

BORIDE LAYER GROWTH KINETICS OF THE BORIDED HIGH ALLOYED COLD WORK TOOL STEEL

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ABSTRACT

In this study, the case properties and diffusion kinetics of high alloyed cold work tool steel borided in Ekabor-2 powder were investigated by conducting a series of experiments at temperatures of 1123, 1173 and 1223 K for 2, 4 and 6 h. The boride layer was characterised by optical microscopy, X-ray diffraction technique and micro-Vickers hardness tester. X-ray diffraction analysis of boride layers on the surface of the steels revealed the existence of FeB, Fe₂B, CrB, Cr₂B and MoB compounds.

Depending on the chemical composition of substrates and boriding time, the boride layer thickness on the surface of the steel ranged from 13.14 and 120.82 µm. The hardness of the boride compounds formed on the surface of the steels ranged from 1806 to 2342 HV_{0.05}, whereas the Vickers hardness value of the untreated steels was 428 HV_{0.05}. The activation energy (*Q*) of the borided steel was 287.019 kJ/mol.

Keywords: boride layer, microhardness, kinetics, activation energy.

AIMS AND BACKGROUND

Boriding is a thermochemical surface hardening method which can be applied to practically any ferrous material, as well as some nonferrous materials (Mo, Nb, Ti alloys)^{1–5}. The boriding process is applied in order to increase the hardness and the wear resistance of the surfaces of metals and alloys, and in order to retain these properties at high temperatures and increase erosion resistance. Therefore, studies on the properties of the borided layer have focused on the properties of hardness, wear and corrosion^{6–14}. Boriding is mostly applied to iron-based alloys in industry. Nowadays the boriding process can be carried out in solid, liquid, gas and plasma media, between 700 and 1000°C for 1–10 h. The most useful boriding method is box boriding, which is easier to apply and more cost effective than other methods. The method applied during boriding, boron powder, type of borided material and process parameters are the factors which affect the shape of the boride layer and its mechanical properties.

The thickness of the boride layer formed on the surface of the metal by the boriding process is affected by the composition of the boriding medium, degree of purity, treatment temperature and time and metal composition subjected to boriding¹⁵⁻¹⁹. In general, the presence of alloying elements reduces the diffusivity of boron in the steel and consequently decreases the thickness of the borided layer. For example, while carbon, molybdenum and tungsten dramatically reduce the borided layer thickness, silicon, chromium and aluminum have moderate influence, and nickel, manganese and cobalt have only marginal influence²⁰.

In this study cold work tool steel was borided considering these advantages of pack boriding. Characterisation and growth diffusion of the obtained boride layer were calculated. The main objective of this study was to investigate the diffusion kinetics and the effect of process parameters, such as temperature, time and chemical composition, on the boride layers formed on cold work tool steel after powder pack boriding at different processing temperatures and times.

EXPERIMENTAL

Boriding and characterisation. The high alloyed cold work tool steel (Uddeholm Sleipner) essentially contained 0.90 wt.% C, 0.50 wt.% Mn, 7.80 wt.% Cr, 2.50 wt.% Mo and 0.50 wt.% V. The test specimens were cut into Ø 25×10 mm dimensions, ground up to 1000 G and polished using diamond solution. The boriding heat treatment was carried out in solid medium containing an Ekabor-2 powder mixture placed in an electrical resistance furnace operated at temperatures of 1123, 1173 and 1223 K for 2, 4 and 6 h under atmospheric pressure. The microstructures of polished and etched cross-sections of the specimens were observed under a Nikon MA100 optical microscope. The thickness of borides was measured by means of a digital thickness measuring instrument attached to an optical microscope (Nikon MA100). Thickness values given in the results section are averages of at least 15 measurements. The contour diagrams, showing the variation in boride layer thickness with respect to the boriding temperature and time, were plotted using the Sigma plot 12.5 program. The presence of borides formed in the coating layer was confirmed by means of X-ray diffraction equipment (Shimadzu XRD 6000) using Cu K α radiation. The hardness measurements of the boride layer on each steel and unborided steel substrate were made on the cross-sections using a Shimadzu HMV-2 Vickers indenter with a 50 g load.

