

SYNTHESIS OF TITANIUM OXIDE (TiO₂) NANOTUBES ON TITANIUM ALLOY IN-SITU VOLTAGE CONDITION FOR BIOMEDICAL APPLICATIONS

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology degree in Biomedical Engineering

By

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**DEPARTMENT OF BIOMEDICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

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

Accredited with Grade "A++" by NAAC

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APRIL - 2024



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **DHARANI K (40240008)** who have done the project work and carried out the project entitled "**SYNTHESIS OF TITANIUM OXIDE (TiO₂) NANOTUBES ON TITANIUM ALLOY IN-SITU VOLTAGE CONDITION FOR BIOMEDICAL APPLICATIONS**" under our supervision from November 2023 to April 2024.

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I, DHARANI K (40240008) hereby declare that the Project Report entitled "SYNTHESIS OF TITANIUM OXIDE (TiO₂) NANOTUBES ON TITANIUM ALLOY IN-SITU VOLTAGE CONDITION FOR BIOMEDICAL APPLICATIONS" was done by me under the guidance of Dr. A. Sabarivani, M.Tech., Ph.D., (Internal Guide) and Dr. V. Sivaprakash, M.E., Ph.D., (External Guide) is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Biomedical Engineering.

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ACKNOWLEDGEMENT

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

I would like to express my sincere and deep sense of gratitude to **Dr. T. Sudhakar, M.sc., Ph.D.**, Head of the department, Dept. of Biomedical Engineering, Internal Project Guide **Dr. A. Sabarivani, M.Tech., Ph.D.**, Dept. of Biomedical Engineering, and our External Guide **Dr. V. Sivaprakash, M.E., Ph.D.**, Dept. of Mechanical Engineering, for their valuable guidance, suggestions, and constant encouragement paved the 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 Biomedical Engineering who were helpful in many ways for the completion of the project.

I would like to express my thanks to my parents for their unwavering support to me during these years.

ABSTRACT

Materials and alloys are highly preferable for biomedical, clinical, energy and solar application. Electrochemical anodization of surface modification technique is highly preferable for biomedical application. The coating thickness surface treatments are remarkable for enhancing the material performance. A quality high-resolution surface morphological analysis is used in various research areas. Alpha and beta are high quality materials, and they are highly demanded in use. Usage of titanium and their application in the biomedical field is trending under research. The lightweight and high strength titanium are highly motivated to be usable in the Orthopedic (dental and bone) replacements. In this research work anodization of TiO_2 nanotubes on titanium alloys, are fabricated with the use of ethylene glycol, NH_4F , addition of super materials. The nanotubes surface morphology of nanotubes has been analysed by HR-SEM, The phase were characterized by XRD analysis , The surface wetting behaviour annealed by contact angle method. Fabrication TiO_2 Nanotubes under 40V.

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LIST OF ABBREVIATIONS

EC-SERS	Electromechanical Surface-Enhanced Raman Spectra
EDX	Energy-Dispersive X-Ray Spectroscopy
EG	Ethylene Glycol
FE-SEM	Field Emission Scanning Electron Microscopy
PVD	Physical Vapor Deposition
PLA	Polylactic Acid
p-NP	P-NITROPHENOL
SPEs	Screen-Printed Electrodes
TNA	Titanium Nanostructures
TNTs	TIO ₂ NANOTUBES
XPS	X-ray Photoelectron Spectroscopy
XRD	X-RAY DIFFRACTION

CHAPTER 1

INTRODUCTION

1.1. TiO₂ NANOTUBES

Titanium (Ti) is one of the most promising materials for biomedical devices due to its high corrosion resistance and specific combination of strength and biocompatibility. Researchers have recently been focusing close attention on the processes involved in fabricating biomedical devices. Because titanium alloys are very adaptable in the biological environment, they are the ideal material to use when creating biomedical implants. But the substance is deadly to humans since it has poor biocompatibility properties in its received state. It is necessary to modify the outermost layer of a Ti alloy having toxicity behavior to improve its bio adaptability. A variety of methods, including sol-gel, hydrothermal, chemical, physical, even electrochemical anodization, can be used for altering the surface of materials. The most simple and convenient method for surface modification among all these procedures is electrochemical anodization. When compared to alternative methods, electrochemical anodization requires a minimal amount of area and energy to create stable, compact TiO₂ nanotubes. (Krajewski.,et.2023)

