

Titanium Silicide Coating by Using Magnetron Sputtering

**Submitted in partial fulfillment of the requirements for the award
of Master of Science degree in Physics**

By

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**DEPARTMENT OF PHYSICS
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SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade “A” by NAAC

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MAY – 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **DEEPIKA. D (40590010)** who carried out the project entitled "**Titanium Silicide Coating by Using Magnetron Sputtering**" under my supervision from **NOVEMBER 2021 to MARCH 2022**

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ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to the Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. S. Ravichandran**, Head of the Department, and Department of Physics for providing me necessary support and details at the right time during the progressive reviews.

I am thankful to **My Parents and Family members** for their constant support in completion of the project.

I would like to express my sincere and deep sense of gratitude to my External Project Guide **Dr. K. Gobi Saravanan** and Internal Project Guide **Dr. J. Anita Iett** for their valuable guidance, suggestions and constant encouragement that paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Physics who were helpful in many ways for the completion of the project.

Finally, without the help of my friends, who supplied intriguing discussion, I could not have been able to accomplish this project.

(DEEPIKA. D)

ABSTRACT

Hard coatings comprise excellent wear-resistant and corrosion-resistant properties which are mainly used in load-bearing applications. In the present study, TiSi_2 coating was developed by the magnetron sputtering method. The deposited film was characterized by X-ray diffraction, scanning electron microscope and EDS to find crystallinity and surface morphology and elemental analysis of the coating, respectively. Further, surface hardness and wear analysis were determined by nanoindentation and tribometer analysis, respectively. Further, the electrochemical behaviour of the coatings in presence of artificial blood plasma was also carried out.

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LIST OF ABBREVIATIONS

- SEM –SCANNING ELECTRON MICROSCOPE
- XRD – X-RAY DIFFRACTION
- EDX – ENERGY DISPERSIVE X-RAY
- TiSi_2 - TITANIUM DISILICIDE
- CVD – CHEMICAL VAPOUR DEPOSITION
- PVD – PHYSICAL VAPOUR DEPOSITION
- AFM – ATOMIC FORCE MICROSCOPE

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CHAPTER 1

INTRODUCTION

Nano biomaterials are made up of nanoparticles and it covered by monolayers of silica inert like materials. It is also known as nonviable material which is used in biological systems. It can be a natural or synthetic substance and it can be used for any tissue, organ, strength, nano-apatite grains, nano-membrane etc (1). The Nano-biomaterial have unique properties which can be utilized by biomedical applications like biosensing, drug, wound healing, medical implant and diagnostic systems like protein. The fast growth of nanotechnology has helped researchers produce nanoparticles, nanofibers, Nano-coating, and nanocomposites for biomedical applications (2).

It has been 60 years in these biomaterials fields there are many paths to development in nano-bio materials. The conventional biomaterials with their medical applications and characteristics in biomaterial, metals and alloys, ceramics like polymers, polyethylene, joint replacement and titanium, titanium alloys are used in fracture fixation, bone and joint replacement dental implants, pacemakers because of their high biocompatibility, low thermal and electrical conductivity, bioactive corrosion and compression resistance (3). Most alloys are used in their decades as permanent load-bearing metal implants due to their fatigue resistance and tensile strength. When biomaterials are implanted in the body, it depends upon size, shape, surface and roughness etc.

The nano which is very small The nanostructured biomaterials have a great attention for human beings' tissue regeneration and these Nano-particle usually forms the core of Nano-biomaterial. Not only in dental implants but also used to assay for blood proteins in the laboratory, grow cells in culture, mostly ceramic hydroxyapatite is used in bone replacement applications (4). Nanotechnology is developed in the biological process by its nanoscale size, and dimensions of the material. Richard Feynman won the Nobel prize winner in 1957 for explaining the basic concept of nanotechnology (5).

The term nanotechnology was first proposed by Norio Taniguchi in 1974 found the technology as the ability for material at the nanoscale. The development of

nanotechnology leads to the two main types of the invention the imaging of molecules and the surface at the nanoscale which gives importance to nanotechnology (6). Examples like ten hydrogen atoms are equal to 1 nm dimensions when they are placed in a row. Many material changes into a nanoscale range their properties also change with them like when platinum is changed into a nano range they behave like a catalyst, the silicon which is an insulator in nanoscale it behaves like the conductor. When the element is in nanoscale they all show different properties. One billionth of a meter is equal to one nanometer.

Nanomaterials are keys to nanoscience and nanotechnology. The nanoscale materials are least than one dimension. It is approximately 100,000 smaller than the diameter of human hair. Nanomaterials occur naturally but at the same time, it can be found in cosmetics, stain-resistant clothing, sunscreens, and sporting goods. The main two properties why the material having nanoscale has different properties is new quantum effects which are used to determine the material properties, characteristics, electrical, magnetic behaviours and increase in relative surface area so the chemical reactivity and depending on strength. Nano-coating and nanocomposites are used in diverse consumer products, like sports equipment, bicycles and automobiles. UV-blocking coatings on glass bottles which protect beverages from damage by sunlight, and longer-lasting tennis balls using butyl-rubber composites.

1.1 IMPORTANCE AND ADVANCES IN NANOMATERIALS

Nanomaterials started immediately after the big bang when nanostructures form in meteorites. Michael Faraday 1857 reported the colloidal gold particles synthesized. Nanostructured catalysts also have been investigated for over 75 years. Nanosized amorphous silica particles have been used in more applications in everyday human life. In the 1970's metallic nanopowders for magnetic recording, tapes were developed. In 1976 the nanocrystals were produced by the inert gas evaporation technique were published for the first time by Granqvist and Bugrimna. Recently it has been found that Maya blue paint is a nanostructured hybrid material. The origin of this color and its resistance to acids and bio-corrosion is still not understood but studies of authentic samples from Jaina Island showed that the material is made of needle-shaped palygorskite (clay) crystals that form a superlattice with a period of

1.4 nm, with intercalates of amorphous silicate substrate containing inclusions of metal (Mg) nanoparticles. This made the blue color when these nanoparticles and superlattice are present.

Nowadays nanophase engineering expands fast with the growth in the number of structural and functional materials in both organic and inorganic, optical, electric, catalytic functions. Nanomaterials can be in one dimension, two and three dimensions. They can exist infused, aggregated, single tubular it can be in irregular shapes. Common types of nanomaterials include dendrimers, quantum dots, nanotubes and fullerenes. The nanomaterial can be characterized as ultrafine grain size less than 50 nm. Zero dimensions nanomaterial anyone can be outside nanoscale that is all the three dimensions are in nanoscale range examples like clusters, quantum dots, fullerenes. One dimensions nanomaterial is outside the nanoscale and two other dimensions in the nanoscale range examples like nanowires, nanorods, biopolymers. Two dimensions nanomaterial is only one dimension in the nanoscale range and it exhibit plate like shapes, examples like nanolayers, surface coating and thin films. Three dimensions nanomaterials are bulk materials made up of a collection of Nano-particles examples like dispersions of nanoparticles, bundles of nanotubes and multiple nanolayers.

Due to the high ductility at elevated temperatures of nanophase ceramics as compared to the coarse-grained ceramics. Nanostructured semiconductors show non-linear optical properties. These particles have some special properties like luminescence in silicon-germanium quantum dots as infrared optoelectronic devices. They are used as window layers in solar cells. Nanosized metallic powders have been used for gas-tight materials, dense parts and porous coatings. Single nanosized magnetic particles are mono-domains and one expects that also in magnetic nanophase materials the grains correspond with domains.

Once titanium was rare material, it is the most important metal in the industry (7). They are mostly used in medical fields due to their lightweight critical applications. It can be used in hard tissue replacement materials and orthopedic and orthodontic (joints and teeth) applications (8). In earlier days, the surgery to replace bones and joints was a big challenge. To overcome that these titanium alloys have been extensively used in these fields. Titanium alloys are high specific strength and

excellent corrosion resistance. The biocompatibility of an implant that gives the materials implantation is been done. The mechanical properties of biomedical materials determine the implants for specific applications. Titanium alloys are classified on the basis of metallurgy and their shape, and orientation. Commonly these alloys contain aluminium and vanadium, whose ions may be detrimental to the nervous system. Even the titanium alloy is differentiated in their grades. The best titanium for use in bio implantation is Grade 4 titanium alloy.



Fig 1.1: Titanium alloy

The most preferred method in dental by titanium silicide due to its good balance between strength and ductility. These titanium silicides can be prepared by a dental casting machine. So when there is an addition in silicon in titanium alloy there will be a change in microstructural in titanium alloys. Various types of titanium alloys have been developed in the aerospace industry and for illustration, titanium alloys are used in the construction of turbocharger rotors, valves etc. Many methods have been introduced to make titanium alloys more scaling resistant like chemical vapor deposition (CVD), physical vapor deposition (PVD), alloying, sol-gel, etc. At high-temperature oxidation of titanium alloys are significantly accelerated and there will be a small effect on the mechanical properties of titanium alloys.

When compared to stainless steel and CoCrMo alloys, Ti alloys are particularly good implant materials due to their great corrosion resistance. Titanium has lots of properties like it is easily alloyed with other metals, to give very strong, lightweight, wear corrosion-resistant alloys. The atomic number of titanium is 22 which is on a

d-block having a melting point of 1941K and boiling point of 3560K. The Silicon has an atomic number of 14 which is on a p-block having a melting point of 1687K and boiling point of 3538K. Silicon is the most important element in biology for skeletal structures and it is deposited in many plant tissues. Titanium is almost present in every rock. Other than in medical fields it is used in frames, phones, surgical tools, jewellery etc. There are different types of titanium alloy that depend upon their grades, (grade 5, 23, 12, 6).

Titanium silicide is commonly known as titanium disilicide which has a melting point of 1470°C and boiling point of 1540°C. The special characterization of titanium silicide is excellent oxidation resistance at high temperatures. The implant is key for an active lifestyle nowadays. To determine the stiffness of the implant material the elastic modulus is also considered as a part generally, the titanium alloys have 100Gpa. The biomedical alloys cannot be used directly it has some limitations like sufficient strength level can be used, only bio tolerant elements are used and to maintain the level of elastic modulus.

Nano coating states that coating a thin film where the Nanostructures give a network of molecules on the surface. Nano coating develops material properties like friction reduction, corrosion properties and life span of the materials which are used in medical implants. Most the Nano-coatings are eco-friendly and they are non-toxic gases in the atmosphere (9). Anti-corrosive coatings, metals components are coated with these coatings so that they protect the component against the atmosphere, salt spray, oxidation etc., Waterproof and non-adhesive coatings, these coatings repulse water thus making the surface waterproof. These features are mostly found in the food packaging industry. Antibacterial coatings are such type coatings in health care industries that play a crucial role in stopping infections. Anti-abrasion coating is another type in which coatings are used to strengthen the material and it is used where there is no use of lubricants (10).

