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Investigation of the Influence of NiBSi/NiCrBSi Coatings Applied by Flame Spraying with Simultaneous Fusing on the Substrate Material

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Abstract: The aim of this research is to investigate the influence of nickel alloy type from the same group, the parameters of flame spraying, as well as the preparation of the substrate and the heat treatments of the substrate on the microstructure of the coating/substrate system. Due to the possibility of applying nickel alloys in corrective maintenance of tools used on elevated working temperatures, hot work tool steel X38CrMoV5-1 was selected as a substrate material. The investigation of the microstructure of the coating/substrate system was carried out according to the factorial design of experiment, where the input factors were varied on two levels. The factors that were varied are: Ni-based self-fluxing alloys - NiCrBSi and NiBSi; distance of the burner from the workpiece - small (6 mm) and large (20 mm); preparation of the substrate - roughened and non-roughened and the heat treatments of the substrate - soft annealed and tempered condition. Ni-based self-fluxing alloys were applied on samples (12,5 × 25 × 25 mm) by flame spraying with simultaneous fusing process. Analysis of the microstructures of the coating/substrate system was carried out on the Leica DM 2500M light microscope. After the conducted analysis the paper concluded that by spraying the selected coatings onto the X38CrMoV5-1 tool steel base, poor quality coatings are obtained, due to the appearance of cracks (NiCrBSi) or separation of the coating from the substrate (NiBSi). This is attributed to the formation of martensitic structure of the substrate after spraying and the presence of residual stresses.

Keywords: flame spraying with simultaneous fusing; hot work tool steel X38CrMoV5-1; microstructure; Ni-based self-fluxing alloys

1 INTRODUCTION

In order to extend the service life of damaged and worn parts and subsequently aim to reduce maintenance costs, thermal spraying processes can be used to apply different types of coating materials to different substrate materials. Thermal spraying is widely used in almost all branches of industry due to the large number of substrate materials on which coatings can be applied. There is also a large number of coating materials that can be applied, but the most commonly used and researched are coatings based on nickel, iron, and cobalt. Due to their exceptionally good properties, nickel-based coatings are widely used in conditions where good wear resistance, capability to work on elevated temperatures, and corrosion resistance are required. Nickel alloys are suitable for all thermal spraying processes: flame spraying, HVOF, HVAF, a detonation gun, an electric arc, plasma spraying, supersonic plasma spraying, plasma spraying with a transferred arc and laser [1-11], but after the spraying process, a subsequent fusing procedure is often applied, in order to reduce porosity and create a metallurgical connection between the coating and the substrate [1-4].

Due to their wide applicability, flame spraying and nickel-based coatings have been researched in almost all aspects, from resistance to various types of wear and corrosion to researching the influence of external mechanical loads and residual stresses [12-14]. All the aforementioned research was almost always accompanied by microstructure analysis. In almost 90% of microstructure research, the authors analysed mainly the microstructure of the coating. Some authors do not even mention the type of substrate or state it in general (for example, carbon steel or low-carbon steel), which can be understandable when investigating a property that is important only for the coating (i.e., the resistance of the coating to different types of wear). For example, in papers [15-18], where the material of the substrate is mentioned, it is most often low-carbon steel, and the impact of spraying nickel alloys on the material of the substrate itself is not mentioned. Hot work tool steel was chosen as the substrate in a few papers. Hot work tool steels are subjected to different types of wear and thermal fatigue [19, 20], and their protection with different types of coatings has been investigated in order to extend their service life [21-23]. The systematic application of surface engineering procedures for improving the properties of hot work tool steels is presented in the paper [21], in which it is stated that the application of nickel alloys by flame spraying is a possibility. In the paper [23], the results of the investigation of the properties of the NiCrBSi+Ni/MoS2 coating previously applied and then fused using a laser on hot work tool steel substrate (H13, X40CrMoV5-1, Utop Mo2) were presented, but without investigating the effect of the process on the substrate. Difficulties in applying flame spraying for corrective maintenance of molds for high-pressure casting of aluminium castings, which are made of tool steel for working in a hot state, are stated in the paper [24].

Due to the above-mentioned small number of papers investigating the influence of the spraying process, type of coating, spraying parameters, and type of substrate on the microstructure of the coating/substrate system, this paper aims to investigate the effects of the mentioned parameters on the microstructure of the coating/substrate system. The investigation of the microstructure of the coating/substrate system was carried out according to a factorial design of the experiment where four factors were simultaneously changed on two levels: the type of coating (NiBSi and NiCrBSi), the spraying distance during the deposition during flame spraying with simultaneous fusing (small and large), substrate preparation (roughened and non-roughened) and heat treatment of the substrate (soft annealed and tempered condition).

2 EXPERIMENTAL PART

Two types of coatings from the same group of nickel alloys - NiBSi and NiCrBSi (Fig. 1) were applied by flame spraying with simultaneous fusing using Super Jet Euttaloy oxy-acetylene gun (acetylene pressure 50 kPa and oxygen pressure 200 kPa) on samples measuring 12,5 × 25 × 25 mm (substrate - hot work tool steel) [14].

