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Examination of Wear and Rockwell-C adhesion Properties of Nitronic 50 Steel Coated with Pack Boriding Method

Ersan MERTGENÇ*¹

Abstract

The Nitronic 50 steel is a nitrogen containing stainless steel, which has a high corrosion resistance, and high strength but its surface resistance against wear is low, making it extremely limited to use in areas subject to wear. In this study, in order to improve the material surface and to investigate its effect on tribological properties, boronizing process was carried out by used pack boriding method at 850 °C, 900 °C and 950 °C for 4 hours. As a result of coating process, the boride layer has a smooth and flat structure in SEM investigations, the coating thickness varies between 9 µm and 36 µm and the boride layer thickness increases with increasing temperature. While the hardness of the uncoated material was around 250 HV_{0.05}, the surface hardness of the material reached up to 1.712 HV_{0.05} with the coating process and increased about 7 times. According to XRD analysis, the surface of the coating layer consisted of phases FeB, CrB, Ni₃B, Fe₂B, Cr₂B and MnB. Wear behavior was performed by ball-on-disk wear test in dry environment. The friction coefficient and wear rate decreases with increasing temperature, while the wear resistance is increased by 20 times compared to unboronized sample. When the wear tracks were examined, the uncoated Nitronic 50 had an adhesive wear mechanism, on the other hand the boronized samples had an adhesive and abrasive wear mechanism together. The Rockwell-C adhesion test was carried out under a load of 1.471 N and the resulting surface damages were evaluated according to the quality map. Boronized steel at 850 °C is defined as HF3 type, at 900 °C and 950 °C at boronized steel it is defined as HF4 type and adhesion is acceptable.

Keywords: Nitronic 50, pack boriding, Rocwell-C adhesion, wear

1. INTRODUCTION

Nitronic steels are seen as an erosion resistant alternative to 13/4 martensitic stainless steel currently used [1]. Nitronic steels are mainly austenitic stainless steels containing nitrogen as an alloying element in the matrix [2]. By adding nitrogen to the content of stainless steels; strength,

ductility, toughness, work hardening capacity, erosion and corrosion resistance increases [3-5].

Nitronic 50 is a high nitrogen, weldable stainless steel and superior to SAE 304 and 316 and 316L in strength and corrosion resistance [6] and is also known as 22-13-5 stainless steel [7]. They have almost two times higher yield strength and good corrosion resistance than standard 316 L stainless steel [8], which is due to the high content of

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chromium and molybdenum in the content, thus rivaling nickel alloys in seawater. Unlike most austenitic stainless steels, they are not magnetic when working in cold or sub-zero temperatures [7]. Nitronic 50 exhibits good mechanical properties at low and high temperatures where corrosion resistance and strength are required to be combined and is used extensively in a variety of applications such as naval construction, pumps, fittings, cables and heat exchangers [9].

Although stainless steels exhibit high corrosion resistance, low surface hardness is a major disadvantage, especially when exposed to wear, and it is important to improve the tribological and mechanical properties of their surfaces. In recent years, researchers have been working extensively on various coating techniques such as Physical Vapor Deposition (PVD) [10-12], Diamond-like Carbon (DLC) [13-17], Plasma Nitriding [18-20], Boronizing [21-23] to increase surface hardness through surface modification.

Fundamentally boronizing is a thermo-chemical process [24] diffusing the boron atoms from the surface of materials by means of solid, liquid and gas [25]. Boronizing can be carried out in solid, liquid and gas environments. Boron method is widely used because of its low cost and simple. [26]. Pack boronizing is carried out in the temperature range of 700 °C - 1000 °C for 0,5 to 10 hours [27].

When the studies on Nitronic 50 stainless steel are examined; It has been observed that mainly the corrosion properties of the material have been studied. It is noteworthy that different methods have been used for coating, but the effects of pack boring on the material have not been investigated.

In this study, by using pack boriding method, Nitronic 50 steel was treated at 850 °C, 900 °C and 950 °C temperatures for 4 hours, the acceptability of the coating was evaluated by Rockwell-C adhesion test and the effect of coating on the wear behavior was investigated.

2. MATERIALS AND METHODS

Nitronic 50 stainless steel was used in this study. The chemical composition is given in Table 1.