Normal orthopaedic implantation of material will not allow people to live their normal life. So to reduce the wear debris Nanotechnology has been introduced which gives exceptional results in orthopaedic implants by implementing a coating system on the biocompatible implant material, corrosion resistance and helps in the growth of bones and teeth (11). The coating has been developed by many methods like laser

surface alloying, and powder siliconizing, the silicon coating is very effective (12). Compared to uncoated material the Si-Ti coated has a great factor when it involves oxide scales and it forms Ti_5Si_3 (13). At high temperatures, these Ti_5Si_3 show oxidation resistance and have a high melting point (14). To improve the oxidation resistance of Ti-based alloys there are many methods are there. It has been studied that when silicon is added there is high-temperature oxidation (15). There are many methods that help to improve the oxidation resistance Ti-based on alloys and the addition of silicon is observed (16). It has been proved that the addition of Si increases the temperature oxidation resistance of these alloys, also their wear and creep resistance. The mechanical properties Ti-Si alloys are one the promising elements. Due to various inherent qualities of the materials, titanium and its alloys are typically thought to be difficult to machine. Because of its limited thermal conductivity, the temperature at the tool interface rises, reducing tool life. Furthermore, its machinability is further by its high strength at high temperatures and poor modulus of elasticity. (17).



Fig 1.2: Coated titanium alloy

It was not possible by the titanium-ceramic dental for their restorations some affected their bond-like Stress generated during cooling due to a thermal expansion mismatch at the porcelain oxide Ti interface, adherence of the self-formed oxide to the Ti substrate, bonding by fusion of the self-formed oxide with the porcelain, and stress generated during cooling due to a thermal expansion mismatch at the porcelain oxide Ti interface. They concluded that Si_3N_4 coatings increased the spallation strength of porcelain on titanium by a factor of six when compared to uncoated titanium. Si_3N_4 coating on Ti with or without heat cycles had much greater

adhesion strength values than unprotected oxidised Ti samples, according to adhesion strength tests(18).

With excellent mechanical qualities, excellent corrosion resistance in a physiological environment, and good formability Ti is the ideal contender for orthopaedic applications because of these features. However, when they are surrounded by tissue in an in vivo environment, they lack osteointegraty in limited occurrences. The Ti-6Al-4V is a commonly used titanium alloy that accounts for roughly half of all titanium alloy production worldwide. One of the most fundamental factors limiting Ti-6Al-4V's applicability is its poor wear resistance when compared to stainless steel and CoCr-based alloys. The plasma nitriding process significantly increased the wear resistance of Ti-6Al-4V alloy (30–90%). However, the nitrogen concentration in the surface region played a large role in this improvement. it has been studied that the effects of plasma immersion ion implantation heating treatment on Ti-6Al-4V alloy, finding that the material treated at 800°C exhibited excellent wear resistance. To increase the wear resistance of Ti-6Al-4V alloy, a laser surface alloyed with gaseous nitrogen was used(19).

Titanium silicides (Ti_5Si_3) have gotten a lot of attention recently, and they've been used as "the most fascinating silicide-based material." Ti_5Si_3 is expected to be a competitor to the major candidate, material Titanium silicide (Ti_5Si_3) and its composites show potential for applications at temperatures above 1000°C.(20) Metallic silicides are well-known for their high-temperature oxidation resistance and can be utilized as coatings to protect metals. Their research has demonstrated the high resilience of this compound when used as the primary component of a Ti-Si coating on titanium alloys. After oxidation with 100% oxygen, X-ray diffraction examination revealed that TiO_2 futile was the only crystalline phase in the scale (21).

Thin films of titanium disilicide (TiSi_2) with a face-centred orthorhombic structure were produced. The thickness of thin films affects the development of the TiSi_2 crystalline phase, and the quantity of the TiSi_2 crystalline phase in the thin film is proportional to the mole ratio of TiCl_4 . This result was done by chemical vapour deposition(CVD) it is only good for roughing. So, for this coating, there is a need for high temperatures, and size is limited to the chamber capacity. When titanium disilicide is coated in the CVD method we get a result of the sample with a large

thickness, so when physical vapour deposition is used it allows for thin coating and sharp edges(22). The pack cementation coating method is a promising approach for producing silicide diffusion coatings on titanium alloys that give oxidation resistance at high temperatures. Solid-state diffusion governs the growth rates in all of the silicide layers for coatings generated by packs with the less stable silicide layers, according to a model for multiple layer growth(23).

The oxidation behaviour of silicide on ceramic coating is observed, the silicide coating was mostly made up of TiSi_2 and SiC , and during its preparation, a number of fine cracks appeared in the coating's outer layer. The oxidation rate was enhanced when these fissures permeated the entire covering during cyclic oxidation, reducing the oxidation resistance. Due to the cracks, the oxidation rate was increased(24). Metal implant corrosion is significant because it can compromise biocompatibility and mechanical integrity. The substance utilized must not have any negative biological effects on the body, and it must be stable and preserve its useful properties. Due to the high melting temperatures of titanium alloys, the casting of these alloys was not conceivable until recently.

In the dental laboratory, recently. The first appearance of a vertical centrifugal casting machine with an argon-electric arc and a vacuum pressure casting with an argon electric arc. The casting high melting temperature was done by machine. titanium alloys with a high melting point are also possible. So, it was proved that pure titanium alloy cannot be used directly in the biomedical field due to its lack of biocompatibility, with the support of other materials it can easily achieve its properties so we have used the latest technology of magnetron sputtering and inert gas argon in it(25). Titanium is very resistant to corrosive bodily fluids, after saline water treatments, their surfaces are softer and less impacted than after acid treatment(26).



Fig 1.3: Titanium Implanted on teeth

The corrosion resistance of the crystalline titanium silicides is well known that when these materials are dipped in HCL solution their corrosion-resistant property is increased. The Ti-Si amorphous alloys are expected to have good corrosion resistance when it is kept in HCL solution(27). At high temperatures, most metallic alloys are shielded from oxidation by the creation of a thick layer. The creation of an impermeable and adhering SiO_2 layer on the surface protects the interior from further oxidation, giving silicides their oxidation resistance. In the case of silicon and aluminium oxide, it gives an impervious protective coating, In all circumstances, oxidation resistance is a function of temperature, and it may not be protective beyond a specific temperature depending on the fault concentration in the oxide coating oxidation(28).

The use of a DC magnetron sputtering in both research institutes and industrial manufacturing use this form of sputtering. The magnetron is a device in which the magnetic field is applied perpendicular to the electric field and parallel to the target, and it is a PVD method. The same innovative PVD method was approached by it didn't give a high-power supply as the DC magnetron sputtering, it failed he give the power supply to the target(29). When the magnetron sputtering is done in the TiSiN coatings in the temperature range of 25–800°C, coatings deposited at bias voltages of 50 V and higher generate a stable phase composition consisting of (Ti, Si) embedded in a Si_3N_4 , with smooth surface morphology and increased hardness. When the temperature is increased its stability and phase have been decreased(30).

Apposition, attachment and penetration are the three phases of the implantation process. The unstable adherence of the blastocyst to the endometrial surface is known as apposition(31). The most common technique for introducing contaminants into solids in a uniform and repeatable manner is ion implantation. Its primary application is in the semiconductor sector, where it is frequently used for semiconductor electrical doping. In VLSI circuits, ion implantation is commonly employed. Wafer cooling, dielectric charging, particle contamination, and process control are the main issues in using ion implantation in VLSI processing(32). An implant material must exhibit specific properties in order to work well in vivo features that are well-controlled and appropriate to each application Biomaterials for bone applications are utilised in a range of situations, from orthopaedic to dentistry, but biomaterials characterization is critical in all circumstances to ensure that implants perform as intended.

One of the most important factors to consider when choosing a material for use as an implant is its needed mechanical performance in the specific skeletal application. Mechanical qualities are significant on various length scales because the bone is extremely sensitive to its mechanical environment. Cellular responses such as differentiation towards bone cells and mineralization are significantly influenced by microscopic mechanics. Hardness tests are a type of indentation resistance test that is quick, simple, and non-destructive. (33).

Metals, plastics, polymers, and compounds generated from cells and tissues, such as collagen, are some of the most common biomaterials. In history, biomaterials and biomedical devices have been utilised to diagnose and cure illnesses. Implants are utilised as grafts or additions in a variety of tissues, including bones and joints, to replace missing or damaged tissue and restore normal structure and function. Various autogenous, allogeneous, etc., bone grafts have been utilised by surgeons for over a century to aid in the healing of bone defects, particularly big or non-union lesions. These issues, as well as the need for materials with a variety of properties that can be used in a variety of applications, have prompted extensive research and development of bone implants and bone substitute materials(34).



Fig 1.4: Titanium Implanted in bone

Using argon-ion in the implantation such as Ar42, have been demonstrated to boost biomaterials' antibacterial efficacy. An in vitro model cell culture system and surface analysis was used to examine the utilization of ion-implantation to promote osseointegration. By using titanium the implantation was done on various ions like Ca, K, and Ar ions. It was concluded that when they use Ar ion in implantation the Polyurethanes have been shown to lessen tissue inflammation(35). Implantation is a method for achieving superior titanium corrosion resistance in hostile situations.

The implant should not be short-term, it should provide long-term biocompatibility. the mechanical and biological biocompatibility of implants, the cost of manufacture, and the use of various alloying elements to build a strategic technique for reducing the use of rare metals, it appeared that Ti alloys would be the future metallic implants for long-term use(36). Lower SiOZ concentration glasses have a more open network structure, allowing for faster proton-alkali exchange and are rich in pH. It has been discovered that some glasses in the system have great bioactivity, and that by regulating the production parameters, coatings with good adhesion to Ti and Ti alloys may be fabricated(37).

CHAPTER 2

LITERATURE SURVEY

Serhii tkachenko et al carried out All Ti–Si–Zr alloys demonstrated lower coefficients of friction than the commercial Ti–6Al–4V alloy. It also showed 5 times lower wear rates than the Ti–6Al–4V. The Ti–Si–Zr alloys are high hardness. There will be uniform friction and wear resistance compared to titanium alloys. Due to the periodic local fracture, it showed some fluctuations. They stated the Wear and friction properties of experimental Ti–Si–Zr alloys for biomedical applications. The lower wear resistance of the Ti–Si–Zr alloys in comparison to the commercial CoCr alloys did not show such material transfer. The alloy's microstructure and hardness were investigated using optical microscopy, XRD, scanning electron microscopy (SEM) and Vickers indentation(38).

Xuanyong Liu et al observed that with more new surface modification technologies titanium and its alloys can improve their properties to the need of the situation. The proper surface treatment expands the use of titanium and titanium alloys in the biomedical fields. Some of the recent applications. They have proved the wear, corrosion resistance and biological properties of titanium and its alloys and they can improve by proper surface coating techniques. They have. So if proper techniques are followed titanium alloys have a big role in biomedical fields they can use different methods like sol-gel, chemical and electrochemical treatment, ion implantation and some latest research going has been discussed(39).

