

Boronizing Kinetics of 30CrNiMo8 Steel

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Abstract: Boronizing is a thermochemical treatment performed to obtain hard surface layers that improve wear resistance. In this study, the boronizing kinetics of pack boronized 30CrNiMo8 steel is investigated. Boronizing was performed at 830, 880 and 930 °C for 4, 6 and 8 hours in DURBORID® G powder mixture. The boride layers obtained characteristic but less pronounced sawtooth morphology, which is typical for the boronizing of low-alloy steels. The average layer thicknesses were between 32,9 and 122 µm, depending on the boronizing temperature and duration, with the temperature having a greater influence. The kinetics of boride layer growth was investigated using the Arrhenius equation, and the results confirm that the boronizing process follows the parabolic law, i.e. that the kinetics of boride layer growth is governed by the diffusion of boron atoms. The activation energy is estimated to be 173,45 kJ/mol and the frequency factor to be $1,54 \times 10^{-5}$ m²/s. Based on these results, a mathematical model was established to describe the relationship between the boride layer thickness, temperature and duration of boronizing of 30CrNiMo8 steel. This model is suitable for practical application as it allows a quick and easy estimation of the boride layer thickness based on the boronizing parameters.

Keywords: activation energy; Arrhenius equation; boronizing; growth kinetics; steel

1 INTRODUCTION

Many tools and machine components are exposed to considerable friction and wear during their work, often in high-temperature and corrosive environments. Under these conditions, surface properties are critical to ensuring durability and reliability. The growing demand for improved wear and corrosion resistance has led to the development of various surface treatments, such as carburizing, nitriding, carbonitriding and boronizing (or boriding). Among the processes mentioned, boronizing stands out due to its ability to achieve exceptional surface properties, especially in terms of hardness at room and elevated temperatures [1-3].

Boronizing is a thermochemical treatment in which boron atoms diffuse from a boron-containing medium into the surface of the treated part and combine with atoms of the base material to form boron compounds (borides). The resulting layers are extremely hard, which significantly improves abrasion resistance both at room temperature and at higher temperatures. In addition, boride layers improve resistance to adhesive wear, corrosion resistance in dilute acids and alkaline media, oxidation resistance and resistance to the influence of liquid metals [4-6].

Boronizing can be carried out in solid (paste and powder), liquid, gaseous, plasma and plasma paste media [3, 6, 7]. Despite the disadvantages such as high temperatures, long duration and difficult control of process variables [3, 5, 8], pack boronizing is the most commonly used due to its simplicity and cost efficiency [4, 6, 7]. In addition, the boronizing agent can be reused, no special equipment is required (if the metal container is properly sealed, pack boronizing can be carried out in the furnace without a protective atmosphere) and it is possible to change the chemical composition of the powders as required [2, 9, 10].

Boronizing can be carried out on a wide range of ferrous and non-ferrous materials (steels, castings, nickel-based alloys, cobalt-based alloys, molybdenum and titanium) [11, 12]. Most steels, with the exception of steels with a high aluminium or silicon content, are suitable for boronizing [13]. Boronizing of steels is usually carried out at temperatures of 800 - 1050 °C and for a duration of

0,5 - 10 hours [7, 14], whereby single-phase (Fe₂B) or two-phase (FeB/Fe₂B) layers can form on the surface. Whether a single-phase or two-phase layer form depends on the treatment method and on parameters such as temperature, duration, chemical composition of the base material and boron potential [1]. Although the FeB layer is harder than the Fe₂B layer (1600 - 2100 HV and 1400 - 1600 HV respectively [10]), its formation is undesirable due to its brittleness. In addition, FeB and Fe₂B have different coefficients of thermal expansion, which leads to the formation and propagation of cracks at the FeB/Fe₂B interface, causing the FeB to fall off in service. Boride layers also differ from other surface layers in their appearance: they exhibit a more or less pronounced sawtooth morphology. In terms of quality, a pronounced serration is preferred as it ensures good adhesion of the layer to the substrate. For all these reasons, single-phase Fe₂B layers with a pronounced sawtooth morphology are preferred in most industrial applications [1, 6, 7, 15].

