iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 5 **Issue:** XII **Month of publication:** December 2017

DOI:

www.ijraset.com

Call: 08813907089 | E-mail ID: ijraset@gmail.com

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor :6.887
Volume 5 Issue XII December 2017- Available at www.ijraset.com

Study of the microstructural and mechanical properties of plasma sprayed HAp and HAp + 15 wt-% Al_2O_3 coatings on pure titanium

Vikas Rattan[1], T.S.Sidhu[2], Manoj Mittal[3]
[1] *Research Scholar, Inder Kumar Gujral Punjab Technical University, Kapurthala, Punjab, India*
[2] *Shaheed Bhagat Singh State Technical Campus, Ferozepur, Punjab, India*
[3] *Inder Kumar Gujral Punjab Technical University, Kapurthala, Punjab, India*

Abstract: ***The poor mechanical properties like brittle nature, low wear resistance, abrasion, fatigue, and hardness of hydroxyapatite coatings resist it to be used as a coating on load bearing implants. To improve the mechanical properties of hydroxyapatite coatings it is reinforced with secondary bioinert material. In this study an attempt has been made to improve the mechanical properties of the plasma sprayed hydroxyapatite (HAp) coatings by reinforcing it with 15wt-% Al_2O_3. The feedstock and coatings were characterized by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDAX) and X-ray diffraction analysis (XRD). Surface roughness for reinforced HAp coatings was found to be more than pure HAp coatings. Tensile bond strength was found to be increased with the increase in alumina (Al_2O_3) content of the coatings.***

Keywords: Biomaterials, Hydroxyapatite, Reinforcement, Alumina, Plasma spraying

I. INTRODUCTION

Calcium phosphate ceramics have received much attention as a potential candidate for replacing and augmenting bone tissue. The most widely used calcium phosphate based bioceramics are hydroxyapatite and β-tricalcium phosphate[5]. Hydroxyapatite [(HAp), $Ca_{10}(PO_4)_6(OH)_2$], is the crystalline phase of calcium phosphate with a Ca/P ratio of 1.67 and is the main constituent of bone and teeth in the human body. It is a promising material for biomedical applications. It helps in promoting bone growth and has the ability to bond directly to the bone because of its highly bioactive nature [1]. It suffers from poor mechanical properties, and is not preferred to be used as a bulk material under severe loading conditions [2]. One of the solutions to this problem is to use the HAp as a coating material on the bioinert metallic substrates, e.g. titanium and titanium alloys such as Ti-6Al-4V. Several methods are used to deposit HAp onto implant surfaces, for example, thermal spray techniques (plasma spraying, high-velocity oxyfuel spraying method), ion beam sputtering, pulsed laser deposition, sol–gel, electrophoretic deposition method, electrochemical deposition and press and sinter method[3-5]. Another method to overcome the mechanical limitations of HAp is to reinforce the HAp with bioinert materials with better mechanical properties and then coat it on the metallic substrates. By doing this, one can take the advantage of the bioactive properties of the ceramic coating and the mechanical properties of the metallic substrate. A significant improvement in mechanical properties can be attained by reinforcing HAp with yttria-stabilized zirconium oxide, titanium oxide, aluminum oxide and carbon nanotubes etc. without compromising its biocompatibility [6]. Researchers have reported that reinforcing HAp with ZrO_2 or BaO and Al_2O_3 significantly improved the mechanical properties of hydroxyapatite [7]. Singh et al. [8] have reported improvements in the properties of the HAp by reinforcing with alumina and other materials. Results of pure and reinforced HAp coatings successfully deposited by plasma spraying process show denser and crack-free coatings for the reinforced coatings. Improvement in surface roughness was observed with the reinforcement. Porosity decreased, tensile strength increased, microhardness improved and corrosion current density is higher in the reinforced HAp coating as compared to pure HAp coatings. However, non-favorable phases like TCP (tricalcium phosphate) and TTCP (tetra calcium phosphate) were also observed. These phases are prone to dissolution in the body environment rapidly, which may lead to failure of implant or tissue loss. The present study focuses on the development and characterization of the pure and reinforced hydroxyapatite coatings on the pure titanium substrate via plasma spraying technique.

