

**CHARACTERISATION OF TRICALCIUM PHOSPHATE COATING
OVER SS316L FOR BIOMEDICAL IMPLANTS**

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Engineering degree in

Mechanical Engineering

By

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DEPARTMENT OF MECHANICAL ENGINEERING

SCHOOL OF MECHANICAL ENGINEERING

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade “A” by NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

APRIL – 2021



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DEPARTMENT OF MECHANICAL ENGINEERING
BONAFIDE CERTIFICATE

This is to certify that this Project Report is the Bonafide work of **UTHAYAKUMAR.S (37150197)** and **VIGNESHWAR.G (37150212)** who carried out the project entitled **"CHARACTERISATION OF TRICALCIUM PHOSPHATE COATING OVER SS316L FOR BIOMEDICAL IMPLANTS"** under my supervision from October 2020 to March 2021

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DECLARATION

We, **UTHAYAKUMAR.S (37150197)** and **VIGNESHWAR.G (37150212)** hereby declare that the Project Report entitled **“CHARACTERISATION OF TRICALCIUM PHOSPHATE COATING OVER SS316L FOR BIOMEDICAL IMPLANTS”** done by us under guidance of **Dr. M. SANGEETHA** is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering degree in Mechanical.

1.

2.

DATE:

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I convey my thanks to **Dr. S. PRAKASH M.E. Ph.D.**, Dean, School of Mechanical engineering and **Dr G. Arun Kumar M.E. Ph.D.**, Head of the Department, Dept. of Mechanical Engineering for providing me necessary support and details at the right time during the progressive reviews.

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ABSTRACT

The project is about coating the Stainless steel (SS316L) for the Bio-medical applications like Biomedical Implants, Bio-compactable materials, etc. The process of coating tri-calcium phosphate over SS316L substrates is done by the process of Plasma spraying method. Stainless steel SS316 is chromium-nickel based steel that possess increased levels of resistance against several bimolecular substances, because of the addition of molybdenum in its composition. Molybdenum present in the SS316L allows to be more resistant to corrosion overall and in specific resistance against chlorine pitting. It is coated with tri-calcium phosphate. The Tri-calcium phosphate is also known as TCP. The calcium salt of phosphoric acid is Tri-calcium phosphate. The chemical formula for the Tri-calcium phosphate used is $\text{Ca}_3(\text{PO}_4)_2$. It is also available in various formats like tribasic calcium phosphate and bone phosphate of lime (BPL) and is a white solid of low solubility. Most commercial samples of “tri-calcium phosphate” are in fact hydroxyapatite. The coated material is used for Bio-medical implants, Bio-compactable materials, etc.

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CHAPTER 1

INTRODUCTION

Coating is a process of creating several thin layers of deposition of the dispersion, solution or liquid monomer. They are solidified by means of drying or curing. There are number of coating process are available, based on the material used and the application. In our experiment the application we are about to consider is biomedical implementation and related applications. So here we considered the chemical conversion coating process, which means the base material is coated using different chemical in the form of solution, liquid or the solid component is converted into a thin adhering coating under the soluble compound. The coatings are mainly applied to protect the element against corrosion, to increase the life span of the equipment, to improve the adherence of other coatings, for lubrication purposes and in some cases the coating is just for good appearance. The coating process was done by using the plasma spray process. Plasma spraying process is defined as the process that combines the particle melting, quenching and consolidation in a single operation. In the spray setup, the injection of the material to be deposited into the plasma jet is introduced, where they are melted and that the molten particles strike the substrate surface forming the required adherent coating. For the plasma spraying process the material can be fed in the form of powder (the powder spraying) or the wire (wire spraying)

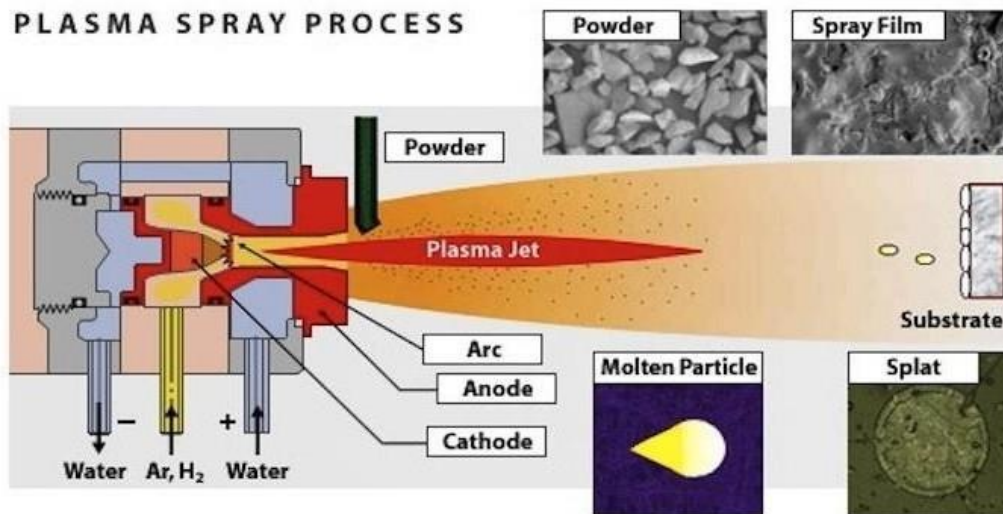


Fig.1 Plasma spraying process

The ideal bio-medical components we considered are all made up of the stainless steel. There are different methods available for the steel coating. For our experiment, we have considered the chemical conversion coating process of phosphate conversion coating. The Phosphate conversion coating is theoretically considered as the most suitable method for the stainless steel parts that it creates a thin adhering layer of iron, zinc or manganese phosphates. That it will be result in the improvement of corrosion resistance, improvement in the lubrication and other benefits. This process is also available as phosphate coating, phosphatization, parkerizing, phosphatizing or phosphating. The different types of the phosphate coatings are manganese, iron and zinc.

1.1 Stainless steel

Stainless steel 316 (SS316L) is sometimes referred to as marine grade stainless steel and also known as the second most common austenitic stainless steel. The SAE grade SS316L is an austenitic chromium-nickel stainless steel which means it has high amounts of chromium and nickel. Also The SS316L containing deliberate amount of molybdenum which increases general corrosion resistance. Especially improves its pitting resistance to chloride ion solutions, so it is resistance

to the toxic salts. It contains silicon, Molybdenum, chromium, manganese, and carbon, with the majority of the composition is being iron. The SS316L is the extra-low carbon version of SS316 that minimizes harmful carbide precipitation during various scenarios like welding. Both the SS316 and SS316L have the highest resistance to corrosion against sulphuric, hydrochloric, formic, acetic and the tartaric acids; as well as it is against acid sulphates and alkaline chlorides.

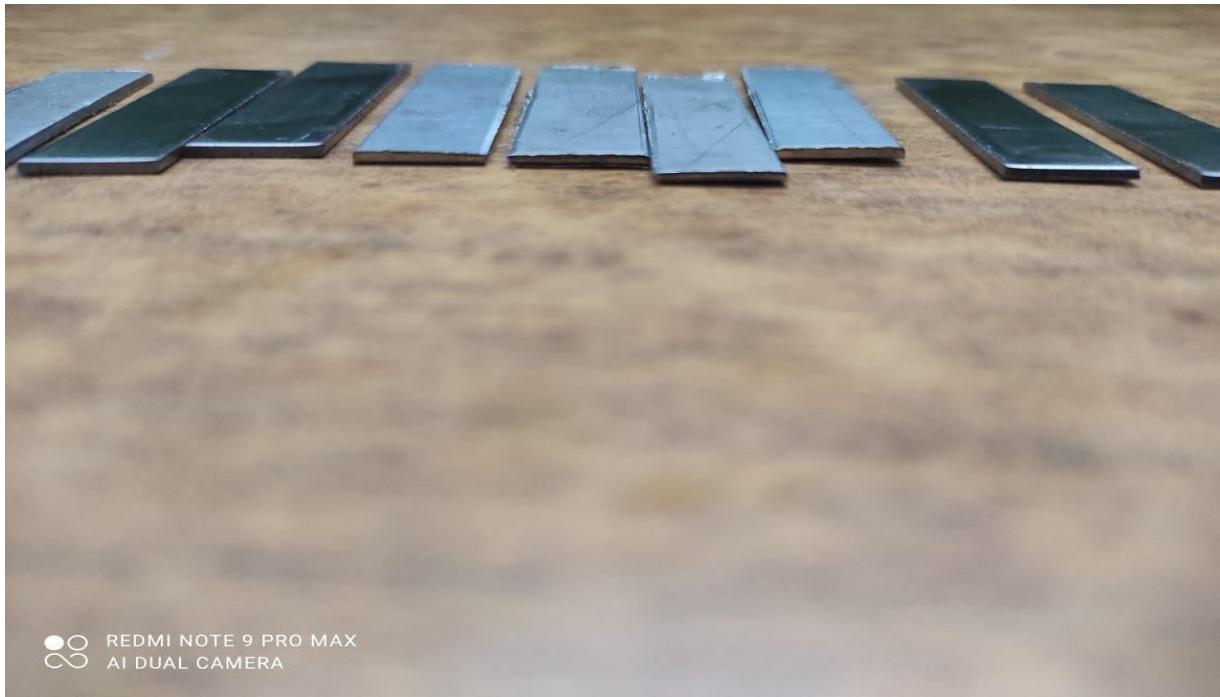


Fig.2 Pieces of SS316L

1.1.1 Properties and composition of SS316L

Density: 0.799 g/cm³ Specific Heat: 0.50 kJ/kg-K (0-100 degrees Celsius), Electric Resistivity: 74 microhm-cm (20 degrees Celsius) Thermal conductivity: 16.2 W/m-k (100 degrees celsius).Modulus of Elasticity (MPa): 193 x 10³ in tension Melting Range: 2,500 – 2,550 degrees Fahrenheit (1,371 – 1,399 degrees Celsius).

Table.1 Chemical composition of SS316L

Element	Chemical composition % (max)
Carbon	0.030
Manganese	2.00
Phosphorus	0.045
Sulphur	0.03
Silicon	0.75
Chromium	16.00-18.00
Nickel	10.00-14.00
Molybdenum	2.00-3.00
Nitrogen	0.10

Common uses for type 316 stainless steel include in the construction of

- Exhaust manifolds,
- Furnace parts,
- Heat exchangers,
- Jet engine parts,
- Pharmaceutical and photographic equipment,
- Valve and pump parts,
- Chemical processing equipment,
- Tank,
- Evaporators.

