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FAILURE MECHANISM AND MECHANICAL CHARACTERISTICS OF NiCrBSi COATING

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Abstract

Mechanical properties of NiCrBSi coatings on plain carbon steel substrate prepared by HVOF (High velocity flame spraying) were studied in this work. Sprayed coatings with average thicknesses of 0.25 mm, 0.5 mm and 1 mm were prepared on preheated steel strips and steel strips without preheating. Chemical analysis of the coatings cross-section offered information about material structure and distribution of chemical elements within the coating. Hardness of the coating was measured with the aim to reveal the influence of coating thickness and coated substrate preheating on coating hardness. Similar hardness was observed regardless coating preparation conditions and thickness. Bend test was used for characterization of failure character of NiCrBSi thermally sprayed coatings under different thermal conditions. Higher coating thickness was beside cracking, accompanied also by coating delamination from the substrate, while also some influence of testing temperature (laboratory temperature vs. -50 °C) on coating failure characteristics were observed. No influence of the coating preparation conditions on the coating pull-off strength was observed.

Keywords: thermal spraying, NiCrBSi coating, bend test, coating failure mechanism, cracks to centimetre ratio, microstructure, HVOF

INTRODUCTION

The method of thermal spraying is a process where coatings of metal, ceramic or cermet material are deposited on the metal substrate (Ambrož, 1990; Kraus, 2000; Heimann, 1996). The deposited material is coated in the molten state, partly molten state or non-molten state. Depending on the used method of spraying, the deposited materials are in the form of powders of different granularity, wires, rods or liquid suspensions (Pawlowski, 2008). The metal substrate is heated to the temperature of around 100 °C during the coating process. It eliminates the deformation of substrate and degradation of its structure (Kraus, 2000). The pre-treatment of the substrate is necessary before the coating deposition. Inclusions and corrosion products are

removed from the surface of the substrate during the pre-treatment process. Blasting is the most used method to treat the substrate before the deposition. Corundum, ballotini, steel or glass grit or cast-iron balls are the most used abrasives for blasting depending on the substrate. Significantly thicker coatings can be reached by thermal spray deposition in comparison with the methods like Chemical Vapour Deposition (CVD), Physical Vapour Deposition (PVD) or electroless plating at the same deposition time (Fauchais, 2008). Methods of thermal spraying can be divided into two main groups - methods using the mixture of fuel and oxygen (flame spraying, D-gun or HVOF) and methods using the thermal energy of electrical source (electric arc spraying or plasma spraying) (Mohyla, 1995).

HVOF method, as a type of thermal spraying, uses the mixture of fuel and oxygen and uses lower process temperature in comparison to plasma spraying (Yilbas, 2005; Espallargas, 2015; Schneider, 2006). The temperature of the feed particles accelerated and heated by the burning fuel decreases as a result of their high velocity and the cooling effect of the air through which the particles move to the substrate. The feeding coating material is used only in the form of powder (García-Rodrígues, 2016). The powder is introduced into the supersonic flame via gas stream. Due to the high speed, deposited particles are uniformly spread and fixed to the surface of the coated substrate. This positively affects the adhesion of the coating to the substrate and the cohesion of coating (Fauchais, 2008). The porosity of the coating is low and the density of the coating is high (Yilbas, 2005; Espallargas, 2015; Schneider, 2006; Taltavull, 2015). Under the proper spraying conditions, compressive stresses can be created within the coating, which positively affects fatigue properties of deposited coatings (Fauchais, 2008; García, 2011). This method has high productivity and a wide range of deposited materials can be used. One of the possible ways how to increase the corrosion and wear resistance is application NiCrBSi coatings by HVOF (high-velocity oxygen fuel) or APS (atmospheric plasma spraying) method (Milanti, 2015).

