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The growth of single Fe_2B phase on low carbon steel via phase homogenization in electrochemical boriding (PHEB)

G. Kartal [a,*], S. Timur [a], V. Sista [b], O.L. Eryilmaz [b], A. Erdemir [b]

[a] *Istanbul Technical University, Department of Metallurgical and Materials Engineering, Istanbul, Turkey*
[b] *Argonne National Laboratory, Energy Systems Division, Argonne, IL, USA*

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ABSTRACT

In this study, we introduce a new electrochemical boriding method that results in the formation of a single-phase Fe_2B layer on low carbon steel substrates. Although FeB phase is much harder and more common than Fe_2B in all types of boriding operations, it has very poor fracture toughness; hence, it can fracture or delaminate easily from the surface under high normal or tangential loading. We call the new method "phase homogenization in electrochemical boriding" (PHEB), in which carbon steel samples undergo electrochemical boriding for about 15 min at 950 °C in a molten electrolyte consisting of 90% borax and 10% sodium carbonate, then after the electrical power to the electrodes is stopped, the samples are left in the bath for an additional 45 min without any polarization. The typical current density during the electrochemical boriding is about 200 mA/cm^2. The total original thickness of the resultant boride layer after 15 min boriding was about 60 μm (consisting of 20 μm FeB layer and 40 μm Fe_2B layer); however, during the additional phase homogenization period of 45 min, the thickness of the boride layer increased to 75 μm and consisted of only Fe_2B phase, as confirmed by glancing-angle x-ray diffraction and scanning electron microscopy in backscattering mode. The microscopic characterization of the boride layers revealed a dense, homogeneous, thick boride layer with microhardness of about 16 GPa. The fracture behavior and adhesion of the boride layer were evaluated by the Daimler-Benz Rockwell C test and found to be excellent, i.e., consistent with an HF1 rating.

1. Introduction

Boriding is a promising thermochemical surface hardening treatment applied to a wide range of engineering components to improve their wear, oxidation, and corrosion resistance [1–3]. Boriding of ferrous materials is a common practice and has been in use since the early1900s [1]. Since then, countless papers have been published on the topic, but we only cite a few key ones in this paper. During electrochemical boriding, boron atoms are extracted from the electrolyte and then deposited onto the surface of steel substrates. Due to their small size and high mobility, these boron atoms diffuse into substrates, and some of them react with substrate atoms and form iron borides. Depending on the specific characteristics of the boriding experiments (such as type of powder mixture, temperature, time, and current density) and chemical composition of the base steel, a single or duplex layer may form, for instance, FeB and/or Fe_2B with low alloy steels. In the case of titanium, the boride layers consist of TiB_2 and TiB phases [4,5].

These dual-phase boride layers offer some tribological advantages due to a gradually declining hardness profile from the surface through the boride layer and well into the substrate. However, as seen in the examples of steel boriding, the higher boron content phase (FeB) on top may not be ideal for mechanical and tribological applications [1,2]. First of all, despite being very hard, the FeB top layer is very brittle and has a substantially different coefficient of thermal expansion. While cooling down after the boriding treatment, high tensile stresses develop in the FeB phase while compressive stresses form in Fe_2B; hence, borided steels with high FeB content develop extensive micro and macro cracks parallel to the borided surface layers [6–9], as shown in Fig. 1 (a and b). Additionally, the brittleness of FeB layers leads to severe flaking and spalling when a high normal or tangential load is applied, as shown in Fig. 1(c). Consequently, a single Fe_2B boride layer is more desirable than a dual FeB–Fe_2B layer.

The preferred methods to prevent FeB growth are (1) diluting the boron concentration by pack boriding of powder mixtures [1,10], (2) applying a much thinner boriding agent [11–13], or (3) working at high boriding temperatures for long enough to transform FeB into

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[*] Corresponding author. Tel.: + 90 212 285 35 45; fax: + 90 212 285 34 27.
E-mail address: kartalgu@itu.edu.tr (G. Kartal).

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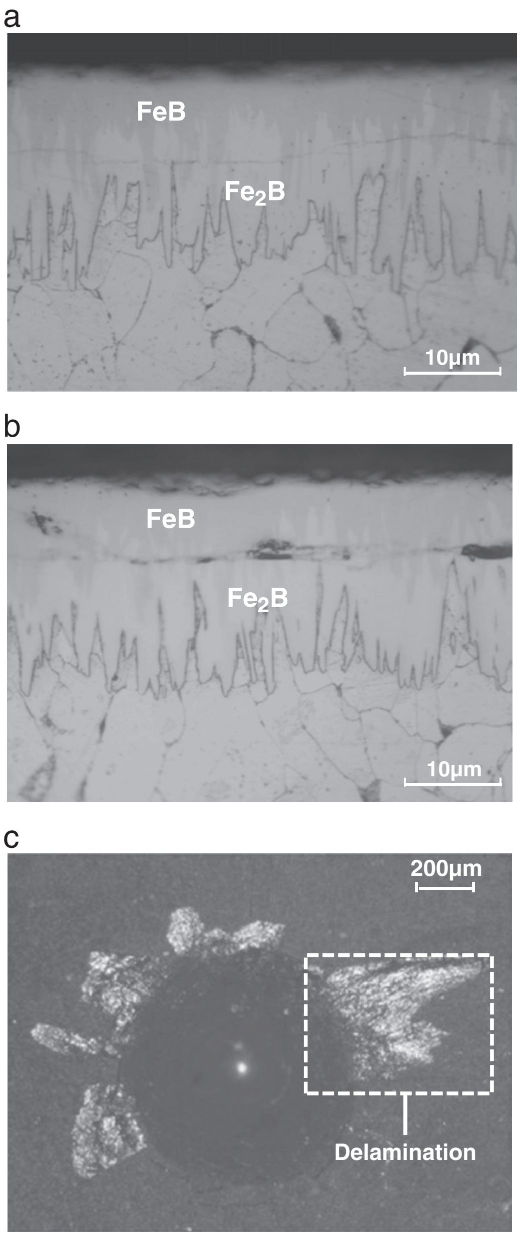