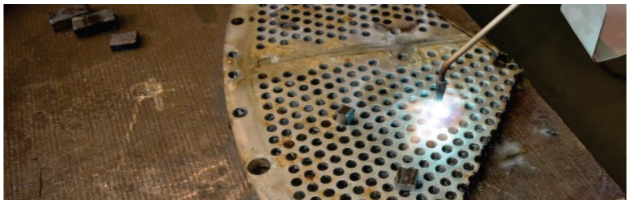


Figure 1 Spraying process using a gas flame with simultaneous fusing

The thickness of the coating is 1 mm and is applied through eight passes. Hot work tool steel X38CrMoV5-1 (Utop Mo1, W300, H11, Č4751, 1.2343) is a high alloy tool steel and one of the most commonly used steels, not only as a hot work tool steel but also as ultra-high strength structural steel. The applied steel has a favourable combination of toughness, hardness, wear resistance, yield resistance with a high limit of elasticity and strength on the elevated temperature and it is air hardening steel [19, 25].

Tab. 1 shows the chemical composition of the substrate material - hot work tool steel X38CrMoV5-1, while Tab. 2 shows the chemical composition of the spraying powder.

Table 1 Chemical composition of the substrate material

Chemical element, % w.t.	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	V	Fe
X38CrMoV5-1	0,36	0,93	0,35	0,018	< 0,001	4,74	0,37	1,00	0,19	0,27	remaining

Table 2 Chemical composition of the spraying powder

Chemical element, % w.t.	C	Cr	Fe	B	Si	Ni
NiCrBSi (*Eutalloy 10009, BoroTec*)	0,7	15	3,5	3,2	4,4	remaining
NiBSi (*Eutalloy 10185, BronzoChrom*)	0,1	0,5	0,5	2,5	3	remaining

The microstructure of the coating/substrate system was investigated according to the factorial design of the experiment, where four factors were changed simultaneously on two levels with two repetitions of combinations of factor levels. The factors changed on two levels are the type of nickel alloy from the same group (type of coating) - NiCrBSi and NiBSi, the spraying distance during the deposition - small (6 mm) and large (20 mm), substrate preparation - roughened and non-roughened and heat treatment of the substrate - soft annealed and tempered condition. Since the spraying process using a gas flame with simultaneous fusing creates a metallurgical connection between the coating and the substrate, the substrate preparation factor is defined on two levels. In addition to the standard preparation of the substrate (level - roughened), as the second level of this factor, it was chosen that this standard preparation will be omitted, and the substrate will be cleaned from impurities and fats only with ethyl alcohol (level - non-roughened). The roughening of the substrate has a significant impact in the so-called cold spraying process using a gas flame, in which a mechanical connection is formed between the coating and the substrate [26]. It can also have a significant impact on the two steps flame spraying process of the investigated alloys - when the alloys are first sprayed with a classic cold process (where the spraying distance from the workpiece during the deposition is up to 220 mm, or even 300 mm [27] and the coating is connected to the substrate by mechanical connection [16, 18, 28], and then subsequent fusing is applied). As the paper investigates the process of spraying using a gas flame with simultaneous fusing (hot process in one step), where the spraying distance from the workpiece is ten and more times less than in the cold process, the coating and the substrate are not connected by mechanical connection, but rather by heating the substrate material to the fusing temperature that allows the diffusion of chemical elements between the coating material in a semi-molten state (between solidus and liquidus temperature) and the substrate material and creation of metallurgical connection. Thus, in research [29, 30], the authors even prepared the substrate to extremely low roughness - mirror finish, with the aim of avoiding sand-sandblasting, which can affect the deterioration of the dynamic properties. The heat treatment of the substrate was chosen as a factor due to the frequent case of applying coatings on heat-treated materials in practice, mainly in corrective maintenance, and due to the observed smaller number of studies related to the impact of the spraying process on the applied substrate, particularly on the pre-heat treated substrate [31, 32]. For this factor (heat treatment), along with the level of tempering of tool steel, the soft annealed state was chosen as the second level. Hot work tool steels are not used in a soft annealed state, but this process allows machining before the tempering. In this paper, however, this condition was chosen as a level, with the aim of proving whether, with the spraying process using a gas flame with simultaneous fusing (the temperature of fusing is similar to the austenitizing temperature of the selected tool steel), a significant change in the structure of the substrate is possible - due to the fact of good quenchability of this type of steel even in case of slow cooling, which is the case after the applied spraying process.

The microstructure of the coating/substrate system was investigated according to the test plan shown in Tab. 3.

2.1 Preparation of Samples for Metallographic Analysis

Fig. 2 shows the dimensions and shape of the samples for testing the microstructure of the coating/substrate (X38CrMoV5-1 steel) system after the spraying process.

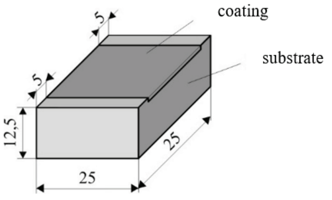


Figure 2 Dimensions and shape of the samples for testing the microstructure of the coating/substrate system

In order to obtain the surface required for metallographic analysis, after the spraying process, the samples were cross-cut on a wire EDM machine (Fig. 3) with intensive water cooling, and then the surface was prepared for metallographic analysis.