Ni-based coatings are used in cases, where the increase of wear resistance while increasing the hot corrosion resistance is necessary. They are also most frequently used in automotive, aerospace or petrochemical industry (Houdková, 2014). Chromium, as another component of the NiCrBSi coating, improves the oxidation and corrosion resistance (Houdková, 2014). Chromium with a combination of other elements in coating increases the hardness due to the formation of hard precipitating phases in the coating. Boron, as the chromium, contributes to the formation of hard phases. Boron with silicon reduce the melting point due to the presence of a eutectic phase at 3.6 wt. %. The addition of boron and silicon increases the self-fluxing capabilities of Ni-based coatings (Miguel, 2003; Shabana, 2015). Silicon forms hard carbides in coatings. These carbides increase the resulting hardness, wear and abrasion resistance of NiCrBSi coating. Further increase in hardness could be reached by incorporation of hard carbides, such as WC, TiC, B_4C, etc. into the coating (Al-Mutairi, 2015; Watanabe, 2006; Mayrhofer, 2015).

This paper deals with the mechanical properties of the coating and with the mechanical failure characteristics under different conditions of the measurement. Coatings properties were tested under temperature gradients which can be encountered in an industrial environment. Quantitative crack propagation and failure mechanism after the thermal cycles as a life prediction methodology of NiCrBSi coatings was studied.

MATERIALS AND METHODS

Experimental procedure

Substrate material and spraying conditions

Carbon steel strips with dimensions 100 × 20 × 3 mm were used as substrates for the deposition of NiCrBSi coating. Surfaces of substrates were blasted using brown corundum (F36 mesh) to reach uniform surface roughness and air-dried during the pre-treatment before coating application. Some of the mild steel strips were preheated to 100 °C to favour the residual thermal stress reduction. The steel strips were thermally sprayed using HVOF JP-5000 gun under the following conditions: Fuel flow (kerosene): $21 \cdot 8\ l \cdot h^{-1}$, Oxygen flow: $920\ l \cdot min^{-1}$, Nitrogen pressure: 6 bar, Spraying distance: 360 mm.

Applied powder was provided by FST b.v. The powder (composition: Ni balance, 16% Cr, 4% Fe, 3% B, 4.25% Si and 0.7% C) had a spherical morphology, provided by the gas atomization process, with a typical size – 53 + 20 μm.

NiCrBSi coating was applied on steel strips from the powder based material mixture at elevated temperature. The average thicknesses of prepared coatings were 0.25 mm, 0.5 mm and 1 mm.

Coating structural characterization

Prepared coatings were studied using scanning electron microscope SEM ZEISS EVO LS-10 with EDS Oxford Instruments Xmax 80 mm^2 detector and the AZtec software. Chemical analysis of coated specimens cross-sections was performed with the aim to characterise chemical homogeneity of prepared coatings and eventually reveal elemental segregation.

The porosity of prepared coatings was evaluated on their cross-sections in three different locations using the image analysis (ImageJ software).

Coatings mechanical properties and failure analysis

LECO AMH43 automatic Hardness Tester was used for coatings hardness measurement. The load 1000 g with a dwell time of 10 s according to the standard ISO (ISO 6507-1:2005E, 2005) was adopted for coatings characterization. The measurement was performed on coatings cross-sections to eliminate the influence of substrate in the case of low coating thickness. The measurement was performed on all the specimens; covering each coating thickness, preparation conditions (substrate without preheating and with preheating) and bend test conditions.

For the coating cracking and failure mechanism characterization, four sets of coated strips were chosen. According to literature (Harding, 1987) all specimens were bent to 10° angle on a cylinder with the diameter of 25 mm. Two sets of specimens were used to identify the influence of the testing temperature on the coatings behaviour. The third set of specimens was used to analyse the influence of thermal cycling on the coatings characteristics and the fourth set of specimens was used to identify the influence of coated substrate preheating and thermal cycling on coating characteristics. Thermal cycling consisted of steps of cooling the coated specimen to the temperature of –50 °C and following reheating to the laboratory temperature (± 20 °C) five times (5 cycles) in a row. The bending test was either conducted at specimens reheated to the laboratory temperature and on the specimens cooled to –50 °C after applied 5 heating cycles (marked as the temperature at bend test).

