Characterization of Tungsten boride coating realized on AISI D2 steel

Ugur Sen, Eray Abakay, Saduman Sen
Faculty of Engineering, Department of Metallurgical and Materials Engineering, Sakarya University, Turkey

Abstract:

In this study, tungsten boride coating was realized on AISI D2 steel by boro-tungstenizing treatment. Steel samples were boronized at 1000 °C for 2 h in the first step of the coating process, and then tungstenizing treatment realized by thermo-reactive deposition (TRD) method in the powder mixture consisting of tungsten powder, ammonium chloride and alumina at 1100 °C for 1–4 h. Samples were characterized by optical microscopy, X-ray diffraction (XRD) analysis and Vickers micro-hardness tests. Coating layer formed on the steel samples is smooth, compact and well bonded to the steel matrix. The depth of the tungsten boride layer formed on the steel samples was changed between 7.35±0.73µm to 18.45±1.73 µm, depending on treatment time. The average micro-hardness value of the coating layer was about 2094±211 $HV_{0.005}$. The layer consisted of WB, Fe_2B, FeWB borides beside Fe_3W_3N and Fe_6W_6C phases.

Keywords: Coating, boro-tungstenizing, thermo reactive deposition

1. Introduction

The use of surface coatings opens up the possibility for material designs 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, thermal loads and corrosion. Surface treatments offer remarkable choices for a wide range of tribological applications where the control of friction and wear are of primary concern [1]. Multicomponent boriding is a thermochemical treatment involving consecutive diffusion of boron and one or more metallic elements such as aluminum, silicon, chromium, vanadium, tungsten and titanium into the component surface. This process is carried out at 850 to 1050 °C and involves two steps: (i) boriding by conventional method and (ii) diffusing metallic elements through the powder mixture or borax based melt in to the borided surface [1–2]. Multi-component boronizing is a thermo-chemical surface hardening process which involves boronizing and metalizing. By this process, boron and one or more metallic elements diffuse into the metal substrate and form boride phases and/or solid solution metallic boride layers. The boron-based coating can provide high wear resistance, corrosion resistance and high temperature oxidation resistance on metal substrates [3-5]. Multi-component and multiphase thin films are of increasing interest as protective coating system, because of their excellent combination of physical, chemical, and mechanical properties [6-10].

In the present study, boro-tungstenizing treatment was applied on AISI D2 steel substrate by the duplex treatment. The first step in the process was the boronizing treatment in order to produce iron boride phases on the substrate for boron source. The second step was a tungstenizing treatment for the production of tungsten based interstitial compounds on the surface of the pre-boronized steel. The main goal of the study was to investigate the structural

*Corresponding author: Address: Faculty of Engineering, Department of Metallurgical and Materials Engineering, Sakarya University, Turkey. **E-mail address: ugursen@sakarya.edu.tr**

characterization of boro- tungstenized layer formed on the pre-boronized steel by TRD method.

2. Materials and Method

The material used in the boro-tungstenizing treatment was AISI D2 steel which consisted of 1.56% C, 0.27% Si, 11.2% Cr, 0.30% Mn, 0.75% Mo, 0.93% V, 0.137% Si, 0.02% P and 0.001% S 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 to 1200 grid emery paper and cleaned in ultrasonically for 15 min in ethanol. The boronizing treatment was carried out in a slurry salt bath consisting of borax, boric acid and ferro-silicon at 1000°C for 2h in the first step of the boro-tungstenizing treatment which was detailed at ref [11]. In the second step, pre-boronized steel samples were tungstenized by TRD method in the powder mixture consisting of tungsten, ammonium chloride, alumina and naphthalene at 1100°C for 1–4 h. The samples were directly immersed in the powder mixture in an alumina crucible. An alumina lid was used to close to the box and alumina cement was used for sealing the crucible. After the treatment, the samples were cooled in the coating pot and atmospheric conditions for 1 h.

The boro-tungstenized samples were ground from cross-sections and polished up to 0.3 μm alumina paste 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 were utilized using Future-Tech FM-700 micro-hardness tester under the loads of 5 gf. X-ray diffraction (XRD) analyses was realized from the surfaces of the coated sample with 2θ varying from 10° to 80°, using Cu K_{α} radiation.

