SURFACE FATIGUE AND WEAR RESISTANCE OF THIN AND THICK HARD COATINGS[1]

Priit Kulu[2]
Mart Saarna[2]
Fjodor Sergejev[2]
Andre Gregor[2]
Alina Sivitski[2]
Hartmut Müller[3]

Abstract

Among the wear resistant coatings and deposition techniques physical vapour deposition (PVD) of hard thin coatings and high-velocity oxy-fuel (HVOF) spraying of thick WC-Co-based powder coating have provided good wear protection against various types of wear including impact wear. Different PVD coatings (mono- and multilayer) on the different substrates (hardmetal, cold work tool steel and nitrided steel) and from the thermal spayed coatings hardmetal based HVOF sprayed coatings were under the study. Influence of the post-treatment of the PVD and HVOF spray coated surfaces to the structure and properties of substrate and coatings were also studied. Cracking resistance and surface fatigue at cyclic indentation and impact wear resistance of coating systems were determined. As the result of the study of different coating systems the criteria for the coatings selection and recommendations for the improving of their wear resistance at different conditions of exploitation are proposed.

Keywords: Hard coatings; Wear resistance; Surface fatigue.

---

[1] *Technical contribution to the 18th IFHTSE Congress - International Federation for Heat Treatment and Surface Engineering, 2010 July 26-30th, Rio de Janeiro, RJ, Brazil.*

[2] *Professor, Department of Materials Engineering, Tallinn University of Technology, Tallinn Estonia.*

[3] *Abteilung Strahltechnik, Günte –Köhler–Institut für Fügetechnik und Werkstoff-prüfung. Otto–Schott–Straβe 13, 07745 Jena, Germany.*

1 INTRODUCTION

Thin hard PVD coatings often are not able to work in conditions of high loads on a relatively soft substrate material. To wide their applications areas (not only by the substrates hardmetals and high-speed steels) one of the ways is using coating followed heat treatment.[1] For production of gradient coatings using nitriding with following hard coatings (duplex coatings) on nitriding steels is also prospective.[2] A subsequent heat treatment of the hard coated low-tempering steels by using high-energy beams, especially laser beam for substrate hardening, causes a notable improvement of its loads support.[3,4]

Thermal sprayed hardmetal coatings are very prospective for wear protection and provide good wear and corrosion resistance.[5] WC-Co hardmetals are the most widely used materials for different wear applications owing to their excellent combination of high wear resistance and good strength-toughness. At impact wear the exposed surface should be able to withstand repeated deformation. The plastic deformation, direct brittle and fatigue fracture are dominating and there is a controversy between the hardness and fracture toughness of coatings. To increase their load support heat treatment of substrate is also prospective.[6]

2 MATERIALS AND METHOD

2.1 Substrate Materials and Coatings

Three different substrate–coatings systems: hardmetal–coating, cold work tool steel–coating and nitrided steel–coating were studied. The heat treated samples of size 20x20x5 mm were grinded and diamond polished. Three different PVD coatings, monolayer TiN, TiCN and multilayer TiAlN, were deposited using the PLATIT *П*80. The properties of substrates and coatings are given in Tables 1 and 2. The microstructure of coated systems was observed by the SEM. The thickness of the coatings was measured by Kalotester KaloMax®.

Table 1. Substrate materials and their properties

Type of substrate	Designation	Condition	Surface hardness HV, GPa	Modulus of elasticity E, GPa	H^3/E^2
Hardmetal (HM)	H10 (WC10Co)	As produced	15.5 HV0.3	590	0.011
Cold work tool steel (CWTS)	Vanadis 6	Hardened	8.4 HV0.01	210	0.013
Nitrided steel (NS)	38CrMoAl8	Nitrided	8.5 HV0.01	275	0.008

