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An Experimental Analysis to Investigate the Effect of Zirconia Coating on Titanium-Based Implant Surfaces for Enhanced Performance

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Abstract: The work accentuates on antibacterial action of the surface built prosthetic Ti-6Al-4V combination, which is considered as a potential embed material with low modulus, erosion obstruction and great biocompatibility. In any case, the helpless wear obstruction showed by Ti implant material is a test to be tended to. To upgrade the service time of Ti-6Al-4V composite, an exertion has been made to expand the mechanical and natural properties of the implant by covering it with micron-nano coating materials utilizing plasma spraying, which empowers to accomplish thick coatings. The surface altered examples were tested using a Scanning Electron Microscope (SEM), scratch conduct, hardness and Studies on antibacterial activities of the coatings were evaluated. This work manages the creation of monolayer coatings, for example, AT and YSZ and bilayer coatings, for example, AT/YSZ and YSZ/AT on Ti-6Al-4V compound by utilizing the plasma showering process. Among the four coatings created, the bilayer AT/YSZ covering showed higher hardness than different coatings. The antibacterial action uncovers that the monolayer AT covering showed better antibacterial movement with 29.1% lesser bacterial settlements contrasted with monolayer YSZ covering, 25.1% lesser to bilayer AT/YSZ covering and 46.1% lesser to bilayer YSZ/AT covering on Ti-6Al-4V compound, individually.

Keywords: Aluminium-titanate, Plasma Spraying, Hard coatings,Antibacterial Studies.

I. INTRODUCTION

A biomaterial should be cost-effective with superior mechanical and physical properties that exhibits structural capability and without any adverse effects on the body. Over the decades, materials like 316L-stainless steel, cobalt chromium alloys and commercially pure titanium alloys have been employed as implant materials (Azevedo, 2003) [1]. Failure of an implant is the most important aspect which directly influences the material of choice. Establishing the cause of failure provides information for design improvements, working methods and the components used. The major causes for implant failure can be categorized into infection/periimplantitis, surgical errors, dislocations, design errors, improper bone-bonding, osteomyelitis, stress-shielding, corrosion, wear and aseptic loosening.Yang et al., developed nanostructured Al2O3-TiO2-Zr2 composite powders for plasma spraying by using the spray drying granulation technique. In this work, the spray drying parameters on the surface morphology and properties of coatings were investigated. The results obtained from their study clearly reveal that the powder particle size as well as the powder flow ability initially increases with an increase in the solid slurry content and the binder content. This study clearly demonstrated that the coating developed by using composite powder feedstock with spherical geometry, homogenous distribution and finer microstructure leads to highly dense and finer microstructure of the coating. Further to this, due to less lamellar structure and homogenous composition, the nanostructured coating showed superior hardness and toughness when compared with the conventional micro structured coating (Yang et al. 2013) [2].Among genuine impacts, implants leading to contamination and infections are the most serious effects in orthopaedic applications. In orthopaedic surgery, one of the main challenges is the prevention of bacterial formation resulting in bacterial biofilm on implant surfaces (Kazemzadeh-Narbat et al. 2013) [3]. The bioactivity, biocompatibility and its physical properties are vital for an implant material, so do the antibacterial activity that plays a significant role in enhancing the service period of an implant. In order to prevent the infection on and around titanium implants, the antibacterial coatings are performed on titanium implants (Busscher et al. 2010) [4].

II. OBJECTIVE

1. To develop hard coatings utilizing micron and nano alumina and yttria Stabilized zirconia on Ti-6Al-4V through plasma spray process.
2. To assess the hardness and scratch conduct of hard coatings utilizing Vickers Hardness and Scratch tester.
3. To survey the antibacterial property of coatings utilizing colony count method.

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III. EXPERIMENTAL DETAILS

A. Substrate Preparation

Ti-6Al-4V was purchased in the form of a rectangular plate and was cut into 10x10x01mm work pieces using electro-discharge machining (EDM) wire cutting machine. The specimens were polished to mirror like finish with SiC paper (220-2500 grits), followed by diamond polishing using 1µm diamond paste prior to plasma spraying. Contaminations or oil on the substrates surface significantly lowers the coating adhesion and may cause splitting or delamination. To remove the contamination, substrates were washed with distilled water and ultrasonically cleaned in acetone for 10 minutes. Prior to coating process the substrates were grit blasted and preheated to a temperature of 400◦C with plasma torch, in order to enhance the adhesion of the coating.

Fig. 1 Titanium sample

B. Coating Deposition

Depending upon the different melting temperatures of the powders used in coatings, the various process parameters have been employed for both hard and soft coatings and these coatings were prepared and the process parameters utilized for the hard coatings are tabulated in Table 1.

