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# THE INFLUENCE OF THE MULTILAYER STRUCTURE OF HARD COATINGS ON THEIR RESISTANCE TO MICRO-IMPACT FATIGUE WEAR

# WPŁYW WIELOWARSTWOWEJ BUDOWY TWARDYCH POWŁOK NA ICH ODPORNOŚĆ NA MIKROUDAROWE ZUŻYCIE ZMĘCZENIOWE

**Key words:** hard coating, micro-impact, fatigue wear, crater evolution.

**Abstract:** Fatigue cracking of thin hard anti-wear coatings occurs, among others, in the tribological contact of sliding friction pairs, in the top layers of cutting tools coatings, as well as in the surface of elements subjected to erosion processes. Coating fatigue wear is initiated as a result of cyclic interactions with micro-roughness of counterpart or other elements or particles that repeatedly impact the surface. The selection of appropriate coatings can increase the durability of machine components that are subjected to fatigue impact loads. The paper presents the results of tests on micro-impact fatigue wear of elements covered with single TiN and DLC coatings, as well as multi-layer (Ti/TiN)×8 type. Fatigue tests were carried out using the micro-impact method by cyclic impact of the surface of the coating with a diamond ball. The experiments were performed using a special laboratory stand. The correlation between fatigue life of coatings and their micromechanical properties such as Young's modulus and hardness were also examined. The hardness and Young's modulus were determined by an instrumented indentation method. The test results proved that the (Ti/TiN)×8 multilayer coating demonstrates wear 1.4 times smaller than the sample with the TiN coating and 1.2 than the sample with the DLC coating.

**Słowa kluczowe:** twarda powłoka, mikroudar, zużycie zmęczeniowe, ewolucja krateru.

**Streszczenie:** Pękanie zmęczeniowe cienkich twardych powłok przeciwzużyciowych występuje m.in. w styku tribologicznym ślizgowych węzłów tarcia, w warstwach wierzchnich pokryć narzędzi skrawających, a także w powierzchni elementów poddanych procesom erozyjnym. Zużycie zmęczeniowe powłok inicjowane jest na skutek cyklicznych interakcji z mikronierównościami przeciwelementu lub innymi elementami bądź cząstkami, które wielokrotnie uderzają w powierzchnie. Dobór odpowiednich powłok może zwiększyć trwałość elementów maszyn, które poddane są obciążeniu udarowemu o charakterze zmęczeniowym. W pracy przedstawiono wyniki badań odporności na mikroudarowe zużycie zmęczeniowe elementów pokrytych powłokami pojedynczymi TiN oraz DLC, a także wielowarstwowymi typu (Ti/TiN)×8. Testy zmęczeniowe przeprowadzono metodą mikroudarową poprzez cykliczne uderzanie w powierzchnię powłoki kulą ceramiczną. Eksperymenty wykonano przy zastosowaniu specjalnego stanowiska pomiarowego. Zbadano również występowanie korelacji pomiędzy trwałością zmęczeniową powłok i ich właściwościami mikromechnicznymi takimi jak moduł Younga oraz twardość, które wyznaczono statyczną metodą wciskania wgłębnika. Wyniki badań dowiodły, że wielowarstwowa powłoka (Ti/TiN)×8 wykazuje zużycie 1,4 razy mniejsze od próbki z powłoką TiN oraz blisko 1,2 od tej z powłoką DLC.

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## INTRODUCTION

Due to the continuous increase application field of anti-wear coatings, it is necessary to investigate their properties under specific conditions. This requires the design of new test equipment, including that intended for testing the resistance to cracking of coatings under impact fatigue **[L. 1–3]**. In these tests, the ball-shaped indenter, which is most common, impacts the surface of the coating cyclically. Tests are run using a device in a vertical arrangement in which the indenter makes a vertical movement and impacts the flat surface of the coating perpendicular to it. Another solution is to provide the indenter horizontal movement in a direction perpendicular to the flat surface of the sample **[L. 4]**. In the Laboratory of Tribology and Surface Engineering of AGH in Krakow, the investigations of the coatings strength against micro-impact surface fatigue are carried out using a device operating in a horizontal system.

The process of coatings cracking is a complex issue, and its recognition can reduce the wear of coatings and prevent serious breakdowns of machines or devices. Currently, a lot of research is being carried out to describe this phenomenon in detail for specific materials, but primarily to determine the durability of the coatings due to cyclic loading **[L. 4]**.