3. Results and Discussion

Figure. 1 shows the optical micrograph of the boro-tungstenized AISI D2 steel at 1100°C for 2h. The coating layer includes three distinct regions; these are (i) boro-tungstenized layer which formed on the pre-boronized steel samples, (ii) iron boride layer which took place under the boro-tungstenized layer and (iii) matrix. The coating layer formed on the substrates were compact and homogeneous. XRD analysis of the coated samples at 1100°C for 2h showed that the phases formed on the coated steel samples are FeWB, W_2B and Fe_2B phase beside Fe_6W_6C and Fe_3W_3N minor phases, as seen in Figure. 2. Suwattananont et. al explained that the phases formed in the multi component boronizing treatment including tungsten are W_2B, Fe_2B, FeB [3]. In the present study, tungstenizing treatment resulted to formation of FeWB, Fe_6W_6C (minor) and Fe_3W_3N (minor) beside the W_2B and Fe_2B phases. In the coating bath include ammonium chloride and naphthalene which caused to nitrogen and carbon supplier materials and the coating layer can be react with C and N. So, Fe_6W_6C and Fe_3W_3N phases took place on the coated samples as minor phases

Boro-tungstenzing is a duplex treatment which includes boronizing and then TRD processes. The possible reactions took place in boronizing treatment are [1];

$$2B_2O_3+3Si = 4B + 3SiO_2$$
$$2Fe+B = Fe_2B$$
$$Fe_2B+B=2FeB$$

Boride phases formed on the steel sample react with tungsten in the second steps of the treatment. Possible reactions realized during the TRD treatment are; (i) Ammonium chloride decomposed to the ammonia and hydrochloric acid [12];

$$NH_4Cl_{(s)} = NH_{3(g)} + HCl_{(g)}$$

and then the ammonia decompose to nitrogen and hydrogen [13];

$$2NH_{3(g)} = 2N^{+}_{(g)} + 3H_{2(g)}$$

Tungsten reacts with the hydrochloric acid as follows;

$$W_{(s)} + xHCl_{(g)} = WCl_{x(g)} + x/2H_{2(g)}$$

Tungsten chloride produced in the process reacts with iron boride layer. Possible boride phase reactions in the process are as follows;

$$2WCl_{x(g)} + FeB_{(s)} = FeCl_{x(g)} + W_2B_{(s)} + x/2Cl_2$$
$$2WCl_{x(g)} + Fe_2B_{(s)} = W_2B_{(s)} + 2FeCl_{x(g)}$$
$$W_2B + Fe_2B = 2FeWB_{(s)}$$

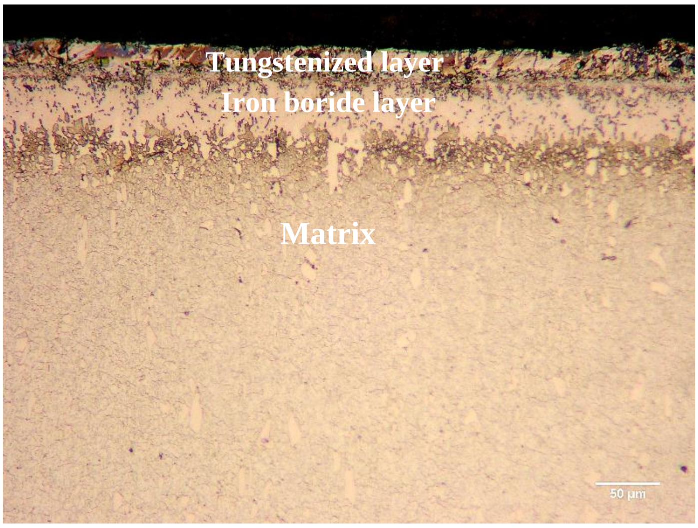

Figure 1. Optical micrograph of boro-tungstenized AISI D2 steel at 1100ºC for 4h.

