Effect of loading conditions on the scratching behaviour of a TiBN and different DLC top-layered coatings applied on X210Cr12 cold work tool steel

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Abstract

Coatings and coated tools are more and more commonly used in the automotive industry due to their performance-increasing ability. During their application, they have to perform good tribological resistance, including scratch resistance, which may be influenced by many factors such as loading force, type of coating, etc. In the current research work, we investigated the scratching behaviour of four different industrially applied wear-resistant coatings – TiBN, TiBN+DLC, TiAlN+DLC, and CrN+DLC – under different constant loadings of F = 10, 15, 50, and 100 N. Based on the obtained results, we established that the monolayer TiBN has a higher resistance to scratch loading than the DLC top-coated systems. Morphological failure analysis revealed that the damage mechanism of the multilayer coatings with different sublayers is dominated by the DLC top layer independently of the type of underlayers.

Keywords: wear-resistant coating, TiBN, DLC, scratch test, tribology

1. Introduction

The automotive industry is at the forefront of technical innovation, requiring constant improvements in manufacturing technologies and materials to meet the always-changing sustainability, efficiency, and performance criteria. Tool steels are essential to the industry's success since they are used in the shaping, forming, and moulding of different automotive parts. However, the durability and effectiveness of these tool steels could be deteriorated by severe operating circumstances, which include high temperatures, abrasion, and corrosive environments. Hard coatings on the tool surfaces may enhance the resistance to wear and corrosion, optimising the frictional behaviour and benefiting the overall durability of tools.

Recent advancements in coating technologies have witnessed the integration of nanomaterials, such as nanostructured carbides and nitrides, further enhancing the hardness and wear resistance of tool steels [1]. Additionally, innovations in deposition technologies, including Physical Vapor Deposition (PVD) [2] and Chemical Vapor Deposition (CVD) [3] or the combination of the two, Plasma Assisted Chemical Vapor Deposition (PACVD) [4], have enabled the precise control of coating thickness and composition, allowing for the optimisation of performance characteristics.

1.1 Monolayered coatings

The automotive industry increasingly implements monolayered coatings on tools because of their exceptional wear resistance, low friction, and outstanding corrosion resistance, which is essential during cutting and shaping. Titanium nitride (TiN), titanium carbonitride (TiCN), aluminium nitride (AlN), aluminium carbonitride (AlCN), etc. coatings have been commonly used in the automotive industry to enhance the performance and durability of tool steels and to improve their ability to withstand corrosion and oxidation, which is vital in the demanding operating conditions of the automotive industry. They are applied to the tool's surface by PVD or CVD processes.

1.2 DLC coatings

Diamond-like carbon coatings are types of amorphous carbon film that have characteristics similar to both diamond and graphite. These coatings consist of carbon atoms hybridised in sp^2 and sp^3 configurations, giving them exceptional hardness, resistance to wear, and, depending on the loading conditions, extremely low friction properties. In addition, they have chemical inertness and exceptional adherence to different substrates. These features render them exceedingly sought-after for automotive applications and are utilised to improve durability, minimise frictional losses, and promote fuel efficiency across diverse components. DLC coatings are well-suited for protecting surfaces exposed to severe circumstances, such as engine components, gearbox parts, and fuel system elements. DLC coatings are typically used in a multilayer rather than a monolayer structure, where an intermediate layer is applied on the substrate below the DLC top layer. The purpose of the intermediate layer is to provide a transition between the substrate's lower hardness and the DLC topcoat's significantly higher hardness.

1.3 Multilayered coatings

The typical structure of the multilayered ceramic coatings comprises several layers of ceramic materials, each with unique qualities customised for the intended application. The primary advance of this architecture consists in decreasing the high gradient of the mechanical and thermal characteristics between the metallic substrate and ceramic coating. The composition of the different layers may consist of oxides, nitrides, and carbides, offering a diverse framework for optimising hardness, thermal conductivity, and chemical resistance. The purposeful arrangement of these layers enhances the general effectiveness of the coating.