The article attempts to assess the durability of selected thin coatings subjected to cycle impact-fatigue load. This type of coating wear, caused by repetitive impacts, can be observed in the top layer of cold working forming tools, cutting tools, or, in case of the dynamic impact of surface micro-roughness, e.g., in sliding friction pairs. The effect of long-term work of these elements is the accumulation of micro cracks caused by the stress field and fatigue of the coating, which leads to plastic deformation of the substrate as well as crumbling and chipping of the coatings' layers.

The carried out research was aimed to determinate the impact of the multilayer structure of Ti/TiN coatings on their resistance to micro-impact fatigue wear compared to single coatings TiN as well as DLC.

## TESTED MATERIALS

The subject of the investigation were hard coatings: titanium nitride (TiN), multilayer titanium/titanium nitride ((Ti/TiN)×8), and carbon coating type of DLC (Diamond Like Carbon). The coatings were deposited on X10CrNi18-8 austenitic steel by the PLD method (Pulsed Laser Deposition). The TiN coating was applied as a single layer with a thickness of 2 μm. The multilayer (Ti/TiN)×8 coating with a thickness of 1 μm consisted of 8 alternately layers of Ti and TiN. In the comparative analysis, a DLC coating 1 μm thick was also used.

## TEST METHOD

The main part of the experiment was tests of coating resistance to micro-impact fatigue carried out on the Impact Tester device. The device allows one to perform tests consisting of cyclic impact of the sample surface with an indenter with a given force. **Figure 1** shows

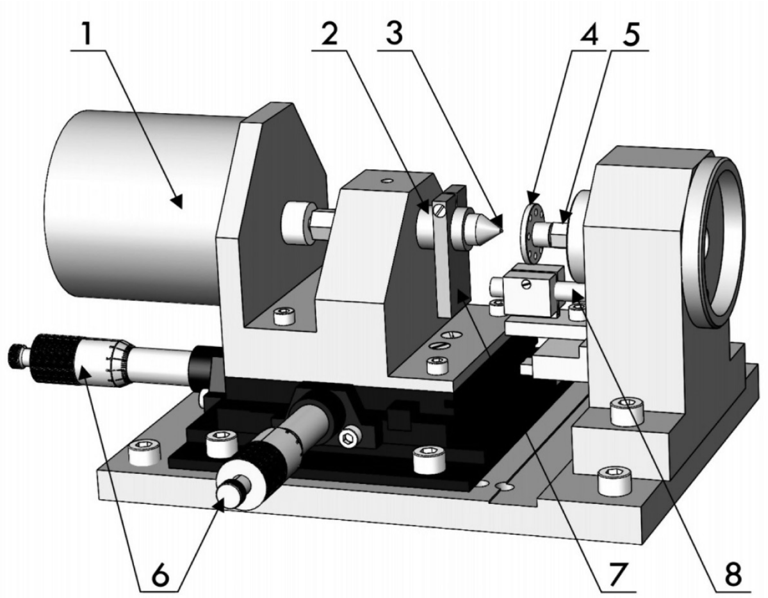


**Fig. 1. Impact Tester scheme: 1 – inductor, 2 – head, 3 – indenter, 4 – sample holder, 5 – force sensor, 6 – micrometer screws, 7 – mirror, 8 – optical displacement sensor**

Rys. 1. Schemat Impact Testera: 1 – wzbudnik, 2 – głowica, 3 – wgłębnik, 4 – uchwyt na próbkę, 5 – czujnik siły, 6 – śruby mikrometryczne, 7 – zwierciadło, 8 – czujnik przemieszczenia the scheme of the Impact Tester device. It is built of an inductor (1), attached to a head (2) which perform reciprocating motion. The essential element of the head is an indenter (3) ended with a spherical working element, most often diamond. The indenter holder has been prepared so that the working element can be changed if tests require using a tip made of a different material or of a different size and shape. The force sensor (5) is used to measure the impact force of the indenter on the sample mounted in the fixed holder (4). The optical displacement sensor (8) together with the mirror (7) allows one to measure the position of the indenter relative to the sample. In addition, the device is equipped with two micrometre screws (6), which allow positioning the body, including the inductor and head, and set the appropriate position of the indenter relative to the sample, as well as adjusting the distance of the indenter relative to the sample depending on the thickness of the tested material **[L. 5].**

In addition to the working parts described above, the device is equipped with a measurement and regulation system for device control and measurement data acquisition, the schematic diagram of which is shown in **Fig. 2**. The generated inducement signal is amplified and provides power of the inductor **(pos. 1 Fig. 1)**. Signals from displacement and force measurement systems **(pos. 5 and 8, Fig. 1)** are sent to the oscilloscope, which is triggered by a signal from the inducement generator. This allows the registration of the force and displacement from a value equal to zero. The values measured with an oscilloscope are sent to a computer that saves data. It is possible to control the inducement signal generator using a suitable computer program, which increases the stability of the given force. The forcing signal generator allows one to control the signal in such a way as to avoid repeated impact and reflection of the indenter from the sample in one cycle. This behaviour would lead to an overstated number of load cycles and, as a result, to make test results unreliable.