Fig. 1. Potential failure of dual-phase boride layers: (a) crack formation at the interface of FeB and Fe_2B layers, (b) splitting of FeB layer, and (c) spalling of boride layer under loading conditions. The boride layer formed after 30 min electrochemical boriding at 950 °C and 200 mA/cm^2.

Fe_2B phase [1,13–15]. Alternatively, Gopalakrishnan et al. [16] suggested use of thermal cycling in the pack or liquid boriding ("interrupted boriding") to hinder FeB phase formation. Even though they grew a single Fe_2B boride layer on a steel substrate, the proposed process requires a long duration (4 or 5 h) and extra safety precautions because either a pack boriding box must be taken in and out of a furnace 4 or 5 times during the pack boriding, or the sample must be removed from a liquid boriding bath. Another promising approach to reduce the amount of highly brittle FeB phase is post-laser heat treatment after the boriding process. However, this treatment not only alters the structure and chemistry of the boride layer but also significantly decreases the surface hardness of the boride layer from 1800 HV to around 900 HV on top [17,18].

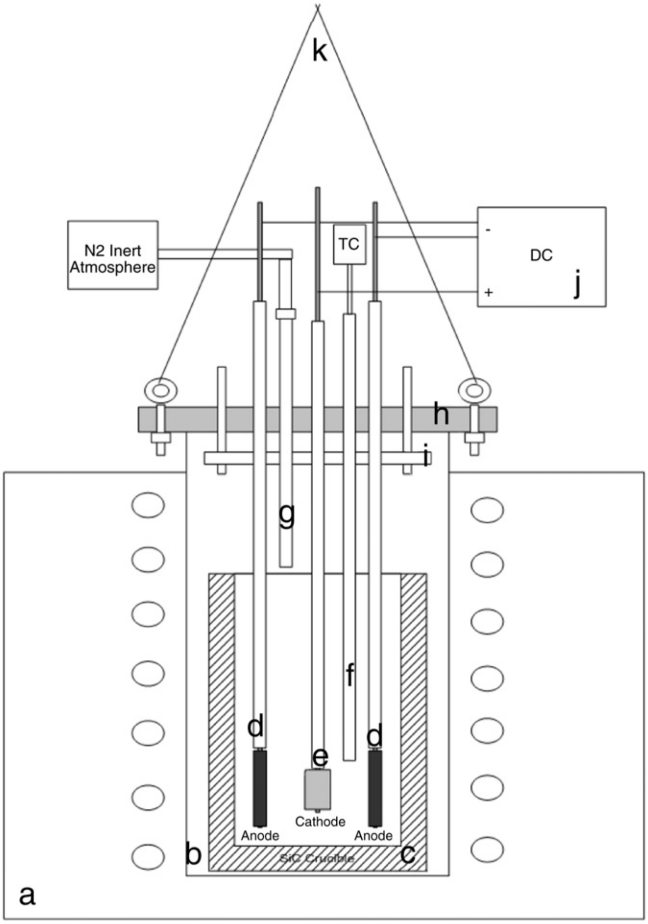

Fig. 2. Schematic illustration of electrochemical cell: (a) electric furnace, (b) alumina tube, (c) SiC crucible, (d) graphite anode and stainless steel rod with alumina tube shield, (e) cathode and stainless steel rod with alumina tube shield, (f) thermocouple with SiC tube shield, (g) N_2 purge line, (h) water cooled aluminum cap, (i) alumina plate, (j) DC power supply, and (k) crane sling.

In this study, we developed a new method that can deposit a single-phase Fe_2B layer on steel substrates in a fast, safe, and green fashion. We call the new process "phase homogenization in electrochemical boriding" (PHEB), where electrochemical boriding is combined with a phase homogenization treatment in the same boriding bath without any extra effort. The single Fe_2B phase was inspected by glancing-angle x-ray diffraction (XRD) and scanning electron microscopy (SEM) in backscattering mode. Additionally, Vickers micro-hardness examination and the Daimler-Benz Rockwell C test were carried out to determine the hardness variation and the fracture behavior, as well as adhesion of the new boride layers.

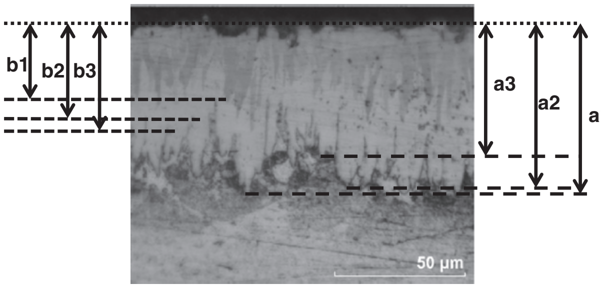

Fig. 3. Typical boride layer formed after 15 min EB: a and b represent peaks and valleys of the total boride layer, respectively.