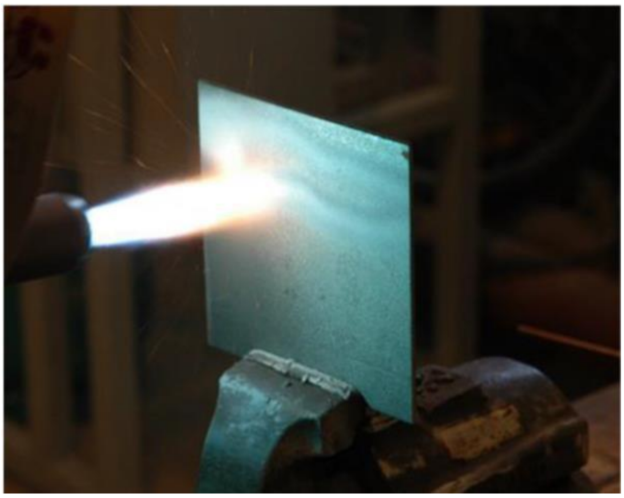

Fig. 2 Plasma spray coating on Titanium substrate

Table 1. Parameters used for plasma spray process in hard bilayer coatings

Parameters	AT coating	YSZ coating
Plasma current (A)	600	450
Plasma voltage (V)	50	55
Ar gas flow pressure (NLPM)	42	42
42H2 gas flow pressure (NLPM)	9	8
Carrier gas flow rate (psi)	45	45
Powder feed rate (gram/minute)	45	45
Spray distance(cm)	10	10

C. Hardness, roughness and scratch testing

Micro hardness values for all the coatings were measured using Vickers micro hardness ester. The measurements were carried at different points on the cross section of as-sprayed coatings with a load of 200g for 15s and the average value is reported. On the other hand, surface roughness of the monolayered and bilayered coatings was measured. Scratch resistance of the coatings was evaluated using a commercial micro scratch tester on the obtained hard mono/ bilayered coatings, micron/nano coatings and soft composite coatings. The scratch on the hard and soft coatings, was performed with progressive loading from 0 to 100N by using a diamond stylus of the 200μm radius. The loading was varied in steps at 2 N/mm. The scratch tracks were observed through an optical microscope.

D. Antibacterial Testing

The anti-bacterial assay was performed by using Staphylococcus aureus and the culture was maintained in a nutrient broth (Himedia). A single colony was inoculated into 5ml of sterile broth and incubated at 37◦C for 18 hours, and the turbidity was later measured at absorption of 562 nm. A value of 0.52-0.54 has to be obtained which shows a confluence of 10*9cells/ml. A dilution of 108 organisms was organized for the experiment. The obtained coatings were sterilized in 70% ethanol for 30 minutes and washed once with phosphate buffer saline (PBS). In a 12-well plate, 2ml of culture was added along with the samples and incubated at 37◦C for 24 hours. After incubation, the bacterial culture was removed and the samples were rinsed thrice with PBS. The solution was serial diluted after three washes till 10*-4 dilution. 0.1 ml of 10*-3 and 10*-4 dilutions were placed on sterilized nutrient agar (Himedia) plates and incubated at 37◦ C for 18 hours and the total numbers of bacterial colonies were finally counted.

IV. RESULTS AND DISCUSSION

A. Micro hardness and roughness for hard mono and bi-layered coatings

Five measurements were taken across the cross-section of the coatings using Vickers Hardness testing machine by applying a load of 200 g at a dwell time 15s. The average micro hardness values of AT, YSZ, BL-AT/YSZ and BL-YSZ/AT coatings are 804±5 HV, 644±4 HV, 1053±5 HV and 1037±4 HV, respectively. The average surface roughness and hardness values are reported in table. The variation in micro-hardness observed for different coatings is because of the dissimilarity in the surface morphology and its phase composition obtained in the coating. The micro hardness value for the BL-AT/YSZ coating was found to be higher, which is associated with low porosity in the coating. The average surface roughness for AT, YSZ, BL AT/YSZ, and BL YSZ/AT coatings was measured as 6.92µm, 7.82µm, 6.84µm, and 7.36µm, respectively.

Table 2. Roughness and hardness measurements for hard monolayer and bi-layered coatings.

Coatings	Roughness (Ra) (µ m)	Hardness (HV)
Monolayer AT coating	6.92	804±5
Monolayer YSZ coating	7.82	644±4
BL AT/YSZ coating	6.84	1053±5
BL YSZ/AT coating	7.36	1037±4

B. The scratch behaviour of hard mono- and bi-layered coatings

Among the coatings, AT, BLAT/ YSZ and BL-YSZ/AT coatings exhibited tensile crack, whereas the YSZ mono-layered coating revealed micro tensile crack, which confirms that all coatings undergo cohesive failure. Generally, the tensile cracks are formed due to the frictional stress present behind the trailing edge of the stylus, and these stresses balance the compressive frictional stresses ahead. Due to the higher thickness, only cohesive failure occurs and it is not possible to evaluate the adhesion strength of the coatings. In the present work, it is observed that the substrate was not exposed as the coatings did not spall during the scratch test, which implies that all coatings have undergone only cohesive failure. Smear particles are observed on the scratch track of BL AT/YSZ coating. The type of coating failure for all the coatings was measured and tabulated in Table 4.2. From the scratch, width of AT coating is minimal and amongst the bilayer coatings, the scratch width of AT/YSZ BL coating is minimal which clearly indicates that monolayer AT coating and BL AT/YSZ coating possess higher cohesive strength.

