

## DETERMINATION OF CONDITIONS FOR EVALUATING FRICTION-WEAR PROPERTIES OF COATINGS

Huu Chien NGUYEN, Emil SVOBODA, Jiří PROCHAZKA, Tomáš DRAŽAN, Jakub HORNÍČEK,  
Dang Long LE

*UNOB - University of Defence in Brno, Brno, Czech Republic, EU,*  
[huuchien.nguyen@unob.cz](mailto:huuchien.nguyen@unob.cz)

<https://doi.org/10.37904/metal.2021.4168>

### Abstract

This study was conducted to investigate the determination of the conditions for evaluation friction-wear properties of the thin layers and coatings on structural 16MnCr5 steel. The AlCrVN coatings with the thickness of 2.0  $\mu\text{m}$  were generated on the un-nitrided and nitrided materials under the same conditions. The friction-wear properties of the layers and coatings were evaluated by the ball-on-flat method on The Universal Mechanical Tester 3 (UMT-3) with different test forces. The obtained results showed that the friction-wear properties of AlCrVN-coated materials changed markedly when the force reaches a certain value. The most suitable conditions for evaluating the properties of thin hard coatings have been determined.

**Keywords:** Plasma nitriding, AlCrVN coating, duplex coating, friction-wear properties, ball-on-flat test

### 1. INTRODUCTION

It has been reported that the duplex coatings composed nitrided layers and thin hard coatings improved markedly mechanical, friction-wear properties of steels [1], [2]. The plasma nitriding technology was mostly used primarily to increasing of surface hardness, wear resistance, corrosion resistance and fatigue strength [3], [4], [5], [6], [7], [8]. Nitrided layers formed by plasma nitriding improved adhesion and wear resistance of the thin hard coatings [9]. The AlCrVN coating have been investigated in many studies to improve the friction-wear properties of materials [9], [10]. The AlCrVN coatings exhibited low friction coefficient of 0.2 and good wear resistance at high temperature (700 °C) [10].

The ball-on-disc and ball-on-flat methods are the most common laboratory methods for evaluating the tribological properties of materials, especially the evaluation of adhesive, abrasive wear [11], [12], [13]. The advantages of the ball-on-flat method over the ball-on-disc method are the simplicity of test specimen preparation and the accurately of wear evaluation. Due to the limited adhesion of the thin hard coatings to the substrate, the coating could be delaminating damaged under high stress during the test. In this case, the results of the experiments do not correctly reflect the friction-wear properties of the coatings. Therefore, it is important to determine appropriate test conditions, in particular the load when evaluating the tribological properties of layers and coatings using the ball-on-flat method.

This study was conducted to determine of the conditions for evaluation friction-wear properties of the thin compound layer and AlCrVN coatings. As the substrate, DIN 16MnCr5 structural steel, which was widely used for higher stressed components production, such as gears, shafts, crankshafts, connecting rods, camshafts, etc., was selected. The AlCrVN coatings were coated on both un-nitrided and nitrided substrates under the same conditions. Friction-wear tests were conducted on the UMT-3 tribometer with different test forces for evaluating friction-wear properties of the materials. The profiles of the wear track cross sections were obtained by Talysurf CLI 1000 stylus profilometer using absolute inductive position sensor.

## 2. MATERIALS AND EXPERIMENTAL PROCEDURES

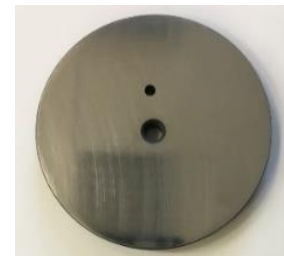
### 2.1. Materials

Chemical composition of the 16MnCr5 structural steel used in this study was evaluated by spark emission equipment Q4 TASMAR. The measurement chemical composition of steel was performed five times and the averages are showed in (Table 1).

