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Characteristics and wear behavior of cenosphere dispersed titanium matrix composite developed by powder metallurgy route

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Abstract: The cenosphere dispersed Ti matrix composite was fabricated by powder metallurgy route, and its wear and corrosion behaviors were investigated. The results show that the microstructure of the fabricated composite consists of dispersion of hollow cenosphere particles in α-Ti matrix. The average pore diameter varies from 50 to 150 μm. The presence of porosities is attributed to the damage of cenosphere particles due to the application of load during compaction as well as to the hollow nature of cenospheres. A detailed X-ray diffraction profile of the composites shows the presence of Al_2O_3, SiO_2, TiO_2 and α-Ti. The average microhardness of the composite (matrix) varies from HV 1100 to HV 1800 as compared with HV 240 of the as-received substrate. Wear studies show a significant enhancement in wear resistance against hardened steel ball and WC ball compared with that of commercially available Ti−6Al−4V alloy. The wear mechanism was established and presented in detail. The corrosion behavior of the composites in 3.56% NaCl (mass fraction) solution shows that corrosion potential (φ_{corr}) shifts towards nobler direction with improvement in pitting corrosion resistance. However, corrosion rate of the cenosphere dispersed Ti matrix composite increases compared with that of the commercially available Ti−6Al−4V alloy.

Key words: Ti matrix composite; sintering; wear property; corrosion; microstructure

1 Introduction

Titanium and its alloys are widely used for dental and orthopedic implants due to their excellent chemical stability, good mechanical and biocompatibility properties [1]. In this regard, it is relevant to mention that porous Ti−6Al−4V alloy is a future bio-material as it can provide not only a favorable environment for bone ingrowth, but also similar elastic modulus and stiffness to those of the surrounding bone, which would be expected to reduce the extent of stress shielding [2−3]. In the past, porous titanium was reported to be developed by powder metallurgy route [4], space holder method [5], rapid prototyping method [6], and the replication of polymeric sponge [7]. However, a precise control of pore structure (e.g., porosity, pore size, pore shape, interconnection between pores, and pore alignment) for the achievement of improved mechanical properties and biological performance is a real challenge [8]. Furthermore, porous titanium has poor friction and wear properties. Although these properties can be improved by using particulate reinforced composites, the weak interface between the particles and matrix often results in debinding or loosening of the particles. In the wear process, the release of these hard particles can cause further damage to the implant and a cascade of cellular activity that degenerates bone.

Among the reinforcements used for synthesizing MMCs, hollow fly ash particulate (cenospheres) has recently gained a significant attraction due to its excellent energy absorption properties [9]. Considerable studies have been conducted on measuring the mechanical properties of metal matrix syntactic foams [10−13] and coefficient of thermal expansion of aluminum based composites [14]. It has been observed that hollow spherical cenosphere particles in aluminum matrix provide excellent wear resistance in both dry and lubricating conditions [15]. Because of spherical nature of these particles, these particles may not cause wear to the counter surface or the implant. In the past, the development of cenosphere dispersed titanium foam was presented by powder metallurgy route with an average porosity of 45%−70% and elastic modulus of 25−42 GPa

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by the present authors [16].

In this work, the wear and corrosion behavior of the cenosphere dispersed titanium matrix composite was studied in detail and compared with those of the commercially available Ti−6Al−4V alloy.

