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Evaluation of Microstructure and Mechanical Properties of Plasma Sprayed HA+10% wt. Al_2O_3 Coatings on Titanium and SS 316 L with and without Bond Coat

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Abstract
The investigation of bioactive and biocompatible is required so that service life of body implants in harsh body environment can be prolonged. This study involves the performance evaluation of plasma sprayed hydroxyapatitite and 10 percent weight alumina (HA +10% wt. Al_2O_3) coatings on pure titanium and SS 316 L. Optical microscopy, X-ray diffraction, scanning electron microscopy techniques were used to characterize powder, specimens and as sprayed coatings.

Keywords
Plasma Spray Process, Bioactive Coatings, Biocompatible Coatings, Hydroxyapatite

I. Introduction
Degradation of metallic implants in harsh body environment by wear, corrosion are the main failure modes of components in human body. Very low carbon stainless steels, titanium and its alloy like Ti – 6Al – 4V, cobalt alloys and magnesium alloys have been developed for hip and knee prostheses, dental implants and bone replacement parts, but they are not able to meet requirements of both bioactivity and biocompatibility. The strength of these materials is very good, but they do not make bond with natural bone and tissues. One possible way to overcome these problems is the use of thin bioactive, biocompatible corrosion and wear resistant coatings.

Coatings can add values to products by allowing the mechanical properties of the substrate materials to be maintained while protecting them against corrosion inhuman body. Bioactive and biocompatible coatings used for repair and replacement of bone or tooth must withstand extremely severe environmental condition. Diffusion coating and overlay coating are two basic systems that are currently used on metallic implants to provide improved resistance in body environment and thereby extend service life of the component. Deposition rate of diffusion coatings is very low hence these are not commercially viable. Overlay coatings can be deposited by various thermal spray techniques such as flame spraying, arc spraying, high velocity oxy-fuel (HVOF) and plasma spraying [1-2].

Plasma spraying is a thermal spray technique and is capable of producing coatings with lower porosity, higher hardness, superior bond strength and less decarburisation than many of the other thermal spray techniques with exception of HVOF. Plasma sprayed hydroxyapatite (HA) coatings on titanium implants provide excellent biocompatibility and bioactivity for orthopedic and dental prostheses [3 – 6].

The composition and structure of coatings are typically determined by role that they play in various material systems and performance environments. The coating should have a composition that will react with the environment to produce the most protective layer possible, provide resistance to corrosive environment, should have long term stability and have resistance to cracking or spallation under stresses induced under operation of components [7].

Current laboratory animal studies on plasma sprayed HA coating on titanium implants have that HA coating displays many biological advantages, such as formation of firm chemical bonding with bone [8-9], the enhancement of tissue growth into the pores of HA coated metallic implants [10] and protection of surrounding bone against metal ion release from metallic implants [11-12]. The ideal HA coating on titanium implant should have long stable life in physiological environment and induce natural bone growth around the implants.

II. Materials and Methods

A. Powder and Coating Preparation
Synthetic hydroxyapatite spray dried powder ($Ca_{10} (PO_4)_6 (OH)_2$) was procured from IFGL Bioceramic Limited, Kolkata, India with an average particle size of 52 mesh and aluminum oxide (Al_2O_3) was procured from S D fine-chem limited, Mumbai, India. The average particle size of HA was 52 mesh and that of aluminum oxide was 100 mesh and purity of these powders were 100% and 98% respectively and were 100% crystalline. Feed stock for plasma spray coating was made by mixing 10 wt. % aluminum oxide with HA powder by mechanical method for 4 hours at 150 rpm in planetary ball mill. Before spraying the powder was kept in air oven at 150ºC for 2 hours to remove moisture if any present in the mixture.

Plasma spray system (Dyne Technology, United Kingdom) with robotic arm was used to ensure the repeatability of the process. Argon and hydrogen were used as plasma gas and argon as carrier gas. In plasma gas argon was primary gas and hydrogen was secondary gas. The net energy of plasma torch for coating was 20 kW with 750 A current and 50V voltage. The plasma arc pressure was 60 psi (pond per square inch) and powder pressure was 80 psi. The external nozzle diameter was 20mm and internal one was 8 mm and stand off Distance (SOD) was 10 cm with powder feed rate of 30 g/min. The temperature of plasma flame was 12000 ºC but specimen temperature was less than 200 ºC. The flame diameter was 20 mm which allowed melting of majority of injected particles with high efficiency.

The substrate material used for the HA/ alumina coating was pure titanium and SS – 316L (very low carbon stainless steel). The specimens were cut into size of 20x15x5 mm^3 from the strip procured from Guru steel and Engineering Company, Mumbai India. The specimens were polished with SiC papers down to 220 grit sand blasted with alumina powder (grit 45) to roughen their surface to increase the surface area, so the adhesivity of coating increases. Before apply coating on these specimens, these were cleaned in ultrasonic bath (Soltec, Italy).

B. Elemental Analysis
The elemental analysis of bare specimens was done using Energy Dispersive X-ray Analyzer (EDX) fitted with Field Emission Scanning Electron Microscope (FESEM). The analysis of Titanium and SS316 L is shown in Table 1.