LiMei Kang et al stated that titanium alloys are the microstructure by controlling the impurity content therefore there are different composites that have different lattice, morphology, phase scale. They have stated the strengthening approaches, microstructures, their mechanisms and mechanical properties. What are the advances in the development of high strengthened titanium has and a lot of examples have been given on how the high strength titanium alloys are developed based on different methods like amorphous, semi-solid, crystallization was presented. They have given detailed tabulation on. Different preparation techniques obtained mechanical properties of representative bimodal titanium and titanium alloys(40).

Devaraju. A et al stated that the materials combined with more dissimilar materials to achieve good properties are Metal Matrix Composites. In this study, they successfully fixed the powder metallurgy technique. Composites of different materials gives important in modern technology, their demand increased in the industry. So, the different studies the fly ash, Cu and Sic Metal Matrix Composites were studied and their friction properties were studied by tribological methods. It has been concluded that copper and sic fly ash show better at low loading conditions compared to higher loading conditions. The optimum level is given at average sliding velocity. Their compact shows a uniform distribution which was studied by scanning electron microscope and their particle size is shown as approximately 1 mm(41).

Shivendra. K et al observed the types of coating materials and their role and what methods were been used. This is based upon nowadays the lifestyle of human beings are changing like having knee joints problem, bone-related the damages it is known as Osteoarthritis. So to cure that implantation is the main role. So they used stainless steel the most preferable alloy as titanium as it has a good bio-compatible. They have gone through tribological studies have gone through for the wear rate and friction between the implants. Nano coating is used for implantation on the knee for the life saviour. Nanotechnology plays an important role in the implant. This paper will be useful in how the biocompatible implants and how to reduce friction how works on knee implanting(42).

Josef Strasky et al studied that Ti-Nb-Zr-Ta by alloying with low amounts of silicon found 20% the increase in strength and decrease in elastic modulus compared to titanium alloy. This property will be suitable for biomedical use due to its mechanical properties. In that condition the alloy's elastic modulus was 65GPa and the alloy containing O and other materials were having 80GP this led to the β phase to increase the electrons atom ratio. They have studied about 12 different Ti-Nb-Zr-Ta based alloys alloyed with O, Si, and Fe by arc melting and hot forging methods. In this alloy when it was in the β phase with low stability it achieved in lowest elastic modulus. They have been observed by SEM for their silicide precipitates(43).

Elzbieta Godlewska et al observed that depositing silicide coating on a Ti-6Al-1Mn alloy by pack cementation method to upgrade the oxidation resistance at a

temperature above 700°C. They have also focused the parameters on composition and morphology to get the coating. They also said about the next silicide coatings were tested for oxidation resistance. After the silicide coating their morphology and composition were analysed by different methods like transmission electron microscopy, energy-dispersive X-ray spectroscopy, electron microscopy, scanning transmission electron microscopy etc. The oxidation resistance was improved by ascertained so as a result, they got coating deposition. The coatings were developed by several layers with different stoichiometry, the predominant one being TiSi_2 (44).

Kathleen Chou et al have said about how to improve the oxidation resistance of titanium alloys by oxidation of pure titanium coated with a thin silicon layer. The titanium oxide external grows like crystals from titanium which is outward diffusion. The comparison with the uncoated titanium oxidized they found it their morphology of internal growing oxide scale for silicon-coated titanium show few voids and Nano-crystalline grain over longer exposures. After coating the new bilayer structure of SiO_2 and Ti_5Si_3 is formed with Nano-wedge shaped and Nano-crystalline, compact(45).

Minagar. S et al studied that aqueous and non-aqueous TiO_2 , ZrO_2 , ZrTiO_4 nanotubes were fabricated. They have concluded the percentage of cells attached to the surfaces of different pore sizes and roughness parameters of TiO_2 , ZrO_2 , ZrTiO_4 nanotubes fabricated in aqueous and non-aqueous electrolytes after cell culturing for 7 days. With a variety of non-aqueous electrolytes their nanotube size, bone cell attachment and morphologies possess were compared to those fabricated in an aqueous electrolyte. The implant surface is affected by the cell response by shape, migration, differentiation by the studies of mechanical and topography(46).

Dalibor Vojtech et al stated that titanium and its alloys are attractive materials due to their corrosion resistance and superior weight ratio. The behaviour of titanium silicon alloys with the composition of TiSi_2 and TiSi_8 when it is high temperature in oxidation air. The alloys were in around 30hr at the temperature range of 850 °C. The composition and morphology of scales also their oxidation kinetics studies were gone with comparing pure titanium. When the process was going the titanium silicon alloy didn't show any of the changes up to 850°C. The materials like Ti_5Si_3 particles

in the TiSi_8 alloy plays a major role in the oxidation process. Here their process is increased due to only a continually release of silicon into the scales. So the oxidation resistance increases with time(47).

Andrea Skolakova et al observed that due to excellent corrosion resistance, biocompatibility and high mechanical properties so these titanium and titanium alloys are used in implantology. They found that comparing human bone the young's modulus was too high. To overcome that they have pores were made in the material, this helped in improving the osseointegration. The pore-forming agent was created by reactive sintering and this was compared with pure titanium and titanium alloys. Osseointegration happens within a short time with the great property of biomaterials, with good quality. They have stated about the vitro bioactivity tests, vitro cytotoxicity testing, corrosion resistance, mechanical properties etc. By powder metallurgy method porous alloys are prepared. All pores containing alloy were almost matched to the bones(48).

Oukati Sadeq. F et al studied that the synthesis of f Ti–Si–Al coatings on the surface of Ti–6Al–4V alloy hot-dip siliconizing method was introduced it is so fast, echo route, simple to synthesis. On the surface of the bare alloy three layers were formed in which the first layer the substrate had mainly the composition of TiAl, the second layer consisted of TiSi_2 and Ti_5Si_3 and phases and the third layer had $\text{Al}_{11}\text{Ti}_5$ and TiSi_2 phases. When the temperature is increased and the soaking time there is thickness formed of titanium- silicide intermediate layer. It also noticed that it follows the Arrhenius relationship with activation energy at 100 kJ/mol for the parabolic rate constant for the growth. For crack-free coating, the melt temperature was 850 °C. To form the compositions they lead high-temperature oxidation resistance coated on Ti–6Al–4V alloy(49).

Revesz. P et al stated that to study the measure of the growth of titanium silicide layers by silicon samples by implanting with high doses of phosphate, arsenic, oxygen, argon to titanium. By four-point probe measurement, by x-ray diffraction, Rutherford backscattering the annealing behaviour of the samples. The samples phosphate, oxygen, arsenic showed their thickness less as compared to nanoimplanted samples. Arsenic atoms started to be distributed homogeneously in the silicide layer. The implantation depth and at high annealing temperatures it is

observed that argon and oxygen the implanted samples precipitate the titanium atoms. The presence of the unstable TiSi phase was measured by X-ray diffraction but after annealing at 700 degrees Celsius there was no trace of TiSi₂ was found(50).

CHAPTER-3

AIM AND SCOPE

- To coat TiSi_2 by magnetron sputtering method on titanium alloy.
- To find crystallinity nature by XRD.
- To find surface morphology and elemental analysis by SEM.
- To assess surface hardness by nanoindentation.
- To assess wear analysis by tribometer.
- To estimate protein adsorption.
- To estimate platelets adhesion.
- To evaluate the cytotoxic nature
- To analyse the corrosion studies

CHAPTER-4

MATERIALS AND METHODS

4.1 TITANIUM AND TITANIUM SILICIDE

Titanium is considered one the most biocompatible metal, its alloys are safe to use in critical structures as in aircraft, other computational materials engineering tools. Titanium and titanium alloys show different properties when they are modified in low and high temperatures. There is various phase transformation that can be achieved according to their stable nature, mostly their combinations depend on the ductile phases based upon hcp and bcc allotropic. There are non-toxic in nature and due to their combination, they determine the plasticity and fracture behaviour of their phases mixtures. Largely used in pragmatic engineering and their simulation which reduces the time and cost of qualifying the materials(51).

Titanium silicide is commonly known as titanium disilicide. It is mostly used in the semiconductor industry; it helps in reducing the sheet resistance of transistors connections. It is an inorganic chemical compound. Nowadays this material is favoured for contact metallization of silicon devices as it has low resistivity. the formation of TiSi_2 proceeds as a solid phase reaction between the Si substrate and the deposited Ti(52).

4.2 MAGNETRON SPUTTERING

The titanium silicide is coated on the titanium alloys by the magnetron sputtering method. This method is used because it targets the materials on the substrate it gives a large amount of ionization rate of the deposition. It is a high-speed method which is introducing a magnetic field on the surface of the targeted cathode and application of the magnetic field to the charged particles to increase the sputtering rate. There are many types of methods like direct current (DC) magnetron sputtering, radio frequency (RF) magnetron sputtering, Ion beam sputtering, reactive gas sputtering it has a different principle, procedure, used in different applications. Here we have used dc magnetron sputtering used magnets to negatively charged target materials so they get bombarded the substrate, which allows the faster deposition rates. Sputtering on metals is considered an easy method for ignition and sputtering.



Fig 4.2: Magnetron sputtering machine

DC sputtering is a thin film physical vapour deposition it is a coating technique where a target material for the bombardment purpose. This is the basic method for coating the materials. The major advantage of using this method is high deposition rates at low power consumption and it is low in and simple when we are doing coating on metals(53). So there are two systems like upward and downward sputtering, like in upward process the substrate is placed on top and the targeted guns in the bottom, in downward sputtering the guns, are presented in top and substrate holder is presented in the bottom. We have used the upward sputtering process by physical vapour deposition. This system works by the inert gas argon (sputtering gas) and the magnetic field is used for the control of uniformity, increasing the deposition rate and lower voltage needed over-reactive sputtering.

The DC magnetron sputtering plays a major role in a high rate of vapour deposition. It consists of three guns inside like they can fix the target in any of them namely cathode RF one and two and cathode DC. The first substrate holder is taken with the help of double-tap cello tape our material is taken titanium alloy have been taped it due to we are doing upward sputtering. We are going to use only one gun, if we

want to use more than one target we can use another hence we are using only one target we used to mask the other guns with aluminium foil. The whole magnetron sputtering is controlled by a control panel.



Fig 4.2.1: Target guns and shutter

A shutter is present inside the sputtering machine first when we start the machine the titanium silicide is coated on the shutter so if any impurities are present it will be coated on the first shutter then after 5-8 minutes we can remove the shutter. The control panel consist of some control buttons pump, pumping, wet, temperature, heat etc...Before that, we have in the vacuum chamber before 5-6hrs we have to fix our titanium alloys on the substrate holder and close them. The inert gas argon is used in this magnetron sputtering, 500V is applied to the machine.