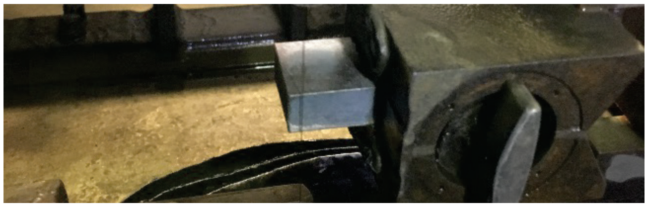


Figure 3 Cutting the sample on the wire EDM machine

All samples were etched with a nital, the aim was to etch the structure of the substrate and not of the coating. Microstructure imaging was carried out on the Leica DM 2500M light microscope according to the test plan shown in Tab. 3, where the Sample column represents the sample labels. For each combination of factor levels, e.g. (substrate material: steel X38CrMoV5-1 - soft annealed; coating type: NiCrBSi; spraying distance: small and substrate preparation: non-roughened) there were two repetitions, which is why the designations are e.g. 1_1-1 and 1_1-2 [14].

The overview of microstructures is given below.

Table 3 Microstructure of coating/substrate microstructure test plan, substrate material - steel X38CrMoV5-1

Substrate material	Coating type	Spraying distance	Substrate preparation	Sample	
Steel X38CrMoV5-1 - soft annealed	NiCrBSi	Small	Non-roughened	1_1-1	1_1-2
	NiCrBSi	Large	Non-roughened	1_2-1	1_2-2
	NiBSi	Small	Non-roughened	1_3-1	1_3-2
	NiBSi	Large	Non-roughened	1_4-1	1_4-2
	NiCrBSi	Small	Roughened	1_1_0-1	1_1_0-2
	NiCrBSi	Large	Roughened	1_2_0-1	1_2_0-2
	NiBSi	Small	Roughened	1_3_0-1	1_3_0-2
	NiBSi	Large	Roughened	1_4_0-1	1_4_0-2
Steel X38CrMoV5-1 - tempered	NiCrBSi	Small	Non-roughened	2_1-1	2_1-2
	NiCrBSi	Large	Non-roughened	2_2-1	2_2-2
	NiBSi	Small	Non-roughened	2_3-1	2_3-2
	NiBSi	Large	Non-roughened	2_4-1	2_4-2
	NiCrBSi	Small	Roughened	2_1_0-1	2_1_0-2
	NiCrBSi	Large	Roughened	2_2_0-1	2_2_0-2
	NiBSi	Small	Roughened	2_3_0-1	2_3_0-2
	NiBSi	Large	Roughened	2_4_0-1	2_4_0-2

3 MICROSTRUCTURE ANALYSIS FOR SUBSTRATE MATERIAL - X38CRMOV5-1 STEEL

In this chapter, microstructures for all combinations of substrate materials - X38CrMoV5-1 steel and coatings are presented and analysed, as follows [14]:

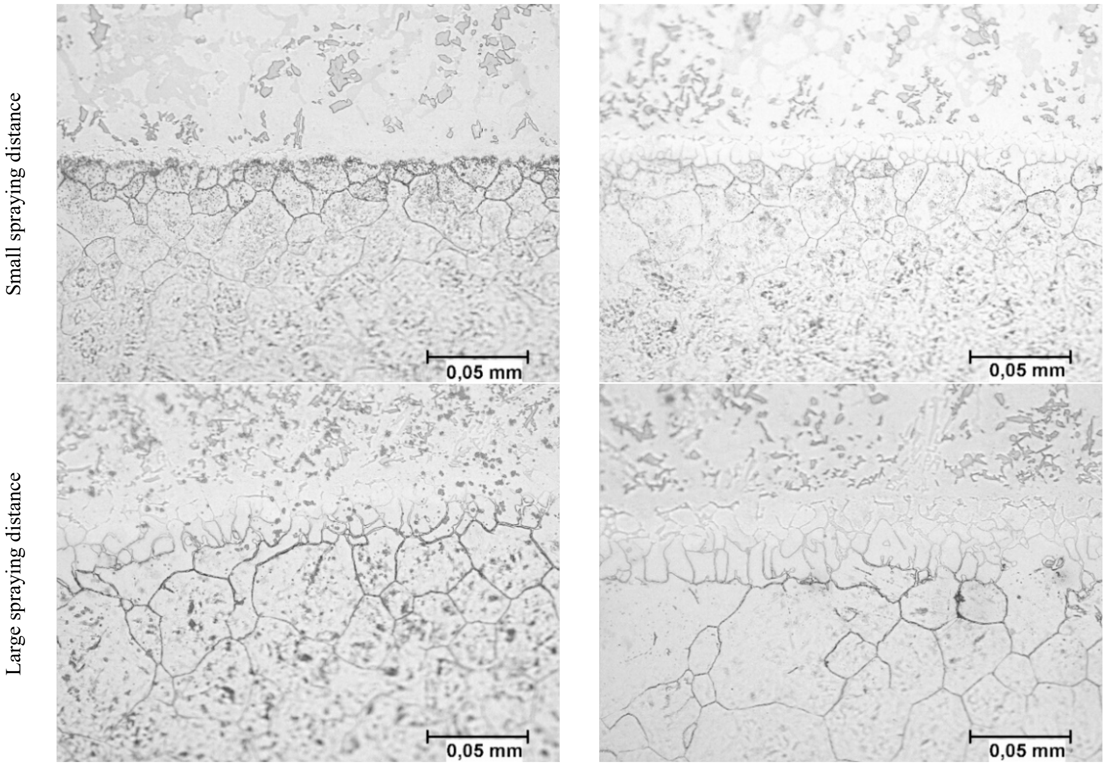
- X38CrMoV5-1 steel, soft annealed - NiCrBSi coating
- X38CrMoV5-1 steel, tempered - NiCrBSi coating
- X38CrMoV5-1 steel, soft annealed - NiBSi coating
- X38CrMoV5-1 steel, tempered - NiBSi coating.

