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METALLURGICAL CHARACTERIZATION AND KINETICS OF BORIDED 34CrNiMo6 STEEL

ABSTRACT

Boriding of 34CrNiMo6 steel was performed in a solid medium consisting of Ekabor-II powders at 1123, 1173 and 1223 K for 2, 4 and 6 h. Morphological and kinetic examinations of the boride layers were carried out by optical microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). The thicknesses of the boride layers ranged from 22 ± 2.3 to 145 ± 4.1 depending on boriding temperature and time. The hardness of boride layer was about 1857 HV_{0.1} after boriding for 6 h at 1223 K, while the hardness of the substrate was only around 238 HV_{0.1}. Growth rate constants were found to be between 1.2×10^{-13} – 9.8×10^{-13} m²/s depending on temperature. The activation energy for boron diffusion was estimated as 239.4 ± 8.6 kJ mol⁻¹. This value was comparable to the activation energies reported for medium carbon steels in the literature.

Keywords: *Boriding; 34CrNiMo6 steels; boride layer; Ekabor II; growth rate constants; activation energy*

INTRODUCTION

34CrNiMo6 steel is a typical medium-carbon low-alloy steel widely used in the automobile industry for its high strength, good ductility, and excellent corrosion resistance [1-3]. However, its surface properties are often considered inadequate for certain engineering applications requiring high resistance to wear such as ring gears, valve stems, connecting rods, shackle pins, etc. [4,5]. Surface treatments are therefore required to overcome this disadvantage. Boriding (also known as boronizing) is a thermochemical surface hardening process in which boron atoms are diffused into the surface of a workpiece to form single-or multi-phase boride layers with the base metal [4,6-8]. The boriding temperature and time, as well as the alloying elements in the material to be borided, affect the formation of boride(s) and thickness of the boride layer formed [4,9]. The formation of a boride layer on the surface of the materials can greatly influence mechanical properties [10,11]. Compared to conventional surface hardening processes, it is possible to obtain more than twice the

hardness of the substrate at the surface by the boriding process in iron-based materials [12]. Gunes et al. [13] reported that the surface hardness of Ni 201 significantly increases (1642–1854 HV_{0.05}) due to the formation of hard boride phases, whereas the hardness of substrate material was on average 185 HV_{0.05}.

To control the boriding process, evaluation of kinetic parameters is essential. Many models have been developed over the years to optimize the thicknesses of boride layers obtained in industrial boriding applications [14–16]. Ortiz-Domínguez et al. [16] and Zuno-Silva et al. [17] indicated that boron diffusion was dependent upon the mass balance at the Fe₂B-substrate interface on ASTM A36 and AISI 1045 steels. Another model was developed by Keddad and Kulka [18] to explain the boriding kinetics of AISI D2 steels. This method is an integral method, which takes into account the effect of boride incubation time of the total boride layer (FeB + Fe₂B). Another approach, based on the integral method, was proposed by Zuna-Silva et al. [19]. The common result of these three models is that the growth kinetics of boride layer is governed by a parabolic growth law.

In the literature, studies are available on the boride layer growth kinetics of medium-carbon low-alloy steels such as 34CrAlNi7, AISI 1020-1035, AISI 4130 and AISI 4140 [20–24]. However, at the time of this paper, there are no studies that describe the boriding properties of 34CrNiMo6 steel in detail. This study aims to therefore investigate whether the boriding process applies to 34CrNiMo6 steel, and if so, to examine the diffusion kinetics of and microhardness of borided 34CrNiMo6 steels in the 1123–1223 K temperature range for variable exposure times of 2–6 h.