The thickness of the boride layer depends on both the process parameters (temperature, duration, boronizing agent) and the chemical composition of the base material. The thickness of the resulting layers increases with increasing temperature and duration of the boriding process. Regarding the chemical composition, the higher the content of carbon and/or alloying elements, the thinner the boride layers formed. The chemical composition of the base material not only influences the thickness of the layer, but also its morphology. The higher the content of carbon and/or alloying elements, the thinner and less serrated the boride layers [1, 3, 14, 16].

The dependence between the layer thickness and the boronizing parameters has been the subject of numerous studies. Over the years, several approaches for the kinetic study have been proposed in the literature, such as the integral diffusion model [17], the dimensional kinetics model [18], the fuzzy neural network-based approach [16] and the stochastic approach [19]. Although many authors have used different approaches to study boronizing kinetics, most studies are based on the Arrhenius equation and the determination of the activation energy for different materials. When investigating the boronizing kinetics of AISI H13 steel, Karakaş et al. established empirical

equations to predict the boride layer thickness [14]. Considering the amount of matter involved, Ruiz-Trabolsi et al. estimated the activation energy to be 148,3 kJ/mol for pack boronizing of AISI 1018 steel. They also reported that the layer thickness increases as a function of both the boronizing parameters and the sample size [20]. Based on Fick's second law, Delai et al. established a diffusion model to predict the thicknesses of FeB and Fe₂B layers on 4Cr5MoSiV1 steel [21]. Using the Arrhenius equation, Žanetić et al. determined the values of frequency factor and activation energy for the boronizing of X90CrMoV-18 martensitic stainless steel [10]. Orihel et al. investigated the boronizing kinetics of Sleipner steel in Durboride powder and determined activation energies in FeB and Fe₂B of 193,53 kJ mol⁻¹ and 179,70 kJ/mol, respectively [22]. Using the same approach based on the Arrhenius equation, Duan et al. calculated the activation energy for the boronizing of a Ti6Al4V alloy to be 231,177 kJ/mol [23].

The understanding of boronizing kinetics is crucial for the practical application of boronizing, as it allows the optimization of the process by choosing the boronizing parameters to obtain boride layers with the desired thickness. In this study, the boronizing kinetics of 30CrNiMo8 steel is investigated using the Arrhenius equation. The 30CrNiMo8 is a low-alloy structural steel commonly used in the automotive and engineering industries for highly stressed components such as shafts, crankshafts, connecting rods, rollers, gears, pinions and other transmission elements [24, 25].

2 MATERIALS AND METHODS

2.1 Experimental

The steel 30CrNiMo8 with a chemical composition (wt. %) of C: 0.32; Mn: 0.53; Si: 0.233; P: 0.014; S: 0.022; Cr: 2.1; Ni: 2.01; Mo: 0.377; was used as substrate material. A 3²-factorial experimental design with 3 repetitions each was selected for the experiment, so that 27 specimens with nominal dimensions of $\varnothing 16 \times 7$ mm were prepared. Before boronizing, the samples were sanded with emery paper up to 600 grit.

After preparation, the samples were packed into the steel containers together with the Hef-DurferritDURBORID® G powder mixture (for boronizing temperatures of 850 - 1000 °C) and sealed with fireclay. Boronizing was carried out at 830, 880 and 930 °C for 4, 6 and 8 h in the Nabertherm B180 laboratory furnace without protective atmosphere. After boronizing, the samples were cut in cross-section and mounted in cold mounting acrylic resin. For the metallographic examinations, the samples were prepared by grinding (with emery paper up to 1500 grit), polished with alumina and etched with 3 % Nital.

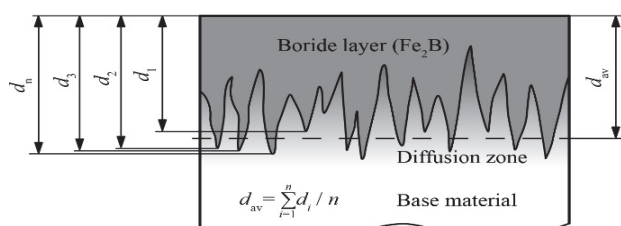


Figure 1 Determination of the boride layer thickness

The microstructure was examined using a Leica DM 2500 M light microscope. For each experimental setup, the boride layer thickness was determined as the mean value of the distances from the surface to the tips of the boride teeth (as shown in Fig. 1), measured with the Leica Q550 MW Imaging Solution.