According to the idea of field-assisted separation, the presence of ion nanoparticles in the electrolyte during the anodization process allows for continuous etching, which forms oxide layers over the nanotubes. Nanotubes can also be fabricated using Ti alloys as substrate materials. The completed shape depends significantly on anodization parameters including anodization duration, voltage, temperature, and most importantly the electrolyte composition. (Bencina.,et.2019). Since no nanostructures occur in the electrolyte without water addition and either or nanotubes are created with increasing water content, we studied the influence of water content on the development of nanostructures using Ethylene Glycol (EG) based electrolytes. Anodic oxidation is also called electrochemical anodization, because of its affordability and ease of use, the anodic oxidation procedure is one of the most often used methods. The anodizing parameters are easily modifiable to control various geometric factors, such as the diameter and length of the nanotubes. (Abukabda.,et..2016).

Maintaining the right temperature in electrolytes produces good results. At around 36°C, different input potentials and temperatures result in a nanotube length of approximately 7 μm , depending on the anodization period, water content, and input conditions. Based upon the anodizing duration, water content, or input conditions, a nanotube with a length of roughly 7 μm can be produced at varying temperatures and input potentials around 36°C. This was found that the development of the nanotubes development onto the materials was greatly stimulated by a proper degree of water content. It was possible to identify the rib formation and the nanotube creation on an electrolyte with a high-water content. To attain sufficient attachment of cells with bone and associated regions which are just in direct touch with the thin surface layer nanotubes are required. The water content percentage in the electrolyte had a significant impact on the shape of the nanotubes that developed because it influenced the breakdown of F^- or H^+ ions throughout the anodization process. (Haghi.,et.2017)

Clinical implant applications in the biomedical area often failed due to the implant surface's inability to support new bone growth, resulting in the implant's weak bonding to adjacent bone and the subsequent creation of tissue that is fibrous. To get around this, the implant's surface needs to be changed. TNT cultivated in calcium phosphate (HAp) has excellent osseointegration and biocompatibility, making it a promising material for implants. To the best of our knowledge, there are currently no published reports on the usage of these implants in people. However, some reports are available on the use of TNT as an implant in the animals. Over the past 15 years, research on the electrochemical anodization method of TiO_2 nanotube synthesis has been published. Very few studies on the impact of electrolytes water percentages have been published, even though many are being conducted on nanotubes. The current study examines the effects of the water concentration in the electrolyte by assessing that mechanical, physical, or chemical characteristics of TiO_2 nanotubes as well as the length of the nanotube. (Elahi.,et.2018)

According to the findings, the mechanical and physical behavior of the nanotube can be enhanced, and consequently, the material performance, by increasing its length and crystallinity. (Denchai.,et.2018). Also, the surface morphologies of the nanotubes were examined using a high-resolution scanning electron microscope (HR-SEM), nanoindentation was used to assess the nanomechanical properties, and corrosion behavior was investigated.

In all corrosion analyses were carried out using simulated body fluid (SBF) solution.
(Adan.,et.2016).