**Table 1** 16MnCr5 structural steel chemical composition [%]

| Elements      |      | C              | Mn             | Si             | Cr             | P                | S               |
|---------------|------|----------------|----------------|----------------|----------------|------------------|-----------------|
| EN 10084-2008 | max. | 0.19           | 1.40           | 0.37           | 1.10           | 0.035            | 0.035           |
|               | min. | 0.14           | 1.10           | 0,17           | 0,80           | -                | -               |
| Measured      |      | 0.18<br>± 0.01 | 1.10<br>± 0.05 | 0.35<br>± 0.02 | 1.10<br>± 0.01 | 0.023<br>± 0.005 | 0.01<br>± 0.005 |

The specimens used in the experiment are disc-shaped in diameter of 6.5 cm and a thickness of 5 mm. The surface of the samples was wet grinded by Struers LaboPol-60 grinder to obtain the same surface roughness Ra (0.09 µm) in order to eliminate the effect of roughness on the friction-wear properties. For grinding, the Silicon Carbide Grinding papers grit 120, 220, 800, 1200 were used. The metallographic test sections of the specimens were polished to mirror surfaces using Leco CAMEO Disc Platinum 1, 2, 3, 4 and diamond polishing paste with grain size 1 µm by Leco PX-500 Grinder/Polisher.



**Figure 1** Feature of samples

Four types of materials were prepared for this study. The untreated material, plasma-nitrided material are called UN, PN materials, respectively. The AlCrVN coating to untreated material was performed and this AlCrVN-coated material is called AlCrVN material (AlCrVN coating). Hereafter, the AlCrVN coating to nitrided material was carried out. This hybrid-surface-treated material is called PN/AlCrVN material (AlCrVN duplex coating).

Plasma nitriding process was carried out in the PN 60/60 Rübige equipment at a temperature of 470 °C for 4 h in a 280 Pa gas mixture of H<sub>2</sub> flow 8 l/min and N<sub>2</sub> flow 24 l/min. The AlCrVN coating were performed in the company LISS using cathodic-arc deposition method.

### 2.2. Experiment procedures

The surface roughness parameter Ra was measured by a profilometer Talysurf CLI 1000. Ra is widely used because the surface roughness assessment according to Ra is relatively inexpensive [14].

After polishing to mirror surfaces, the test cross sections were etched by 2 % Nital etchant (a solution of ethanol and nitric acid). The microstructure of the nitrided layer and the feature of AlCrVN coatings were observed on the cross sections of the disc specimens by Olympus DSX500i opto-digital microscope. The thickness of the AlCrVN coatings was measured on the cross sections using optical method by Olympus LEXT 3000 laser confocal microscope. The microhardness profile of the nitrided layer was obtained in the range from the surface to depth of 0.5 mm on the polished cross section of PN material with the load of 0.981 N by Vickers method on the LECO LM247/AT Microhardness Tester. Based on the results of the microhardness test, the thickness of the nitrided layer was determined according to DIN 50 190-3 [15].

The friction-wear properties of the materials were evaluated using the linearly reciprocating ball-on-flat sliding wear test on Bruker UMT-3 tester. To obtain stable results, Tungsten carbide balls with the hardness of 1800 HV10 were used as the opposite material. The conditions of the test are showed in (Table 2).

**Table 2** Conditions of the friction-wear tests [12]

| Parameter | Ball diameter (mm) | Stroke length (mm) | Oscillating frequency (Hz) | Test duration (s) | Ambient temperature (°C) | Relative humidity (%) | Lubrication |
|-----------|--------------------|--------------------|----------------------------|-------------------|--------------------------|-----------------------|-------------|
| Value     | 6.35               | 10                 | 5                          | 1000              | 22 ± 0.5                 | 40 - 60               | None        |

The above test parameters were remained stable to eliminate the effect of these parameters on the friction-wear properties of materials for all friction-wear tests. The friction-wear tests were performed with different test forces to select the most suitable force to evaluate the tribological properties of layers and coatings. The test forces of friction-wear test were 5 N, 10 N, 12 N, 20 N and 25 N.

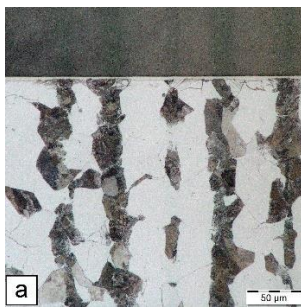
The cross-sectional profiles of the wear track were evaluated using stylus profilometer Talysurf CLI 1000 with the absolute inductive sensor at eleven locations equally spaced along its length. From the recorded data, the mean cross-sectional profiles were obtained. The track cross-sectional area and track maximal cross-sectional depth (wear depth) were determined using the Tallymap Platinum software. The total volume of material removed during sliding (the wear volume) was computed from the track cross-section area and the length of stroke [11].