Kinetics. On the condition that boron diffuses and grows parabolically, the alteration of boride layer thickness with time can be described by the following equation:

$$x^2 = D t \quad (1)$$

where x is the depth of the boride layer (mm); t – the boriding time (s); D – the growth rate constant that depends on the diffusion element (in this case boron) and the diffusion coefficient. It is a well-known fact that the main factor limiting the growth of a layer is the diffusion of boron into the substrate. It is possible to argue that the relationship

between growth rate constant, D , activation energy, Q , and the temperature in Kelvin, T , can be expressed as an Arrhenius equation (equation (2)):

$$D = D_0 \exp(-Q/RT) \quad (2)$$

where D_0 is a pre-exponential constant; Q – the activation energy (J/mol); T – the absolute temperature (K), and R – the gas constant (J/mol K). The activation energy for the boron diffusion in the boride layer is determined by the slope obtained in the plot of $\ln D$ versus $1/T$, using equation (2). The formation rate of the boride layers was examined and kinetic equations and parameters were determined at a temperature range of 1123–1223 K for periods of 2–6 h.

RESULTS AND DISCUSSION

Characterisation of boride coatings. The cross-sections of the optical micrographs of the borided cold work tool steel at the temperature of 1123 and 1223 K for 2 and 6 h are shown in Fig. 1. As can be seen, the borides formed on the cold work tool steel substrate have a smooth morphology due to higher alloy content. It was found that the coating/matrix interface and matrix could be significantly distinguished and the boride layer had a columnar structure. Depending on the chemical composition of substrates and boriding time, the boride layer thickness on the surface of the steel ranged from 13.14 to 120.82 μm (Fig. 2).

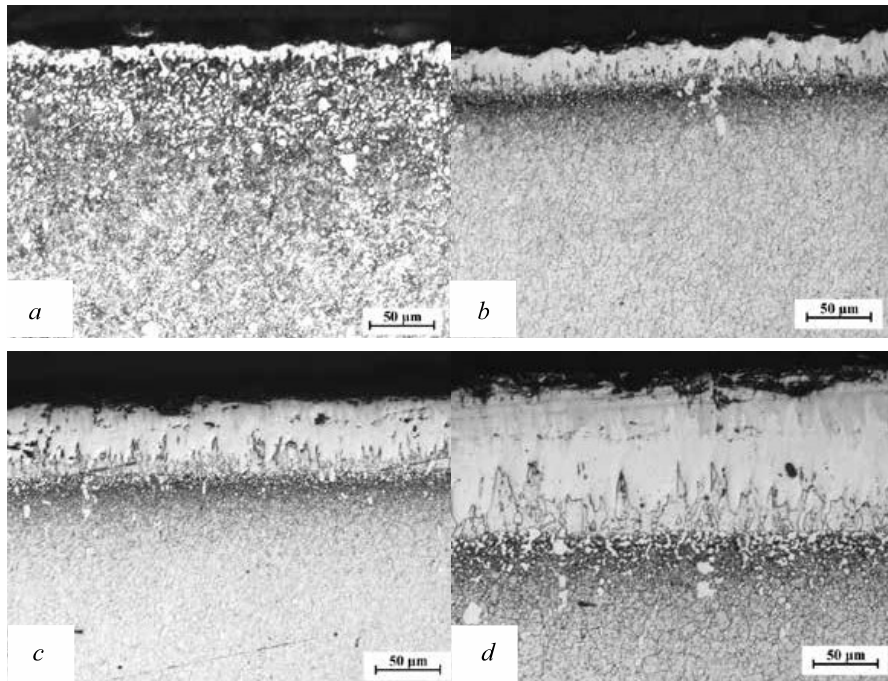


Fig. 1. Cross-sections of the borided cold work tool steel: 1123 K – 2 h (a); 1123 K – 6 h (b); 1223 K – 2 h (c), and 1223 K – 6 h (d)