II. EXPERIMENTAL

A. Coating Feedstock and Substrate Material

Hydroxyapatite powder (Capitol 60-1) with mean particle Size: d (50) =45.5µm, procured commercially from the Plasma Biotal Limited, UK has been used as the feedstock powder to develop coatings. The Al_2O_3 powder (plasma spray grade) was commercially

procured from Inframat Advanced Materials LLC, USA. The micrographs of the feedstock used for plasma spraying are shown in Fig.1. It can be seen from the micrographs that HAp powder has a spherical morphology, while Al_2O_3 powder has angular /sharp morphology.

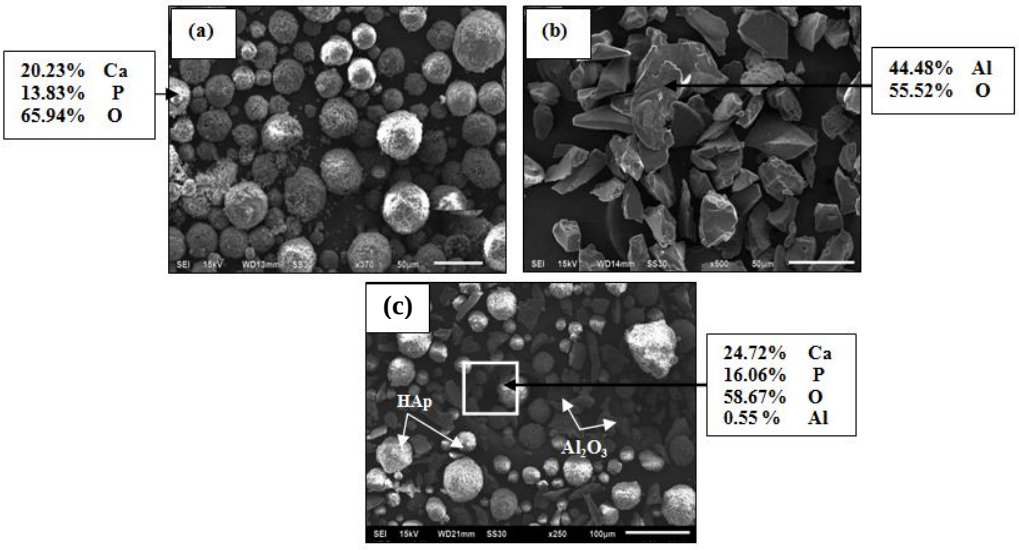


Fig. 1 SEM morphology and EDAX analysis of (a) HAp powder, (b) Al_2O_3 powder, and (c) HAp+15 wt-% Al_2O_3 powder

The feedstock used for the coating was prepared by blending 15wt-% Al_2O_3 powder with HAp using mechanical mixing. The commercially pure titanium (cp-Ti) (Grade 2) supplied by Steel Emporium, Mumbai, India has been used in the present study as the substrate material and was coated using 40 kW Miller thermal plasma spray apparatus at Anod Plasma Limited, Kanpur, India. The coating process parameters employed for plasma spraying are represented in Table I. The samples for the present investigation has been prepared by cutting the strip in the form of samples of dimensions 20 x 15 x 5 mm. Polishing of the uncoated substrate samples was done using SiC Emery papers of 180, 220, 400, 600 grit size.

Table I Plasma spraying process parameters employed for coating the specimens

Process parameter	Value
Arc current (A)	500
Arc voltage (V)	50
Powder feed rate (g/min)	40
Standoff distance (mm)	100
Plasma arc gas pressure (MPa)	0.41
Powder carrier gas pressure (MPa)	0.31

B. Characterization of Coatings

SEM/EDAX analysis of the feedstock powder and the coated specimens has been done on the Scanning Electron Microscope (JEOL, JSM-6610LV), Tokyo, Japan with EDAX attachment (Oxford-instruments, England). XRD analysis for the feedstock powder and coatings has been done using X-ray Diffractometer (PANalytical, XPERT-PRO).

