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Characterization and determination of Fe_xB layers' mechanical properties

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ABSTRACT

The aim of this study is to obtain microstructural characteristics and investigate the mechanical properties such as hardness, Young's modulus and fracture toughness of the boride layer depending on process time and temperature. The produced double layers (FeB and Fe_2B) were extensively analyzed with respect to X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRD pattern of the boride layers, which were formed on SAE 1020 and 1040 quality steel at 900 °C for 2 h, 4 h and 6 h, include only FeB phase on surface with (1 1 1), (2 1 0), (1 0 1) and (1 1 1) planes. SEM cross-sectional investigations show that double-phase boride layer existence from surface to inside of substrate. The structural compositions of layers consist of boron rich phase (FeB) and iron rich phase (Fe_2B), respectively. Surface roughness value of samples is important parameter for micro-indentation test with Dynamic Ultra Micro Hardness Tester. After diffusion controlled boriding process, material surface roughness can be high for micro-indentation test. So, surface polishing process is applied for decreasing roughness of FeB layers. Surface roughness values of 2 h, 4 h and 6 h borided SAE 1020 and SAE 1040 quality steel were decreased from 0.9 μm to 0.05 μm by polishing process. Mechanical properties of layers were examined by Shimadzu Dynamic Ultra-microhardness test machine for estimating Young's modulus due to load–unload sensing analysis and in addition to mechanical investigation hardness–depth curves of the layer were obtained for estimating indentation depth and load dependency of mechanical properties. Load depended elastic modulus (125–624 GPa) and hardness (17–33 GPa) were obtained at 80 mN, 160 mN, 320 mN and 640 mN applied peak loads depending on boriding process time. Fracture toughness properties of FeB surface layers were calculated by Vickers Fracture Toughness method for 1 N applied peak load with measuring crack length after loading stage was finished.

1. Introduction

Serviceable engineering components not only rely on their bulk material properties but also on the design and characteristics of their surface. This is especially true in high hardness components, as their surfaces must perform many industrial functions in a variety of complex environments. The surface of industrial component may require treatment to enhance the surface characteristic. Surface treatments that cause microstructure changes in the bulk material include heating

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and cooling/quenching through induction, flame, laser and electron beam techniques, or mechanical treatments (Celik et al., 2006).

One of the thermo chemical surface treatments of steel-based material is boriding process. Boriding (or boronizing) is an important thermo chemical treatment applied to enhance the surface hardness and wear resistance of ferrous and non-ferrous alloy components (Yu et al., 2005; Sinha et al., 1991; Chatterjee-Fisher, 1989). During boriding process, the diffusion and subsequent absorption of boron atoms into metallic lattice of component surface form interstitial boron compounds (Graf von Matuschka, 1980). The resulting layer may consist of either a single-phase boride (FeB or Fe_2B) or polyphase boride layer (FeB and Fe_2B). The mechanical properties variations of FeB and Fe_2B layers depending on process time and temperature are important for surface treatment application. In addition to this, the microhardness of the boride layer also depends strongly on the composition and structure of the boride layer and composition of the based material. The pack boriding has the advantages of simplicity and cost-effectiveness in comparison with other boriding processes. In this technique, the boriding agent in powder form is placed into a heat resistant box and samples are embedded into this powder under inert gas atmosphere. At the end of boriding time, the box is cooled at room temperature and then, dust over the samples is removed (Keddam and Chentouf, 2005). In this study, prepared samples were borided by pack boriding process, for 2 h, 4 h and 6 h at 900 °C Pack boriding process was applied to SAE 1020 and SAE 1040 quality low-alloy steel substrate for achieving advantages such as:

- Hardness of the boride layer can be retained at higher temperatures than, for example, that of nitrided cases.
- A wide variety of steels, including through-hardenable steels, are compatible with the processes.
- Boriding, which can considerably enhance the corrosion–erosion resistance of ferrous materials in nonoxidizing dilute acids and alkali media, is increasingly used to this advantage in many industrial applications.
- Borided surfaces have moderate oxidation resistance (up to 850) and are quite resistant to attack by molten metals.
- Borided parts have an increased fatigue life and service performance under oxidizing and corrosive and corrosive environment (Sinha et al., 1991).

As known from previous studies (Yao, 2005; Laursen and Simo, 1992), the qualities of small size and non-destructive test capability make the indentation technique superior to the tension test for determination of mechanical properties of surface coatings, films and layers. For very small volumes of material, the uniaxial test is inapplicable. Furthermore, the structural materials may not be removed to do the tension test in most cases, for instance, the materials used in the electronic solders or engineering welds. Indentation technique can evaluate the material properties while keeping the structural integrity (Yao, 2005). The advantage of the indentation test, in comparison with a uniaxial tensile test, is of course the relative simplicity of the experimental setup. On the other hand, an obvious drawback is the very complicated mechanical problem arising owing to inelastic and/or inhomogeneous deformation in the indented materials. Therefore, until recently the interpretation of indentation tests have relied heavily on semi-empirical formulae. The work by Tabor (Tabor, 1951) is perhaps the best example of this, with no or little theoretical foundation. With the advent of modern computers and advanced numerical methods, however, the understanding of the mechanics involved during ball indentation (Hill et al., 1989; Kral et al., 1993; Giannakopoulos et al., 1994), cone indentation (Laursen and Simo, 1992) and Vickers Indentation (Giannakopoulos and Suresh, 1999), has increased rapidly in recent years.

Regarding mechanical properties, hardness testing provides useful information on the strength and deformative characteristics of the materials (elastic modulus, elastic recovery hardness, etc.). Hardness is a mechanical parameter which is strongly related to the structure and composition of solids. Hence, microhardness is not only a mechanical characteristic routinely measured but it has also been developed as an investigation method of structural parameters in recent years. Therefore, hardness experiments have become more and more important to characterize a material (Giannakopoulos and Suresh, 1999; Uzun et al., 2005; Elmustafa and Stone, 2002; Atar et al., 2003).

The characteristic ability of a material to resist penetration of an indenter allows evaluation of a parameter that we know hardness. The indentation hardness of materials is measured in several ways by forcing an indenter having specific geometry (ball, cone, and pyramid) into the specimens' surface (Gogotski et al., 1999). The conventional microhardness value can be determined from the optical measurement of the residual impression left behind upon load release. In recent decades, the development of depth-sensing indentation equipment has allowed the easy and reliable determination of two of the most commonly measured mechanical properties of materials, the hardness and Young's modulus (Zhu and Bartos, 2000). The depth-sensing (or dynamic) micro-indentation method offers great advantages over the conventional Vickers microhardness testing in two aspects. First, apart from microhardness (or microstrength), the method can also provide well-defined mechanical parameters such as elastic modulus of the interfacial zone. Secondly, as load and depth of an indentation are continuously monitored, optical observation and measurement of diagonal length of the indent/impression, which can be difficult and subjected to inaccuracy, is no longer required (Uzun et al., 2005).

According to the previous study, mechanical properties of boride layer consisted of fracture toughness and hardness determinations were studied (Atar et al., 2003; Sen et al., 2001, 2005; Ozbek and Bindal, 2002; Sen and Sen, 2003) but indentation study of FeB layers were not investigated in details depending on applied load, process time and temperature. In this study, it was aimed to examine the dynamical hardness measurements of boride layer to determine modulus and hardness values under different applied peak loads and show the load and indentation size dependency of the hardness and modulus. In addition to mechanical investigation of coatings, films and layers; fracture toughness determination with Vickers Hardness Indentation was proposed by Evans and Charles (1976) and later extended and modified by Niihara