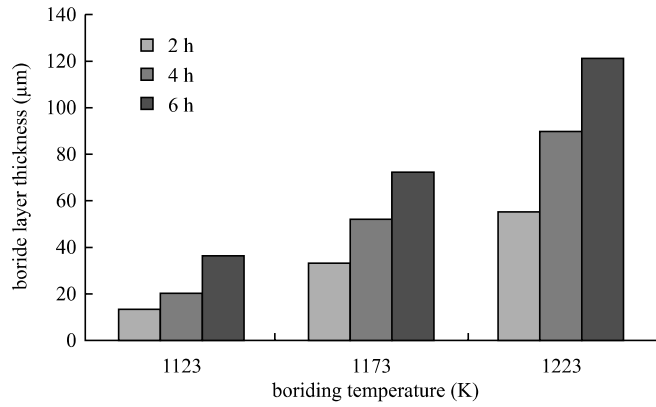


Fig. 2. Thickness values of boride layers with respect to boriding time and temperature

The amounts of C, Cr, Ni and Mo appear to be lower than that of iron in the boride layer because of less solubility. Thus, the deficiency of C, Cr, Mn and Mo results in a negative effect on the boride layer in terms of both thickness and morphology^{21–24}. Efe et al. borided 31CrMoV₉ and 34CrAlNi₇ steels with Ekabor-2 boron powder at 1123–1223 K for 2–8 h and reported that some alloying elements (Cr, Ni) had a negative impact on the diffusion of boron atoms into the steel surface²⁵. Gunes¹³ borided gear steels with Ekabor-2 boron powder at 1123–1223 K for 2–6 h and obtained a 15–260 µm boron layer and alloying elements had an effect on both the morphology and the thickness of the boron layer.

In this study, the presence of borides was identified using XRD analysis (Fig. 3*a–d*). XRD patterns show that the boride layer consists of borides such as SB and S₂B (S = metal: Fe, Mo, Cr).

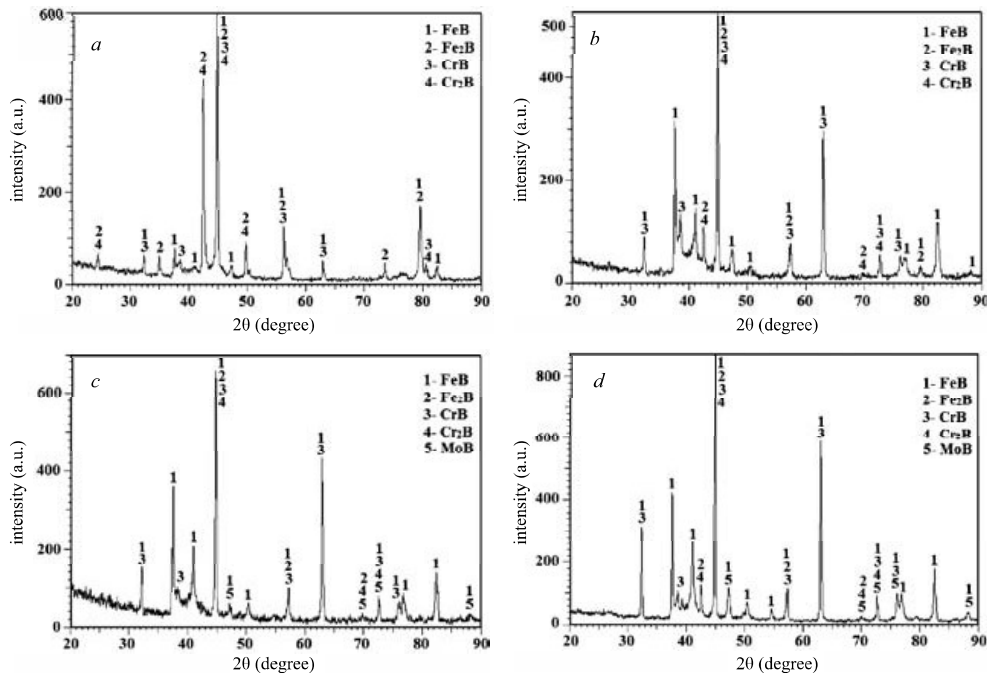


Fig. 3. X-ray diffraction patterns of borided cold work tool steel: 1123 K – 2 h (a); 1123 K – 6 h (b); 1223 K – 2 h (c); 1223 K – 6 h (d)