### 3. RESULTS

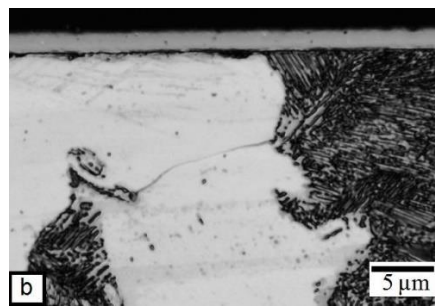
#### 3.1. Microstructure and hardness profile

**Figure 2** shows the features of the nitrided layer and the PN/AlCrVN duplex coating. **Figure 3** shows the microhardness profile of the nitrided layer.

According to the results of the measuring by laser confocal microscope, the thickness of the AlCrVN coatings was  $2.0 \pm 0.1 \mu\text{m}$ . As showed in (**Figure 2**), a compound layer with thickness  $2 \pm 0.5 \mu\text{m}$  was generated on surface of PN material. The thickness of the nitrided layer was  $185 \mu\text{m}$ , as can be understood from (**Figure 3**).

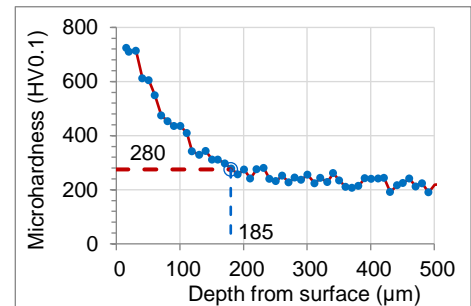


a)



b)

**Figure 2** Features of a) nitrided layer, b) PN/AlCrVN duplex coating



**Figure 3** Hardness profile measured on cross section of PN material

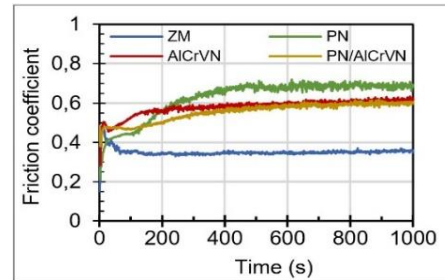
#### 3.2. Friction-wear properties

**Figure 4** shows the relationship between the test time and the friction coefficients at test force of 5 N. **Figure 5** shows the relationship between the test time and the friction coefficients, the features of the wear tracks, the mean cross-sectional profiles of the wear track for other test force. **Figure 6** shows the wear volumes and wear depth of materials at different test forces.

