Application of the indentation method for cracking resistance evaluation of hard coatings on tool steels

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Abstract. In this paper cracking resistance and fatigue properties of five hard coatings – TiN, TiCN, TiAlN, AlTiN and nanocomposite nc-AlTiN/a-Si$_3$N$_4$ (nACo®) on tool steels Weartec[TM] and Vanadis 6 are evaluated by means of the cyclic Vickers indentation method. The analytical part covers an evaluation of damage evolution of the coated system versus the number of cycles. The comparative adhesion testing was conducted with the use of the Rockwell C technique. It was found that the type of cracks formed in the coated systems under cyclic loading depends on the Young modulus/hardness ratio. The data obtained permits to compare the cracking and fatigue resistance of the coated systems and to select the optimal coating for bulk and sheet metal forming tools.

Key words: PVD, hard coating, multilayers, indentation method, cracking, delamination.

1. INTRODUCTION

The functional properties of hard coatings have gained increasing importance as their application for sheet metal cold forming tools has grown recently. Fatigue and cracking resistance are the most important properties in such long-term applications where alternating loads are applied. Investigations of the coating crack development intensity and fatigue are commonly carried out using the indentation method for its ability to measure mechanical properties in the microscopic range [1–5].

The main elements of damage evolution and fatigue response in coated brittle systems are mechanical properties like the Young modulus/hardness ratio (E/H ratio) [4,6], microstructure and deposition technique. Two major crack types were observed in brittle materials during indentation tests: cone and radial (Palmqvist)

cracks [1,4,5]. The elastic brittle hard coatings do not show plastic deformation up to tensile fracture (normal stress) and may not suffer fatigue damage. Thus the crack propagation of the coated system may be attributed to the ratcheting deformation of the substrate under cyclic indentation. Two damage modes of coated systems: the tensile-driven cone cracking ("brittle" mode) and shear-driven microdamage accumulation ("quasi-plastic" mode) were determined [5].

This paper investigates cracking resistance of hard ceramic coatings subjected to cyclic Vickers indentation. The dependence of the crack types and the intensity of crack development on the coating type, deposition technique and substrate material are determined.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

A spray formed (SF) cold work tool steel – Weartec™ and powder metallurgical (PM) high-speed steel (HSS) Vanadis 6, produced by Uddeholm, were used as substrates for coating deposition (Table 1). According to the manufacturer, PM steels have smaller carbide size and more homogeneous structure than SF steels resulting in better chipping/cracking resistance.

Five different PVD coatings, among them monolayer TiN and multilayer gradient TiCN, nanocomposite nc-(AlTi)N/a-Si_3N_4 (nACo®), TiAlN and AlTiN, were studied. TiAlN and AlTiN coatings were deposited only on Weartec™ substrate to reduce the number of experiments. Cross-sections of coated specimens, observed on the scanning electron microscope (SEM) are presented in Fig. 1. Mechanical properties of the coatings (Table 2) were obtained by MTS Nano Indenter XP® in a depth mode with a target depth of 150 nm and average indentation force of 12 mN. The thicknesses of the coatings, measured by Kalotester kaloMax®, was about 2.3 μm. The results presented in Table 2 are in good correlation with those available in the literature. In [7] the TiN coating with a thickness of 2.1 μm on HSS had the nanohardness of 27 GPa and Young's modulus about 305 GPa. Fouvry et al. [8] investigated a TiCN coating with a thickness of 2.5 μm on HSS which had Young's modulus of 550 GPa. The substrate material and coating surface roughness were measured by a surface roughness measuring instrument of Taylor Hobson Ltd. Surtronic 3+ (using CR filter) with an accuracy of 2% (Tables 1 and 2).