Fig 4.2.3: Substrate holder

4.2.1 APPLICATIONS

Used in integrated circuit processing. In optical applications like for the antireflection coatings on glasses. Decorative coatings, corrosion-resistant and low friction coatings. Used in automotive that is wheels and rims, data storage, laser disk. Nowadays they are widely used in microelectronics due to the semiconductors properties and gate dielectric, passive thin-film components and sensors. Mostly well-known fields in war coating and medical implants(54).

4.3 X-ray diffraction

This is used to identify the crystallinity of the material and it gives the unit cell dimensions information. It was founded in 1895 by Wilhelm Rontgen later it was developed by Max von Laue in 1912. It is mostly used in research fields, it can be obtained by orientation at different temperatures but it doesn't give information about its chemical nature. It is a powerful non-destructive device.

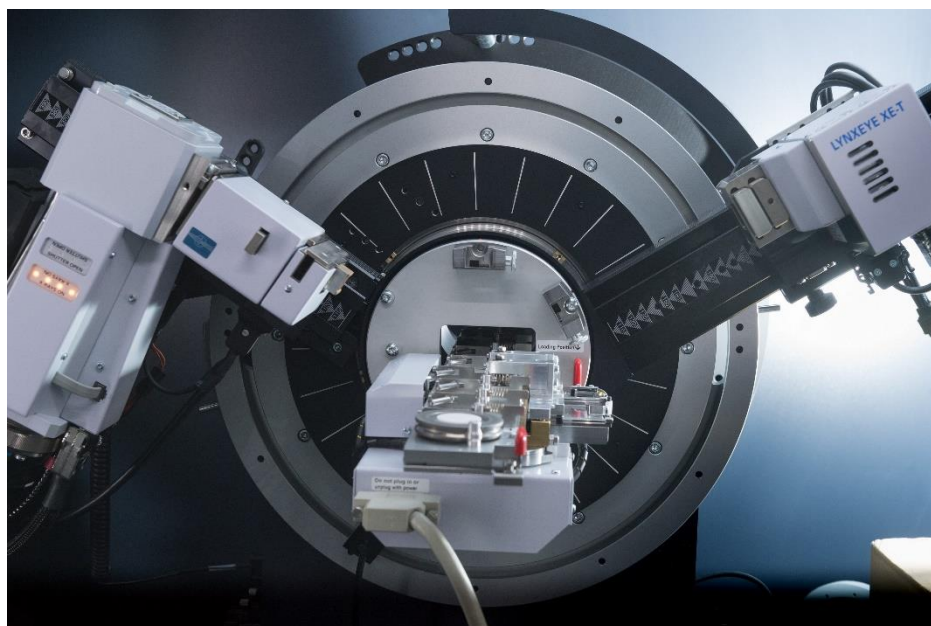


Fig 4.3: X-ray Diffraction

x-rays are waves of electromagnetic radiation. When we place our materials they scatter incident x-rays through interaction with the atom's electrons. This is known as elastic scattering. The electrons strike heavy target materials that are cathode rays. The regular array of scatterers gives a regular array of spherical waves. Thus it has some condition that successive lines of the grating will be equal to the wavelength of light. In most of the directions, the destructive interference cancels each other but they add constructively in a specific direction by Bragg's law. The wavelength of light should be Armstrong range and the most important condition is that spacing between atoms should be equal to the wavelength of x-ray. It consists of a glass tube that is to evaluate for high temperature. There are two parts cathode which contains filament and anode for x-ray targeting. Bragg's law states:- $2d\sin\theta = n\lambda$, where d is the spacing between diffracting plane, θ is the incident angle, n is an integer and λ is the wavelength. Continuously x-ray diffraction patterns show electromagnetic waves spotting on a regular array of scatterers(55).

4.3.1 APPLICATIONS

To find the crystalline or amorphous materials whether it is minerals or an inorganic compound and it is a non-destructive method. It requires only a minimal sample for analysis. To identify the orientation and to determine structural properties like lattice parameters, phase composition, strain and epitaxy. To measure the thickness of thin films and multilayers and to determine the atomic arrangement.

4.4 SCANNING ELECTRON MICROSCOPE

Scanning electron microscope(SEM) is the improvement of an electron microscope. It scans a focused stream of electrons over a surface to form an image. It was founded by Stintzing in 1927 later it went through a lot of improvements. Its principle involves the interaction of electrons with the atoms of an object. The image is formed by detected signal and merging beams. It has been built by a single electron gun, with no uses for auxiliary lenses(56)

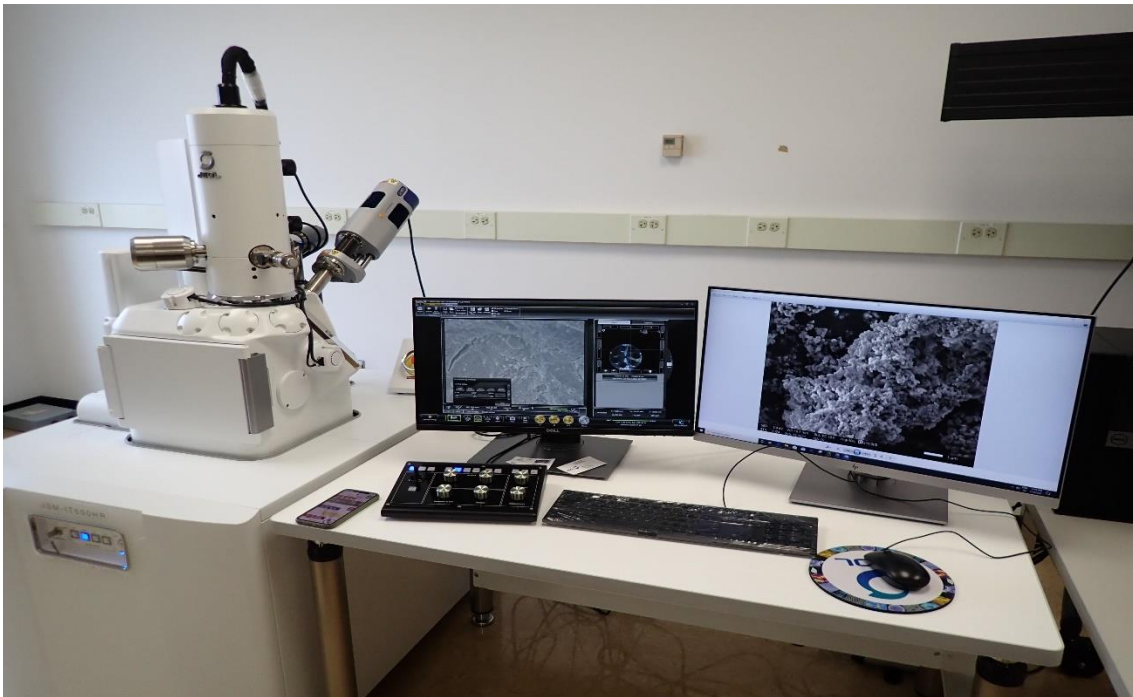


Fig 4.4: Scanning Electron Microscope

When primary electrons strike's the samples it produces the secondary electron, in this secondary electrons contains information about the object and give the 3D images of the samples. The SEM consists of an electron gun that produces a high energy electron beam a voltage is applied for the control of the emission current and the tungsten tip, the magnetic condensing lens is used for condensing the electron beam. The scanning coil is placed between condense lens and sample, the scintillator is present to collect the secondary electrons which convert into an electrical signal. This electrical signal can be fed into CRO through a video amplifier. It can be directly viewed on-screen, to examine large thickness and used in a large depth of focus. When the rays hit on the sample there will be emission of three types of beam outside of the sample that is x-ray, back reflection and secondary electrons.

4.4.1 APPLICATIONS

Used in examining the structure of very large 3D images. The results of the samples get with resolution down to the nanometer scale. Mainly used for the surface that is for topography that how it looks texture, detectable features. Then we can get the details by elemental analysis of how the atoms are arranged. It helps to examine a wide range of materials at high and low magnification without focusing on depth.

4.5 ATOMIC FORCE MICROSCOPE

It is also known as a scanning tunnelling microscope it is very high resolution that is very closely spaced with objects. It can be seen when two objects are present when it is tiny. It's a high-resolution type of scanning probe microscopy. It works on the principle of interatomic forces.

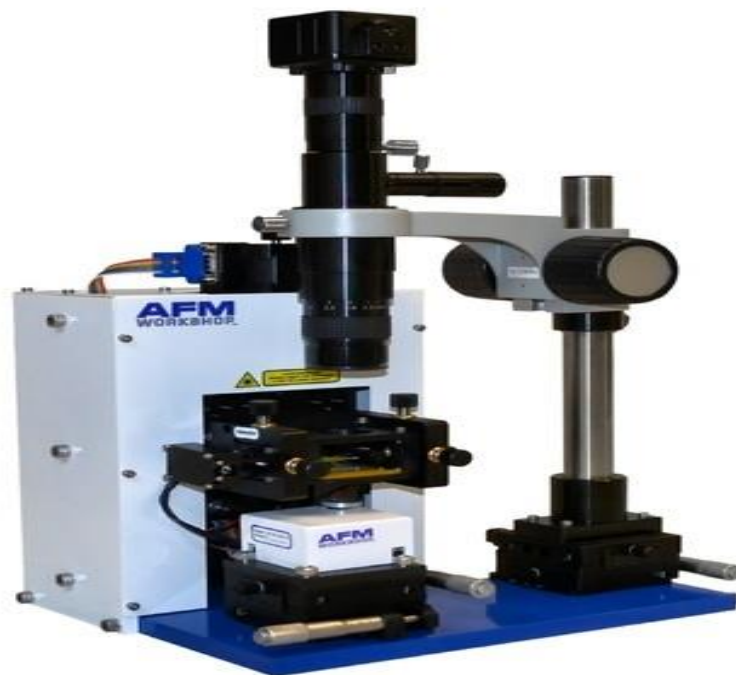


Fig 4.5: Atomic Force Microscope

There will a laser of light has been sent on the cantilever. Here they do not touch the sample they just analyze the sample so we can get the information about the sample. The sample is scanned by a tip that is mounted by a cantilever. A topographic image of the sample is taken(57).

The piezoelectric table act as a holder that allows moving in all direction. The counterpart of the tip is the sample is most convenient geometry is a planar surface. Due to sample roughness being greatly reduced in the AFM compared to other surface force techniques. The samples only need to be smooth on the scale when compared to the radius of curvature at bottom of the tip. Here they are three types of modes-contact mode here the distance between tip and sample is very less, here it has high resolution. In non-contact mode here the distance between tip and sample is very large but the resolution is low. Tapping mode is the distance between tip and sample is intermediate, the resolution is better when compared to non-contact mode. So mostly this method is preferred for a good method for topography images.

