



Electrochemical boriding and characterization of AISI D2 tool steel

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ABSTRACT

D2 is an air-hardening tool steel and due to its high chromium content provides very good protection against wear and oxidation, especially at elevated temperatures. Boriding of D2 steel can further enhance its surface mechanical and tribological properties. Unfortunately, it has been very difficult to achieve a very dense and uniformly thick boride layers on D2 steel using traditional boriding processes. In an attempt to overcome such a deficiency, we explored the suitability and potential usefulness of electrochemical boriding for achieving thick and hard boride layers on this tool steel in a molten borax electrolyte at 850, 900, 950 and 1000 °C for durations ranging from 15 min to 1 h. The microstructural characterization and phase analysis of the resultant boride layers were performed using optical, scanning electron microscopy and X-ray diffraction methods. Our studies have confirmed that a single phase Fe₂B layer or a composite layer consisting of FeB + Fe₂B is feasible on the surface of D2 steel depending on the length of boriding time. The boride layers formed after shorter durations (i.e., 15 min) mainly consisted of Fe₂B phase and was about 30 μm thick. The thickness of the layer formed in 60 min was about 60 μm and composed mainly of FeB and Fe₂B. The cross sectional micro-hardness values of the boride layers varied between 14 and 22 GPa, depending on the phase composition.

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1. Introduction

AISI D2 and other D class tool steels are high carbon and chromium-containing alloyed steels which are used extensively in a variety of machining and manufacturing operations. They have the ability to retain their desirable mechanical properties intact upon cycling over a range of temperatures, hence D2 and other group D steels are important for demanding metal-forming and manufacturing operations. Some of the typical industrial applications of D2 steel include punches, piercing and blanking dies, spinning tools, shear blades, slitting cutters, as well as a variety of higher-end wood working tools [1–4].

Application of a variety of hard coatings on D2 steel can enhance its wear resistance further. At present, many different types of thin hard coatings (mainly Ti-based coatings) are applied on tools and dies made from D2 steels by chemical vapor deposition (CVD) [5], physical vapor deposition (PVD) [6,7], and plasma assisted chemical vapor deposition (PACVD) techniques. However, most of these techniques may require up to 12 h of processing time to achieve sufficiently thick (i.e., 5 to 10 μm) hard coatings needed for good performance long durability in machining and metal-forming applications.

Among the many other surface treatment processes, boriding, offers high potential to achieve very thick and hard boride layers [8] with good resistance to wear and oxidation [3,9–12]. Since it is not an add-on

coating, problems associated with adhesion may not be a major concern either. Boriding could be achieved in various ways: powder pack boriding, molten salt boriding, gas boriding, fluidized bed boriding plasma boriding and electrochemical boriding, in which electrochemical boriding offers the fastest generation of desired boride phases in shortest time [8–28]. In all these boriding processes, boron atoms are extracted or reduced from the boron-rich media and deposited on the surfaces of the substrates (to be borided). Subsequently, the diffusion of boron atoms from the surface into the substrate results in a variety of boride phases especially FeB and Fe₂B in the case of iron [12]. The growth rate of boride layer obeys a parabolic law with the following equation.

$$d^2 = Kt \quad (1)$$

where d is the thickness of the boride layer, K is the growth rate constant and t is the duration for the boriding process. The growth rate constant (K) depends on the diffusion of boron and the temperature according to the Arrhenius equation (Eq.(2))

$$K = K_0 \exp\left(\frac{-Q}{RT}\right) \quad (2)$$

where K_0 is pre-exponential constant, Q is the activation energy, T is the temperature and R is the universal gas constant. In particular, boriding has been very successfully employed in low-carbon steels for a variety of applications.

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Boriding of high carbon steels with high alloy content (like AISI D2) has been somewhat difficult in the past. Casteletti et al. performed molten salt boriding of AISI D2 steel with borax ($\text{Na}_2\text{B}_4\text{O}_7$) as the main active ingredient and ferrotitanium and aluminum as the reducing agents at 800 °C and 1000 °C for 4 h. They reported maximum thickness of around 40 μm [24]. In the case of conventional pack boriding process, the thickness attained was around 45 μm and it took about 8 h [25]. In general, the diffusion of boron into these steels has been greatly suppressed mainly because of the secondary carbide phases acting as diffusion barriers.

In this study, we explored the possibility of faster and more efficient boriding of AISI D2 steel using an electrochemical process. To the best of our knowledge, this is the first attempt on the electrochemical boriding of AISI D2 steel as there are no published papers or reports that we could find in open literature. This study also reveals the fact that although thin hard coatings on AISI D2 steels are useful for many industrial applications, their service life could be further improved by a prior electrochemical boriding step. But in this study, we mainly concentrated on electrochemical boriding of D2 steel in order to achieve thick and hard boride layers in very short process durations. Based on the results of our study, we can state that electrochemical boriding of D2 is feasible, and the process is fast, cheap and clean since it produces no gaseous emissions or solid wastes. Characterization of borided D2 steel samples reveals uniform 30–60 μm thick boride layers in 15–60 min durations respectively.