It should be noted that, for the images of the microstructures that follow, the upper part of the image represents the coating, and the lower part of the image represents the substrate. The exceptions are images with cracks (Figs. 5 and 7), where the upper part of the picture shows the substrate and the lower part the coating.

a) X38CrMoV5-1 Steel, Soft Annealed - NiCrBSi Coating

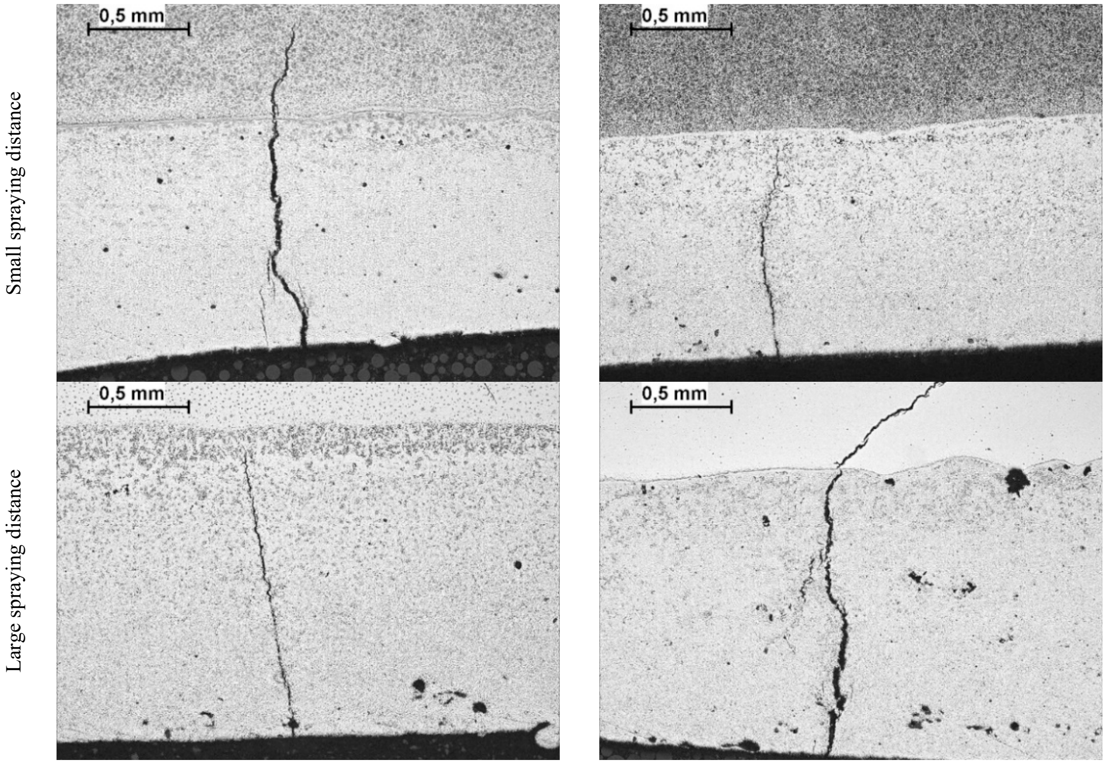
From Fig. 4, it is evident that when spraying on the non-roughened substrate surface, there was no separation of the coating from the substrate and that, as for the roughened substrate, spraying with both smaller and greater spraying resulted in the creation of a metallurgical connection between the coating and the substrate. The white zone is clearly pronounced between the coating and the substrate - the so-called diffusion zone. However, two zones are clearly visible during spraying on the non-roughened substrate surface with a long spraying distance as well as on the roughened surface (with a small and large spraying distance). Below the white zone of the so-called diffusion zone is a zone with austenitic grains, which is wider when spraying with a long spraying distance. In the paper [33], which describes the research results on spraying nickel alloys using flame spraying with subsequent fusing procedure and vibration, the authors also mentioned two zones. They state that the solidification of the coating starts from the coating/substrate boundary, where the substrate has the role of heat dissipation, and that at this boundary, an almost single-phase coating is created - Ni matrix without or with some eutectics. The second zone in the coating is a boron-free zone that has a greater thickness for vibration-subjected samples. The authors [34] also conclude that closer to the substrate, there are fewer precipitates in the coating itself (they cite the reason is the poor fusion that would encourage the growth of precipitates); A similar conclusion is made in the paper [35]. However, in the investigation of the corrosion resistance of NiCrBSi coatings [16], it was concluded that close to the coating/substrate boundary, there is a greater amount of a certain type of precipitates, which, due to the higher mass, are located at the bottom of the coating. In the research of the authors [36, 37], who studied plasma spraying of NiCrBSi coatings with subsequent flame fusing, the coating/substrate boundary was considered, and it was concluded that the coating/substrate boundary is constituted of two zones.

