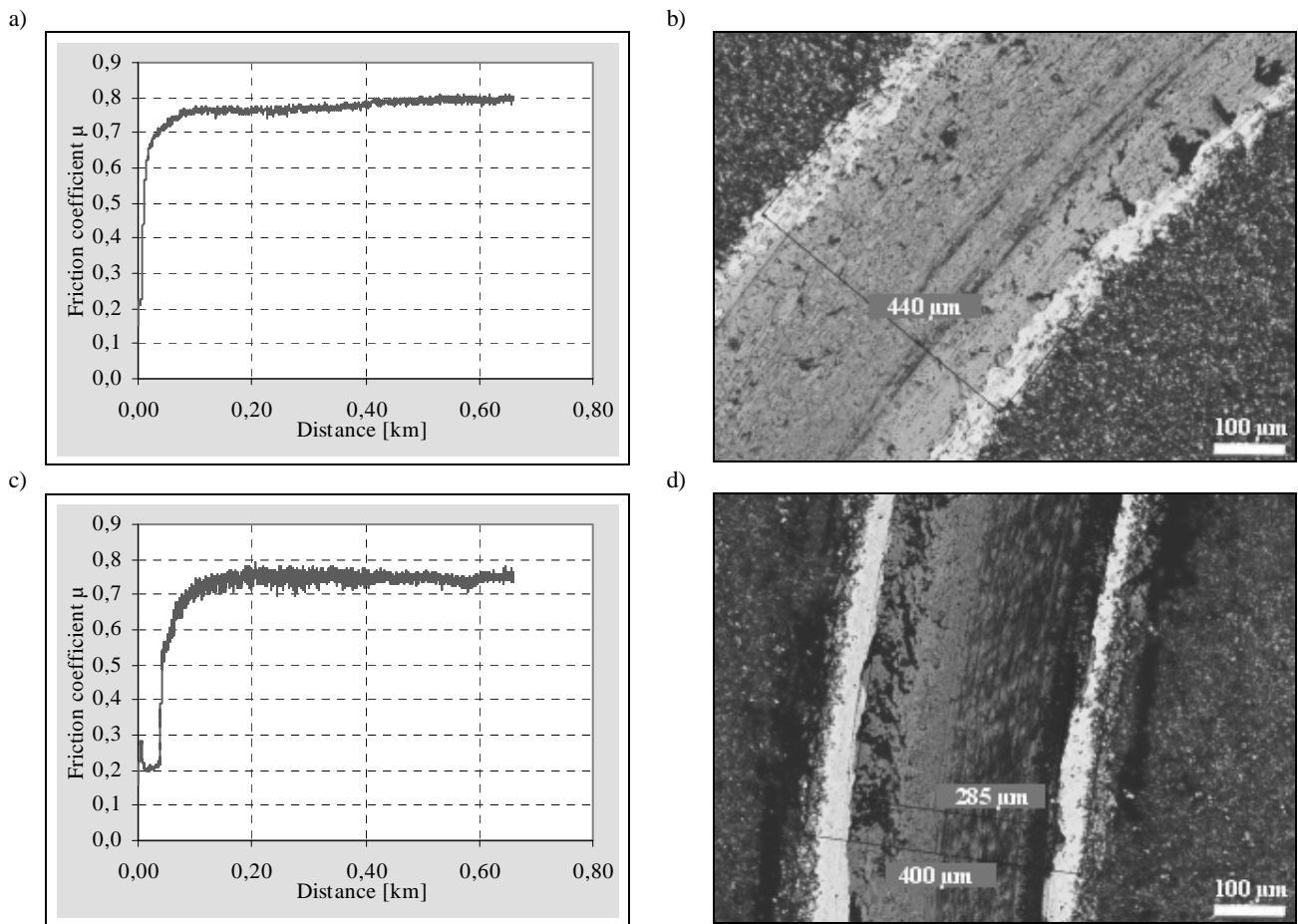
a)

b)

c)

d)

Fig. 5. Tribological properties of TiN+multiTiAlSiN+TiN coatings deposited on oxide ceramic substrates: a) relationship of the friction coefficient versus friction trace length during the pin-on-disc test for coating deposited on $Al_2O_3+ZrO_2$ substrate, b) measurement of the tribological failure trace width on the coating surface on $Al_2O_3+ZrO_2$ substrate, c) relationship of the friction coefficient versus friction trace length during the pin-on-disc test for coating deposited on Al_2O_3+TiC substrate, d) measurement of the tribological failure trace width on the coating surface deposited on Al_2O_3+TiC substrate

4. Summary

The quantity of the internal stresses of the investigated coatings may be assumed to be small for the values up to 500 MPa, and medium, for values about 1000 MPa, compared to the data obtained in other research project reports, in which the coating stresses are in the range from -1000 to -6000 MPa. It has been stated that the minimal absolute value of tension stress as well as the maximal absolute value of compressing stress influence both the micro-hardness and the adhesion and resistance to abrasive wear of the examined coatings.

On the basis of "pin-on-disc" tests carried out, it has been noted that for the majority of coatings deposited on oxide ceramic tools, there is no full damage of the coating after less than n=30000 number of cycles, what means good tribological properties of the deposited coatings.

As a result of the deposition of anti-wear PVD and CVD coatings on the examined oxide and nitride ceramic tools, a significant increase of the hardness of samples has been achieved what allows for the improvement of the usable properties of examined cutting tools. The interrelations shown, between the value of residual stresses and the hardness and adhesion, may be crucial while designing new techniques of coating depositions.

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