It is also used in pulp, paper, and textile processing equipment and any parts exposed to marine environments.

1.1.2 Properties of tricalcium phosphate

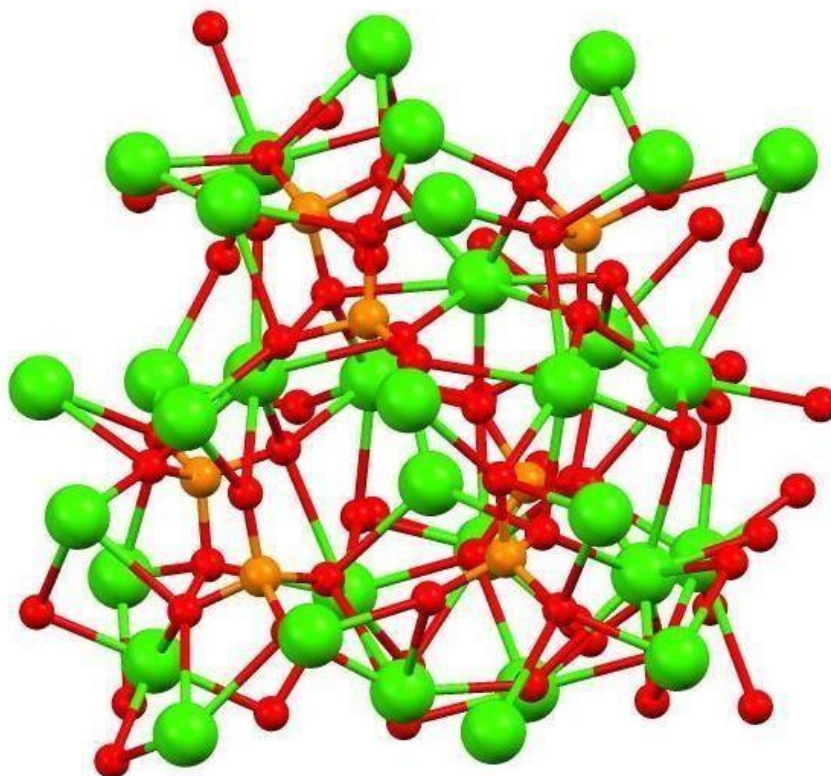


Fig.3 Molecular bonding of tricalcium phosphate

Table.2 Properties of tricalcium phosphate

Properties	
Chemical formula	$\text{Ca}_3(\text{PO}_4)_2$
Molar mass	310.18
Appearance	White amorphous powder
Density	3.14 g/cm^3
Melting point	Liquefies under high pressure at 1670 K (1391 °C)
Solubility in water	0.002 g/100 g

1.2 Bio-medical implants

The Bio-medical implants are the man-made devices that are fabricated by different methods but mostly the modern bio-medical implants are made up of

additive manufacturing methods. The fabricated implantation will be inside the body to replace or support a biological structure, together with delivering drugs and monitoring body functions. They can remain in the body for some temporary period or permanently. The latest biomedical implantation includes sensory devices like prosthetics (artificial functional organs like hand, leg, etc.), sutures and wound dressings, spinal and dental implants, cardiovascular (related to heart) devices such as vascular grafts, stent, heart valves, pacemakers, retinal implants, cosmetic and structural implants such as rods, braces, craniofacial, hip and knee replacements. Also as ophthalmic (eye) devices including glasses and contact lenses as well as insulin delivery devices.

The biomedical implants must be made up of bioactive materials, since it will be associated with the human muscles, tissues and other internal organs as well. The term “bioactive” means the materials that can be affect its surrounding tissue chemically or biologically. Scaffolds are made up of synthetic or natural polymers were developed to regenerate the damaged or deteriorated tissues and also for several drugs deliver for some significant locations. Scaffolds can include molecules that promote a biological response in the region where they are implanted, Also included in promote the healing and regeneration.

There are several risks related to the surgery during the placement and removal of the implant, include infection and implant failure. Also there are several side effects like inflammation reaction against the material or rejection needs to be taken into the consideration. There are several biomaterials available such as Bio ceramics,

1.2.1 Bio ceramics

Ceramics are the physical element which is chemically inert and possess low thermal and electric conductivity as well as physical properties, which make them a suitable material glass for biomedical implants. Bio-ceramics possess the mechanical properties which is almost equivalent to the native bone. But the ceramics are has high brittleness.

1.2.2 Polymers

A variety of natural and synthetic polymers are used as hip and joint

replacements, soft tissue transplant, denture, facial prostheses as well as medical adhesives, sealants and coatings. Polymers are commonly selected based on their mechanical characteristics, composition and their physical properties. **Natural polymers** non-toxic and exhibit protein binding-sites and biochemical which are very important for the tissue regeneration and also the natural polymers possess similar properties to the native tissues. **Synthetic polymers** are developed with tailored physical and chemical properties depending on the desired application to overcome limitations of natural polymers.

1.2.3 Metals and alloys;

Metals are commonly used as load-bearing orthopaedic implants such as wires screws, fixation plates, and artificial joints for hips, knees, shoulders and ankles, as well as for dental and for other joints. Since the mechanical properties, their easy processing and their possibility to sterilise them, metals and alloys are ideal materials for biomedical implants. Novel magnesium alloys, titanium and titanium alloys are the several alloys used in the biomedical implantations.

1.3 Considerations

Biomedical implantation involves in the different consideration such as type of material used, cost, life span of the implant, most important thing while designing a biomedical implantation is that, could we use the implant for human being / will it be accepted by the human body, like that there are number of considerations are taken into account. In the following topic we will be briefly looking into the implantation considerations.

1.3.1 Surface properties

Surface properties are an ideal consideration for bio-medical implantation. The implanted object will be in contact with the cells, muscles, bones, etc. So the surface properties will be responsible for cell adhesion, cellular and tissue responses. And in most of the implantation cases the object should have considered the roughness, texture and the orientation of the irregularities and the tension is determined by the wetting fluid such as blood or water. The texture of the surface will be defined from concave or convex. The concavity of the surface will because of the treatments like hydroxyapatite coatings and the convex property of the surfaces are

created through the etching and the blasting process. And the surface properties are also directionally dependent like isotropic or anisotropic. In most of the cases the object should be highly porous surfaces on additively manufactured implants which in turn into the benefits like good ingrowth of bone tissue. By means of using the calcium tri-phosphate spray coating process we can reduce the capillary action on blood and relevant growth factors or bone cells is inhibited.

1.3.2 Metal corrosion

The life span of the implantation element will be decided based on the various factor, Corrosion is one of the major factors to be considered during the design of the implant and also if the corrosion is happening while the implant is placed inside the skin means it will create some unwanted or bad effects like inflammation, thus it will be result in the failure of the biomedical implantations. Actually the meaning of the implant corrosion is that the involuntary breakdown of the metals by means of the electrochemical reactions. The function of corrosion will be like the loss of ions from the metal surface in an acidic, an alkaline or a neutral environment. There are several biomedical implantation corrosions are available such as **crevice corrosion** which is commonly occurs in narrow regions in the biomedical implants because of the metal ions creates the localised positive charge, this crevice corrosions will occurs in the objects which has interfaces between screws/plates and bone; **pitting corrosion** which will occurs in the implants with the small surface pit due to the metal ions reacts with the chloride ions resulting in rough surfaces, it will happen in the object which will in the orthopaedic and the dental implants; **Galvanic corrosion** is occurs due to the electrical gradient reaction between Co-Cr alloys, Ni-Cr, Ag-Pd, Au-ternary Ti. This corrosion will be in the oral/dental implants and screws and nuts; **Corrosion fatigue and fretting** is generally occurs due to the cyclic stress, which will be happen in the bone cement, femoral implants, bone plates & screws at the bone-stem interface and stem-cement interfaces of modular hip implant.

1.3.3 Polymer degradation

The Polymer degradation is also called as the biodegradation which is occurred because of the process called as hydrolysis. The polymer surface has the ability to react or to attack by the organisms which will secrete enzymes breaking down into the ester bonds in the macromolecules. And the end product which is the

smaller polymer molecules will be further converted into the carbon dioxide and water. This process varies for every polymer but all the polymers will lose their mechanical integrity at the end of the degradation process.

1.3.4 Biocompatibility

The biocompatibility is defined as the object or the element which has some desired response to its biological surrounding which is depends on biodegradability and to the corrosion. There is a standard published by the international standard organisation **ISO 10993** standard series elements are used to assess the biocompatibility of the medical grade materials and the medical devices. The ISO 10993 standard is created by various testing such as toxicity, irritation, cytotoxicity, sensitization, implantation and the biodegradation. In most cases the materials which meets these above mentioned criteria are all noble metals and titanium, their alloys, cobalt based alloys, in some cases alumina, zirconia, quartz, fused silica, bio glass, silicon. The biocompatibility polymers are epoxies, silicones, polyurethanes, polyimides, silicon-polyimides, polycyclic-olefins, silicon-carbons and the liquid crystal polymers.

1.3.5 Foreign body response:

Foreign body response means the immune reaction of the human body towards the implanted materials. There are certain inflammatory reactions which will happen in the response to the surgical implantation of biodegradable or to the non-biodegradable materials present in the medical devices or implants.