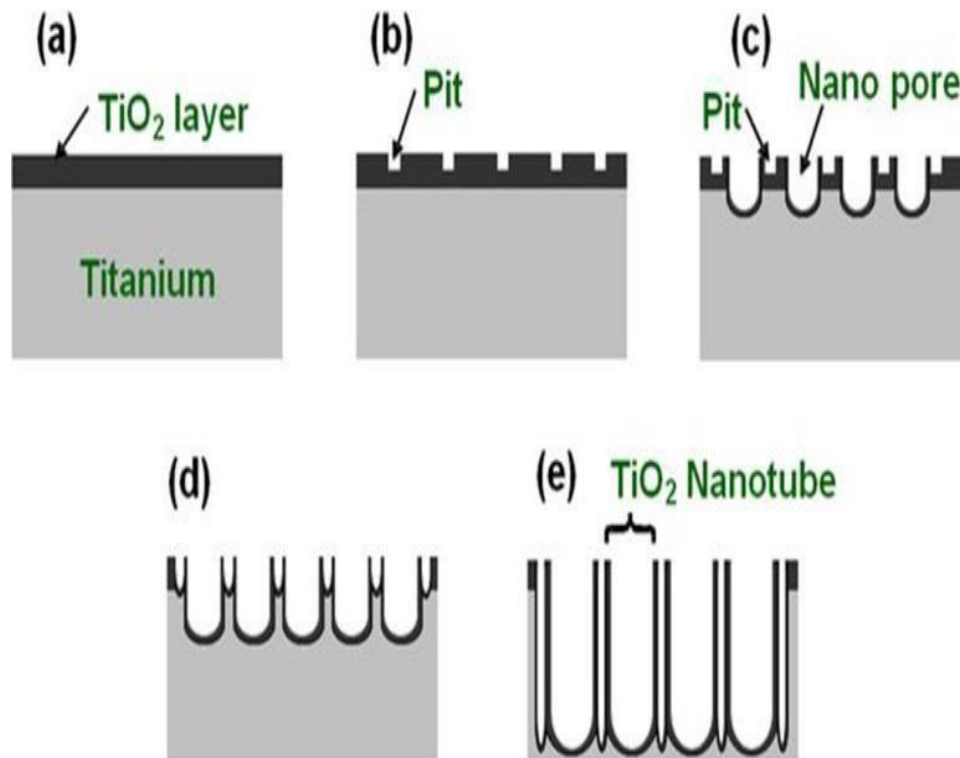


Fig:1.1: Schematic illustration of anodic synthesis TiO_2 nanotubes.

CHAPTER 2

LITERATURE SURVEY

2.1 LITERATURE REVIEW

Saharudin et al., (2018) concentrated on this work have said one of the multifunctional metal oxides that has been studied extensively is TNA. Because of certain advantages, including a substantial area of surface, a comparatively long electron diffusion length, and biological compatibility, it is possible to grow permeable anodic nanostructure, specifically self-organized and highly organized TNA through anodization.

C.Adan et al., (2016) of this work discussed that a highly organized TiO₂ nanotubes array of photoanodes' physical and photoelectrochemical characterization has revealed a strong association with their role for photocatalytic or photo electrocatalytic reactions. Due to their larger surface area, an increase of the nanotubes' length significantly accelerates both the observed reaction rates for photocatalysis and photo electrocatalysis.

Zhang et al., (2013) of this work have created an effect of water content fluoride concentration, applied voltage oolites at room and anodization time on the crystal structures and morphologies of the anodic TiO₂ films prepared in EG based electrolytes were NH₄F added, applied, and investigated. It is shown that well crystalline TiO₂ (anatase) films can be grown with higher applied voltage, longer anodization time, higher fluoride concentration and appropriate adding water.

Junkar et al., (2018) investigates about TiO₂ nanotubes, prepared by electrochemical anodization of a Ti foil, were annealed to obtain the transition of the amorphous to anatase crystal phase. The effect of the crystalline material of the interactions with whole-blood-derived platelets was studied. The paper investigates the performance of annealed TiO₂ nanotubes in interactions with blood platelets.

Nesheva et al., (2019) of this work looked at anodic oxidation that has been used to develop an array of nanotubes made of TiO_2 with four distinct inner diameters (60, 65, 80, and 120 nm) on Ti6Al4V alloy substrates. The surface structure of as-prepared samples was examined using FE-SEM, which revealed that nanotubes are evenly spaced across the Ti 6 Al 4 V surface and are open at the top. The amorphous nature of the TiO_2 NTs in the as-prepared samples was discovered by Raman scattering.

Pishkar et al., (2018) the author has discussed about the highly ordered, densely packed hexagonal TiO_2 nanotubes. that were successfully fabricated using the two-step anodization procedure are detailed in this paper. The Ti substrate had a periodic pattern in the shape of a hexagon after the NTs had been anodized and subsequently peeled off. It investigated how the Ti substrates affected the surface shape and physical characteristics of the produced TiO_2 NTs.

Mansour et al., (2019) in this paper looked at conveys that findings of the mechanical testing show that the 3D printed nanocomposites' modulus, strength, and hardness have all increased due to the addition of multifunctional graphene. When compared to pristine PLA samples, the damping as determined by cycle compression and modal testing was much higher.

C.G.Farling et al., (2021) have comes to the conclusion that screen-printed electrodes (SPEs) covered with silver thin films are an efficient and effective way for creating high-quality electromechanical surface-enhanced Raman spectra (EC- SERS)substrates. PVD-coated SPEs exhibit robust and consistent EC-SERS enrichment for the identification of probe particles, with coefficients of variation for 5- (pyridine-4-yl)- 1,3,4-oxadiazole-2-thiol (PYOT) and 4-aminothiophenol (p-ATP) being 8.2% and 5.5%, respectively. By using PVD, non-uniformity problems with other techniques, like colloidal gel or coated metallic films, are eliminated, leaving the SPEs with uniform nanostructured films.