2. Experimental

AISI D2 steel plates (whose nominal composition was C 1.48, Cr 11.91, Mo 0.98, V 0.76, Si 0.96, Mn 0.45 wt.% and balance Fe) were obtained from commercial sources and cut into smaller rectangular test pieces to fit into our electrochemical boriding unit. All samples were progressively ground and polished down to 1200 grit size using a series of SiC emery papers to remove the oxide layers and other contaminants from the surface. The electrochemical boriding experiments were carried out in molten borax ($\text{Na}_2\text{B}_4\text{O}_7$) at 850, 900, 950 and 1000 °C and at a current density of 200 mA/cm^2 . Samples were attached to the cathode holder while two graphite plates served as

anodes. The SiC crucible which contained the electrolyte was externally heated by electrical heating elements in a furnace. Before being attached to the cathode holder of the electrochemical cell, the samples were ultrasonically cleaned in acetone and blow-dried.

The process duration varied between 15 and 60 min. The molten bath was purged with dry nitrogen in order to protect electrodes and crucible from oxidation and corrosive attack of the molten electrolyte. Electrolysis was conducted by a direct current source. The temperature of the electrolyte was measured by a K-type thermocouple placed in a Hexaloy tube. Cell potential and temperature were recorded and monitored by a data acquisition system during the electrolysis.

After each experiment, borided D2 steel samples were withdrawn from the electrolyte and left for air-cooling. The residual electrolyte left on the surface was cleaned by washing the samples in boiling water. The borided samples were then cross sectioned and polished to mirror finish for further characterization. The boride layer thickness and morphology were investigated in cross-section using an Olympus STM6 optical microscope. Scanning electron microscope with back scattered electron imaging (BEI) was also performed by using FEI Quanta 400 F to determine the contrast difference between the phases of boride layers. X-ray diffraction studies used a Philips Xpert2 diffractometer with $\text{Cu K}\alpha$ radiation of wavelength 1.54 Å. Both regular 2θ - ω and glancing angle scans were carried out to determine the phases of the boride layers. Microhardness measurements were performed using Wilson/Tukon equipment (Model 300) under a 100 g load. We also used Rockwell C equipment with 150 kg load to obtain information on the adhesion of boride layers according to a protocol developed by Daimler-Benz.

3. Results and discussion

3.1. Thickness and morphology

The cross sectional morphologies of boride layers formed on AISI D2 steel were examined by both optical microscopy and SEM (in back scattering mode). Fig. 1 shows the optical images of the boride layers formed at different process durations. As is obvious, the thickness of the resultant boride layers increases with boriding time. In these optical

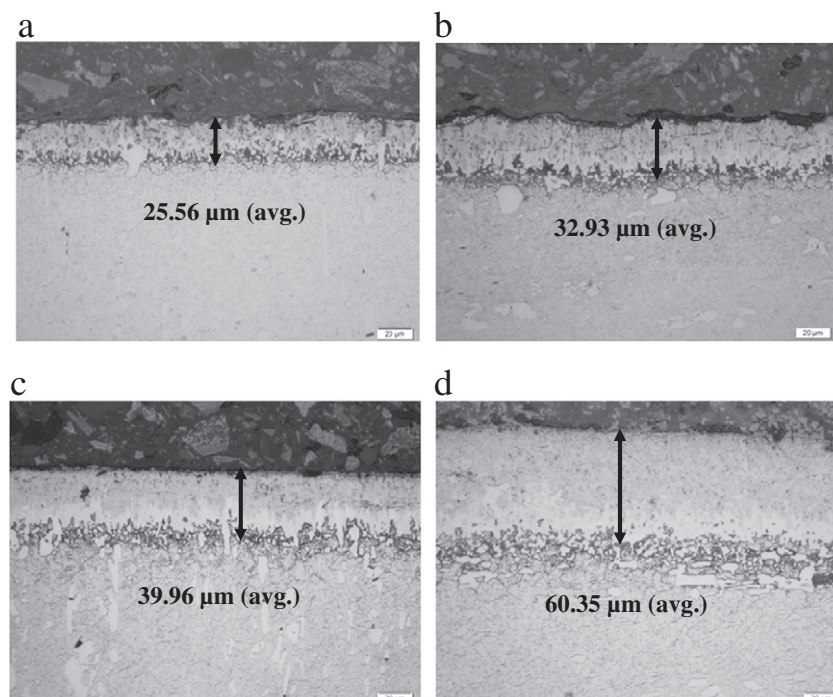


Fig. 1. Optical micrographs of boride layers formed at 950 °C for different boriding durations (a) 15 min (b) 30 min (c) 45 min (d) 60 min.

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