

Adhesive Behavior of the Pack-Borided AISI 304L Steel with Microwave Hybrid Heating

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Abstract

AISI 304L stainless steel material was pack-borided with microwave hybrid heating method at temperatures of 850, 900 and 950 °C for 2, 4 and 6 hours. The morphology of the boride layer formed on the surface of the samples was examined by an optical microscope. The X-ray diffraction (XRD) analysis showed the presence of the FeB, Fe₂B, Cr₂B and Ni₂B phases on the surface of the borided samples. The Daimler-Benz Rockwell-C adhesion test was carried out to evaluate the adhesive strength of the boride layer to the substrate material. The tests were repeated at least 3 times for each of the samples pack-borided at all process temperatures and times. After the adhesion tests, macro and SEM images of indentation traces were taken. By analysing the indentation craters, it has been determined whether the damages are acceptable or not with reference to the VDI 3198 standard. The indentation craters formed on the surfaces of the samples were pack-borided at 850 °C for all process times, at 900 °C for 2 and 4 hours, and at 950 °C for 2 hours have the best adhesion quality in the HF1 category of the VDI 3198 norm. The pack-boriding treatment with microwave hybrid heating contributed positively to the adhesion strength, but in additions to this, the test results revealed that adhesion decreased with increasing boriding temperature and time.

Keywords: Adhesion test, Daimler-Benz Rockwell-C, microwave hybrid heating, pack-boriding, VDI 3198 norm.

1. Introduction

Boriding changes the microstructure and composition of the material surface by thermochemical diffusion of boron atoms to the material surface; creates a functional surface layer with optimum surface properties. High hardness and low friction coefficient are two important mechanical properties that the boride layer that forms as a result of boriding gives the material surface. Thanks to these features, the major problem that shortens or ends the service life of engineering materials, namely surface wear, is prevented. Morón et al. [1], in their study, reduced the friction coefficient of the AISI H13 steel, which was initially in the range of 0.64-0.71, to 0.10-0.11 values, which they applied pack-boriding at 950 °C for 6 hours, and increased the abrasion resistance by 23 times in the lubricated environment. Kayali et al. [2] achieved a 30-fold reduction in the abrasion rate of the AISI 316L stainless steel alloy that borided with 2 and 6

hours of hold times at 800 and 900 °C. Material surfaces working in contact with each other are connected with micro-welds after a while. With the ongoing relative movement breaking these bonds, the relatively less hard material is transferred to the opposite element, thus creating gaps on the less hard material surface and protrusions on the other surface. This surface wear resulting in material loss is called adhesive wear. Surface hardening is the leading measure to be taken to increase the adhesive strength on the material surface. However, conventional surface hardening methods cannot be applied to austenitic stainless steels. Because they protect their austenitic microstructure from room temperature to high temperatures. For this reason, boriding is widely preferred in order to increase surface hardness due to its easy applicability and economy. In their study, Alias et al. [3] increased the surface hardness of AISI 304 austenitic stainless steel alloy 5 times with the pack-boriding process at 850 °C and for 8

hours. In many studies in the literature, high surface hardness values have been obtained in different types of steels as well as austenitic stainless steels by applying boriding process [4-19]. Thanks to boriding, the surface hardness of the material not only increases; since the chemical reactivity of boron against oxygen is high, a thin oxide film forms on the boride layer. This layer acts as a solid lubricant, reducing the friction coefficient [20, 21]. Boride layer has a low tendency to cold weld so it does not need to be oiled to prevent adhesive wear. Taktak ve Tasgetiren [22] subjected AISI H13 and AISI 304 steels to boriding in a slurry salt bath at 800–950 °C for 3, 5 and 7 hours. The quality of adhesion strength of the boride layers formed on both steel materials borided at 800 °C is in the HF1 and HF2 category and is very high. However, it has been reported that the adhesion quality of the boride layer decreases as the process temperature and time increases. The increase in the quality of adhesion strength between the boride layer and the matrix material increases the adhesion resistance. As the adhesion quality deteriorates, crack formation starts due to internal stresses and progresses, resulting in delaminations on the material surface.

Krelling et al. [23] applied pack-boriding process on AISI 1020 steel with a 4 hour retention time at 1000 °C. After Rockwell C indentation tests, they obtained HF1 quality adhesion strength between the boride layer and the substrate and reported the result that the boriding gave a good adhesion resistance.

This study aimed to increase the adhesion resistance by using a microwave hybrid heating system as a thermal energy source for pack-boriding process. The adhesion strength analysis of the boride layer formed on the surface of AISI 304L stainless steel samples, on which pack boriding was applied for 2, 4 and 6 hours at 850, 900 and 950 °C temperatures with microwave hybrid heating methods, to the matrix material was evaluated by Daimler-Benz Rockwell-C adhesion test.

2. Experimental Procedure

2.1. Pack-boriding process with microwave hybrid heating

The test specimens were manufactured from AISI 304L stainless steel material, whose standard chemical composition is given in Table 1, with a diameter of 20mm and a height of 10 mm. The samples were ground up to 1200 grid and subjected to ultrasonic bath for 30 minutes before the thermochemical treatment. The pack-boriding treatments were carried out in a microwave furnace at 850, 900 ve 950 °C for 2, 4 and 6 h, and the samples were allowed to cool in the open air at the end of the process. Commercial Ekabor-II powder was used as the boriding agent. The samples were placed in AISI 304 stainless steel containers and

covered with boriding powder. The schematic picture of the microwave sintering furnace with the microwave hybrid heating mechanism where the pack-boriding process takes place is given in Figure 1. Heating in the microwave heating system is provided by both microwave radiation and convection heat transfer mechanisms. That's why hybrid heating is in question. Thanks to the microwave absorbing plates around the boriding crucible, which is heated volumetrically by microwave radiation, convection heat transfer accompanies it. This hybrid heating system, which increases thermal diffusion and saves energy with homogeneous heating and homogeneous temperature distribution, is an improvement innovation in the pack-boriding process. The morphology of the boride layer formed on the sample surfaces after the boriding process was examined by optical microscope at x200 magnifications, and the phases formed in the layer were determined by XRD. The samples for microstructural analyses were sanded with SiC abrasive paper up to 1200 grid, polished with 1 µm diamond polishing solution, and etched with Glyceregia solution.

