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Tribological investigation of tantalum boride coating under dry and simulated body fluid conditions

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Abstract

Tantalum is known to be a highly corrosion resistant refractory metal. Boronized tantalum has potential use in machinery components, structures and as a bearing material in joint implants due to its high corrosive resistance and hardness. In this research, the friction and wear characteristics of boronized tantalum were studied against bearing steel E52100 under dry and simulated body fluid (SBF) conditions. Results showed that SBF caused tribochemical reactions leading to an increase in friction corresponding with amorphous debris. Fatigue cracks formed under dry conditions and tribochemical wear cracks formed under SBF conditions.

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1. Introduction

Tantalum is a refractory, transition metal found to be inert to body fluids [1,2]. It has been reported to incite satisfactory local host tissue response [3,4]. Tantalum is a candidate for heavy load bearing skeletal implants [5,6]. Black [7] has reported that tantalum causes vital encapsulation in soft tissue and osteointegration, similar to titanium, in hard tissue. Tantalum therefore has the potential of being considered as a material in joint repair applications.

According to Koleske [8], coatings applied to surfaces have the ability to modify their static and dynamic mechanical properties, flexibility, toughness, adhesion, hardness, abrasion resistance, slip, chemical resistance and stresses. Friction and wear have to be minimized and lubrication maintained, in order to decrease the chances of tribological failure of interacting mechanical components [9]. Several authors have studied the mechanisms of friction and wear on pure metals, as well as on coated surfaces [10].

Boronizing is a surface treatment, thermochemical process, which involves the diffusion of boron into a base metal at high temperatures (815–980°C) to form hard boride coating layers [11–14]. Several authors have studied the effects of boronizing metal surfaces [15,16]. Boronizing of metals has been found to form a metallic boride surface offering high hardness and resistance to acid corrosion [16]. Pure, cold worked tantalum has a hardness value of 200 kg/mm² [17] whereas tantalum boride has been found to have a hardness value in excess of 2000 kg/mm² [18].

Bearing steel has a variety of applications [19–22]. For tribological testing, bearing steel is often used as a standard material. We therefore used this material in the present work [23]. The main cause of artificial joint failure is the effects of wear debris particles from the implants [24]. These particles incite a negative immune response from the body leading to osteolysis. Understanding the debris formation mechanisms could provide information to improve an implant's performance and lifespan. Although the metallic components are usually articulated against a softer polymer in joint replacements, sometimes, chips and pieces of bone and bone cement can cause considerable damage to the metal components due to their hardness. In this study, we investigate the tribological properties of boronized

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tantalum against a relatively hard material, bearing steel E52100.

We conduct tribological tests of dry tantalum boride against bearing steel E52100 to study its basic tribological properties against a ferrous metal. These tests were also carried out in simulated body fluid (SBF) as a lubricant. The SBF is similar in composition to blood plasma [25]. This test gave us information on the tribochemical behavior of tantalum boride coating if used as an implant material and as a machinery component working in a corrosive environment (e.g., in sea conditions).

2. Materials

Pure tantalum was the substrate material with purity 99.9%. The sample was in the shape of a cylinder with diameter approximately 5 mm and thickness 2.7 mm. Boronizing was performed in a solid medium using Ekabor powders that had grain sizes less than 850 μm and had a nominal chemical composition of 90% SiC, 5% B₄C and 5% KBF₄. The pure tantalum test specimen was placed in contact with Ekabor powders and then transferred to an electrical resistance furnace in a stainless steel crucible with a diameter of 5 cm and height 8 cm. It was then heated from room temperature to 940 $^{\circ}$ C in 50 min under atmospheric pressure and held in the furnace for 4 h. This was followed by cooling in air at atmospheric temperature and pressure.

The counter pin was bearing steel E52100 (0.98–1.10% C, 0.2–0.35% Si, 0.25–0.45% Mn, \leq 0.025% P, \leq 0.025% S, 0.9–1.15% Cr, \leq 0.25% Ni and \leq 0.08% Mo). It was in the form of a sphere 6 mm in diameter. It was made of a martensitic AISI 52100 steel, quenched at a hardness value of approximately 60 HRC.

The SBF was originally developed by Kokubo [25]. It was buffered at a pH of 7.25 with Tris(hydroxymethyl) aminomethane and HCl. The ionic concentrations in SBF are given in Table 1 [26]. The method of preparation of SBF is provided below [26].

1. Dissolution of chemicals

- (i) Using a 100 ml beaker, heat 700 ml of ion-exchange water to 36.5 °C, stirring continuously with a magnetic stirrer.
- (ii) Combine the first nine chemicals from Table 2, according to the amounts shown.

2. Adjustment of pH

(i) Calibrate a pH meter with fresh standard buffer solutions.

Table 1 Relative ionic concentrations in SBF

Ion	Concentration (mM)	
Na ⁺ K ⁺ Mg ²⁺ Ca ²⁺	142.0	
K ⁺	5.0	
Mg^{2+}	1.5	
Ca ²⁺	2.5	
Cl-	147.8	
HCO ³⁻	4.2	
HPO^{4-}	1.0	
HPO ⁴⁻ SO ₄ ²⁻	0.5	

Table 2 Chemicals included in SBF

Chemical	Quantity	
NaCl (g)	7.996	
NaHCO ₃ (g)	0.35	
KCl (g)	0.22	
$K_2HPO_4(g)$	0.174	
$MgCl_2 \cdot 2H_2O(g)$	0.305	
1 M HCl aqueous solution (ml)	~40	
CaCl ₂ ·2H ₂ O (g)	0.368	
$Na_2SO_4(g)$	0.071	
Tris(hydroxymethyl)aminomethane (g)	6.057	
1 M HCl aqueous solution (ml)	~10	

- (ii) After the first nine chemicals are added, confirm that the temperature is at 36.5 °C and titrate the 1 M HCl solution with a pipette to adjust the pH to 7.25.
- (iii) After adjusting the pH, transfer the solution to a 1000 ml volumetric flask.
- (iv) Add ion-exchange water to the flask until the total volume is 1000 ml and allow the solution to cool to room temperature. Once cooled, add more ion-exchanged water to achieve 1000 ml of solution.
- (v) The SBF should be stored in a refrigerator at 5-10 °C.

Before the tribological tests started, a drop of SBF was applied to the tantalum boride surface using a clean pipette.

3. Experiments

3.1. Boride layer characterization

The microhardness was measured on boronized Ta and substrate Ta using a micro-diamond indenter equipped with a Zeiss optical microscope. The applied load was 10 gm and the test duration was 5 s. Before measurements, samples were polished to a roughness in the submicron range.

3.2. Tribological tests

The tribological tests were conducted using a CSM instruments pin-on-flat tribometer. Prior to testing, the pin and flat specimens were cleaned in acetone. E52100 was used as the pin. The test specimen (boronized tantalum) was mounted on the tribometer so that its upper surface was in a horizontal position. The sliding was a linear reciprocating motion at 2.5 cm/s. The amplitude was 1 mm. A normal load of 5 N was applied. The instrument measured the tangential force between the two contacting surfaces and the coefficient of friction was calculated as the ratio of the tangential force to the normal load by the software TriboX2.7 (CSM Instruments). The tests were conducted for 1.5 h so that enough information of wear debris and wear mechanisms could be collected.

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