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ANALYSIS OF THE BORONIZED LAYER ON K 190 PM TOOL STEEL

ANALIZA BORONIZIRANE PLASTI V ORODNEM JEKLU K 190 PM, IZDELANEM PO POSTOPKU METALURGIJE PRAHU

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This work deals with the characterization of the phases in boronized layers formed for particular processing parameters on tool steel of the ledeburite type. The high-alloyed tool steel K190 PM prepared by powder metallurgy was used as a substrate. X-ray diffraction (XRD) measurements were carried out at different depths of boronized layers, as well as of transient layers, using a Philips PW1710 diffractometer. The analysis revealed that the surface consisted of FeB (MeB) and an unidentified crystalline phase (or phases), which amount decreases with increasing distance from the surface. Also, a gradual increase in the amount of Fe_2B (Me_2B) at the expense of MeB was observed with increasing distance from the surface. The layer neighboring the substrate consists mainly of Me_2B and CrB. The transition layer between the borides and the base material was found to contain the $Me_{23}(C,B)_6$ phase.

Key words: tool steel, boronized, layer, constitutive phases, microhardness

Predstavljena je karakterizacija faz v boronizirani plasti, pripravljeni na ledeburitnem orodnem jeklu. Kot podlaga je uporabljeno jeklo K 190 PM, pripravljeno po postopku metalurgije prahu. Difrakcija rentgenskih žarkov je bila izvršena pri različnih globinah boronizirane in prehodne plasti z difraktometrom Philips PW 1710. Analiza je pokazala na površini fazo FeB (MeB) in neidentificirano kristalno fazo (ali faze), katerih vsebnost se zmanjšuje z razdaljo od površine. Ugotovljena je tudi postopna rast količine faze Fe_2B (Me_2B) na račun faze MeB z oddaljevanjem od površine. Plast na meji s podlago je iz Me_2B in CrB. V prehodni plasti med boridi in osnovnim materialom smo našli faze $Me_{23}(C,B)_6$.

Ključne besede: orodno jeklo, boronizirana plast, sestavne faze, mikrotrdota

1 INTRODUCTION

In addition to the popular techniques of carburizing and nitriding, boronizing also plays an important, though less common, role in thermo-chemical treatments. Boronizing usually leads to the formation of a very hard and wear-resistant layer on the surface of the material. Due to the diffusion of the boron into the surface of the carbon steel, iron borides with very high hardnesses are formed. The boronized layers exhibit a higher hardness than the layers prepared with carburizing or nitriding. Generally, low-carbon and low-alloyed steels are boronized. This process can also be applied to austenite steels, non-ferrous alloys (nickel or cobalt), heat-resistant metals such as Ti and Ta and also to sintered carbides, such as WC-Co, with the aim of improving their wear resistance [1,2]. Boronizing is currently also used for a number of applications where no surface treatment has been used so far. It is applied to various forming tools, components working in an abrasive environment, dies for the injection casting of non-ferrous alloys and others. Furthermore, it is suitable for preparing functional layers with an extended lifetime of the substrate material due to the increased hardness of the surface. During the boronizing of plain steels, FeB or Fe_2B is formed in the surface layers. The thickness of the boride layers typically varies in the range from 20 to 300 μm [3,4]. The aim of this paper is to present a detailed XRD characterization of the boronized layer formed on the surface of K 190 PM high-alloyed tool steel.

2 EXPERIMENTAL

The boronized layer investigated was formed on K 190 PM tool steel prepared by powder metallurgy. The chemical composition of the steel is shown in **Table 1**.