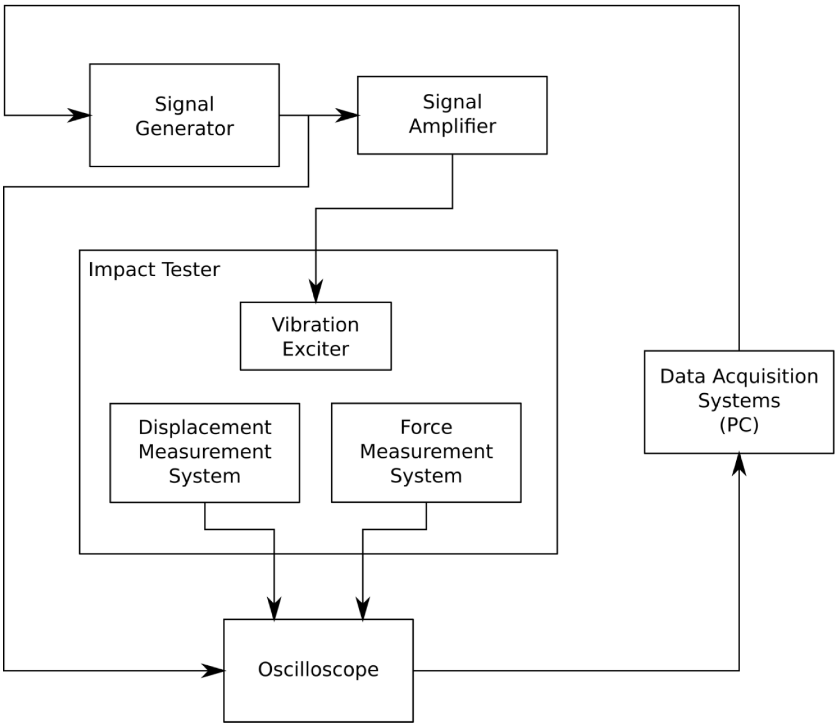


**Fig. 2. Schematic diagram of the control and measurement system for Impact Tester device**
Rys. 2. Schemat ideowy układu kontrolno-pomiarowego dla stanowiska Impact Tester

Fatigue strength tests were carried out with the following parameters:

- Load of indenter: 4 N,
- Indenter shape: spherical,
- Indenter radius: 0,2 mm,
- Indenter material: diamond,
- Inducement signal: sinusoidal.

The numbers of load cycles that were realized during the tests are summarized in the **Table 1.**

**Table 1. Tests plan of micro-impact fatigue of the coatings**
Tabela 1. Plan badań mikroudarowego zmęczenia powłok

| Coating | Single indentation (static load) | 500 cycles | 1000 cycles | 2000 cycles | 4000 cycles | 5000 cycles | 200 000 cycles |
|---|---|---|---|---|---|---|---|
| **(Ti/TiN)×8** | × | × | × | × | × | × | × |
| **TiN** | × | × | × | × | × | × | – |
| **DLC** | × | × | × | × | × | × | – |

As the criterion of coating durability, the number of impacts with a constant force (cycles) at which the coating has detached from the substrate in the central part of the crater was adopted. The measurement of the wear of the coating-substrate system was assumed as the cross-sectional area of the crater formed as a result of the impact of the indenter. This area was determined based on the measurements of the geometrical dimensions of the crater which were made using a Profilm 3D contactless company Filmetrics, USA. Micro-mechanical properties of the coatings were also tested using a CSM Instruments Micro-Combi-Tester (MCT). The research included indentation tests with a Vickers diamond indenter **[L. 6]**. On the basis of indentation measurements by the use an instrumental indentation method, the hardness and modulus of elasticity of the coatings were determined using the well-known Oliver and Pharr model **[L. 6]**. Six replicates were performed for each sample at a load of 10 mN.