EXPERIMENTAL METHOD

Boriding and characterization

The chemical composition of the 34CrNiMo6 steel was 0.34 wt% C, 0.40 (max) wt% Si, 0.65 wt% Mn, 1.50 wt% Cr, 0.23 wt% Mo and 1.50 wt% Ni. The samples were cut to dimensions of 10 mm × 10 mm × 50 mm from a 10-mm thick plate. The samples were packed in commercial Ekabor-II boron source and ferro-silicon as an activator. The samples were borided in a muffle furnace at temperatures of 1123, 1173 and 1223 K for durations of 2, 4 and 6 h. After boriding, the samples were removed from the furnace and cooled in air. Cross-sectional samples were then prepared and cold-mounted in epoxy resin, ground with 180, 320, 600 and 1000 grit SiC papers, and polished using 6 µm diamond paste for studying microstructures and microhardness. The polished samples were then dried up and metallographically etched with a 4% Nital solution.

The phases formed on the surface of the borided 34CrNiMo6 steels were determined using a Rigaku D-MAX 2200 X-ray diffractometer with CuKα radiation of 0.15418 nm wavelength. Metallographic cross-sections were prepared and the morphological details and microstructures of the boride layer were examined by optical microscopy. High magnification imaging was carried out using a JEOL 5600LV scanning electron microscope.

Fig. 1. Optical micrographs in the cross-section of the specimen borided at 1223 K for 2 (a), 4 (b) and 6 h (c)

To construct the hardness profiles of the borided 34CrNiMo6 steels, a Vickers microhardness tester with an indentation load of 100 g and dwell time of 10 s was used. Many indentations were made on surfaces of borided steels under each experimental condition to check the reproducibility of hardness data. Boride layer thicknesses were measured by the method provided by Yu et al. [25].

RESULTS AND DISCUSSION

Microstructures of the borided specimens

Optical micrographs of the cross-sections of 34CrNiMo6 steel borided at 1223 K for 2, 4 and 6 h are given in Fig. 1. The boride layers formed on the surface of the borided steel have a sawtooth morphology. As boriding is a thermochemical diffusional process, the thickness of the boride layers and the diffusion zone increase with boriding time.

The average boride layer thicknesses of borided 34CrNiMo6 steels ranged from 22 ± 2.3 to 145 ± 4.1 µm depending on boriding time and temperature (Fig. 2). The obtained values show similarities with results obtained for other medium carbon steels reported in the literature [20,21,26,27]. Corresponding to this, Sen et al. [26] and Altinsoy et al. [27] showed that boride layer thickness of medium carbon steels (AISI 4340 and AISI 1020) changed from 21 to 238 µm and from 20.5 to 216 µm between 1073 and 1273 K for durations of 2, 4, 6 and 8 h, respectively.

Fig. 2. Square of the boride layer thickness of borided 34CrNiMo6 steel as a function of boriding time and temperature

Fig. 3. SEM micrographs of 34CrNiMo6 steel borided at 1123 K (a), 1173K (b) and 1223 K (c) for 6 h

Cross-sectional SEM images of the 34CrNiMo6 steel borided at the temperatures of 1123, 1173 and 1223 K for 6 h are shown in Fig. 3. In this figure, two distinct regions are identified on cross-sections of the borided specimens: (i) the boride layer and (ii) the substrate essentially unaffected by boron diffusion. Characteristic FeB (outer) and Fe₂B phases (inner) in the boride layer are distinguishable. Furthermore, small Fe₂B boride particles are also observed beneath and in between the Fe₂B needles adjacent to the matrix.

X-Ray diffraction analysis

Diffraction patterns of 34CrNiMo6 borided at 1123 K and 1223 K for 6 h are presented in Fig. 4. At 1123 K, XRD patterns reveal that FeB and Fe₂B phases are the dominant phases in the boride layer (Fig. 4a). The diffraction patterns also show peaks belonging to chromium iron carbide (Cr,Fe)₂₃C₆ (Cr_{15.58}Fe_{7.42}C₆). This phase was also identified by Yamazagi et al. [28] and Ohta et al. [29]. When the boriding temperature is increased to 1223 K, Fe₃O₄ (magnetite) is detected in addition to the FeB and Fe₂B phases. This observation confirms that the surface of borided samples have been oxidized because of non protective atmosphere of the boriding medium as indicated in Reference [30].

