

EFFECT OF THE ABRASIVE GRIT SIZE ON THE WEAR BEHAVIOR OF CERAMIC COATINGS DURING A MICRO-ABRASION TEST

VPLIV VELIKOSTI BRUSNIH ZRN NA VEDENJE KERAMIČNIH PREVLEK PRI MIKROABRAZIJSKEM PREIZKUSU OBRABE

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Micro-abrasion tests are commonly used to perform the wear tests on hard coatings. In this study, AISI 1040 mild-steel specimens were coated with ceramic coatings including oxides with different hardness values. The free-ball micro-abrasion testing method was used to examine the wear behavior of the ceramic surfaces over different test durations. The coating-hardness measurements were carried out with a micro-hardness tester. In the experimental studies, the SiC abrasive particles with three different grain sizes (800, 1000 and 1200 mesh), were used to explore the effect of the abrasive particle size on the wear ratio of ceramic coatings. The abrasive effect of the SiC particles in the abrasive slurry was evaluated by examining the SEM micrographs of the coating surfaces. According to the test results, the sample surface coated with Cr₂O₃ exhibited a higher wear resistance than those covered with Al₂O₃ and its compositions with TiO₂. It can also be concluded from the results that an increase in the percentage of the TiO₂ powders in the Al₂O₃ mixture leads to a decrease in the wear resistance.

Keywords: micro-abrasion, ceramic coating, abrasive grain size

Za preizkušanje obrabe trdih prevlek se navadno uporablja mikroabrazijski preizkus. V tej študiji so bili vzorci mehkega jekla AISI 1040 prevlečeni s keramično prevleko iz različno trdih oksidov. Za ugotavljanje vedenja keramičnih površin pri obrabi z različnim trajanjem preizkusa je bila uporabljena metoda mikroabrazijskega preizkusa s tremi prostimi krogli. Trdota prevleke je bila izmerjena z merilnikom mikrotrodote. Pri eksperimentih so bili za ugotavljanje vpliva velikosti abrazijskih delcev na obrabo keramične prevleke uporabljeni abrazijski delci SiC s tremi različnimi velikostmi (zrnatost 800, 1000 in 1200). Abrazijski učinek SiC-delcev in abrazijskih odpadkov je bil ocenjen in preiskan s SEM-posnetki površine prevleke. Glede na rezultate imajo vzorci, prekriti s Cr₂O₃, večjo odpornost proti obrabi kot vzorci z Al₂O₃ in njihova kombinacija s TiO₂. Iz rezultatov se lahko tudi ugotovi, da povečanje deleža TiO₂ v mešanici z Al₂O₃ povzroči zmanjšanje odpornosti proti obrabi.

Ključne besede: mikroabrazija, keramična prevleka, velikost abrazijskih zrn

1 INTRODUCTION

Generally, the coatings used for improving the performance of industrial parts are available in various types, ranging from hard coatings with high abrasion resistance to soft, lubricating coatings and applications requiring low friction coefficients.¹

Ceramics stand out in the friction and abrasion applications in the industry on account of their high hardness, high chemical stabilities, high oxidation-resistance values, high temperature- and thermal-barrier features. However, the high costs involved in their manufacturing and their brittle characteristics limit the use of ceramics. For these reasons, ceramics are preferred for the formation of anti-abrasive layers, applied with thermal-spray methods rather than being used as bulk materials.² For years, various metallic and ceramic coatings have been applied on materials in order to form abrasion-resistant surface layers using the powders sized between 10–100 μm with thermal-spray methods.³ The essence of the atmospheric-plasma-spray (APS) method, which is a

thermal-spray-coating method, is to form a plasma jet for melting the powder material. Powder particles are injected by means of a protective gas and the powders derive their speed and temperature from the plasma jet via the thermal and kinematic transfer. The particulates form abrasion-resistant, rapidly stiffening and thin layers on the surface to be coated.⁴⁻⁷

