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Impact of the acidity of the medium on the corrosion resistance of the boronized surface layer – based on the cross-section of the boronized layer obtained on X6CrNiTi18-10 steel

Wpływ kwasowości środowiska na odporność korozyjną na przekroju poprzecznym naborowanej warstwy wierzchniej stali X6CrNiTi18-10

The paper presents the results of research on the impact of the acidity of the environment on the corrosion resistance of X6CrNiTi18-10 steel after the boronizing process. The assessment of the corrosion resistance of the surface layer was carried out using the method of progressive thinning, which consists in performing corrosion tests on increasingly deeper areas of the layer. The use of such methodology in corrosion tests allows to track changes in individual parameters read from the potentiokinetic polarization curves and, consequently, to plot their depth profiles. The resistance of the boronized layer to general corrosion was tested in acidified sulphate solutions with three pH values: 1, 2 and 4. The structure and properties of the resulting surface layer were assessed on the basis of structural tests and microhardness changes on the cross-section of the surface-treated material. It has been found that the corrosion rate of the general surface layer is 3-4 times higher than the corrosion rate of core material, but only for strongly acidic environments. The characteristics of the passive state of steel deteriorate in particular, which is visible by the increase in the value of the critical passivation current, the minimum current in the passive range and by limiting the tendency to secondary passivation. For less acidic environments, boronization generally leads to deterioration of corrosion resistance, however, this deterioration is less than expected.

Keywords: corrosion, stainless steel X6CrNiTi18-10, boronized layers

W pracy przedstawiono wyniki badań dotyczących wpływu kwasowości środowiska na odporność korozyjną warstwy wierzchniej na stali typu X6CrNiTi18-10 po procesie naborowywania. Ocenę odporności korozyjnej warstwy przeprowadzono, wykorzystując metodę postępującego ścieniania, która polega na wykonywaniu testów korozyjnych na coraz głębiej położonych obszarach warstwy. Zastosowanie takiej metodyki w badaniach korozyjnych pozwala śledzić zmiany poszczególnych parametrów odczytywanych z potencjokinetycznych krzywych polaryzacji oraz wykreślić ich profile głębokościowe. Badania odporności na korozję ogólną warstwy naborowanej wykonano w zakwaszonych roztworach siarczanowych o trzech wartościach pH: 1, 2 i 4. Budowę i własności powstałej warstwy wierzchniej oceniono na podstawie badań strukturalnych oraz zmian mikrotwardości na przekroju poprzecznym obrobionego powierzchniowo materiału. Stwierdzono, że szybkość korozji ogólnej warstwy wierzchniej jest 3-4-krotnie większa niż szybkość korozji osnowy tylko dla silnie kwaśnych środowisk. Szczególnemu pogorszeniu ulegają charakterystyki stanu pasywnego stali, co uwidacznia się przez wzrost wartości krytycznego prądu pasywacji, minimalnego prądu w zakresie pasywnym oraz przez ograniczenie tendencji do pasywacji wtórnej. W wypadku środowisk mniej kwaśnych naborowanie prowadzi generalnie do pogorszenia odporności korozyjnej, jednak pogorszenie to jest mniejsze od oczekiwanego.

<u>Słowa kluczowe</u>: korozja, stal nierdzewna X6CrNiTi18-10, warstwy naborowywane

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Fig. 1. Microstructure on the cross-section of the surface layer of X6CrNiTi18-10 steel after the boronization process with division into zones and characteristic hardness for each zone

Rys. 1. Mikrostruktura na przekroju poprzecznym warstwy wierzchniej stali X6CrNiTi18-10 po procesie naborowywania, z podziałem na strefy oraz charakterystycznymi twardościami i grubościami dla każdej ze stref

1. Introduction

Boronization, i.e. saturation of the steel surface with boron, is a thermochemical treatment in which boron atoms diffuse into the metal substrate and form a very hard and wear-resistant surface layer [1]. Many types of steel are suitable for boronization, including: carbon steels, low-alloy steels, high-alloy steels, tool steels, cast irons, stainless steels, cobalt alloys and nickel alloys. During the boronization of ordinary carbon steel, two phases of borides are formed, namely: FeB with 16.1% B by mass. and Fe₂B with 8.8% B, the second of which, due to its lower brittleness, is definitely more desirable in industrial applications [2, 3]. During boronization of alloy steels, additionally formed borides may include some alloying elements, such as chromium (Cr) or nickel (Ni) [4]. The amount and type of boride phases formed on the surface depends not only on the chemical composition of the steel, but also on other process parameters, i.e. boronization method, temperature or process time [5].