CHAPTER 2

LITERATURE SURVEY

The biomedical implantation, bioactive materials and biodegradable, non-biodegradable materials. Selection consideration of the materials. The detailed biomedical implantation results in the human body and the reactions takes place while taking a metal as implant material. Benefits and demerits of using the already existing coating materials are explained clearly. There are various biomaterials available such as bioceramics, natural polymers, synthetic polymers, metals and their alloys. The implant properties also explained such as the biocompatibility, surface properties, metal corrosion & the types of metal corrosion, polymer degradation and the foreign body response (FBR) taken from Andrea Domingues Goncalves et al. [1]

The review paper is about the significant research and development in the field of bio-medical implants has evoked the scope to treat a broad range of orthopaedic ailments. This includes the fracture fixation, total bone replacement, dental screws, and others. The coating of the nanomaterial has been employed to enhance the antibacterial activities of the bio-implant. The titanium and titanium alloys merits and demerits are explained with the mechanical properties taken from Harbhajan Ahirwar et al. [2]

The research paper is about the usage of the tricalcium phosphate as an osteoconductive scaffold (biomaterials) for the potential clinical application for the bone regeneration. The tricalcium phosphate was studied for the tissue engineering principles taken from Myat Nyan et al. [3]

Processing and characterization of tricalcium phosphate (TCP) laminates the fabricated by stacking the individual TCP sheets for the bone-tissue engineering taken from Yasuhiro Tanimoto et al. [4]

This paper is an attempt to give the details of the variants of surfacing techniques, bonding mechanism, factors selection of process parameters to apply on different combinations of materials and confirming the same with experimental test

results. Effect of process parameters on the responses like width, height, surface roughness, tensile strength shear strength and ratio of tensile strength and shear strength are discussed in details. Testing of deposits for quality evaluation also carried out such as visual inspection, adhesion test, and microstructure and hardness distribution to find potential industrial applications taken from S. Ravisekhar et al. [5]

The surface treatment for any metals is step by step illustrated and the suitable method for coating of the SS316 is explained. The process and the types of plasma spray coating and the thermal spray coating is explained in detail is taken from Joseph Edwards et al. [6]

CHAPTER 3

AIM AND SCOPE OF THE PRESENT INVESTIGATION

This project is about to replace the titanium in the biomedical implantation. In the biomedical fields, they are used for bone repairing and even for the production of artificial limbs. The tricalcium phosphate over SS316L can also be provided to the required calcium for the broken bone growth, this helps for faster bone growth. The SS316L is 60% lesser than the titanium in terms of weight. This coating process can be done by plasma spraying process. This can also be used in food processing, chemical processing and the photography, etc.

Chapter 4

MATERIALS AND EXPERIMENTAL METHODS

4.1 Materials

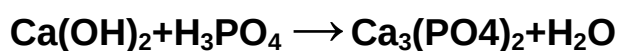
The substrate plate used here is SS316L. The material used as the coating is tricalcium phosphate which is white amorphous powder. The stainless steel SS316L is first designed using CAD software. Then it is manufactured using machining process. The manufactured element is then introduced into the plasma spray coating process. There are ten number of samples are manufactured and plasma spray coated and ready for the testing.

Table.3 Chemical composition of SS316L

Materials	C	Mn	P	S	Si	Cr	Ni	Mo	N	Fe
% of composition	0.030	2	0.045	0.03	0.75	16	12	2-3	0.10	reminder

4.2 Tricalcium phosphate

The tricalcium phosphate is actually the calcium salt of the phosphoric acid, which is represented in the chemical formula of $\text{Ca}_3(\text{PO}_4)_2$. The tricalcium phosphate is generally looking like white amorphous or crystalline powder that has no taste and does not possess any particular taste. The tricalcium phosphate is derived from the calcium products such as bones, milk, teeth, and ground. But for the experimental purpose we just purchased the tricalcium phosphate powder from the shop. The



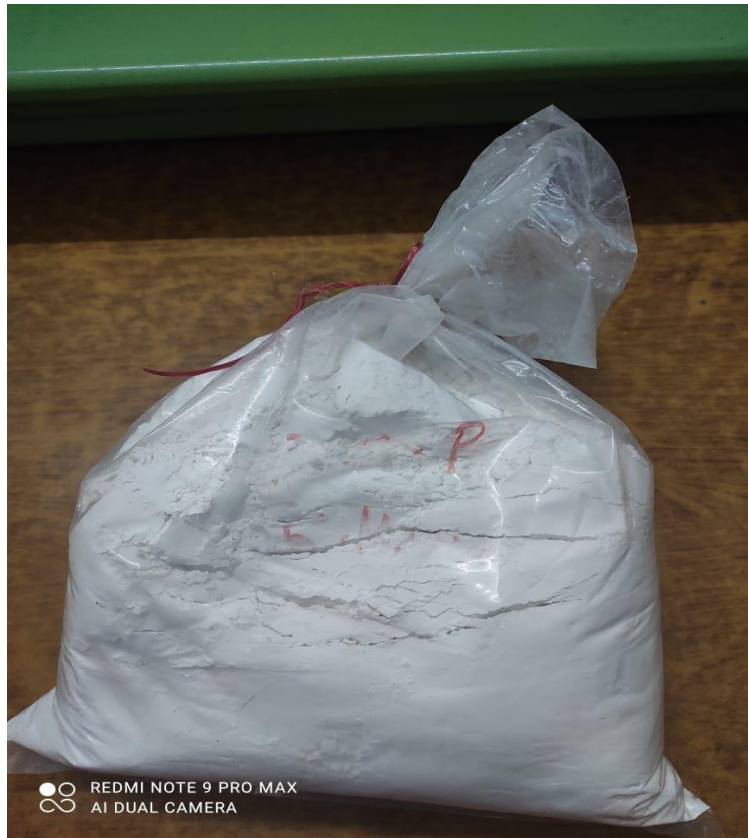


Fig.4 Tricalcium phosphate powder

4.3 Experimental process and parameters

Plasma spraying process is a thermal spray coating technology that the heat ionizes the gas. The gas itself becomes the heat source for this process. The object (SS316L) is placed inside the chuck and the process get started once the plasma is get ready. There after the coating material melts and is propelled onto the substrate's surface where it solidifies immediately. Usually the process can be applied under the ambient pressure or in vacuum under an inert gas. It is particularly ideal for higher melting point material such as ceramic coatings which performs extremely well in high temperature corrosion resistance and hardness. The object (the biomedical implant) is placed on the bed. The injection of the tricalcium phosphate material which is to be deposited into the plasma jet, where they are melted and the molten particles are strike the substrate surface forming the adherent coating.

CHAPTER 5

RESULTS AND DISCUSSION

The biomedical implant sample is prepared for cleaning the surface, plasma spray coated and it was undergone various testing as follows. In the beginning itself the implant which is about to coat is clear and we determined in each and every step. The test results are all almost equivalent to the already existing implants. There are several improvements in the final outcome than the titanium in terms of weight and other certain category, which are all briefed bellow.

5.1 Primary inspections

The coating quality of the calcium deposits are primarily evaluated at the shop floor after the plasma spray deposition. These are for the qualitative analysis, to address the status of the deposit whether it is acceptable or not. The NDT techniques such as the appearance and adhesion tests such as lifting test, impact test and the chisel and hammer test are all performed for the evaluation of the bonding quality and for the testing of the spray coating adhesiveness.

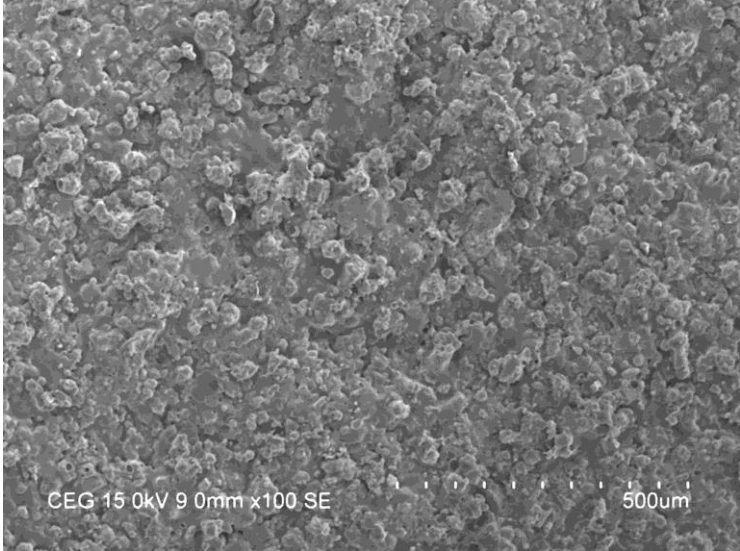
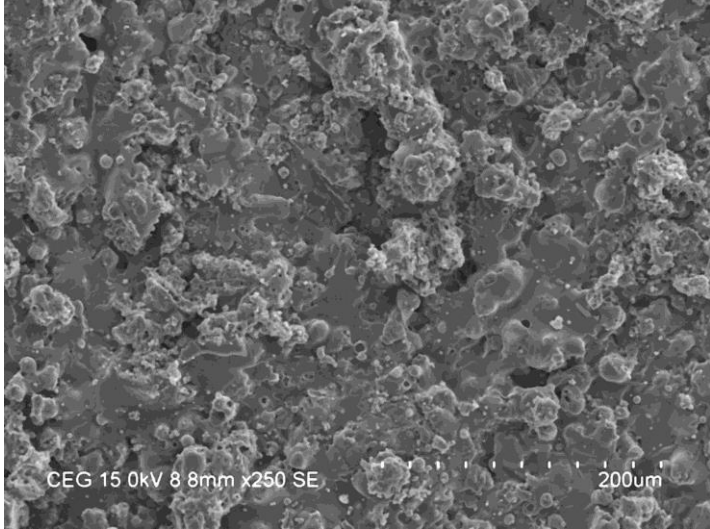
5.2 Visual inspection