4.5.1 APPLICATIONS

It is used in various fields like cell biology, molecular biology, surface chemistry, tribology and thin film and coatings. It also measures the modulus at the surface of the material for more accuracy termed as local modules. Nowadays it has been used in vision research to find the proteins within a cell membrane for high-resolution imaging. It helps to how the changes take place in mechanical, physical properties. Used in nanotechnology, semiconductors, electronics industry.

4.6 TRIBOLOGY

This study is done for wear, friction how they interact with surfaces. From this study, we get proper grade lubricant for the specific application. The wear and friction occur through the superposition of mechanisms that challenges the control and quantify of the material. Tribological tests are so crucial for estimating tribological behaviour. It consists of different types lie solid, boundary, mixed friction. In which solid friction states that the friction is done between direct contact with solid surfaces. There will be high wear rates due to direct contact in surfaces and solid friction. It should satisfy two conditions that the force friction acting between two sliding surfaces is directly proportional to the load pressing the surfaces together and the force of friction is independent of the apparent area of contact between the two surfaces. Lubricants are used to reduce the friction between the surface and wear control of the materials.



Fig 4.6: Tribometer

4.6.1 APPLICATIONS

It is mostly used in sliding components, like bearings, seals, brakes. These are commonly used in various machines which have relative motion and requires sliding motion. It differs from macro to nanoscales which will be used in transport and manufacturing sectors. Used in various fields, like biotribology, classical tribology, green, geo, nanotribology. From this method, we can say how our materials stability. The material stability is noted based upon how and where the material can be used is fixed because their lot of energy loss take place to minimize that this study is very useful.

4.7 EPIFLUORESCENCE MICROSCOPE

A mercury lamp is used to provide ultraviolet light. The light enters and hits the dichroic mirror that reflects and allows another range to pass through them, the reflected light which falls up to the specimen. The ultraviolet light excites fluorescence within molecules in the specimen. The fluorescent wavelength is collected by objective wavelength, so the light passing through barrier filter and dichroic makes the image which is visible from the eyepiece, this fluorescence microscope combines and forms another type of microscope with some added

features to it. The fluorescent molecules with specimen can occur naturally by self-dye too. Here a high-intensity light source is used which excites a fluorescent species in the sample. Its species turn to a lower energy light when a longer wavelength produces the magnified image(58).



Fig 4.7: Epifluorescence Microscope

4.7.1 APPLICATION

It is more powerful focused light sources like led, lasers etc., mostly used in getting high resolution of 3D images of specimens. They produce exact images of thick samples by going depth point by point taking images that are visible from the eyepiece. Used to see structures and measure physiological events mostly in microbes. To study Ph, DNA, enzymes various fluorescent indicators are used. Imaging specific cells with the larger population and also with small specimens.

4.8 PROTEIN ADSORPTION STUDIES

Protein adsorption is the first process when the implantation is done. After the implantation, this is the coated titanium alloy is taken and the coated surface is equilibrated in isotonic tris buffer of 0.197g which is diluted in 25ml of water for about

20hrs. After that titanium alloy is taken out slightly washed in distilled water then it is transferred into the plasma of 1.5ml for 3hrs. The plasma consists of different types of proteins present in our human body like collagen. This is done so the protein which is present in plasma get attached to alloy. Then again it is taken out again washed in distilled water is transferred into 1ml of Sodium dodecyl sulphate (SDS) this is for the protein which is attached in alloys will be bonded to break that It is placed in 20hr in SDS solution.

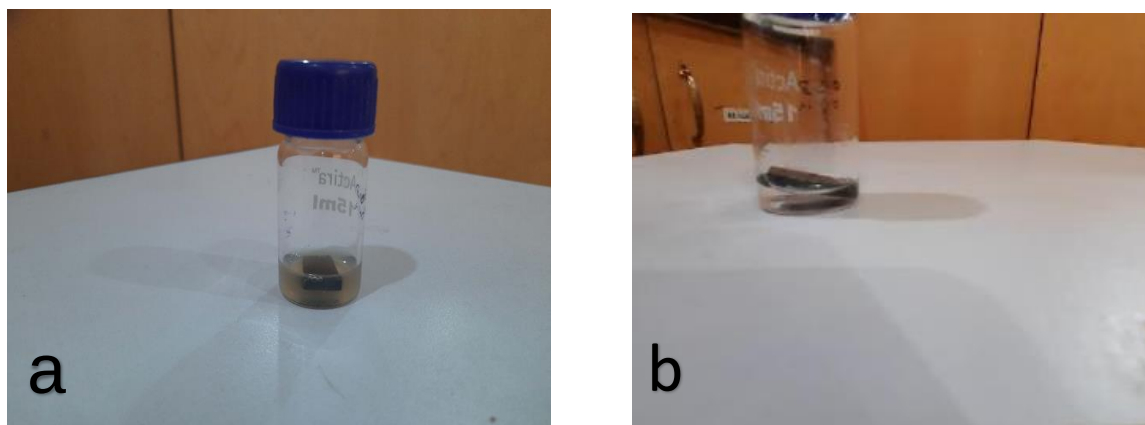


Fig 4.8: (a) Coated sample is dipped in plasma (b) Coated sample is dipped in sodium dodecyl solution.

Now the solution is taken out separately this solution is taken out for the protein estimation by Lowry's method. First to prepare two types of reagents I and II. The first reagent is prepared by taking 0.1 N of NaOH which is prepared by 0.4g of NaOH is taken and dissolved by 100ml of distilled with it 2g of Na_2CO_3 is added into it. Then 1% of NaK tartrate and 0.5% of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is taken both are dissolved separately by 50ml of distilled water. Now, 48ml of the mixed solution of NaOH and Na_2CO_3 is taken and 1ml of NaK and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is mixed with 48ml of solution. Now the 50 ml is ready which is our reagent I. The II reagent is floin phenol. We have to prepare a standard solution of bovine serum albumin (BSA) which is prepared by 1mg/1ml. This BSA solution is taken 5 different test tubes and one more test tube is our SDS solution which was kept for 20hrs with alloy is taken. Now to add in each test 4.5ml of I reagent is added then it is incubated for 10mins. After that 0.5ml is added and incubated for 30mins. When this second solution is added it will be in yellow colour so after 30mins it changed into blue colour then it is

observed under the ultrasonic where the OD is determined. Then a graph is drawn between OD and the concentration. From the graph, we can find protein adsorption.



Fig 4.8.1: Presence of protein solution

4.9 PLATELETS ADHESION STUDIES

Thrombogenicity study is been done by platelet adhesion analysis. A two set of samples coated and uncoated is taken and made to dissolve in distilled water for 24hr. The human blood is collected by the volunteer by venipuncture into ethylenediaminetetraacetic (EDTA) containing sterile tubes. Then the blood was centrifuged at 3000 revolutions per minute(rpm) for 3mins to get platelet-rich plasma(PRP). The PRP is taken out and kept separately then again the PRP is centrifuged at 4000rpm for 10mins. Then the platelets have been separated and one drop of platelet is taken mixed with one drop of acridine orange so that the image of platelets can be viewed clearly in the optical microscope.

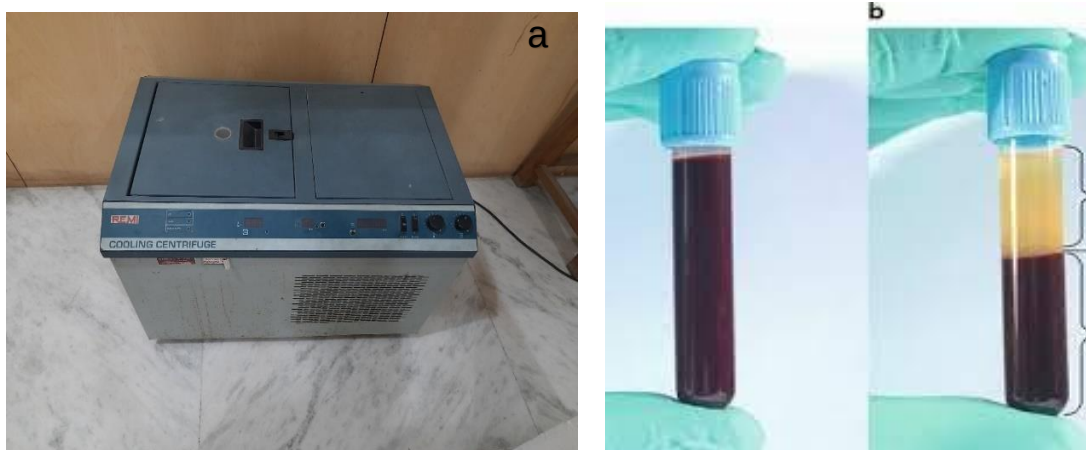


Fig 4.9: (a) Centrifuge machine (b) Separated blood and plasma

Then the separated platelets are re-suspended and diluted with 1X phosphate-buffered saline(PBS). We are using PBS solution because it is having a constant pH and it is isotonic and non-toxic to cells. Both the coated and controller should be immersed in the PBS solution of 1ml. for an hour. Then the platelets are collected separately by pipet then mixed with the 5ml of PBS solution slowly this process is done slowly so platelets do not active. After it gets mixed we have to take 2ml of the mixed solution and keep it in the glass slide in which our material is kept. Then to add formaldehyde solution it the for 15mins then at different concentrations ethanol is taken about 50%, 70%, 100%. Form each a drop is taken out to mix with a solution which is kept in glass slide for 3mins separately. After a few minutes when it gets dry then it is observed under an optical microscope.

4.10 BIOMINERALIZATION

This study is done so that which mineral deposits on the surface at what concentration can be achieved. It shows the heterogeneous accumulations, of both organic and inorganic. It has two types extracellular and intracellular in which the mineral formation within the cell is intracellular and outside the cell is extracellular biomineralization which mostly involves inorganic(59). It includes all the studies of biologically produced materials like bone, teeth, shells. After implantation, the first procedure that occurs is biomineralization.

The ions present in our body fluid like calcium, potassium get attached to the coated surface. First, take SBF (simulated body fluid) solution is taken it contains ions that are present in our body fluid then our coated and uncoated material is dipped into

it. This process takes place for a few days, after that the weight of the samples reading is taken, then we have discarded the solution again to add a new solution and counted for a few days for the next reading. This procedure is followed for 14 days in which three times the solution has been discarded. The graph is a plot between the weight of the sample and the number of days.