2. Experimental work

2.1 Base material

The base material of the tested samples was K100 steel. The EN number of the cold-forming tool steel with a ledeburitic microstructure is X210Cr12, indicating that it's a highly alloyed steel with a 12% chromium content (Table 1.)

Table 1. Chemical composition of K100 tool steel in weight% [5]

C	Si	Mn	Cr	Mo	Ni	V	W	Other
2.00	0.25	0.35	11.50	–	–	–	–	–

Due to the high chromium carbide content, it has good corrosion and wear resistance, which allows it to be used for plastic-forming tools, such as punches and dies, cut-off tools, stamps, pressing and hole flanging tools, or moulds.

2.2 Investigated coatings and test procedure

In the current research work, four different types of coatings, which are commonly used in the automotive industry, have been investigated: a monolayered TiBN coating and three different DLC top-layered coatings, i.e. TiBN+DLC, TiAlN+DLC, CrN+DLC. In the case of each multilayered coating, there is a thin WC transfer layer between the underlayer and the top functional DLC layer.

Instrumented scratch tests with different constant loading forces have been performed to evaluate the tribological performance of the coatings. The basis of ranking the investigated coatings was the critical loading force causing the total delamination of the coating layer while considering the subcritical damage mechanisms, as well. The layer thickness of the coatings was measured by the ball cratering (Calotest) method.

In terms of tribological behaviour, there is a complex relationship between the thickness of the coating and certain characteristics such as residual stress, adhesion, or hardness [6]. The total layer thickness and the thickness of the different underlayers may influence the tribological performance.

During the Calotest method, a ϕ30 mm hardened steel ball was used as a wearing tool, rotating with a speed of 3000 1/min for 3 minutes at each measurement. The diameters of the created craters were then measured using optical microscopy. The measured layer thickness values are compared in Figure 1.

The lowest (4.23 µm) layer thickness was obtained for the single-layer TiBN coating, while the highest (7.91 µm) layer thickness was measured for the TiBN+DLC coating system. The average thickness of the TiAlN+DLC and CrN+DLC coating systems were similar, 6.78 µm and 6.14 µm, respectively. The average thickness of the DLC overlay coatings was 2.96 µm for all coating types. Compared to the DLC top coatings, the thickness of the individual single-layer TiBN coatings was lower by ~1.9-3.7 µm.

The results of the Calotest clearly show that the WC-Co layer is a thin underlayer for all DLC coating systems, which – despite the small thickness – provides the proper adhesion of the DLC and supports it under mechanical stress.

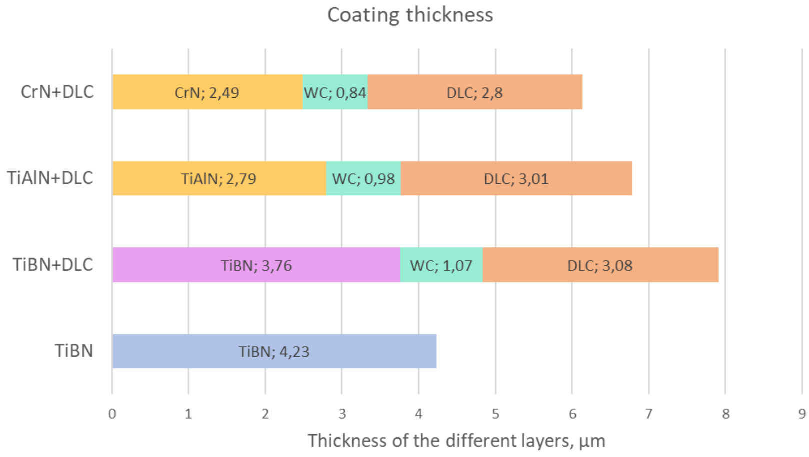


Figure 1. Layer thickness of the tested coatings measured by the ball cratering method

2.3 Constant load scratch test

The most widely used method for testing the adhesion of wear-resistant coatings is scratch testing, which has been used since the 1970s to evaluate and compare the tribological performance of the different PVD and CVD coatings.