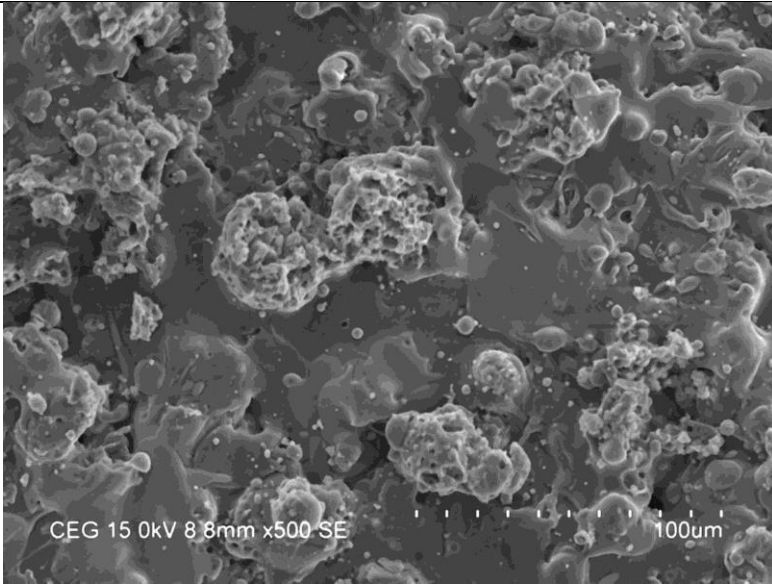
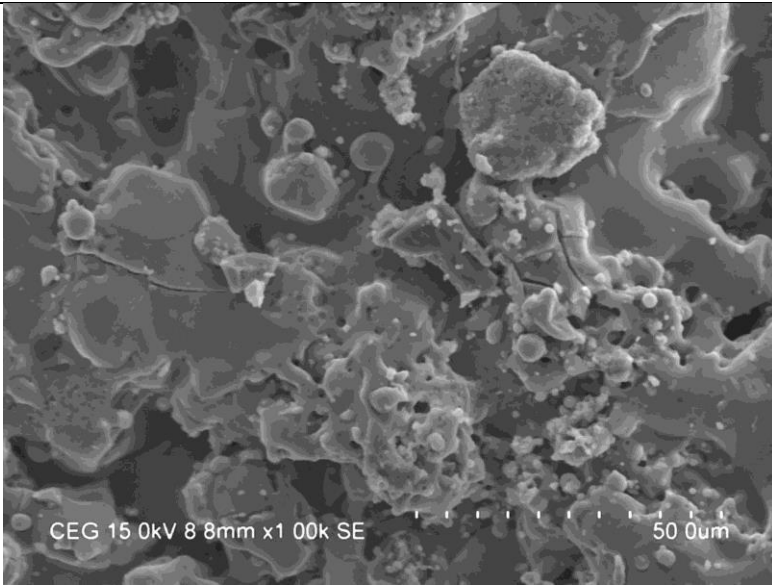
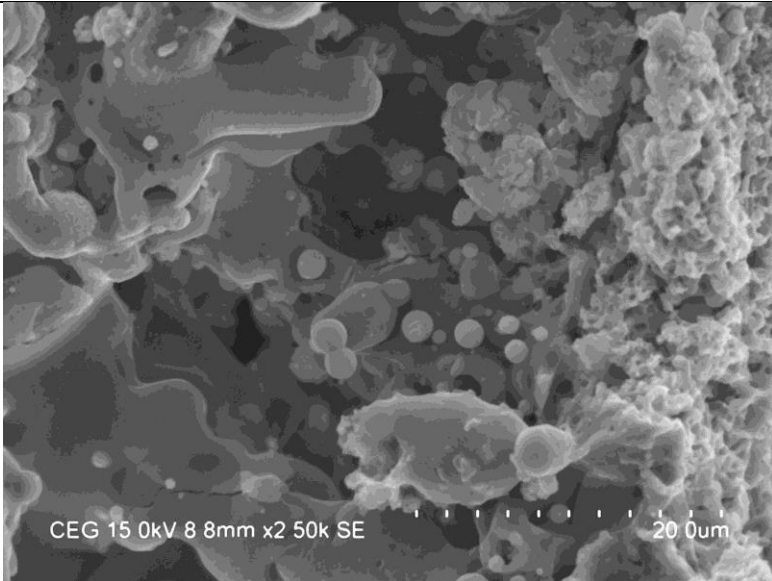
The deposit is inspected for the physical appearance and the damage. It is also inspected by the visual inspection using magnifying glass and scanning electron microscope (SEM) for the visible defects such as the blisters, pits, roughness and the cracks and the undercut areas.

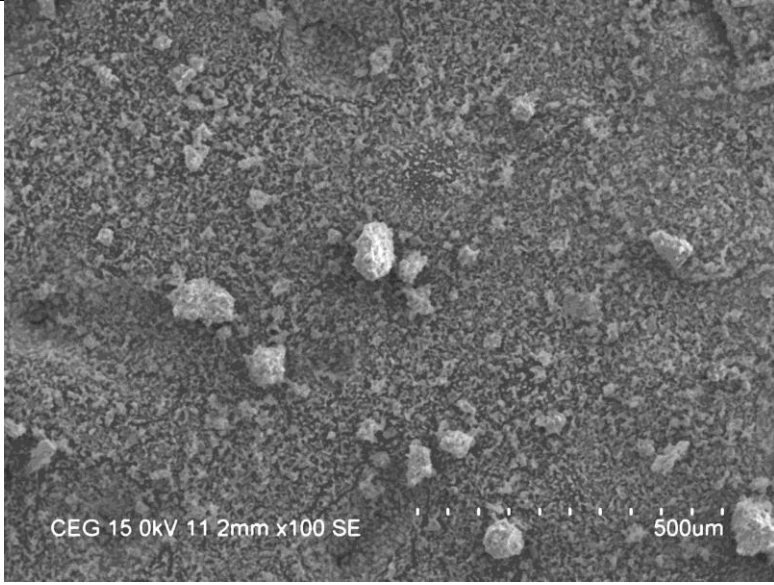
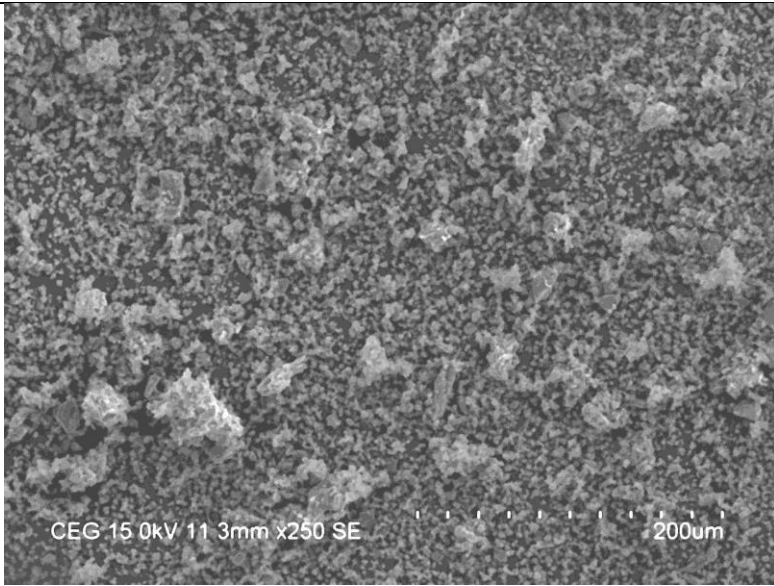
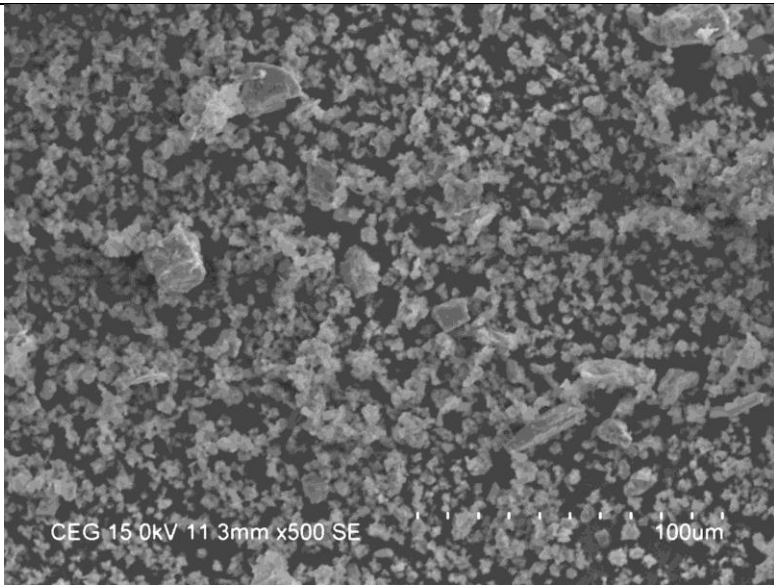
5.3 Scanning electron microscope (SEM)

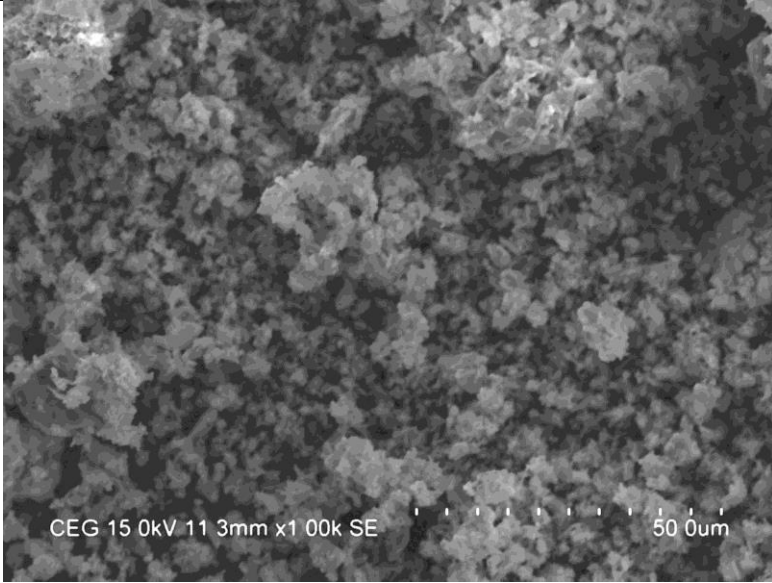
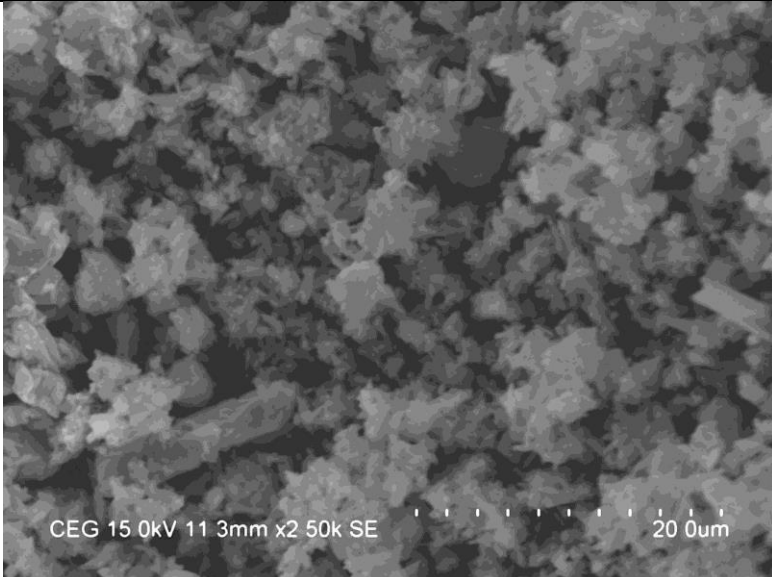
The scanning electron microscope is used to produces the images of the samples by doing the scanning of the surface with the focused beam of the electrons. The electron from scanning electron microscope will be interacted with the atoms in the sample which is to be examined. The SEM is accurate up to nanometre. In our case there are ten different cases is considered by means of changing the area to be scanned and the accuracy of the scanning.