2.2. Adhesion test and characterization

The Daimler-Benz Rockwell-C adhesion test was used to evaluate the adhesion of boride layers. The Rockwell-C indentation test is specified according to the VDI 3198 norm, as a quality test for coated materials. In this test, the plastic deformation that occurs as a result of the penetration of the conical end indenter into the surface of the coated material determines the adhesion quality of the coating. The thickness of the sample must be at least 10 times greater than the indentation depth. The type and volume of damage to the coating gives information about the adhesion of the coating layer at first glance and the fragility secondly. The principle of the method and the quality categories of the adhesion strength of the damage in the coating layer after the test are shown in Figure 2. Grades between HF1-HF4 indicate the presence of sufficient adhesion in the coating layer; the HF5 and HF6 categories indicate insufficient adhesion strength, ie failure of the coating [24, 25]. Indentation processes, BMS 200-RB brand Rockwell hardness measurement device in accordance with VDI 3198 indentation test standards, with 120° conical tip and 150 kg load after repeating 3 times, the craters formed on the sample surfaces were examined using a stereo microscope and SEM (scanning electron microscope). By analyzing the cracks, delaminations and fractures occurring in the boride layer of each sample, it was determined whether the damages were acceptable by referring to the damage categories (HF1-HF6) of the VDI 3198 indentation test standard in Figure 2.

Table 1. AISI 304L stainless steel material chemical composition

Element	C	Mn	P	S	Si	Cr	Ni	N
Wt %	≤ 0.030	≤ 2.00	≤ 0.045	≤ 0.030	≤ 0.75	18-20	8-10	≤ 0,10

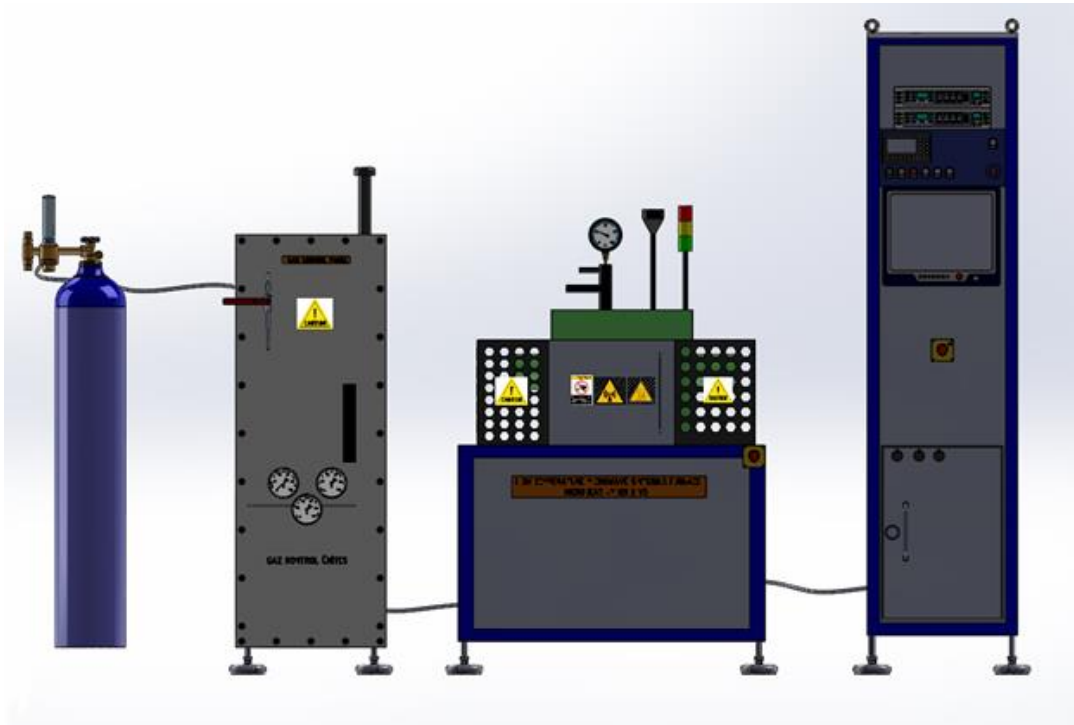


Figure 1. Schematic picture of microwave sintering furnace

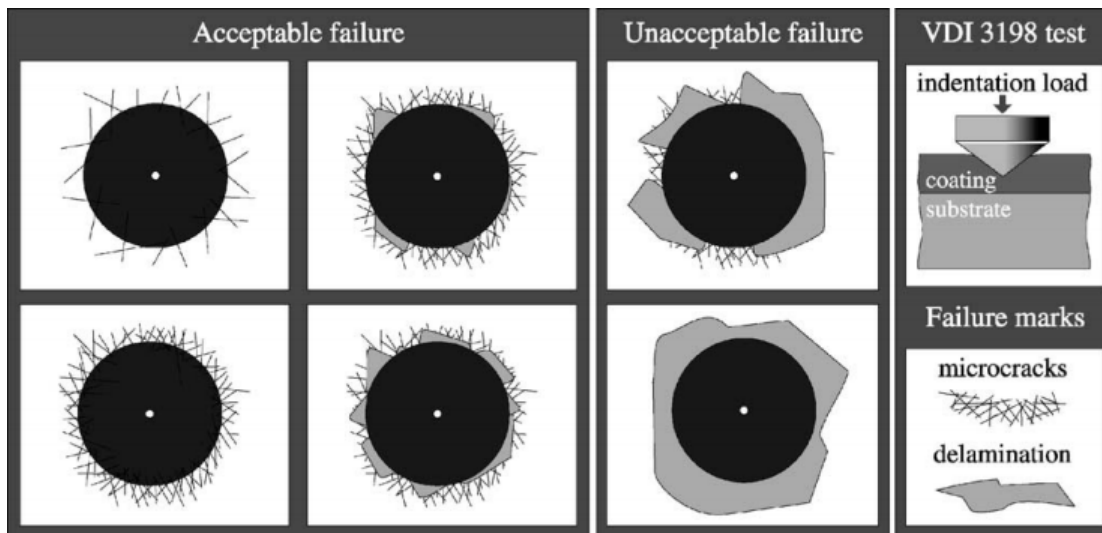


Figure 2. VDI 3198 indentation test adhesion quality classification [24]

3. Results and Discussion

3.1. The Characterization of Boride Layers

In the optical microscope images, the boride layers formed after boriding on the cylindrical samples of AISI 304L stainless steel are bilayer ($Fe_2B + FeB$) and can be clearly distinguished from the base material. As can be seen from Figure 3, the increase in process temperature

and retention time increased the boride layer thickness. Due to the high amount of alloying elements in the structure of AISI 304L stainless steel material, the boride layer formed on its surface has flat and smooth morphology. In Figure 4, XRD analysis showed the presence of Cr_2B and Ni_2B phases in the boride layer besides Fe_2B and FeB phases.

