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Niobium boride coating on AISI M2 steel by boro-niobizing treatment

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Abstract

In this study, niobium boride coating was applied on pre-boronized AISI M2 steel by the thermo-reactive deposition technique in a powder mixture consisting of ferro-niobium, ammonium chloride and alumina at 950 °C for 1–4 h. The coated samples were characterized by X-ray diffraction, scanning electron microscope and micro-hardness tests. Niobium boride layer formed on the pre-boronized AISI M2 steel was smooth, compact and homogeneous. X-ray studies showed that the phases formed on the steel surfaces are NbB, Nb_3B_2, FeB and Fe_2B. The depth of the niobium boride layer ranged from 0.97 μm to 3.25 μm, depending on treatment time. The higher the treatment time the thicker the niobium boride layer observed. The hardness of the niobium boride layer was 2738±353 $HV_{0.01}$.
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Keywords: Borides; Thermo-reactive deposition; Niobium boride; AISI M2 steel

1. Introduction

The use of hard coatings obtained by the thermo-chemical treatments opens up the possibilities for materials designed, in which the specific properties are located where they are most needed. The substrate material can be designed for strength and toughness, while the coating is responsible for resistance to wear, corrosion and oxidation and thermal loads [1]. Hard coatings with boride, carbide, nitride or carbonitride of transition metals are common to improve the wear resistance of ferrous materials [2,3]. Transition metal boride coatings have attractive properties such as high melting points, high mechanical strength and chemical inertness [4]. Especially, borides, carbides and nitrides of niobium, vanadium, chromium and titanium are famous among the transition metals [5–8].

Niobium borides are recognized among the transition metal borides 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, good chemical stability and high wear resistance [9–12]. This type of coating can be produced by several methods along with CVD and PVD, both of which have their respective advantages [3]. Besides CVD and PVD techniques to deposit niobium boride coating, thermo-reactive deposition technique can also be utilized for this coating on pre-boronized steel base alloys. Multi-component boriding is a thermo-chemical treatment involving consecutive diffusion of boron and one or more metallic elements such as niobium, aluminum, chromium, vanadium and titanium on to the component surface. This process is carried out at 850 to 1100 °C and involves two steps: (i) boriding by conventional method and (ii) diffusing metallic elements

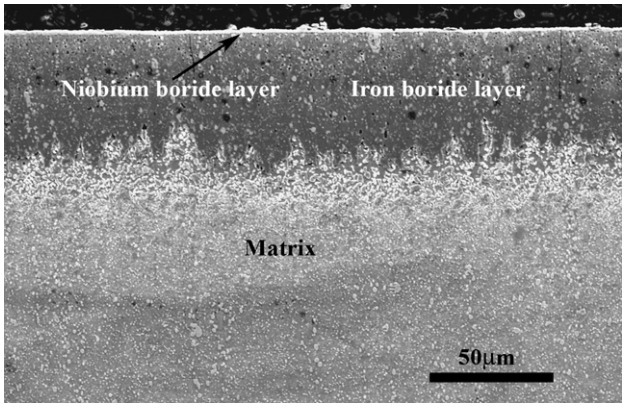


Fig. 1. SEM image of boro-niobized AISI M2 steel at 900 °C for 2 h.

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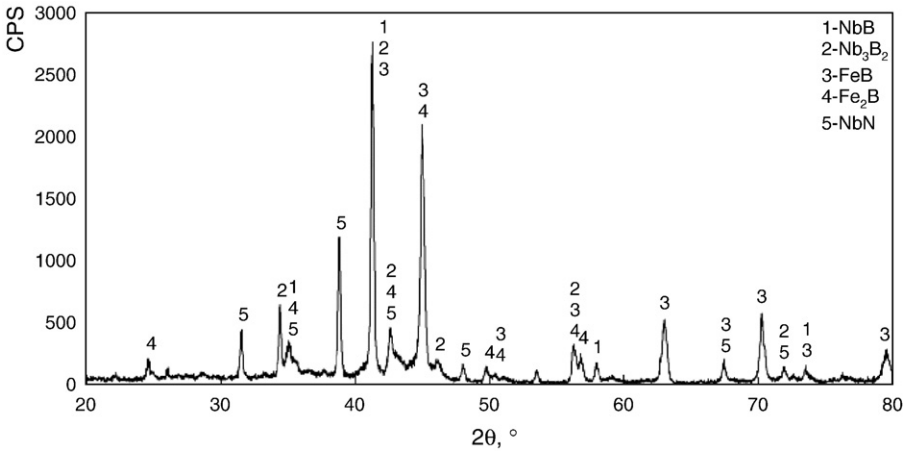