Table 1. Chemical composition of Nitronic 50 steel (wt. %)

| C | Si | Mn | P | S | N | Cr | Mo | Ni | V |
|-------|-----|------|-------|--------|------|-------|------|-------|------|
| 0.032 | 0.6 | 5.04 | 0.027 | 0.0005 | 0.30 | 21.49 | 2.17 | 12.06 | 0.16 |

The test samples were cut into $\varnothing 16 \times 8$ mm dimensions and ground up to 1200 grid and polished using diamond solution. Samples were placed in a stainless-steel box with Ekabor-II commercial powder. Then, they were boronized in a temperature controlled chamber type furnace under atmospheric pressure for 4 hours 850 °C, 900 °C and 950 °C. After the completion of boriding process, test specimens removed from the sealed box were eventually allowed to cool down in still air.

For microstructure analysis, samples were cut along longitudinal section, sanded with SiC abrasive paper up to 120 - 1200 grit, polished with 1 μ m alumina suspension and etched with stainless steel etch (HCl: HNO₃: H₂O to 1: 1: 1). Coating layer, matrix microstructure, wear tracks and boride thickness measurements were examined by using a Leo 1430VP scanning electron microscope (SEM). The presence of boride in the coating layer was confirmed by x-ray diffraction (Shimadzu XRD-6000) using CuK α ($\lambda = 1.5406$ Å) radiation at 20° - 90°.

Shimadzu HMV-2 micro-hardness tester was used for hardness measurements. The measurements were made under conditions of 50 g load for 10 seconds from the cross-sectional surfaces of the coated samples to the matrix.

The wear tests of Nitronic 50 stainless steel coated by pack boring method were carried out in the ball disk system ball-on-disc system, dry conditions at room temperature, under 5 N load, 0.3 m / s wear rate and 500 m distance. 8 mm diameter WC-Co balls were used in the wear tests. The surface of

each sample was cleaned with alcohol before and after the abrasion wear tests. After the tests, the wear volumes of the samples were quantified by multiplying cross-sectional area by the width of wear track obtained from a Tribotechnic Rugosimeter. The wear rate was calculated with the following formula.

$$\text{Wear rate} = \text{Wear volume} / (\text{Applied load} \times \text{Sliding distance}), \text{ mm}^3 / \text{Nm}.$$

The Rockwell-C adhesion test was developed in Germany and standardized in VDI 3198. The Rockwell-C adhesion test, also known as the quality test for coated compounds, is applied at a load of up to 1471 N. It is mapped between HF1 and HF6 according to the condition of damage to the coating in figure 1 [28]. According to this test, which is easy to apply and evaluated especially during quality control processes during production; HF1-HF4 describes adequate adhesion, ie the acceptability of the coating, whereas HF5 and HF6 represent insufficient adhesion [29,30].

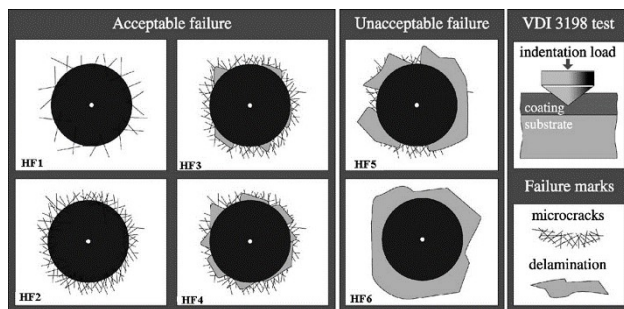


Figure 1. Principle of the VDI 3198 indentation test [28]

3. RESULTS AND DISCUSSIONS

3.1. Characterization of Coating Layer and Thickness

The microstructures of Nitronic 50 steel from the cross section of 850 °C, 900 °C and 950 °C for 4 hours are shown in figure 2.