2.2 Kinetic Study

In this study, an approach based on the Arrhenius equation was used to investigate boronizing kinetics. Boronizing is a thermal diffusion process in which the kinetics of boride layer growth is controlled by the diffusion of boron atoms into the surface [20, 26, 27]. Given the diffusion nature of boronizing, it can be assumed to obey a parabolic law described as follows:

$$d^2 = D \cdot t \quad (1)$$

where d is the thickness of the diffusion layer (i.e. the boride layer) (m), D is the growth rate constant (m²/s) and t is the treatment duration (s). Eq. (1) describes the dependence of the thickness on the duration of the process. It can also be represented in another form by rooting it:

$$d = \sqrt{D \cdot t} \quad (2)$$

Eq. (2) shows a linear relationship between the layer thickness and the square root of the duration, and the proportionality constant is the square root of the growth rate constant. The growth rate constant D depends on the temperature, and this relationship is expressed by the Arrhenius equation as follows:

$$D = D_0 \cdot \exp\left(-\frac{Q}{RT}\right) \quad (3)$$

where D_0 is the frequency factor (m²/s), Q is the activation energy (kJ/mol), T is the diffusion temperature (K), and R is the universal gas constant (kJ/(mol·K)). The frequency factor (pre-exponential constant) reflects the rate of collisions between the molecules of the reacting species. The activation energy is the minimum energy required to overcome the energy barrier for a reaction to occur. The Arrhenius Eq. (3) can be represented in a different form by taking the natural logarithm of the equation.

$$\ln D = \ln D_0 - \frac{Q}{R} \cdot \frac{1}{T} \quad (4)$$

Eq. (4) shows the linear relationship between the natural logarithm of the growth rate constant and the reciprocal of the diffusion temperature, where the proportionality constant is the quotient (Q/R) and $\ln D_0$ is the intersection of the straight line with the ordinate.

By combining Eq. (1) and Eq. (3), Eq. (5) is obtained as follows:

$$d = \sqrt{D_0 \cdot t \cdot \exp\left(-\frac{Q}{RT}\right)} \quad (5)$$

Eq. (5) can be used to calculate the coating thickness as a function of temperature and duration of diffusion (in this case boronizing).

3 RESULTS AND DISCUSSION

3.1 Microstructure

Fig. 2 shows optical micrographs of the boronized 30CrNiMo8 steel. Three different zones can be seen on the cross-sections of the samples: the surface layer (the

outermost zone), the diffusion zone (under the layer) and the substrate (zone not affected by boron diffusion).

As can be seen in Fig. 2, the layers obtained have a typical but less pronounced sawtooth morphology. In the literature, the sawtooth morphology is attributed to the preferential growth of iron borides along the crystallographic direction [001], where boron atoms diffuse faster and produce a needle-like morphology [11, 19, 20, 26, 28-30].