Fig. 2. X-ray diffraction patterns of boro-niobized AISI M2 steel at 900 °C for 2 h.

through the powder mixture or borax based melt on to the borided surface [13]. There is a great deal of study about duplex treatment, which involves first conventional surface hardening methods likes carburizing, nitriding, boronizing and then, metallizing like niobizing, vanadizing, chromizing, etc. [13–18].

In the present study, the niobium boride coating was applied on the steel substrate by a duplex treatment. The first step in the process was the boronizing treatment in order to produce iron boride phases on substrate. The second step was a niobizing treatment for producing niobium boride phases on the pre-boronized steel. The main goal of the study was to investigate the structural characterization of niobium boride layer formed.

2. Experimental procedure

The steel used for the boro-niobizing treatment was AISI M2 steel which consisted of 0.95% C, 0.32% Si, 0.18% Mn, 0.032% P, 0.049% S, 4.66% Cr, 4.43%Mo, 0.77% Ni, 1.92% V, 6.8% W and iron (balance). Before treatment, the samples were cut in to the dimensions of 20 mm in diameter and 5 mm in length, and ground up to1200 grid emery paper and 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 1000 °C for 2 h in the first step of the boro-niobizing treatment. 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 900 °C for 1–4 h. 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 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. In the

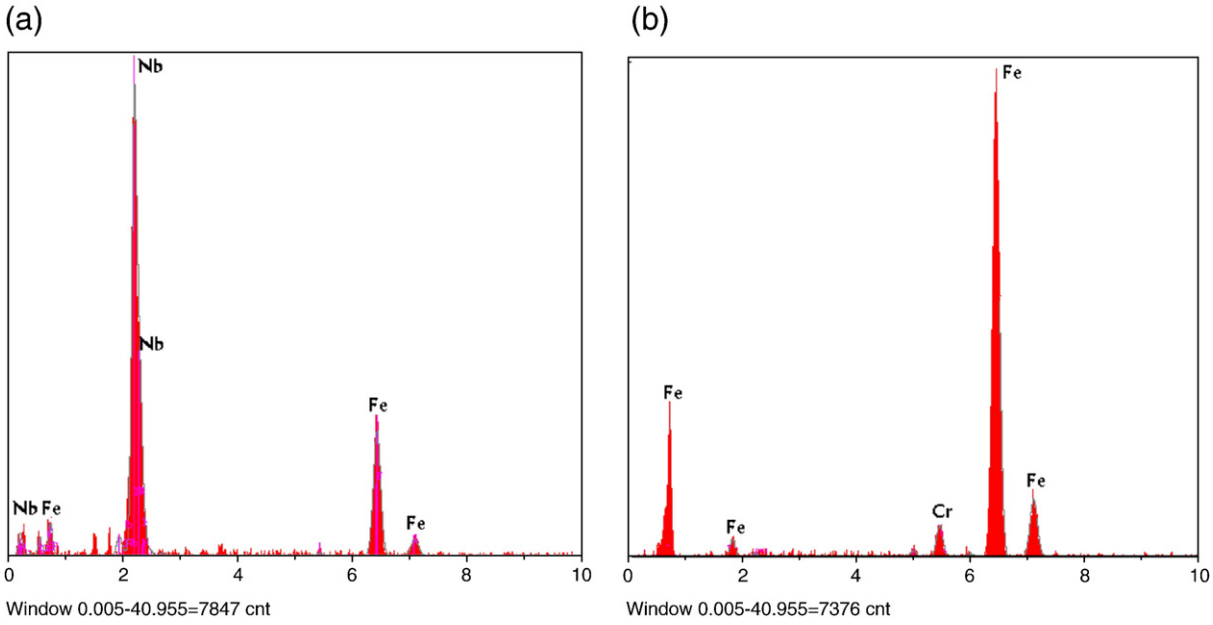


Fig. 3. EDS analysis of (a) niobium boride layer and (b) matrix of boro-niobized AISI M2 steel at 900 °C for 2 h.