Table 1: Chemical composition of K 190 ISOMATRIX PM steel
Tabela 1: Kemijska sestava jekla K 190 ISOMATRIX PM

Chemical element	Fe	C	Si	Mn	P	S	Cr	V	Mo
Content *w*/%	balance	2.29	0.5	0.32	0.026	0.019	12.3	4.06	1.06

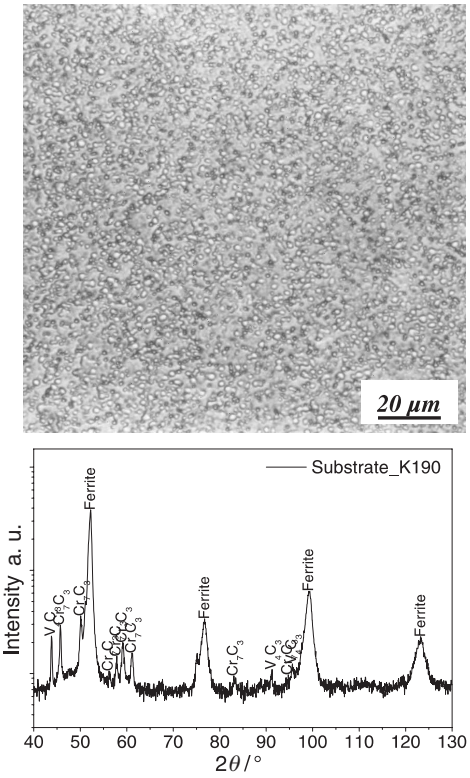
The steel was delivered as a rod with a diameter of 10 mm. For the purpose of the boronizing experiment, the rod was cut into 10-mm-long pieces. The pieces were cleaned and degreased and boronized at at 1000 °C for 8 h in a (80 × 85 × 125) mm hermetic container filled with a *DURBORID* powder mixture. After boronizing the samples were cooled to room temperature in containers in the open air. The microstructure of the boronized K 190 PM tool-steel layer consisted of a boride and a transient layer. The microhardnesses of the boride layer,

the transient layer, and the substrate were measured with a Hannemann indenter fitted to a Zeiss Neophot 21 light microscope.

The XRD patterns of the boride layer, the transient layer and the substrate were recorded with a Philips PW 1710 diffractometer using Fe-filtered CoK_α radiation. The experimental device was equipped with a secondary graphite monochromator and a pulse-height proportional detector. Data were recorded in the range 20–144° (2θ) using a constant step of 0.05° and a 10 s exposure time and samples rotating. Qualitative and quantitative analyses of the phases were also carried out using X-ray diffraction on the as-delivered substrate of soft-annealed K 190 PM steel.

Exploiting the natural penetration depth of the X-ray radiation, the analysis was performed in consecutive steps. First, the as-prepared boronized substrate without any mechanical cleaning was examined using XRD and microhardness measurements. Next, a predetermined volume of the surface layer was removed with grinding with APEX diamond discs. The new surface was then examined using the same experimental tools. The series contained a total of 10 XRD patterns, taken from the as-prepared surface and the surfaces at the depth of (2, 7, 20, 30, 40, 50, 60, 70, 80) μm. It should be emphasized that the information extracted from a single diffraction pattern taken from a boride layer represents the volume corresponding to the penetration depth of X-rays. Therefore, it cannot be considered as a characterization of the surface but rather of a layer of a certain thickness located at various depths.

The identification of the peaks in the XRD patterns was based on a comparison of the calculated hkl and the d_{hkl} list of relevant phases determined from the chemical composition of the substrate, further revised with data on the phase identification in the boride layers [1-5]. For the quantitative treatment of the XRD data the Powder Cell 2.4 computer program and, in special cases, the MAUD program based on a Rietveld algorithm [6-8]) were used. All the crystallographic data used for the calculation of the theoretical diffraction patterns were taken from Pearson's handbook [9].



Phase	Ferrite	V_4C_3	Cr_7C_3
Quantity φ / %	72.1 ± 1.4	5.6 ± 0.1	22.3 ± 0.4

Figure 1: Microstructure and XRD pattern of the soft-annealed K 190 PM steel. A table summarizing the amounts of the different phases is included

Slika 1: Mikrostruktura in XRD-posnetek mehko žarjenega jekla s tabelo z vsebnostjo različnih faz

3 RESULTS AND DISCUSSION

The microstructure of the soft-annealed substrate of K 190 PM is shown in Figure 1. It consists of a uniform distribution of small spheroidized M_7C_3 and MC carbides in the ferrite matrix, which is characteristic for the soft annealed K 190 PM tool steel. The results of the quantitative phase analysis of the substrate determined from the XRD (Figure 1) by the Rietveld method using the Maud computer program are shown in the table in **Figure 1**.