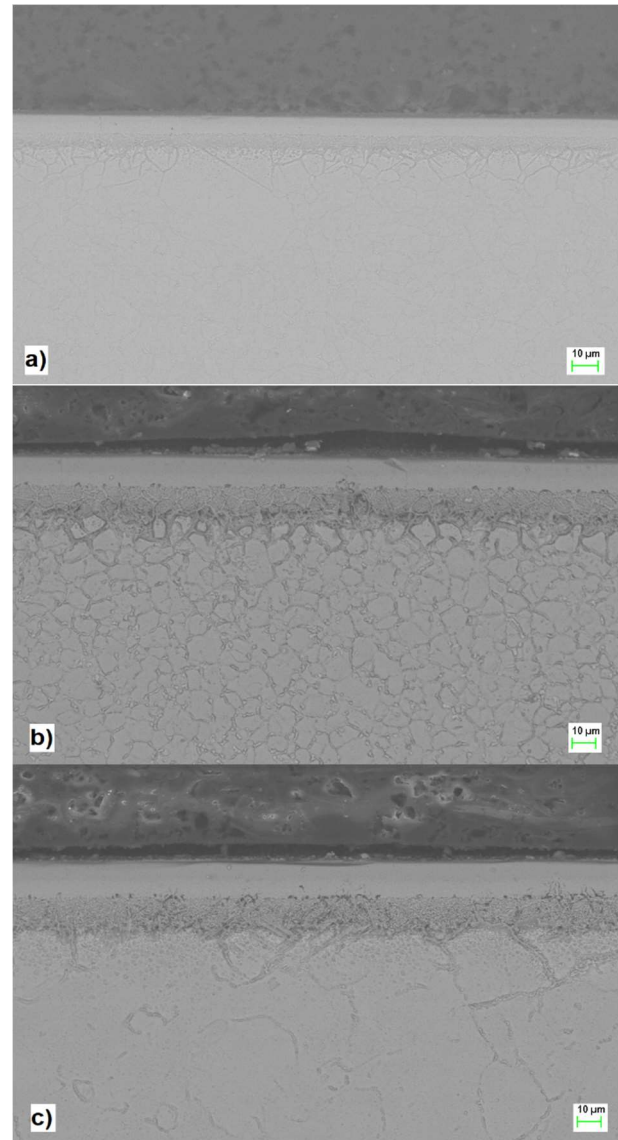


Figure 2. SEM cross-sectional views of boronized Nitronic 50 steel for 4 hours at a) 850 °C, b) 900 °C c) 950 °C

Boride layer has a flat and smooth structure and increase in boride layer thickness with increasing temperature [31, 32].

In steels, the thickness of the boride layer is strongly influenced by the alloying elements as a chromium, nickel and carbon in the substrate material [33]. Especially the presence of chromium makes it difficult to diffuse boron atoms due to its lower solubility compared to iron and prevents the growth of iron borides [34, 35]. The presence of as high as about 21 % chromium in Nitronic 50 steel results in a reduced boride layer thickness compared to steels. boride layer thickness was given table 2 and measured at

850 °C 13 μm , 900 °C 29 μm and 950 °C 36 μm . It is observed that an increase in the boride layer thickness is caused by the change in the process temperature for a treatment time of 4 h. This is due to the fact that the substrate material has high alloying elements, especially chromium and nickel.

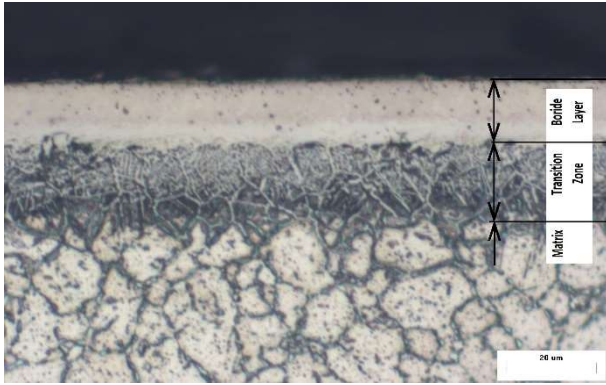


Figure 3. Zones of the coating layer of boronized Nitronic-50 steel for 950 °C and 4 hours

When the figure 3 is examined, it is seen that there are 3 zones in coated Nitronic 50 steel; zone 1: coating layer FeB/CrB and composed of rich Fe, B, Cr, Ni alloys, zone 2: diffusion zone with higher hardness than matrix but lower hardness than boride layer, zone 3: steel matrix free of boron [36, 37].

3.2. X-Ray Diffraction Analysis

Figure 4 shows the x-ray diffraction analysis of boronized Nitronic 50 steel at 850 °C, 900 °C and 950 °C for 4 hours.

