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Multi-scale wear of a boride coating on tungsten

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Abstract

In this work, wear mechanisms of boride coating on pure tungsten using a pin-on-disk tribometer were investigated. The surface analysis after wear tests was conducted using an atomic force microscope (AFM). Research results showed that the boride coating underwent different types of wear modes: abrasion within a length scale from micrometer to millimeter and fracture within a few nanometers. Tungsten showed wear through plastic deformation and adhesion. These mixed wear modes could only be seen under the atomic force microscope.

Keywords: Multi-scale wear; Atomic force microscopy (AFM); Thin film; Tungsten boride coatings

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1. Introduction

Boride coatings have been applied to metal surfaces in order to improve their corrosion resistance, electrochemical properties, tribological performance, and to prolong service life [1]. Efforts have been made in studying material properties of boronized steels. Anthymidis et al. [2] did a comparative study of boride coatings on a steel substrate obtained by the pack-cementation method and by fluidized bed technology. Pack-cementation is an in situ self-generated chemical vapor deposition (CVD) coating technology. In the fluidized bed method, the part to be coated is preheated in an oven and dipped into the fluidized bed of the target powder. Powder particles melt as they come in contact with the hot parts, and solidify, forming a uniform coating. Coatings were characterized as good adherence and only one-phase belonging to Fe_2B was formed during the treatments. The pack-cementation procedure resulted in coatings with residual stresses and preferred orientation, while the coatings produced by fluidized bed were characterized as strain-free grains with random distribution. Xu et al. [3] compared superplastic and conventional boronizing. They reported that superplastic boronizing retarded the formation of high boron containing boride phases and reduced the growth texture of the borides. With superplastic boronizing (0 0 1) FeB growth texture was eliminated and (0 0 1) Fe_2B growth texture was reduced significantly. In the same report, researchers noted that superplastic boronizing processes suppressed the formation of Si-rich zones which otherwise would have formed between boride grains. The microhardness of the boride layer processed by superplastic boronizing was more uniform than that produced by conventional boronizing. Palombarini et al. [4] studied the role of oxygen in iron boronizing. Their results indicated that the residual gaseous oxygen in the reaction atmosphere in boronizing of Armco iron in B_4C–KBF_4–SiC powder mixtures was detrimental. It reacted with boron to form gaseous boron oxides rather than reacting with carbon in B_4C to liberate active boron for the process. The presence of oxygen from powder mixtures containing Fe_2O_3, Fe_3O_4, or SiO_2 also lead to a decrease of B_4C reactivity as shown by a lower production of surface Fe_2B. Surface structure, texture and growth of boride coatings on Armco iron and Fe–C-1.26% Cr was studied by SEM and X-ray diffraction [5]. Single-phase Fe_2B

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and two-phase FeB/Fe_2B layer structures were examined. It was found that there is no simple relation between texture and interphase morphology. The columnar morphology of Fe_2B on the substrate interface was explained by a tip-enhanced growth mechanism. FeB was a hard phase growing in hard Fe_2B matrix, whereas Fe_2B grew in the ductile substrate matrix. This resulted in different local stresses and lattice distortions at the interface. Rai et al. used an atomic force microscope (AFM) to analyze the surface of the Ni–B coatings treated in different processes. It was found that the microhardness was related to the surface topography of coating and the types of borides, which emerged in deposit. The authors observed nanometer sized protrusions of 20–40 nm in height [6]. Not much work has been done on boronizing of refractory metals. Boronizing is a thermo-chemical diffusion surface treatment in which boron atoms diffuse into the surface of the work piece to form hard borides with the base materials [7].

Wear mechanisms of boronized steels have been reported. In a previous study, formation and self-lubricating mechanisms of thin boron oxide and boric acid films on surfaces of boronized steel were reported. The ultra-low friction behavior of boronized steel surfaces was due to the layer–lattice structures of these films [8–12]. Martini et al. [13] studied the sliding and abrasive wear of boronized coating on steel. He found that there was a difference in values of surface height in different regions of the coatings. The difference was due to various crystallographic orders of iron borides. The wear resistance was initially poor when there was a thin, friable layer constituting disordered crystals. The wear resistance increased to a maximum value in regions with ordered Fe_2B crystals.

The motivation of this research is to understand the most fundamental tribological issues of boride coatings and their associated nonferrous substrates. To date, there is not much literature available on the tribological properties of boronized nonferrous metals in general and boronized refractory metals in particular. To investigate wear mechanisms, a pin-on-disk type tribometer and an atomic force microscope (AFM) were utilized. Boride coating on tungsten substrate was the main focus of this work. Tungsten is one of the hardest elements in the periodic table. With a boronized layer, its tribological applications will widen.

2. Experimental details

2.1. Materials

Annealed polycrystalline tungsten substrate (99.95% pure) was obtained from Alfa Aeser®. It has body-centered cubic crystal structure. Cylindrical samples (size of 6.3 mm diameter and 6 mm high) to be coated were placed in contact with Ekabor powder, which has the composition of 90 wt% SiC, 5 wt% B_4C and 5 wt% KBF_4. The coatings were produced by heating samples to 940 °C in a resistance heating furnace for different duration of time of 2, 4, 6 and 8 h in the presence of Ekabor powder. Samples were then air-cooled.

Surface roughness is defined as the finer irregularities of the surface texture that usually result from the inherent action of some production processes. The surface roughness of the coating was studied by using the Talysurf profilometer (Taylor Hobson) with diamond tip diameter of 5 μm. The scan length was 4 mm. The tests were conducted at three different places on the surface of the coated samples. Reported Ra value is the average of the three values with standard deviation.

2.2. Friction and wear tests

Tribological experiments were carried out using a reciprocal pin-on-disk tribometer (CETR Micro-Tribometer Model UMT-2). In this equipment the bottom part is movable and the top pin or ball stays stationary. In the present study, the bottom movable flat surface was the boronized tungsten and upper fixed surface was the steel ball with diameter of 6 mm. The cylindrical samples (size of 6.3 mm diameter and 6 mm high) were used for tribological study. The wear tests were conducted on the flat surface of the cylinders. The reciprocating amplitude was 4 mm which formed a wear track of the same length. The sample surfaces were first cleaned using an ultrasonic cleaner in acetone bath for 10 min to remove physically bonded foreign particles and dust.

The tribometer monitors the actual dynamic normal load, frictional force, coefficient of friction, and the pin height reduction during the test. The tests were conducted at an initial applied load of 2 N for 120 s at 100 rpm. After the friction stabilized, the applied load was increased to 5 N for 3600 s at 100 rpm at room temperature (about 20 °C). The relative humidity of the room was 55–60%. The surface heights and the coefficient of friction were calculated. The test procedure was similar to a previous report [14].

2.3. Surface characterization

Metallographic observation was conducted using a scanning electron microscope (SEM) to observe the morphology of borides formed on the substrate. Borides were characterized by X-ray diffraction with a Rigaku X-ray diffractometer. The microhardness of boronized tungsten and underlined substrate along the cross section were measured by an Instron microhardness tester attached with a Vickers diamond indenter at a constant load of 100 g (0.98 N). Samples were prepared by using a conventional metallographic technique before the measurements. According to the W–B phase diagram, there are four different types of tungsten borides: W_2B_5, WB, WB_4, and W_2B. They are all nonstoichiometric intermetallic compounds that have a range of compositions.

The surface properties and wear mechanisms were investigated using surface characterization techniques. The finer scale analysis of wear mechanisms was conducted using an atomic force microscope (AFM) (Pacific Nanotech Inc.