4.11 CONTACT ANGLE

This is a unit of measurement for the wetting of a solid by a liquid (wettability). This is defined as the angle formed by the liquid at the three-phase boundary. The surface wettability of coated and uncoated samples was found by contact angle measurement. By using water which is wetting liquid is taken through pipet 3 microliter of water is taken and dropped on the material for the configuration between the measurements. After one min the wettability characteristic of the

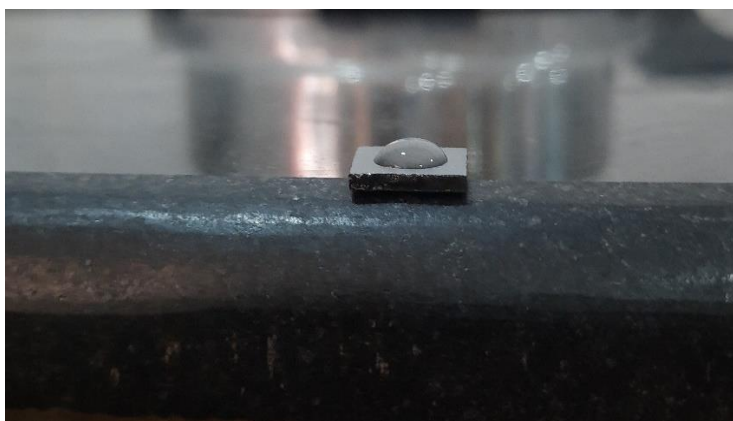


Fig 4.11: Sample with liquid for Wettability test

material is measured by the expression $W_{adh} = \gamma_{lv}(1 + \cos \theta) = -\Delta G$, which has the surface tensions of the testing liquid and contacts angle of the samples(60).

4.12 CORROSION STUDIES

The electrochemical behaviour of the test samples was determined in artificial blood plasma solution (ABP) which have the composition of diverse particles like sodium, calcium, potassium, at room temperature. The study was done in a standard three-cell electrode setting with an uncovered zone of 1cm². Open circuit potential (OCP) was measured about before corrosion tests to accomplish a steady potential for the working electrode. Electrochemical impedance spectroscopy (EIS) was carried out in the frequency range of 100 kHz to 10 MHz with an AC amplitude of 10 mV. After

EIS analysis, potentiodynamic polarization (PDP) was studied at a potential of ± 500 mV vs SCE with a scan rate of 10 mV/s. The corrosion potential (E_{corr}) and corrosion current density (I_{corr}) were determined by the tafel plot(61).

4.13 BACTERIAL ADHESION STUDIES

The bacterial strain *Streptococcus mutans* (MTCC-497) was obtained from the Microbial Type Culture Collection and Gene Bank (MTCC) in India. The bacterial strain was incubated in the cardiac infusion broth at 37°C for the entire night. Following incubation, 0.1 mL of bacterial culture was transferred to a conical flask containing 200 mL of heart infusion broth. The coated titanium silicide and uncoated titanium alloy samples were then immersed in a conical flask containing bacterial culture and incubated in a shaking incubator for 24 hours. The samples were gently rinsed with distilled water after a day of incubation to eliminate unattached bacterial cells. The samples were then ultrasonically separated from adherent bacterial cells in a phosphate-buffered saline (PBS) solution. Finally, a serial dilution was performed, and 0.1 ml of bacterial suspension was put to agar plates, which were then incubated for 24 hours at 37 degrees Celsius in a bacterial incubator. To measure the anti-adhesion efficacy of coated and untreated samples, the number of colony forming units per square centimetre (CFU/cm²) was computed(62).

4.14 CYTOTOXIC ANALYSIS

For the cytotoxicity study, the National Centre for Cell Sciences (NCCS) in Pune, India, provided mouse fibroblast cells (NIH 3T3 cell line). The cells were grown in GIBCO's Dulbecco's Modified Eagle Medium (DMEM), which included 10% foetal bovine serum (FBS) and 1% non-essential amino acids (GIBCO). Every two days, the medium was subcultured and kept at 37° C in a 95 percent humidified atmosphere with 5% CO₂. The cells were treated for 5 minutes with 0.1 percent trypsin and 0.1 percent ethylene diamine tetraacetic acid (EDTA) to remove them from the walls. The cytotoxicity of test materials was assessed using a direct technique based on ISO-10993-parts. The test samples were grown at 37°C on a 12-well plate with fibroblast cells in DMEM.

4.15 STYLUS PROFILOMETER

This machine is used to detect the surface. A stylus profilometer is slower than non-contact approaches because it involves physical motions in X, Y, and Z while keeping contact with the surface. The coated sample has been placed in a stylus profilometer so through the probe the how much level of coated is done when it is compared to non-coated sample is identified.



Fig 4.15: (a) Stylus profilometer (b) Electron tape

The electron tape is placed on the sample before coating. So, where the tape is present it deflects the ions it doesn't allow any sample to coat it. After coating with the help of a stylus profilometer with one sample it can be determined how much thickness of material have been coated.

4.15.1 APPLICATION

Stylus profilometry is frequently used to measure feature sizes and step heights in patterned and etched materials. It is a method to extract topographical information from the surface of the earth. Profilometry is used to determine surface morphology, step heights, and surface roughness. This can be done with either a physical probe or light.

4.16 POLISHING MACHINE

This machine is used to polish soft metals like copper, titanium, plastics and brass. There are many types of polishing machines even the paints are been removed, it removes the extremely fine imperfection. It works like a magnet is placed with its a silicide carbide sheet is taken there are different types of grade sheets is available we have taken 2000A grade sheet and fix into the magnet. The time is fixed at about 10mins with 1000 rounds per min. when the machine starts rotating slightly water is poured. With the help of double cello tape, the sample is made to stick in our fingers and kept in a rotating silicide carbide sheet. After 10mins all the impurities present in the sample (titanium alloy) is removed. This is how the machine works.



Fig 4.16: Polishing machine

4.16.1 APPLICATIONS

Polishing can be used to improve and restore the appearance of metal parts and objects such as metals, cookware, kitchenware, plastics. The process of buffing is also used to make light reflectors. It removes a substantial amount of material.

CHAPTER-5

RESULT AND DISCUSSION

5. RESULTS

5.1 SEM ANALYSIS

SEM stands for scanning electron microscope is used to get the morphology of the sample.

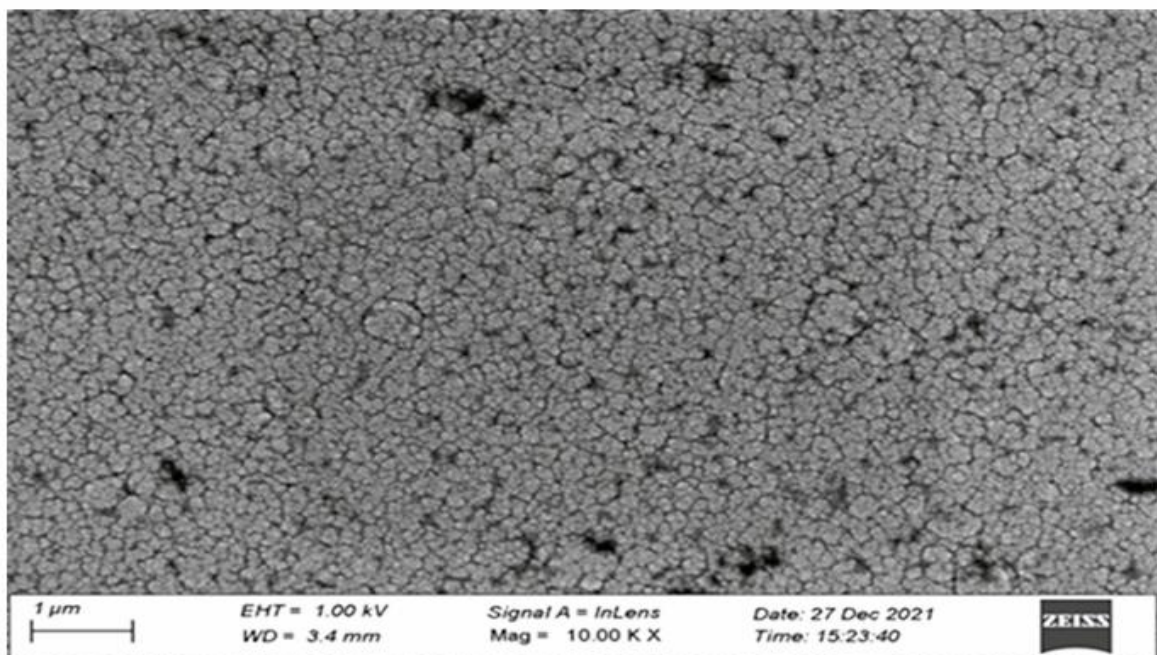


Fig 5.1.1: Morphology of the sample

The electron high tension is displayed in the data zone in the range of 200v to 30kv. The working distance at which the beam is focused up to 2μm the length is measured. Here they have used an objective lens at 5X, the eyepiece is watched under 10X so that the morphology will be 50times closer than the normal samples.

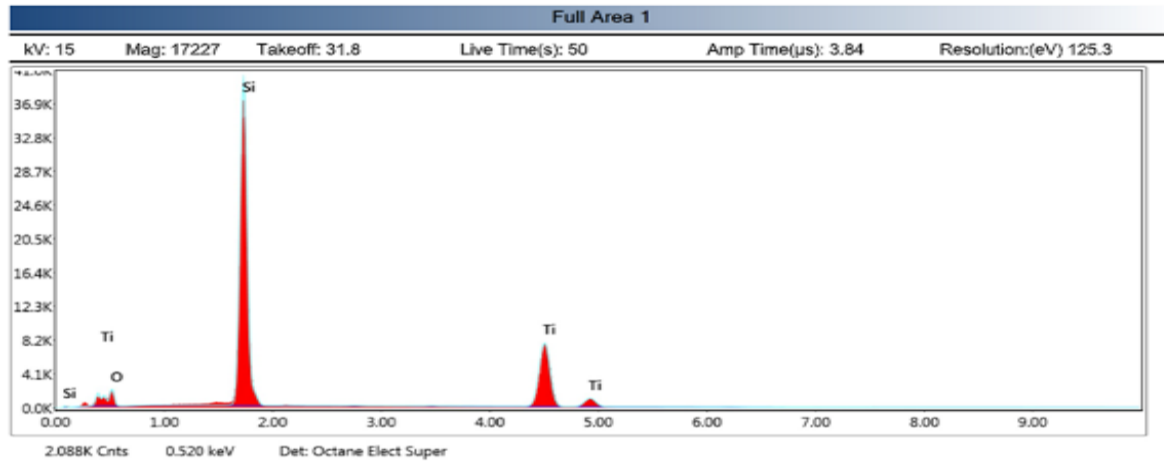


Fig 5.1.2:EDAX Spectrum

Here to identify and quantify the present sample x-ray energies are used. The x-axis is energy (Ev), the y-axis is counts.

Table 5.1: Weight and Atomic percentage

Element	Weight%	Atomic%	Error %
O K	8.71	17.12	11.28
SiK	49.44	56.39	2.99
TiK	41.85	27.49	2.4

So, from the above table, it can be concluded that the weight percentage of oxygen is 8.71, Silicon is 49.44 and titanium is 41.85. The atomic percentage of oxygen is 17.12, silicon is 56.39 and titanium is 27.49.