XRD results showed that boride layers formed on the cold work tool steel contained the FeB, Fe₂B, CrB, Cr₂B and MoB phases, respectively in Figs 3a–d. With increasing time and temperature, the Fe₂B phase content decreases and the FeB and CrB phases content increases for the steel. The boride layers mainly consist of intermetallic phases (FeB, Fe₂B, CrB and Cr₂B) as a result of diffusion of boron atoms from the boriding compound to the metallic lattice with respect to the holding time.

Microhardness measurements were carried out from the surface to the interior along a line in order to see the variations in the boride layer hardness, transition zone and matrix, respectively. The microhardness of the boride layers was measured at 10 different locations at the same distance from the surface and the average value was taken as the hardness.

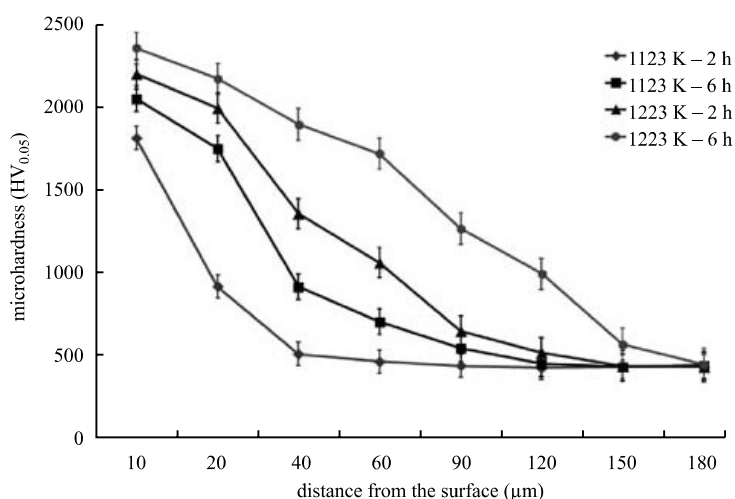


Fig. 4. Variation of hardness of borides formed on the surface of cold work tool steel from surface into interior

Microhardness measurements were carried out on the cross-sections from the surface to the interior along a line (Fig. 4). The hardness of the boride compounds formed on the surface of the steels ranged from 1806 to 2342 HV_{0.05}, whereas the Vickers hardness values of the untreated steels was 428 HV_{0.05}. When the hardness of the boride layer is compared with the matrix, boride layer hardness is approximately four times greater than that of the matrix.

Contour diagram showing boride layer variations due to boriding temperature and time is given in Fig. 5. Contour diagrams can be used for two purposes: (1) to predict the coating layer thickness with respect to the process parameters, namely time and temperature; (2) to determine the value of process time and temperature for obtaining a predetermined coating layer thickness^{18,26}. The boride layer increased with the increase in boriding time and temperature in cold work tool steel.

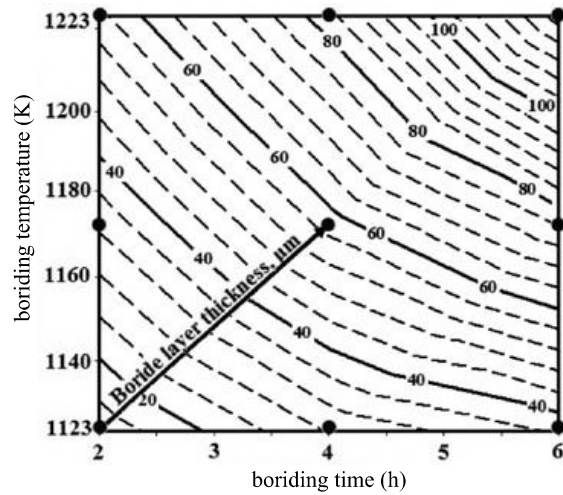


Fig. 5. Contour diagram and thickness values of boride layers with respect to holding time and test temperature

Kinetics. In this study, the effects of the processing temperature and boriding time on the growth kinetics of the boriding layer were also investigated. Kinetic parameters such as processing temperature and time must be known for the control of the boriding treatment and the growth rate constants were calculated using equation (1) (Fig. 6).