Fig. 4. XRD patterns of borided 34CrNiMo6 at 1123K (a) and 1223K (b) for 6 h

In the literature, it has been reported that the alloying elements can lead to the formation of mixed borides in the boride layer of alloy steels [4]. Corresponding to this, Azakli et al. [31] noted that Mo₂FeB₂ precipitates were formed in the boride layer and transition zone of borided Fe–Mo binary alloys. In the present study, complex borides were not observed, but Ni-Fe was observed at 1223 K (Fig. 4b).

Hardness tests

Vickers microhardness tests were carried out across the cross-sections of the borided specimens to obtain the hardness profiles (Fig. 5).

Fig. 5. Optical images of the indentation prints on the 34CrNiMo6 steel borided at 1223 K for 6 h

Fig. 6 shows the microhardness profiles of borided 34CrNiMo6 steel as a function of boriding temperature and time. It can be seen from the figure that the boriding process significantly increases the hardness of the boride layer compared to the matrix. There is more than a 7-fold increase in hardness at the surface. This is a consequence of the presence of hard

boride phases (FeB, Fe₂B, and additionally from Ni-Fe due to solid solution hardening) as determined by the XRD analysis in this region. On the other hand, the decrease in the hardness value towards the matrix is explained by the increasing proportion of the matrix surrounding the iron boride needles [23,32,33]. In addition to this, the outer FeB phase in the boride layer was found to be much harder than the inner Fe₂B phase (Fig. 5 and 6) because the bonds in FeB were slightly stronger than those in Fe₂B [34]. The obtained hardness values are very close to the values obtained for medium carbon steels. For similar boriding conditions (1123 K and 4 h), the hardness values of the boride layer obtained are around 1600 HV_{0.1}, 1800 HV_{0.1} and 1700 HV_{0.1} for borided medium carbon steels 32NiCrMo6.4 [4], SAE 1035 [32] and AISI 1040 [35], respectively.

Fig. 6. Microhardness profiles of specimens borided at 1123 and 1223 K for 2 and 6 h

Boriding kinetics

Boriding parameters such as processing temperature and time have to be known to predict the thickness of the boride layer which is formed by the diffusion of boron atoms into the substrate. In the literature [17,21,32,36], the relationship between square of boride layer thickness and boriding time obeys a parabolic growth law and is given as follows;

$$d^2 = Kt \quad (1)$$

where d is the thickness of boride layer (m), t is the boriding time (s), and K is the growth rate constant for the boriding temperature (m²/s). Meanwhile, the relationship between growth rate and the boriding temperature can be described by an Arrhenius equation of the form [7,10,13,14,35]:

$$K = K_0 \exp \left(-\frac{Q}{RT} \right) \quad (2)$$

where K_0 is the collision factor (m²/s), Q is the activation energy for the particular reaction (J/mol), T is the absolute temperature (K) and R is the universal gas constant (J/(mol·K)).

Fig. 7. Growth rate constant of 34CrNiMo6 steel borided with Ekabor II powders as a function of reciprocal of temperature

The effect of the boriding temperature and time on the growth kinetics of the boride layer was also investigated. The growth rate constants were calculated using Eq. 1 for the three boriding temperatures. The change in K with T is plotted in Fig. 2 and also listed in Table 1. The growth rate constant K determines the rate at which borides grow on the surface of the steel, and Table 1 shows that higher process temperatures result in higher K values. When $\ln K$ is plotted with respect to the reciprocal of absolute temperature (Fig. 7), the activation energy Q and collision factor K_0 can be calculated from the slope and y-intercept, respectively (Eq. 2).

The calculated Q and K_0 values are given in the Table 1. A direct comparison of K and Q values for 34CrNiMo6 steel could not be made, since no boriding studies on 34CrNiMo6 steel could be found. Comparisons were therefore made using results from other borided medium carbon steels. However, it can be seen from Table 2 that the Q values of medium carbon steels also depend on the type of boriding process. Table 2 clearly shows that the Q values obtained through plasma paste boriding method were established to be quite low compared to those obtained through traditional boriding methods. The high rate of boriding by the plasma paste method has been explained by the extensive amounts of active boron generated by the plasma, which does not exist in the pack boriding method [37]. Campos et al. [38] have shown that the boron potential can be controlled by modifying the thickness of the paste and by changing water/powder ratio in the paste.