Scratch tests are usually performed by applying an increasing or constant load. The test procedure is governed by the ASTM Standard G171 (03). The primary information derived from these tests is the critical normal load leading to the total failure (i.e. delamination) of the coating. Microscopic examination of the scratch produced on the coated surface can provide additional information on the measure and mechanism of the coating's damage. The ASTM C1624 standard represents useful guidance providing a uniform basis for the interpretation and designation of the subsequent damages [7,8].

We have completed the measurements with four different loading forces, i.e. F = 10, 15, 50, and 100 N, while the specimen holder table travel speed was v = 5 mm/min, identical for each test.

The friction coefficient – scratch length diagrams obtained for 10 and 15 N normal loads are shown in Figure 2.

The curves of the individual coatings overlap and show no significant difference. However, for both loadings, the highest friction coefficient was obtained in the TiAlN+DLC coating. It can also be established that the steady-state friction coefficient values were systematically lower for the higher normal load (F=15 N) cases. Namely, it was μ~0,3 for F=10 N while μ ~0,23 for F=15 N loading.

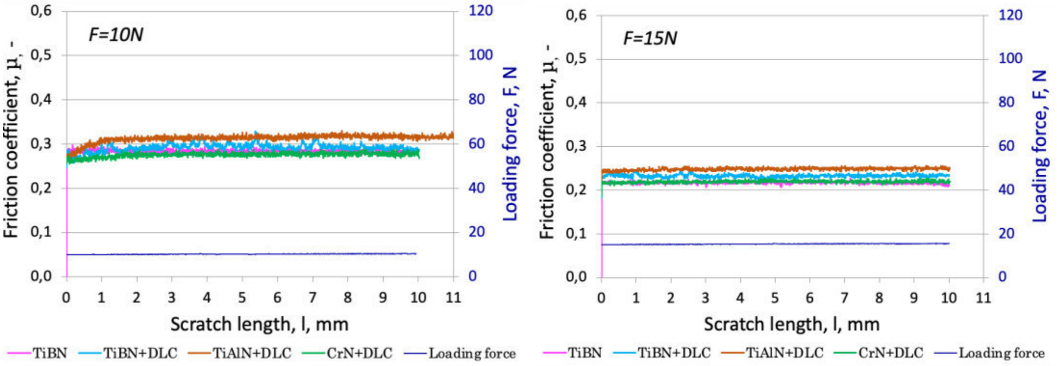


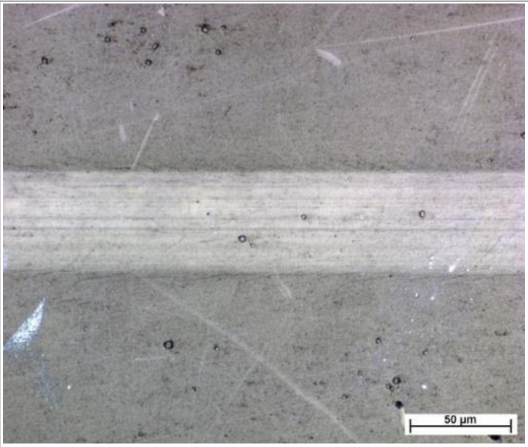
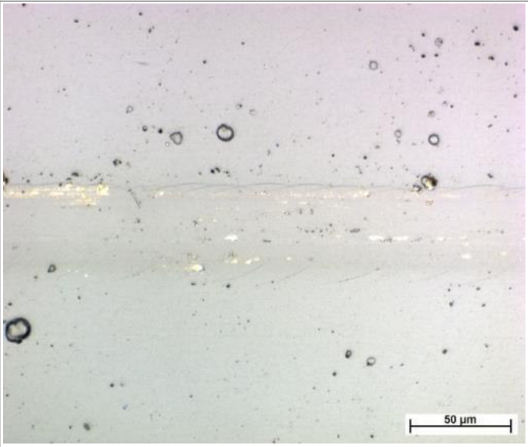
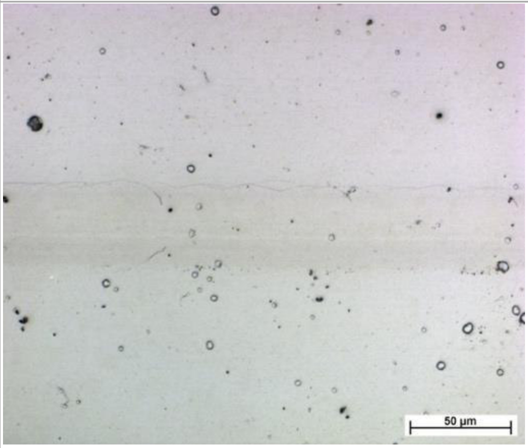
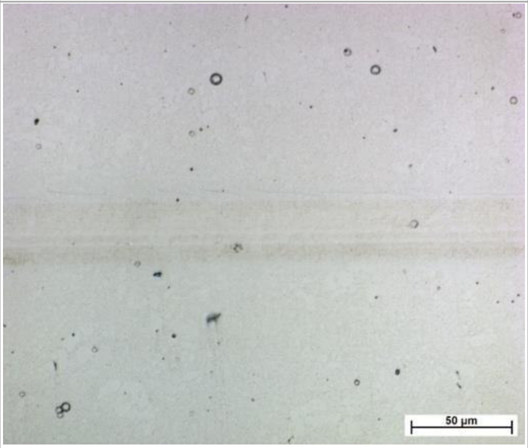
Figure 2. μ and F vs. l diagram of the investigated coatings for a constant normal load of F = 10 N (left) and F = 15 N (right)

The optical microscopic images of the scratches created by the 10 N loading force are shown in Table 2. In contrast, micrographs illustrating the scratches produced by F = 15 normal loads are shown in Table 3.

Analysing these images, it can be established that in the case of F = 10 N constant load, the TiBN-coated sample showed no functional damage, while this loading caused partial tensile cracking, which appeared for each DLC top-coated sample. The characteristic of such damage is the 45° angle between the axis of the cracks and the scratch groove.

Visualising such minor injuries by optical microscopy is difficult since these damages occur at loadings well below the critical one.

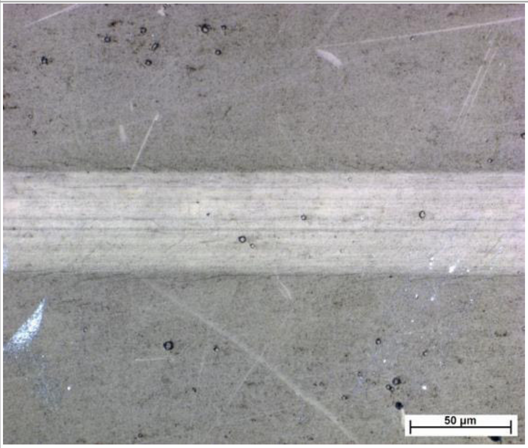
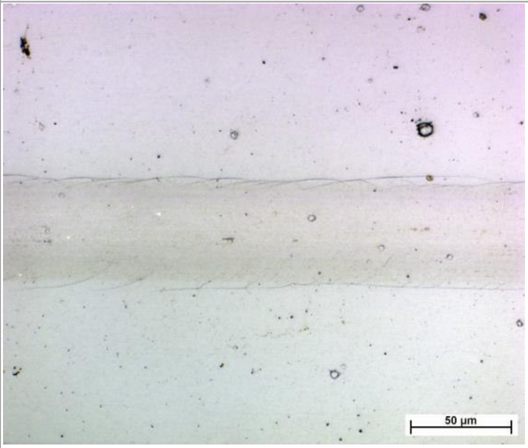
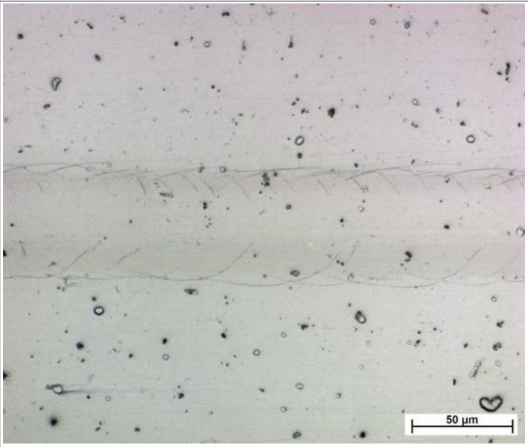
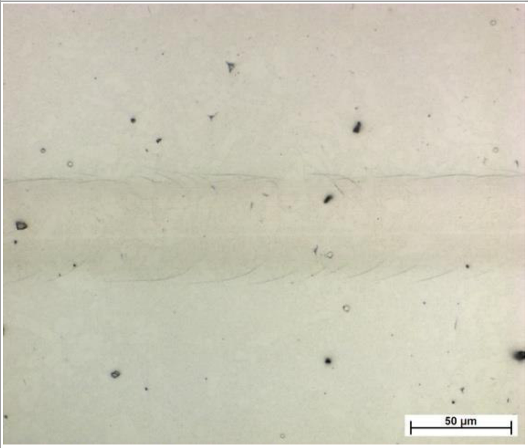
Table 2. Analysis of the damage mechanisms of the coatings at F = 10 N loading force