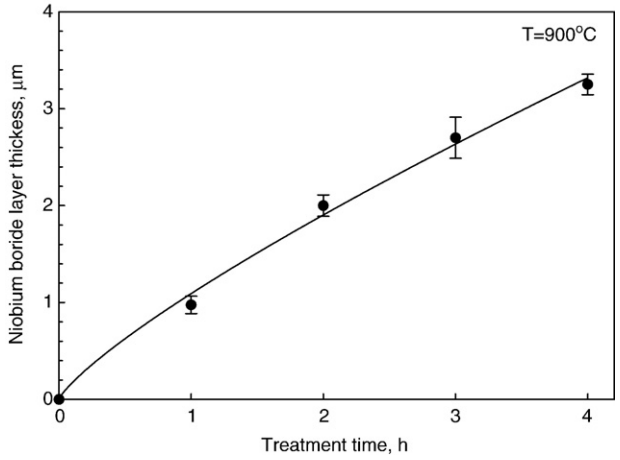


Fig. 4. Niobium boride layer thickness depending on niobizing time.

scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS), the samples were analyzed on the cross-sections. Micro-hardness measurements of the layers from surface were performed 10 times using Future-Tech FM-700 micro-hardness tester under the loads of 10 g. 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 20° to 80°, using CuK_α radiation.

3. Results and discussion

Fig. 1 shows a SEM micrograph of the boro-niobized AISI M2 high alloy steel in the powder mixture consisting of ferro-niobium, ammonium chloride and alumina at 900 °C for 2 h. Coating layer is composed of three distinct regions; these are (i) a niobium boride layer formed on the surface of the coated steel, (ii) an iron boride layer took place under the niobium boride layer and (iii) a matrix. The niobium boride layers formed on the AISI M2 steels were smooth and homogeneous and compact. But the coating layer was thinner than that of the other thermo-reactive deposition coatings, such as niobium carbide, titanium nitride, vanadium boride, chromium nitride [1,14,19]. XRD analysis of the boro-niobized AISI M2 steel sample at 900 °C for 2 h showed that, the phases formed on the coated steel sample are NbB and Nb_3B_2 beside FeB and Fe_2B phases as seen in Fig. 2. This result agrees with earlier studies of Riberio et al. [20] and Usta et al. [21]. EDS analysis showed that niobium concentrated in the coating layers formed on the steel surface. The layer formed on the surface of the coated steel is rich in niobium, which is the component of the boride identified by the XRD analysis. 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 Fig. 3(a) and (b).

The depth of the niobium boride layer ranged from 0.97 µm to 3.25 µm, depending on the treatment time (see Fig. 4). 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 the temperature causes to an increase in the coating layer thickness. Bath composition, substrate, treatment time and temperature all affected the coating layer thickness in the thermo-reactive deposition processes according to Ref. [14]. The hardness of the niobium boride layer was 2738±353 $HV_{0.01}$. This is in good agreement with Riberio et al. [20]. These are due to the presence of hard borides (NbB and Nb_3B_2) in the boride layer as verified by XRD analysis (Fig. 2).

4. Conclusions

AISI M2 steel was the substrate used for the deposition of niobium boride coating by thermo-reactive deposition technique. In the first step, boron concentration on the surface layer of the steel sample was increased by boronizing treatment, in order to form a boride-based coating by this technique. After that, pre-boronized steel samples were niobized by pack method in the powder mixture consisting of ferro-niobium, ammonium chloride and alumina. The results showed that:

- Niobium boride coating can be produced by thermo-reactive deposition technique on the pre-boronized AISI M2 steel sample.
- Coating layers formed on the AISI M2 steels were homogenous, smooth and compact.
- XRD analysis showed that the coating layer include NbB and Nb_3B_2 phases.
- The hardness of the niobium boride layer formed on the pre-boronized steel samples was significantly high (2738± 353 $HV_{0.01}$).
- The treatment was proved to be efficient in the production of boride base coatings.

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