Table.4 Scanning electron microscope output

S. No.	SEM image	Output
1		MicronMarker=50000 Condition Vacc=15kV Mag=x1.00k WD=8.8mm
2		MicronMarker=100000 Condition Vacc=15kV Mag=x500 WD=8.8mm

3		MicronMarker=100000 Condition Vacc=15kV Mag=x500 WD=8.8mm
4		MicronMarker=50000 Condition Vacc=15kV Mag=x1.00k WD=8.8mm
5		MicronMarker=20000 Condition Vacc=15kV Mag=x2.50k WD=8.8mm

6		MicronMarker=500000 Condition Vacc=15kV Mag=x100 WD=11.2mm
7		MicronMarker=200000 Condition Vacc=15kV Mag=x250 WD=11.3mm
8		MicronMarker=100000 Condition Vacc=15kV Mag=x500 WD=11.3mm

9		MicronMarker=50000 Condition Vacc=15kV Mag=x1.00k WD=11.3mm
10		MicronMarker=20000 Condition Vacc=15kV Mag=x2.50k WD=11.3mm

5.4 Tensile strength

The study was taken, and the materials are cut to 2mm thickness, 32 mm gauge length and 12mm width with the coating material. The specimen is prepared in such a way that the deposited material can be separated only by tensile load. The tensile load is then noted and the bonding strength is then found. The tensile load shows the bonding strength between the substrate and the Tricalcium phosphate material.

Table. 5 General Tensile Test –S.I. Units

Crosshead Speed	2.0000 mm/min
Second Speed	0.0000 mm/min
Sample Rate (pts/secs)	10.0000
Full Scale Load Range	50.0000 kN
Interface Type	2300/3300
Third Speed	0.0000 mm/min
Sample Identification	1
General Tensile Test	S.I. Units
Test type	Tensile
Test Date	Monday, March 15, 2021
Humidity (%)	50
Temperature	73 F
Series IX Automated Materials Testing System	8.33.00



Fig. 5 Universal Testing Machine

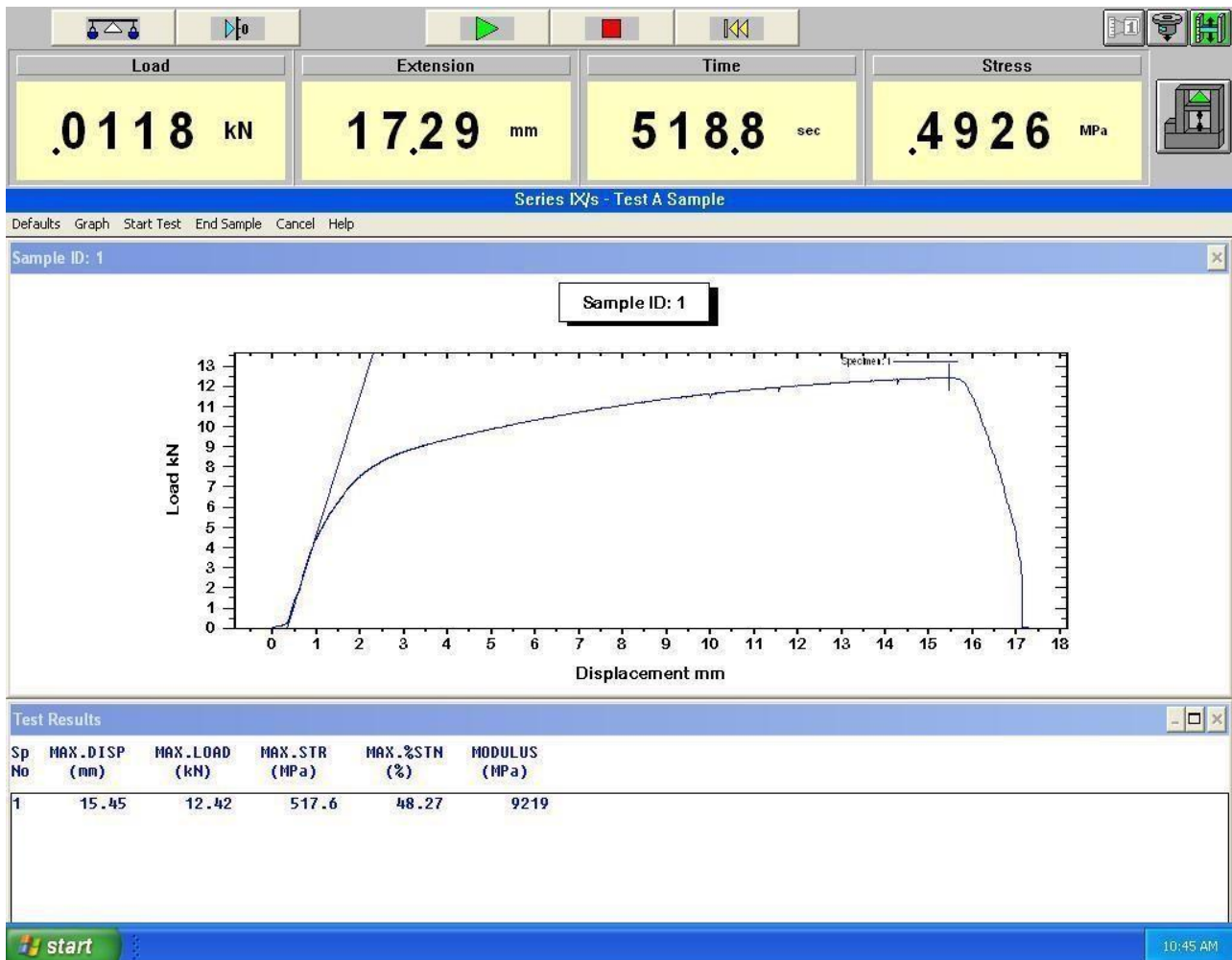


Fig.6 Tensile strength Graph

5.5 Element Energy Dispersive Spectroscopy

The element energy dispersive spectroscopy is otherwise referred as the energy dispersive X-Ray analysis. This EDAX technique is used to identify the elemental composition of materials. The Element energy dispersive spectroscopy is a non-destructive testing method and the specimens of the interest can be examined in a situation with little or no sample preparation. The EDS systems are the simple mechanism which is added or attached to the electron microscopy instruments like Scanning Electron Microscopy (SEM) or the Transmission Electron Microscopy (TEM). The result of the material composition in terms of net count, weight in percentage and the number of atoms in percentage.

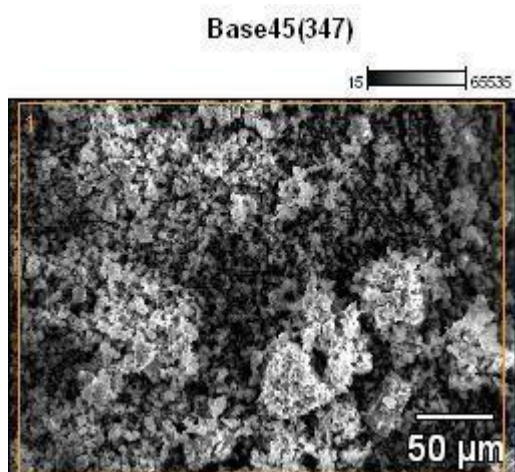


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Accelerating Voltage: 15.0 kV

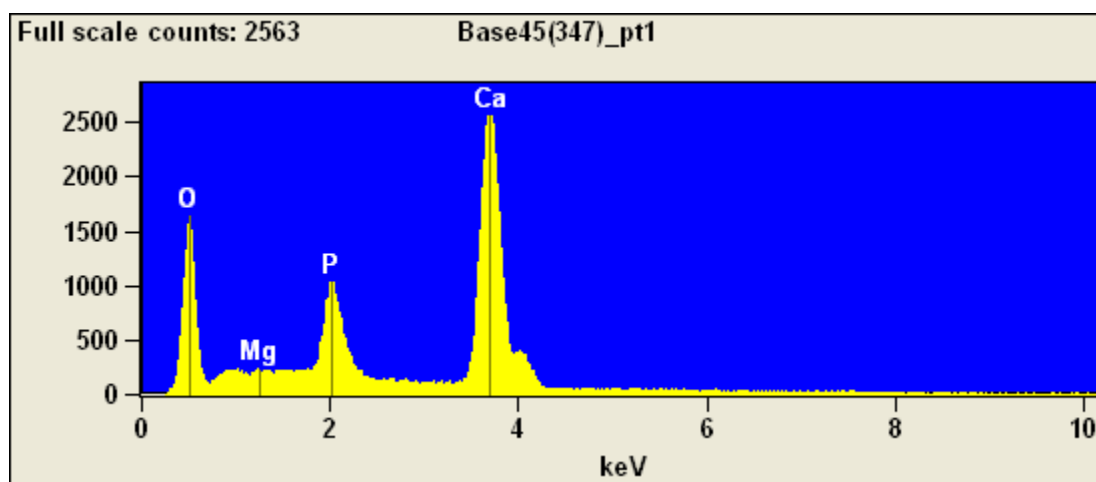


Fig. 7 Element energy dispersive spectroscopy of Tricalcium phosphate

This graph represents relationship between the full scale counts of the element present in the powder of Tricalcium phosphate in terms of Kiloelectron Volts KeV.