First two sets included specimens with coatings with thicknesses of 0.25 mm and 0.5 mm prepared on steel strips without preheating. Bend tests were performed at laboratory temperature and after cooling the specimens to the temperature of –50 °C. The third set of specimens contained specimens with coatings with thicknesses of 0.25 mm, 0.5 mm and 1 mm prepared on steel strips without preheating. The fourth set contained specimens with coatings with thicknesses of 0.25 mm, 0.5 mm and 1 mm prepared on steel strips with preheating. The bending tests were conducted on specimens after the thermal cycling. The last two series of specimens were prepared to evaluate whether the preheating, or thermal cycles have any influence on the cracking progression of the coating during bending.

The failure characteristics of coatings were studied on bent specimen cross-sections using the inverted light optical microscope Zeiss Axio Observer Z1m. As a useful parameter, to evaluate the failure characteristics of coatings, a crack to centimetre ratio was introduced. This parameter was calculated from the equation (1):

$$C = \frac{n}{d}, \tag{1}$$

where C is the calculated ratio, n is the number of cracks caused by bending and d is the distance between the first and the last crack in centimetres measured in the coating.

The pull-off strength of coatings with thicknesses of 0.25 mm, 0.5 mm and 1 mm was tested according to the ASTM A370 standard. The test was performed on NiCrBSi coatings prepared on the top of steel cylinders (without and with preheating) with a diameter of 40 mm. Two specimens coated at the same conditions were fixed together (coating on coating) using extra strong adhesive (MasterBond) capable of sustaining pulling force of 80 MPa. The adhesion strength between NiCrBSi coating and the coated steel substrate was determined using Instron 5985 machine with the test strain rate 1 $mm \cdot min^{-1}$.

RESULTS AND DISCUSSION

Coating chemical composition

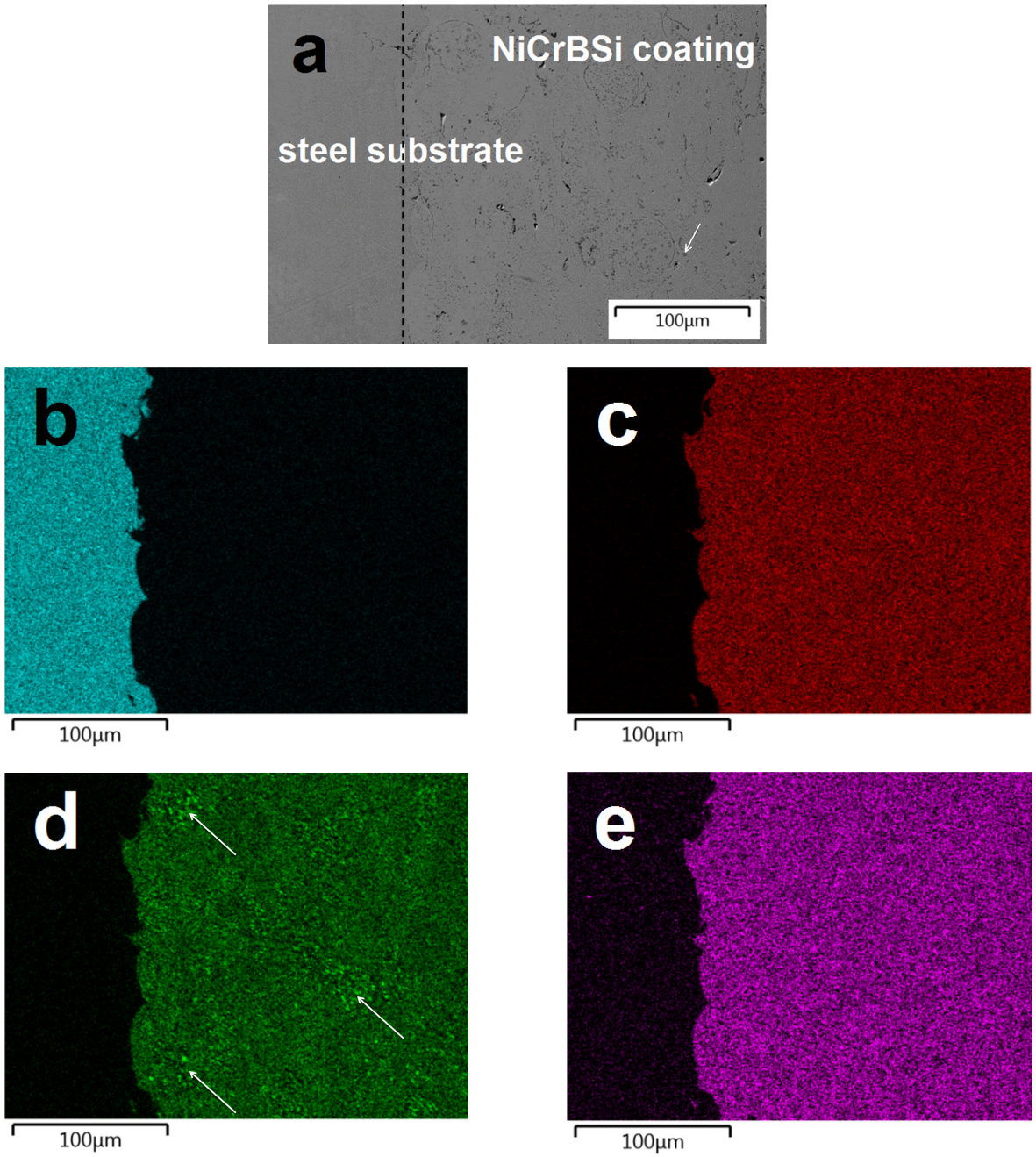
Characterization of the NiCrBSi coating prepared by thermal spraying on plain carbon steel was performed with the aim to estimate the influence of coating thickness (0.25, 0.5 and 1 mm). Sprayed coating with the average thickness of 0.25 mm was analysed using SEM and the EDS analysis (Fig. 1). Some microstructural features and porosity were characteristic for the coatings (Fig. 1a). The average porosity value for all coatings was ~2 %. From Fig. 1a can be observed some dark spots corresponding to borides Cr_2B and oxides of Cr and Ni (Sharma, 2015). EDS analysis revealed