5.2 CONTACT ANGLE

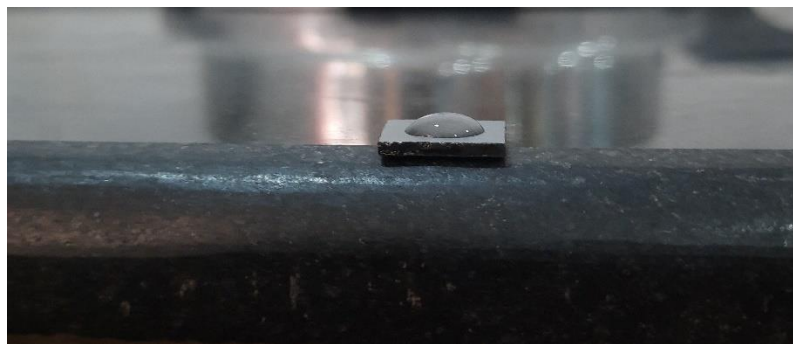


Fig 5.2: Liquid placed on a coated sample

To check the wettability of the coated sample by placing 3 microliters of liquid on it. It has been proved that when the contact angle is above 90° then it is hydrophobic and below 90° it is hydrophilic.

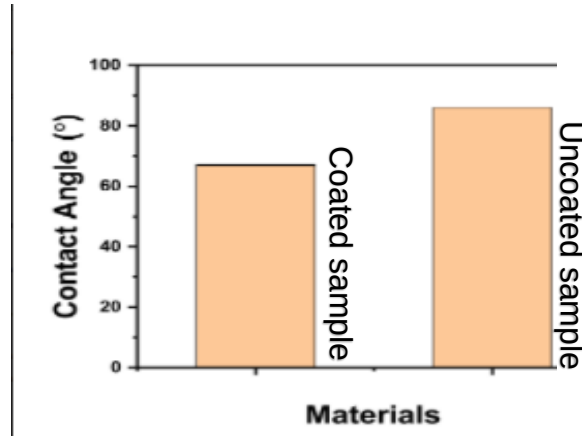


Fig 5.2.1: Graph for measurement of contact angle

It has been observed that the coated sample has a 67° and the uncoated sample has around 85 to 87° . Hence the coated sample has a hydrophilic nature. The x-axis shows the contact angle and in the y-axis are our coated and uncoated materials.

5.3 BIOMINERALIZATION

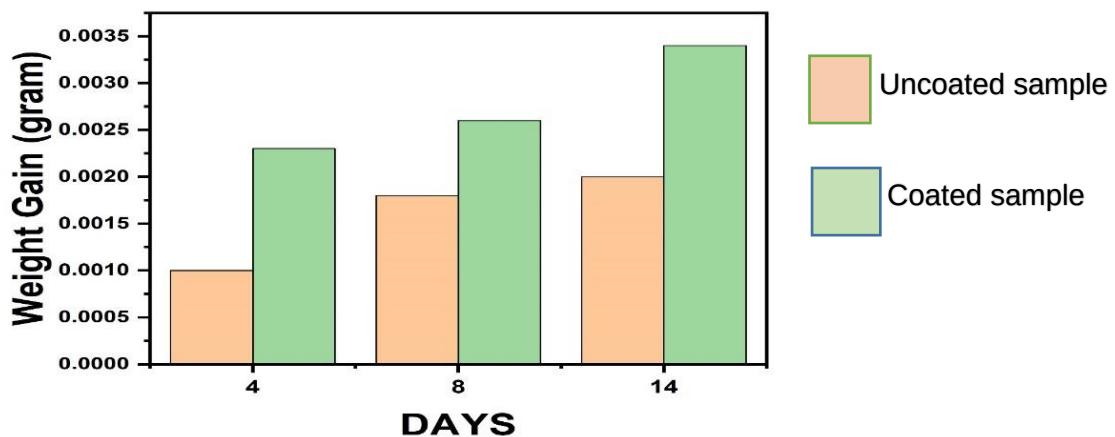


Fig 5.3: Graph for biomineralization

After implantation, biomineralization is the first procedure to take place. Both coated and uncoated sample has been dipped in simulated body fluid for 4 days then their weight is noted, like that same as 8 then 14 days. Its weight is measured and the

graph is plotted. So in fig 5.3, the graph is plotted between weight gain in the x-axis and the number of days on the y-axis. From the graph, it can be concluded that on an increase in the number of days there is also an increase in weight gain so there is deposition minerals is taking place. It can be noticed that comparing uncoated samples the coated sample has high weight gain. Hence the coated sample shows well in the biocompatible surface.

5.4 BACTERIA ADHESION

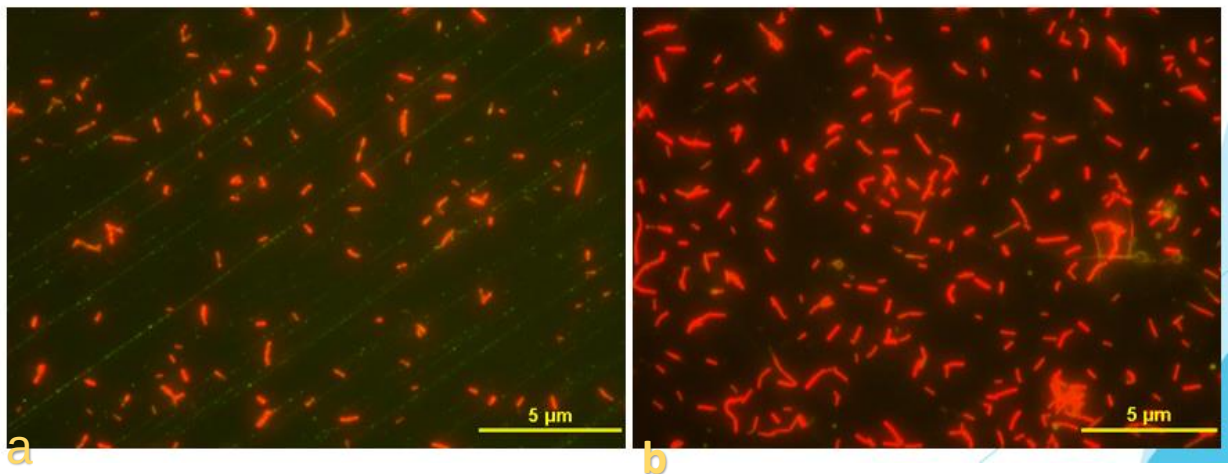


Fig 5.4 (a) Coated sample (b) Uncoated sample

From the above fig 5.4, it can be concluded that the bacteria in the uncoated sample is high when compared to coated sample (titanium silicide). It is an important process that allows bacteria to adhere to the surfaces. Differentiating the samples, the titanium silicide is less toxic. It prevents tooth decay-causing *Pseudomonas aeruginosa* bacteria invasion rather than titanium alloys substrate. It can be said my sample is non-toxic.

5.5 CYTOTOXIC ANALYSIS

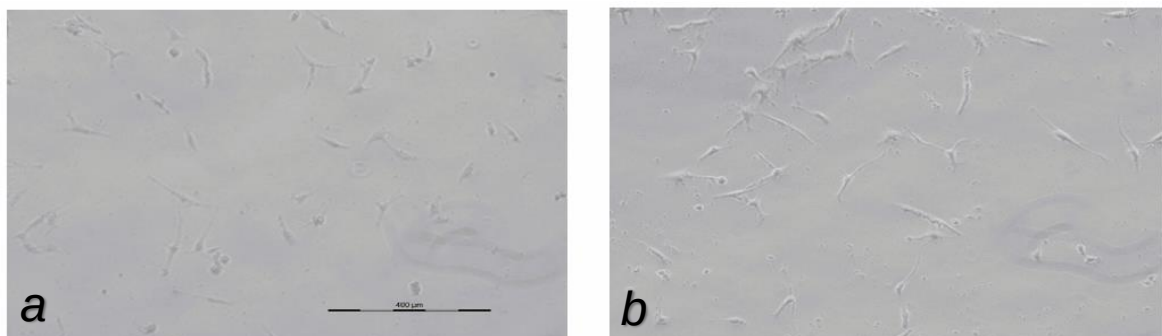


Fig 5.5: (a) Uncoated sample (b) Coated sample

The cytotoxic analysis is an in vitro test that determines whether a medical device may kill cells owing to hazardous compounds leaking or direct contact. It will be determining a test substance's potential toxicity. So, from fig 5.5 it is observed that the coated sample has better wound healing capacity compared to uncoated titanium alloy.

5.6 PLATELETS ADHESION STUDIES

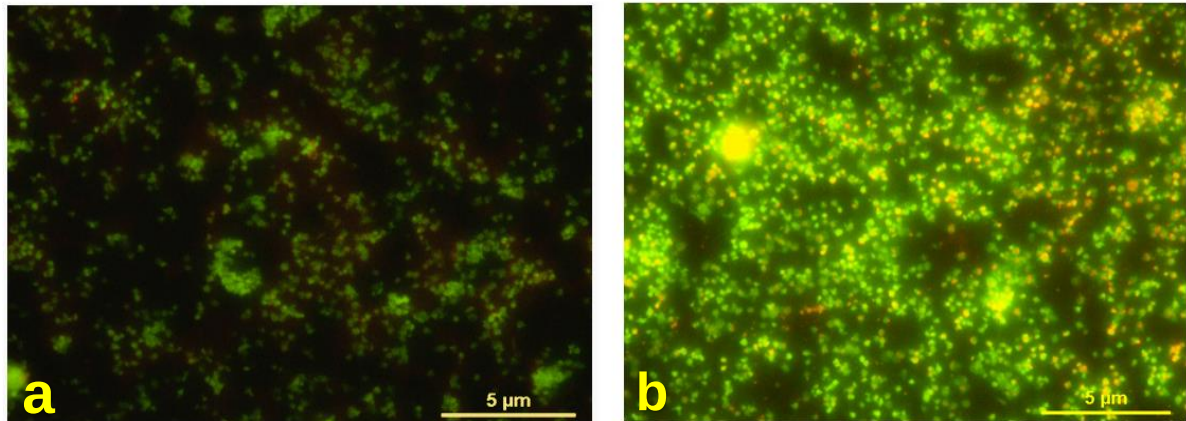


Fig 5.6: a)Titanium silicide coated sample b)Titanium alloy uncoated sample

Platelet adhesion is a critical role in the response to vascular damage, and it's usually thought of as the initial stage in the process. Platelets bind together or form individual connections with extracellular surfaces. The platelet behaviour has been shown in fig 5.6 it shows that in titanium silicide there is no platelet activation were in titanium alloy there is a large number of platelet activated cells can be noticed.