Table 1. Chemical compositions, mechanical and surface properties of substrate materials

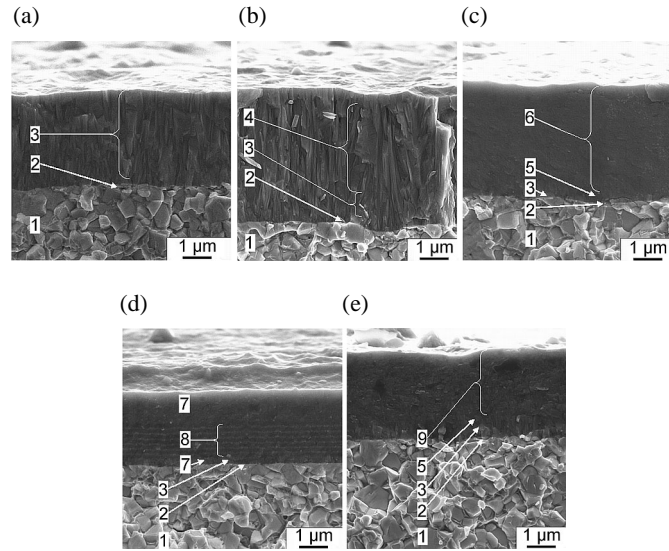| Substrate	Chemical composition, wt%						Hardness HRC/HV30	Young's modulus E, GPa	Surface roughness R_a, μm
	C	Si	Mn	Cr	Mo	V			
Weartec™	2.8	0.8	0.7	7.0	2.3	8.9	64/843	199	0.51±0.10
Vanadis 6	2.1	1.0	0.4	6.8	1.5	5.4	64/843	210	0.51±0.10

Fig. 1. SEM images of fractured surface of PVD coatings: (a) TiN; (b) TiCN; (c) nACo; (d) TiAlN; (e) AlTiN. 1 – substrate; 2 – Ti adhesion layer; 3 – TiN layer; 4 – TiCN gradient coating; 5 – TiAlN gradient layer + AlTiN gradient layer; 6 – nc-AlTiN/a-Si$_3$N$_4$ gradient coating; 7 – TiAlN layer; 8 – TiAlN/AlN multilayer coating; 9 – AlTiN gradient coating.

Table 2. Mechanical and surface properties of coatings

Coating/ Substrate	Type	Young's modulus E, GPa	Nano-hardness, GPa	E/H ratio	Surface roughness R_a, μm
TiN	Monolayer	438 ± 80	28.5 ± 0.6	15.4	0.08 ± 0.01
TiCN	Multilayer	500 ± 90	26.6 ± 1.4	18.8	0.10 ± 0.04
nACo®	Nanocomposite	323 ± 13	29.0 ± 1.5	11.1	0.10 ± 0.04
TiAlN	Multilayer	301 ± 90	19.9 ± 1.2	15.2	0.10 ± 0.04
AlTiN	Multilayer	336 ± 13	23.8 ± 1.0	14.1	0.05 ± 0.01

2.2. Coating deposition procedure

Heat-treated samples (plates) of the size $20 \times 20 \times 5$ mm of two different substrate materials were grinded, diamond polished (powder grain size 1 μm) and degreased ultrasonically in the phosphate-alkali solution before deposition. Coatings were deposited using an arc ion plating PVD technique, on π-80 Platit equipment. The parameters of the deposition process varied from coating to coating and are presented in Table 3.

Table 3. Deposition parameters

Coat-ing	Bias voltage, V	Pressure, mbar	Ti–Al/AlSi cathode arc current, A	Temper-ature, °C	Ar	N_2 flow, sccm	C_2H_2
TiN	−75…−120	8×10^{-3}	(100…125)/–	450	6	200	–
TiCN	−60…−120	$(5…7) \times 10^{-3}$	(120...130)/–	450	6	165…180	7…39
nACo	−75…−150	$9 \times 10^{-3}…1.2 \times 10^{-2}$	(82…125)/(65…100)	435…475	6	200	–
TiAlN	−60…−150	$8 \times 10^{-3}…1.5 \times 10^{-2}$	(85…125)/(65…115)	475	6	200	–
AlTiN	−60…−150	$4 \times 10^{-3}…1.2 \times 10^{-2}$	(60…125)/(52...130)	430…450	6	150…200	–

2.3. Cyclic indentation test procedure

A servo hydraulic fatigue test system INSTRON 8800 and Vickers diamond pyramid indenter were used in the indentation experiments. The total indenting load of 500 N (mean compressive load of 275 N, alternating load of 225 N), stress ratio of 0.1 with a sinusoidal loading pattern and loading frequency of 0.5…15 Hz were applied. The optical microscope Axiovert 25 (ZEISS) with 500× magnification and Buehler Omnimet Image Analysis System 5.40, including the package for crack length measurement (Palmqvist method [9]), was used. The qualitative evaluation criteria of the cracking (0…VI) from crack formation, propagation to delamination (Table 4) were considered.