that the distribution of Ni and Si was homogeneous in the entire cross-section of the coating (Fig. 1c and e). The local increase of intensity in the case of chromium was observed in the coating (Fig. 1d), while this effect can be attributed to the local formation and segregation of chromium intermediate phases in NiCrBSi coating (Fig. 1a).

The same microstructure and elemental distribution were observed for all the thicknesses of the prepared coatings (0.25, 0.5 and 1 mm) while no influence of coated steel preheating on coatings microstructure or chemical analysis results was observed. In all the cases the presence of chromium phases was revealed by the EDS analysis.

Hardness of deposited coatings

The hardness of prepared coatings was measured on all the prepared specimens' cross-sections, covering each coating thickness (0.25, 0.5 and



1: *Microstructure and EDS mapping analysis of thermally sprayed NiCrBSi coating with the thickness of 0.25 mm prepared on specimen without preheating,*
a) Microstructure of NiCrBSi coating in the cross-section, b) Iron, c) Nickel, d) Chromium, e) Silicon

I: *Individual hardness values of specimens with different thickness*

Coating thickness [mm]	As-sprayed	Thermal cycling	Pre-heated substrate
0,25	670 ± 60	670 ± 40	650 ± 60
0,5	710 ± 60	720 ± 40	–
1	700 ± 60	680 ± 50	660 ± 40

1 mm), preparation conditions (substrate without preheating and with preheating) and bend test conditions. Resulting average value of hardness of all the specimens was determined as 690 ± 50 HV 1 (Tab. I). No influence of coating thickness, coated substrate preheating or temperature (eventually heat cycling) used for the bending testing on the hardness of the coatings was observed. However, the hardness is strongly influenced by the presence of pores and secondary phases in the as-sprayed coating.