## RESULTS

Firstly, the micromechanical properties were investigated. **Figure 3** shows the determined hardness and modulus of elasticity of the tested coatings.

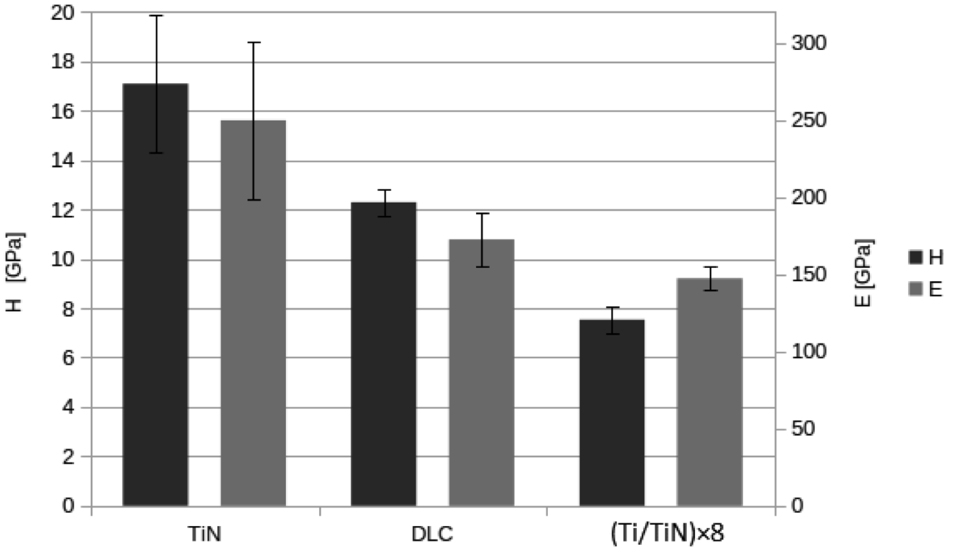


**Fig. 3. Hardness (H) and modulus of elasticity (E) of the tested coatings**
Rys. 3. Twardość (H) i moduły sprężystości (E) badanych próbek

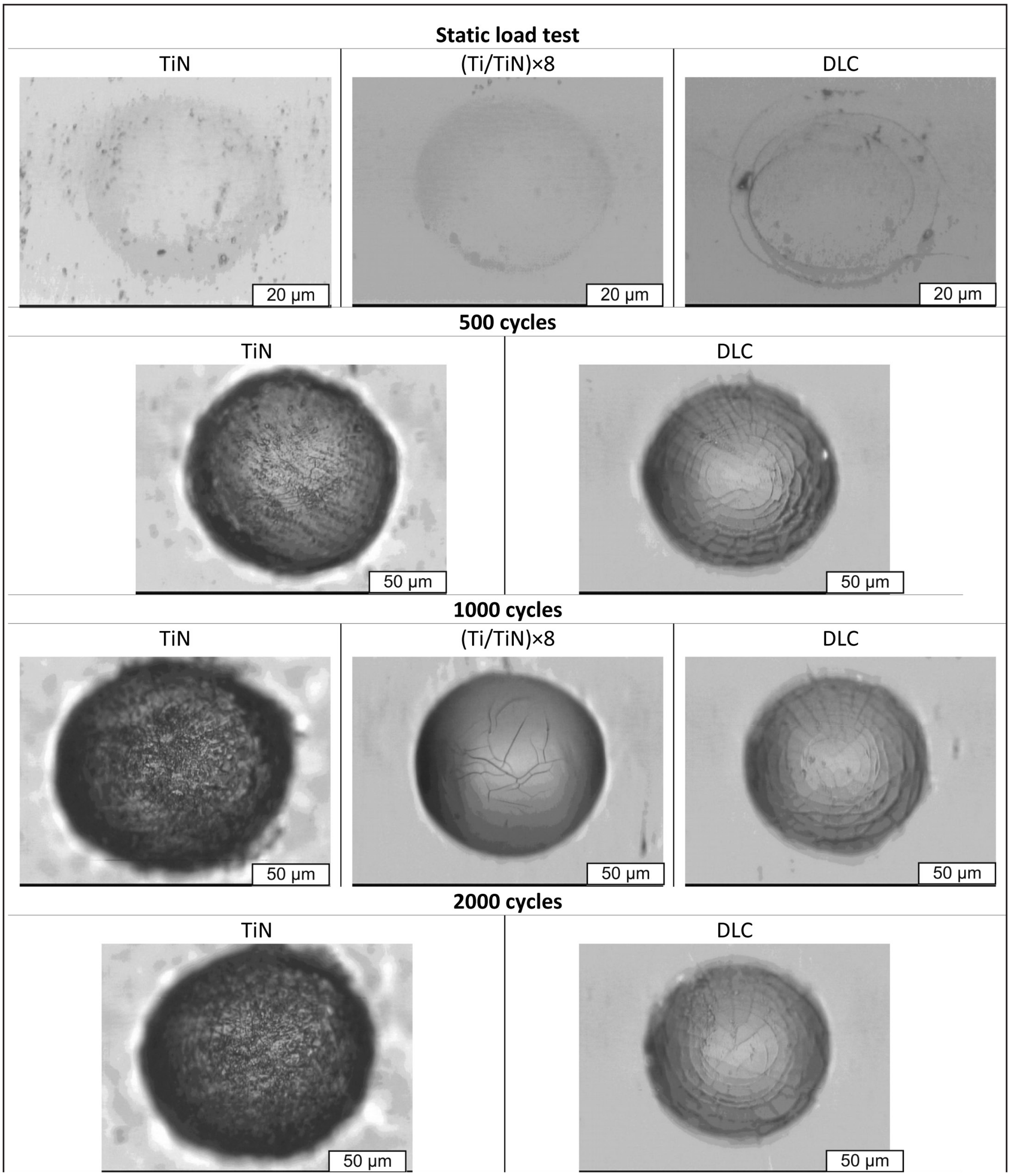
The TiN coating exhibited the highest hardness and modulus of elasticity, which were 17.1±2,8 GPa and 249.8±51 GPa, respectively. The deposition of the (Ti/TiN)×8 coating in a multilayer system resulted in a significant reduction in hardness to the level of 7.5 ± 0.6 GPa and elastic modulus to 147.2 ± 7.6 GPa. In contrast, the DLC coating showed intermediate values of 12.3 ± 0.5 for hardness and 172.5 ± 17.1 GPa for the modulus of elasticity.