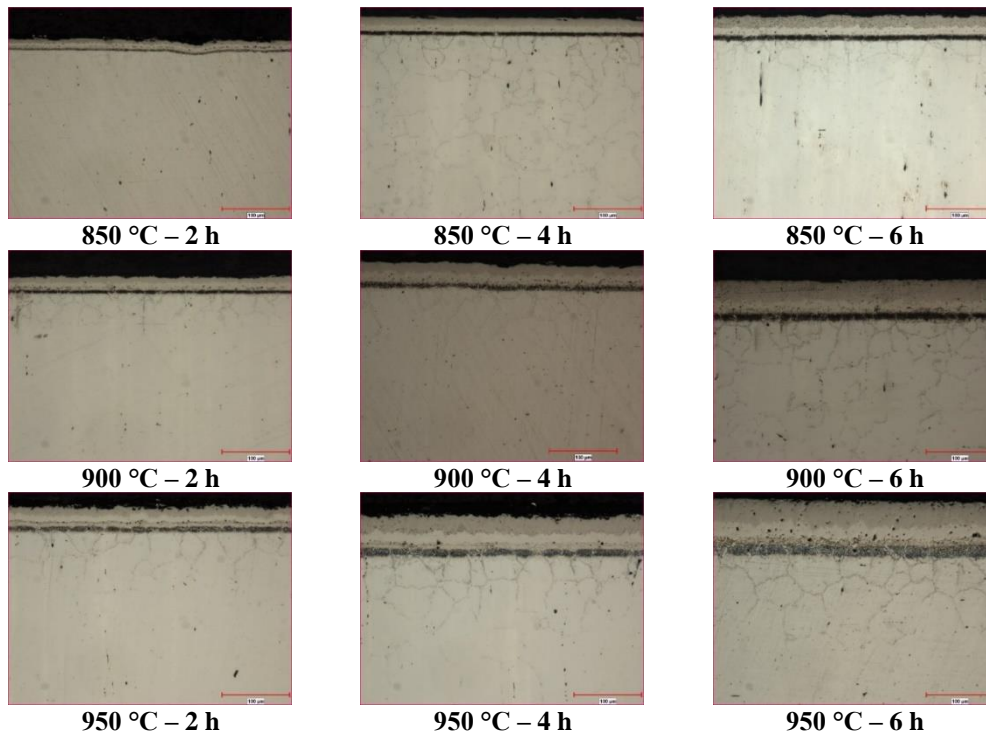


Figure 3. Optical microscope images of AISI 304L samples pack-borided at process temperatures and times with microwave hybrid heating: x200 magnification

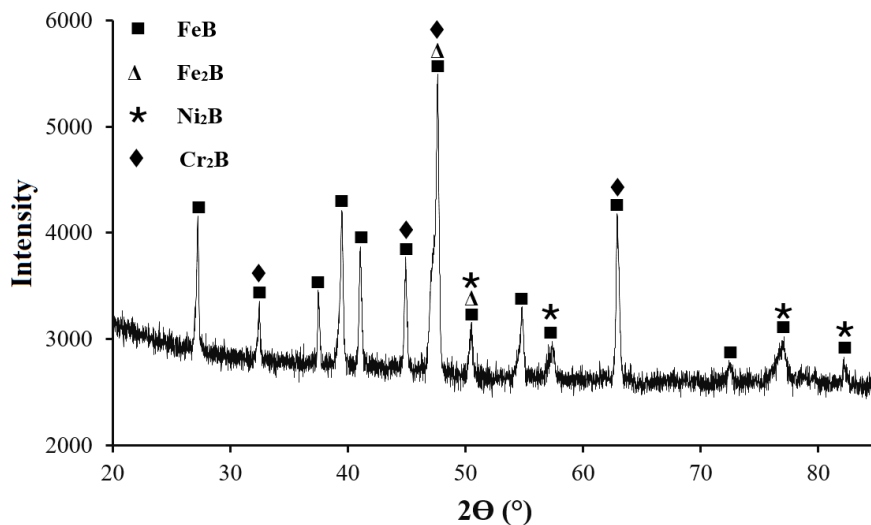


Figure 4. XRD pattern of the pack-borided AISI 304L steel at 900 °C for 6 hours

3.2. The adhesion resistance of boride layer

Macro and SEM images of the indentation traces taken after the adhesion tests on AISI 304L stainless steel samples pack-borided with microwave hybrid heating method are given in Figure 5-18. Whether the appearance of damage occurring in the adhesion test results is acceptable or not was evaluated according to the VDI 3198 norm. In Figure 5, the indentation craters formed on AISI 304L stainless steel sample surfaces, which are pack-borided at 850 °C for all process periods, at 900 °C for 2 and 4 hours, and at 950 °C for 2 hours by microwave hybrid heating method, are as in

the HF1 category of the VDI 3198 and are acceptable. HF1 has the best adhesion quality. Therefore, the adhesion strength of the samples pack-borided at these process temperatures and times is quite good. However, as can be seen in Figure 6, the boride layer on the surface of one of the samples, which was pack-borided at 900 °C for 6 hours, was broken up during the indentation test, and in another there are spillings. A spalling was observed in the boride layer of one of each sample which was pack-borided at 950 °C for 4 and 6 hours. Damages in these samples are of HF5 quality and are unacceptable.