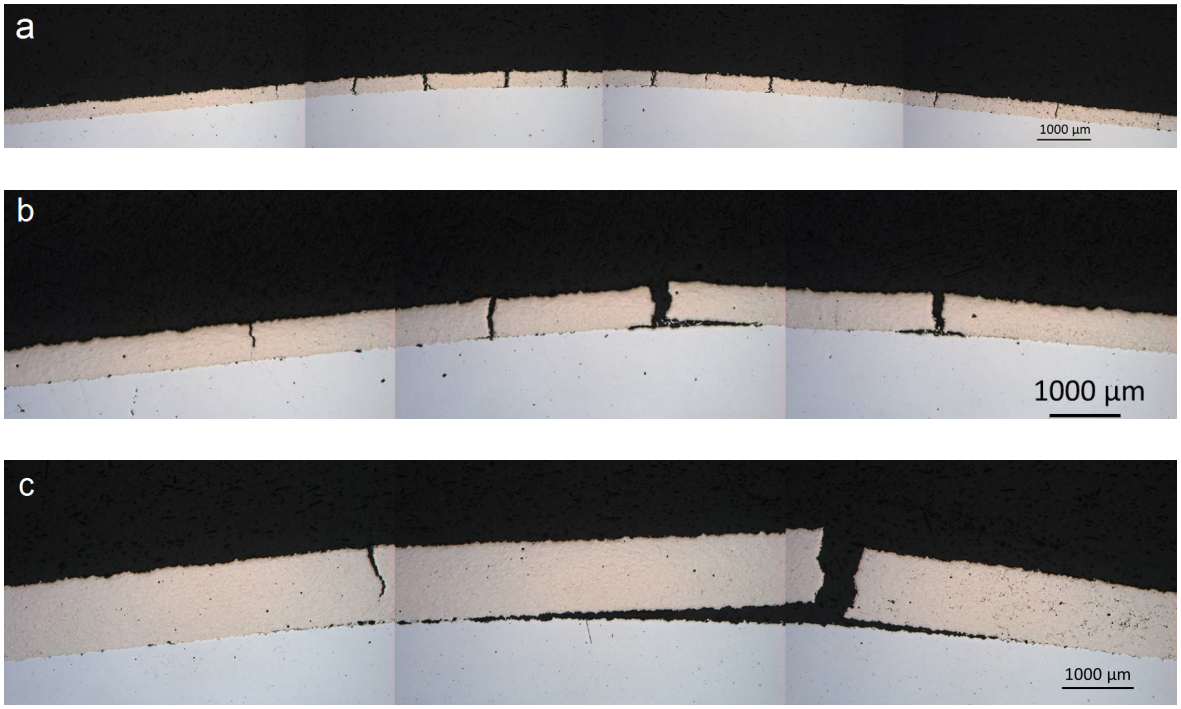
The quite high standard deviation can be connected to the porosity of the coating and the presence of borides and oxides in the coating. The calculated porosity of analysed coatings was approximately 2% (Tabs. II-V). It was found that the differences between porosity of individual coatings are not correlating to any coating preparation parameters (coating thickness, preheating of substrate steel) while different values of porosity were obtained in different parts of the same coating. The presence of porosity is to be expected in these types of coatings, as it is almost impossible to prepare fully compact coating from extra hard compounds (powder used for the coatings preparation) with limited plastic properties of the powder particles. High level of pores can have negative influence to the mechanical, frictional and corrosion properties of the coated materials.

Cracking properties

The coatings cracking after bending the coated specimens to the angle of 10° is documented in Fig. 2. Bending of the coating with the thickness of 0.25 mm resulted in a formation of several cracks in the deformed area (Fig. 2a). With increasing coating thickness, the number of the crack formed due to the bending loading decreased, however, coating spallation and delamination occurred (Fig. 2b-c). Dividing the number of cracks by the length in which they appeared (Equation 1) give a useful parameter for estimation of the effect of coating thickness, the temperature of the specimen during the bending test and preheating of the specimen on the coating cracking progress. The bending tests conditions and the obtained cracks to centimetre parameter are provided in Tabs. II–V. Bending test revealed a strong correlation between the formation of cracks (coating failure) to the coating and bending test temperature. Decreasing the bending test temperature from laboratory temperature (approximately 20 °C) to –50 °C led to increase of the cracks to centimetre parameter of approximately by 50%. Hard ceramic coatings are generally brittle and sub-zero temperatures lead to even greater susceptibility to brittle deformation (Karihaloo, 1996). The increase in the number of cracks to centimetre parameter give similar results for the 0.25 mm thick coating as for 0.5 mm thick coating, but the character of crack differ Fig. 2. In the coating with the thickness of 0.25 mm many small cracks formed in the bend area, however, in the thicker coating (0.5 mm) few small linear cracks similar to the cracks in the mm coating but also 2 large cracks developed during the bending loading. Delamination of the coating substrate regardless of the bending temperature was a mechanism accompanying the large cracks present in the coating, Fig. 2b. This behaviour can be attributed to the higher cohesive strength of the thicker coating than the adhesive strength to the substrate (Fauchais, 2008). In the case of 1 mm thick coatings, only one crack was formed through the entire coating. From this vertical crack, the horizontal crack spread along the interface coating/substrate (Fig. 2c).