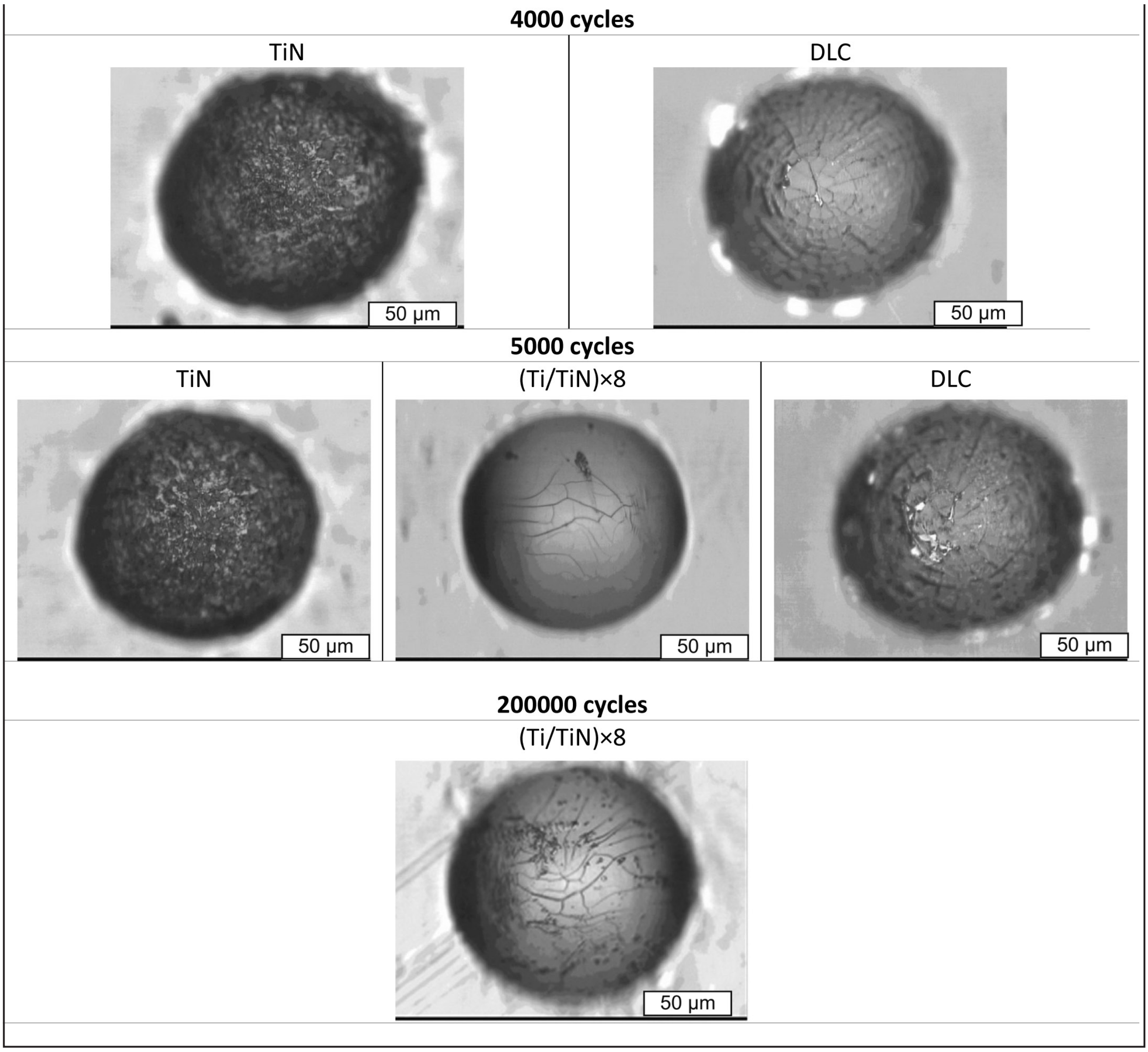
After fatigue tests, the surface quality of the coatings in the crater site was observed using a light microscope. In **Table 2,** the images of craters of individual coatings after a specified number of cycles were collated.