Table 6. Net Counts of Tri calcium phosphate

	O	Mg	P	Ca
Base45(347)_pt1	23285	564	13150	51122
Weight %				
Base45(347)_pt1	52.54	0.28	6.48	40.69
Atom %				
Base45(347)_pt1	72.65	0.26	4.63	22.46

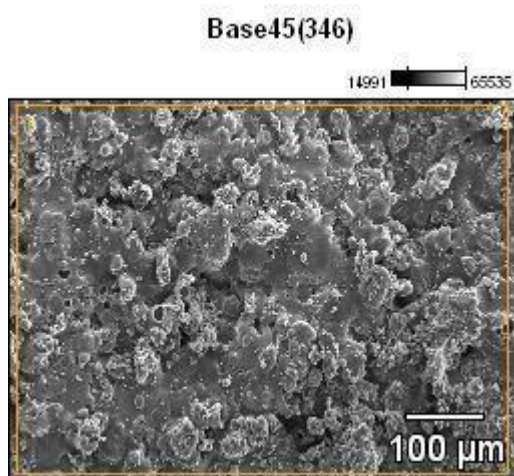


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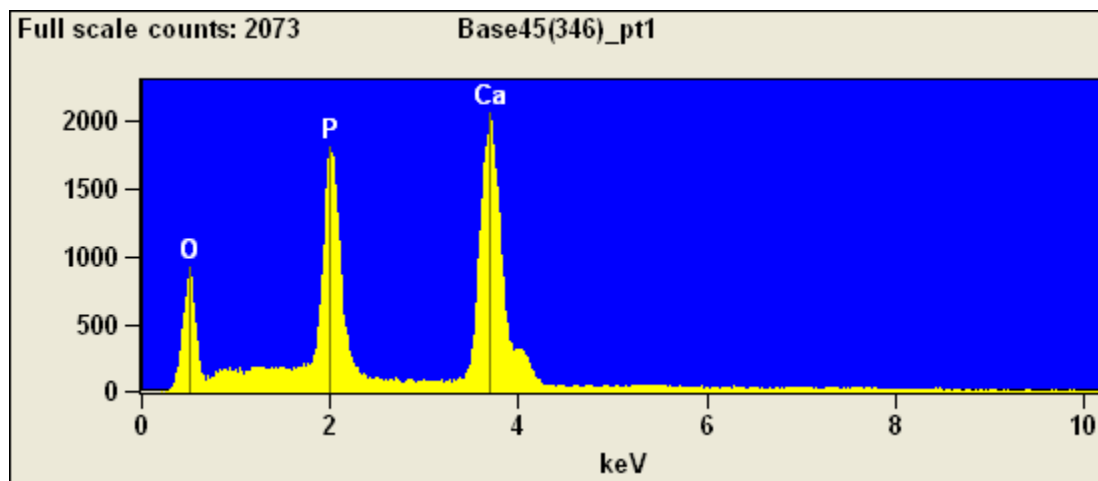


Fig. 8 Element energy dispersive spectroscopy of SS316L

This graph represents relationship between the full scale counts of the element present in the coating of Tricalcium phosphate over the stainless steel SS316L in terms of Kiloelectron Volts KeV

Table 7. Net Counts of SS316L

	O	P	Ca
Base45(346)_pt1	12687	28824	39738
Weight %			
Base45(346)_pt1	40.96	17.87	41.17
Atom %			
Base45(346)_pt1	61.48	13.86	24.67

5.6 Electrochemical corrosion test

Since the object we are designing is a biomedical implant, we should have to do an electrochemical corrosion test. All the corrosion occurs because of the electrochemical process of oxidation and reduction reactions are called as electrochemical corrosion. If the corrosion takes place with the release of electrons from the metals means it is oxidation and if the process involves in the gain of the elements means, it is called as the reduction process under the presence of corroding solution. Because of the flow of electrons (current) in the corrosion reaction, we can measure and also can be controlled electronically.

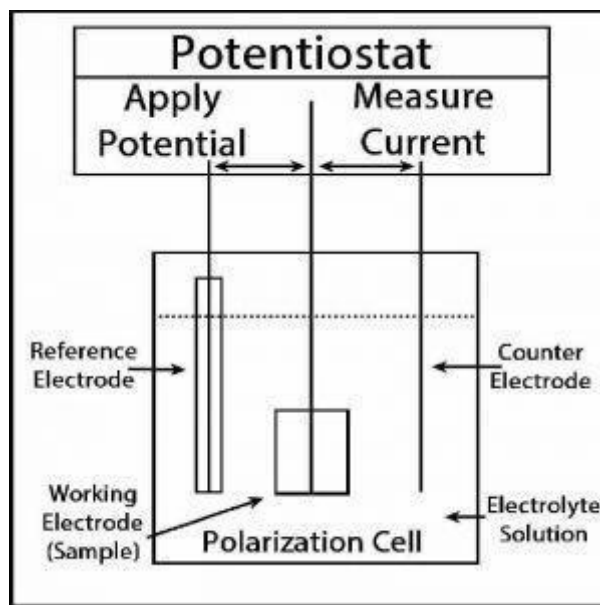


Fig.9 Electrochemical Corrosion Testing

For the testing purpose the polarization cell is setup consisting of an electrolyte solution. Here the sample (the biomedical implant) is considered as electrode. The electrochemical corrosion is measured by using the potentiostat as an energy difference between the working electrode and to the reference electrode.

This electrochemical corrosion experiments is used to measure and/or control the potential and current of the oxidation or the reduction reaction. In most experiments imposes the potential on the working electrode and measures the resulting current.