O.Zakir et al., (2023) investigated the effect of anodic TiO₂ NTs' morphological, fundamental, electrochemical, or photocatalytic properties were examined in relation to the anodizing time. The length of the TiO₂ NT tubes was discovered to be controlled by the anodization period, gradually growing to reach a maximum of 3.32 μ m after 90 min. The anatase and rutile phases in the TiO₂ NTs were mixed, as shown by Raman spectroscopy and XRD. Mott-Schottky analysis showed that the titanium dioxide NTs that are corresponded with an n-type semiconductor. The rate of methylene blue degradation went up from 31.6% through 93.1% when the pH of the solution was raised from 3 to 12.

Denchai et al., (2018) says that in this study the authors come to an idea that a novel spin coating method for the regulated and consistent attaching of AuNPs on TNTs is presented. The spin coating method circumvents the drawbacks of traditional techniques like heat evaporation or conservative heating by enabling fine control over the bonding of AuNPs to the TNTs substrate. Through FESEM microscopy and EDX analysis, the manufactured AuNPs-TNTs demonstrated excellent morphological structures and validated AuNP deposition over the TNTs. These AuNPs-TNTs were also examined for their application in catalysis by reducing p-nitrophenol (p-NP) catalytically. The author emphasizes the value of utilizing supportive materials, such as TNTs, to stop AuNPs from aggregating and to exert greater influence over their attachment.

G.Padmapriya et al., (2016) the author emphasizes that microwave combustion was used to successfully create spinel Ni_xZn_{1-x}Fe₂O₄ tiny particles, or its fundamental, morphological, or opto-magnetic properties were investigated. Through XRD & EDX examination, the synthesis of natural phase spinel ferrites was verified. According to HR-SEM and HR-TEM analysis, the specimens showed decreased aggregation and sphere-like particle morphology. The specimens displayed ferromagnetic while superparamagnetic behavior, and the magnetization values rose as the Ni content did as well. With the addition of Ni, the Ni_xZn_{1-x}Fe₂O₄ the spinel nanocatalysts revealed improved photocatalytic activity and achieved greater success in the photocatalytic destruction of the blue dye methylene.

H.Sogawa et al., (2020) the author aimed to evaluate Utilizing FE-SEM and XPS techniques, the study sought to assess the surface structure the elemental composition of the epidermal layer of spider draglines. The spider dragline was found to have a hierarchical structure, made up of a collection of microfibrils that caused the surface to exhibit weak fibrilar and network-like patterns. The biochemical components of the large ampullate gland or spiders can be linked to the existence of Na, P, or S elements, which were discovered using XPS analysis. The findings of this study offer early understandings of the chemical changes that occur in silk proteins and the development of hierarchy during the dragline spinning process of spiders.

Gokalp et al., (2018) the author's view on the relevance of skid resistance in guaranteeing the safety of roadway pavement is highlighted in the research paper's conclusion. Skid resistance information from multiple pavements with varying aggregates were collected for the study, and performance was evaluated using both a friction tester and a laser profilometer. For several pavement sections, field tests were carried out for more than ten years.

M.Krajewski et al., (2023) the author in this research paper demonstrated the crystal violet (CV) and Congored (CR) dyes were highly effectively adsorbed into iron-iron oxide core-shell nanochains (Fe NCs) from aqueous solutions. The iron core of the Fe NCs was surrounded by a thin Fe₃O₄ shell, displaying a typical core-shell structure. A contact period of 90 minutes with pH 7 or 150 minutes at pH 8 were shown to be the most effective for removing CV and CR, respectively. The Temkin model was discovered to be the most appropriate for both dyes, and the adsorption procedure matched the pseudo-first-order kinetic model. Another option for CR could be the Redlich-Peter son model. After five phases of adsorption/desorption, the Fe NCs showed outstanding adsorption capacity for both dyes, making them interesting materials for both purification of water and environmental applications.

H.Zhang et al., (2022) said that the objective of the study by author was to quantitatively analyze a Ti-3Al-5Mo-4.5 V alloy utilizing a variety of techniques, including X-ray diffraction (XRD). Peaks overlapping or preferred orientation (PO) were problems that were solved using the XRD entire pattern fitting method. The fitting quality was enhanced by carefully scanning the 2 diffraction pattern and altering the PO with the March-Dollase and spherical harmonics functions. The trustworthiness of the XRD method was demonstrated by the final results from XRD, which were in good agreement with those of Electron Back-Scattered Diffraction (EBSD). The Ti-3Al-5Mo-4.5 V alloy's calculated phase fractions were found to be 73.65% phase and 26.35% phase. The study showed that a trustworthy and practical method for quantified phase analysis of alloys made of titanium can be using the XRD entire pattern fitting method.