Table 1: Chemical Composition Analysis of Substrate Materials

	Chemical composition (wt. %)										
	C	Si	Mn	P	S	Cr	Ni	Mo	Fe	Ti	Coating Thickness (µm)
SS 316 L	0.023	0.645	1.96	Traces	---	17.56	8.98	2.16	Bal.	---	230-260
Titanium	---	---	---	---	---	---	---	---	---	100%	230-260

C. Microstructure Characterization

The inverted optical microscope (Leicca Microsystems Imaging Solutions limited, England) and was used to study the microstructure of powders and coatings. The microstructure of coated samples were taken with the inverted optical microscope.

D. Porosity and Examination

The computerized inverted optical microscope (Olumpus) equiped with Material Plus software was used to examine porosity of plasma spray coatings. The surface image of HA coating was captured by optical microscope and image was analysed using image analysing software which determined porous area according to colour difference between pores and coating. Thirty readings were taken for each type of coating on various samples and afterwards average of these measurements was taken.

E. Mechanical Characterization

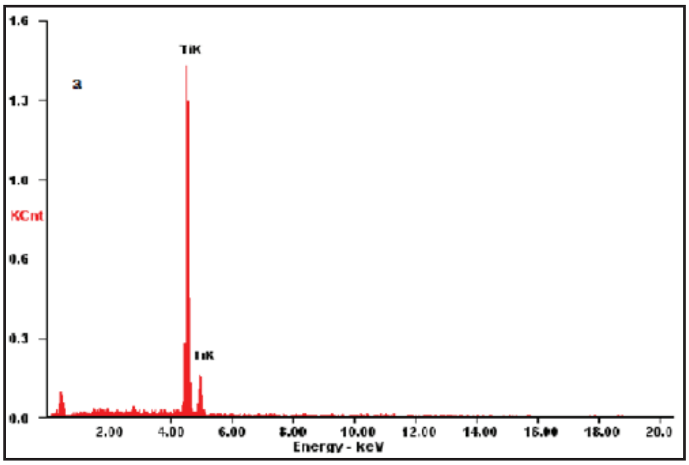
As ASTM C-633, bond strength of coating was checked. Samples of one inch diameter and one inch in length were prepared. The specimen was prepared with sand blasting with alumina grit and then plasma spray coated with HA/alumina powder. The coating thickness of 150µm was maintained at all the samples. The coupling specimen was also grit blasted. Both the specimens (coated and bare) were then glued with thermoplastic and then joined under pressure in a fixture. The couples were then cured for specific time and were subjected to tensile tests.

The microhardness of coating was determined using digital micro Vickers hardness tester with square pyramidal diamond indenter with a load of 2.942N. For micro hardness measurement the sample were cut to view the cross section. The samples were mounted in transoptic powder (Buehler, USA). The average of 10 indentations along the interface was taken from one specimen.

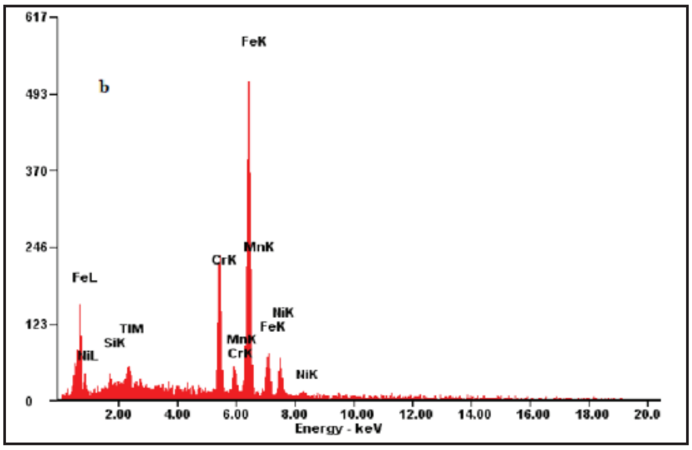
III. Results and Discussion

A. EDAX Analysis of Substrates and Powders

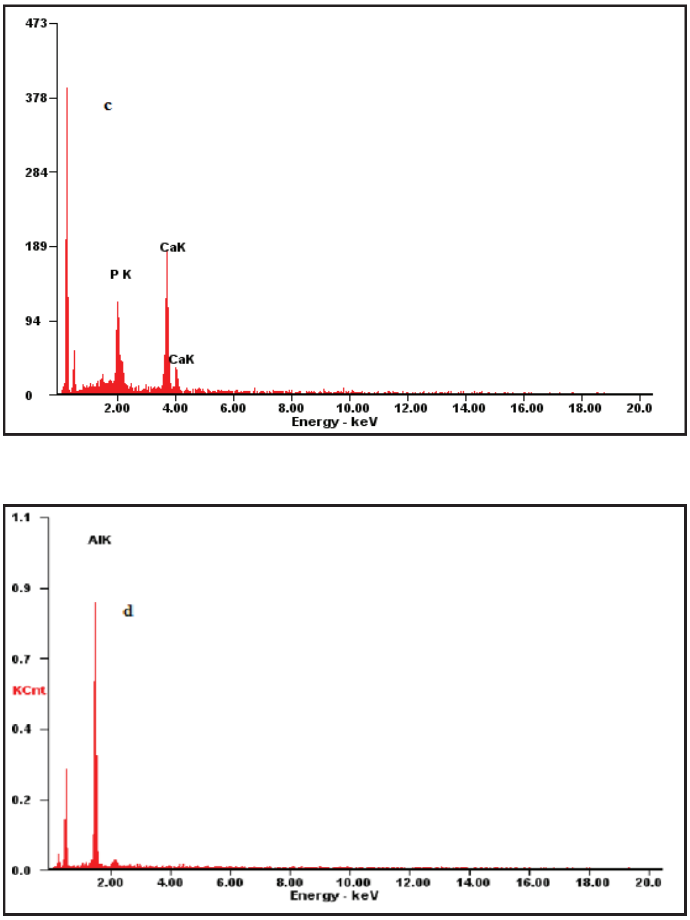
The EDAX analysis of titanium, SS 316 L, HA powder, alumina powder and composite feedstock is shown fig. 1.



