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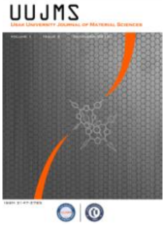

Research article

Niobium boride coating on the surface of AISI 1010 by a duplex treatment

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Abstract

In this study, niobium boride layers were deposited on the surface of AISI 1010 steel by a duplex surface treatment. In the first step of duplex treatment, boronizing treatment was performed at 900°C for 2 h and then pre-boronized steels were niobized at 850, 900, 950°C for 1, 2, 3 and 4 h using thermo-reactive deposition/diffusion (TRD) method. Presence of the niobium boride phases were confirmed by Xray diffraction (XRD), scanning electron microscope (SEM) and microhardness methods. The hardness of coating layer was 2598±482. Experimental results show that the depth of the coating layers changes between 1.5 μm to 7.19 μm. The higher temperature the higher thickness of coating layer was obtained.

Keywords: *Niobium boride, Thermo-reactive deposition/diffusion method, AISI 1010 steel*

1. Introduction

Deposition of hard thin coatings with a few microns in thickness such as carbide, nitride, carbonitride and boride of transition metals is a common method to improve the performance of tools, dies, molds and for many different ferrous metals. To fabricate thin hard coatings, two kinds of processes namely chemical vapour deposition (CVD) and physical vapour deposition (PVD) have been put into practical application in worldwide industries due to their respective advantages. CVD usually involves high processing temperatures (700–1200°C) in order to achieve deposition of the coating material. However, this high temperature can lead to the heavy distortion of the treated part. PVD process, which is usually performed at 200–500°C, well below the tempering range of tool steels, requires expensive and complicated equipment. Due to the limited amount of diffusion that occurs during PVD process, adhesion strength of the coating layer is weaker than that of thermoreactive diffusion (TRD) treatments [1-3]. TRD is an alternative method of coating steels with a hard, wear-resistant layer of carbides, nitrides, carbonitrides or borides [4]. Conventionally, this is a high temperature process, operating at 800–1200°C. In the process, one of carbide, nitride and/or boride forming

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elements (Cr, V, Ti, etc.) is deposited on the surface by a thermo-chemical process. These elements react with carbon, nitrogen and boron atom containing the substrate. The main limitation of the TRD process is that the steel substrate must contain enough carbon, nitrogen or boron content; otherwise the coating thickness becomes severely limited. For this purpose, steel's that contain enough carbon, nitrogen or boron is suitable for the coating treatment, or the surface composition of the steel can be enriched with C, B, and N elements by carburizing, nitriding, carbo-nitriding or boronizing treatments before the TRD coating [5].

In the present work, the niobium boride layers were deposited on the surface of AISI 1010 steel substrates. The first step of the duplex treatment was the boronizing treatment to produce iron boride phases on substrates. The second step was niobizing treatment for producing niobium boride phases on the pre-boronized steel substrates. The main goal was to investigate the structural characterization of niobium boride layer formed.

2. Material and Method

In this study, AISI 1010 low carbon steel was used as substrates, the composition of which is given in Table 1. Before the duplex treatment, the samples were cut in to the dimensions of 20 mm in diameter and 5 mm in length, and ground up to 1200 grid emery paper and then washed ultrasonically for 15 min in ethyl alcohol. The boronizing treatment was carried out in a slurry salt bath consisting of borax, boric acid and ferro-silicon at 900°C for 2 h in the first step of the duplex treatment.

Table 1
Chemical composition of AISI 1010 steel

Sample	Chemical composition (wt%)							
	C	Si	Mn	P	S	Cr	Mo	Fe
AISI 1010	0.12	0.11	0.05	0.003	0.023	0.012	0.02	Balance

In the second step, pre-boronized steel samples were niobized by pack method in the powder mixture consisting of ferro-niobium, ammonium chloride and alumina at 850-900-950°C for 1–4h. The samples were directly immersed in the powder mixture in the alumina crucible. An alumina lid was used to close the box and alumina cement was used for sealing the crucible. After the treatment, the samples were cooled in the box for 1 h in the air. The boro-niobized samples were grounded and polished up to 0.3 μm with alumina paste and then etched with 3% nital for metallographic examinations. Nickon Epiphot 200 optical microscope with optical micrometer was used for measuring the depth of coating layer formed on the steel samples. Micro-hardness measurements of the layers from surface were performed 10 times using Shimadzu HV2 micro-hardness tester under the loads of 10g. Average value of the hardness of the coating layer and standard deviation was calculated. X-ray diffraction (XRD) analyses of the layers were performed on the surfaces of the coated sample with 2θ varying from 10° to 90°, using CuKα radiation.

3. Results and Conclusion

3.1. Results

Borides of niobium are recognized as potential candidates for high-temperature structural applications, due primarily to their excellent properties such as high melting temperature, high strength, high thermal and electrical conductivity, and good chemical stability. The phase diagram of the Nb–B system in Fig. 1 shows the presence of five borides, including Nb_3B_2, NbB, Nb_5B_6, Nb_3B_4, and NbB_2 [6].

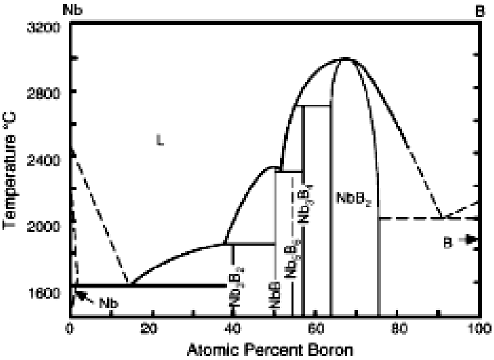

Fig. 1 Phase diagram of Nb-B system

In our study, coating layer was composed of three distinct regions on the surface of niobium boride coated substrates; these are (i) a niobium boride layer formed on the surface of the coated steel, (ii) an iron boride layer formed under the niobium boride layer and (iii) a matrix as shown in Fig. 1. SEM cross-sectional view shows that niobium boride layer formed on the pre-boronized AISI 1010 steel surfaces are of compact, smooth and porosity-free morphology and homogeneous the entire surface of steel sample, and niobium concentration in the coating layer is much higher than that of the inner part of the coated steel, and the iron concentration of the inner part of the coated steel is much higher than that of the surface, as seen in Figs. 2a, b and c. These results are in agreement with Ref. [7].

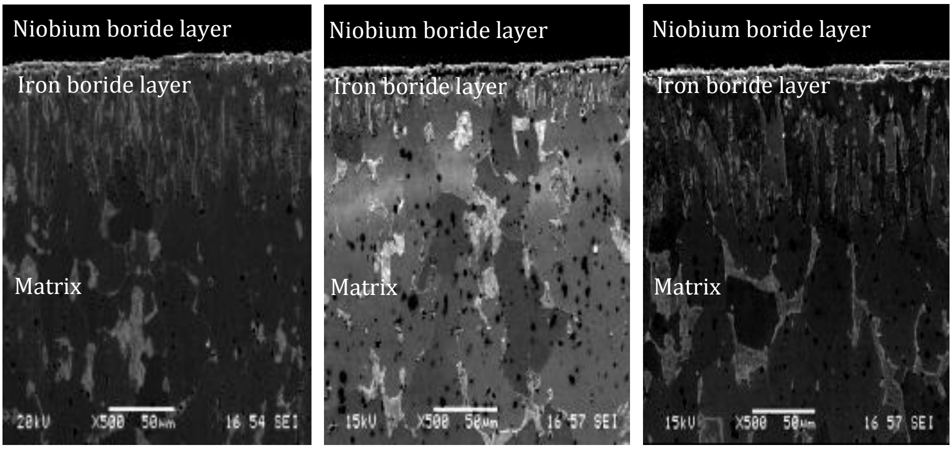

Fig. 2 SEM images of boro-niobized AISI 1010 steel at (a)850°C, (b)900°C, (c)950°C for 4h

The hardness of the niobium boride layer was 2598±482 $HV_{0.01}$ which was much harder than boronized layer (1452 HV) and matrix (126 HV). These are due to the presence of hard niobium boride phases such as NbB, NbB_2 and Nb_3B_4 at the coating layer as verified by electron dispersive spectrometer (EDS) and XRD analysis as shown in Fig. 3 and Fig. 4. This kind of niobium boride phases were also obtained at earlier studies [6, 8-11].

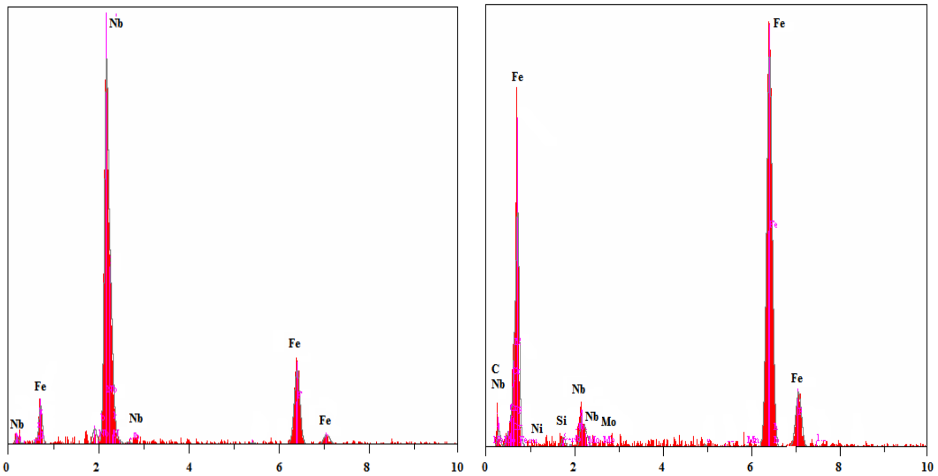

Fig. 3 EDS analysis of boro-niobized substrate at 950°C for 4h (a) niobized layer (b) matrix material

The depth of the niobium boride layers changes between 1.5 μm to 7.19 μm, depending on the treatment temperatures and times as shown in Fig. 5. The higher the treatment time, the thicker the niobium boride layer became. In the thermo-chemical coating processes, an increase in the process time and temperature cause to increase in the coating layer thickness. Composition, substrate, treatment time and temperature all affect the coating layer thickness in the thermo-reactive deposition processes according to Ref. [5]. Depth of the niobium boride layers on the AISI 1010 steel is lower which may explain by lower diffusivity of boron atoms into niobium compared to earlier studies [12,13]. This may be explained by the lower diffusivity of boron atoms into niobium compared to vanadium or tungsten.

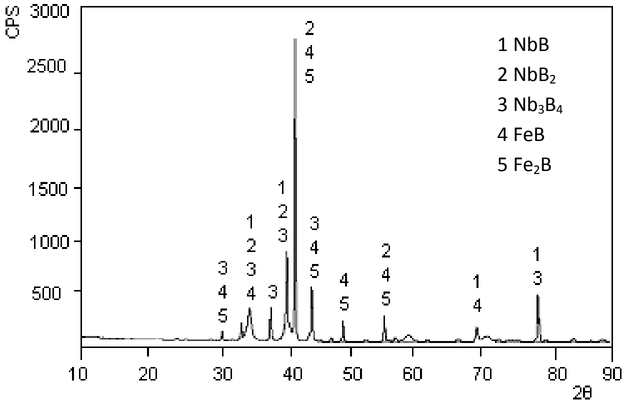

Fig. 4 XRD patterns of boro-niobized AISI 1010 steel at 900°C for 2h

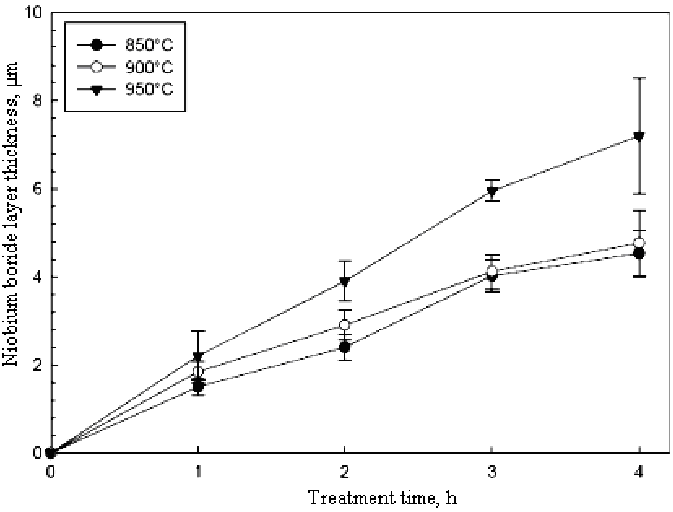

Fig. 5 Niobium boride layer thickness depending on boro-niobizing temperatures and times

3.2. Conclusion

- Niobium boride coating can be formed on the pre-boronized steel sample by TRD method.
- The coating layer has denticular, dense and porosity-free morphology.
- The thicker niobium boride layer was obtained at 950°C for 4h.
- Niobium boride layer thickness on the AISI 1010 steel ranged from 1.5 μm to 7.19 μm depending on treatment times and temperatures.
- The Vickers microhardness of the nibium boride layer was measured to be 2598±482 $HV_{0.01}$.

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