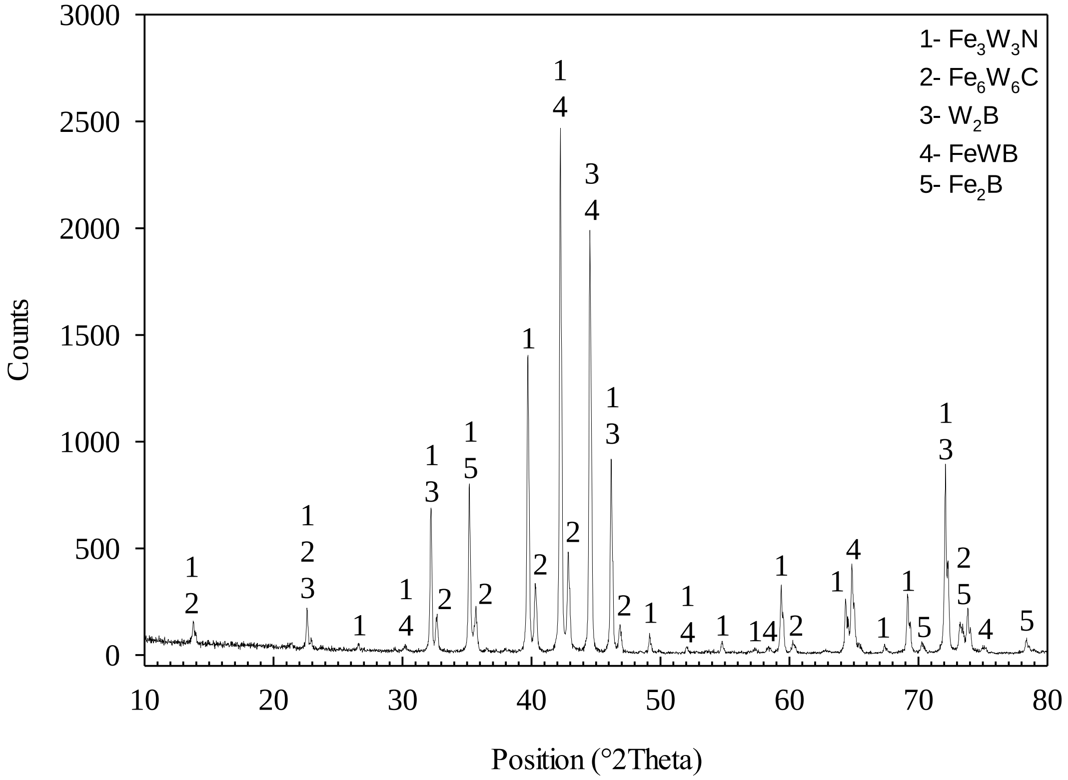

Figure 2. X-ray diffraction patterns of boro- tungstenized AISI D2 steel at 1100ºC for 2h.

The depth of the boro-tungstenized layer ranged from 7.35±0.73 μm to 18.45±1.73 μm, depending on the treatment time (see Figure 3). Increase in the process time caused to increase in the coating layer thickness. In the thermo-chemical coating processes, an increase in the process time and the temperature causes to increase in the coating layer thickness. Bath composition, substrate, treatment time and temperature all affect the coating layer thickness in the thermo reactive deposition processes [14]. The average hardness of the boro-tungstenized layer was 2094±211 $HV_{0.005}$. It is good agree with the study of Khor et al. In the study, the hardness of tungsten borides are changing between 1938 HV and 2500HV [15]. These are consequences for the presence of hard phases (FeWB, W_2B and Fe_2B) in the boro-tungstenized layer as verified by XRD analysis (Figure 2).

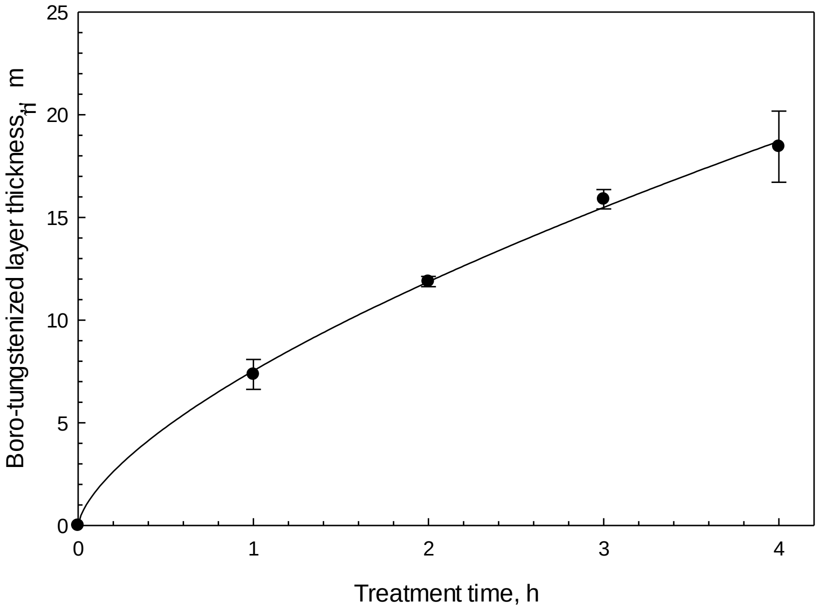

Figure 3. Boro-tungstenized layer thickness depending on process time.

4. Conclusions

The main conclusions of the study should be summarized as follows:

- Based on the results, the boro-tungstenized treatment can be applied on the AISI D2 steel.
- The obtained layer was compact and homogeneous.
- The coating layer includes three distinct regions; these are (i) a boro- tungstenized layer formed on the surface of the steel, (ii) an iron boride layer took place under the boro- tungstenized layer and (iii) a matrix.
- The layer thickness was changed between 7.35±0.73 μm to 18.45±1.73 μm depending on treatment time (1-4h) at 1100°C. Increase in the process time caused to increase in the boro- tungstenized layer thickness.
- The average hardness of the layer was 2094±211 $HV_{0.005}$.
- The phases formed on the coated samples were FeWB, W_2B and Fe_2B phases beside Fe_6W_6C and Fe_3W_3N minor phases.

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