Images of the surfaces craters formed in coatings after a certain number of impacts.

Analysis of the craters obtained after static load indicate that a pronounced cracks were formed only in a DLC coating. After 500 cycles of impacts, some cracks in TiN and DLC coatings appeared on the entire surface of the crater bottom, thus in the place of direct contact of the indenter with the coating. In the range of up to 500 cycles of impacts, no detachment of the coating from the substrate was found. For the TiN coating, each subsequent test at 1000, 2000, 4000, and 5000 cycles caused its destruction, according to the adopted criterion. The TiN coating partly broke off and crumbled, and its fragments were driven into the substrate. DLC coating, after tests of 500, 1000, and 2000 cycles of impacts, had a similar wear mechanism as in previously observed ones, i.e. cracks occurred in the coating but pieces do not chipped off. After 4000 cycles, the DLC coating material detached from the substrate. An increase in the

**Table 2. Images of the surfaces craters' formed in coatings after a certain number of cycles**
Tabela 2. Obrazy powierzchni kraterów powstałych w powłokach po określonej liczbie cykli





number of cycles up to 5000 produced the progress in the crack and detachment process of the coating.

The (Ti/TiN)×8 multilayer coating showed the highest resistance to micro-impact fatigue. A few small cracks in this coating being the result of the cyclic impact of the indenter were observed only after 1000 load cycles – much later than for harder TiN and DLC coatings. An increase in the number of cycles obviously increases the number of cracks, but even after 5,000 cycles, their amount was comparatively marginally. A network of micro cracks appeared in the coating over the entire surface of contact with the indenter but without being detached coating from the substrate. An example of a three-dimensional image of image of the sample surface after impact tests and the cross-sectional profile of the crater are shown in **Fig. 4**, which was obtained from the Profilm 3D device.

Thanks to the measurements of crater geometry, it was possible to determine a depth of the craters formed during fatigue tests **(Figs. 4–6).**

Coating wear was determined by the crater's cross-sectional area. In the single indentation at static load, wear was many times smaller than the wear determined after tests with a greater number of cycles at dynamic load. Analysing the results of these tests **(Fig. 5)**, it can be concluded that the wear of the TiN coating after 500 and 1000 cycles was similar. Comparable cross-sectional sizes of craters were also found after 2000, 4000, and 5000 cycles, and the profiles of the craters cross-sections do not differ significantly. Each subsequent fatigue test deepens the crater of the sample with (Ti/TiN)×8 multilayer coating, and, unless its diameter does not change significantly, the depth increases for each impact sequence, which is illustrated in **Figure 6**.