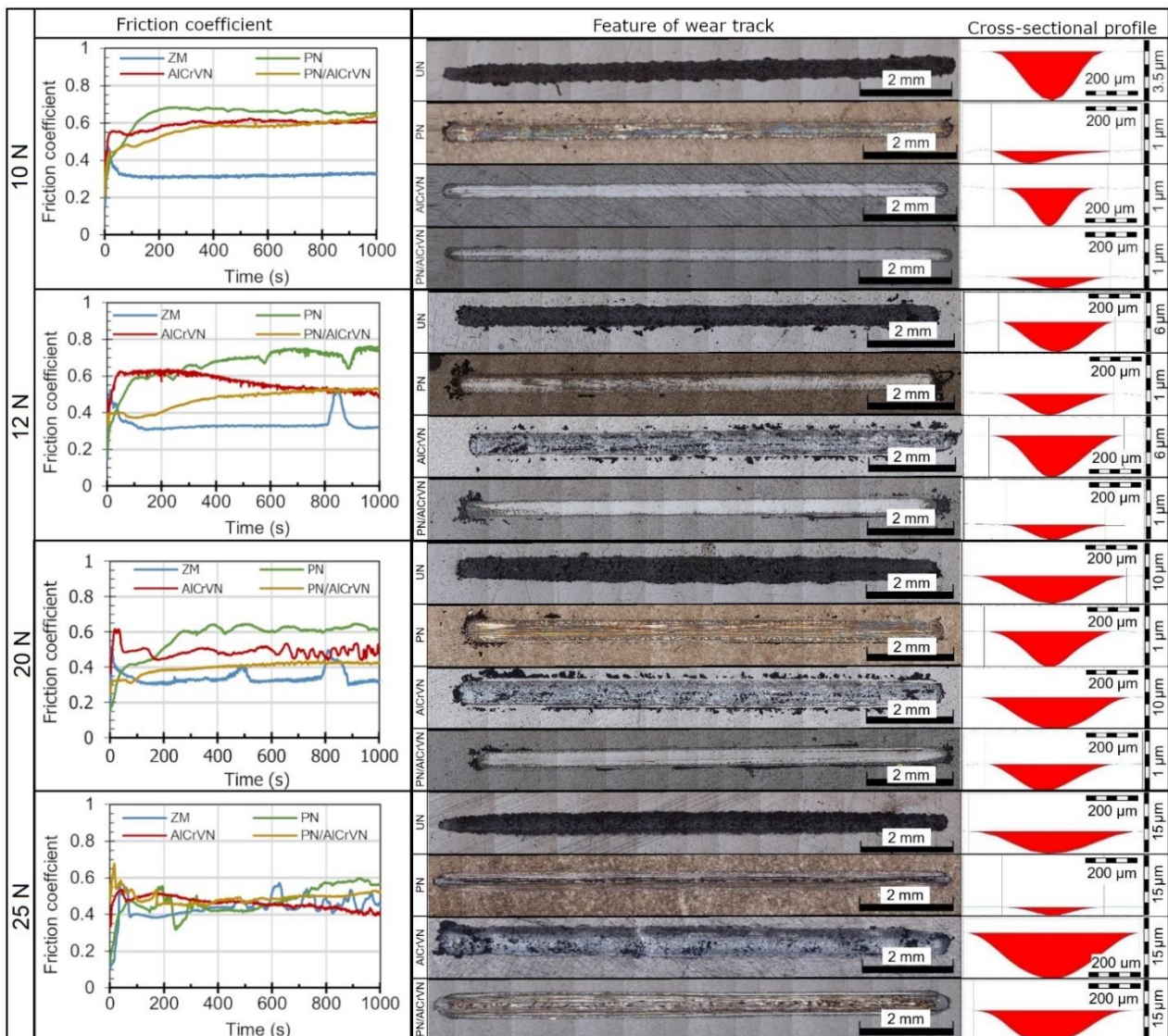
At a test force of 5 N, there was no sharp change in the friction coefficients after the running-in period. The friction coefficients of all materials fluctuated slightly.

With a test force of 5N, the wear amount of the samples is very small. Therefore, it was so hard to accurately evaluate the wear volume and wear depth of materials, especially for the PN and PN/AICrVN material.

As can be understood from (Figure 5), the relationship between the time test and the friction coefficients at a test force of 10 N were almost similar that at test force of 5 N for all materials. The wear volume of the PN/AICrVN material was the lowest (Figure 6a). The wear volume of the PN material was lower than the value of the AICrVN. The wear volume of the UN material was highest. During the test, there were not failures of coatings for all AICrVN-coated materials (Figure 5).

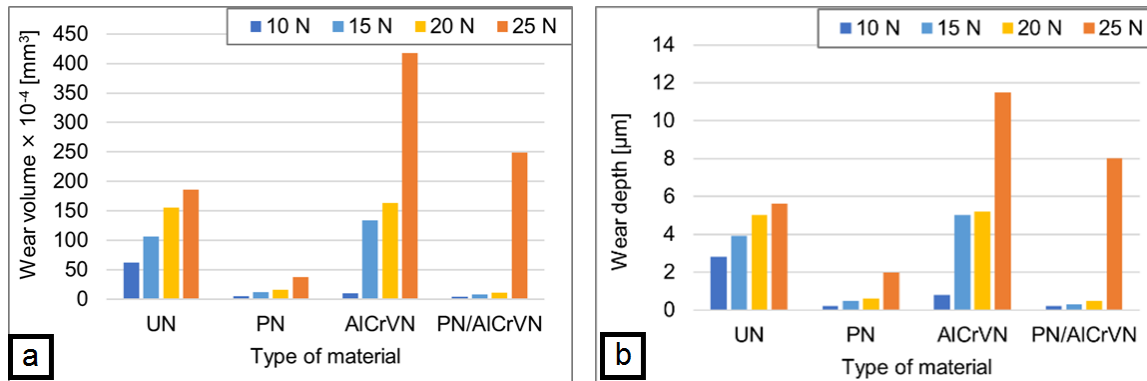


**Figure 1** Relationship between test time and friction coefficients at 5 N



**Figure 2** Relationship between time test and friction coefficients; features and mean cross-sectional profiles of wear tracks

The friction-wear properties of each material at a test forces of 12 N and 20 N were similar. At a test force of 12 N, from the test time of 300 s, the friction coefficients of the AlCrVN material started decreasing because the AlCrVN coating was gradually damaged and wear proceeded to the substrate. At a test force of 20 N, from the test time of 50 s, the friction coefficients of the AlCrVN material dropped because the AlCrVN coating was markedly damaged. In contrast, there were not failure of the AlCrVN coating on PN/AlCrVN material. At a test force of 12 N and 20 N the wear volume of the AlCrVN material was higher than that of the UN material due to the damage of the AlCrVN coating. The wear volume of the UN material was markedly higher than the value of PN material. The wear volume of the PN/AlCrVN was lowest (**Figure 6a**).