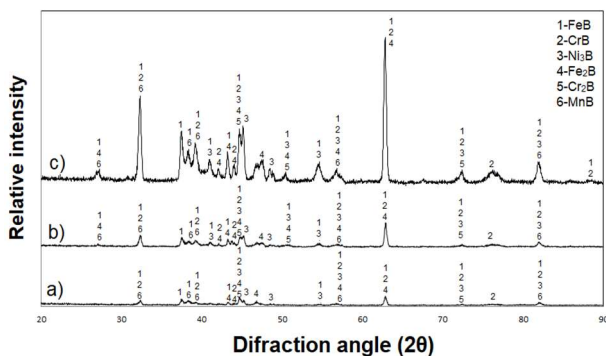


Figure 4. X-ray diffraction patterns of boronized Nitronic 50 steel for 4 hours at a) 850 °C, b) 900 °C c) 950 °C.

According to xrd analysis, FeB, CrB, Ni₃B, Fe₂B, Cr₂B and MnB phases were formed on the surface of the material depending on the process temperature. FeB and CrB phases increase with increasing temperature in boronizing treatment. The reason for this is that the diffusion process is accelerated with increasing temperature and the layer thickness increases. Due to the high amount of alloying elements such as Mn, Mo, Cr and Ni in Nitronic 50 steel, different phases were obtained due to these elements. The microstructure and the mechanical properties of the boronized material depend largely on the chemical composition of the material, the boriding temperature and the boriding time [38]. It is the reason why chromium based phases settle in dominant peaks due to the chemical composition of the material. On the other hand, the presence of FeB phase as the dominant peak in iron-based peaks is close to the surface, while Fe₂B phase settles between FeB phase and matrix [35, 39, 40]. The coexistence of the CrB and FeB peaks is due to the fact that these phases are very difficult to distinguish due to their similar structure [41].

3.3. Hardness of Boride Layer

Micro-hardness measurements were performed from cross-sectional area from surface to matrix (figure 5).

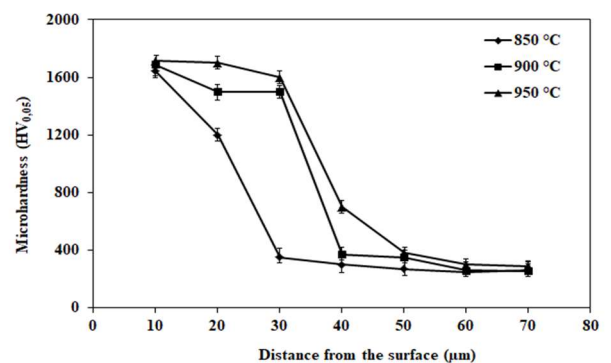


Figure 5. Micro-hardness profiles of boronized samples at 850 °C, 900 °C and 950 °C for 4 h.

The hardness of boronized Nitronic 50 steel was between 1080 HV_{0.05} and 1.712 HV_{0.05}, while the hardness of untreated steel was found to be (<) 247 HV_{0.05}. Hardness of boronized Nitronic 50

steel increased by seven times compared to the hardness of non-treated Nitronic 50 steel.

3.4. Wear Properties

In Table 2, coating thickness, friction coefficient and wear rate values of non-boronized and boronized samples are given.

Table 2. Friction coefficient and wear rates of boronized Nitronic 50 steel.

| | Conditions | | | |
|--|------------|--------------|--------------|--------------|
| | Uncoated | 850 °C – 4 h | 900 °C – 4 h | 950 °C – 4 h |
| Coating layer | - | 13 | 29 | 36 |
| Friction coefficient | 0.71 | 0.64 | 0.63 | 0.61 |
| Wear rate x 10 ³ (mm ³ / Nm) | 81.15 | 10,85 | 5.42 | 3,43 |

When the wear test results of Nitronic 50 steel were examined, it was found that the friction coefficient was 0.71 in the non-boronized sample, whereas the friction coefficient in the boronized samples at different temperatures for 4 hours ranged from 0.64 to 0.61. Wear rate was 81.15 mm³ / Nm in non-boronized sample and lower values were obtained in boronized samples. The lowest wear rate was measured as 3.43 mm³ / Nm in boron-treated sample at 950 °C for 4 hours.