The micro-scale abrasion test is implemented successfully in assessing the abrasive performances of various materials. This technique is applied on metallic and non-metallic bulk and coating materials along with various abrasive slurry media.⁸⁻¹¹ The abrasion crater obtained in this way is measured with optical or profilometric methods and thus the abrasion results are evaluated in terms of the volume and abrasion mechanisms.¹²⁻¹⁴ The ceramic coatings such as Cr₂O₃, Al₂O₃ and TiO₂ applied with the thermal-spray method were examined tribologically at high temperatures as well as at room temperature, in dry and lubricant sliding conditions.² However, there are not enough studies for an

abrasion analysis of the mentioned ceramic coatings by means of the micro-abrasion technique. For this reason, in this study, the abrasion behaviours of the Cr_2O_3 , Al_2O_3 , $\text{Al}_2\text{O}_3 + 13\% \text{TiO}_2$ and $\text{Al}_2\text{O}_3 + 40\% \text{TiO}_2$ coatings were examined using the micro-abrasion test.

2 MATERIALS AND METHOD

In this study, the AISI 1040 steel with a diameter of 20 mm and length of 50 mm was selected as the substrate material. Firstly, the surface was cleaned of the unwanted residues (oil, dust and residual metals) with a blasting process to achieve a certain roughness value. After this process, the powders with a composite rate of 80 % Cr and 20 % Ni and baked at 100 °C were applied to the substrate material so as to form an intermediate surface with a thickness of 30 μm . The aim of this process is to ensure that a stronger bond is established between the substrate surface material and the ceramic coating material. Finally, the ceramic-powder coatings were applied, in two stages, on the substrate surfaces using the plasma method. The coating thickness was about 150 μm and the process parameters used are shown in **Table 1**. In accordance with the mixture composition of the ceramic powders, different hardness values for the ceramic coatings were obtained (**Table 2**). The SEM micrographs regarding the Cr_2O_3 and Al_2O_3 coatings that include 40 % of TiO_2 are given in **Figures 1** and **2**, respectively.

Table 1: Atmospheric-plasma-spraying conditions

Tabela 1: Pogoji pri naprševanju v atmosferski plazmi

Arc flow rate	80–100 L min ⁻¹
Arc pressure	0.689 MPa
Auxiliary gas flow rate	5–15 L min ⁻¹
Auxiliary gas pressure	0.345 MPa
Spray rate	2.7–6.8 kg/h
Arc voltage	61–68 V
Arc current	400–600 A
Spray distance	70–100 mm

Table 2: Hardness values of the coatings

Tabela 2: Trdota prevlek

Coating material	Hardness (Hv)	Roughness ($R_a/\mu\text{m}$)
Cr_2O_3	960	0.353
Al_2O_3	820	0.326
$\text{Al}_2\text{O}_3 + 13\% \text{TiO}_2$	730	0.237
$\text{Al}_2\text{O}_3 + 40\% \text{TiO}_2$	630	0.250

The aim of the micro-abrasive wear test is to generate "the wear craters" on the specimen. So, one can calculate the wear volume (V), using the crater depth (h) and the crater diameter (b)^{15,16} after the wear tests. The volumetric mass-loss value can be calculated using both the crater radius and the crater height. The formulas required for the calculations are clearly specified in¹⁷.

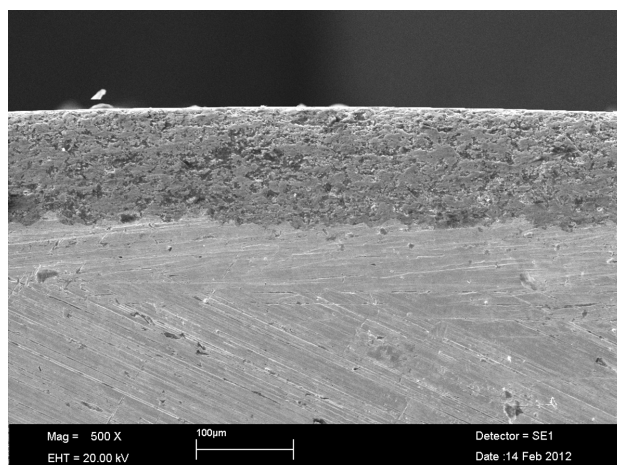


Figure 1: Cross-section of the Cr_2O_3 coating layer
Slika 1: Prečni prerez prevleke iz Cr_2O_3