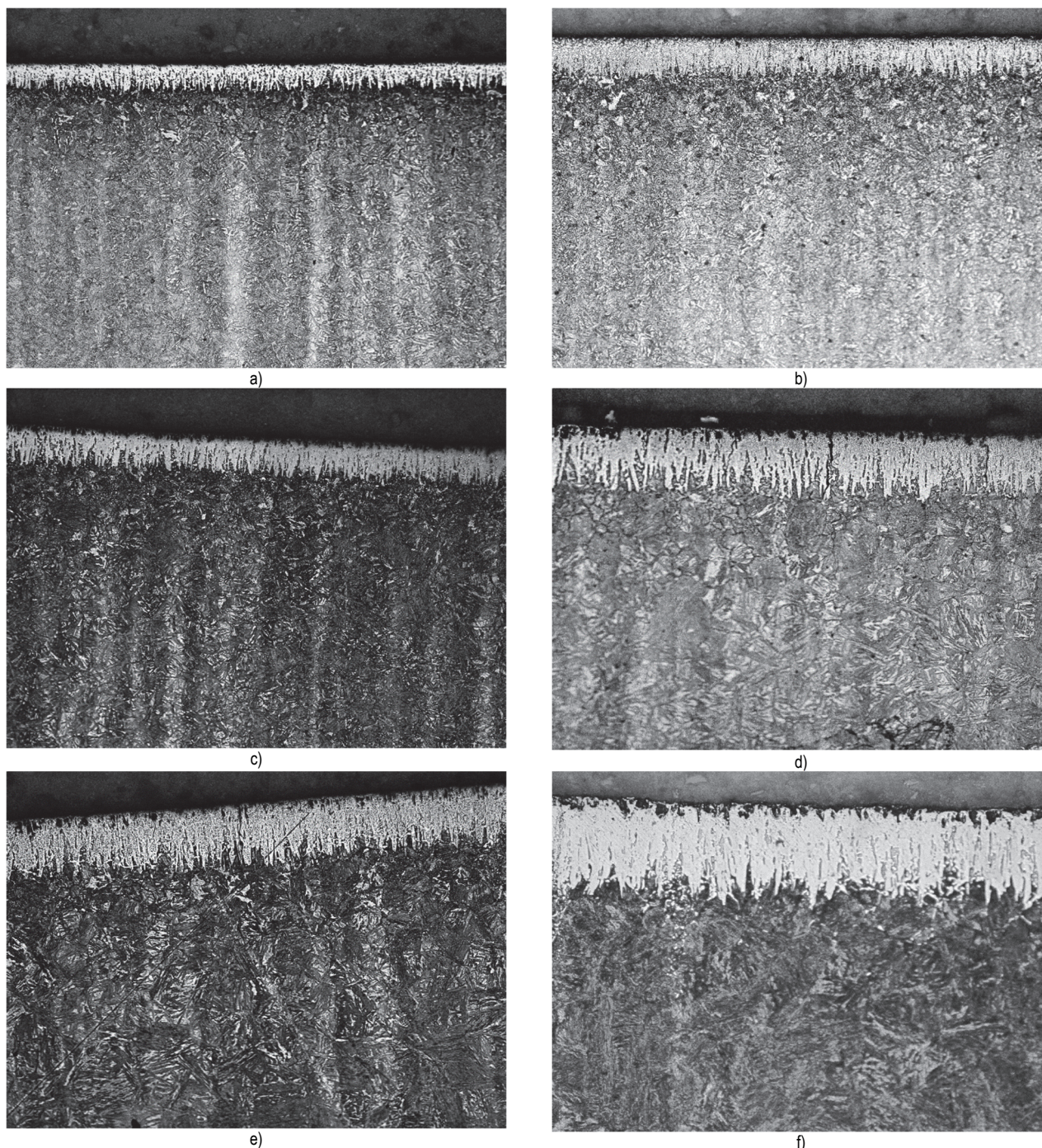


Figure 2 Microstructure of boride layers on 30CrNiMo8 steel, 200:1 magnification, obtained at: (a) 830 °C, 4 h; (b) 830 °C, 8 h; (c) 880 °C, 4 h; (d) 880 °C, 8 h; (e) 930 °C, 4 h; (f) 930 °C, 8 h

Compared to the layers on low-carbon steels reported in [1, 7, 18-20], the boride layers on 30CrNiMo8 steel are less serrated. The medium carbon content and the presence of alloying elements in 30CrNiMo8 steel are reflected in

the morphology. In order to investigate the influence of the chemical composition of the base material on the microstructure of boronized steels, Drajewicz et al. carried out boronizing on 13 steel grades. They reported that the

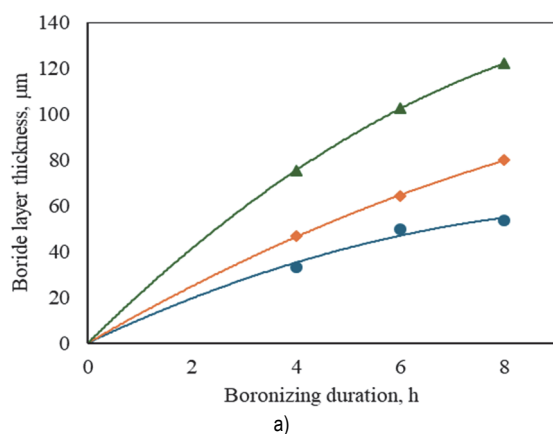
morphology of the boride layer changes with increasing carbon and alloying element content, from thin and sharp boride needles to thicker and blunted needles to a smooth interface between the boride layer and the base material [31]. Many studies also report that the morphology of the boride layers becomes smoother with increasing carbon and/or alloying element content. [13, 14, 17, 22, 26, 29, 31, 32].