TiBN coating	**TiBN+DLC coating**
	
TiAlN+DLC coating	**CrN+DLC coating**
	

Increasing the loading force from 10 N to 15 N resulted in no considerable changes in the scratching behaviour of the TiBN coating; only a slight indication of lateral crack formation is worth mentioning. Besides, as experienced before, partial tensile cracking was observed for the TiAlN+DLC coating system. In contrast, the damage was more expressed in the case of the TiBN+DLC and CrN+DLC coatings, where continuous tensile cracks could be seen on the two sides of the scratch grooves.

Based on the morphological analysis of the scratches created by 15 N constant loading, we can conclude that the failure is still subcritical, while the extent of damage on the DLC top-layered coatings is visibly higher.

Table 3. Analysis of the damage mechanisms of the coatings at F = 15 N loading force

TiBN coating	**TiBN+DLC coating**
	
TiAlN+DLC coating	**CrN+DLC coating**
	

Increasing the loading force further up to F = 50 N, the friction coefficient values continued to decrease slightly, having the average values close to μ = 0.2, as shown in Figure.

Another difference in the friction behaviour for this loading was observed in the case of the TiBN+DLC and TiAlN+DLC coatings, where the friction coefficient showed a significant fluctuation that was not typical for smaller loadings.

The character of the related μ-l curves indicates that the integrity of the coating started to be loose, and the debris accumulating in the scratch grooves ahead of the stylus obstructs the continuous progress of the tool motion and causes a local increase in the coefficient of friction.

Figure 3 also provides information on the friction coefficients registered in the case of the highest, i.e. F = 100 N normal load, displaying μ values being strikingly higher for all investigated coatings compared to the previously demonstrated lower loading cases.

Analysing these μ-l curves, it is seen that the friction coefficient values rapidly increase in the initial stages of the scratching operation. The range of the friction coefficient falling between 0.45÷0.55 suggests that the coatings suffer total delamination at an early stage of the scratch test, and the stylus penetrates the metallic substrate, deforming it plastically.

From these scratching diagrams obtained for the F= 50 and 100 N, it can also be concluded that the critical load falls between these values.

The usual method of finding the F_{crit} during constant load scratch test is to refine the load increments until we can find the highest load that does not yet cause coating separation from the substrate while finding the lowest load that already leads to complete damage to the coating.

It is evident that this critical parameter is easier to find in a test with an increasing loading force regime; however, the identification of typical damage modes occurring at a given load can be done with much higher reliability during constant loading tests.