Table 2. Studied PVD coatings

Type of coatings	Substrate	Coating thickness, µm	Coating hardness $^{2)}$, GPa	Modulus of elasticity E, GPa	*H/E*
TiN, monolayer	HM, CWTS	2.3	– / 28.5	438 ± 80	0.065
TiCN	HM, CWTS	2.3	– / 31.0	345 ± 31	0.090
TiAlN, multilayer	HM, CWTS	2.3	– / 19.9	301 ± 90	0.066
TiN, duplex	NS	300 / 2.3$^{1)}$	8.5 / 28.5	275 / 438	0.031 / 0.065$^{1)}$
TiAlN, duplex	NS	300 / 2.3	8.5 / 19.9	275 / 301	0.031 / 0.066
TiN, laser hardened	CWTS	1400 / 2.3	4.3 – 5.6 / 28.5	275 / 438	0.018 / 0.065

$^{1)}$ *nitrided layer or laser hardened zone / PVD coating*
$^{2)}$ *microhardness of nitrided layer or hardened zone / nanohardness of PVD coating*

The cross-sections of TiN, TiCN and TiAlN coatings on the different substrates are given in Figures 1 and 2.

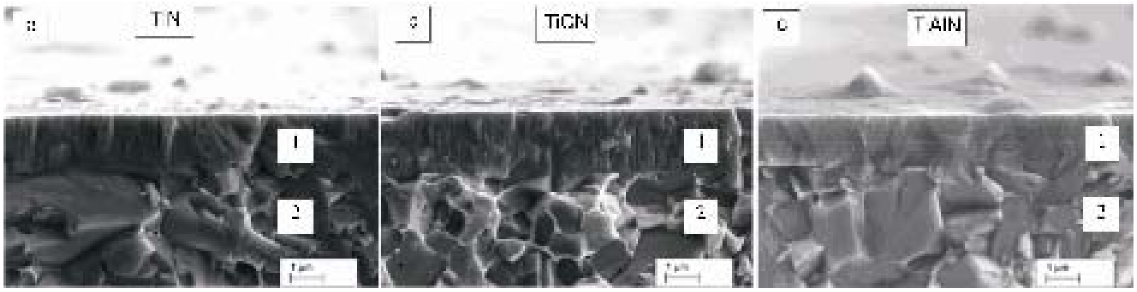

Figure 1. SEM images of fractured surface of PVD coatings on hardmetal: a– monolayer TiN, b– monolayer TiCN and c– multilayer TiAlN.

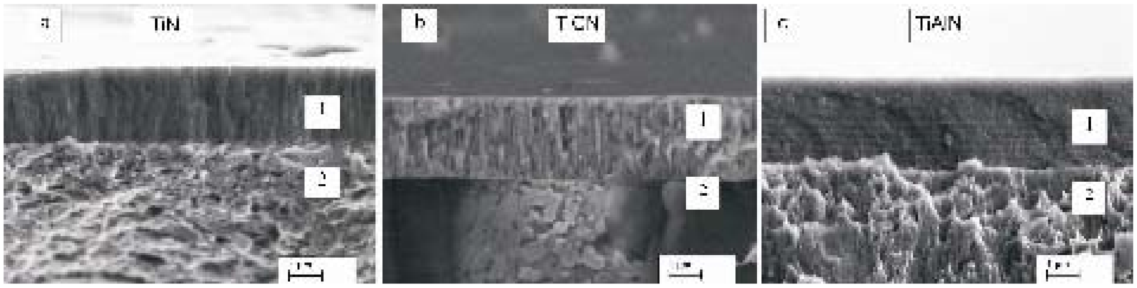

Figure 2. SEM images of fractured surface of PVD coatings on nitrided steel: a– monolayer TiN, b– monolayer TiCN and c– multilayer TiAlN.

From HVOF sprayed coatings WC–17Co hardmetal coating (Tafa 1343) sprayed by Tafa JP 5000 on substrate steel C45 was studied. Laser hardening of coated specimens using the Haas HL 4006 D 4 kW Nd: YAG laser was performed.[6] Vickers micohardness of coating was measured using 0.1 – 0.3 N load. The microstructure of HVOF sprayed and laser hardened coating is shown on Figure 3b.