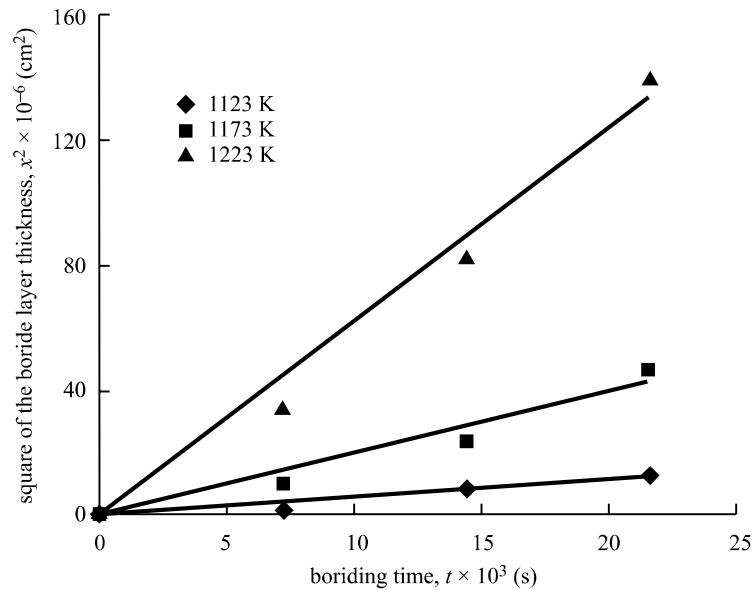


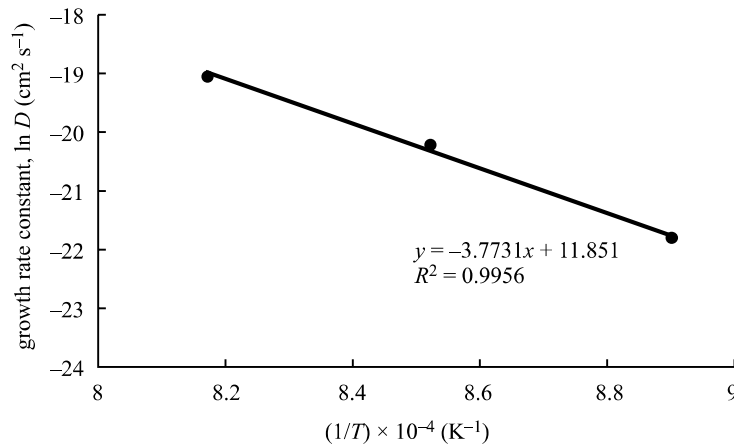
Fig. 6. Square of the boride layer thickness versus holding time in cold work tool steel

As a result, the effective growth rate constants with respect to boriding temperature are 1.38×10^{-10} , 4.27×10^{-10} , and 13.56×10^{-10} cm² s⁻¹ for the cold work tool steel (Table 1). The plot of $\ln D$ versus $1/T$ exhibits a linear relationship (Fig. 7), and activation energy of 287.019 kJ/mol for the cold work tool steel was obtained from the slope of the straight lines.

Table 1. Growth rate constant (D) and activation energy (Q) as a function of boriding temperature and steel

Steels	Growth rate constant ($\text{cm}^2 \text{s}^{-1}$)			Activation energy (kJ/mol)
	temperature (K)			
	1123	1173	1223	
Cold work tool steel	1.38×10^{-10}	4.27×10^{-10}	13.56×10^{-10}	287.019

Activation energy values obtained in this study vary in accordance with the chemical composition of the steel. In steel with low carbon and alloying elements, activation energy decreases with the boride layer thickness increases at the boriding treatment. The values calculated in this study are compatible with the values reported in the literature as seen in Table 2 (Refs 9, 27–29). The present study gives the highest activation energy. It can be considered that carbon and alloying elements acted as a diffusion barrier, inhibiting the diffusion of active boron.

