Contents lists available at ScienceDirect





Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Characterization of AISI 4140 borided steels

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ARTICLE INFO

Article history: Received 2 October 2009 Accepted 20 October 2009 Available online 24 October 2009

Keywords: Boriding Growth kinetics Characterization Diffusion model Fracture toughness Hard coatings Residual stresses

ABSTRACT

The present study characterizes the surface of AISI 4140 steels exposed to the paste-boriding process. The formation of Fe₂B hard coatings was obtained in the temperature range 1123–1273 K with different exposure times, using a 4 mm thick layer of boron carbide paste over the material surface. First, the growth kinetics of boride layers at the surface of AISI 4140 steels was evaluated. Second, the presence and distribution of alloying elements on the Fe₂B phase was measured using the Glow Discharge Optical Emission Spectrometry (GDOES) technique. Further, thermal residual stresses produced on the borided phase were evaluated by X-ray diffraction (XRD) analysis. The fracture toughness of the iron boride layer of the AISI 4140 borided steels was estimated using a Vickers microindentation induced-fracture testing at a constant distance of 25 μ m from the surface. The force criterion of fracture toughness was determined from the extent of brittle cracks, both parallel and perpendicular to the surface, originating at the tips of an indenter impression. The fracture toughness values obtained by the Palmqvist crack model are expressed in the form $K_C(\pi/2) > K_C > K_C(0)$ for the different applied loads and experimental parameters of the boriding process.

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1. Introduction

Boriding is a thermochemical surface treatment, in which boron is diffused into, and combines with, the substrate forming a single or double phase metal boride layer at the surface. In industry, boriding is generally applied to ferrous alloys to enhance their surface hardness and wear resistance [1]. In addition to being a selective method, the paste-based treatment reduces manual work compared with the powder-based boriding process [2,3]. Depending on the boron potential that surrounds the material surface, the chemical composition of the substrate, temperature and treatment time, two phases can be identified in the surface layer, i.e. an outer phase, FeB, with a boron content of 16 wt.%, and an inner phase, Fe₂B, with a boron content approximately of 8 wt.% [4,5]. Previously, it was found that the interfaces of FeB/Fe₂B and Fe₂B/substrate, which are present at the surface of different ferrous and nonferrous alloys in the boriding process, have a rough or saw-toothed morphology. However, when the alloying elements increase in concentration on the substrate, the formation and morphology of the growth interface at the surface of the sample tends to be flat [6].

The present study characterizes the surface of AISI 4140 steels hardened by the paste-boriding process. The evaluation of

the growth kinetics of Fe_2B layers at the material surface was done at different boride incubation times to estimate the boron diffusion coefficient at the hard coatings. The GDOES technique showed the concentration profile of alloying elements that diffused in the borided phase. The XRD technique was used to estimate the magnitude and distribution of residual stresses along the boride layer. Finally, the fracture toughness of the borided phase was evaluated under the experimental parameters of 6 and 8 h of treatment for the different temperatures of the boriding process, considering the length of brittle cracks, parallel and perpendicular to the surface that originate at the tips of an indented impression.

2. Diffusion model

The growth kinetics of borided layers has received significant attention during the last 20 years for the automation and optimization of the boriding process [7-10].

In this work, some assumptions were considered for the diffusion model:

- (i) The boron concentration C_{Fe₂B}(x(t)) at the Fe₂B phase (Fig. 1) depended only on the position x(t).
- (ii) The growth kinetics was controlled by the boron diffusion in the Fe₂B layer.
- (iii) The growth of the boride layer occurred as a consequence of the boron diffusion perpendicular to the specimen surface.

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^{0169-4332/\$ -} see front matter 0 2009 Elsevier B.V. All rights reserved. doi:10.1016/j.apsusc.2009.10.070

- (iv) The boron concentration values along the Fe_2B /substrate interface were unknown.
- (v) Differences in specific volume per solvent atom for the Fe₂B phase were accommodated fully in the diffusion direction.
- (vi) The influence of the alloying elements on the growth kinetics of the layer was not taken into account.

