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

## High temperature tribological behaviour of borided surfaces based on the phase structure of the boride layer

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#### ARTICLE INFO

Article history: Received 12 July 2013 Received in revised form 23 October 2013 Accepted 26 October 2013 Available online 12 November 2013

Keywords: Diffusion treatments High temperature Sliding wear Steel Thermal effects Wear testing

### ABSTRACT

This study focuses on the high temperature tribological behaviour of the boride layers generated on 4140 steel by pack boriding process. Boriding at 750 and 800 °C resulted in a single phase (Fe<sub>2</sub>B) boride layer, while a dual phase (Fe<sub>2</sub>B+FeB) boride layer was developed at 850 and 900 °C. Sliding wear tests conducted by rubbing an alumina ball revealed that single and dual-phase boride layers exhibited almost similar tribological performances and superior wear resistances at room temperature. With increasing testing temperature, boride layers exhibited different wear behaviours depending on their phase structure so that, dual phase boride layer yielded better wear resistance than single phase boride layer, especially at the testing temperature of 300 °C.

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#### 1. Introduction

Surface nature plays a crucial role on the service life and performance of engineering components utilized in corrosion and/or wear related technological applications. One of the solutions for preventation of surface failures caused by friction and contact loads is surface hardening via thermal or thermo-chemical processes and/or covering the surface with hard layer via deposition processes [1]. In the past decades, thermo-chemical processes such as carburizing, nitriding, carbo-nitriding, nitro-carburizing and boronizing have been recognized as attractive surface hardening methods for ferrous alloys. Among these processes, boriding is the primary choice for wear control as well as high surface hardness. It also has the potential to provide enhancements in corrosion and oxidation resistances [2,3]. The borided surface retains its high hardness up to 1000 °C. Furthermore, the remarkable feature of the borided surface is its ability to retain its hardness even after additional heat treatments [4].

In boriding of ferrous alloys, boron potential of the boriding media, the chemical composition of the substrate, process temperature and time govern the phase structure of the boride layer. Depending on the concentration of the diffused boron, the phase structure of the boride layer can be consisted of tetragonal Fe<sub>2</sub>B ( $\sim$ 8 wt% B) and/or orthorombic FeB ( $\sim$ 16 wt% B). In general FeB

phase can be characterized by its higher hardness and rigidity but lower toughness as compared to  $Fe_2B$  phase [4,5]. In this respect, presence of FeB phase in the boride layer is not desirable because of its more brittle nature causing spalling when exposed to high external loads [6,7].

Superior contribution of the boride layer on the room temperature (RT) wear resistance of ferrous alloys has been well established [2,4,6–12]. However, publications on high temperature tribological performance of borided surfaces are very limited in the open literature. In a previous study, conducted on 52,100 and 440C steels, it has been reported that boride layer can maintain its protective nature against wear even at testing temperature of 600 °C, when compared to the unborided state [13]. This study has been initiated with the aim of examining high temperature tribological performances of the borided low alloy 4140 steel. Although the boride layers of 4140 steel are known to be compact, hard, adherent and wear resistant at RT [10,14,15], this study more specifically focused on high temperature tribological performance of the boride layers depending on their phase structures.

#### 2. Experimental details

In the present study, disc shaped samples (35 mm in diameter and 6 mm in height) machined from quenched and tempered (300 HV) 4140 steel (0.39 wt% C, 0.93 wt% Cr, 0.85 wt% Mn, 0.21 wt% Mo, 0.22 wt% Si, 0.023 wt% P, 0.015 wt% S) were used as the





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substrate. Before boriding at 750, 800, 850 and 900 °C for 12 h in an EKabor<sup>®</sup>2 powder containing stainless steel box, the samples were ground using 1000 grit SiC paper and washed in distilled water and ultrasonically degreased in acetone. A stainless steel box was covered with Ekrit<sup>®</sup> powder to prevent oxidation. Fresh powder was used for each boriding process. After the completion of boriding, the stainless steel box was removed from the furnace and cooled in air.

Characterization investigations of the borided surfaces were carried out by structural survey and hardness measurements after gently grinding. The structural survey consisted of light optical microscope (LOM-Leica-CTR 6000, Germany) and energy dispersive X-ray (EDX) spectrometer equipped scanning electron microscope (SFEG SEM, Philips, Holland) examinations and X-ray diffraction (XRD, GBC, Australia) analysis. LOM and SEM examinations were conducted on the cross-sections of the borided samples by utilizing standard metallographic procedure and etching with 2% Nital solution. XRD analyses were conducted on the surfaces of the samples with  $CuK\alpha$  radiation. Hardness measurements were also made on the surface of the borided samples with a Vickers pyramid indenter by utilizing a conventional micro-hardness tester (Shimatzu, HMV2, Japan) under indentation loads, varying between 100 and 2000 g. For each load level, the final hardness results were reported in HV scale as the average of five subsequent measurements.

Wear performance of the borided samples were determined using a ball-on-disc type wear tester (CSM High temperature Tribotester, Switzerland) under dry sliding contact of an alumina ball with a diameter of 6 mm. Wear tests were conducted under normal load of 3 N at RT, 300 and 500 °C with a sliding speed of 0.1 m/s along a circular path of 5.5 mm in radius for 500 m distance. The samples were heated in normal atmospheric condition and the test was started when the desired temperature was reached. For each boriding parameter, at least three samples were tested for the reproducibility of data. During wear testing, frictional force data was continuously recorded. After the wear tests, wear tracks were analysed by using a contact surface profilometer (Dektak-6M, Veeco, USA). Worn surfaces and cross-sections of the wear tracks were also examined in a SEM.

#### 3. Results

Cross-sectional LOM micrographs and the XRD patterns of the samples borided at temperatures of 750, 800, 850 and 900 °C for 12 h are presented in Figs. 1 and 2, respectively. The boride layers covering the surfaces of the samples exhibited saw-tooth morphologies at the interface of the substrate without any evidence of discontinuity. Increase in boriding temperature not only contributed to increase in the thickness of the boride laver but also caused coarsening of the microstructure in the substrate. It should be noted that the outermost sections of the boride lavers formed at 850 and 900 °C (Fig. 1c and d) were darkly etched in comparison to the brightly etched inner sections. In the case of the boride layers developed at 750 and 800 °C (Fig. 1a and b) such contrast differences were not identified. On the XRD patterns of the samples borided at 750 and 800 °C, the peaks of the Fe<sub>2</sub>B phase were detected (Fig. 2a and b), belonging to the homogeneously bright etched boride layers shown in Fig. 1a and b. However, on the XRD patterns of the samples borided at 850 and 900 °C only FeB peaks were present (Fig. 2c and d). This



Fig. 2. XRD patterns of the 4140 steel borided at: (a) 750 °C, (b) 800 °C, (c) 850 °C, and (d) 900 °C for 12 h.



Fig. 1. Cross-section LOM micrographs of 4140 steel borided at (a) 750 °C, (b) 800 °C, (c) 850 °C, and (d) 900 °C for 12 h.

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