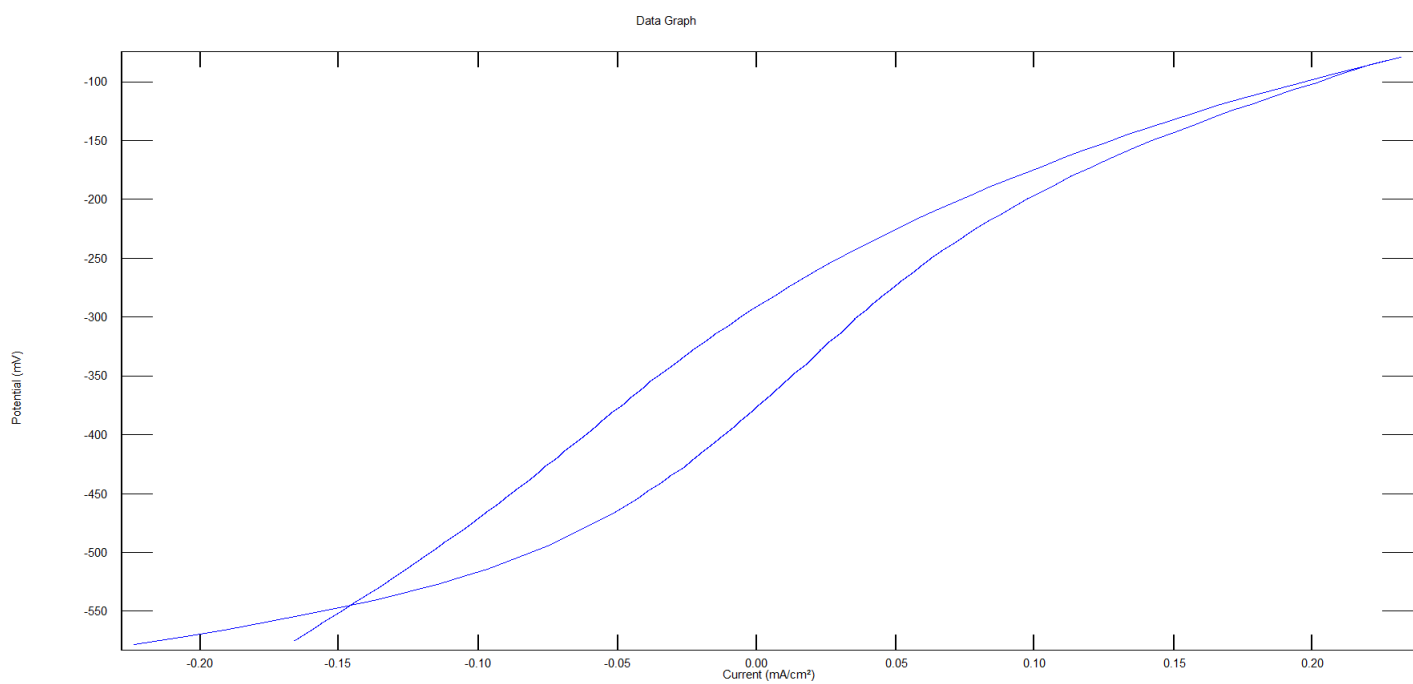


Fig.10 Current VS potential

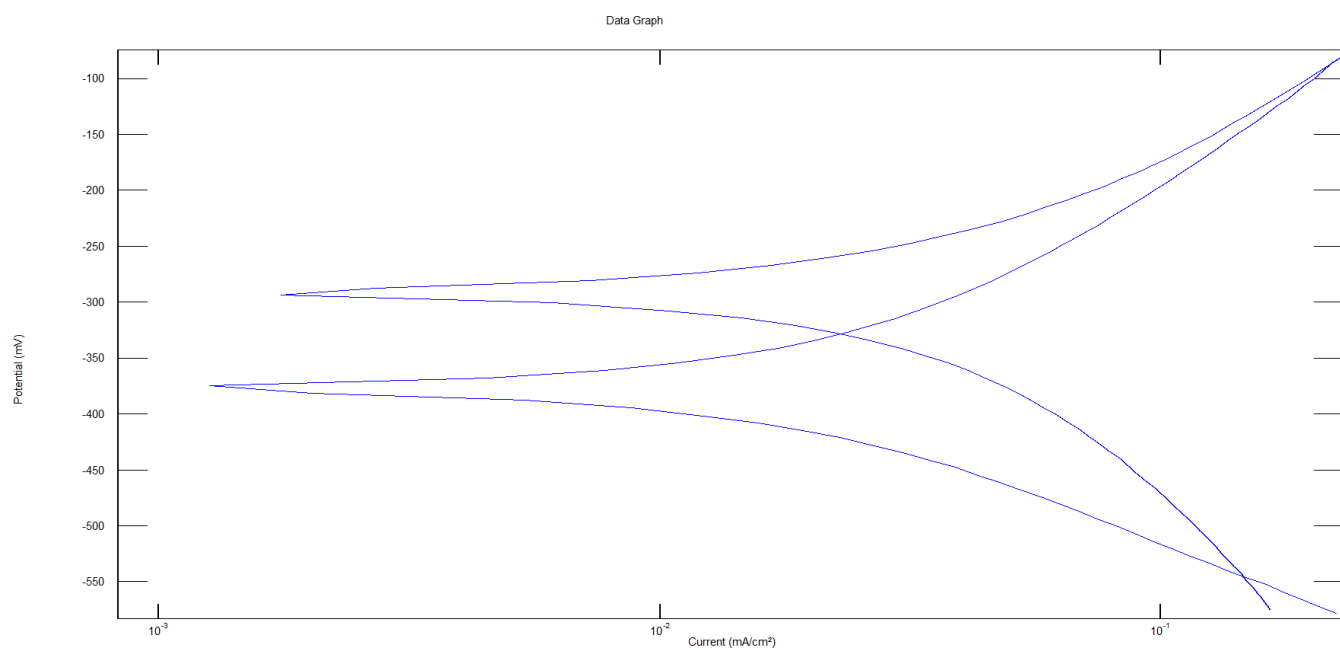


Fig.11 TAFEL

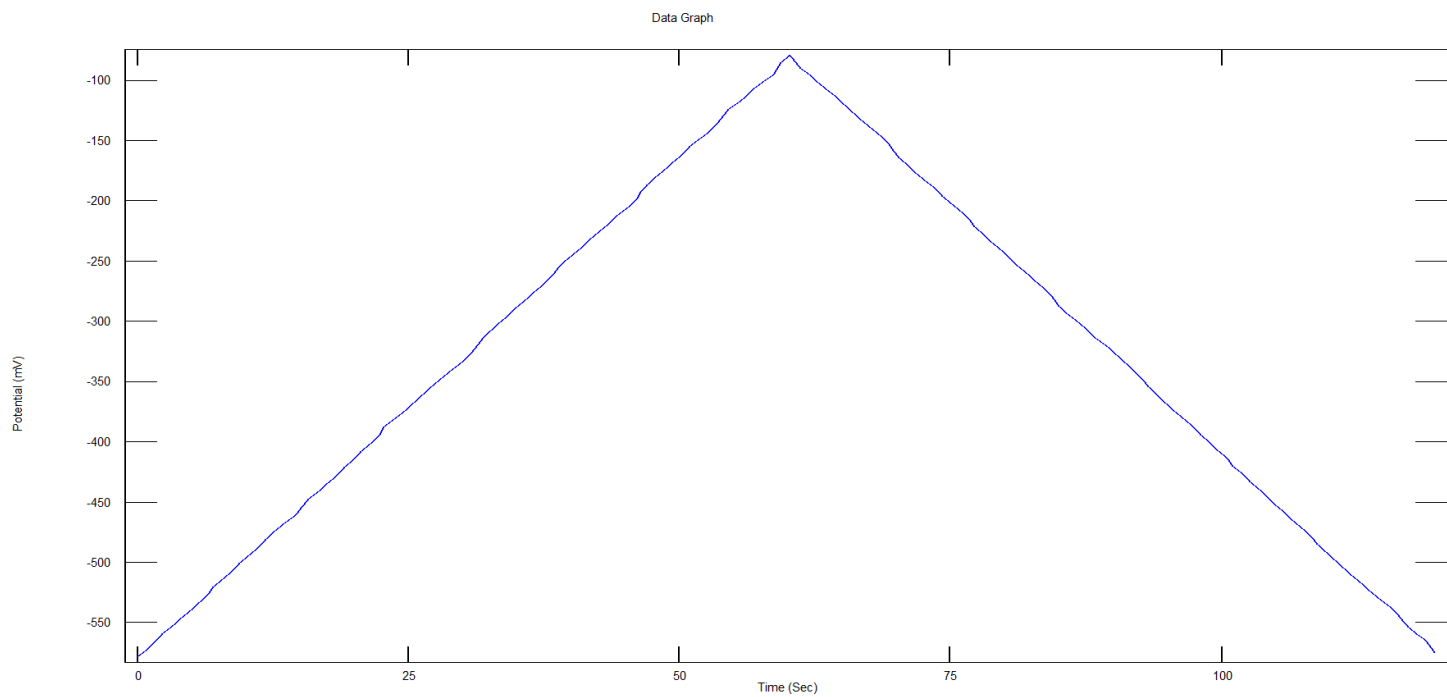


Fig.12 Potential VS Timing

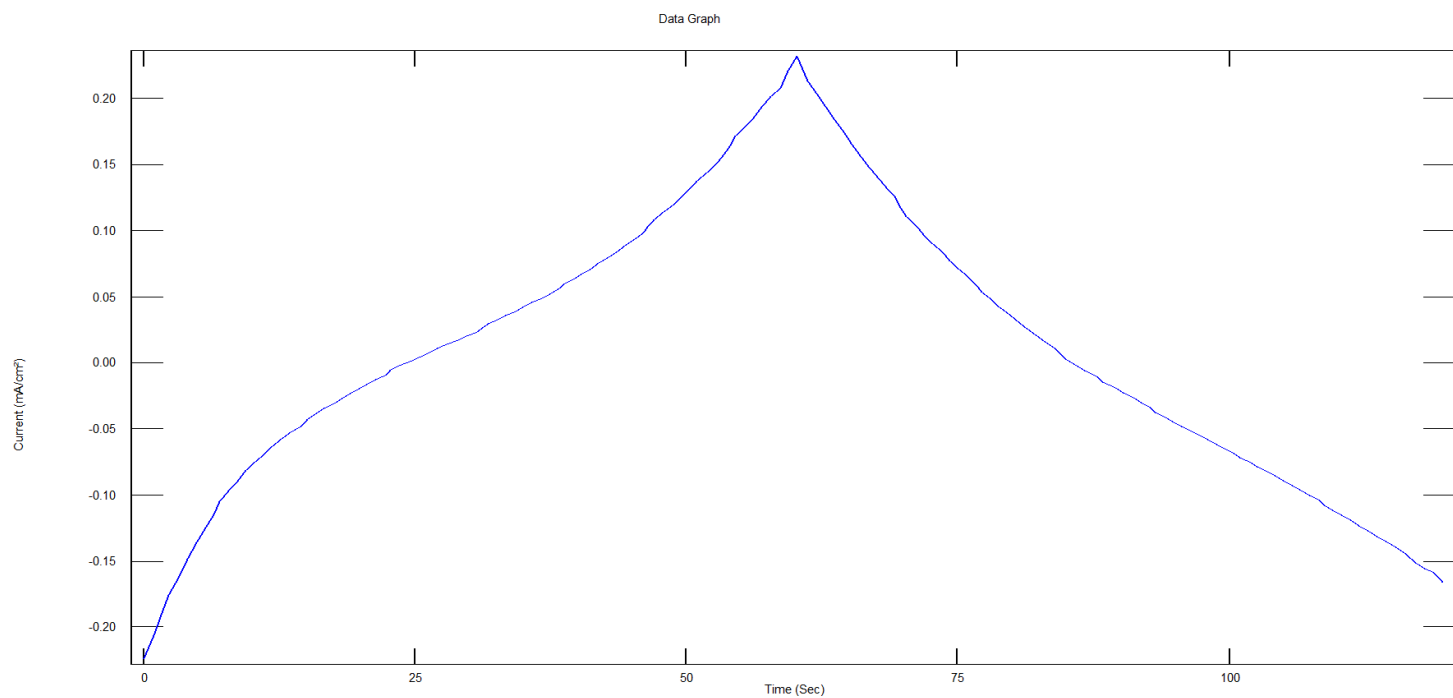


Fig.13 Current VS Timing

5.7 Salt spray test:

The salt spray testing is a laboratory setup for the corrosive saline environment. This setup is used to accelerate means of testing the ability of the surface coatings to withstand the atmospheric corrosion. This salt spray test is also known as the salt fog testing. The biomedical implantation sample is placed inside the salt spray testing chamber. Then the apparatus will spray the saltwater and then the object is observed with various parameters as follows.



Fig.14 Salt Spray Testing Chamber

The details of the tests, inferences observed and the conclusions arrived are given below.

5.7.1 Details of testing parameters:

- 1) Humidity : 98% as measured by hygrometer during the test.
- 2) Temperature of the test : 25 to 27 Degrees centigrade.
(Continuously indicated).
- 3) Pressure of Air for atomizing : 2 to 3 bar continuously by pressure regulator.
- 4) Composition of the salt solution : For 1 liter of solution.

3.5% of Sodium chloride

1% of Magnesium chloride

De-ionized water 95.5%
- 5) PH of the solution : Maintained at 7.5 by addition of buffer solution.
- 6) Measurement of pH : Measured once in 8 hours.
- 7) Type of loading of specimens : Tied with plastic wire and hung in the hangers

Table.8 Salt spray test results

S. Id	Initial Weight in g	Final Weight in g	Weight Loss in g	Width in mm	Thickness in mm	Height in mm	volume in cm ³	Density g/cm ³	Area in cm ²	Corrosion mm/year	Corrosion mils/year
1	8.732	8.719	0.013	52.55	1.4	1.3	0.95641	9.129975	146.1936	1.77749E-05	0.008389774

5.8. XRD test

XRD stands for X-ray powder diffraction. XRD is a rapid analytical technique which is primarily used for the phase identification of a crystalline material and can provide the information about a unit cell dimensions. Using this analysis we can find out the finely ground material, homogenized and the average bulk composition is determined. The input X-Ray for scanning has 30kv, 12 mA. The detector used is SC-70 in the continuous mode of scanning is done. The scanning range is 10 to 80 deg. And the scanning axis is 2 Theta/Theta. Step width is 0.02 deg. and the scan speed per duration time is 8 deg./min.