The surface roughness of the coated specimens were measured using the surface roughness tester (SJ-201, Mitutoyo). Surface roughness values represented here are the average of the five readings taken along different directions.

The tensile bond strength of coatings (pure and reinforced) was measured according to ASTM C 633 standard. Two cylindrical specimens of dimensions (ϕ25.4 mm x 25.4 mm) were used for the study. One plasma coated sample with its coated side was glued to other bare (uncoated) sample with epoxy adhesive. Both the specimens were joined and fixed in a vice and cured for 24 h at room temperature. The tensile bond strength test was performed with the tensile machine (Hounsfield, UK). The load was applied to the one arm while the other arm was kept fixed. The transverse speed of the arms was kept at 0.1mm/min for all the tests. The samples

having an average coating thickness of 155 µm were tested in the tensile bond strength test. The test was performed on three similar samples for each type of coatings and the mean tensile bond strength has been reported.

III. RESULTS AND DISCUSSION

A. Coating Analysis

SEM images with EDAX analysis of the pure and reinforced coated specimens of the pure Ti are represented in Fig. 2.

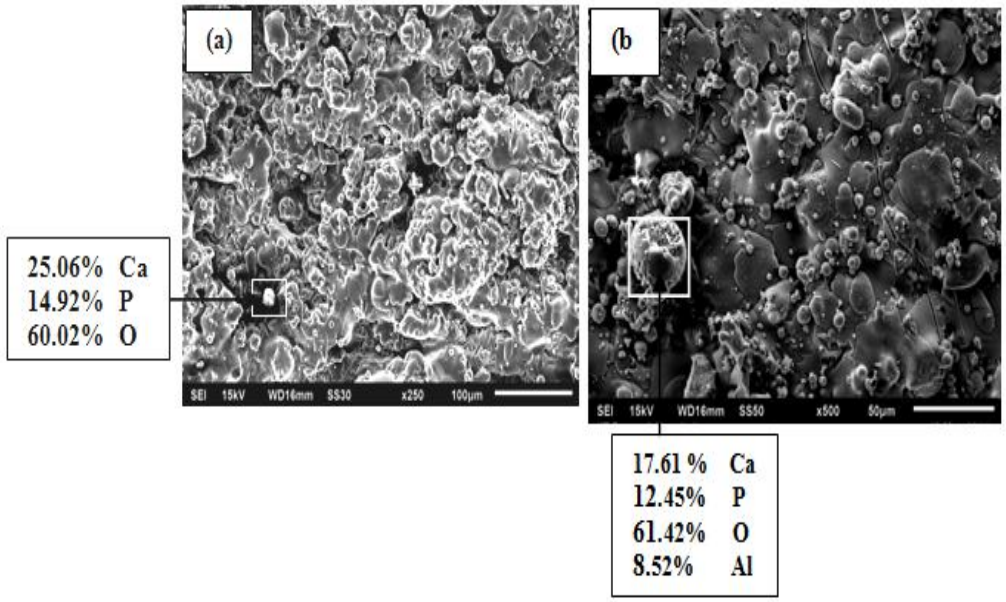


Fig. 2 SEM morphology and EDAX analysis of plasma spray coating on pure Ti (a) Pure HAp coating and (b) HAp+15 wt-% Al_2O_3 coating

As sprayed and reinforced coating microstructure consists of some unmelted, partially melted and fully molten splats on its surface. The EDAX analysis of the coatings shows that the coatings are mainly formed of the elements like Ca, P, O, and Al. Some cracks can also be seen on the surface of the coatings. This may be caused due to the rapid cooling of the coating as compared to the substrate and the stresses induced in it. As the coatings are much thinner than the substrate supporting it, so most of the stress is induced in the coatings and hence the cracks are developed in the coatings [9].