When the wear tests of steels are examined in the literature, the coefficient of friction changes between 0.5 – 0.7 [42, 43, 44] and this shows that this study is consistent with the literature. On the other hand, surface investigators such as Kovaci et al. [16], Cuao-Moreu et al. [45], Teng et al. [46] and He et al. [47] have studied the tribological properties of metal specimens by coating different metal surfaces with different materials, temperature, time and method. The reason for the serious decrease in wear rate is the wear resistance of the hard coating layer on the surface of the material [48-50]. As seen from the XRD analysis, the amount of high hardness phases such as FeB and CrB increased.

The SEM images of the wear marks on the ball on disk system, against WC-Co balls, at 5 N load, 0.3m / s wear rate and 500 m distance are given in figure 6.

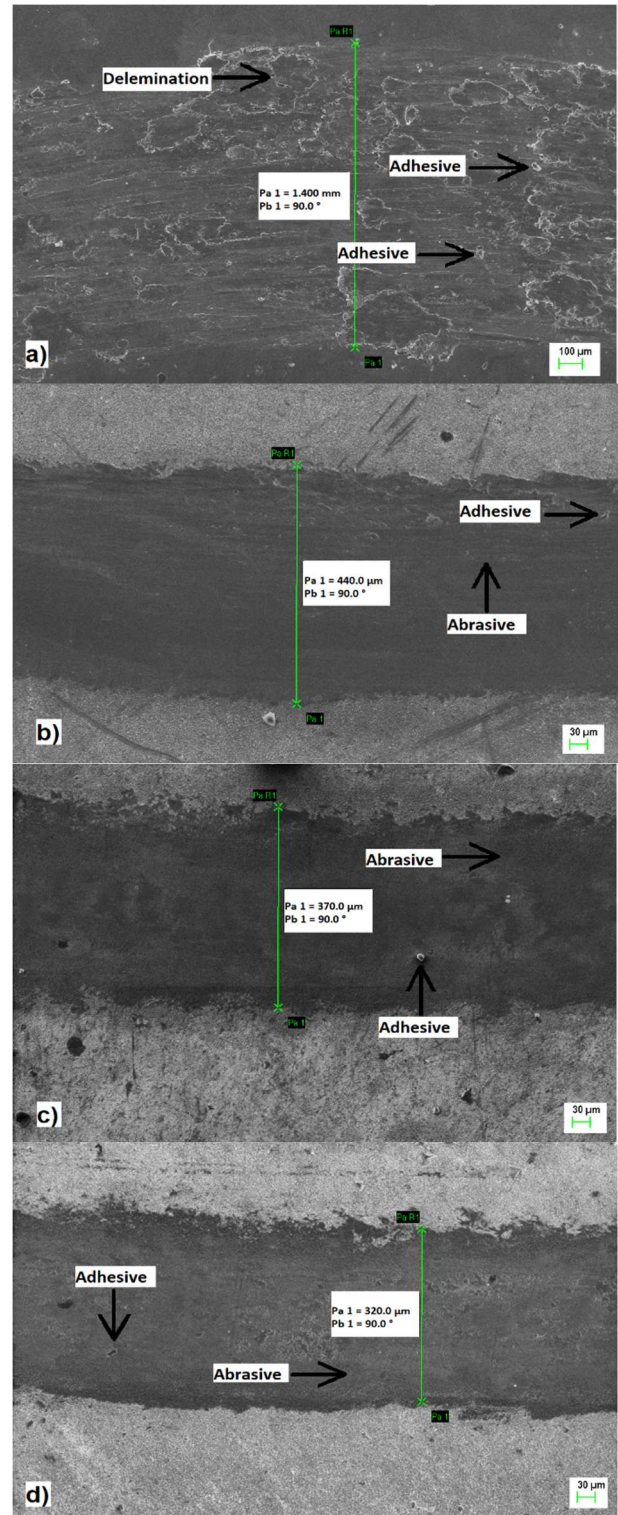


Figure 6. Wear traces of boronized Nitronic 50 steel a) non-boronized, b) 850 °C – 4 h, c) 900 °C – 4 h, d) 950 °C – 4 h

Wear traces vary according to boride layer thickness [33, 51, 52]. When the surface morphology of the wear marks is examined, adhesive wear is seen in the non-boronized

sample, while abrasive wear is seen in both the adhesive and abrasive wear by boring samples repeatedly cutting the coating surface by friction pairs. The width of the wear tracks ranges from 1.400 μm to 320 μm and when the tracks are examined, it is seen that the largest wear tracks and damages such as smearing (plastic deformation) in the wear direction are non-boronized specimen (figure 6 (a)) and the narrowest wear tracks are boronized at 950 °C (figure 6 (d)), the highest temperature used for 4 hours in the experiment. With the reduction of boride layer thickness, wear marks become more deeper [53].

3.5. Rockwell-C Adhesion

SEM images obtained from the adhesion test using a standard Rockwell-C hardness tester are shown in figure 7. Damage to the boride layer and adhesion strength are defined according to quality maps. According to the quality map, HF1-HF6 is grouped between HF1-HF4 and HF5 and HF6 are defined as inadequate adhesion [28].

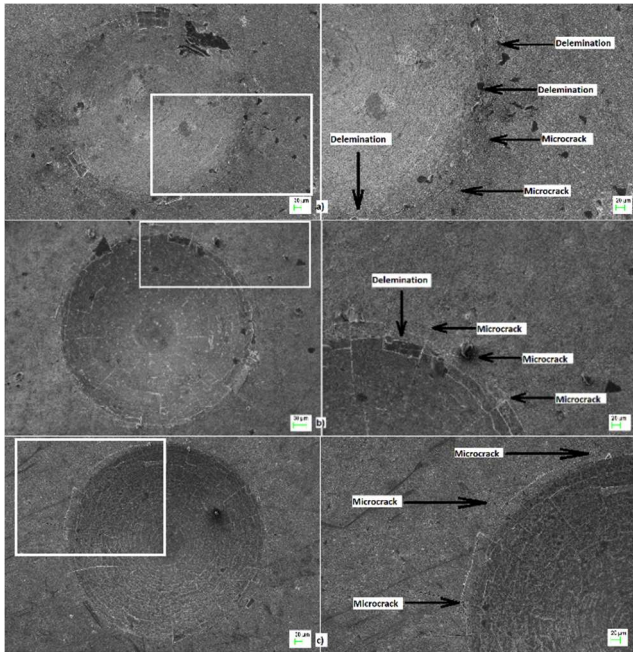


Figure 7. SEM micrographs and radial cracks of VDI adhesion test on boronized Nitronic 50 steel for 4 hours at a) 850 °C, b) 900 °C c) 950 °C.

When the damage images of boronized Nitronic 50 steel are examined for 4 hours, it is observed that radial cracks occur around the craters in the

boring at 850 °C (figure 7 (a)) and it is compatible with HF3 according to the quality map. In samples boronized at 900 °C (figure 7 (b)) and 950 °C (figure 7 (c)), delamination occurs along with radial cracks around the indentation craters and the amount of delamination increases with increasing boronizing temperature. This is due to the increase at the process temperature and the depth of the hard and brittle structure FeB phase. However, the images of samples according to the quality test were identified as HF4 and represent sufficient adhesion.

4. CONCLUSIONS

The results obtained from the investigation of tribological properties by coating the Nitronic 50 steel with pack boriding method at 850 °C, 900 °C and 950 °C for 4 hours are given below;

- The coating and the matrix interface exhibit a flat structure, which can be clearly seen the SEM observations. Boride layer thickness increased with temperature increase and reached 13 μm , 29 μm and 35 μm respectively.
- FeB, CrB, Ni₃B, Fe₂B, Cr₂B and MnB phases were formed in all boriding conditions and FeB, CrB phases increased with increasing temperature.
- The matrix hardness is around 250 HV_{0.05}, while the hardness of the boride layer ranges from 1080 HV_{0.05} to 1712 HV_{0.05}, and the surface hardness of the Nitronic 50 steel has increased about 7 times.
- The friction coefficient and wear rate of Nitronic 50 steel boronized by pack boring method decreased with increasing boriding temperature. While wear rate was 81.15 mm³ / Nm for unborided sample, with boriding treatment decreased to 3.43 mm³ / Nm and the wear resistance of the material surface increased about 20 times.
- As a result of the wear tests, non-boron steel has been exposed to intensive adhesive wear along with delaminations. In boronized samples,

adhesive and abrasive wear types are seen together.

- In the Rockwell-C adhesion test, the boronized sample at 850 °C represents HF3 according to the quality map, while the 900 °C and 950 °C samples were damaged in the HF4 type. Adhesion for coating is acceptable.

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