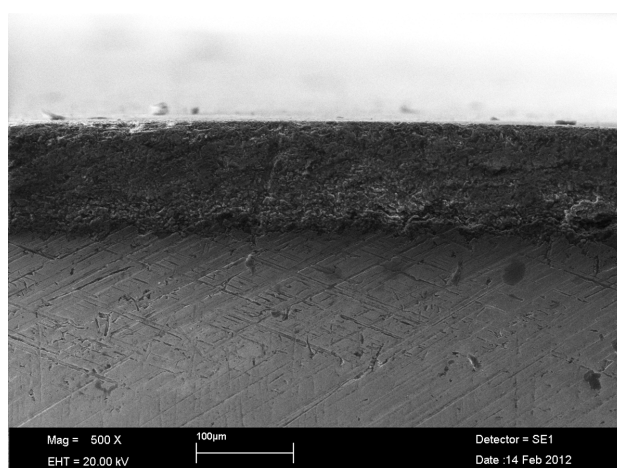


Figure 2: Cross-section of the $\text{Al}_2\text{O}_3 + 40\% \text{TiO}_2$ coating layer
Slika 2: Prečni prerez prevleke iz $\text{Al}_2\text{O}_3 + 40\% \text{TiO}_2$

After the coating process, the free-ball micro-abrasion wear test was applied to each sample. The free-ball micro-abrasion method is a simple test technique for determining the wear behavior of coatings and is explained in detail in some studies.^{15,18–20} The ball used in this test was made from the AISI 52100 steel and had a diameter of 25.4 mm. Silicon carbide (SiC) was used as the abrasive with three particle sizes (800, 1000 and 1200 mesh). The abrasive slurry that was created was composed of 25 % of SiC and 75 % of distilled water. Each test was repeated three times. During the tests, the abrasive slurry droplet was implemented as one drop per 20 seconds. To determine the experimental test combinations, the abrasive grit size, the test duration, the coating material and the spindle speed were selected as the test factors (**Table 3**). Each factor has three levels except for the coating material that has four levels. A total of forty-five test combinations were carried out in accordance with the factorial design. Conventional characterization techniques, such as scanning electron microscopy (SEM), micro hardness and X-ray diffraction, were

employed to study the microstructure of the coating zone.

Table 3: Factors and their levels

Tabela 3: Dejavniki in njihova veličina

Factor	Level 1	Level 2	Level 3	Level 4
Coating material	Cr ₂ O ₃	Al ₂ O ₃	Al ₂ O ₃ + 13 % TiO ₂	Al ₂ O ₃ + 40 % TiO ₂
Abrasive grit size (mesh)	800	1000	1200	
Test duration (min)	1	2	3	
Spindle speed (r/min)	115	160	230	

3 RESULTS AND DISCUSSION

After the micro-abrasion tests, the volumetric wear values were calculated and so, the influence of each test factor on the results could be determined using an ANOVA table (**Table 4**).

The wear loss values for all parameters are given in **Table 5**. The factors that have significant effects on the mass-loss value of the coatings were determined by analyzing the ANOVA table. Hence, from the ANOVA table, the significance of the factors, according to their effects on the mass-loss values, can be presented in a descending order such as the grit size, the test duration and the coating material. However, the spindle speed does not have a significant effect on the wear behavior, if the selected confidence interval is 95 %. This case can be attributed to the reduced effect of free-mass weight of steel ball with increasing the tangential velocity. Increasing the tangential velocity also leads to a decrease in abrasive effect by applying lower pressure to the surface of coating. The effects of the abrasive grit size and the coating-material factors on the mass loss are given in **Figure 3**.

