The item deals with the possibilities of utilization HVOF coatings in thermal cyclic loading conditions. There were evaluated three types of coatings based on WC-Co, WC-Co-Cr and Cr₃C₂-25NiCr. The quality of coatings was evaluated in terms of their adhesion as sprayed and also during the cyclic thermal loading, EDX analysis and evaluation of microhardness. Construction and structure of coatings were studied using optical and electron microscopy. There was also evaluated resistance of the coatings against erosive wear.

Key words: HVOF coatings, adhesion, erosion, thermal loading.

Mogućnosti rabljenja visoko brzinskih kisikovih (VBK) prevlaka u uvjetima toplinskog cikličkog opterećenja. Članak objašnjava mogućnosti rabljenja VBK-a prevlaka u uvjetima toplinsko cikličkog opterećenja. Vrijednosti su za tri prevlaka na temelju WC-Co, WC-Co-Cr i Cr₃C₂-25NiCr. Kakvoća analize je provedena na temelju prijanjanja u početnom stanju i tijekom cikličkog toplinskog opterećenja sa EDX elektronskim mikroskopom. Vrijednovane su također otpornost prevlake na erozivno trošenje.

Ključne riječi: VBK prevlake, prijanjanje, erozija, toplinsko opterećenje

INTRODUCTION

Thermally-sprayed coatings belong to the dynamically developing field of surface engineering [1,2]. These high-quality functional coatings are applied in the basic industry, as well as in renovations [3], mainly due to their excellent properties, which are characterized by high wear resistance [4-8], corrosion resistance and resistance against high temperatures [9,10]. Thanks to wide range of different combinations coating-substrate material, thermal spraying offers as many possibilities as no other technology of coatings deposition. HVOF (High Velocity Oxygen Fuel) is one of the technologies, which formed coatings with very small porosity (<1 %) compared with the basic material and high adhesion strength (> 80 MPa). There are minimal thermal changes of substrate during spraying and also roughness of coating surface is low.

Area of utilization thermally sprayed coatings is due to a wide variety of usable materials and their combinations very broad, Figure 1. It is possible to deposit coatings of wide materials spectrum, from pure metals up to special alloys. Resistance of coating based on cermets is determined predominantly by type, morphology and size of hard particles and their volume fraction in a tough matrix.

The item presents experimentally obtained results aimed at assessing selected coatings applied by HVOF technology. The coatings were subjected to cyclic thermal stress. Their tribological properties were evaluated in conditions of erosive wear. The quality of coatings was evaluated by pull-off test, measuring the microhardness, and by EDX analysis. Conditions of experimental works were chosen in order simulate the operating conditions in the iron manufacturing in basic oxygen furnace (BOF).

MATERIALS AND METHODS

Substrate for application the coatings was made of structural carbon steel 12 020 (STN 41 2020, C15E, 1.1141). Chemical composition of the steel is listed in Table 1.

Mechanical properties of the steel substrate: tensile strength 740 – 880 MPa, yield strength ≥ 440 MPa.
Test samples were made from round bar Ø50 mm with a height of 15 mm.

**Substrate pre-treatment**

Test samples were pre-treated by air grit blasting: air pressure of 0.5 MPa, abrasive - brown corundum, grain size 1.0 mm.

**Material of coatings**

There were deposited three types of coatings by HVOF technology on pretreated samples. On the first group of samples coating of WC-729-1/1 343 VM (WC-17Co) was applied, on the second group of samples coating of WC-731-1/1 350 VM (WC-Co-Cr) deposited and on the third group of samples coating CRC-300-1/1 375 VM (Cr3C2-25NiCr) was deposited. Materials were supplied as a powder, agglomerated and sintered, produced by Praxair, Inc., USA. Table 2 shows chemical composition of the powders.

<table>
<thead>
<tr>
<th>Coating</th>
<th>C</th>
<th>Co</th>
<th>Si</th>
<th>W</th>
<th>Cr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-17Co 343</td>
<td>5,5</td>
<td>16,2</td>
<td>0,036</td>
<td>78,4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>WC-Co-Cr 350</td>
<td>5,5</td>
<td>9,9</td>
<td>0,02</td>
<td>80,58</td>
<td>3,9</td>
<td></td>
</tr>
<tr>
<td>Cr3C2-25NiCr 375</td>
<td>10</td>
<td></td>
<td></td>
<td>68,5</td>
<td>21</td>
<td></td>
</tr>
</tbody>
</table>

For the coating deposition equipment JP-5000, Praxair TA was used; it deposits coatings using system HP/HVOF (High Pressure / High Velocity Oxygen Fuel) with System Powder Feeder 1264. The surface of deposited coatings was not further modified after spraying. Parameters of spraying are listed in Table 3.

**RESULTS AND DISCUSSION**

Thickness of the coatings as sprayed, were as follows: 1 343 – 234 μm, 1 350 – 356 μm and 1 375 – 393 μm. The highest microhardness values was shown by coating 1 350 (1 447 HV 0,1) which was caused by a high content of tungsten and addition of cobalt compared to the coating 1 343, which also contains tungsten but at lower concentrations and had lower values of microhardness (1 010 HV 0,1). The lowest microhardness values were shown by coating 1 375 with a high content of chromium, tungsten-free (975 HV 0,1).

Figure 3 shows macro views, fractures, cross-sections and appearance of surface the coatings after thermal cycles and after pull-off test.

To determine the basic properties of coatings microhardness was measured according to STN ISO 4516 on Shimadzu HMV-2E test equipment, load 980,7 mN (10 g), dwell time 15 s. Samples were subjected to cyclic thermal load in electric chamber furnace according to the following mode:

1. heating of the samples in electric chamber furnace at 900 °C,
2. dwell in the furnace for 20 minutes,
3. free cooling of samples on still air to ambient temperature.