The boride layer thicknesses are calculated as the mean value of at least 45 measurements. The results are listed in Tab. 1.

Table 1 Thickness of the boride layers on steel 30CrNiMo8, μm

	4 h	6 h	8 h
830 °C	32,9	49,9	53,6
880 °C	46,6	64,3	79,8
930 °C	75,3	102,6	122,0

The results presented in Tab. 1, together with the micrographs in Fig. 2, show that the boride layer thickness increases with increasing boronizing temperature and duration. This agrees well with the results of other studies carried out on different materials [12, 20, 26, 32]. It can also be observed that increasing the temperature has a greater effect on the thickness than increasing the duration. Using a statistical approach, VillaVelázquez-Mendoza et al. [33] investigated the effects of temperature, duration and substrate roughness on boride layer thickness. Their results showed that temperature had the greatest effect (66.96%), followed by duration (15.48%) and substrate roughness (7%). This greater effect of temperature compared to duration is also confirmed by Delai et al. [21] and Morales-Robles et al. [33].



Comparing the thicknesses from this study with those from other studies, it can be seen that the boride layers formed on 30CrNiMo8 steel are thinner than those on unalloyed and low-carbon steels [4, 10, 18, 21], but thicker than those on high-alloy steels [1, 7, 34]. Several studies have reported that not only the morphology but also the thickness of the boride layer is influenced by the chemical composition of the base material. In particular, the boride layer thickness tends to decrease when the content of carbon and/or alloying elements in the steel increases. During the growth of the boride layer, carbon and alloying elements (which are insoluble in iron borides) are pushed towards the core. Their increased concentration in the diffusion zone creates a barrier that suppresses further growth of the boride layer. Consequently, the boriding of steels with a higher content of carbon and alloying elements leads to thinner and smoother boride layers [10, 16, 29, 31].

A higher proportion of carbides below the layer, which appears as a darkly etched area, also results from an increased carbon content in the diffusion zone compared to the core. Several studies [3, 5] have also reported that the formation of additional carbides is due to the depletion of carbon by the growth of the boride layer and its accumulation in the diffusion zone. This increased carbide content increases the hardness of the substrate in the diffusion zone [10].

3.2 Boronizing Kinetics

The results shown in Tab. 1 are graphically illustrated in Fig. 3.

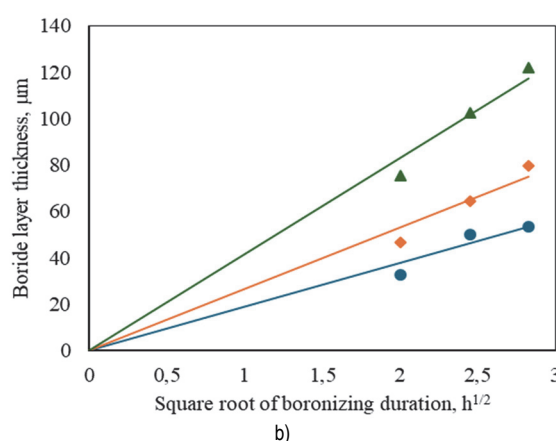


Figure 3 Boride layer thicknesses as a function of: (a) the boronizing duration; (b) the square root of the boronizing duration

The graphs in Fig. 3a confirm a parabolic relationship between the boride layer thickness and the boronizing duration, as described in Eq. (1). Fig. 3b shows the relationship between the boride layer thickness and the square root of the boronizing duration, showing a linear trend in agreement with Eq. (2). These results confirm that the boronizing process follows the parabolic law Eq. (1) and its modified version Eq. (2), suggesting that the kinetics growth of the boride layer is governed by the diffusion of boron atoms. This diffusion-controlled nature of boronizing has been confirmed in several studies [4-8, 12-15, 20-23].