P.Kumar et al., (2018) the authors emphasize the sensitivity and excellent linearity of the MTT assay, which allows for accurate measurements of cell viability even at high cell densities (up to ~106 cells per well). It also notes that the MTT assay can detect subtle changes in metabolic activity, making it useful for assessing cell stress in response to toxic agents, even in cases where there may not be immediate cell death. Additionally, the paper likely provides details on the standardized protocol for conducting the MTT assay in 96-well plates, which can be easily scaled up or adapted for different plate formats.

C.H.Lee et al., (2012) says that the combination of TiO₂ nanotubes (TNTs) and TiO₂ nanoparticles considerably increased the effectiveness in dye-sensitized solar cells (DSSCs), according to the paper's conclusion. This improvement was made possible by a fabrication procedure that used the alkali hydrothermal conversion for TiO₂ powders and the sol-gel approach for TNTs. To create the DSSCs, the finished elements were then screen printed onto FTO glass and coated. Ruthenium (II) (N-719) was used in the study as an electrolyte (I³/I³⁻) and dye for the DSSCs. The materials' crystalline structure and shape were validated by X-ray diffraction or scanning electron microscopy characterization. A UV-Vis spectrometer was also used to detect absorption spectra.

K.S. Brammer et al., (2012) gives the conclusion of this paper which highlights the significant potential of titanium oxide (TiO₂) surface nanostructures, specifically TiO₂ nanotubes created through electrochemical anodization, for applications in orthopedic implant design. The study emphasizes that these 3D tube-shaped nanostructures have profound effects on osteogenic cells, suggesting enhanced bone regeneration potential. The paper also notes a growing body of evidence supporting the benefits of using TiO₂ nanotubes for orthopedic implant surfaces. Overall, the current trends discussed in the paper indicate a promising future for the clinical use of TiO₂ nanotubes in orthopedic research and applications.

D.Lei et al., (2018) view on this is the digital image correlation (DIC) approach is described by the author in the paper to evaluate the implantation effect of the acetabulum after hip joint prosthetic replacement. By comparing speckle pictures of the undeformed material and its distorted counterpart, the DIC technique calculates the strain field. To confirm the viability of the DIC approach for the socket installation deformation test, three groups of tests were carried out. The deformation caused by plastic can be effectively decreased with the right installation angle, potentially extending the useful life of acetabulum prosthesis.

T.J.Joyce et al., (2021) have said that after hip and knee replacement, shoulder joint replacement is also the next most common joint replacement treatment, and it is expanding more quickly. The opposite shoulder prosthesis, which was primarily created to treat severe rotator cuff injuries but is now utilized for other therapeutic indications as well, is the most widely used and effective shoulder joint replacement. There are a variety of synthetic shoulder joint designs available, including some that use cutting- edge design principles and new biomaterials. The author emphasizes the necessity for standardized testing procedures for shoulder implants as well as the rising popularity of shoulder joint replacement, particularly the usage of reverse shoulder prostheses.

P.Morales-Gil et al., (2023) have using a reline deep eutectic solvent, the author describes the electrical alteration of titanium oxide nanotubes (TiO₂NTs) with gold (also known as Au or silver (AgNPs) nanoparticles. The production of silver as well as gold nanoparticles happens through repeated dimensional nucleation and diffusion-controlled development, according to research on the formation and development mechanisms of metal nanoparticles. XRD, SEM, and XPS analyses of the altered Ti/TiO₂NTs surfaces supported the development of 100 nm-diameter, quasi-spherical silver or gold nanoparticles. The author provides details on the motion or morphology of the silver and gold nanoparticles that were successfully electrodeposited onto TiO₂ nanotubes.

M.Coha et al., (2021) in this study gives the purpose of removing organic contaminants from generated water, advanced methods of oxidation (AOPs) like Fenton-based oxidation, which heterogeneous catalysts, electro-oxidation, photo-assisted procedures, and homogeneous AOPs have undergone substantial research. The Fenton-based processes, heterogeneous photocatalysis, anodic oxidation processes, and ozonation are the AOPs in produced water treatment that have received the most research. To speed up the following biological steps and membrane separation procedures, oxidation can be utilized as an additional polishing stage in effluents that have already been treated or as a primary or secondary treatment phase. To better apply advanced oxidation in generated water treatment and speed up its deployment on a large scale, the author also discusses obstacles, research requirements, and future implications.