B. Surface Roughness

Surface roughness plays a vital role in the effective attachment and prolonged working of the implant in the human body. Studies by L.Ponsonnet et al. [10] showed that the cell behavior on biomaterial surface is largely affected by surface properties like roughness, texture, chemical composition and morphology. The surface roughness parameters (R_a, R_q, and R_z) for the plasma coated pure Ti substrates are shown in Table II.

Table II Roughness values (in µm) of HAp and HAp+15 wt-% Al_2O_3 plasma coated cp- Ti substrates

Parameter	HAp	HAp+15wt-% Al_2O_3
R_a (µm)	6.520	7.213
R_q (µm)	7.82	9.58
R_z (µm)	35.44	41.79

The average surface roughness (R_a) value for pure HAp and HAp+15 wt-% Al_2O_3 coated specimens were 6.520 ± 0.743 µm, and 7.213 ± 0.67 µm respectively. The roughness values represented are the average of the five readings taken along different directions of the substrates. The higher roughness values may be attributed to the presence of alumina as reinforcement.

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor :6.887
Volume 5 Issue XII December 2017- Available at www.ijraset.com

C. XRD Analysis

The XRD pattern obtained from feedstock powders is shown in Fig. 3.

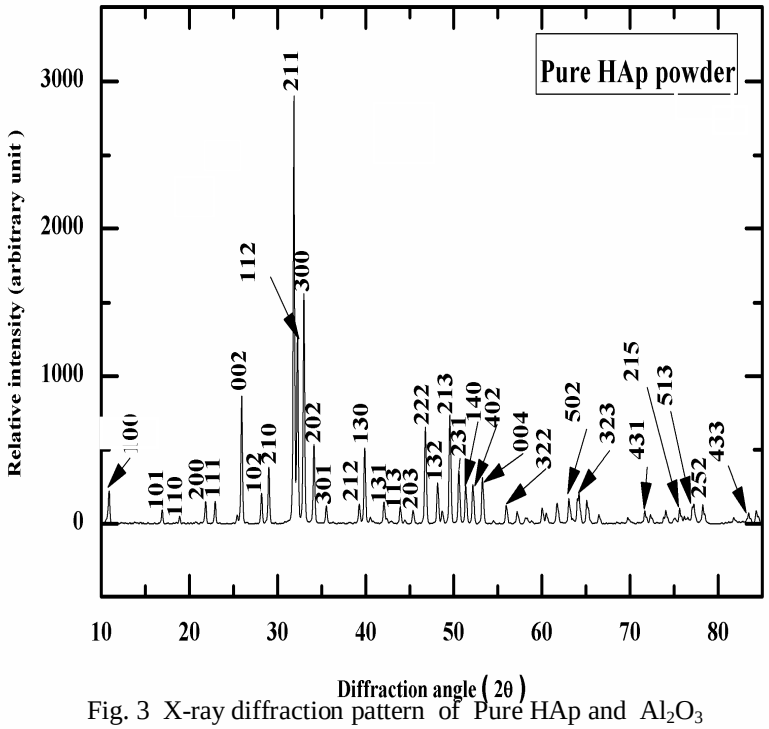
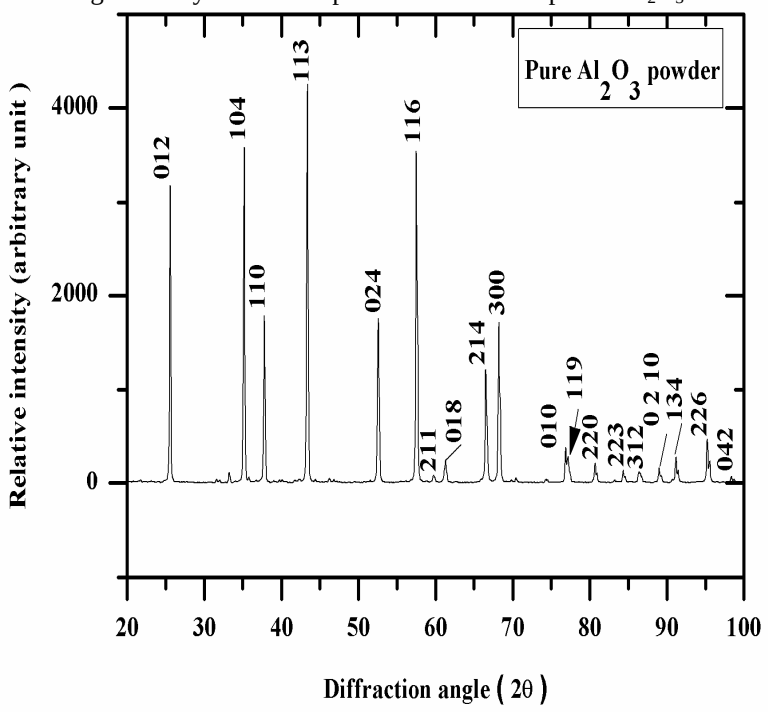