Table 3. Studied HVOF sprayed coatings

Type of coating	Substrate	LHZ 1) / coating thickness, µm	Hardness HV0.2, GPa 2)
WC–17Co	C45, normalized	– / 200	2.0 / 13.4
(Tafa 1343)	C45 laser hardened	180 / 200	2.0 – 4.1 / 7.0 –10.8

1) LHZ – laser hardened zone
2) LHZ hardness / coating hardness

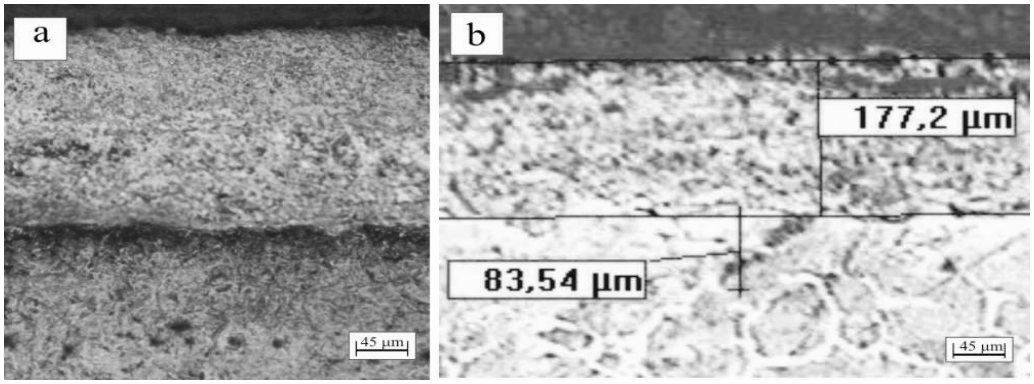

Figure 3. Microstructure of cross-section of HVOF sprayed WC–17Co coating on steel C45: a – as sprayed, b – laser hardened.

2.2 Adhesion and Surface Fatigue Testing

A Rockwell adhesion test (Rockwell "C" indentation test) method was used in order to verify the adhesion quality of the coatings. Tests were performed on hardnessmeter Zwick 8150 at load of 1471 N (150 kgf). Indentations were made in a direction perpendicular to the specimen surface. The indented samples were then analysed with an optical microscope and results were classified into the categories given in the CEN/TS 1071 – 8 standard.[7]

A single point indentation method was used to evaluate the surface fatigue of the coatings. In the indentation experiments Vickers diamond pyramid indenter and Instron 8800 servohydraulic fatigue test system were exploited (Figure 4). The number of indentation cycles varied from 1 up to 10 000 and total indenting load of 100 and 500 N with stress ratio R = 0.1, sinusoidal loading pattern and loading frequency of 0.5 – 15 Hz was applied. The optical microscope with 500x magnification and Buehler Omnimet Image Analysis System 5.40 including the package for crack length measurement by Palmqvist method was used.[8]

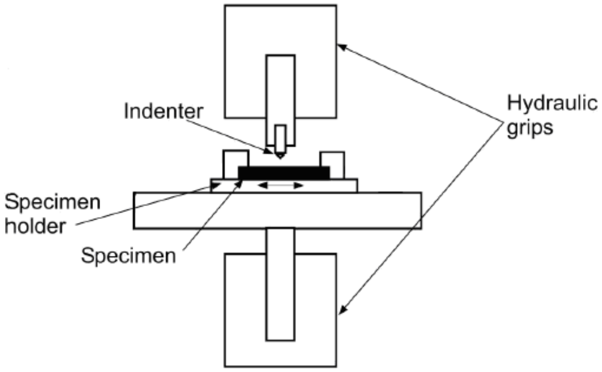

Figure 4. The schematic of surface fatigue tester.

2.3 Impact Wear Testing

The impact wear testing was performed on impact wear tester (Figure 5) designed and produced at TUT. The special design using ball indenters up to 30 mm in diameter enables to study the behaviour of coatings under conditions of dynamic compressive stresses (flat-to-flat contact) that are characteristic to blanking. The dynamic load is transferred from the hammers that are connected to, and accelerated by the rotating disk. The transferred energy depends on speed and mass of hammers and can be adjusted. Hammers fastened on the periphery of the disk allow increasing the frequency at least up to 100 Hz. To minimize the wear of moving parts and its effect on the change of contact conditions during testing the arm type construction was applied. The force measurement system (force sensor) with the protection from overloading was used for monitoring of the contact parameters. Applied force was equal to 16 N, the speed of hammer at the impact point was 2.75 m/s. The failure of the surface was characterized by the ratio *FR* (ratio of the highly damaged area to the overall contact area).[9]