M.A Castro-Rojas et al., (2021) say that the conclusion of this paper highlights the potential of nanotechnological approaches, specifically the incorporation of carbon nanotubes, in advancing materials for restorative dentistry. The authors emphasize that both glass ionomer cements and resin-based composites, while promising, have limitations in terms of mechanical properties and susceptibility to fracture. The integration of carbon nanotubes into these materials shows promise in addressing these issues, leading to improved mechanical properties. Additionally, the review highlights the potential applications of carbon nanotubes in drug delivery and tissue

engineering for treating oral infections and lesions. The author concludes by underscoring the status of this state-of-the-art technology and envisions a future where carbon nanotubes play a significant role in advancing dental practice.

Ayşe, Baldemir, Kılıç et al., (2023) gives the conclusion of this paper which highlights the paper which is provided has no reference to "electrochemical anodization-based paper." This work describes the development of an electrochemical paper-based analytical device (ePAD) for the simultaneous measurement of three important biomarkers for cardiovascular disease (CVD): procalcitonin (PCT), troponin I (cTnI), and CRP. The development on electrochemical paper-based immunosensors in biomarker detection is presented in the paper. The developed immunosensors performed well in both clinical and biological diagnostic applications. Paper-based analytical gadget made of origami (oPAD). Paper-based electrochemical analytical device (ePAD).

Magdalena Jarosz et al., (2020) gives the conclusion of this paper which highlights The provided research discusses the effects of anodization settings for surface morphology of nanostructured matter oxide layers and the electrochemical oxidation process of Ti15Mo alloy. This work examined in detail the surface morphology of nanostructured oxide layers that were electrochemically generated on Ti15Mo and were adjusted by varying the anodization settings. The investigation focused on the surface morphology of nanostructured materials oxide layers on Ti15Mo alloy. The oxide layers' pore dimensions and interpore distance were influenced by anodization parameters. Ti15Mo alloy anodization in a single step Analysis techniques include energy dispersive X-ray spectroscopy (EDS), X-ray diffractometry (XRD), and scanning electron microscopy (SEM).

Hong Zhou et al., (2022) gives the conclusion of this paper which highlights the application for titanium oxide in biomedical applications is not the main topic of the study; instead, it covers the use of low oxide titanium coating for medical device applications. This article describes the construction of a solid conical shroud for plasma spraying. The results indicate that the conical shroud attachment was crucial

in safeguarding the titanium particles during the plasma spraying procedure. Titanium particles were shielded during plasma spraying by the shroud attachment. The titanium coating sprayed with plasma exhibited a moderate porosity and an improved microstructure. Veiled plasma spray Analysis using X-ray spectroscopy with energy dispersive and scanning electron microscopy.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The aim is to enhance the biocompatibility of titanium alloys for biomedical applications. TiO_2 nanotubes possess a high surface area and can promote better cell adhesion and proliferation, making them suitable for applications like orthopedic implants and dental implants. The use of in-situ voltage conditions allows for precise control over the morphology, dimensions, and properties of the TiO_2 nanotubes. This ensures that the nanotubes are optimized for specific biomedical applications, such as drug delivery, osseointegration, or antimicrobial properties. The TiO_2 nanotubes must demonstrate stability and durability over an extended period. This is crucial to ensure that they can withstand the harsh physiological environment within the human body without degradation or loss of functionality.

3.2 SCOPE

The scope is to investigate the parameters affecting the growth of TiO_2 nanotubes, such as voltage magnitude, electrolyte composition, temperature, and processing time, to achieve optimal nanotube characteristics suitable for biomedical purposes. Evaluate the mechanical properties and chemical stability of TiO_2 nanotube coatings under physiological conditions, ensuring their long-term performance and durability in biomedical environments. The exploration of in-situ voltage conditions for synthesizing TiO_2 nanotubes on titanium alloy, emphasizing its potential impact on biomedical applications. It delves into the advantages, challenges, and prospects, providing a comprehensive overview for researchers and practitioners in the field.

CHAPTER 4

MATERIALS AND METHODOLOGY

Replacement and reconstruction in a human body enhance the life of the human being for that the selection of materials surface modifications technique and electrolyte preparation and the anode and cathode selection are important in this section we discuss about the material selection (Titanium as anode and Platinum as cathode) ethylene glycol and ammonium fluoride has the electrolyte and electrochemical anodization process is the major surface modification techniques has choosed for the fabrication TiO_2 .(Abdulla.,et.2017).