Stainless steels, with their high resistance to aggressive environments, do not always have sufficiently high hardness and strength. It seems advisable to find a surface treatment that will improve the strength of this material while minimizing the loss of corrosion resistance. This work undertakes research aimed at learning about changes in corrosion resistance on the cross-section of the boronized surface layer depending on the acidity of the corrosive environment. Using progressive thinning method, depth profiles of the most important parameters determining the corrosion resistance of the material were plotted. Such profiles, combined with the knowledge of the structure and phase changes in the layer, make it possible to properly design the thermo--chemical treatment and the subsequent proper use of the already processed material.

The aim of this work is a comprehensive assessment of corrosion properties in a non-oxidizing acidic environment of boron--modified layers over their entire cross-section. In particular, determination of depth profiles of corrosion parameters enriched in boron of surface layers. These studies are a continuation of earlier scientific works [6, 7], in which the authors focused on the characteristics of the structure and mechanical properties of boronized layers.

2. Materials and Methods

The subject of the tests were samples of austenitic X6CrNiTi18-10 steel, the surface of which was subjected to saturation with boron at 900°C for 6 hours. As a source of boron the Ekabor II composition, Bortec GmBH (main ingredients: silicon/boron carbides; powder size <850 μ m) mixed together with KBF₄ activator and alumina filler has been applied. The metallographically prepared specimens (etching in HCI–HNO₃ solution) were observed with the use of Keyence VHX 7000 optical microscope. Observations were carried out on cross-sections, which allowed to isolate individual, differently etching zones of the boronized layer. The hardness distribution in the surface layer was measured using the Vickers method at a load of 0.4903 N (HV 0.05).

Potentiokinetic polarization curves were determined in 0.5 M sulphate solutions acidified to pH = 1, 2 or 4. During the measurements, the solutions were deaerated with argon. The samples for electrochemical tests were in the form of rotating discs with a working surface area of 0.2 cm². Before each potentiokinetic measurement, parallel layers of 4-10 µm were grounded from the surface of the disc electrodes, moving from the surface samples (boronized surface) into the core material. The average thickness of the grounded layer was determined on the basis of the mass loss of the tested disc electrode in relation to the initial mass with an accuracy of ±0.02 mg. The adopted methodology in analogy to the works [8-10] referred to as the progressive thinning method. Potentiokinetic tests were carried out at a temperature of 25°C ±0.1°C, at a disk rotational speed of 12 rpm and a potential scanning speed of 5 mV \cdot s⁻¹, using its shift from the potential value of about 20 mV more from negative corrosion potential (E_{corr}) to $E_{stop} = 1.9$ V. Before polarization tests, the samples were immersed in a corrosion solution for 0.5 hours to establish the stationary potential (open circuit potential - OCP). Values of all electrode potentials are relative to a 1 M silver chloride electrode. The corrosion rate (i_{corr}) was determined on the basis of the measurement of the polarization resistance (R_p) [11]. In the range of potentials slightly different from $E_{\rm corr}$ the external current density is a linear function of the potential, and the tangent of the angle of inclination of this relationship is inversely proportional to the corrosion rate. Assuming that the Tafel slopes are analogous to those for iron in sulfate solutions [12],

it can be concluded that $i_{corr} = 0.0130 R_p^{-1}$ for pH = 1 and 2 and $i_{corr} = 0.0174 R_p^{-1}$ for pH = 4 (where i_{corr} is expressed in A \cdot cm⁻² and R_p in $\Omega \cdot$ cm²).

3. Results and discussion

Figure 1 shows an exemplary image of the microstructure obtained on the cross-section of the surface layer after boronization. The obtained layer shows a typical three-zone structure, where a zone of M_2B precipitates extends under the MB-type precipitates, which then turns into a diffusion zone – such three-zone model can also be observed in other scientific papers [6]. In the near-surface area, the columnar structure characteristic of borides is not observed [1, 13], but the morphology of the layers is flat due to the high surface tension resulting from the alloying elements present in the material. If the content of alloying elements, mainly chromium and nickel, in the steel is increased, the development of the jagged boronized/ core material interface is inhibited and a smooth interface is formed [14–16].

The point analysis of the chemical composition for different layer depths [7] suggests that the iron borides (FeB and Fe₂B) are present in the structure, and due to the increased content of chromium and nickel in the tested steel, also the possibility of precipitates: CrB, Ni₂B and (Fe,M)₂B, where M = Cr or Ni [17–20]. With the applied process parameters, structural changes are observed down to a depth of approx. 70–80 µm. The hardening of the surface is also observed to a comparable depth, with the maximum hardness of 2500 HV 0.05 characterized by the outermost layer of the MB type reaching a depth of about 35 µm. In the deeper areas of the boronized layer, the hardness systematically decreases, assuming values of 1400–2000 HV 0.05 for M₂B borides, and at depths greater than 75 µm, the values characteristic of non-boronized X6CrNiTi18-10 steel – about 300 HV 0.05 (Fig. 1).