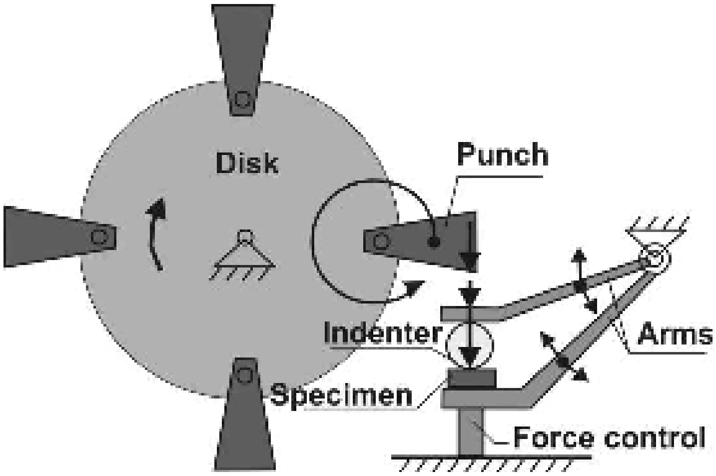

Figure 5. Schematic of impact wear tester.

3 RESULTS AND DISCUSSION

3.1 Hardness of Coating Systems

Microhardness values of the substrates measured using microhardness method and Vickers pyramid at load of 0.1 – 3 N varied from 1500 HV0.3 (by hardmetals) up to 840 HV0.01 (by tool and nitrided steels).

Hardness distribution of PVD coated nitrided steel given in Figure 6 shows that the core microhardness of the plasma nitrided substrate is 350 HV0.01 and the microhardness of the nitrided surface, having case depth of 300 μm, is around 850 HV0.01 *e.g*. the surface hardness is increased by a factor of 2.4.

The hardness distribution in HVOF sprayed WC-Co coated sample, brought in Figure 7, shows that laser treatment does not affect the microhardness of the coating, which stays at the level of the one of the sprayed coating. The microhardness of the laser treated coating ranges from 750 HV0.2 to 1060 HV0.2, whereas no clear influence of the laser treatment can be tracked. The microhardness of substrate steel C45 stays on the level of normalized steel.

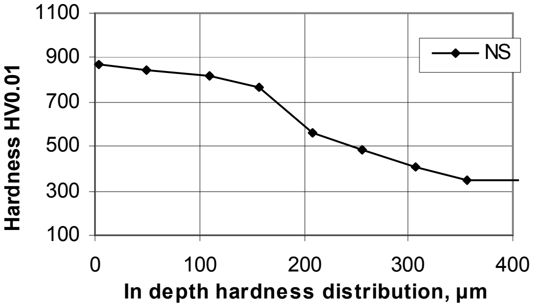

Figure 6. The hardness distribution of nitrided steel (NS) substrate (38CrMoAl8).

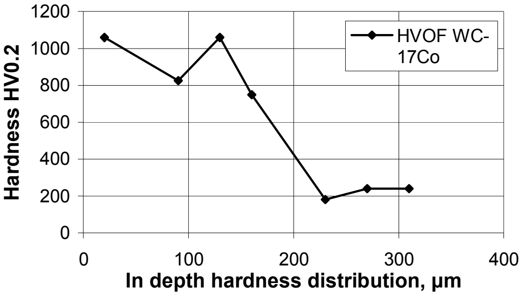

Figure 7. The hardness distribution in laser treated HVOF sprayed WC-Co samples.

3.2 Adhesion Evaluated by Rockwell C-scale Test

The results of the indentation test of PVD coatings are presented in Figures 8 and 9. The type and the volume of a failure zone indicate to coating adhesion and its brittleness, which correspond to the microstructure and the mechanical properties of the coatings. Coatings of higher hardness and lower Young's modulus (higher *H/E* ratio, TiAlN) withstand the load without adhesive delamination of the coating, but with nucleation of long conical cracks. However, considerable amount of long radial cracks of 100 – 150 µm were generated, causing the partial adhesive delamination of coating that is a typical behavior of TiN coating under loading (Figures 8a and 9a).[9] The same features are seen with larger magnification in the case of TiAlN coating with only difference – short radial cracks accelerate chipping on the bordering area of coating-indenter contact (Figure 8b and 9b). Emerged chips tend to make connections with the closest on the sides forming the ring or conical crack. The size and number of the chips is not considerable.[9] The structural defects presented on the surface, such as pores and non-metallic inclusions, simplify chipping/ delamination action.