4.1. MATERIALS

- Titanium (Ti6Al4V)
- Ethylene Glycol
- Ammonium Fluoride
- Platinum wire
- Di - Water

4.1.1. *Titanium dioxide (TiO_2)*

Titanium dioxide (TiO_2) nanotubes are a unique and versatile form of TiO_2 with a tubular nanostructure. They have gained significant attention due to their exceptional properties and wide range of potential applications in various fields TiO_2 nanotubes are characterized by their hollow, tubular structure. (Sogawa.,et.2020) Titanium dioxide (TiO_2) plays a crucial role in the electrochemical anodization process, particularly in the creation of TiO_2 nanotubes. This process involves the controlled electrochemical oxidation of titanium to form a layer of nanotubes on its surface in the electrochemical synthesis of TiO_2 nanotubes, titanium dioxide (TiO_2) plays a central role as the material from which the nanotubes are formed. Titanium dioxide is used in the process with 1mm. It is widely used in coating for dental and bone replacements because of its lightweight and high strength. TiO_2 on SBF increases the biocompatibility of materials

increase the cell growth. Titanium dioxide nanotubes are a remarkable nanomaterial with a wide range of applications in various fields, including biomedicine, environmental remediation, energy, and electronics. Their unique structure and properties make them a subject of intensive research and development for innovative applications in the future. The figure 4.1 shows the Titanium oxide (TiO_2). TiO_2 is valued for its ability to provide whiteness and opacity while also enhancing the durability and weather resistance of products it is incorporated into. Beyond its role in pigmentation, titanium dioxide also serves as a photocatalyst in environmental applications, aiding in the degradation of pollutants in air and water. Its inert nature and biocompatibility make it a common ingredient in pharmaceuticals and food products. However, concerns regarding its potential health and environmental impacts have led to ongoing research and regulatory scrutiny in recent years. (Elahi.,et.2018).

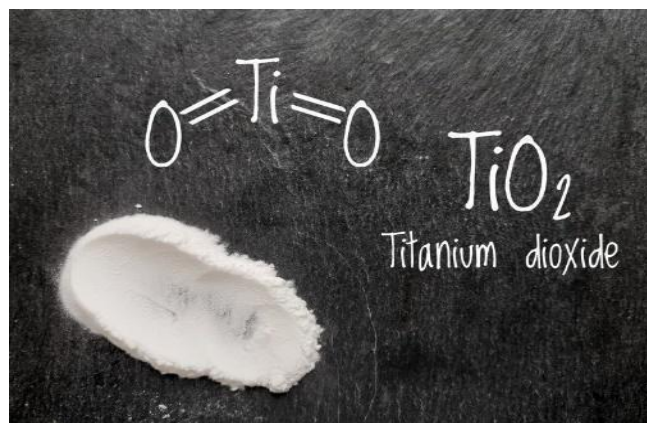


Fig: 4.1: Titanium dioxide (TiO_2)

4.1.2. Ammonium fluoride (NH_4F)

Ammonium fluoride (NH_4F) is a chemical compound composed of ammonium ions (NH_4^+) and fluoride ions (F^-). It is a white crystalline solid with a characteristic ammoniacal odor. Titanium dioxide (TiO_2) nanotubes are a different material altogether. These are nanoscale tubes made primarily of TiO_2 , with a tubular structure. They can be created through processes like electrochemical anodization. (Zhang.,et.2013). The fluoride ions from NH_4F play a critical role in regulating the growth and morphology of the TiO_2 nanotubes. They influence factors like nanotube diameter, length, and ordering. It can play a role in electrochemical synthesis of TiO_2 nanotubes particularly when an anodization process is used. As a part of the electrolyte solution enable the controlled growth of TiO_2 nanotubes through the anodization process. It influences the final characteristics of the nanotubes produced. The structure of Ammonium fluoride (NH_4F) is shown in figure 4.2.(Zahir.,et.2023)

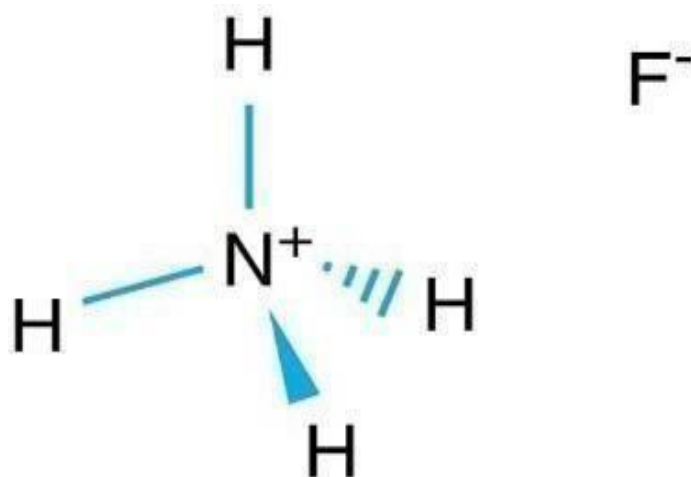


Fig: 4.2: Structure of Ammonium fluoride (NH_4F)