Table 1. Calculated growth rate constants and activation energy for the 34CrNiMo6 steels

Temperature T (K)	Growth rate constant K (m^2/s)	Activation energy Q (kJ/mol)	Collision factor K ₀ (m^2/s)
1123	0.12×10^{-12}	239.4 ± 8.6	1.65×10^{-2}
1173	0.34×10^{-12}		
1223	0.98×10^{-12}		

The activation energy value obtained in our study is 239.4 ± 8.6 kJ/mol which differs slightly from the activation energy values obtained for other medium carbon steels for the

same boriding temperature in Ekabor II. The difference is probably due to the chemical composition of the substrate. Regarding the alloy element concentrations in the four medium carbon steels borided with Ekabor II in Table 2, there are differences in Cr, Ni, Mo and Mn content between each. While C, Mo and W in the alloy dramatically reduce the rate of the boriding process, Si, Cr and Al have moderate influence [38-41]. Similar discussions have been given in Ref. 42. In that study, it has been noted that activation energy values change with the chemical composition of the steel because alloying elements can often act as a barrier for boron diffusion. It has also been shown [4,16,42,43], that the activation energy also depends on the methods used for measuring the thickness of the boride layer, surface preparation, the type and size of the boriding powders, and whether a single-phase (FeB) or double-phase boride layer (FeB + Fe₂B) is formed (due to boron potential).

Table 2. The comparison of the activation energies obtained for medium carbon steels exposed to boriding

Steels	Temperature range (K)	Q (kJ/mol)	Boriding process	Reference
AISI 4340	1073-1223	234	Salt bath	Sen et al. [26]
32NiCrMo6.4	1123-1223	294.3	Ekabor II powders	Gunes [41]
AISI P20	1123-1223	256.4	Ekabor II powders	Kayali [7]
		213.9	Microwave	
34CrAlNi7	1123-1223	270	Ekabor II powders	Efe et al. [20]
34CrAlNi7	1123-1323	169	Ekabor I	Topuz et al. [21]
AISI P20	1073-1223	200	Ekabor II powders	Uslu et.al [8]
AISI 1035	1073-1273	227.5	Salt bath	Kaouka et al. [32]
AISI 1038	1123-1273	207.8	Liquid	Mebarek et. al. [44]
AISI 1045	1193-1273	179	Paste	Campos et.al. [38]
AISI 8620	973-1073	124.7–138.5	Plasma paste	Gunes et.al.[45]
34CrNiMo6	1123-1223	239.4±8.6	Ekabor II powders	Present study

The collision factor K_0 is constant and is a measure of the effective collisions between reacting species. From the calculated values of Q and K_0 , an empirical equation was developed for predicting the boride layer thickness by the classical kinetic diffusion theory for 34CrNiMo6 steel for given the time and temperature.

$$d = 12.84 \times 10^{-2} \sqrt{t \exp\left(-\frac{28794.8}{T}\right)} \quad 1123 \text{ K} < T < 1223 \text{ K}$$

The correlation coefficient between the predicted and experimental boride layer thickness values for borided 34CrNiMo6 steel was found to be 0.912.

CONCLUSIONS

Boriding of 34CrNiMo6 steel was successfully carried out using the pack boriding technique. The following points can be concluded from the present study:

- The dominant phases formed on the surface of 34CrNiMo6 steel after boriding are FeB and Fe₂B.
- The boride layer thickness ranged between 22±2.3 to 145±4.1 μm depending on temperature and time.

- After boriding, a significant improvement in surface hardness was observed. The microhardness of boride layer was about 1857 HV_{0.1} for 6 h at 1223 K, while that of unborided steel substrate was about 238 HV_{0.1}
- The activation energy for boron diffusion value was found to be $239.4 \pm 8.6 \text{ kJ mol}^{-1}$, which is close to the values expected for medium carbon steels.

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