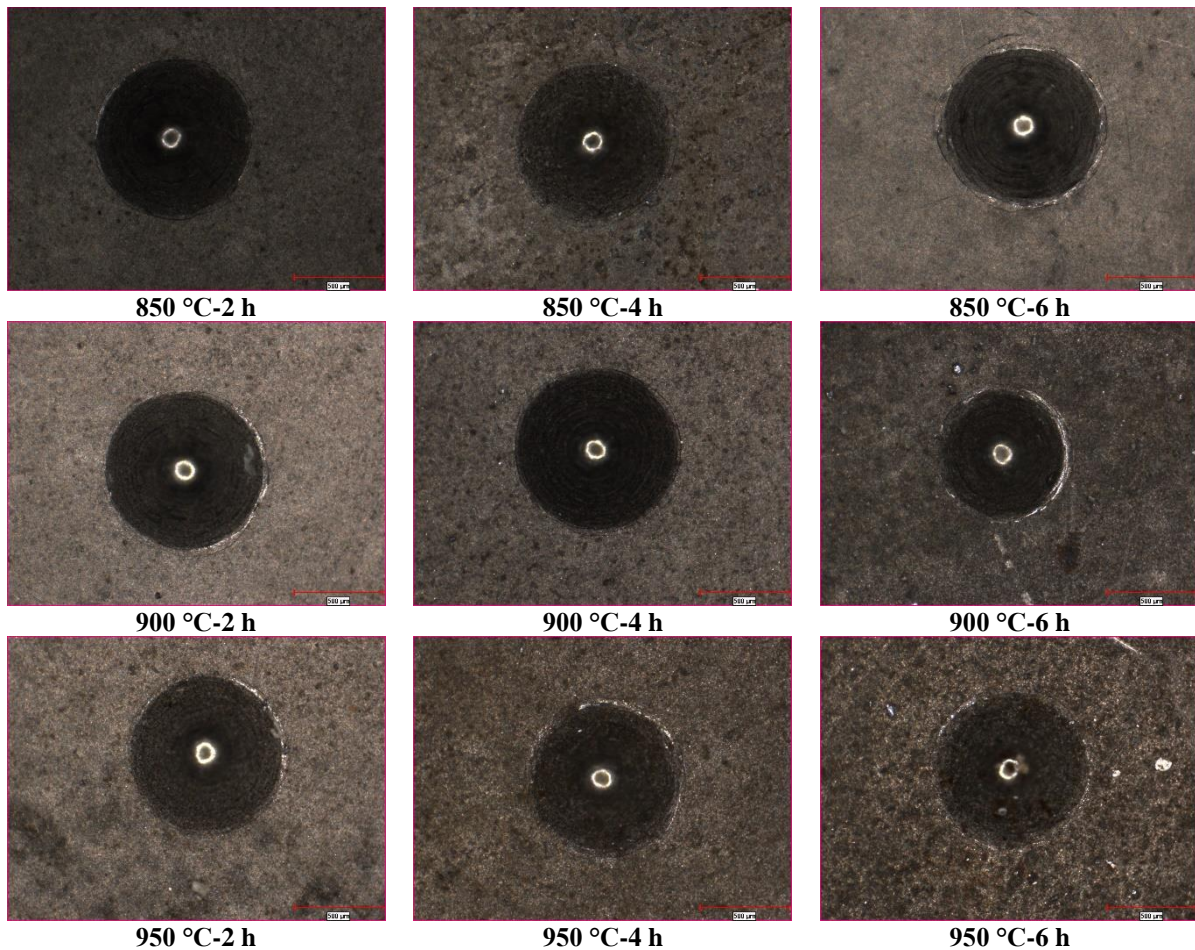


Figure 5. Macro images of the crater formed after the adhesion test on AISI 304L samples pack-borided with microwave hybrid heating



Figure 6. Boride layer removals occurring after the adhesion test in AISI 304L samples pack-borided with microwave hybrid heating

When the SEM images of the indentation traces of the boride layers of AISI 304L stainless steel samples pack-borided with microwave hybrid heating method in Figure 7-11 and Figure 14 are examined, it is more clearly seen that the acceptable damage in these samples

are HF1 quality micro cracks. These micro cracks are in the form of lateral cracks and generally capillary, and belong to the best quality category according to the VDI 3198 norm. Figure 12 shows the SEM image taken after the adhesion test of one of the AISI 304L samples pack-

borided with microwave hybrid heating method at 900 °C with a retention time of 6 hours. It is seen that the adhesion strength quality belongs to the HF3 category due to the curvilinear cracks and occasional spallings in the boride layer on the surface of this sample. The SEM image taken after the adhesion test of another sample pack-borided at the same process temperature and time is given in Figure 13. There are spallation and wear debris in the boride layer of the sample. The adhesion strength of this sample between the boride layer and the base material is HF5 quality and is in unacceptable category.

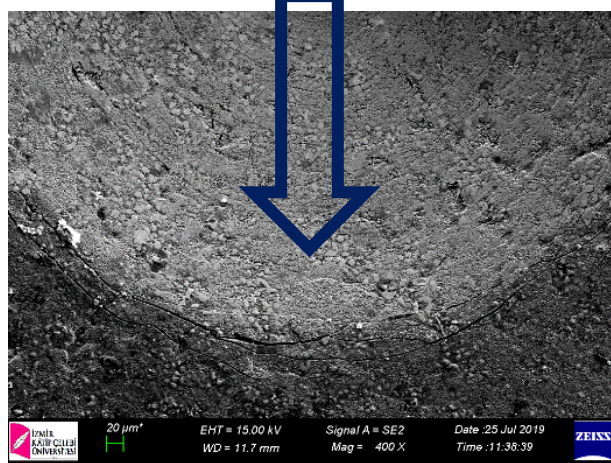
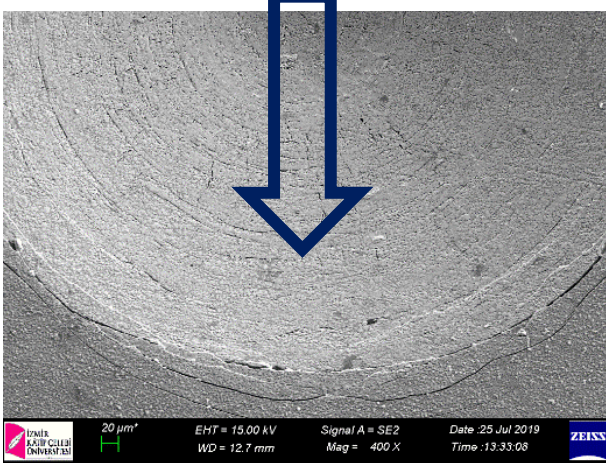
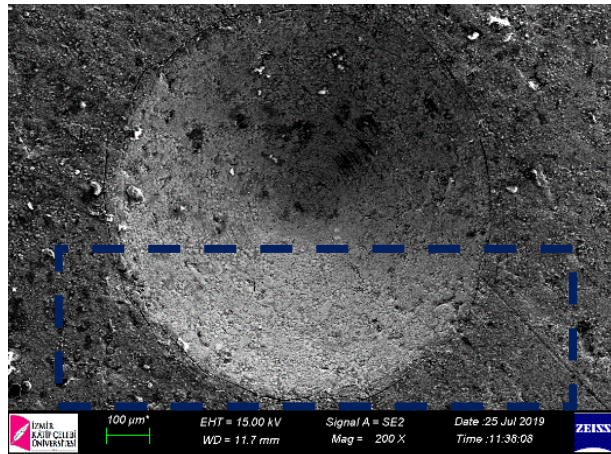
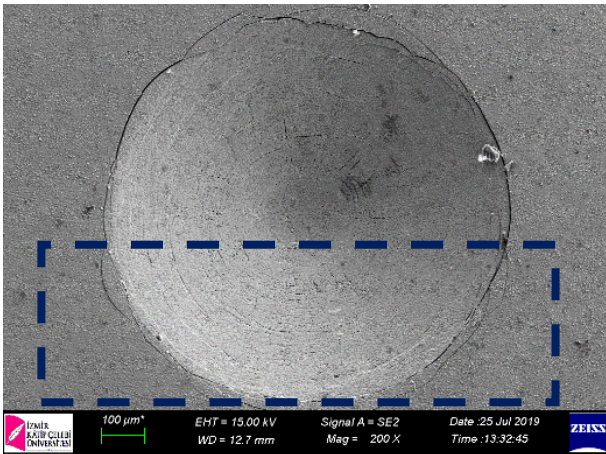