Element	Wt%	At%
TiK	100.00	100.00
Matrix	Correction	ZAF



Element	Wt%	At%
SiK	01.32	02.64
TlM	03.83	01.05
CrK	17.56	18.99
MnK	01.96	02.01
FeK	64.65	65.08
NiK	10.68	10.23
Matrix	Correction	ZAF



Element	Wt%	At%
AlK	100.00	100.00
Matrix	Correction	ZAF

Fig. 1: EDAX analysis of as received powders and substrate: (a) Titanium substrate material (b) SS 316 L (c) Hydroxyapatite powder and (d) Alumina powder

B. Microstructure of HA/Alumina Coating

The microstructure examination of as spray coated specimens was done with Leicca inverted microscope and the micro structures are shown in fig. 2.

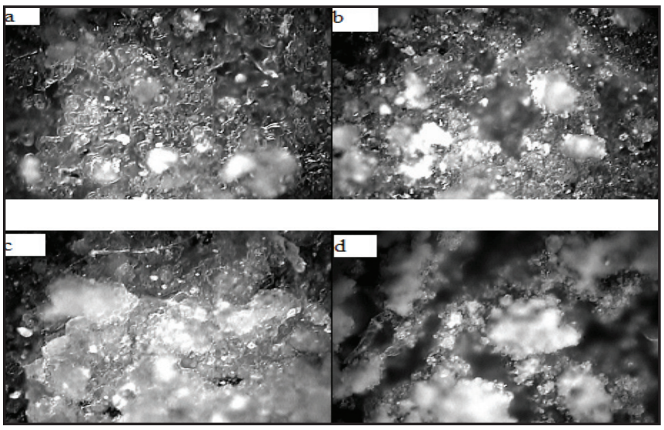


Fig. 2: Microstructure of as coated (a) SS316 L (b) SS 316 L with intermediate layer of Ti_2O_3 13% Al_2O_3 (c) Titanium and (d) Titanium with intermediate layer of Ti_2O_3 13% Al_2O_3

C. Porosity of Coating

There is a significant role of porosity to play as far as biological properties of coating are concerned. Porosity measurements of plasma spray coated specimens were found to be less than 3%.

D. Microhardness of Coating

The microharness data of coatings are compiled in fig. 3 which shows the microhardness profile along the cross-section of coating as function of distance from coating – substrate interface.

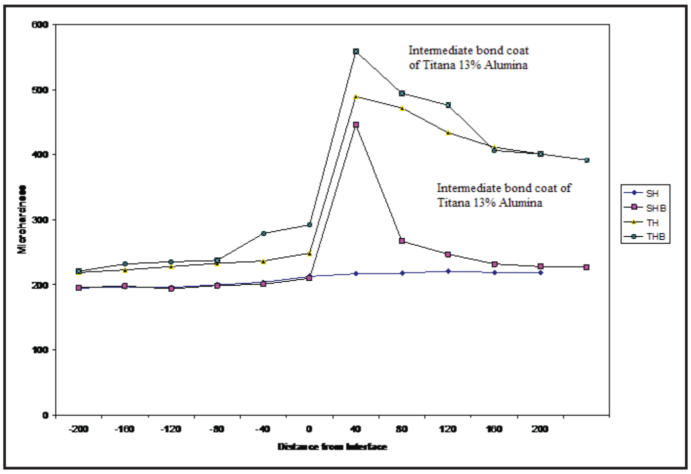


Fig. 3: Microhardness of coatings with bond coat and without bond coat on Ti and AISI 316L SS

E. Bond strength of Coating

Bond strength of coatings were evaluated and found that irrespective of substrate the bond strength of coating with bond coat was much higher than that of without bond coat.

IV. Conclusion

1. The coatings were found to be made of completely molten splats
2. The porosity of coatings were found to be less than 3%.
3. Microhardness of coating was found to be maximum near interface and decreased at a distance from interface, moreover microhardness of bond coat was found to be maximum.
4. Bond strength of coating with bond coat was higher than that of without bond coat, but it was almost same irrespective of substrate materials.

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