When spraying with a small spraying distance, for both types of preparation of the substrate, the grain boundaries are clearly expressed in the area under the coating and the substrate under the coating is fine grained with distributed carbides along the grain boundaries and inside the grains. Further away from the coating/substrate boundary towards the core the roughening of the austenitic grains is evident. Due to the longer heating time when spraying with larger spraying distance, for both types of the substrate preparation, in the substrate under the coating, the grain coarsening is greater.



Non-roughened substrate preparation Roughened substrate preparation

Figure 4 Microstructure system: steel substrate X38CrMoV5-1 (soft annealed) - NiCrBSi coating



Non-roughened substrate preparation Roughened substrate preparation

Figure 5 Cracks appearance in a system: steel substrate X38CrMoV5-1 (soft annealed) - NiCrBSi coating

The initial structure of the substrate before the spraying and the fusing process was ferrite with homogeneously distributed spheroidized chromium carbides, with a hardness of 192 HV10. After the process of spraying and fusing at a temperature of about 1040 °C and slow cooling in the air, there was a change of the structure throughout the whole substrate. The austenitizing temperature of X38CrMoV5-1 steel is 1030 °C, which is close to the fusing temperature. At a distance of 2 mm from the edge of the coating, where the cooling is faster, there was a partial transformation, and a bainite-martensitic structure was achieved. By moving away from the edge of the coating to the core due to slower cooling, a bainite structure was achieved. It has been proven that the simultaneous fusing process was actually the process of austenitizing of soft annealed steel X38CrMoV5-1, and cooling in the air still allowed the structure change.

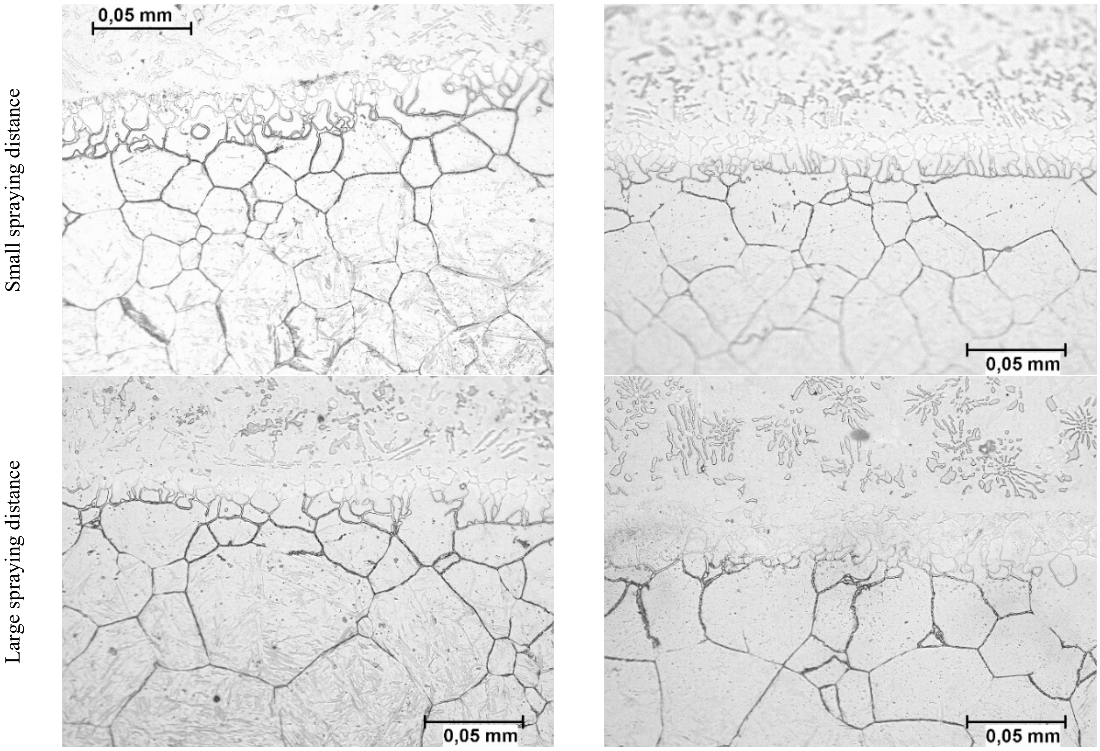
There was an appearance of cracks on all samples, and it is assumed that the cracks were due to residual stresses in the substrate material because the fusing temperature of the NiCrBSi coating (1040 °C) is close to the austenitizing temperature of X38CrMoV5-1 steel (1030 °C). Due to heating the substrate material to this temperature and cooling in the air, a partial transformation into martensite occurred in the area under the coating. After the spraying process, x38CrMoV5-1 steel remained in a quenched state with no tempering following, and due to residual stresses and relaxation of these tensions, cold cracks appeared.