**Figure 3** a) Wear volume, b) wear depth at different test forces

At a test force of 25 N, the friction coefficients started dropping from test time of 50 s for AlCrVN material and from 30 s for PN/AlCrVN material due to marked damage of the AlCrVN coatings and wear proceeded to the substrate. The friction coefficients of PN material markedly fluctuated in interval (180-330) s because the compound layer was gradually damaged. The wear volumes of the AlCrVN-coated materials were higher than the value of the UN material; however, the wear volume of the PN material was lower (**Figure 6a**).

For the test forces above 10 N, there occur upward peaks in the friction coefficient curve after the running-in period for UN material. These changes of the friction coefficient were caused by the changes of properties wear debris layer formed on the worn substrate [16], [17]. The chemical composition of the wear debris layers can be evaluated using the method energy dispersive X-ray spectroscopy (EDX) on the transmission electron microscope [18].

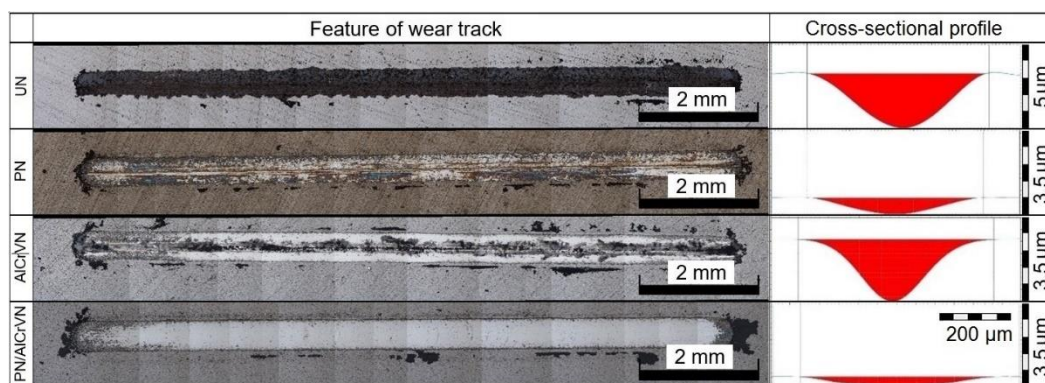
As can be understood from (**Figure 6**), with the increasing test force the wear volume and wear depth has increased. The wear volume of AlCrVN material increased dramatically with the increasing test force from 10 N to 12 N and from 20 N to 25 N. However, the wear volume of PN/AlCrVN material and PN material increased dramatically only with the increasing test force from 20 N to 25 N. This kind of increasing in wear volume with increasing test force did not occur for UN material.

At test forces above 10 N for AlCrVN material and above 20 N for PN/AlCrVN material, the wear depth was greater than the AlCrVN coating thickness. At test forces above 20 N for PN material, the wear depth was greater than the compound layer thickness. This means that, after the layers had been damaged, the wear proceeded to the substrate for AlCrVN-coated materials. After damaged compound layer, the wear proceeded to the diffusion layer for PN material (**Figure 5**). With damaging of coatings, small pieces of the coating were peeled off and formed hard particles. These particles acted in the frictional process as the abrasive particles between the specimen surfaces. Hence, after damaged coating, the wear speed of the AlCrVN-coated materials increased markedly. The wear volume of AlCrVN-coated materials was much greater than the untreated material (**Figure 6**). In this case, the obtained result cannot be used to evaluate the friction-wear properties of the AlCrVN coatings.