In sub-zero temperatures and thick coating the cohesive strength of the coating itself completely dominate over the adhesive strength of the coating to the substrate, therefore only a lower number of cracks can be developed and mostly delamination from the substrate occurred with the increasing thickness of the coating (Fig. 2). Sub-zero conditions (–50 °C) led to significant changes in the cracking progress in all coating thicknesses during the bending test.

Preheating of the coated substrate neither coated specimens heat cycling did not reveal any significant influence on coatings cracking (Tab. IV and V).



2: *Coating failure after the bending test conducted at laboratory temperature on specimens coated without substrate preheating, a) 0.25 mm, b) 0.5 mm, c) 1 mm*

II: *First series of specimens with 0.25 mm coating without preheating*

Specimen	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8
Temperature at bend test (°C)	20	20	20	20	–50	–50	–50	–50
C – cracks to centimetre	6	7	7	7	10	10	11	10
Porosity (%)	2.2	2.0	2.1	1.6	2.4	2.1	1.5	2.4

III: *Second series of specimens with 0.5 mm coating without preheating*

Specimen	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8
Temperature at bend test (°C)	20	20	20	20	-50	-50	-50	-50
C – cracks to centimetre	2	2	2	3	5	5	3	4
Porosity (%)	2.7	2.7	2.7	2.3	2.8	2.2	1.3	2.6

IV: *Third series of specimens coated substrate without preheating*

Specimen	4.1	4.2	4.3	4.4	4.5	4.6	4.7	4.8	4.9	4.10	4.11	4.12
Coating thickness (mm)	0.25	0.25	0.25	0.25	0.5	0.5	0.5	0.5	1	1	1	1
Thermal cycles	0	5	0	5	0	5	0	5	0	5	0	5
Temp. at bend test (°C)	20	20	–50	–50	20	20	–50	–50	20	20	–50	–50
C – cracks to centimetre	9	8	9	10	2	5	4	5	2	2	1	1
Porosity (%)	1.7	1.3	2.5	2.6	1.1	2.2	1.8	0.5	0.9	2.3	1.7	1.8

V: *Fourth series of specimens coated substrate with preheating*

Specimen	3.1	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	3.10	3.11	3.12
Coating thickness (mm)	0.25	0.25	0.25	0.25	0.5	0.5	0.5	0.5	1	1	1	1
Thermal cycles	0	5	0	5	0	5	0	5	0	5	0	5
Temp. at bend test (°C)	20	20	–50	–50	20	20	–50	–50	20	20	–50	–50
C – cracks to centimetre	9	8	7	10	3	4	5	5	2	2	2	2
Porosity (%)	1.6	2.2	2.8	2.6	2.8	1.7	2.7	1.3	0.5	2.2	2.1	1.9