Figure 2 shows the XRD data collected at different depths of the boride and transient layers. The XRD pattern taken from the as-prepared surface and from 2 μm and 7 μm into the boronized layer consist of the dominant FeB (MeB), the minor Fe_2B (Me_2B), CrB, and an unidentified phase (or phases).

The diffraction peaks attributed to the unidentified phase (or phases) did not appear in the diffraction pattern taken from the surface located at a depth of 20 μm. The intensity of the reflections attributed to the Me_2B phase increased slightly between the 7-μm and 20-μm scan.

The observed trend of increasing intensity of the Me_2B phase was confirmed with a series of XRD scans taken from depths of 30 μm and 40 μm. It can be concluded from the next layer in the series, taken from a depth of 50 μm, that the MeB phase becomes a minor phase. It is also possible to observe two strong reflections of the Me_2B phase with increasing intensity. Moreover, the reflection observed at approximately 52° (2θ) suggests an increasing diffraction intensity of the ferrite matrix.

Both the MeB and the Me_2B observed in the depth ranges from 0 μm to 60 μm were influenced by epitaxial growth of the (020) and (002) planes, respectively.

When the material from the boronized layer is removed down to 70 μm or 80 μm the XRD patterns change significantly. It was found that the layer down

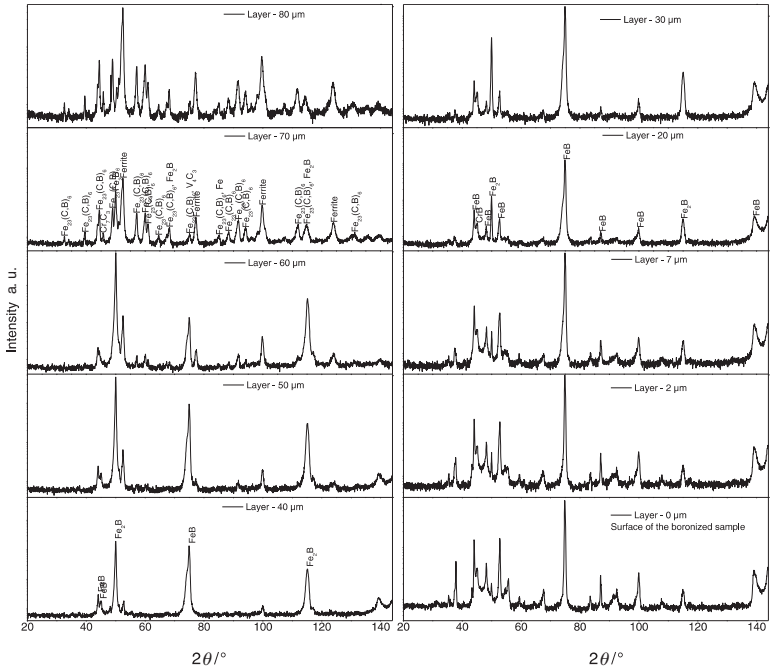


Figure 2: XRD patterns taken from a series of depths in the boronized layer formed on K190 PM tool steel
Slika 2: XRD-posnetki pri različnih globinah boronizirane plasti na jeklu K 190 PM

below 70 µm contains complex iron-chromium carboboride, with a crystal structure corresponding to $Me_{23}(C,B)_6$, and ferrite. Neither MeB nor Me_2B were identified in this depth range. It is, therefore, suggested that the patterns taken from depths of 70 µm and 80 µm are already located in the transient zone between the boride layer and the substrate. The presence of this phase agrees well with earlier publications, where Cr was considered to be an element less soluble in the boride layer. As a result of this behavior, Cr is enriched in the transient zone ahead of the boride layer and forms the complex Cr-based carbide $Me_{23}(C,B)_6$ or carbo-borides, where carbon is partially replaced with boron [5].

A simplified representation of the phase composition in the boride and the transient layers is shown in **Figure 3a** in the form of a color map of dominant phases as a function of layer depth. The map shows that Fe_2B (Me_2B) and FeB (MeB) are present in both the boride and the transition layers after 8 hours of boronizing at 1000 °C.