The DLC fatigue wear resistance is higher than for TiN. This is evidenced by the measurement results in **Tab. 3** and **Figs. 6–7**. The irregular shape of the profiles indicates that parts of the chipped coating have deposited on the walls of the crater. Based on the crater profiles, it was possible to calculate their cross-sectional areas

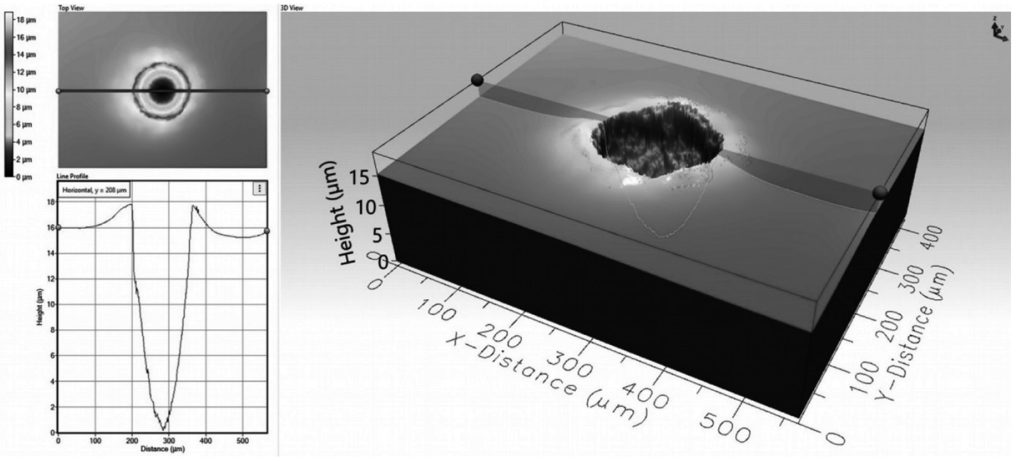


**Fig. 4. 3D image of the surface of sample with TiN coating after 5000 cycles**
Rys. 4. Widok powierzchni próbki z powłoką TiN po 5000 cyklach

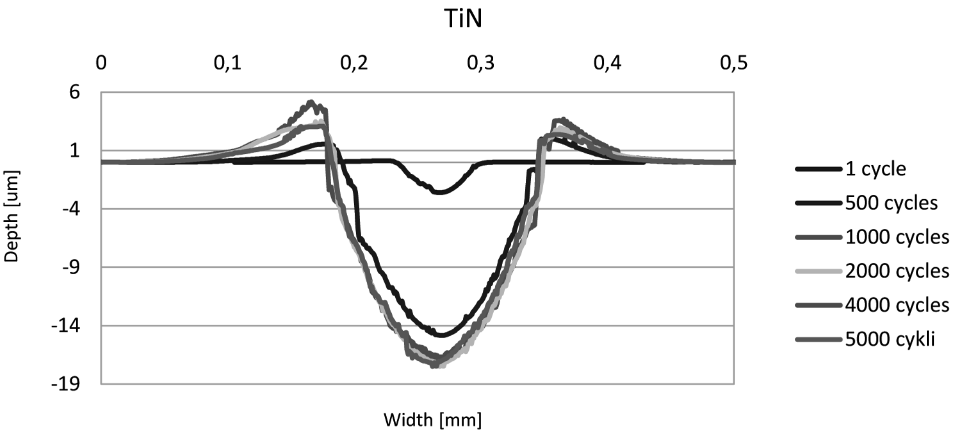


**Fig. 5. Crater mid-section profiles for a TiN-coated sample**
Rys. 5. Profile przekroju środkowego kraterów dla próbki z powłoką TiN