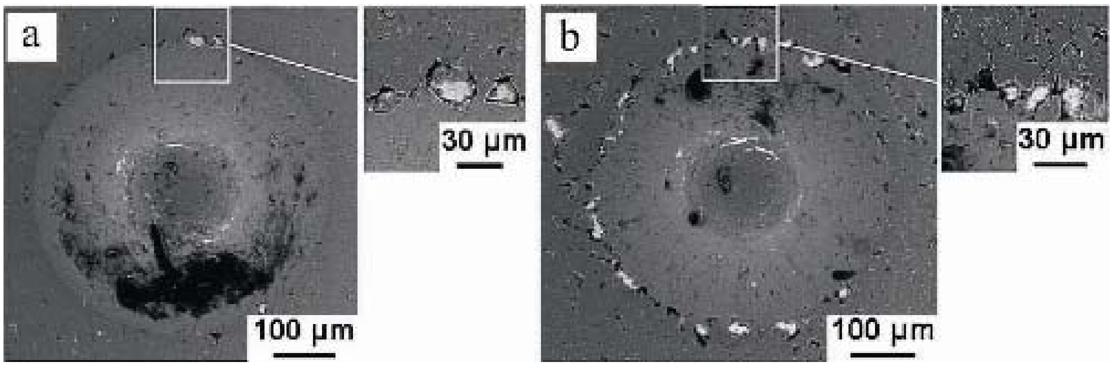

Figure 8. SEM images of the adhesion test of PVD coatings on HM: a – TiN , b – TiAlN.

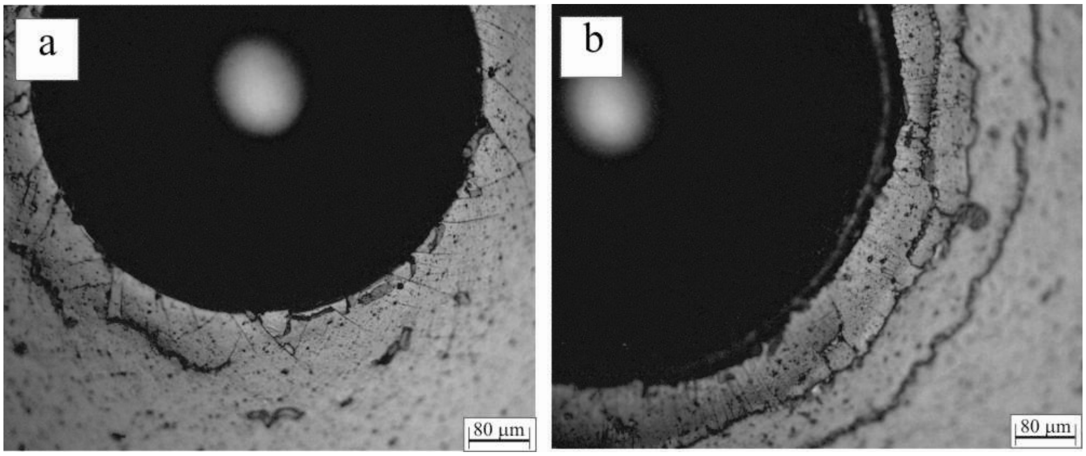

Figure 9. Images of the adhesion test PVD of coatings on NS: a – TiN, b – TiAlN.

3.3 Surface Fatigue Testing by Cyclic Indentation

Fatigue properties of two hard PVD coatings – TiN, TiAlN on nitrided steel and HVOF sprayed coating on steel C45 evaluated by means of the cyclic Vickers indentation method are given in Figures 10 and 11. After the first indentation, mostly contact region model of coating deformation was presented due to indenting load of 100 and 500 N applied.