In Fig. 5, cracks are visible in both the substrate and the coating material on the samples that are not ground.

b) X38CrMoV5-1 Steel, Tempered - NiCrBSi Coating

Fig. 6 shows that, as with the combination of soft annealed steel X38CrMoV5-1 - NiCrBSi coating (Fig. 4), there was no separation of the coating from the substrate when spraying it onto an untreated surface. At the coating/substrate border, now, the two zones are also visible for all combinations of spraying distance and surface preparation.

When spraying with a small spraying distance, the diffusion zone (0.0005 mm of thickness) is less pronounced, which could now be measured, while when spraying with a large spraying distance, it is more pronounced (0.001-0.002 mm of thickness). For this spraying distance, there was a coarser grain in the substrate due to the longer heating time for both types of substrate preparation.

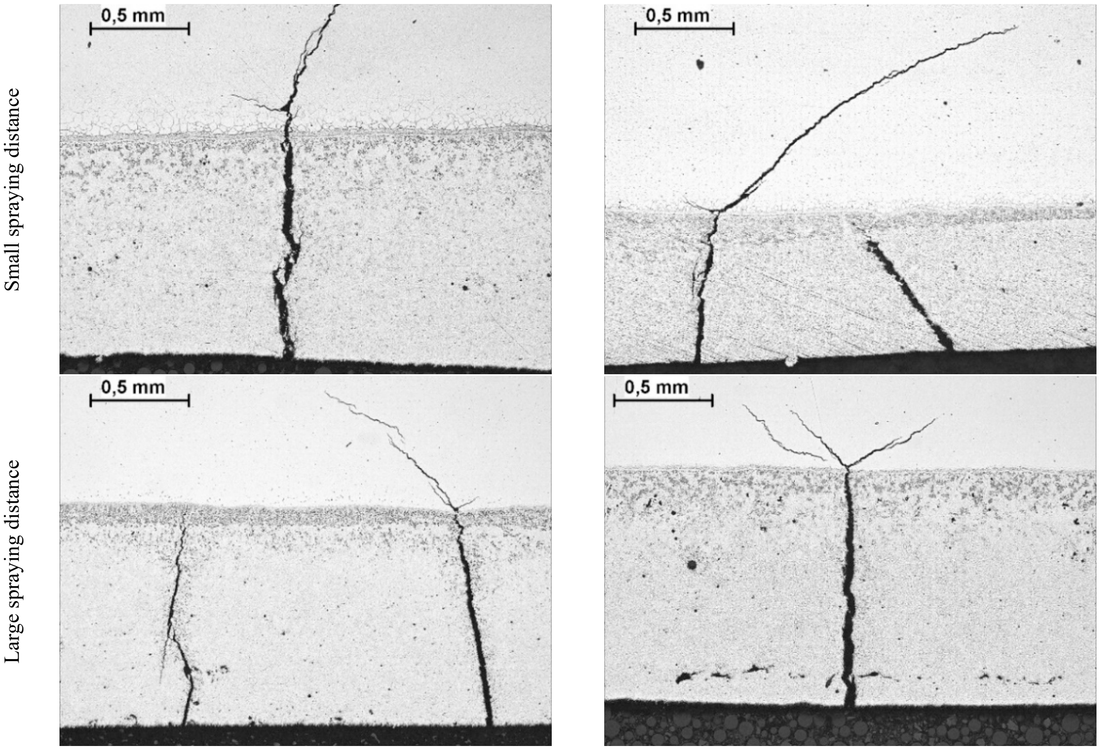


Non-roughened substrate preparation

Roughened substrate preparation

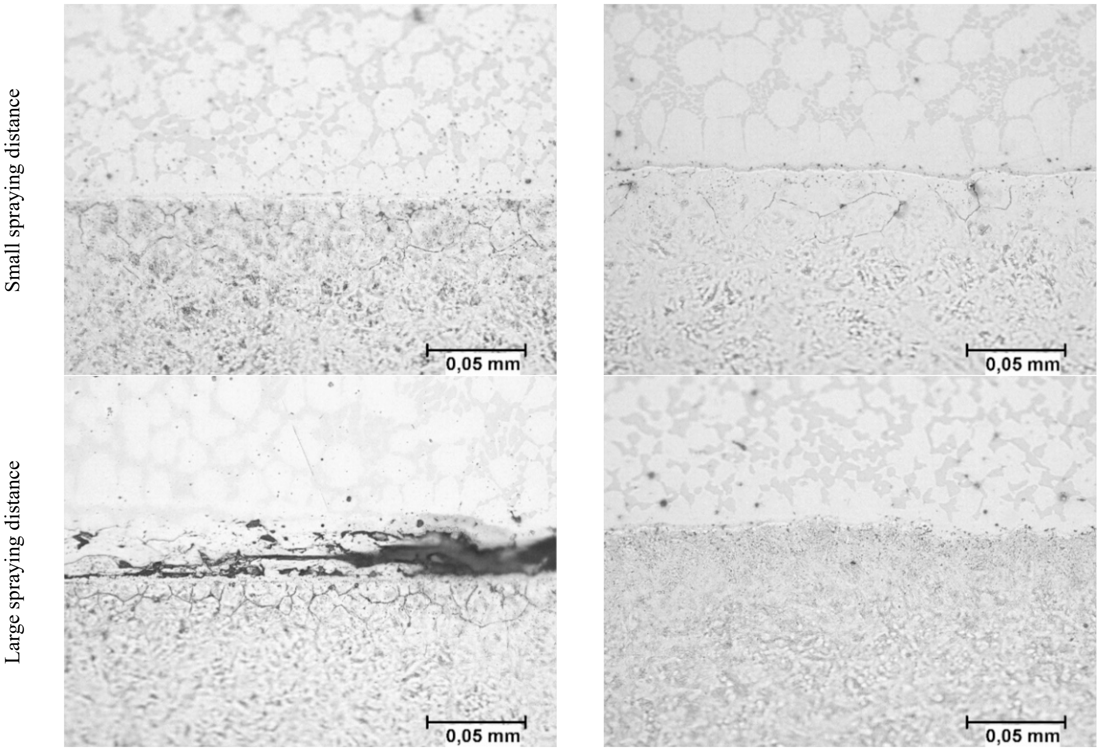
Figure 6 Microstructure system: steel substrate X38CrMoV5-1 (tempered) - NiCrBSi coating

The initial structure of the substrate before the spraying and fusing process was tempered martensite, with a hardness of 52-54 HRC. After the process of spraying and fusing at a temperature of about 1040 °C and cooling in the air, there was a change of structure throughout the substrate sample. During the spraying and fusing process, there was a re-quenching of the substrate material since the austenitizing temperature of X38CrMoV5-1 steel, 1030 °C, is close to the fusing temperature. At 2 mm from the edge of the coating, a martensitic structure with pronounced boundaries of the primary austenitic grain remained [38]. Moving away from the edge of the coating towards the core reduces the