Fig. 3 X-ray diffraction pattern of Pure HAp and Al_2O_3



The XRD pattern shows peaks corresponding to HAp and alumina and no amorphous phase are present in the pattern which indicates that the feedstock powder is highly crystalline material. All the major peaks of HAp powder and Al_2O_3 powder matches the Joint Committee on Powder Diffraction Standards (JCPDS) cards 73-0293 and 42-1468 respectively. XRD analysis was also carried out on hydroxyapatite coatings produced by plasma spraying. Fig. 4 (a and b) shows the XRD spectrum of the coatings with pure HAp and HAp + 15 wt- % Al_2O_3.

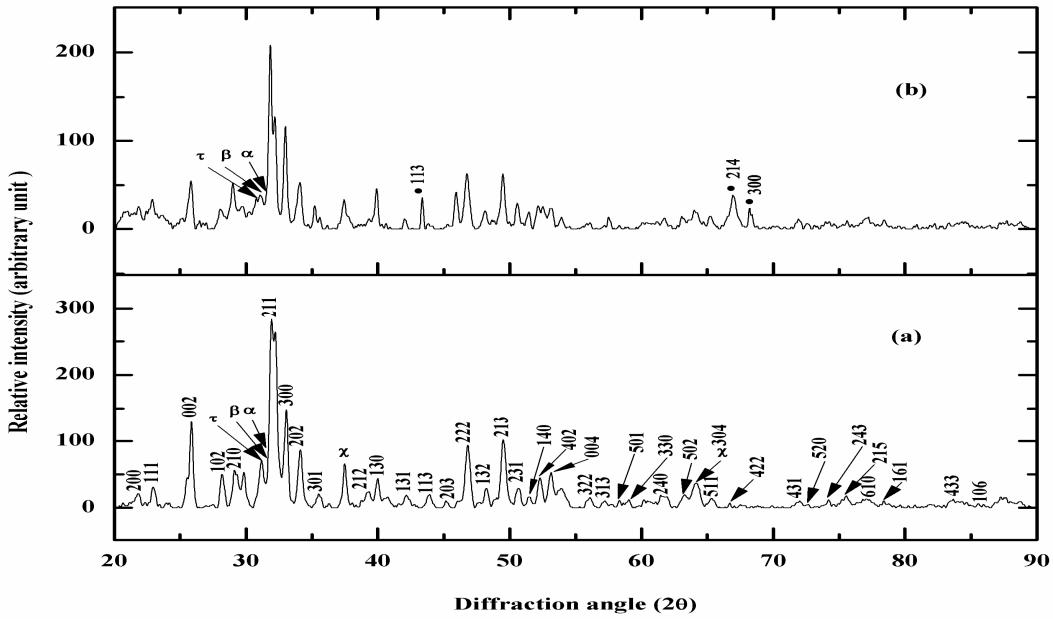


Fig. 4. X-ray diffraction patterns of (a) Pure HAp coatings, and (b) HAp +15 wt-% Al_2O_3 coatings on pure Ti [•represents *hkl* indexing of Al_2O_3 and other represents *hkl* indexing of HAp] [α: α-TCP, β: β-TCP, τ: TTCP, χ: CaO]