To estimate the cracking resistance of the coated system, a quantitative analysis of the samples was performed.[8] The results are presented in the Table 4. Tested PVD hard coatings performed similarly up to 100 indentations. At 1000 indentations some perimeter delamination was observed in case of TiAlN in contrary to TiN. This became even more apparent at 10 000 indentations as shown on Figure 10. Although coatings were awarded with similar evaluation, there are remarkable differences (Table 4 remarks).

HVOF sprayed coating exhibited no delamination but cone cracks starting from 100 indentations and radial cracks at 10 000 cycles (Figure 11).

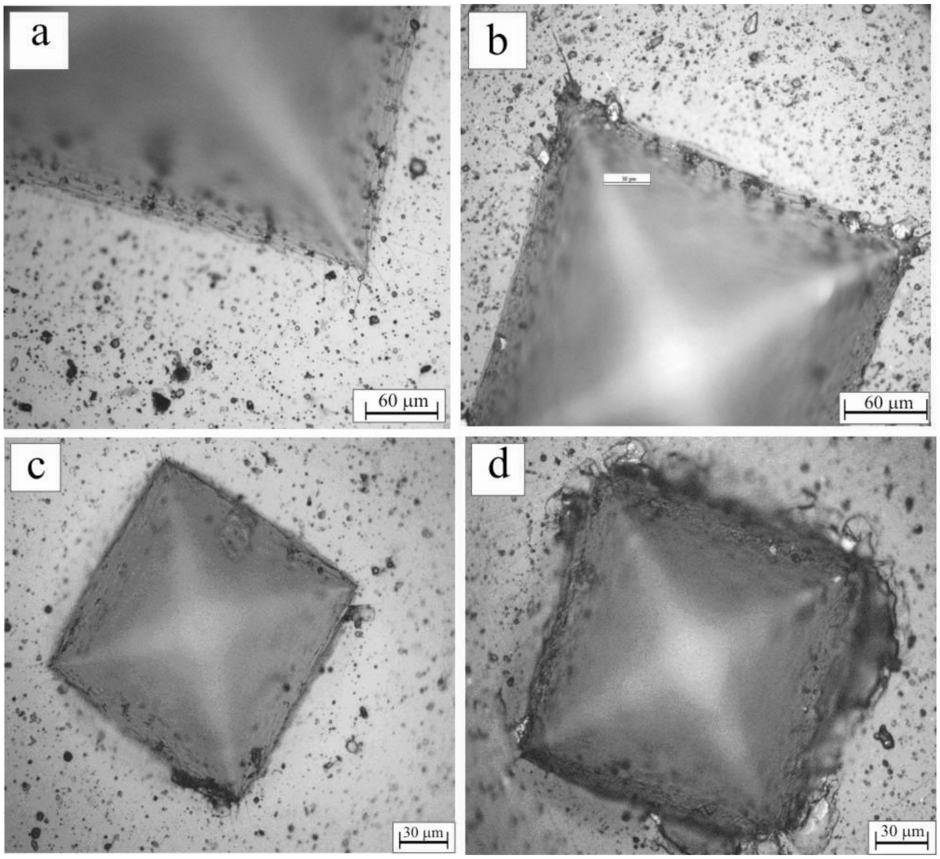

Figure 10. Impressions of PVD coatings on different substrates after 10 000 indentations: a –TiN on CWTS (500 N), b – TiAlN on CWTS (500 N), c – TiN on NS (100 N), d – TiAlN on NS (100 N).

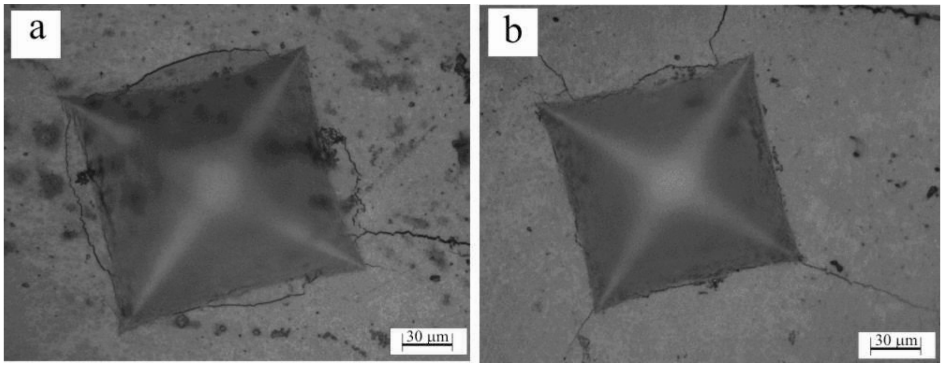

Figure 11. Impression of HVOF sprayed WC–17Co coating on steel C45 after: a – 1000 and b – 10 000 indentations with 100 N load.