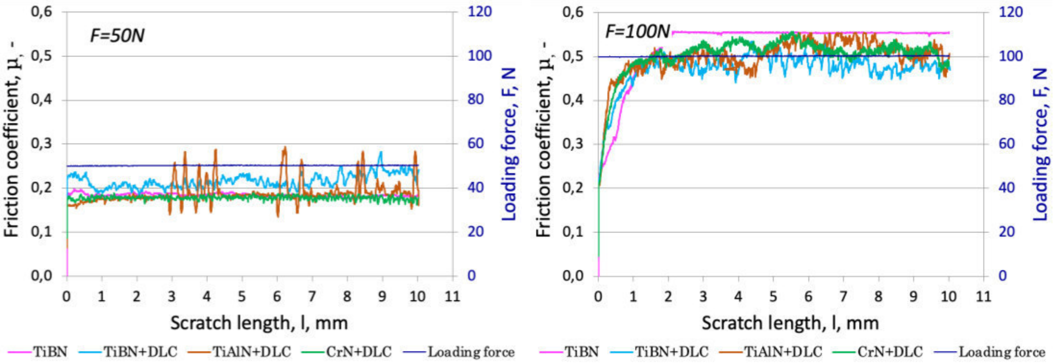


Figure 3. The μ and F vs. l diagrams of the investigated coatings for F = 50 N (left) and F = 100 N (right) constant normal loads

The microscopic pictures of the typical damage mechanisms of the coatings caused by the F = 50 N load are summarised in Table 4.

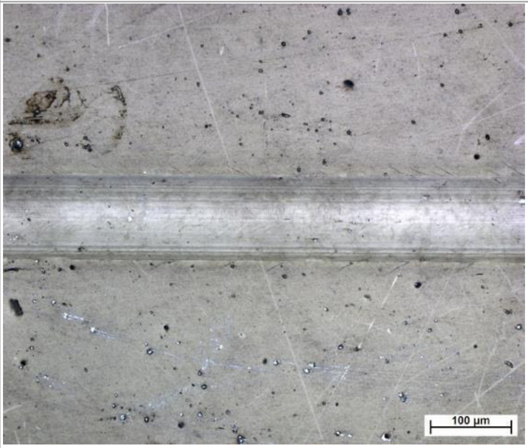
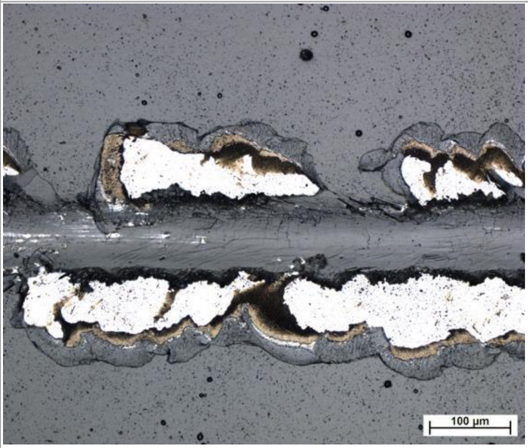
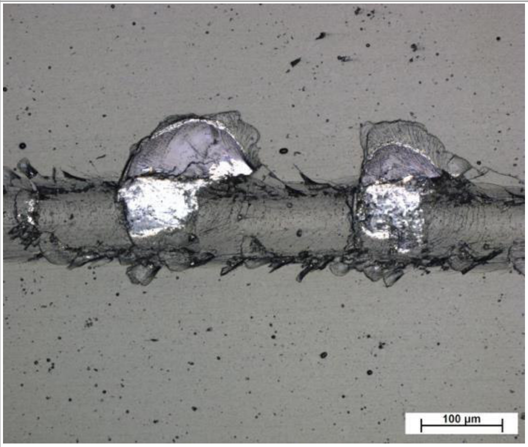
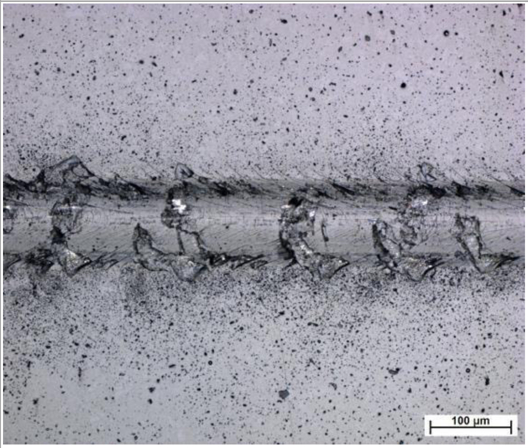
In the monolayered TiBN coating, clear and definite lateral cracks can be recognised along the scratch path with a slight indication of partial recovery spallation.

In contrast, wedging spallation was observed at this loading for all tested multilayered DLC coatings. Still, the measure of the damage was different for the DLC top layered coatings depending on the type of the supporting layer.

At the highest, i.e. F = 100 N normal load, the monolayered TiBN still showed the best scratch resistance with the damage mechanisms of complete recovery spallation and buckling spallation (Table 5.).

Under the F = 100 N load, all the DLC topcoat systems delaminated from the substrate, but the degree of damage was different, which allows for ranking of the multilayer coatings in terms of scratch resistance. The extent of the spalled regions on the two sides of the scratch groove was the highest in TiBN+DLC multilayered coatings. However, the integrity of the coating in the scratch groove was kept to the best. The minor extension of the spalled local region appeared in the case of the CrN+DLC coating, but in this case, the frequency of the spallation was the highest.

Table 4. Analysis of the damage mechanisms of the coatings at F = 50 N loading force

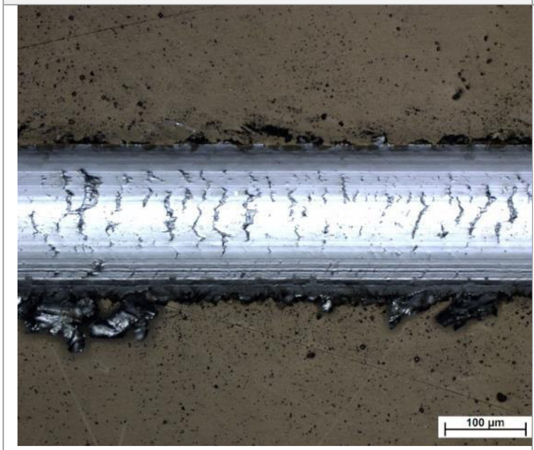
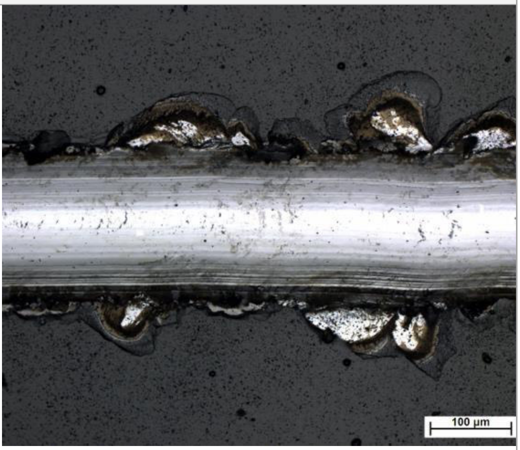
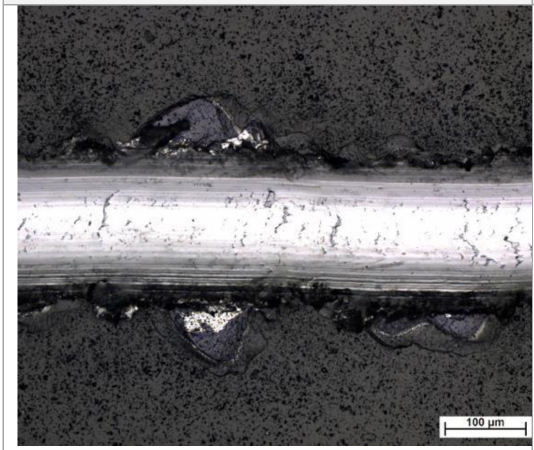
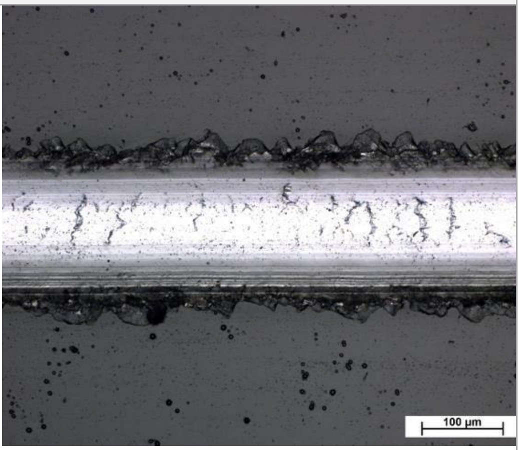
TiBN coating	**TiBN+DLC coating**
	
TiAlN+DLC coating	**CrN+DLC coating**
	

From the above analysis, it is seen that evaluating the mode and severity of the failure needs a careful and complex evaluation process, with attention to several aspects (friction coefficient area, etc.). Therefore, a complete and reliable analysis usually needs microscopic analysis applying visualisation techniques of higher resolution, e.g. fractography using scanning electron microscopy.

Another direction of the scratch morphology analysis is represented by the visualisation of the scratch groove by 2D and 3D profilometry, providing additional qualitative and quantitative information on the characteristic scratch behaviour of the investigated coating systems.

It is also important to mention that the different coatings can possess very different residual stress conditions, highly influencing the scratch behaviour. Therefore, determining their magnitude and nature (tensile, compressive) and analysing their effect must comprise a part of a more comprehensive scratch test evaluation.

Table 5. Analysis of the damage mechanisms of the coatings at F = 100 N loading force

TiBN coating	**TiBN+DLC coating**
	
TiAlN+DLC coating	**CrN+DLC coating**
	

3. Summary

Due to their performance-improving ability, the automotive industry increasingly uses different mono- and multilayered coatings on structural parts and tools. These coatings have several functions, including enhancing resistance to wear, scratch, and corrosion or optimising friction behaviour.

In the current work, we investigated the scratching behaviour of industrially produced and widely applied coatings: a monolayer TiBN coating and three DLC top-layered multilayer coatings with sublayers of TiBN, CrN, and TiAlN. During the constant load scratch test, we applied normal loads of F = 10, 15, 50, and 100 N.

The most important establishments of the research work can be summarised as follows:

1) The coating thickness, measured by the ball-cratering method, was the highest for the

TiBN+DLC multilayered system, which may impact the observed most advantageous performance of this coating under scratch-type loadings in the applied load range.

2) The friction coefficient decreases from 0.3 to 0.2 for each tested coating system with the increase of the normal load from 10 to 50 N, while for F = 100 N μ increases to 0.45- 0.55.
3) The critical average load causing the total delamination of the coatings falls into the range of F = 50-100 N, excepting the TiBN coating, for which the expected F_{crit} value exceeds the upper limit of the applied load range.
4) Morphological analysis of the scratch grooves revealed different damage mechanisms for the TiBN coating compared to the DLC top-coated systems. Under lower (10, 15 N) loadings, each coating showed partial tensile cracking to a different extent, but the most minor damage was experienced for the TiBN monolayer coating. In the case of the DLC top-layered systems, the type of damage was supplemented by partial and complete tensile cracking, becoming more severe when the loading force was increased to 50 and 100 N. At the highest load (F=100 N), complete recovery spallation and buckling spallation occurred in TiBN coating, while TiAlN+DLC and CrN+DLC coatings suffered from total failure.
5) A given loading force caused different failure modes in the different coatings. However, the similarity of scratch damage mechanisms of the DLC top layered coatings indicates the dominancy of the DLC layer in the multilayered system regarding the failure mode.

The direction of the future work is the analysis of the effect of residual stresses on the scratching behaviour of the tested coatings and profilometry analysis to obtain further qualitative and quantitative features of the scratching behaviour of these coatings.

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