The anodic polarization curves of boronized stainless steel at three pH values are shown in Fig. 2a–2c. Among the several dozen polarization curves made for successive layers removed by progress-ive thinning, the figures show only the curves representative for individual zones with a distinctly different structure (see Fig. 1).

The series of curves presented in Fig. 2 characterize the corrosive behaviour of the material at several different depths from the surface previously subjected to thermo-chemical treatment. As can be seen from the graphs, the enrichment of the X6CrNiTi18-10 steel surface with boron deteriorates the anodic behaviour of the surface layers of this material. This deterioration is manifested by high current values both in the state of transition to the passive state and in the passive range. For non-boronized steel (depth >70 µm) above the potential of approx. E = -0.2 V, a clear decrease in the anode current is observed (to approx. 0.1 mA \cdot cm⁻² for pH = 1 and pH = 2) which should be associated with effective passivation. The boronized surface of the X6CrNiTi18-10 steel behaves differently, these layers are not passivated above -0.2 V, but dissolve at high rates up to the potential of +0.5 V. Above this potential value, a short section of the not very effective passive state is visible (from +0.5 to +0.9 V, for pH = 2 with anode currents $i_a \approx 1 \text{ mA} \cdot \text{cm}^{-2}$), after which the boronized surface dissolves transpassively. While the base steel above E = +0.9 V shows a certain tendency to secondary passivation, the boronized zones dissolve in the transpassive range at a considerable speed. Thus, saturation of the surface of acid-resistant steel with boron leads to a significant deterioration of its corrosion resistance in acidic environments (pH = 2), and in particular to the loss of self-passivation ability.

Parallel grinding and electrochemical analysis of successive surface layers of the material leads to the visualization of significant changes in the characteristic corrosion parameters determined from the polarization curves. Successive potentiokinetic curves plot-



Fig. 2. Potentiodynamic polarization curves for the boronized X6CrNiTi18-10 steel and ground away to a specific depth (µm). Potentiodynamic polarization curves obtained in a 0.5 M sulphate solution at three chosen pH values: a) pH = 1, b) pH = 2, c) pH = 4. Experimental conditions: $25^{\circ}C \pm 0.1^{\circ}C$, electrode rotation speed: 12 rps, potential scan rate: $5 \text{ mV} \cdot \text{s}^{-1}$

Rys. 2. Potencjodynamiczne krzywe polaryzacji wykonane dla naborowywanej i zeszlifowanej do określonej głębokości (µm) stali X6CrNiTi18-10 zmierzone w 0,5 M Na₂SO₄ o trzech wartościach pH: a) pH = 1, b) pH = 2, c) pH = 4. Warunki eksperymentu: 25°C ±0,1°C, prędkość obrotowa dysku: 12 obr. · s⁻¹, szybkość skaningu potencjału: 5 mV · s⁻¹

ted using the progressive thinning method, recorded for deeper and deeper zones of the boronized layer, are characterized by a systematic decrease in the critical passivation current (i_{cp}) as well as, to a much lesser extent, the minimum anode current in the passive range ($i_{min,p}$). Figure 3 shows the depth profiles of the critical passivation current and the minimum current in the passive range.



Fig. 3. Depth profiles: a) of the critical passivation current, b) of the minimum current within the passive range, red out from the polarisation curves plotted for the boronized X6CrNiTi18-10 steel in 0.5 M sulphate solutions with pH = 1, 2 and 4

Rys. 3. Profile głębokościowe: a) krytycznego prądu pasywacji, b) minimalnego prądu w zakresie pasywnym, wykreślone na podstawie krzywych polaryzacyjnych wykonanych w 0,5 M roztworach siarczanowych o pH = 1, 2 lub 4 dla naborowanej stali X6CrNiTi18-10



Fig. 4. The effect of pH on the values of i_{cp} and $i_{min,p}$ for the boronized X6CrNiTi18-10 steel at a depths of 20 μm and 40 μm

Rys. 4. Wpływ pH na wartości $i_{\rm kp}$ oraz $i_{\rm min.p}$ dla naborowanej stali X6CrNiTi18-10 na głębokościach 20 $\mu{\rm m}$ i 40 $\mu{\rm m}$