Table 4. Crack types and crack evaluation criteria

	0	I	II	III	IV	V	VI
Crack type							
Crack type description	Very weak secondary radial cracks	Weak secondary radial cracks	Medium secondary radial cracks and weak radial cracks	Medium secondary radial cracks and medium radial cracks	Medium secondary radial cracks and strong radial cracks	Medium radial cracks, delamina-tion of coating and cone (ring) cracks	Strong radial cracks and delamina-tion of the coating

2.4. Comparative adhesion testing

The adhesion test on the Zwick/ZHR 8150 Rockwell hardness tester at an indentation load of 1471 N (150 kgf) was performed to asses the quality of the coatings. The test procedure followed the VDI 3198 (1992) standard [10].

3. RESULTS AND DISCUSSION

3.1. Adhesion testing

The results of the indentation test are presented by micrographs in Fig. 2. The type and the volume of a failure zone indicate to film adhesion and its brittleness, which correspond to the microstructure and the mechanical properties of the coatings. Coatings of higher E/H ratio withstand the load without nucleation of long radial and conical cracks (TiN and TiCN). However, considerable amount of long radial cracks of 10…50 μm were generated, causing the exfoliation of coating layers that is a typical behaviour of TiN and TiCN coatings under loading [11]. The longest radial cracks are present in the TiN coating and with numerous enfolds and larger exfoliations of the coating layer (Fig. 2a). It is obvious that radial cracks predispose that kind of the coating failure. The same features are seen in the case of the TiAlN coating with only difference – the conical cracks are also present (Fig. 2c). It seems that short radial cracks accelerate chipping on the bordering area of the coating–indenter contact. Emerged chips tend to make connections with the closest of the sides forming the ring or conical crack. The structural defects, presented on the surface, such as pores and non-metallic inclusions, simplify this action. Finally, the most drastically fractured case is the nACo® coating (Fig. 2e) with numerous short radial and closed conical cracks. First, the very brittle structure collapses around the indenter and then starts to take up (absorb) fracture energy by the formation of radial cracks. Eventually those are blunted by the perpendicularly formed conical cracks and do not reach the last "ring".

Among all the tested coatings, the TiCN seems to be the most endurable. The mixed failure modes are characteristic of the studied coated system. Most widely
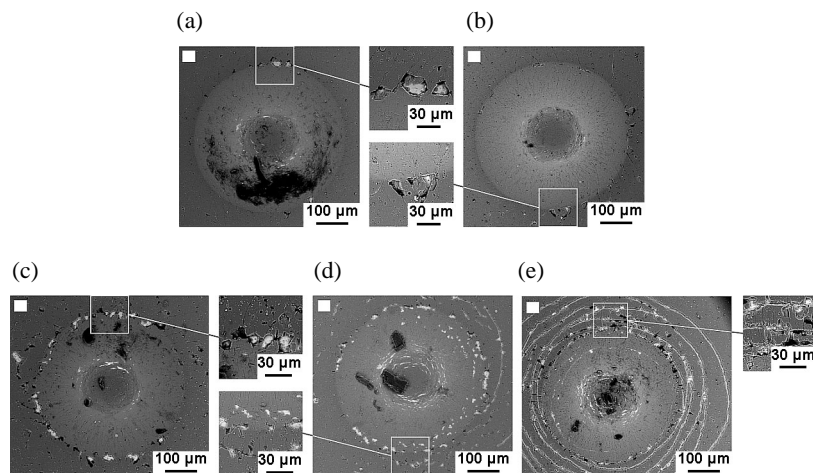

Fig. 2. SEM micrographs of the adhesion test of coatings: (a) TiN; (b) TiCN; (c) TiAlN; (d) AlTiN; (e) nACo®.