The volumetric mass-loss values increased in all the samples as a function of the increasing abrasive particle size. Additionally, it was observed that the Cr₂O₃ coating exhibited the lowest degree of abrasion due to its high hardness values contrary to the expectation. This case can be explained by taking into account both the surface roughness and hardness values of coated samples (**Table**

Table 4: ANOVA table for the main factors

Tabela 4: Tabela ANOVA glavnih dejavnikov

Factor	Degree of freedom	Sum of squares	Mean square	F value	Prob>F
Model	9	4.084E-03	4.538E-04	9.24	<0.0001
Material	3	9.217E-04	3.072E-04	6.26	0.0016*
Spindle speed	2	3.012E-04	1.506E-04	3.07	0.0592
Duration	2	9.419E-04	4.710E-04	9.59	0.0005*
Grit size	2	2.031E-03	1.016E-03	20.69	<0.0001*
Residual	35	1.719E-03	4.911E-05		
Corrected total	44	5.802E-03			

*significant factor

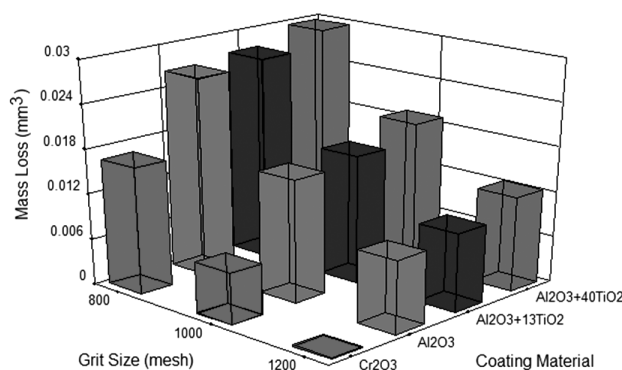


Figure 3: Effect of the abrasive grit size vs. the coating material on the mass loss

Slika 3: Vpliv velikosti zrn abrazijskega sredstva in materiala prevleke na izgubo mase

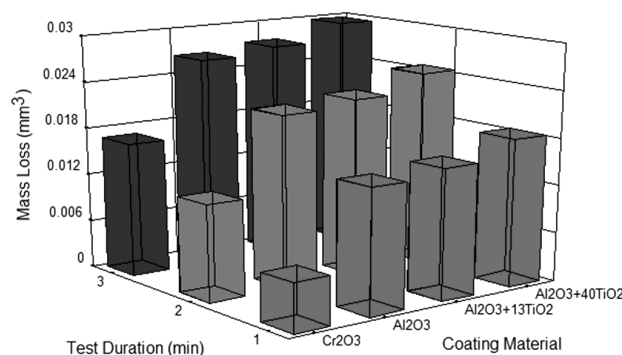


Figure 4: Effect of the abrasive grit size vs. the coating material on the mass loss

Slika 4: Vpliv velikosti zrn abrazijskega sredstva in materiala prevleke na izgubo mase

2), it was observed, contrary to the expectations, that the Cr₂O₃ coating exhibited the lowest degree of abrasion due to its high hardness values. As it is well-known, hardness is one of the most important parameters in increasing the resistance to abrasion. On the other hand, the surfaces with high roughness and hardness values need to be abraded more as they have low rates of toughness. However, the abrasive particles fed into the medium in the form of slurry soon filled in the rough surfaces, thus acting as a bed between the free ball and the material, preventing a rapid abrasion of a sample.

Table 5: Volume loss values for all parameters**Tabela 5:** Zmanjšanje volumna za vse parametre