Figure 7. SEM images of the traces and micro cracks formed after the adhesion test on the AISI 304L sample pack-borided at 850 °C for 2 hours with microwave hybrid heating

Figure 8. SEM images of the traces and micro cracks formed after the adhesion test in AISI 304L sample pack-borided at 850 °C for 4 hours with microwave hybrid heating

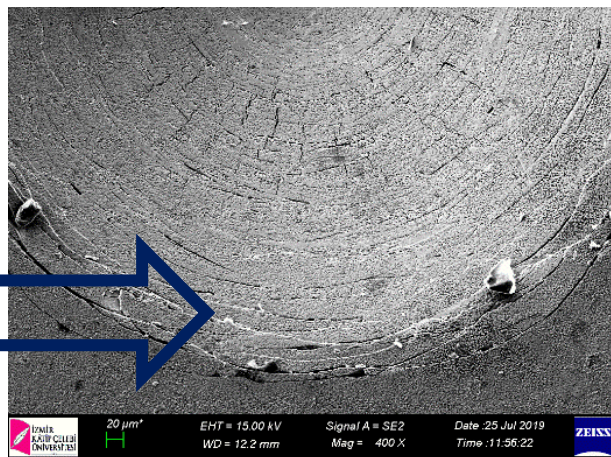
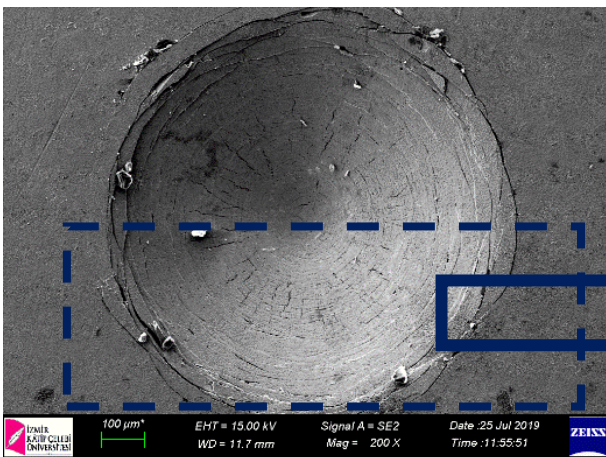


Figure 9. SEM images of the traces and micro cracks formed after the adhesion test on the AISI 304L sample pack-borided at 850 °C for 6 hours with microwave hybrid heating

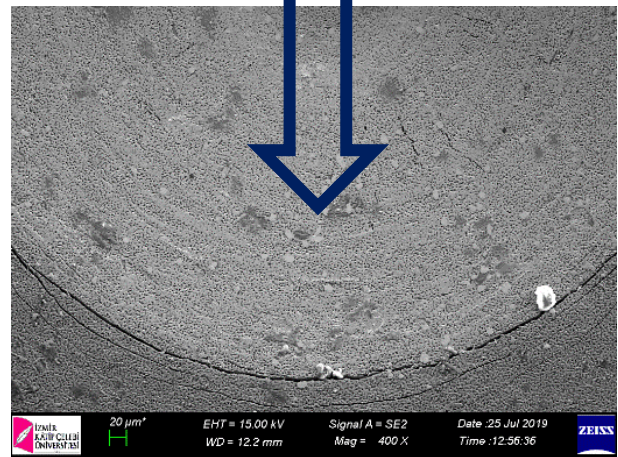
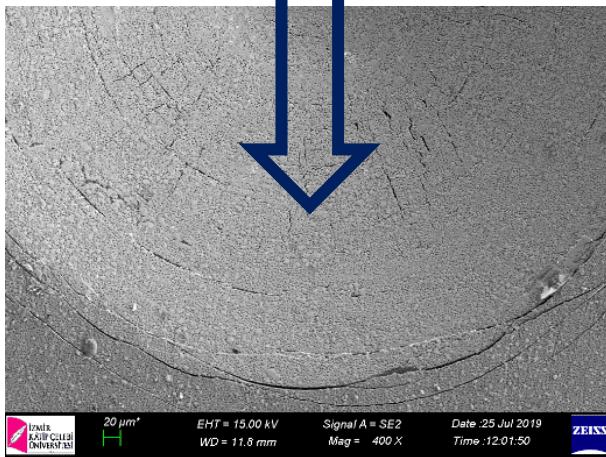
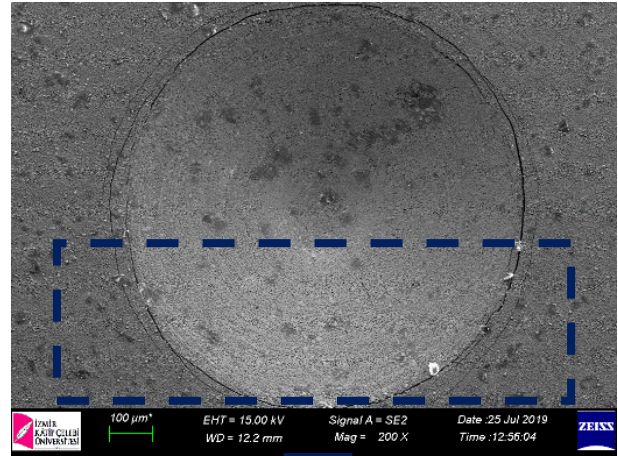
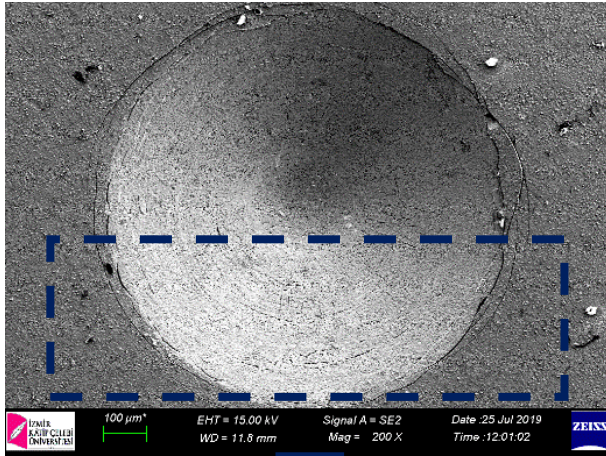