The C_{ads}^{B} is defined as the effective boron concentration at the borided layer [9]. Since only Fe phase is present before boriding, the initial condition can be written as:

$$C_{\text{Fe}_2\text{B}}(x(t>0)=0)=0$$

The boundary conditions of the problem are considered in the form:

$$C_{\text{Fe}_2\text{B}}(x(t = t_0(T)) = 0) = C_{\text{up}} \text{ for } C_{\text{ads}}^{\text{B}} > 8.83 \text{ wt.}\% \text{ B}$$

 $C_{\text{Fe}_2\text{B}}(x(t = t) = u) = C_{\text{low}} \text{ for } C_{\text{ads}}^{\text{B}} < 8.83 \text{ wt.}\% \text{ B}$

 C_{up} is the upper limit of boron concentration at Fe₂B phase and C_{low} is the lower limit of boron concentration at Fe₂B phase.

The mass balance equation at the Fe_2B /substrate interface was set as [11,12]:

$$(C_{\text{low}} - \beta C_0) \left(\frac{dx(t)}{dt} \right) \Big|_{x(t)=u} = -D_{\text{Fe}_2\text{B}} \frac{dC_{\text{Fe}_2\text{B}}(x(t))}{dx(t)} \Big|_{x(t)=u}$$
(1)

 C_0 is the initial boron concentration in the substrate and β is the ratio of the specific volume per solvent atom, $\beta = V_0^m/2V_{Fe_2B}^m (V_0^m \text{ is the molar volume of the substrate and } V_{Fe_2B}^m \text{ is the molar volume of the Fe}_2B$ phase (m³/mol)).

Using the rule for differentiating compositions of functions in Eq. (1) resulted in the following equation:

$$(C_{\text{low}} - \beta C_0) \left(\frac{\mathrm{d}x(t)}{\mathrm{d}t} \right) \Big|_{x(t)=u} = -D_{\text{Fe}_2\text{B}} \frac{\mathrm{d}C_{\text{Fe}_2\text{B}}(x(t))}{\mathrm{d}t} \left(\frac{\mathrm{d}t}{\mathrm{d}x(t)} \right) \Big|_{x(t)=u}$$
(2)

Hence, Eq. (2) is rewritten as follows:

$$(C_{\text{low}} - \beta C_0) \left(\frac{dx(t)}{dt}\right)^2 (dt) \bigg|_{x(t)=u} = -D_{\text{Fe}_2\text{B}} dC_{\text{Fe}_2\text{B}}(x(t)) \bigg|_{x(t)=u}$$
(3)

The growth of Fe₂B layer was governed by the parabolic growth law $x(t) = u = k(t^{1/2} - t_0^{1/2}(T))$ where u is the layer thickness, k is the parabolic growth constant, t represents the treatment time and $t_0(T)$ is the boride incubation time as a function of treatment temperature [8,13–15].

After rearranging, it follows from Eq. (3):

$$C_{\text{low}} - \beta C_0) \frac{k^2}{4} \int_{t_0(T)}^t \frac{dt}{t} = -D_{\text{Fe}_2\text{B}} \int_{C_{\text{up}}}^{C_{\text{low}}} dC_{\text{Fe}_2\text{B}}(x(t))|_{x(t)=u}$$
(4)

The literature indicates that the upper limit of the boron concentration (C_{up}) in the Fe₂B phase is 9 wt.% B [16–18].

Assuming that the boron concentrations remained constant during the treatment at the boride layer, the D_{Fe_2B} value was determined:

$$D_{\text{Fe}_2\text{B}} = 13k^2 \ln\left(\frac{t}{t_0(T)}\right) \tag{5}$$

3. Experimental procedure

3.1. Paste-boriding process

Samples of AISI 4140 commercial steel with dimensions of $8 \text{ mm} \times 8 \text{ mm} \times 5 \text{ mm}$ were used for the thermochemical treatment. The boriding process was carried out in a conventional furnace under a pure argon atmosphere. The temperatures of 1123, 1173, 1223 and 1273 K with 2, 4, 5, 6 and 8 h were selected. Four millimeters of boron carbide paste covered the



Fig. 1. Boron concentration profile for a diffusion controlled of Fe₂B into an initially homogeneous two phase alloy. The hashed area indicates amount of boron needed to advance the Fe₂B phase by *du*.

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