**Fig. 7.** Growth rate constant versus the processing temperature in cold work tool steel**Table 2.** Comparison of activation energy for diffusion of boron with respect to the different boriding media and steel

Steel	Temperature range (K)	Boriding medium	Activation energy (kJ/mol)	References
AISI 1040, P20	1073–1223	pack	168, 200	9
AISI 304, H13	1073–1223	salt bath	253, 244	27
AISI 52100	1123–1223	pack	269	28
AISI 8620	973–1073	plasma paste	100	29
High alloyed cold work tool steel	1123–1223	pack	287.019	present study

CONCLUSIONS

The following conclusions may be derived from the present study:

Boride types formed on the surface of the cold work tool steel have columnar structure.

Depending on the process time, temperature and chemical composition of substrates the depth of the boride layer ranged from 13.14 to 120.82 μm .

The multiphase boride coatings that were thermochemically grown on the cold work tool steel were constituted by the FeB, Fe₂B, CrB, Cr₂B and MoB phases.

The surface hardness of the borided cold work tool steel was in the range of 1806 to 2342 HV_{0.05}, while for the untreated steel substrate it was 428 HV_{0.05}.

Activation energy of 287.019 kJ/mol for cold work tool was determined.

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**BORIDE LAYER
ALLOYED COATINGS**

By: Gunes, I (Gurhan)

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[View Journal Info](#)**Abstract**

In this study, the microhardness of the boride layer on the surface of the Ekabor-2 powder metallurgy (PM) 1173 and 1223 K1 steels was investigated using X-ray diffraction technique. The results showed that the surface of the steels was covered with a boride layer.

Depending on the boron content, the boride layer on the surface of the compounds formed. The microhardness value of the boride layer was 287.019 kJ/mol.

Keywords**Author Keywords:** boride layer; microhardness; kinetics; activation energy**KeyWords Plus:** AISI 8620 STEEL; WEAR BEHAVIOR; GEAR STEELS; MECHANICAL-PROPERTIES; DIFFUSION KINETICS; PASTE MIXTURES; SURFACE; MOLYBDENUM; CHROMIUM; SPEED**Author Information****Reprint Address:** Gunes, I (reprint author)

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BORIDE LAYER GROWTH KINETICS OF THE BORIDED HIGH ALLOYED COLD WORK TOOL STEEL

By: Gunes, I (Gunes, I.)

OXIDATION COMMUNICATIONS

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[View Journal Impact](#)**Abstract**

In this study, the case properties and diffusion kinetics of high alloyed cold work tool steel bonded in Ekabor-2 powder were investigated by conducting a series of experiments at temperatures of 1123, 1173 and 1223 K for 2,4 and 6 h. The boride layer was characterised by optical microscopy, X-ray diffraction technique and micro-Vickers hardness tester. X-ray diffraction analysis of boride layers on the surface of the steels revealed the existence of FeB, Fe₂B, CrB, Cr₂B and MoB compounds.

Depending on the chemical composition of substrates and bonding time, the boride layer thickness on the surface of the steel ranged from 13.14 and 120.82 μm . The hardness of the boride compounds formed on the surface of the steels ranged from 1806 to 2342 HV0.05, whereas the Vickers hardness value of the untreated steels was 428 HV0.05. The activation energy (Q) of the borided steel was 287.019 kJ/mol.

Keywords**Author Keywords:** boride layer; microhardness; kinetics; activation energy**KeyWords Plus:** AISI 8620 STEEL; WEAR BEHAVIOR; GEAR STEELS; MECHANICAL-PROPERTIES; DIFFUSION KINETICS; PASTE MIXTURES; SURFACE; MOLYBDENUM; CHROMIUM; SPEED**Author Information****Reprint Address:** Gunes, I (reprint author)

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