Run	Material	Spindle speed /(r/min)	Duration /min	Grit size mesh	Volumetric mass loss (mm ³)
1	Cr2O3	160	3	1000	0.004794
2	Al2O3+13TiO2	230	3	1000	0.028231
3	Cr2O3	115	1	1000	0.003041
4	Al2O3	160	1	800	0.015952
5	Cr2O3	160	2	800	0.007424
6	Al2O3+40TiO2	160	1	1000	0.012665
7	Cr2O3	115	3	800	0.008543
8	Al2O3+13TiO2	160	2	1000	0.015395
9	Al2O3+13TiO2	115	2	800	0.019840
10	Al2O3+40TiO2	160	1	800	0.016524
11	Al2O3+13TiO2	230	1	1000	0.008543
12	Al2O3+40TiO2	115	1	1200	0.005107
13	Al2O3+13TiO2	115	1	1200	0.003621
14	Al2O3+40TiO2	230	1	1200	0.007531
15	Al2O3+40TiO2	115	3	1000	0.017919
16	Cr2O3	115	2	1200	0.003747
17	Al2O3+40TiO2	160	2	1200	0.005605
18	Al2O3+40TiO2	230	2	800	0.033143
19	Al2O3	115	2	1200	0.002218
20	Al2O3+13TiO2	160	1	1000	0.007748
21	Al2O3+40TiO2	115	1	800	0.009525
22	Al2O3	115	1	1000	0.010870
23	Al2O3+13TiO2	115	3	1000	0.015952
24	Al2O3	160	3	1200	0.001384
25	Al2O3	230	2	800	0.033470
26	Cr2O3	160	1	1200	0.003380
27	Al2O3+40TiO2	160	3	1000	0.017112
28	Cr2O3	230	1	800	0.007009
29	Al2O3+13TiO2	160	1	1200	0.005188
30	Cr2O3	230	2	1000	0.006231
31	Al2O3+13TiO2	160	3	800	0.039037
32	Al2O3+13TiO2	230	2	1200	0.008543
33	Al2O3	230	1	1200	0.002934
34	Al2O3+13TiO2	230	1	800	0.019840
35	Al2O3+13TiO2	115	3	1200	0.009915
36	Al2O3+40TiO2	230	3	1200	0.012197
37	Al2O3	115	3	800	0.032819
38	Al2O3	160	2	1000	0.018125
39	Al2O3	230	3	1000	0.017112
40	Al2O3+13TiO2	115	1	800	0.009784
41	Cr2O3	230	3	1200	0.004281
42	Al2O3+40TiO2	230	3	800	0.059443
43	Al2O3+13TiO2	115	2	1000	0.010729
44	Al2O3+40TiO2	230	1	1000	0.012508
45	Al2O3+40TiO2	115	2	1000	0.015765

The increase in the mass losses as a function of the increasing grit size can be explained as follows: An increase in the size of the abrasive grit causes an increase in the kinetic energy and the contact area of the grit as a function of speed; thereby, the abrasive capacity is increased in a directly proportional way. It is observed that the factor with the second biggest importance is the test duration. An increase in the rate of fresh abrasers along with the extended duration, in accordance with the abrasive slurry dropping frequency (one drop per 20 s),

caused a further loss in the mass (**Figure 4**). Furthermore, the abrasive grits came into contact with a wider area of the sample surface, along with the sliding distance that increased in direct proportion with the test duration.

Thirdly, taking the coating-material factor into account, Cr₂O₃ demonstrated the highest abrasion resistance when compared to the hardness values. Additionally, the hardness value was reduced, along with the

increase in the TiO₂ percentage in the Al₂O₃ content, resulting in an increase in the abrasion amount.

4 CONCLUSIONS

In this study, the wear behaviors of different types of ceramic coatings were evaluated via the micro-abrasion tests. The conclusions can be summarized as follows: Among the specimens, the ceramic coating including Cr₂O₃ has the highest value of hardness. The other coating-hardness quantities, according to their order of magnitude, are pure Al₂O₃, Al₂O₃ + 13 % TiO₂ and Al₂O₃ + 40 % TiO₂.

The most influential factor for the mass loss was the abrasive grit size. The test duration and the coating material were the other effective factors. An increase in the abrasive grit size led to certain differences in the abrasive surface topography. The influence of the plastic deformation of the sample surface with a mesh grit size of 800 is greater than that of 1 200 mesh.

It was observed that the abrasive mechanism changed with the change in the coating composition. The plastic deformation on the surfaces of the Cr₂O₃ and Al₂O₃ coatings was seen to be more prominent, whereas the increase in the rate of TiO₂ in Al₂O₃ brought about a decrease in the hardness, resulting in a reduced plastic-deformation severity and a smoother surface topography. The spindle speed has not much effect on the value of mass loss due to reducing effect of tangential velocity on the pressure applied on surface of coating by the steel ball. The micro-abrasion technique is a method that can be used for comparative assessments, examining the abrasive behaviours of ceramic coatings.

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