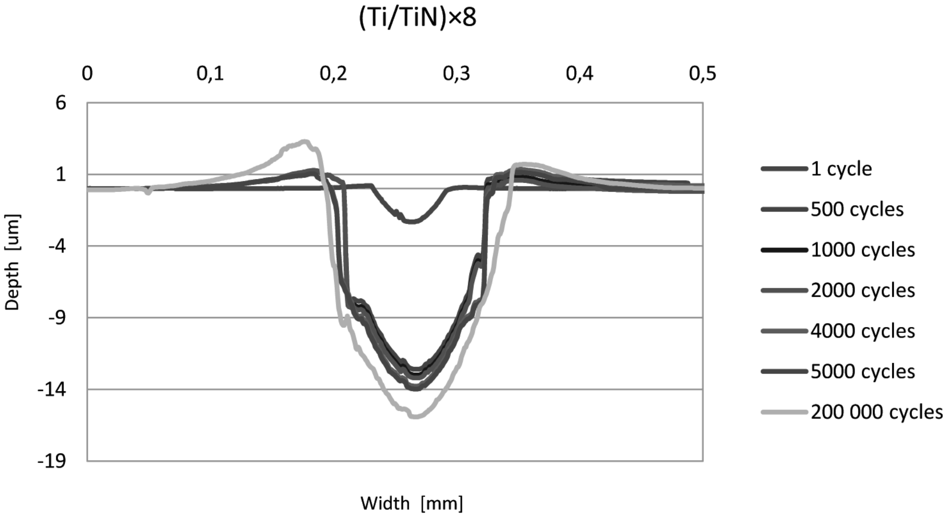


**Fig. 6. Crater mid-section profiles for a (Ti/TiN)×8-coated sample**
Rys. 6. Profile przekroju środkowego kraterów dla próbki z powłoką (Ti/TiN)×8

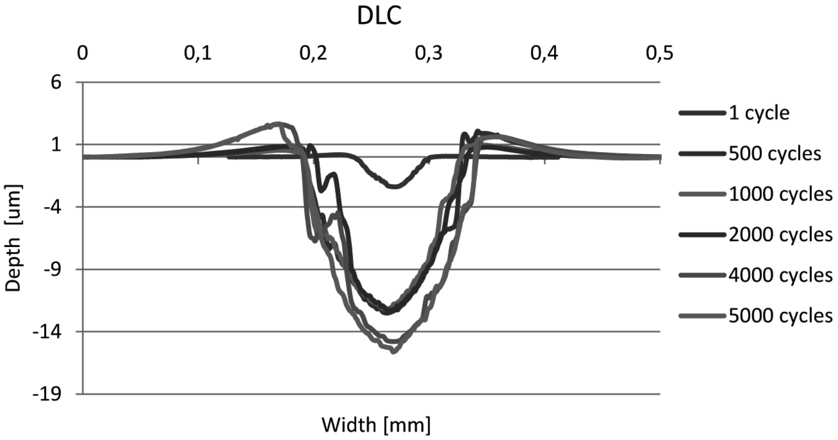


**Fig. 7. Crater mid-section profiles for a DLC-coated sample**
Rys. 7. Profile przekroju środkowego kraterów dla próbki z powłoką DLC

for each sample after static loading, as well as 1000 and 5000 cycles and 200000 cycles for (Ti/TiN)×8. The results presented in **Tab. 3** indicate that the wear of the (Ti/TiN)×8 multilayer coating was the smallest and the single TiN was the the largest.

**Table 3. Values of crater cross-sectional area ($A_C$) and standard deviation (SD) for static load and for selected cyclic impact loads**
Tabla 3. Pole powierzchni przekroju środkowego kraterów podane w µm$^2$ dla obciążenia statycznego oraz wybranych cyklicznych obciążeń udarowych

| Number of cycles | TiN | | (Ti/TiN)×8 | | DLC | |
|---|---|---|---|---|---|---|
| | $A_C$ [µm$^2$] | SD | $A_C$ [µm$^2$] | SD | $A_C$ [µm$^2$] | SD |
| **Single indentation** | 103 | 8 | 89 | 7 | 92 | 5 |
| **1000** | 1 578 | 59 | 1 046 | 12 | 1 102 | 21 |
| **5000** | 1 929 | 48 | 1 316 | 20 | 1 549 | 45 |
| **100000** | × | × | 1600 | 28 | × | × |

## SUMMARY

The applied test method allowed determining the resistance to micro-impact fatigue wear of the coatings. The cyclic impact load of the hard coatings, which were studied in this work, caused their brittle fracture mainly in the crater area. The (Ti/TiN)×8 coating with a multilayer structure showed the highest resistance to fatigue wear. Compared with 56% and 39% harder single TiN and DLC coatings, (Ti/TiN)×8 coating shows much higher fracture toughness. The lower wear of the multilayer coating was found for each number of cycles, not only qualitatively through microscopic observations of the crater surfaces, but also quantitatively, by comparing the cross-sectional areas of the middle craters. The cross-sectional area of the (Ti/TiN) × 8 coating after 5,000 cycles was 1.4 times smaller than the crater cross-section for the sample with the TiN coating and close to 1.2 than that with the DLC coating. Introduced the metallic titanium interlayers in the (Ti/TiN)×8 coating accumulate cracking energy and block the propagation of these cracks, which has a positive effect on increasing the resistance to micro-impact surface fatigue of the multilayer coating. The research allows us to state unequivocally that, in places where impact load is forecasted, it is advisable to use multi-layer coatings, whose structure limits crack propagation.

## ACKNOWLEDGEMENTS

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