The XRD patterns show the sharp peaks corresponding to hydroxyapatite and alumina present in the coatings. There are few extra small peaks at approximately 30-33^o on the XRD spectrum. These extra peaks correspond to the traces of α -tricalcium phosphate (α-TCP), β- tricalcium phosphate (β-TCP) and tetra calcium phosphate (TTCP). Also, the traces of the CaO can be observed between 37-39^o and between 64-66^o for pure HAp coatings. The presence of these phases can be related to the occurrence of the decomposition of hydroxyapatite during the plasma spraying process. It can be inferred that during plasma spraying some hydroxyapatite had decomposed into tricalcium phosphate and CaO with H_2O vapor. These phases are formed due to the thermal decomposition and dehydroxylation of hydroxyapatite powder particles while subjected to a very high temperature of plasma jet (~ 15000^{o}C) [8, 11-16].

D. Tensile bond strength test

Bond strength is one of the important mechanical properties that can affect the coating and ultimately the life span of the implant [17]. The tensile strength test results show that the tensile bond strength for pure HAp and HAp+15wt-% Al_2O_3 coatings was found to be 20.88 Mpa and 23.18 MPa respectively. The tensile bond strength was found to increase with an increase in the Al_2O_3 content to HAp. The results are in close agreement with the studies presented herewith. Quek et al. [17] studied the influence of plasma spraying process parameters like gun transverse speed and spraying current on the HAp/Ti-6Al-4V composite coatings and found higher bond strength for the composite coatings as compared to pure HAp coatings. Similar results were found by Chou et al. [18] in their literature, that the bond strength of pure HAp and ZrO_2/HAp composite coatings to Ti-6Al-4V substrate as per ASTM C-633 were 28.24 and 32.49 MPa.

IV. CONCLUSIONS

Pure and reinforced (HAp+15wt-% Al_2O_3) hydroxyapatite coatings were successfully deposited on commercially pure Ti (Grade2) substrates by plasma spraying process. The following conclusions have been drawn from the study.

A. The plasma sprayed pure HAp coatings has less surface roughness (6.520 ±0.743 μm) as compared to reinforced HAp coatings (7.213 ± 0.67 μm). Higher surface roughness of reinforced coatings was due to the presence of alumina.

B. Some unwanted phases like α-TCP, β-TCP, TTCP and CaO has been observed in the plasma sprayed pure and reinforced hydroxyapatite coatings.

C. Tensile bond strength for reinforced coatings was found to be more than the pure HAp coatings. This may be attributed to the good adhesive strength between the coatings and the substrate and strong mechanical bonds at the interface and among the HAp splats with the addition of Al_2O_3.