Pull-off strenght of coating

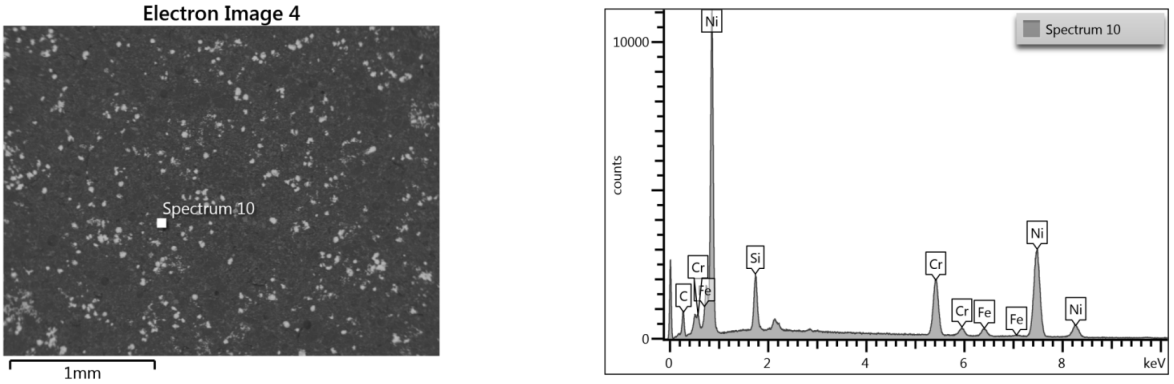
Results of pull-off tests are given in Tab. VI. No significant influence of the thickness of coatings prepared on steel substrates without preheating on coatings adhesion was observed. All the tests specimens (coated substrates) reached the pull-off strength of approximately 50 MPa. However, delamination of tested samples occurred in the adhesive during the test. Similar adhesion values (40.4 MPa) were obtained at work (Coddet, 1998). However, the authors noted that during the pull-off tests the coating delaminated from the substrate. This phenomenon in our experiment was not observed. Only the delamination of the adhesive from the coating occurred. In paper (Li, 2002) is stated that the feedstock powder size of the NiCrBSi has a noticeable effect on the adhesion of the as-sprayed coating. If the powder size is + 45 – 74 μm, the adhesion value is around 35 MPa. For a + 75 – 104 μm powder, the adhesion will increase to almost double (67 MPa).

A small influence of the coated substrate preheating before coating application on coatings pull-off strength was observed. However, in the case of the coating with the thickness of 0.5 mm a large scatter of data for specimens prepared by coated substrate preheating was observed.

Furthermore SEM and EDS analysis of the failure specimens revealed, that the delamination of the adhesive occurs on the surface of the coating

VI: *Pull of strength of coatings with different thickness*

Specimen	Preheating (°C)	Coating thickness (mm)	Pull-off strength of the coating (MPa)
1	no	0.25	50 ± 4
2	100	0.25	57 ± 2
3	no	0.5	51 ± 3
4	100	0.5	33 ± 12
5	no	1	49 ± 9
6	100	1	47 ± 11



3: *SEM and EDS analysis of sample after pull-off test, side with coating, 0.25 mm thickness, steel preheating to 100 °C*

(Fig. 3), as from the coating side, some particles of the coating were visible, yet no trace of coating particles were found on the side of steel specimen, therefore the cohesion of the particles was greater than adhesion to the glue. Which indicate that the test results cannot be used for coating characterization, however, no influence of the coating preparation conditions on the coating pull-off strength were observed. Dark area from Fig. 3 corresponds to the adhesive and the white spots correspond to the underlying coating (which is confirmed by the EDS spectrum).

CONCLUSION

NiCrBSi coatings were prepared on carbon steel by thermal spraying. Three different thicknesses of coatings thickness (0.25, 0.5 and 1 mm) prepared on carbon steel substrate were analysed with the aim to estimate the influence of steel substrate preheating and the temperature of the bending test (laboratory temperature and –50 °C) on coatings properties.

- Prepared coatings were chemically homogenous in all the coating thicknesses and the coating on carbon steel substrates with or without preheating. Chromium rich phases segregation in the coating were observed.
- Observed porosity of specimens was approximately 2%. Thermal cycling, bending test at -50 °C or preheating the steel strips prior coating had no effect on coatings porosity.
- Decreased temperature (–50 °C) during bending test had a significant influence on failure mechanism of all the coating thicknesses (0.25, 0.5 and 1 mm).
- Preheating of the coated substrate neither coated specimens heat cycling did not reveal any significant influence on coatings cracking mechanism during the bending test.
- Increasing the coating thickness led to change of coating failure character. The increase of the coating thickness led, besides the coating cracking, to delamination of the coating from the substrate.

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