Figure 3b shows the quantity of FeB (MeB) and Fe_2B (Me_2B) determined from XRD patterns and the variation of the microhardness as a function of the depth of the layer. The quantity of MeB and Me_2B was determined neglecting other phases (the unidentified phase (or phases), CrB and $Me_{23}(C,B)_6$). The quantitative results were obtained from the Rietveld algorithm implemented in Powder Cell 2.4 [6]. In this graph, the microhardness profile across the analyzed depth profile is also shown. The color map (**Figure 3a**), in combination with the quantity of phases and the microhardness

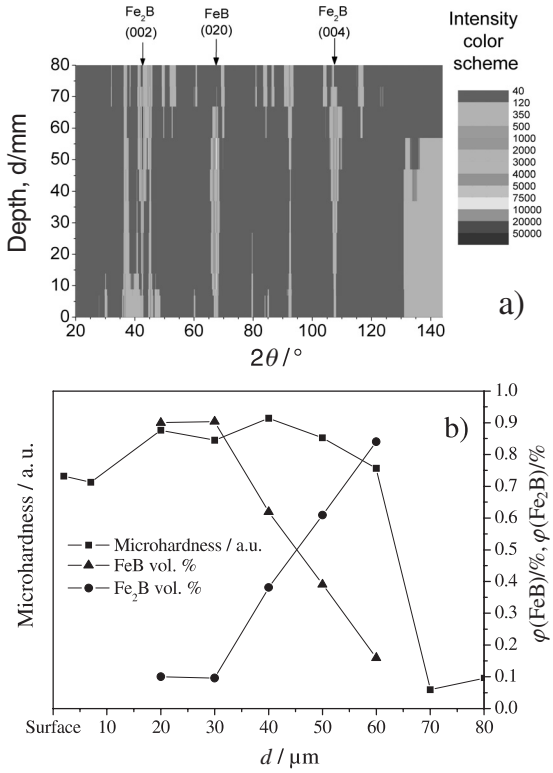


Figure 3: Color map showing the occurrence of the identified phases as a function of the layer depth determined from XRD (**3a**) and the quantities of FeB (MeB) and Fe_2B (Me_2B) determined from the XRD experiment and the microhardness profile plotted as function of the depth of the layer (**3b**).

Slika 3: Barvni prikaz prisotnosti identificiranih faz v odvisnosti od globine plasti, določen z XRD-posnetkom (**3a**), vsebnost FeB (MeB) in Fe_2B (Me_2B) določene iz teh posnetkov in mikrotrdota v odvisnosti od globine plasti (**3b**)

profile shown in **Figure 3b**, is proposed as an effective tool for designers and producers.

4 CONCLUSIONS

The paper presents an estimate of the quantities of MeB and Me_2B at various depths in the boronized layer. The quantitative evaluation was performed, neglecting other phases (an unidentified phase (or phases) or CrB and $Me_{23}(C,B)_6$) present in the boronized layer. It was found that the uppermost layers, down to the depth of 20 μm, consist of MeB and some unidentified phases. The amount of unidentified phase (or phases) decreases with the increasing depth below 7 μm and this phase is not found at the depth of above 20 μm. Most of the MeB was found in the layers from 20 μm to 40 μm. MeB was not found in the layers from 70 μm to 80 μm. The majority of the Me_2B was found in the range from 50 μm to 60 μm. A minor amount of this phase was found in other layers over the entire thickness of the boronized layer. A strong epitaxy into the (020) and (002) planes was observed for the growth of the MeB and Me_2B, respectively. The CrB phase was identified in small quantities from XRD patterns in the range from 0 μm to 50 μm from the surface. Diffraction patterns from depths of 70 μm to 80 μm revealed the presence of $Me_{23}(C,B)_6$, which identifies this region as the transient layer. The highest microhardness of the boronized layer was observed in the range 20 μm to 50 μm from the surface. In the range from 0 μm to 20 μm the measured microhardness was found lower down, probably due to the influence of the unidentified phase (or phases).

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