size of the primary grain and makes the structure more homogeneous. Due to the cooling in the air up to a distance of 3 mm from the edge of the coating, a complete transformation into martensite was achieved. After this distance, the structure of the substrate is, as before the spraying procedure, tempered martensite. In the paper [39], in which the martensitic stainless steel substrate was also quenched and tempered (the austenitizing temperature of 1020 °C is also similar to the fusing temperature), the authors do not refer to the structure of the substrate after the spraying process.



Non-roughened substrate preparation Roughened substrate preparation

Figure 7 Cracks appearance in a system: steel substrate X38CrMoV5-1 (tempered) - NiCrBSi coating



Non-roughened substrate preparation Roughened substrate preparation

Figure 8 Microstructure system: steel substrate X38CrMoV5-1 (soft annealed) - NiBSi coating

As for the samples with a substrate in a soft annealed state, cracks appeared here in the coating and substrate (Fig. 7), which are now more pronounced and were formed due to residual stresses in the substrate material. As x38CrMoV5-1 steel had already been heat treated before the spraying process, during the spraying and fusing process at a temperature of about 1040 °C, the substrate material was re-quenched since the fusing temperature is close to the austenitizing temperature of this steel.

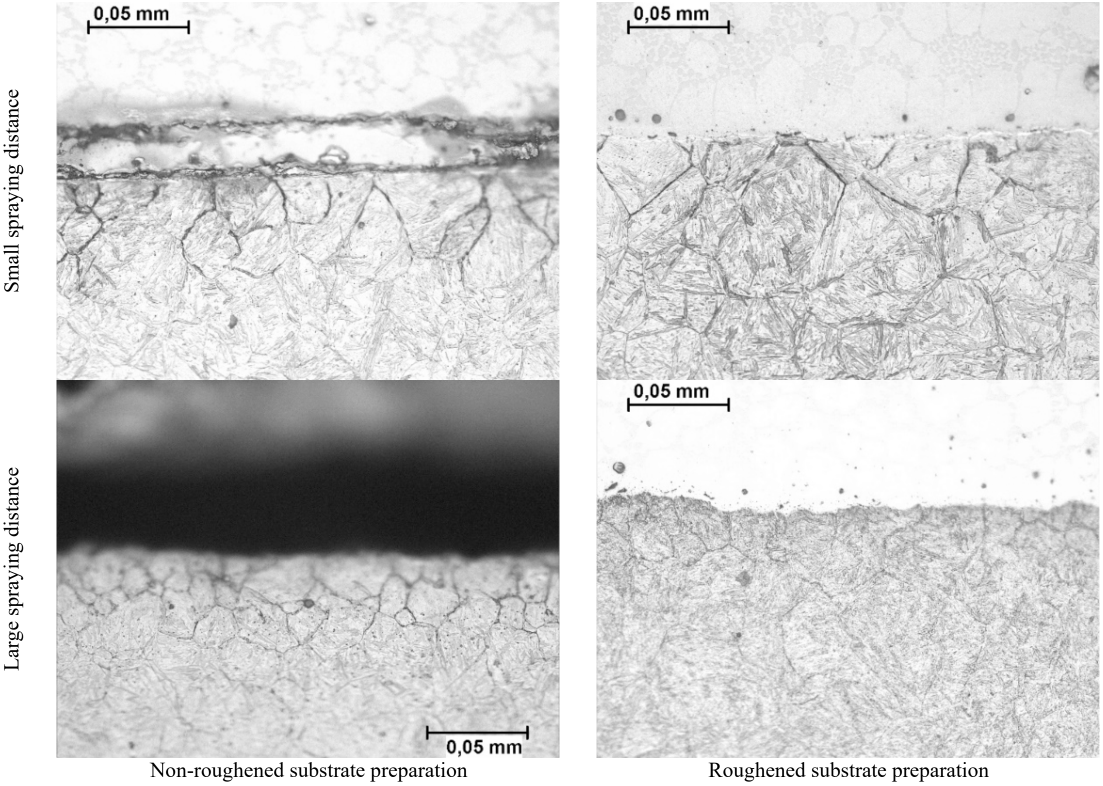
When heating the substrate material to this temperature and cooling the substrate faster in the area under the coating, there was a complete transformation into martensite up to 3 mm from the edge of the coating. After the spraying process, x38CrMoV5-1 steel remained in a quenched state and no tempering followed. Due to the residual stresses cold cracks appeared.