Figure 10. SEM images of the traces and micro cracks formed after the adhesion test in AISI 304L sample pack-borided at 900 °C for 2 hours with microwave hybrid heating

Figure 11. SEM images of the traces and micro cracks formed after the adhesion test in AISI 304L sample pack-borided at 900 °C for 4 hours with microwave hybrid heating

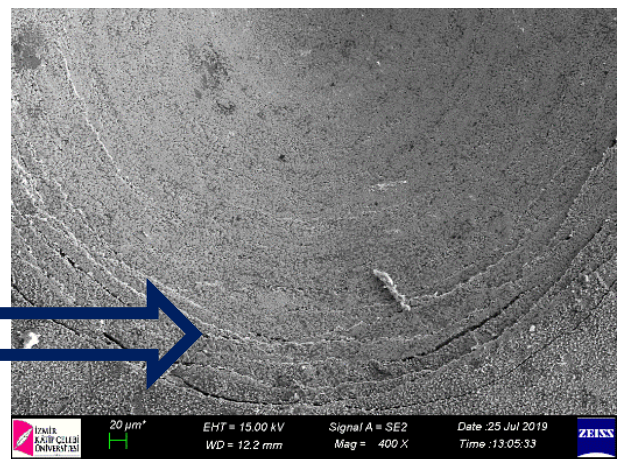
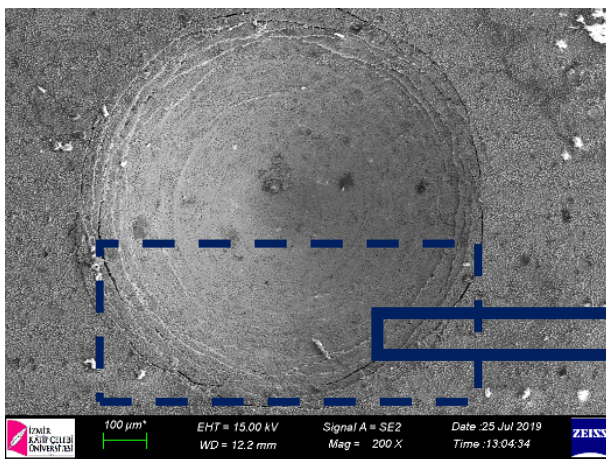


Figure 12. SEM images of the traces and micro cracks formed after the adhesion test on the AISI 304L sample pack-borided at 900 °C for 6 hours with microwave hybrid heating

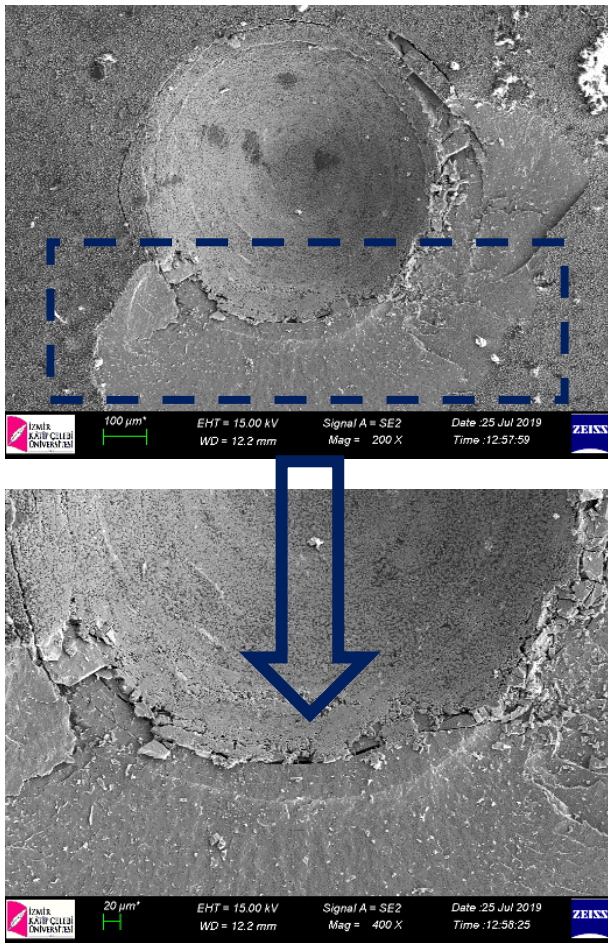


Figure 13. SEM images of the fracture formed after the adhesion test in AISI 304L sample pack-borided for 6 hours at 900 °C with microwave hybrid heating

The indentation craters belonging to one of the pack-borided samples with microwave hybrid heating at 950 °C for 4 hours are shown in Figure 15. There are abrasion scratches and burrs in the boride layer after the adhesion test. This damage is of HF3 type and is acceptable.