From the obtained results, the test force of 5 N is unsuitable (too low) to evaluate the friction-wear properties of AlCrVN coatings. The test force of 12 N, 20 N and 20 N were unsuitable (too high) to evaluate the friction-wear properties of the AlCrVN coating. However, the test force of 12 N and 20 N can be used to evaluate the friction-wear properties of nitrided layer and AlCrVN duplex coating. The test force of 25 N was unsuitable (too high) to evaluate the friction-wear properties of the nitrided layer and AlCrVN coatings. To reduce the test time while ensuring the wear is large enough, the most suitable test force is the largest of the forces that can be used. The most suitable test force to evaluated friction-wear properties for AlCrVN material and for PN/AlCrVN, PN material was 10 N and 20 N, respectively.

At a test force of 10 N after a test duration of 1000 s, the wear volume of the nitrided layer and the duplex coating reached very small values (**Figure. 6**). Due to the small wear volume, the results of experiments cannot clearly show the influence of selected technologies on the wear properties of the steel. Furthermore, with a small wear depth, it was hard to evaluate accurately wear due to the effect of surface roughness.

Under certain test conditions, the wear volume can be increased by extending the test duration. However, the test duration should be chosen so that the wear does not exceed all of the coating. For the AlCrVN material, the wear depth was 0.6  $\mu\text{m}$  for test duration 1000 s. Under the same conditions, the additional friction-wear test with a test duration of 2000 s was performed, in which an increase in the wear depth by 1.2  $\mu\text{m}$  to approximately 2  $\mu\text{m}$  can be expected. **Figure 7** shows the features of the wear tracks and the mean profiles of the wear track cross sections for test force of 10 N, test duration of 2000 s.



**Figure 7** Features and mean cross-sectional profiles of wear tracks at test force of 10 N, test duration of 2000 s

**Table 3** Wear volume and wear depth at test duration of 1000s, 2000 s (test force of 10 N)

| Test duration (s)                               | 1000 |     |        |           | 2000 |     |        |           |
|---|------|-----|--------|-----------|------|-----|--------|-----------|
|   | UN   | PN  | AlCrVN | PN/AlCrVN | UN   | PN  | AlCrVN | PN/AlCrVN |
| Wear volume $\times 10^{-4}$ (mm <sup>3</sup> ) | 62   | 5   | 10     | 4         | 90   | 19  | 47     | 14        |
| Wear depth ( $\mu\text{m}$ )                    | 2.8  | 0.2 | 0.6    | 0.2       | 3.5  | 0.7 | 2.6    | 0.6       |

As can be understood from (**Table 3**), the wear depth of AlCrVN material was higher than the coating thickness. However, the coating AlCrVN only was worn through in the small area of wear track (**Figure 7**). The wear volume of nitrided and AlCrVN-coated materials increased from 3.5 to 4.5 times. The wear depth of PN material and AlCrVN-coated materials increased from three to four times. The test duration of 2000 s was more suitable than that of 1000 s to evaluate wear properties of nitrided layer and AlCrVN coatings.

The obtained results suggested that, the test force of 10 N and the test duration of 2000s was most suitable to evaluate the friction-wear properties for all coatings and layers on 16MnCr5 steel using the ball-on-flat method.

#### 4. CONCLUSION

The suitable conditions of the ball-on-flat test were determined to evaluate friction-wear properties of nitrided layer and AlCrVN coatings on 16MnCr5 steel.

The choice of test force has a great influence on the results of the evaluation of the friction-wear properties of the layers and coatings by method ball-on-flat. The test force of 5 N, at which the wear of treated materials was too low, was unsuitable to evaluate wear resistance. The test force of 25 N, at which all layers were damaged were too high for friction-wear test. There were not failures of AlCrVN duplex coating at test force of 12 N, 20 N. However, the AlCrVN coating on un-treated material was gradually damaged at these test forces. The test force of 10 N, at which damaging did not occur for all coatings was the most suitable force to evaluate friction-wear properties of coatings.

The wear volume increased dramatically with increasing test force to the value, at which the layers, coatings were damaged. To reduce the test time while ensuring the wear is large enough, the most suitable test force is the greatest force, which does not damage the coatings and layers.