Table 4. Evaluation of cracks in studied PVD and HVOF sprayed coatings (evaluation criteria see)[9]

Type of coating and substrate	Indentation cycles				
	1	10	100	1000	10 000
TiN, NS (100 N)	II	II	IV	IV	VI
TiAlN, NS (100 N)	I	I	II	III (some perimeter delamination)	VI (perimeter delamination)
WC-17Co (100 N)	0	V (weak cone cracks, no delamination)	V (weak cone cracks, no delamination)	V, (no delamination)	V, (no delamination, strong RC)

3.4 Impact Wear Resistance

In the current study, the loading under the surface fatigue testing was limited by $1 \times 10^6 - 1 \times 10^7$ cycles since it is often used by authors active in the area of surface fatigue study of PVD coatings[10-12] and since during that number of cycles most of the characteristic fatigue failure modes take place.[13] The extent of failure around the imprint is described through the ratio *FR* which is defined as the ratio of the region in which the substrate is revealed (or highly damaged area) versus the overall contact area[6] after conducted loading cycles, see Figure 12. Each point represents the average value of 3 tests at specified conditions. The scatter of results is in the range of ±10 %.

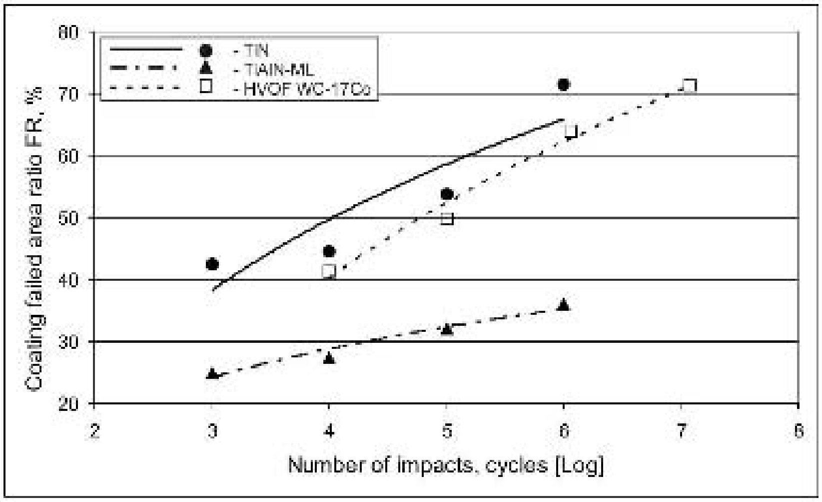

Figure 12. Coating failure development diagram for normal impact under 80 mJ impacts.

High values of *FR* warn of a high risk of coating–substrate adhesive failure mechanisms. The lines on the plot in Figure 12 show the effect of loading cycles number on fracture propagation dynamics. Analogical inference can be marked up between the fatigue curve slopes on the Wöhler plots (fatigue sensitivity) and the appearance of lines on the coating failed area ratio vs number of impacts graph. The steep slope of the curves for TiN and HVOF WC–17Co coatings are the evidence of drastic damage during cyclic loading. These coatings have showed very similar behaviour and sufficiently lower resistance to impact demonstrating the *FR* values up to 2 times higher than that of TiAlN coating at all range of applied cycles. On the contrary, the flat slope for TiAlN indicate better fracture resistance with increase in cycles number

With an increase in the load and number of impact cycles and growth of the imprint area, the dynamic load distributes over the larger area reducing the contact pressure. The *FR* ratio of multilayer TiAlN coating at $10^4 - 10^6$ is stabilized and further increase is not observed. In addition to the base TiAlN gradient layer, this coating has the TiN layer on the top of Ti interlayer. In this system the gradient nature of the top layer and presence of more ductile additional layer provide better performance in impact conditions.