The damage appearance after the adhesion test of another sample, which is pack-borided with microwave hybrid heating at 950 °C for 4 hours, is shown in Figure 16. Delaminations occurred in the boride layer of the sample. According to the VDI 3198 indentation test principles, this damage is of the HF5 type and is unacceptable.

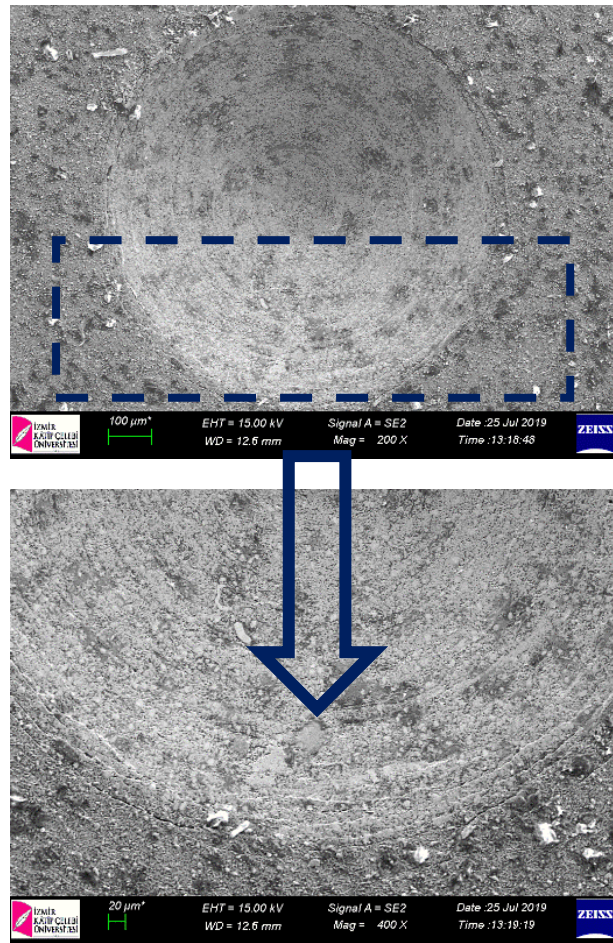


Figure 14. SEM images of the traces and micro cracks formed after the adhesion test of AISI 304L samples pack-borided at 950 °C for 2 hours with microwave hybrid heating

The damage appearance after the indentation test of one of the samples pack-borided with microwave hybrid heating at 950 °C for 6 hours is given in Figure 17. Mosaic cracks and wear lines were formed in the boride layer. This damage is in the HF3 category and is acceptable.

The SEM image showing the damage appearance after the adhesion test of another sample that is pack-borided with microwave hybrid heating at 950 °C for 6 hours is given in Figure 18. Local spallation and delaminations occurred in the boride layer of the sample. According to the VDI 3198 indentation test principles, this damage is of the HF5 type and is unacceptable.

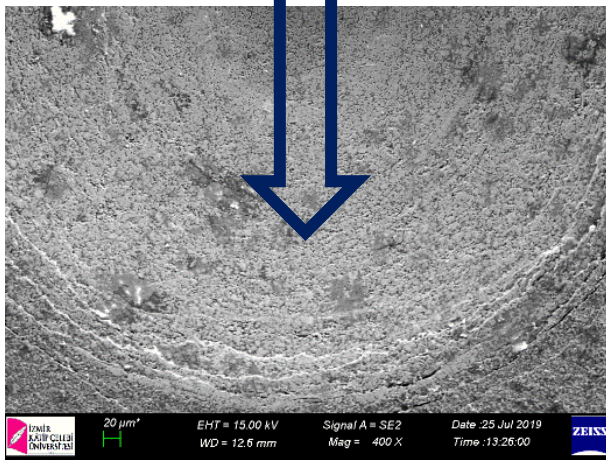
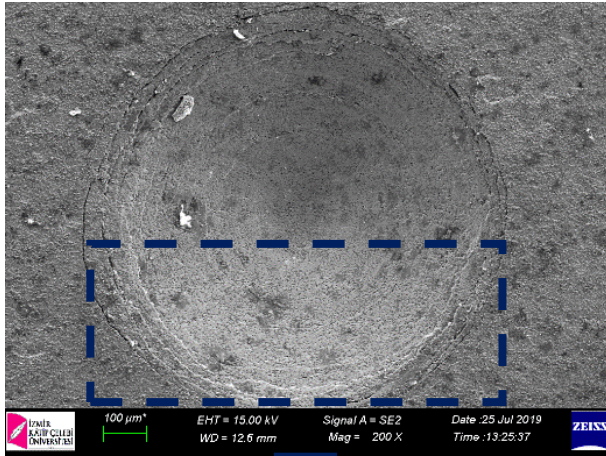


Figure 15. SEM images of the traces and micro cracks formed after the adhesion test of AISI 304L samples pack-borided at 950 °C for 4 hours with microwave hybrid heating

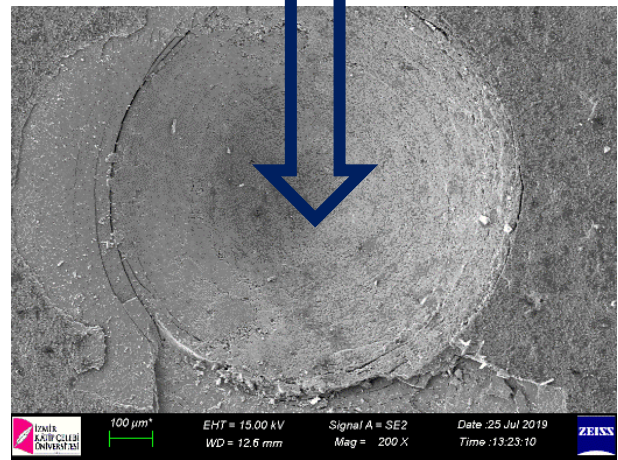
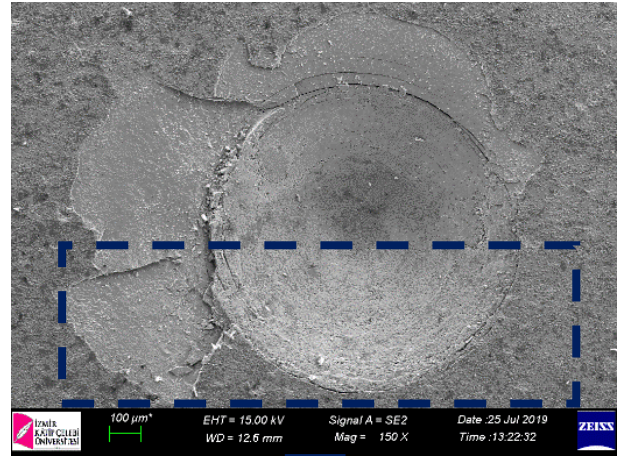