5.7 TRIBOLOGY

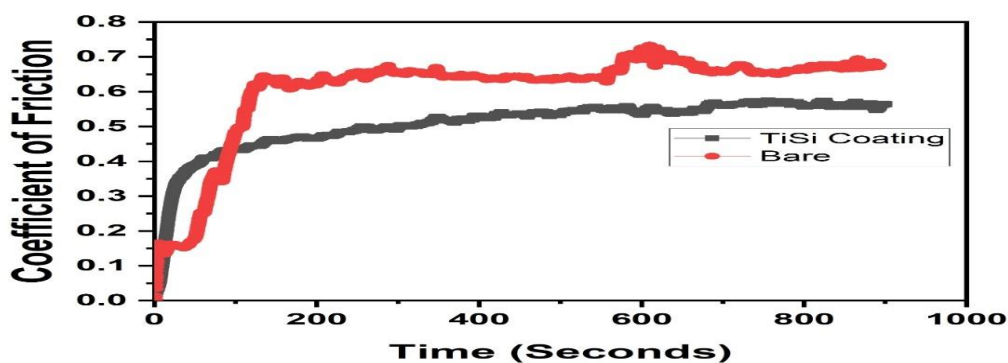


Fig 5.7: Coefficient of friction vs time

Fig 5.7 shows how the sample's friction and wear effects and the sample stability is determined. By this, we can determine the stability of the sample and it has been observed the graph is a plot between the coefficient of friction and time, it is concluded that the coated sample have large stability than the uncoated sample. From it can be concluded that when the coefficient of friction reaches 0.6 the coated sample becomes almost constant and the uncoated alloy reaches more than 0.6 so, the coated material has good stability.

5.8 PROTEIN ADSORPTION

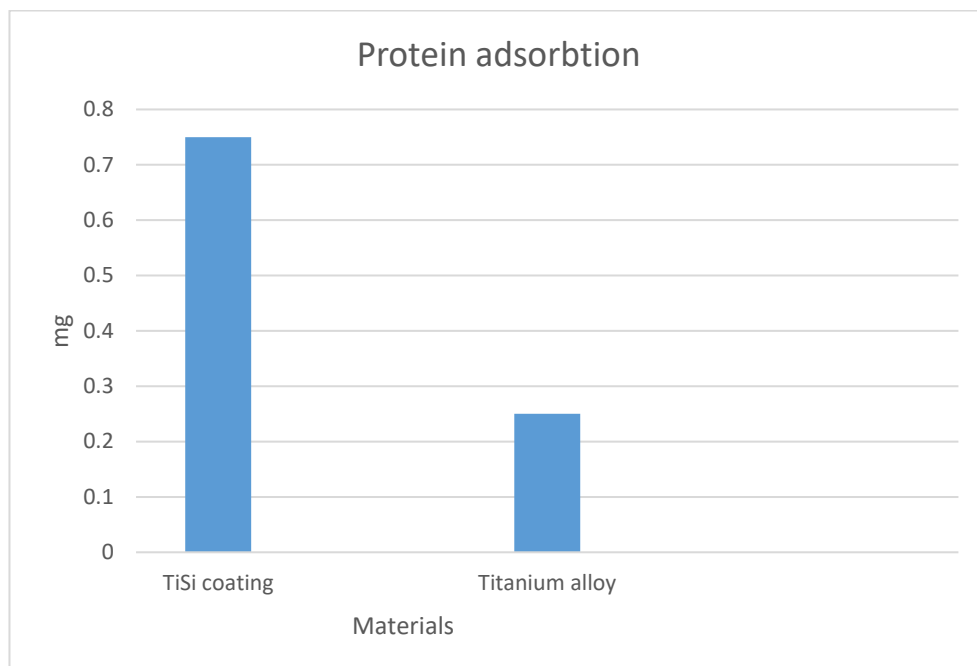


Fig 5.8 Protein adsorption behaviour

Fig 5.8 shows the adsorption of protein is high in the coated sample when it is compared to the uncoated sample. So it can be concluded that the titanium silicide alloy has better biocompatible nature when it is compared to titanium alloys.

5.9 AFM ANALYSIS

Surface roughness measurement with high spatial resolution (AFM) analysis generates images with a near-atomic resolution for surface topography measurement.

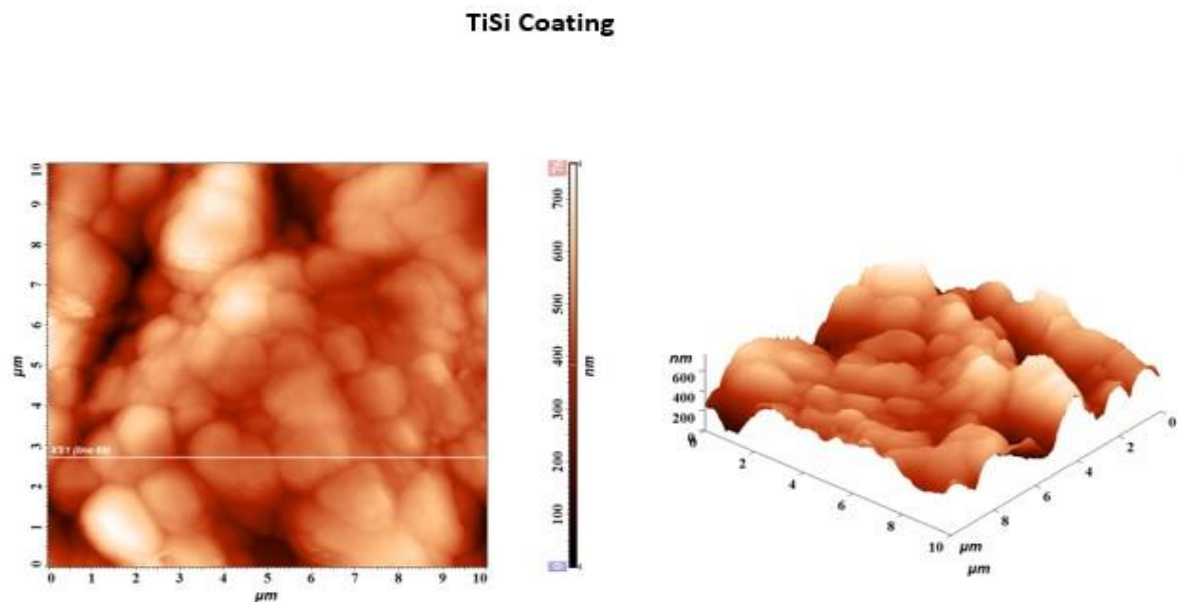


Fig 5.9 Surface topography of titanium silicide sample

AFM was used to assess the coated sample's roughness and topography. Surface topography photos in three dimensions (3D) were taken. The surface of the coating was smooth. The topographical aspects of the coated surface did not show any significant differences.

5.10 CORROSION ANALYSIS

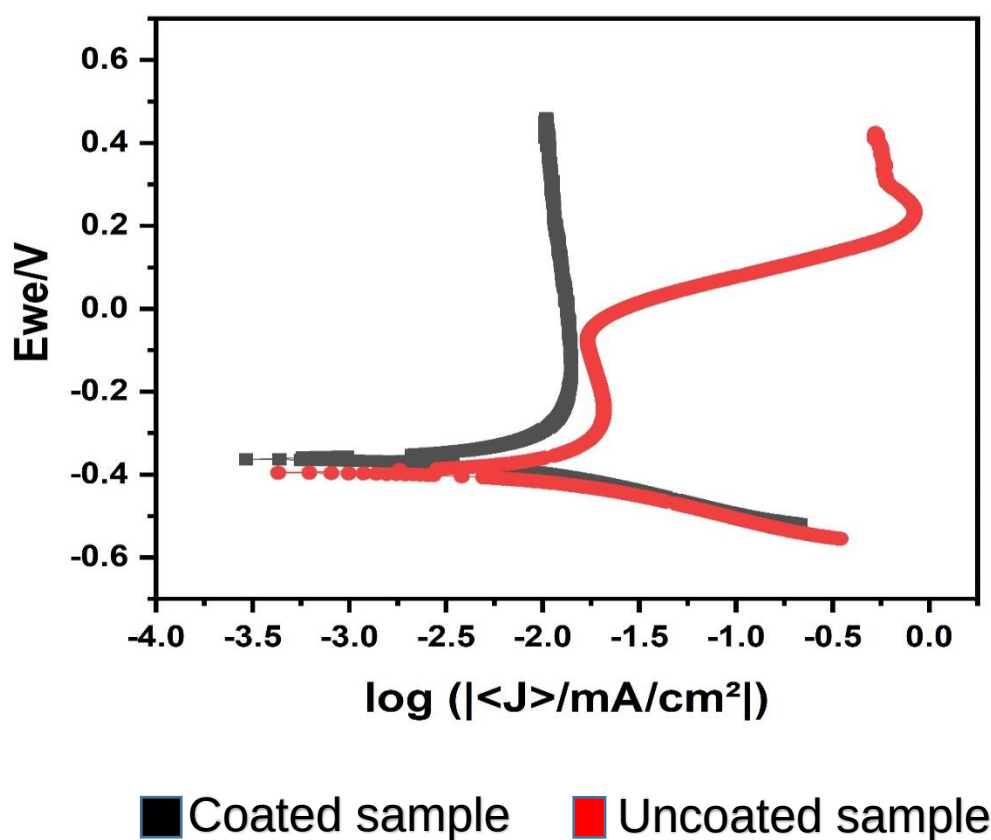


Fig 5.9: The corrosion behavior of coated and uncoated sample

The figure 5.9 represents the polarization curves of titanium silicide coated sample and uncoated titanium alloy. The titanium silicide sample shows strong passive behavior. So it has an excellent corrosion resistance property.

CHAPTER-6

SUMMARY AND CONCLUSION

SUMMARY

In the present study, the titanium silicide was coated on titanium alloy by using the dc magnetron sputtering method by physical vapour deposition, this method is used because of the high rate of deposition on the targeted material. The titanium silicide was our target and the alloy was bought directly from the industry. The coating was done in glass and alloy, after the coating it has undergone SEM analysis for morphology and elemental analysis of the coating was taken by EDAX. X-ray diffraction was studied to find the crystallinity. Further biological studies were done for protein adsorption, platelets adhesion, cytotoxic studies, biomineralization. This is done to check their biocompatibility when it is compared to other metallic biomaterials. These are widely used in biomaterials.

CONCLUSION

In this work, the titanium silicide film was developed by the dc magnetron sputtering method. The surface hardness and wear analysis were determined by nanoindentation and tribometer analyses have been done in which material stability was determined. With the help of EDAX, the weight% and atomic % were investigated. The titanium silicide film surface was confirmed by SEM. Hydrophobic nature was observed in the coated sample compared to the uncoated sample by knowing its wetting ability. With help of human blood, both the platelet and protein tests were done showed good compatibility. Better adhesion property was achieved and showed excellent corrosion resistance when the electrochemical studies were done with artificial blood plasma. In cytotoxic analysis, it has proved that it has better-wound healing capability.

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