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presented are cohesive (chipping, caused by the normal component of the stress tensor) and the delamination with buckling and fracture mode (decohesion of a coating with the formation of microcracks, caused by a combination of shear and normal stresses).

3.2. Coated system behaviour under cyclic loading

The results of cyclic indentation are given in Table 5 and in Fig. 3. The fracture analysis of the TiCN coating revealed that the "quasi-plastic" damage mode with the shortest radial cracks about 20 μm after 10 000 cycles prevailed when the Vickers diamond pyramid was used in the indentation test. In the contrast to the spherical indenters, a sharp indenter penetrates easily into the coating surface and cone cracking formation is suppressed by the radial cracks nucleation (Fig. 3). Radial cracks also become the dominant mode of the indentation fracture of the TiAlN and AlTiN coatings and lead to delamination and accelerated cracking resistance degradation (Fig. 4b). Within multilayer coatings with the "quasi-plastic" damage mode (TiCN, TiAlN, AlTiN) the cracking resistance increased with E/H ratio growth after 10 000 cycles (Fig. 5). Apart from other coatings, in nACo® with the lowest E/H ratio, "brittle" damage mode occurred with the formation of cone cracks, driven by tensile stresses in addition to "quasi-plastic" damage mode (Fig. 4c). On the other hand, the obscured sub-surface damage and the formation of radial cracks could be found below cone cracks. The multilayer coatings are preferable to the monolayer TiN coating with "quasi-plastic" damage mode, one of the lowest cracking resistance and the formation of strong radial cracks, despite the high E/H ratio of TiN (Figs. 4a, 5a).

Table 5. Crack types and crack evaluation criteria

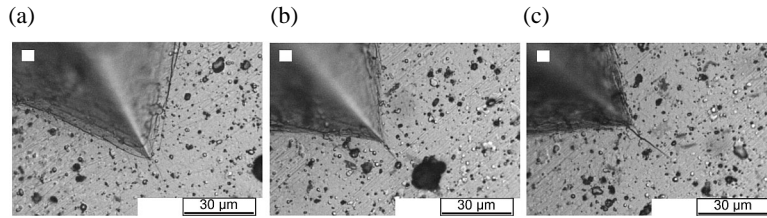|	Cycles					
	1	10	50	100	1000	10 000
TiN/Weartec[TM]	I	I	I	I, II	II	IV
TiN/Vanadis 6	I	I	I	I	II	IV
TiCN/Weartec[TM]	0, I	0, I	0, I	I	I	III
TiCN/Vanadis 6	0	0	0	0, I	0, I	II
nACo®/Weartec[TM]	I	I, II Starting delamination	II Delamination	II Delamination	V Cone crack	V Cone crack
nACo®/Vanadis 6	0	I Starting delamination	V Cone crack	V Cone crack	V Cone crack	V Cone crack
TiAlN/Weartec[TM]	0, I	I	I	I, II	II, III	VI
AlTiN/Weartec[TM]	I	I	I	I	II	VI

Fig. 3. Impression corner of TiCN on Vanadis 6 substrate with the appearance of 0 criteria after 1 cycle (a); 0, I criteria after 1000 cycles (b); II criteria after 10 000 cycles (c).
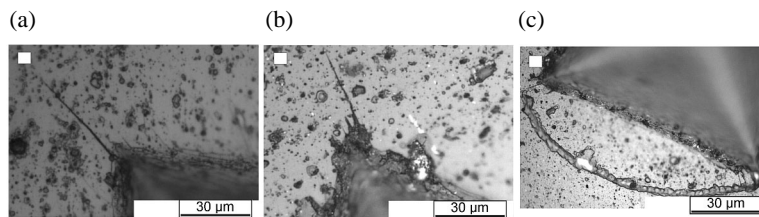