For characterization of resistance to plastic deformation recently new assessment criteria for hard coatings was proposed – resistance of coating system to plastic deformation H^3/E^2.[14] By our case the *H* and *E* are hardness and modulus of elasticity of substrate. It was demonstrated that coating systems with higher H^3/E^2 parameter of substrate had better resistance to the plastic deformation.

4 CONCLUSIONS

In the result of adhesion tests among the tested PVD coatings TiN coatings seems to be most endurable. The mixed failure modes are characteristic, presented are chipping, delamination with buckling and fracture.

In the result of indentation cyclic tests the “quasi-plastic” damage mode with formation of radial cracks prevailed and is typical for high H/E values of coatings. The increase of the number of cycles leads to the radial crack growth. “Brittle” damage with formation of cone cracks as characteristic of the PVD coatings with lowest H/E ratio. Surface fatigue resistance of HVOF sprayed WC-Co coating is low due to the relatively low hardness of substrate.

To assure better resistance of coating systems to plastic deformation and impact wear at high loads the parameter H^3/E^2 must be maximized.

Multilayer coatings had higher impact wear resistance than monolayer coatings with lower H/E ratio.

Acknowledgements

This work was supported by the Estonian Ministry of Education and Research (project SF 0140091s08 and grant ETF 7889).

REFERENCES

1 VDI 3198:1992-08, Coating (CVD, PVD) of Cold Forging Tools. VDI-Verlag, Düsseldorf1991.
2 Handbook of Hard Coatings, G. E. McGuire, S.M., Rossnagel, R., F., Bunshah, Noyes Pub. 2001.
3 SACHER, G., ZENKER, R. Subsequent heat treatment of hard coated steels by electron on laser bean, German – Russian Workshop “Tribology and Surface Engineering: theory, experiment, technologies”, Berlin University of Technology, 28 – 30 March, 2007.
4 ZOESTBERGEN, E. AND DE HOSSON, J. TH. M. Crack resistance of PVD coatings: Influence of surface treatment prior to deposition. *Surf. Eng*., 2002, 18, 283 – 288.
5 KARNEVA, U., LABERBOM, J. AND VUORISTO, P. Development of thermal spary powders for improved tribological and corrosive applications and cost-effecive solutions. *Int. J. of Materials and Product Technology*, 2007, 28, 377–398.
6 Surzenkov, A., Kulu, P., Tarbe, R., Mikli, V., Sarjas, H., Latokartano, J., Wear resistance of laser remelted thermally sprayed coatings, *Estonian Journal of Engineering*, 2009, 15, 4, 318 – 328.
7 CEN/TS 1071–8, Advanced technical ceramics – Methods of test for ceramic coatings – Part 8: Rockwell indentation test for evaluation of adhesion.
8 PALMQVIST, S. The work for the formation of a crack during Vickers indentation as a measure of the toughness of hard metals. *Arch. Eisenhüttenwes*, 1962, 33, 629 – 634.
9 SIVITSKI, A., GREGOR, A., SAARNA, M., KULU, P., SERGEJEV, F. Application of the indentation method for cracking resistance evaluation of hard coatings of tool steels, *Estonian Journal of Engineering*, 2009, 15, 4, 309 – 317.
10 BOUZAKIS, K.-D., SIGANOS, A., Fracture initiation mechanisms of thin hard coatings during the impact test, *Surf. Coat. Tech*. 2004, 185, 150 –159.

11 BOUZAKIS, K.-D. et al., The influence of the coating thickness on its strength properties and on the milling performance of PVD coated inserts, *Surf. Coat. Tech*. 2003, 174 –175, 393 – 401.
12 BOUZAKIS, K.-D. et al., Thin hard coatings fracture propagation during the impact test, *Thin Solid Films*, 2004, 460, 181 –189.
13 MURAKAMI, Y., Metal Fatigue: Effects of Small Defects and Nonmetallic Inclusions, Elsevier Science Ltd., UK, 2002.
14 MUSIL, J., et al. Relationships between hardness, Young`s modulus and elastic recovery in hard nanocomposite coatings, *Surf. Coat. Tech*., 2002, 154, 304 – 313.