Samples were subjected to 10 thermal cycles, and after the 3rd, 5th, 8th and 10th thermal cycle samples were collected to evaluate the adhesion of coatings. Construction, structure and chemical analysis of examined coatings was studied using scanning electron microscopy (SEM) JEOL JSM – 7 000 F. Chemical analysis was conducted using the EDX analyzer INCA, which allows local EDX chemical analysis of the material.

To simulate the working conditions in BOF the impact and flowing of oxides in BOF gas) coatings were subjected to erosion wear in abrasive impact angles 45° and 75°. To simulate the process of oxide impact a laboratory mechanical blasting device KP-1 was used, which allows monitoring the circulation of abrasive. Abrasive used - brown corundum (Al2O3), grain size 1 mm. Intensity of coatings wear was evaluated using gravimetry (mass loss of the coating). Peripheral speed of blasting wheel was 51,0 m/s and output speed of abrasive was 70,98 m/s.

Thickness of the coating was determined by magnetic thickness gauge. Adhesion of coatings was evaluated by pull-off test according to STN EN 582 with help of tensile machine ZDM 10/91.

After pull-off adhesion test, tensile stress necessary to rupture the weakest inter-phase (adhesive fracture) or the weaker component (cohesive fracture) of the test arrangement and also the nature of fraction were determined. Character of fracture was evaluated according to the scheme in Figure 2.
EDX spectral analysis of the coating 1 343 showed the presence of two basic phases - solid particles WC and cobalt surrounding WC particles, which corresponds to the chemical composition of powders for coatings production. EDX spectral analysis of the coating 1 350 shows the presence of WC particles and chrome and cobalt matrix surrounding WC particles. EDX spectral analysis of the coating 1 375 again confirmed the presence of large particles of Cr3C2 and the most extensive component of coating 1 375 - nickel-chromium matrix. Matrix and hard particles of WC and Cr3C2 are well visible on cross-sections and also on fractures of the coatings, Figure 3.

Despite its high hardness, coating 1 350 after 3 thermal cycles showed thermal cracking, Figure 3 – surface after thermal cycles. Surface of coating 1 343 during the

**Figure 3** Macro views, fractures, cross-sections and appearance of surface the coatings after thermal cycles and after pull-off test
thermal cyclic loading was covered with a layer of blue oxides with a strong chalking. Coating 1 375 after thermal cycles retained its aesthetic and tactile qualities. Before pull-off test of the coating, all releasing layers were mechanically removed and samples were degreased with methanol. The surface appearance of coatings during thermal cyclic loading and character of coatings fracture are also shown in Figure 3. Results of coatings adhesion evaluation are shown in Figure 4.

The above results show that the adhesion of coatings has decreased already after three thermal cycles, which remained almost stable during the next thermal loading. The measured values of the adhesion of the coating as-sprayed and the type of fracture showed that in neither case the damage of the coating occurred, therefore the observed initial adhesion values do not correspond to the actual adhesion of coatings, which is definitely higher. Due to very high adhesion of coatings formed by HVOF technology is difficult to determine a real adhesion, because the properties of adhesive used are limiting factor. Although during thermal cyclic loading the coating 1 343 showed a fracture in the coating (90 % B), in fact after the pull-off test on the test dolly side only a very thin layer of coating remained due to strong chalking and oxidation and we cannot talk about the fracture in coating volume. The coating 1 350 after pull-off test has cut off from the substrate along crack area.

The coating 1 375 has not been fractured after pull-off test, so we expect a higher adhesion than the value listed for this coating.

Figure 5 depicts the dependence of erosive wear on the different impact angles of abrasive. For all types of coatings very similar dependences were achieved. Higher weight losses were recorded at an impact angle of 75° in all types of coatings. The references show that for the harder material, which also the evaluated coatings belong to, more intensive wear occurs at a larger impact angles, as confirmed by experiment.

Figure 6 shows 3D appearance the surface of coating 1 343 after erosion test at impact angle 45° (Figure 6a) and 75° (Figure 6b). 3D views were obtained by confocal microscope. Intensity of erosive wear is influenced mainly by the ratio of the coating and abrasive hardness together with the structural characteristics of the coating. Wear intensity of all evaluated coatings was almost the same, higher at impact angle 75°. More complex surface was reached at 75° impact angle. There we can see a displacement of material and creating new configuration of extrusions corresponds to the direction and shape of the incident abrasive.

At larger impact angles, forging effect of the abrasive prevails while at smaller impact angles prevails grooving effect of the abrasive. Wear mechanism of coatings also corresponds to the mentioned facts.

CONCLUSION

Based on the results of the experiments it can be said that the coating 1 350 (1 447 HV 0,1) showed the highest hardness and the coating 1 375 (975 HV 0,1) showed the lowest. To the environment of BOF with high and fluctuating temperatures coating 1 350 cannot be applied, because of its cracking after a few thermal cycles and thereby disruption of its barrier protective effect what creates a precondition for high temperature corrosion of the substrate. In high temperature the coating 1 343 showed strong chalking, which may cause significant losses in weight (and consequently in thickness) of
the coating and its low durability. Coating 1 375 compared with the previous coatings showed a lower hardness, but during the thermal cyclic loading maintains its integrity and adhesion, any other qualitative changes didn’t occur. Resistance to erosive wear of all coatings is approximately the same.

Based on the experimental results obtained, it is possible to recommend for renovation components stressed by extremely high and cyclic temperatures and erosion coating 1 375 (Cr3C2-25NiCr).

Acknowledgement

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Note: Linguistic Adviser / English language Jozef Brezina, Košice, Slovakia.