c) X38CrMoV5-1 Steel, Soft Annealed - NiBSi Coating

From Fig. 8 it is evident that when spraying the NiBSi coating, with a small spraying distance for both types of substrate preparation, as well as for spraying with a large spraying distance on the roughened surface, there is a narrow white zone so-called diffusion zone in which a metallurgical connection between the coating and the substrate is achieved during the fusing at a temperature of about 1070 °C. When spraying with a large spraying distance, and thus with less heat input on non-roughened substrate, there was no complete diffusion between the coating and the substrate material, or there was a subsequent separation of the coating from the substrate, when cooling to the ambient temperature, due to a greater difference in coefficients of thermal elongation between the coating material and the substrate. The coefficient of thermal elongation for the NiBSi alloy is 15.55×10^{-6} K^{-1}. The coefficient of thermal elongation for the substrate material, for the same temperature range, is 14×10^{-6} K^{-1} [40]. For a NiCrBSi alloy, this coefficient is lower than for the NiBSi coating and is 13.62×10^{-6} K^{-1}. A possible cause of partial separation of the coating may also be the local overheating, which can cause damage in the coating or at the coating/substrate boundary [41].

During the metallographic analysis of all the above-mentioned samples, not a single crack through the substrate or coating was observed, which was the case for the NiCrBSi coating on that same substrate.

Based on the structures shown in Fig. 8, it can be concluded that the substrate under the coating has a bainite-martensitic structure, and by moving away from the coating/ substrate boundary towards the core, the hardness of the substrate is lower, and thus the microstructure changes to bainite.



Non-roughened substrate preparation Roughened substrate preparation

Figure 9 Microstructure system: steel substrate X38CrMoV5-1 (tempered) - NiBSi coating

d) X38CrMoV5-1 Steel, Tempered - NiBSi Coating

The combination of tempered steel substrate and NiBSi coating in Fig. 9 shows that when spraying on a non-roughened substrate surface with both larger and smaller spraying distances, there was a partial or complete separation of the coating from the substrate, while for the roughened substrate surface, a metallurgical connection was achieved between the coating and the substrate material. When spraying with a small spraying distance on the roughened substrate surface, the substrate under the coating does not have a coarse grain structure and a weak distribution of carbides across the grain boundaries is visible. Under the coating, due to faster cooling, a martensite structure with a relatively large primary grain was achieved. In the case of a roughened substrate, with spraying with a large spraying distance, the substrate under the coating has less pronounced grain boundaries with partially distributed carbides. The substrate material retained the loosened martensite structure it had before the spraying and fusing process.

On all samples for this type of coating (NiBSi), according to metallographic analysis, not a single crack was observed through the substrate and coating, for the substrate in tempered or in a soft annealed state. On the more fragile NiCrBSi coating, the relaxation of the tension of the substrate (untempered martensite) led to the formation of cracks (Figs. 5 and 7), while, for this tougher NiBSi coating, energy was absorbed, and no cracks were formed.

4 CONCLUSION

When investigating the microstructure of the coating/substrate system using the factorial design of the experiment, along with the investigated coatings, the spraying distance from the workpiece, the preparation of the substrate-non-roughened and roughened and heat treatment of the substrate-soft annealed and tempered condition were systematically combined. It can be concluded that the application of NiCrBSi coating by spraying on a tool steel substrate would not make sense due to the appearance of cracks caused by the formation of martensitic structure of the substrate after spraying and relaxation of residual stresses because the temperature of the fusing was close to the austenitizing temperature of the specified steel, for which conversion into a martensitic structure was enabled even during slow cooling in the air.

For the NiBSi coating, no cracks were observed, as energy was absorbed for this tougher coating. However, due to the greater difference in coefficients of thermal elongation between X38CrMoV5-1 steel and NiBSi coating and the increased volume of martensitic structure, there was a separation of the mentioned coating on a non-roughened surface.

Acknowledgements

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