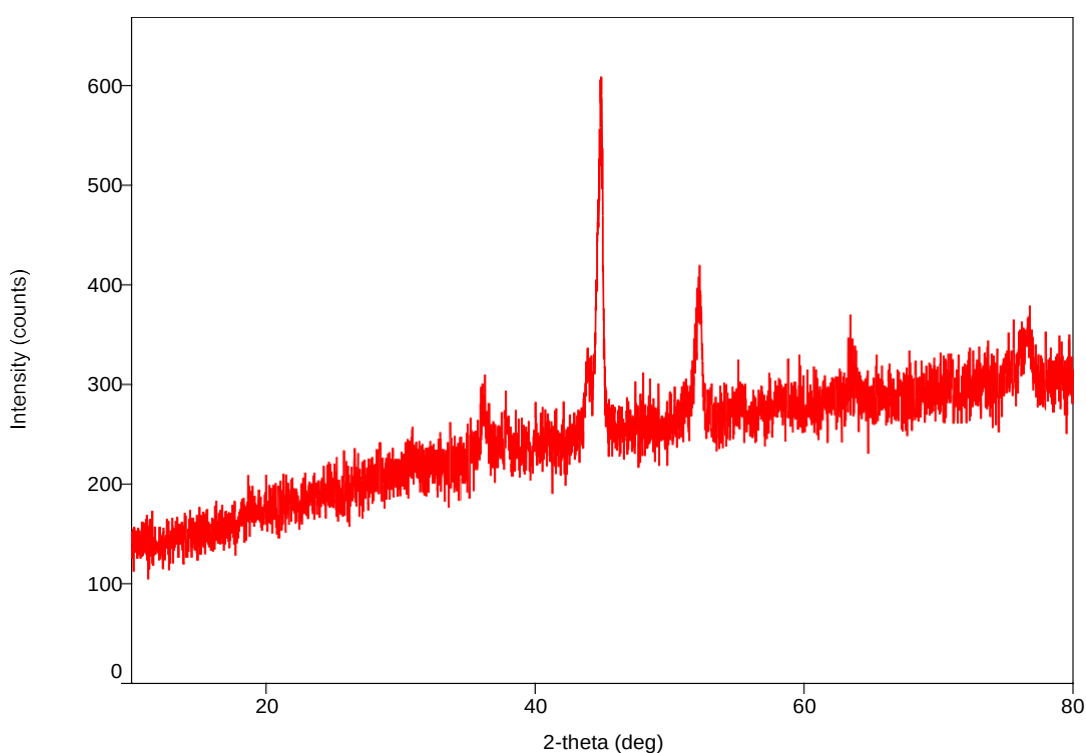


Fig.15 XRD analysis of Tricalcium Phosphate coated over SS316L

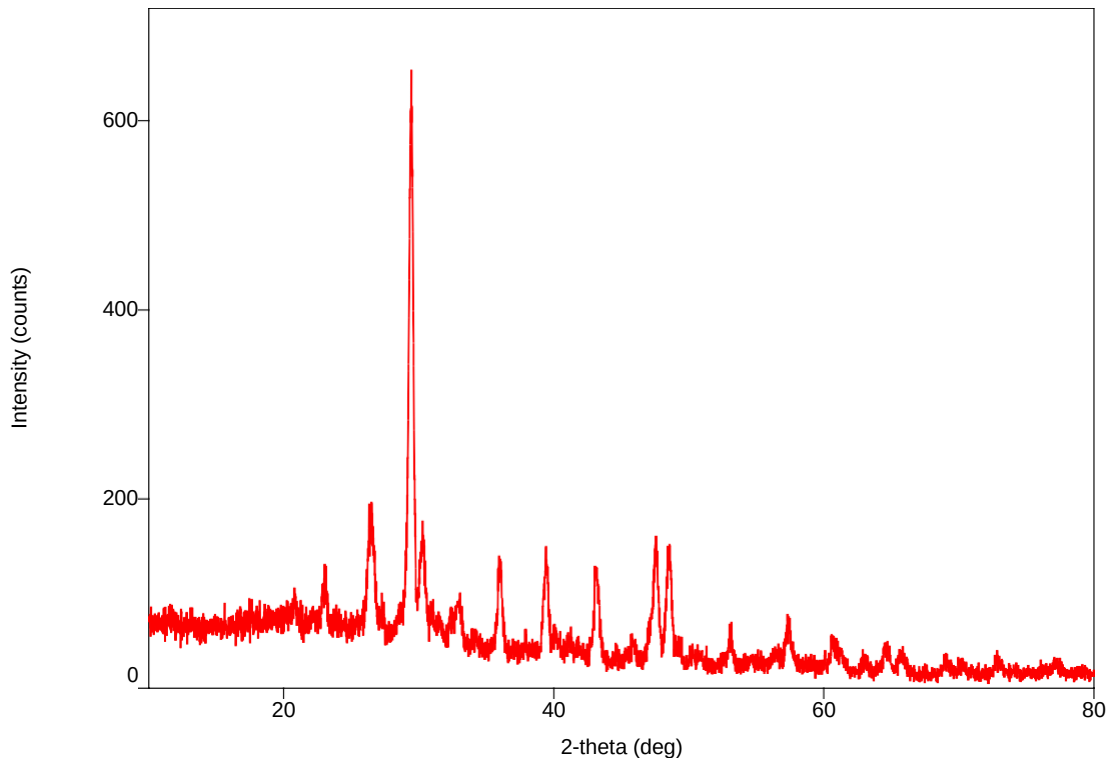


Fig.16 XRD analysis of Tricalcium phosphate

5.9 Microhardness testing

Hardness is the mechanical property which is the tendency of the component or the element to resist the permanent deformation by penetration of another harder material. To determine the amount of the force or the resistance exhibited by the material against the permanent deformation. The microhardness testing, the process is to made on the specimen by a diamond indenter through the testing application of the load P . There is a size variation is the resultant which is measured with the help of calibrated optical microscope. The microhardness is evaluated by the mean stress applied underneath the indenter. Then the measurement of this microhardness is measured with the help of microscope attachment, comprising the indenter and the means for applying small loads. It was then extended till the measurement of the individual phases, orientation effected in single element. Since the biomedical implant will be present inside the body, we should have to more aware about the microhardness measurements.

Table.9 Hardness Test Result

No	04
S.DEV	281.52
MAX	1034.4
MIN	309.3
R	725.1
AVER	555.6
A.CON	NONE HRB
A.IND	-8.49% ..NG
STANDARD VALUE	65535.0
UPPER LIMIT	65535.0
LOWER LIMIT	65535.0



Fig.17 Hardness Test Setup

CHAPTER 6

CONCLUSION

1 In the spray salt corrosion test, compared with any other material Stainless steel SS316L possesses a very good corrosion resistance to salts due to the presence of the higher chromium and nickel content. The weight loss during salt spray test is **0.013 g** and the corrosion rate **1.77749E-05 mm/year**.

2 In the electro-chemical corrosion process, the stainless steel SS316L possesses more corrosion resistance than the other metals and its alloys. Because of the spinel structures of the SS316L formed with the metal oxides the corrosion resistance is high. Thus the SS316L have the better protection with the further corrosion of the metal substrates. The electro-chemical corrosion rate is **0.3683938 mm/year**.

3 The EDAX analysis shows the increase in calcium and phosphate is high, hence the bone repair will be getting faster.

4 Micro hardness test is taken to find the material maximum resistance towards the load. At the maximum load it seems there is a crack of material, but this is enough for the biomedical implantation. This shows the material has good hardness. Microhardness maximum limit is **1034.4 N/mm²** and the minimum limit is **309.3 N/mm²**.

5 Tensile test is taken to find the strength of the stainless steel SS316L coated with tricalcium phosphate. At maximum load the coating is broken. But for the biomedical implantation the tensile strength is good enough than most of the other metals and alloys. The maximum load is **12.4217 KN** and the maximum extension is **17.28687 mm**.

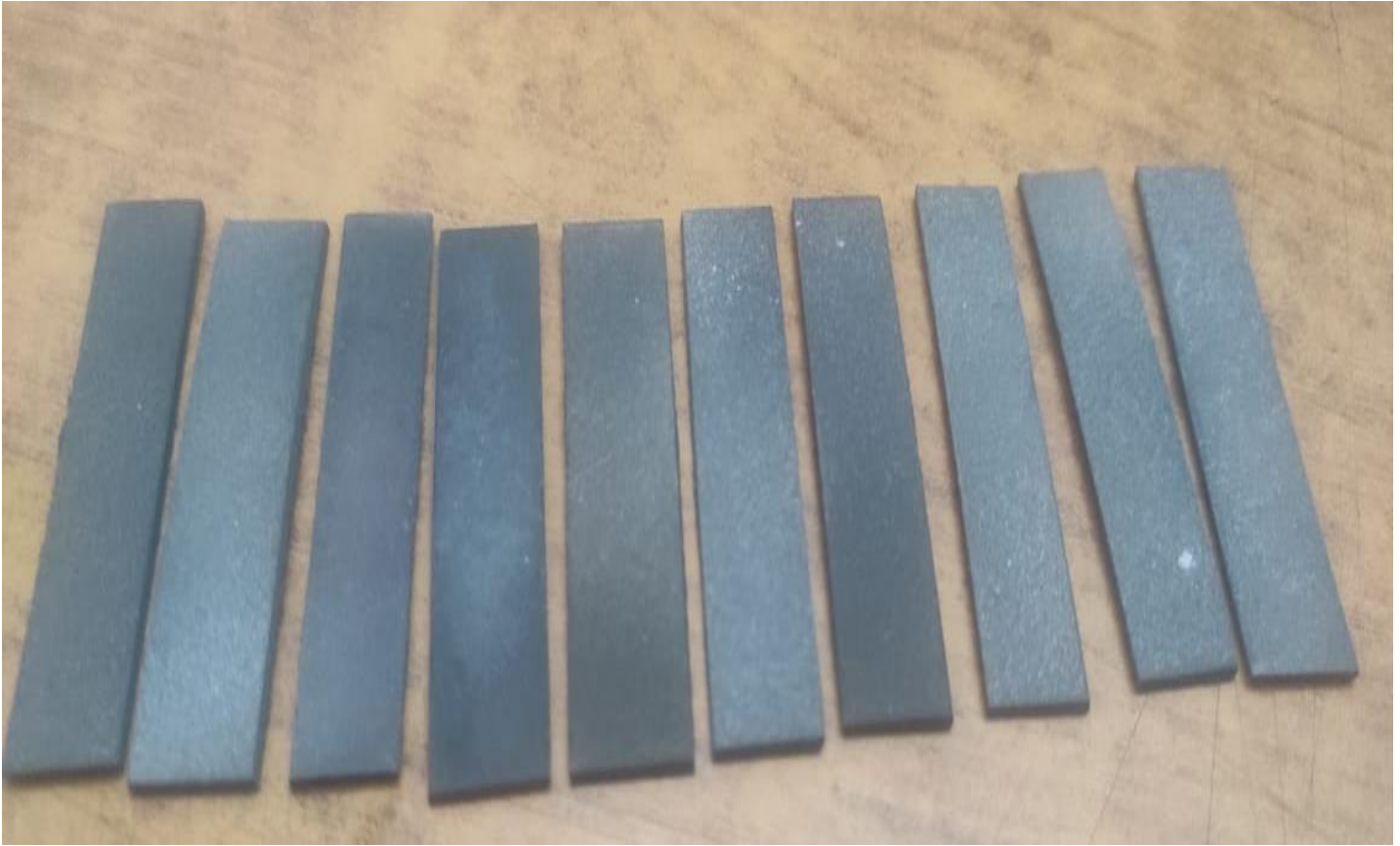


Fig.18 Coated Specimen

CHAPTER 7

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