Figure 16. SEM images of the delaminations and cracks formed after the adhesion test of AISI 304L samples pack-borided at 950 °C for 4 hours with microwave hybrid heating

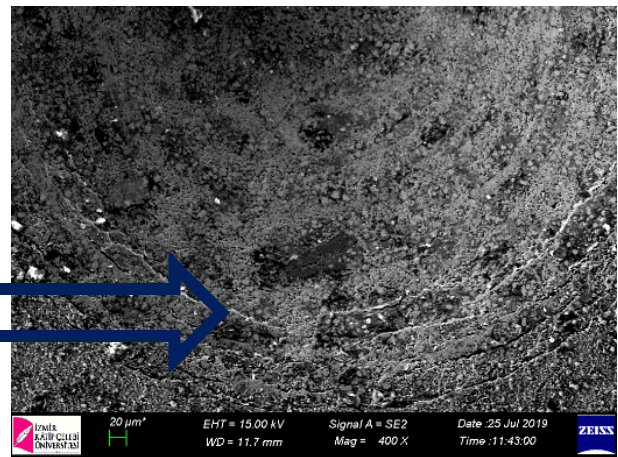
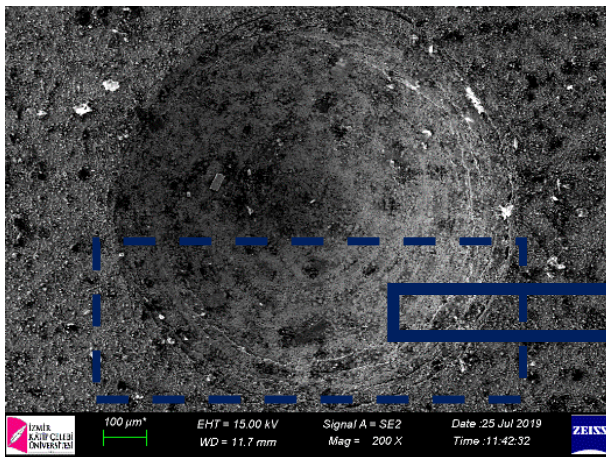


Figure 17. SEM images of the traces and cracks formed after the adhesion test of AISI 304L samples pack-borided at 950 °C for 6 hours with microwave hybrid heating

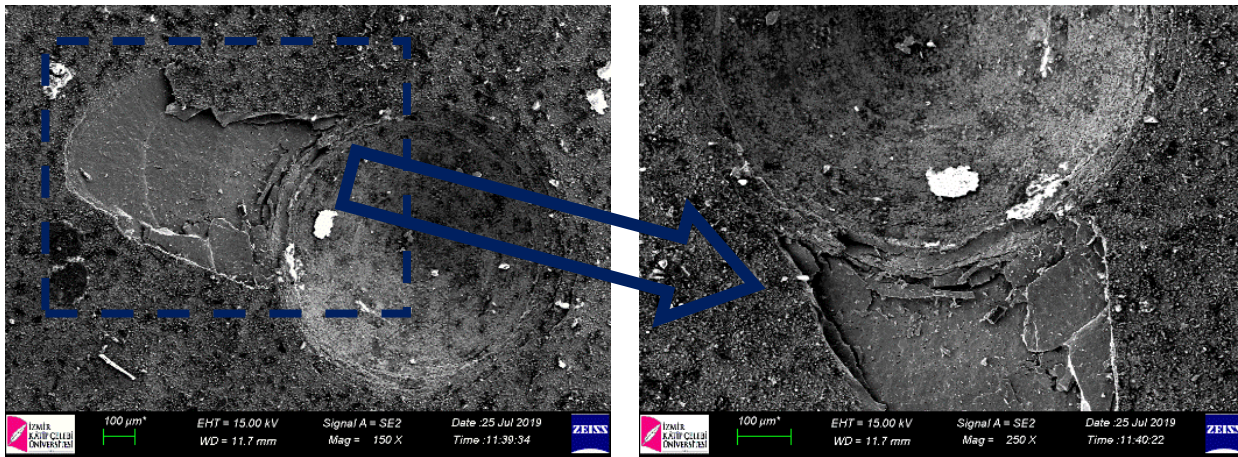


Figure 18. SEM images of the spallation and delaminations formed after the adhesion test of AISI 304L samples pack-borided at 950 °C for 6 hours with microwave hybrid heating

4. Conclusions

Optical microscope examinations showed that both FeB and Fe₂B phase thicknesses increased with the increase of process temperature and retention time in pack-boriding processes performed with microwave hybrid heating method. Uniform and homogeneous boride layers were obtained. However, boride layers formed by the effect of alloying elements are not saw-toothed but flat structure.

XRD pattern analysis showed the presence of FeB, Fe₂B, Cr₂B and Ni₂B phases in the boride layer.

As a result of the Daimler-Benz Rockwell-C adhesion tests carried out, the adhesion strength of the boride layers-substrate of all samples is high, except for the samples that are pack-borided for 6 hours at 900 °C, and at 950 °C for 4 and 6 hours with microwave hybrid heating because the adhesion has decreased with increasing boriding temperature and time. The main reason for the low adhesion strength in these samples is the delamination and spallation of the boride layer as a result of the increase in the thickness of the brittle FeB phase due to the increase in the temperature and retention time of the pack-boriding process.

Author's Contributions

Dilek Arslan: Carried out the experimental processes and examined the results. The manuscript was drafted and written by her.

Recep Onur Uzun: Supervised the experimental procedure and contributed to manuscript preparation.

Ethics

There are no ethical issues after the publication of this manuscript.

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