Fig. 4. Impression corner of coatings on Weartec^{TM} after 10 000 cycles with the appearance of IV criteria, TiN (a); VI criteria, AlTiN (b); V criteria, nACo® (c).
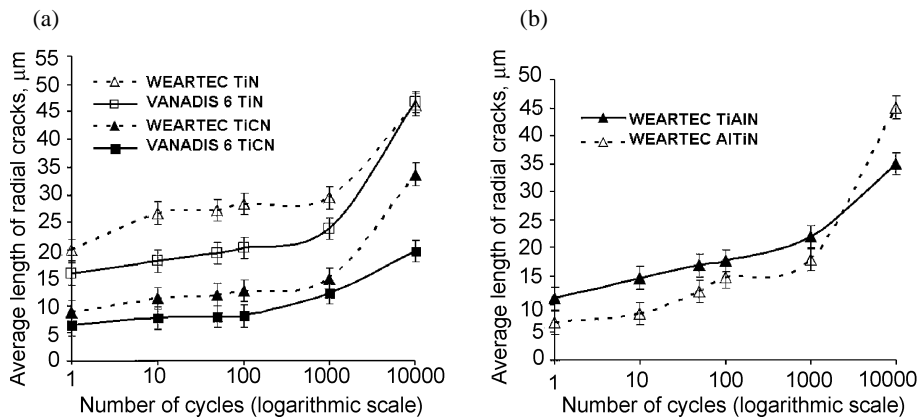

Fig. 5. An average length of radial cracks measured from the indent impression corners depending on the number of indentation cycles (standard deviation bars of 2 μm): (a) TiN and TiCN on Weartec^{TM} and Vanadis 6; (b) TiAlN and AlTiN on Weartec^{TM} substrate.

The observations of both substrates, coated with TiCN, exposed the advantages of PM steel Vanadis 6 over Weartec^{TM}. Vanadis 6 has higher Young's modulus, smaller carbide size and uniform carbides distribution that hampers the propagation of cracks, and crystallographic similarity with the coating, resulting in good adhesion (Fig. 5a). However, Vanadis 6 as a substrate for TiN and nACo® did not show benefits over Weartec^{TM}.

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4. CONCLUSIONS

Indentation cyclic tests of coated tool steels were carried out and the following conclusions can be drawn.

1. The "quasi-plastic" damage mode with the formation of radial cracks prevailed in indentation cycling testing and is typical for high E/H values of coatings. The increase of the number of the indentation cycles leads to the radial crack growth. "Brittle" damage with the formation of cone cracks along with "quasi-plastic" damage mode is characteristic of the cyclically loaded PVD coatings with the lowest E/H ratio.
2. For multilayered coatings with the "quasi-plastic" damage mode (TiCN, TiAlN, AlTiN) the cracking resistance increases with the coating E/H ratio growth. TiCN on Vanadis 6 has the best cracking resistance (II criteria) after 10 000 cycles.
3. Multilayer coatings had higher fatigue resistance than monolayer coatings with higher E/H ratio.

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Tööriistateraste õhukeste kõvapinnete vastupanu hindamine pragunemisele indenteerimismeetodi abil

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On hinnatud viie kõvapinde – TiN-i, TiCN-i, TiAlN-i, AlTiN-i ja nanokomposiidi nc-AlTiN/a-Si$_3$N$_4$ (nACo®), mis on kantud pulbertööriistaterastele Weartec^{TM} ning Vanadis 6 – vastupanu pragunemisele ja väsimusele Vickersi tsüklilise indenteerimise abil. Analüütiline osa sisaldab prao arenemise hinnangut sõltuvalt kasvavate indenteerimistsüklite arvust. Lisaks on tehtud adhesiooni võrdlustest, kasutades Rockwell C tehnikat. On leitud, et pinnatud süsteemi tsüklilisel indenteerimisel tekkivate pragude tüübid on sõltuvuses suhte Youngi moodul/kõvadus (*E*/H) väärtusest. Tööst saadud andmed võimaldavad võrrelda vaadeldud pinnatud süsteemi vastupanu pragunemisele ja on aluseks pinnete valikul külmvormimise (maht- ning lehtvormimise) tööriistadele.