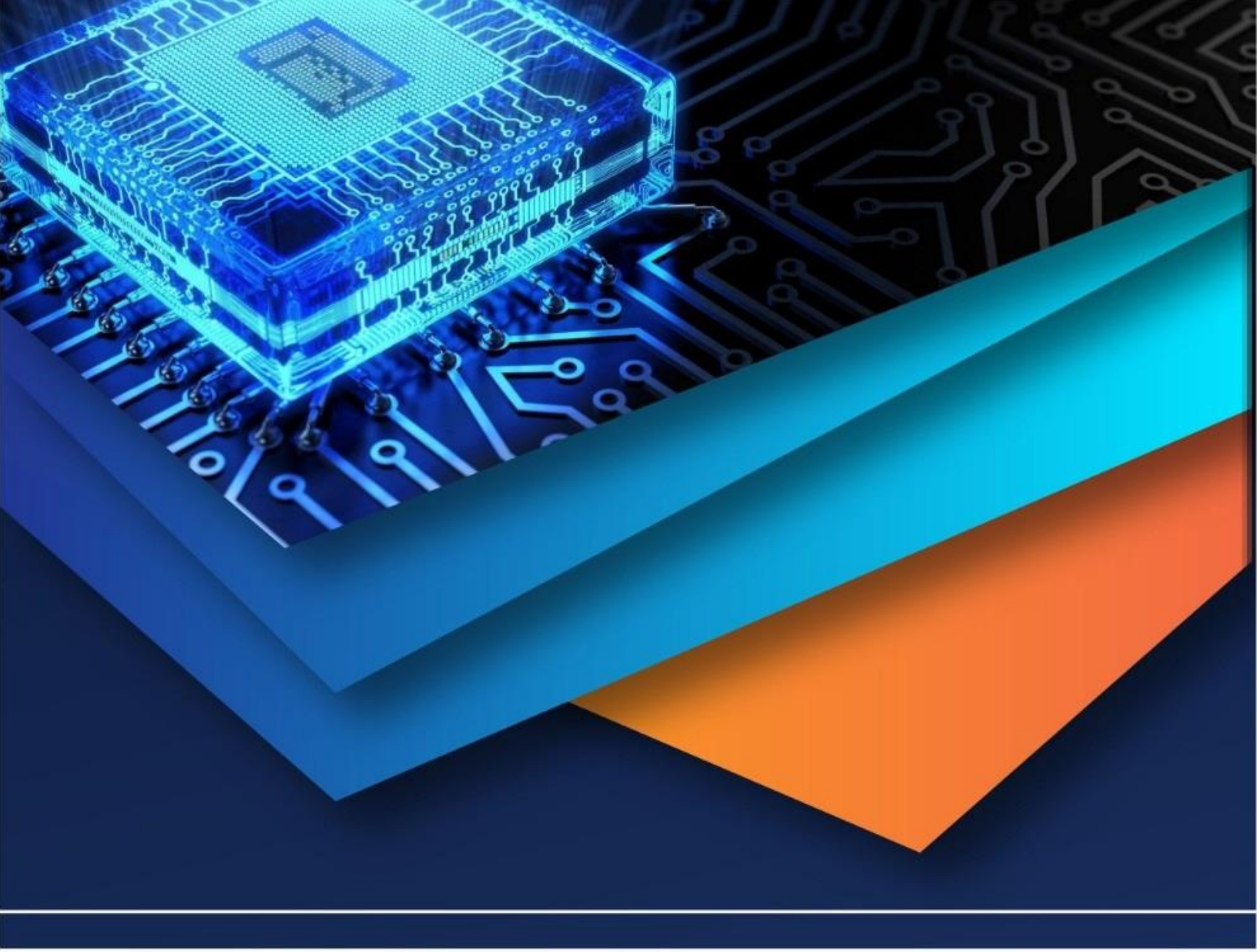
D. No cracks were found on the surface of the pure HAp coatings while reinforced coatings have some minor cracks on their surface.

V. ACKNOWLEDGEMENT

The author Vikas Rattan acknowledges Inder Kumar Gujral Punjab Technical University, Kapurthala, Punjab, India and Anod Plasma Limited, Kanpur, India for providing the facilities to carry out this research work.