#### ACKNOWLEDGEMENTS

***The work presented in this paper has been supported by the specific research project 2020 "SV20-216" at the Department of Mechanical Engineering, University of Defence in Brno and the Project for the Development of the Organization "DZRO Military autonomous and robotic systems".***

#### REFERENCES

- [1] JOSKA, Z., KADLEC, J., HRUBÝ, V., et al. Mechanical properties of duplex treated AISI 316L stainless steel. In: *METAL 2011: 20th Anniversary International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2011, pp. 724-728.
- [2] JOSKA, Z., KADLEC, J., HRUBÝ, V., et al. Investigation of diamond like carbon coating deposited on plasma nitrided austenitic stainless steel. *Chemické listy*. 2012, vol. 106, issue S1, pp. S446-S447.
- [3] KADLEC, J., JOSKA, Z., POKORNÝ, Z. Plasma Treatment of Stainless Steel Surface. *ECS Transactions*. 2010, vol. 32, Issue 1, pp. 69-72.
- [4] POKORNÝ, Z., HRUBÝ, V. Plasma nitriding of deep narrow cavities. *Key Engineering Materials*. 2010, vol. 465, pp. 267-270.
- [5] DOBROCKÝ, D., STUDENÝ, Z., POKORNÝ, Z. Effect of plasma nitriding on the notch toughness of spring steel. In: *METAL 2016: 25th Anniversary International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2016, pp 1037-1045.
- [6] STUDENÝ, Z., KUŠMÍČ, D. Influence of inclusions size on the nitrided components fatigue life. In: *METAL 2014: 23th Anniversary International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2014, pp. 875-880.
- [7] POKORNÝ, Z., DOBROCKÝ, D., KADLEC, J., STUDENÝ, Z. Influence of alloying elements on gas nitriding process of high-stressed machine parts of weapons. *Kovové materiály – Metallic materials*. 2018, vol. 56, issue 2, pp. 97-103.
- [8] PROCHÁZKA, J., POKORNÝ, Z., DOBROCKÝ, D. Service behaviour of nitrided layers of steels for military applications. *Coatings*. 2020, vol. 10, issue 10, Article number 975.
- [9] SINDRA, I., FEI CAI, J.W., et al. Effect of addition of Mo or V on the structure and cutting performance of AlCrN-based coatings. *Coatings*. [online]. 2020, vol. 10, no. 3, pp. 1–11. Available from: <https://doi.org/10.3390/coatings10030298>.
- [10] ROBERT, F., BERNHARD, S., et al. High-temperature, low-friction properties of the Vanadium alloyed AlCrN coatings. *Tribology letters*. [online]. 2006, vol. 23, no. 2. Available from: <https://doi.org/10.1007/s11249-006-9064-1>.
- [11] ASTM G155-04. *Standard Guide for Measuring and Reporting Friction Coefficients*. West Conshohocken, PA, USA: ASTM International, 2004.

- [12] ASTM G133-02. *Standard Test Method for Linearly Reciprocating Ball-on-Flat Sliding Wear*. West Conshohocken, PA, USA: ASTM International, 2002.
- [13] ASTM G99-95. *Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus*. West Conshohocken, PA, USA: ASTM International, 1995.
- [14] BARÉNYI, I., MAJERÍK, J., POKORNÝ, Z., SEDLÁK, J., BEZECNÝ, J., DOBROCKÝ, D., JAROŠ, D., ECKERT, M., JAMBOR, J., KUSENDA, D. Material and technological investigation of machined surfaces of the OCHN3MFA steel. *Kovové materiály – Metallic materials*. 2019, vol. 57, issue 2, pp. 131-142.
- [15] DIN 50190-3:1979. *Hardness depth of heat-treated parts; determination of the effective depth of hardening after nitriding*. German: German Institute for Standardisation, 1979.
- [16] ALFERED, Z. Wear debris: a review of properties and constitutive models. *J. Theor. Appl. Mech.* 2005, vol. 43, no. 1, pp. 3–35.
- [17] HONGXIN, S., SANMING, D., CHAO, S., CHENFEI, S., ZHENGHAI, Y., YONGZHEN, Z. Behaviour of wear debris and its action mechanism on the tribological properties of medium-carbon steel with magnetic field. *Materials (Basel)*. [online]. 2018, vol. 12, no. 1, pp. 14–16. Available from: <https://doi.org/10.3390/ma12010045>.
- [18] ROBL, J., SEDLÁK, J., POKORNÝ, Z., ŃUKSA, P., BARÉNYI, I., and MAJERÍK, J. Analysis of advanced additive technology in direct metal laser sintering and precision casting method. *BULLETIN OF THE POLISH ACADEMY OF SCIENCES TECHNICAL SCIENCES*. [online]. 2020, vol. 68, no. 1, pp. 109-118. Available from: <https://doi.org/10.24425/bpasts.2020.131842>.