Table 3. scratch testing for plasma sprayed hard monolayer and bilayer coatings

Coatings	Nature of crack	Type of coating failure
Monolayer AT coating	Long Tensile crack	cohesive
Monolayer YSZ coating	Micro Tensile crack	cohesive
BL AT/YSZ coating	Long Tensile crack	cohesive
BL YSZ/AT coating	Long Tensile crack	cohesive

C. Antibacterial evaluation coatings

Bacteria are found everywhere and severe infection leading to failure of medical implants will obviously result from surface microbial invasion. Due to the formation of microbes in the implanted site, many failures of implants are widely reported. In order to prevail over the problem of failure due to bacterial adhesion and infection, the implant material should possess superior antibacterial property. In this condition, an anti-bacterial agent is to be used to enhance the performance of the medical implant and to prevent it from bacterial infection. However, the usage of antibiotics leads to the development of multi-drug resistant strains that poses a major threat in the treatment of infection. Amongst the hard coatings, the number of bacterial colonies on AT coating (hydrophilic) was 29% lesser than YSZ and on BL AT/YSZ, it was 28% lesser than that of BL YSZ/AT. This is ascribed from the fact that, although a vast majority of the microorganisms has affinity towards hydrophobic surfaces, they specially adhere to hydrophilic substrates as they will recruit protein and enable apatite deposition. From this observation, it is evident that AT coating provides increased antibacterial property when compared to YSZ coating.

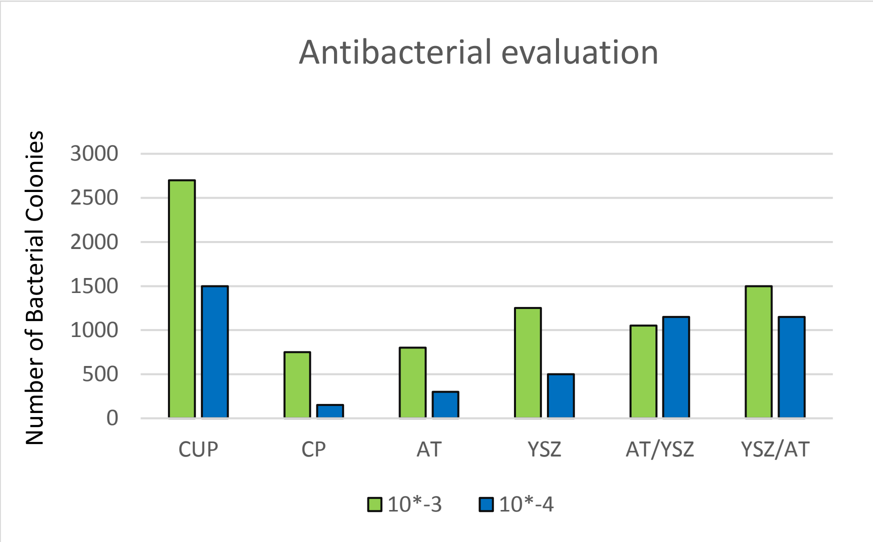

Fig. 3 Antibacterial studies for plasma sprayed hard monolayer and bilayer coatings

V. CONCLUSION

The main objective of this thesis is to identify the best possible coating which can be considered to improve the service period of the orthopaedic implant Ti-6Al-4V alloy whose antibacterial properties are enhanced through coatings. Failure of thin Physical vapour deposition, Chemical vapour deposition and Ti-Nitride coatings in clinical use has prompted us to look for thicker coatings, which will wear at slower rate comparatively to thin coatings and also exhibits improved antibacterial and biocompatibility. Nano coatings are shown to exhibit superior properties when compared to coatings formed using powders of micron size. However, in order to have cost effective solution, we have made an attempt to evaluate the properties of mixture of micron and nano powder compositions such as Al2O3+TiO2, YSZ, were tried and tested for its wear resistance and antibacterial properties. Basically, hard coatings are developed on the femoral head as there is severe wear and tear when it slides against the cup and soft coatings are deposited on femoral stem to enhance their ability to integrate with bone. Our studies on plasma sprayed coatings clearly revealed that amongst the hard coatings, the plasma sprayed mono-layered AT coating and bi-layered nanostructured Al2O3+13wt%TiO2/YSZ coating on Ti-6Al-4V alloy exhibited the best performance in terms of wear resistance and antibacterial activity. The above conclusion is arrived after making a detailed study of the several features associated with the coatings.

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