Given this confirmation of the diffusion-controlled nature of boronizing, the growth rate constants were derived from the slopes of the linear plots in Fig. 3b). The estimated values for the growth rate constants at temperatures of 830, 880 and 930 °C are $9,9 \times 10^{-14}$, $1,95 \times 10^{-13}$ and $4,79 \times 10^{-13} \text{ m}^2/\text{s}$, respectively. The values of the growth rate constants are higher at higher temperatures. This increase indicates that diffusion is faster at higher temperatures, which leads to the formation of thicker boride layers within the same period of time.

The diagram in Fig. 4 was created using the estimated values of the growth rate constant and the modified Arrhenius Eq. (4). The diagram shows a linear relationship

between the natural logarithm of the growth rate constant and the reciprocal of the diffusion temperature. The activation energy is determined from the slope of the straight line, while the frequency factor is determined from the intersection of the extrapolated line with the ordinate.

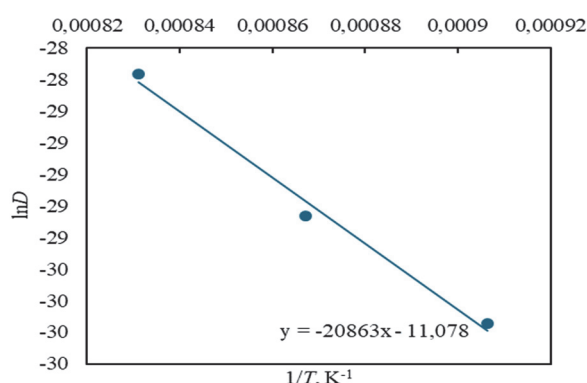


Figure 4 Natural logarithm of growth rate constant as a function of the reciprocal boronizing temperature

The activation energy for the boronizing of 30CrNiMo8 is estimated to be 173,45 kJ/mol and the frequency factor to be $1,54 \times 10^{-5}$ m²/s. When investigating the boronizing kinetics of AISI 4340 (corresponding to the steel grade 30CrNiMo8), Sen et al. reported an activation energy of 243 kJ/mol [35]. Nevertheless, there are differences between these studies, mainly in the chemical composition of the substrate, the type of boronizing medium (pack vs. slurry salt bath) and the temperature ranges investigated. When comparing the values of activation energy reported in different studies, Orihel et al. pointed out the key factors for the scattering of results, which include: the different chemical composition of the base material, the boronizing method and medium, the process parameters, the calculation method or the approach and nature of chemical reactions controlling the process [4].

Substituting the estimated values into Eq. (5), the following expression is obtained:

$$d = \sqrt{1,54 \cdot 10^{-5} \cdot t \cdot \exp\left(-\frac{173450}{RT}\right)} \quad (6)$$

Eq. (6) describes the relationship between the boride layer thickness, the boronizing temperature and the duration for the steel 30CrNiMo8. The resulting mathematical model is suitable for practical application as it allows the calculation of the boride layer thickness based on the boronizing parameters (temperature and duration) or the selection of parameters to achieve a desired thickness.

4 CONCLUSION

In this study, the kinetics of boride layer growth on 30CrNiMo8 steel was investigated. Based on the results, the following conclusions can be drawn:

- The boride layers formed exhibit a characteristic sawtooth morphology, which is less pronounced compared to the layers formed on low-carbon or unalloyed steels.
- The thickness of the boride layers ranges from 32,9 to 122 μ m, with a strong dependence on the boronizing

temperature and duration. Higher boronizing temperatures and longer durations led to the formation of thicker boride layers.

- The results confirm the diffusion-controlled nature of the boronizing process. The activation energy for the boronizing of 30CrNiMo8 steel was estimated to be 173,45 kJ/mol and the frequency factor to be $1,54 \times 10^{-5}$ m²/s.
- Mathematical model was developed to describe the relationship between the boride layer thickness, temperature and duration, which is valid for the boronizing of 30CrNiMo8 steel. This model is suitable for practical application as it allows a quick and easy estimation of the boride layer thickness based on the boronizing parameters.

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