REFERENCES

[1] Sim, P. Cheang, M. Liang, and K. Khor, "Injection moulding of hydroxyapatite composites," Journal of Materials Processing Technology, vol. 69, pp. 75–78, 1997.
[2] H. Li, K. Khor, and P. Cheang, "Titanium dioxide reinforced hydroxyapatite coatings deposited by high velocity oxy-fuel (HVOF) spray," Biomaterials, vol. 23, pp. 85–91, 2002.
[3] C. Yip, K. Khor, N. Loh, and P. Cheang, "Thermal spraying of Ti□6Al□4V/hydroxyapatite composites coatings: powder processing and post-spray treatment," Journal of Materials Processing Technology, vol. 65, pp. 73–79, 1997.
[4] H. Li, K. Khor, and P. Cheang, "Effect of the powders' melting state on the properties of HVOF sprayed hydroxyapatite coatings," Materials Science and Engineering: A, vol. 293, pp. 71–80, 2000.
[5] M. F. Hasan, J. Wang, and C. C. Berndt, "Effect of Power and Stand-Off Distance on Plasma Sprayed Hydroxyapatite Coatings," Materials and Manufacturing Processes, vol. 28,pp. 1279–1285, Feb. 2013.
[6] M. Mittal, S. Nath, and S. Prakash, "Improvement in mechanical properties of plasma sprayed hydroxyapatite coatings by Al2O3 reinforcement," Materials Science and Engineering: C, vol. 33, pp. 2838–2845, 2013.
[7] H. Ji and P. Marquis, "Preparation and characterization of Al2O3 reinforced hydroxyapatite," Biomaterials, vol. 13, pp. 744–748, 1992.
[8] G. Singh, S. Singh, and S. Prakash, "Surface characterization of plasma sprayed pure and reinforced hydroxyapatite coating on Ti6Al4V alloy," Surface and Coatings Technology, vol. 205, pp. 4814–4820, 2011.
[9] V. Sergo, O. Sbaizero, and D. R. Clarke, "Mechanical and chemical consequences of the residual stresses in plasma sprayed hydroxyapatite coatings," Biomaterials, vol. 18, pp. 477–482, 1997.
[10] L. Ponsonnet, K. Reybier, N. Jaffrezic, V. Comte, C. Lagneau, M. Lissac, and C. Martelet, "Relationship between surface properties (roughness, wettability) of titanium and titanium alloys and cell behaviour," Materials Science and Engineering: C, vol. 23,pp. 551–560, 2003.
[11] R.B. Heimann, "Thermal spraying of biomaterials," Surf. Coatings Technol, vol. 201, pp. 2012–2019, 2006.
[12] R.B. Heimann, "Plasma-Sprayed Hydroxylapatite-Based Coatings : Chemical , Mechanical , Microstructural , and Biomedical Properties," Journal of Thermal Spray Technology, vol. 25, pp. 827–850, 2016.
[13] A.Dey, A.K. Mukhopadhyay, S. Gangadharan, M.K. Sinha, and D. Basu, "Development of Hydroxyapatite Coating by Microplasma Spraying," Materials and . Manufacturing . Processes, vol. 24, pp. 1321–1330, 2009.
[14] R.B. Heimann, " Structure, properties, and biomedical performance of osteoconductive bioceramic coatings,"Surface and . Coatings Technology, vol. 233, pp. 27–38, 2013.
[15] S. Vahabzadeh, M. Roy, A. Bandyopadhyay, and S. Bose, "Phase stability and biological property evaluation of plasma sprayed hydroxyapatite coatings for orthopedic and dental applications," Acta Biomaterialia, vol. 17, pp. 47–55, 2015
[16] T.M. Sridhar, U. Kamachi Mudali, and M. Subbaiyan, " Sintering atmosphere and temperature effects on hydroxyapatite coated type 316L stainless steel," Corrosion Sciience, vol. 45, pp. 2337–2359, 2003.
[17] C.H. Quek, K.A. Khor, and P. Cheang, "Influence of processing parameters in the plasma spraying of hydroxyapatite/Ti-Al-4V composite coatings," Journal of Materials Processing Technology vol. 89-90, pp. 550-555.1999.
[18] Bang-Yen. Chou, and Edward. Chang, "Microstructural characterization of plasma –sprayed hydroxyapatite -10wt% ZrO_2 composite coating on titanium," Biomaterials, vol. 20, pp. 1823-1832, 1999.



10.22214/IJRASET

45.98

IMPACT FACTOR: 7.129

IMPACT FACTOR: 7.429

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY