Effect of frictional treatment with a dense cubic boron nitride indenter on the micromechanical properties of the NiCrBSi–Cr₃C₂ coating

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Abstract. Coatings based on NiCrBSi alloys with Cr₃C₂ addition can effectively resist wear, corrosion, and oxidation at high temperatures. The use of frictional treatment by sliding indenters as finishing is a modern way to create high surface quality and to enhance the strength and wear resistance of the surface of parts. The article studies the characteristics determined by instrumented microindentation of the surface of a NiCrBSi–Cr₃C₂ laser clad coating subjected to frictional treatments with a sliding indenter made of dense cubic boron nitride DBN in the air at loads on the indenter of 350, 500 and 700 N, and after grinding. Frictional treatment in the entire considered range of loads contributes to an increase in strength performance, as well as parameters indicating an increased ability of the coating surface to resist elastoplastic deformation. The highest growth of the parameters is observed after frictional treatment at a load of 700 N.

1. Introduction

The formation of protective coatings on the surface of parts is an effective method to increase their durability and reliability. Coatings based on NiCrBSi alloys have good performance under conditions of wear, corrosion, elevated temperatures and are widely used worldwide at present [1, 2].

Gas powder laser cladding is a perspective method for obtaining coatings. The laser beam melts the filler material (powder) and simultaneously the thin surface layer of the base (substrate) during gas powder laser cladding [3]. Compared with other methods of applying surface layers, laser cladding forms coatings with increased hardness and uniformity, as well as with excellent metallurgical adhesion to the substrate [4, 5].

The creation of metal matrix composite coatings is a modern way to improve the performance of coatings. They consist of a rather soft matrix as well as hard reinforcing particles. NiCrBSi powders can be used as a metal matrix material for wear-resistant composite coatings owing to their excellent wear and corrosion resistance. In these alloys, an extremely thermal-resistant (up to 1000 °C) ‘frame-like’ structure of large chromium borides and carbides can be formed by high-temperature annealing [6–8]. This significantly expands the possibilities of using nickel-chromium laser clad coatings for...
high-temperature applications, in particular, in metallurgy [9]. In addition, the considered alloys have a rather low melting point and can serve as a plastic bond for reinforcing phases. Additives of carbides, borides, oxides, and other compounds are used as strengthening phases in NiCrBSi coatings. Coatings based on NiCrBSi with Cr3C2 addition can effectively resist wear, corrosion, and oxidation at high temperatures [10].

Laser clad coatings have significant roughness and undulation [11]. In most cases, these disadvantages are eliminated by grinding with abrasive wheels. However, grinding can provide ‘burns’ and microcracks on the surface, as well as critical tensile stresses, which can lead to a decrease in the service life of parts. The use of surface deformation by sliding indenters as finishing is a modern way to create high surface quality, favorable compressive stresses in the surface layer, and to enhance the strength and wear resistance of the surface of parts [12]. An effective method for obtaining a hardened NiCrBSi coating on metal parts has been proposed. It includes gas powder laser cladding and frictional treatment with a hemispherical indenter made of dense cubic boron nitride (DBN) [13]. The authors also showed that frictional treatments with a DBN indenter at loads of 350–700 N increase the microhardness of the NiCrBSi–Cr3C2 laser clad coating [14].

Instrumented microindentation is one of the methods for assessing the mechanical properties of nickel-based coatings [15–17]. It makes it possible to register diagrams during loading and unloading of the indenter. The method allows evaluating the mechanical properties of materials for which there is no opportunity to conduct standard tests for tensile, compression, and bending [18].

However, the instrumented microindentation method has not been previously used to evaluate the micromechanical characteristics of the NiCrBSi–Cr3C2 coating formed by laser cladding and subjected to frictional treatment. Therefore, the purpose of this work was to research the influence of frictional treatments with a DBN indenter at loads of 350–700 N on the micromechanical properties of the NiCrBSi–Cr3C2 laser clad coating.

2. Material and Experimental Procedure

The cladding material was two powders: 85 wt. % of the NiCrBSi with a dispersion of 40–160 μm and 15 wt. % of Cr3C2 with a dispersion of 50–150 μm. Chemical composition of NiCrBSi powder, wt. %: 14.8 Cr; 2.1 B; 2.9 Si; 0.48 C; 2.6 Fe; the rest is Ni. Two-layer cladding was carried out on plates made of low-carbon steel (0.2 % C) using a CO2 laser in the continuous mode. Cladding was implemented in the following modes: radiation power 1.4–1.6 kW; the size of the laser spot on the surface 2–6 mm; powder consumption 2.9–3.8 g/min; speed 160–200 mm/min; shift 4.0–4.5 mm. An inert gas (argon) was used as a transport gas for the powder mixture. To exclude the undulation, grinding of the surface with a deposited coating was executed on a grinding machine where intensive cooling was provided. After grinding, the thickness of the NiCrBSi–Cr3C2 coatings was 0.7–1.1 mm.

Frictional treatment was performed in a laboratory setup on flat samples with the coatings after grinding their surface. An indenter made of dense cubic boron nitride DBN with hemispherical shape was reciprocating along the sample surface. The load P on the indenter was 350, 500, and 700 N. Frictional treatment was performed with a five-time scanning of the surface with the indenter in air at an speed of 0.013 m/s, a length of stroke of 17 mm, and displacement of an indenter was 0.1 mm per double stroke.

Instrumented microindentation with recording the loading and unloading diagrams was conducted using a measuring system Fischerscope HM2000 XYm. The indenter was a Vickers indenter, a maximum load on the indenter was 0.245 N. Micromechanical parameters were calculated according to ISO 14577 [19]. Loading/unloading time – 5 s. Holding time at maximum load – 20 s. The measurement error of microindentation characteristics from 10 measurements was determined by the standard deviation with a confidence probability of 0.95.
3. Experimental Results and Discussion

Figure 1 presents the diagrams of loading and unloading of the indenter during microindentation of the surfaces of samples with NiCrBSi–Cr3C2 coating after grinding, as well as after frictional treatment with the DBN indenter at loads of 350, 500, and 700 N.

![Figure 1](https://example.com/figure1.png)

**Figure 1.** Experimental curves ‘load $F$ – displacement of the indenter $h$’ during microindentation the surfaces of samples with NiCrBSi–Cr3C2 coating after frictional treatment with the DBN indenter in air at loads of 350, 500 N and 700 N (curves 1, 2 and 3, respectively) and after grinding (curve 4).

| Surface treatment          | $h_{\text{max}}$ (μm) | $h_p$ (μm) | $HM$ (GPa) | $H_{\text{IT}}$ (GPa) | $E^*$ (GPa) | $W_t$ (nJ) | $W_e$ (nJ) |
|---------------------------|------------------------|------------|------------|------------------------|-------------|------------|------------|
| 1 Frictional treatment, $P=350$ N | 1.17±0.03             | 0.83±0.03  | 7.1±0.3    | 10.2±0.6               | 228±6       | 94.0±2.7   | 31.8±0.5   |
| 2 Frictional treatment, $P=500$ N | 1.18±0.04             | 0.83±0.04  | 7.0±0.4    | 10.1±0.7               | 219±9       | 95.1±3.5   | 32.6±0.9   |
| 3 Frictional treatment, $P=700$ N | 1.14±0.03             | 0.78±0.04  | 7.6±0.4    | 11.1±0.7               | 224±4       | 91.2±1.6   | 33.6±0.8   |
| 4 Grinding               | 1.26±0.05             | 0.93±0.06  | 6.1±0.5    | 8.4±0.8                | 229±10      | 103.5±6.3  | 28.9±0.9   |

**Table 1.** The microindentation results of the samples with NiCrBSi–Cr3C2 laser clad coating after different treatments.

Figure 1 shows that the frictional treatment of the coating has a significant effect on the type and position of the lines, shifting them towards lower values of the indenter displacements $h$ during indentation in comparison with the measure of $h$ for the surface after grinding.

The characteristics displayed in table 1 were determined using the curves of loading ($a \rightarrow b$ in figure 1) and unloading ($b \rightarrow c$ in figure 1), according to the method of Oliver and Farr [20]. They reflect the features of the mechanical behaviour of the considered coatings under elastoplastic deformation. The values in table 1 show that the frictional treatment of the NiCrBSi–Cr3C2 coating surface reduces the maximum $h_{\text{max}}$ (to 1.14–1.18 μm) and the residual $h_p$ (to 0.78–0.83 μm) indenter indentation depths compared to the values of the polished surface ($h_{\text{max}} = 1.26$ and $h_p = 0.93$ μm). The lowest values of the maximum and residual indentation depths during indentation are characteristic of the surface of the coating subjected to frictional treatment at a load of 700 N.

Frictional treatment with a DBN indenter at all loads used also increases the values of the hardness on the Martens scale $HM$, which takes into account both plastic and elastic deformation, compared to the values after grinding. Furthermore, treatment at loads of 350–500 N increases the $HM$ values by 15 %, and at loads of 700 N – by 25 % (see table 1). After frictional treatment, the indentation hardness values at the maximum load $H_{\text{IT}}$ increase even more significantly – by 20–21 % after treatment at loads of 350–500 N and by 32 % after treatment at a load of 700 N (see table 1). An increase in $H_{\text{IT}}$ indicates an increase in the resistance to constant deformation [19].

The contact elastic modulus when indenting the surface of the coating $E^*$ after frictional treatment at various loads little (within the error) varies from the characteristics of the polished surface. It is
known that the elastic modulus of metal materials can change during deformation [21–23]. However, in this case, the surface after grinding can also be considered as deformed. It is also important to note that the composite NiCrBSi–CrC2 coating after frictional treatment in air with a DBN indenter at a load of 350 N has a 13 % higher level of the contact elastic modulus ($E^* = 228 \pm 6$ GPa) (see table 1) than the NiCrBSi coating of the same composition, but without CrC2 additives ($E^* = 202 \pm 5$ GPa) [24]. In addition, the contact elastic modulus of NiCrBSi coatings of the same composition not subjected to frictional treatment was 195 ± 5 GPa [25]. This can be explained by the presence in the composite coating of 15 % CrC2 carbides with a significantly increased elastic modulus ($E = 370$ GPa) [26]. Similarly, in [27], an increase in the elastic modulus of a composite material was observed with an increase in the amount of phase with a large elastic modulus.

Table 1 also shows that the values of the total mechanical work of indentation $W_t$, which is determined by the field under the load curve (triangle abd in figure 1) and consists of plastic deformation work and elastic reserve deformation work, inversely depend on the type of treatment compared to the values of hardness $H_M$ and $H_{IT}$, since the more hardened the material, the less it deforms under the indenter and, accordingly, the less work is spent on such deformation. The elastic reserve deformation work $W_e$, which is determined by the field under the unloading curve (triangle cbd in figure 1) and released when the applied load is removed, grows with increasing load on the indenter during frictional treatment.

The determinate values of the work (the total mechanical work of indentation $W_t$ and the elastic reserve deformation work $W_e$) were used in the calculation according to the formula $(1-(W_e/W_t))100$ % of the plastic component of the work when indenting the surface of the coating (table 2). The largest (among the four considered coating states) plastic component of the indentation work (72 %) has a coating after grinding. Growth in the load on the indenter during frictional treatment successively reduces the plastic component from 66 % at a load of 350 N to 63 % at a load of 700 N.

Table 2 presents several calculated parameters determined by microindentation data depending on the type of surface treatment of the coating. The parameters are used to evaluate the resistance of material's surface to elastoplastic deformation under mechanical contact loading. The value of elastic recovery $R = (h_{max} - h_0)/h_{max} \cdot 100$ % [28–30] and the ratio $H_{IT}/E^*$ (specific contact hardness) [31] change in the same way during frictional treatment compared to the state after grinding. The values increase during frictional treatment at loads of 350–500 N and even more significantly at a load of 700 N. It is generally accepted [30, 32] that the elastic recovery of $R$ and the ratio $H_{IT}/E^*$ characterize the proportion of elastic deformation in the total deformation during indentation.

**Table 2.** The plastic component of the indentation work $(1-(W_e/W_t))\cdot100$, elastic recovery $R$, the ratios $H_{IT}/E^*$ and $H_{IT}/E^*/E^{*2}$, calculated from the microindentation results of the samples with NiCrBSi–CrC2 laser clad coating after different treatments.

| Surface treatment | $(1-(W_e/W_t))\cdot100$ (%) | $R$ (%) | $H_{IT}/E^*$ | $H_{IT}/E^{*2}$ (GPa) |
|-------------------|-----------------------------|---------|---------------|------------------------|
| 1 Frictional, $P = 350$ N | 66 | 29.1 | 0.045 | 0.020 |
| 2 Frictional, $P = 500$ N | 65 | 29.3 | 0.046 | 0.021 |
| 3 Frictional, $P = 700$ N | 63 | 31.6 | 0.050 | 0.027 |
| 4 Grinding | 72 | 25.9 | 0.036 | 0.011 |

The values of the $H_{IT}/E^{*2}$ ratio presented in table 2 are regarded to be a parameter of the material’s resistance to plastic deformation after the beginning of the flow since the ratio is proportional to the flow stress $P_0$ of the material [33]. The ratio $H_{IT}/E^{*2}$ has a minimum value for the coating after grinding. Frictional treatment even at a minimum load of 350 N contributes to the growth of this indicator by 82 %. Treatment at a load of 700 N increases this indicator by 145 %.

It was previously shown [34] that there are coarse (50–100 μm) original chromium carbides CrC2 in the structure of the NiCrBSi–CrC2 laser clad coating. Frictional treatment with a DBN indenter at a load of 350 N smooths the NiCrBSi matrix, and protruding initial chromium carbides CrC2 are remarked on the treated surface. The protruding wear-resistant framework on the wear surface made of
the reinforcing phases plays a predominant role (compared with the metal matrix role) in the abrasion resistance of NiCrBSi coatings formed by laser cladding [35]. Therefore, it can be expected that the coating after frictional treatment at a load of 350 N will be characterized by high resistance to abrasive wear [14].

The large initial chromium carbides are partially removed from the surface of the NiCrBSi–Cr₃C₂ coating during frictional treatment at a load of 700 N [14]. The micromechanical properties determined by instrumented microindentation show that increasing the load during frictional treatment to 700 N (compared to treatment at 350–500 N) increases the hardness of the coating and its ability to resist plastic deformation. This indicates that the level of hardening of the metal matrix, in this case, makes a greater contribution to the overall strength properties of the coating measured during microindentation than their possible reduction due to some absence of coarse chromium carbides after frictional treatment at a load of 700 N.

Thus, in comparison with the coating after grinding, the coating after frictional treatments is characterized by both maximum deformation in the elastic region (i.e., by a delayed transition to the plastic stage of deformation) and an increased ability to resist to contact loads after the beginning of the plastic flow. To a greater extent, these features are manifested for the coating after frictional treatment at the highest load used. In [36], the results of microindentation for steel surfaces after frictional treatment correlate with wear mechanisms during sliding friction. The increased ability to resist elastoplastic deformation may indicate better wear resistance of the coating after frictional treatment at a load of 700 N under sliding friction conditions.

4. Conclusion
According to the data obtained with instrumented microindentation, frictional treatment of the surface of the composite NiCrBSi–Cr₃C₂ coating with an indenter made of dense cubic boron nitride in air at loads of 350–700 N causes a significant increase in strength characteristics (Martens hardness HM and indentation hardness at maximum load Hₘ) and does not affect the level of the contact elastic modulus E* compared to the parameters after grinding. Additive 15 wt. % of Cr₃C₂ carbide with a high modulus of elasticity leads to a 13 % increase in the value of the contact modulus of elasticity of the coating subjected to frictional treatment.

In consequence of frictional treatment, the plastic component of the indentation work decreases in all the studied modes, and the values of elastic recovery R as well as the Hₘ/E* and Hₘ/E*² ratios (compared to the corresponding parameters after grinding) increase. This indicates a slow transition to plastic deformation during contact loading of the coating, followed by a more significant resistance to the development of plastic flow.

Frictional treatment at the maximum load on the indenter (700 N), which results in some removal of large initial chromium carbides from the surface, but increases the hardness of the metal matrix, provides a maximum increase in the average values of strength characteristics measured during microindentation, as well as parameters indicating an increased ability of the coating surface after the considered treatment to resist to elastic and plastic deformation.

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