As it results from the plotted profiles (Fig. 3), the values of both the critical passivation current (i_{cp}) and the minimum current in the passive range (imin.p) systematically decrease with increasing distance from the boronized surface. It is worth noting that the values of these parameters are only slightly dependent on the acidity of the environment. Thus, the i_{cp} values in the MB-type precipitation zone (depth up to about 35 μ m) are practically the same for the environment with pH = 1 as for pH = 2 and only slightly lower for pH = 4, in the M₂B-type isolation zone there are comparable for all used environments. However, as can be seen in Fig. 3a, for the volume of the material, the $i_{\rm cp}$ values are definitely lower for the least acidic environment, confirming the ease of transition of stainless steel to the passive state. A small influence of the acidity of the environment is observed for the $i_{\rm min,p}$ values, which are practically convergent for pH = 1 and pH = 2 both in the boronized zone and in the core material. Figure 4 shows the current densities i_{cp} and $i_{\min,p}$ read for each pH from the polarization curves for the MB $(20 \ \mu m)$ and M₂B (40 μm) precipitation zones.

As it has been shown in Fig. 4, for both (MB and M_2B zones), the dependence expressed by the formula (1) applies:

$$\left(\frac{\partial \log i}{\partial p_{\rm H}}\right)_{T = 298 \,\rm K} = -0.1. \tag{1}$$

Similar results and dependencies are also observed for other post-treatment engineering processes – in particular for X6CrNiTi18-10 steel after the nitriding process [21].



Fig. 5. Linear polarization measurements of the boronized X6CrNiTi18-10 steel as a function of the depth of grinding down for different acidities of the corrosive environment: a) pH = 1, b) pH = 2, c) pH = 4

Rys. 5. Pomiary polaryzacji liniowej naborowanej stali X6CrNiTi18-10 w funkcji głębokości szlifowania dla różnych kwasowości środowiska korozyjnego: a) pH = 1, b) pH = 2, c) pH = 4

Figure 5 shows linear polarization relationships for selected zones of the boronized layer for potentials slightly different from the corrosion potential. The slope of the corresponding lines is a measure of the polarization resistance (R_p), which allows the



Fig. 6. The rate of corrosion of the boronized X6CrNiTi18-10 steel at the depths of 20 μ m (MB), 40 μ m and >70 μ m (core) from the surface as a function of pH of 0.5 M sulphate solution

Rys. 6. Szybkość korozji naborowanej stali X6CrNiTi18-10 na głębokościach 20 μm (MB), 40 μm i >70 μm (rdzeń) od powierzchni w funkcji pH 0,5 M roztworu siarczanowego

calculation of the corrosion current ($i_{\rm corr}$), which is a measure of the corrosion rate.

The greatest differences in the slopes of straight lines representing the dependence of the current density on the potential, and thus in the polarization resistance values, between the boronized surface layer and non-boronized steel are visible in the environment with the lowest pH (pH = 1, Fig. 5a). In this environment, for both zones of the surface layer (both MB and M₂B), the polarization resistance takes very similar values, definitely different from the non-boronized zone. For environments with lower acidity, such strong differences in the slope of the above dependence are not observed. The corrosion rate calculated on the basis of the polarization resistance depending on the pH of the environment for selected zones of the boronized layer is shown in Fig. 6.

As could be expected, the highest i_{corr} values are observed for the most acidic medium, because as it is known, the rate of corrosion of metallic materials is strongly influenced by the concentration of hydrogen ions. Differences in material dissolution rates at different depths of the surface layer are observed for individual environments with different acidity. The greatest discrepancies are observed for pH = 1: for the outermost areas of the boronized layer, where MB-type precipitates dominate, i_{corr} is the highest, almost 4 times higher than the area not enriched in boron, and for the zone of M₂B precipitates it is 3 times higher. With the decrease in the acidity of the environment, the differences in the corrosion resistance of individual zones are smaller and smaller.

4. Conclusions

Saturation of the X6CrNiTi18-10 steel surface with boron leads to the formation of a boron-rich surface layer reaching a depth of approx. 70–80 μ m, which significantly improves the mechanical properties of the surface. The obtained layer shows a zonal structure with a different structure and properties

The use of the progressive thinning method makes it possible to track changes in corrosion resistance over the entire cross-section of the surface layers. The enrichment of the steel surface with boron worsens its corrosion resistance the most in the outermost areas of the layer where MB-type precipitates predominate.

Boronization of X6CrNiTi18-10 steel generally leads to a deterioration of its corrosion resistance in acidic sulphate environments, although the deterioration is less than expected. Significant deterioration of corrosion resistance is observed only for strongly acidic environment. For less acidic environments, the differences in material dissolution rates are not so large.

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