2 Experimental

Sintering of cenosphere dispersed Ti-based composite was performed by blending of cenosphere (12%) with particle size of 75−150 μm and Ti with particle size of 20−40 μm for 7 h. The cenospheres primarily contain mullite and sementite. The chemical composition (mass fraction) of cenospheres is as follows: 28.5% Al_2O_3, 58.3% SiO_2, 6.3% Fe_2O_3, 0.4% TiO_2, and 5.8% C(carbon) [17]. The XRD pattern was reported in detail elsewhere [17]. The compaction of the blended cenosphere dispersed Ti was conducted at 75 MPa (this pressure was used to avoid crushing of cenospheres during cold compaction for making pallets) at ambient temperature and at 600 °C for 2 h (a) and 400 °C for 2 h followed by heat treatment at 800 °C for 2 h (b). After debinding (removal of the binding agent used during cold compaction through heating), sintering of the compact was conducted at 1200 °C under vacuum sealed condition for 3 h. Following sintering, the microstructures of the sintered samples (both the top surface, i.e., the surface exposed directly to the atmosphere which was removed through polishing and then used for microstructural characterization and XRD analysis) were characterized by optical microscopy and scanning electron microscopy (SEM). Any reaction did not take place between Ti and Si cenospheres, which was confirmed by the XRD analysis. If any reaction takes place between Ti and Si, there will be the presence of the peak corresponding to Ti_5Si_3 phase. Furthermore, the cenospheres did not melt during sintering process. The top surface here means the inner top surface (about 2 mm inside from the bare top surface). Thus, the XRD pattern of this surface indicates the XRD pattern of inner materials. The XRD pattern did not show any peaks for Ti−Si intermetallics as visible from XRD analysis. The pallets are small and Ti matrix has good thermal conductivity. It is therefore expected that the samples are uniformly heated during debinding and sintering. Conditionally, the samples are porous. Thus, there will be no difference in microstructure and phase constituents at different locations of sintered pallets. A detailed analysis of the phases was carried out by X-ray diffraction technique.

The compositions of the top surface and cross section of the composite were measured by energy dispersive spectroscopy (EDS) analysis attached to the SEM. The microhardness of the surface was measured by Vickers microhardness tester using a 100 g (0.98 N) applied load. Finally, the kinetics of wear of the treated surface was compared with that of the as-received one by a fretting wear tester (DUCOM, TR−283M−M4). Before the test, all samples were mechanically polished to the roughness of 2.5 μm and cleaned using acetone and dried. Commercial bearing AISI E−52100 grade steel ball with the hardness of HRC 58−66 and the diameter of 5 mm and WC ball with the diameter of 5 mm were used as counter body. The wear tests were carried out at varying normal loads of 5 and 10 N for constant testing duration of 30 min and at constant frequency of 10 Hz and constant stroke length of 1 mm. With the help of Winducom 2006 software the cumulating loss of depth with time was computed. The microstructure of the worn debris was analyzed with SEM to understand the wear mechanism. The electrochemical property of the surface in terms of pitting corrosion resistance was compared with that of the conventional Ti−6Al−4V alloy by potentiodynamic anodic polarization study in Hank's solution with the following electrolytic composition: 0.185 g/L $CaCl_2$, 0.4 g/L KCl, 0.06 g/L KH_2PO_4, 0.1 g/L $MgCl_2{\cdot}6H_2O$, 0.1 g/L $MgSO_4{\cdot}7H_2O$, 8 g/L NaCl, 0.35 g/L $NaHCO_3$, 0.48 g/L Na_2HPO_4 and 1.00 g/L *D*-glucose. In the corrosion study, a standard calomel electrode was used as the reference electrode and platinum was used as the counter electrode [15]. Polarization was carried out from −2 to 7 V (vs SCE) at a scanning rate of 2 mV/s. The pitting corrosion behavior was determined by measuring the primary potential for pit formation, φ_{pp1} (the potential at which there is a sudden rise in current density with a small increase in potential) and corrosion potential (φ_{corr}).

3 Results and discussion

The microstructures and phases present in cenosphere reinforced titanium composite were characterized. A detailed investigation of the mechanical properties of the composite in terms of microhardness and wear resistance was evaluated. Finally, the corrosion resistance property of the cenosphere dispersed titanium composite was evaluated and compared with respect to the Ti−6Al−4V alloy in Hank's solution.

3.1 Microstructural characterization of cenosphere Ti matrix composite

Figures 1(a) and (b) [16] show SEM images of the cenosphere dispersed Ti matrix composite debinded at 600 °C for 2 h (a) and 400 °C for 2 h followed by heat treatment at 800 °C for 2 h (b) and sintering at 1200 °C for 3 h, respectively.

Figure 1 shows the presence of globular cenosphere (as shown by arrows) in α-Ti matrix and the composites