4.1.3. Ethylene glycol

Ethylene glycol, also known as ethane-1,2-diol, is a colorless, odorless, best liquid compound. It has the chemical formula $C_2H_6O_2$ and is classified as a diol or glycol. Ethylene glycol is widely used in various industrial applications, but it's most known as the primary ingredient in antifreeze. (Habiballah.,et.2018) Ethylene glycol (EG) is often used in the synthesis of various nanostructures, including titanium dioxide (TiO_2) nanotubes. TiO_2 nanotubes are a type of nanostructured material with a wide range of applications in areas such as photocatalysis, sensors, energy storage, and biomedical fields. Ethylene glycol can serve as a solvent for the titanium precursor compounds in the synthesis process. Titanium precursors are dissolved in ethylene glycol to form a homogeneous solution. This solution is then used to fabricate TiO_2 nanotubes through techniques like electrochemical anodization. EG can act as a stabilizing agent during the anodization process. It helps maintain a stable electrolyte solution, ensuring a consistent environment for the growth of TiO_2 nanotubes. The concentration of EG in the electrolyte solution, along with other parameters, can influence the morphology, length, diameter, and crystal structure of the TiO_2 nanotubes. Adjusting these parameters allows for precise control over the nanotube properties. The structure of Ethylene glycol is shown in the Fig 4.3 (Kumar.,et.2018)

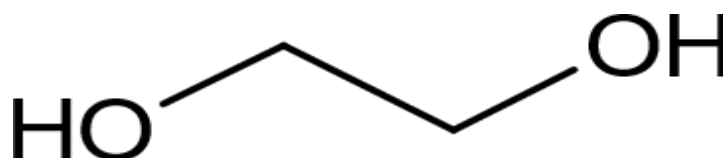


Fig: 4.3: Structure of Ethylene glycol

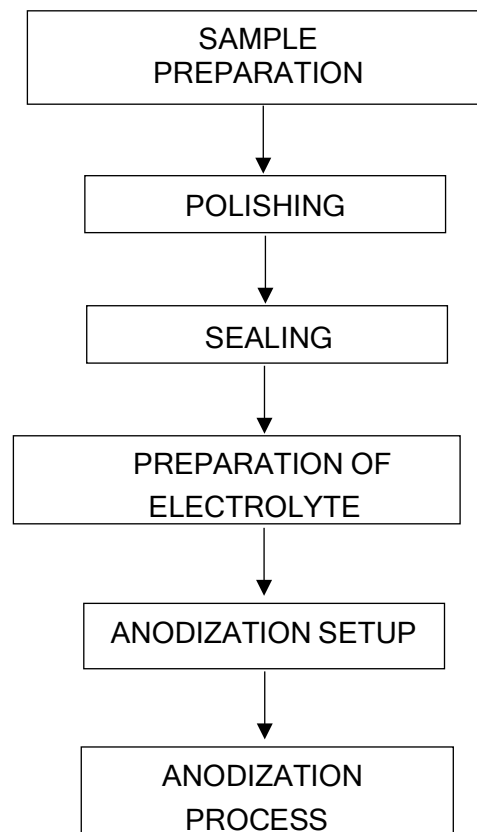
4.1.4. *Platinum wire*

Platinum wire, known for its resilience and conductivity, is utilized across various industries. Its corrosion resistance and high melting point make it indispensable in chemical processes and electrical components. Biomedical applications benefit from platinum's biocompatibility, enabling advancements in surgical implants and therapeutic devices. With its catalytic properties, platinum wire plays a vital role in environmental remediation and industrial processes. Despite its rarity, platinum wire's unique combination of characteristics continues to drive its demand in specialized applications. (Saharudin.,et.2018)



Fig: 4.4: Platinum Wire

4.2. METHODOLOGY OF TiO₂ NANOTUBES



Commercially pure titanium was used to fabricate the TiO_2 nanotubes. To prepare the sample for anodization, titanium plates with a thickness of 1 mm were polished using 350, 650, 850, 1000 and 1500 of microgrid sandpaper until the polish was mirror-like. After polishing, samples were chemically etched using a 1:4:5 ratio of the chemical etchant's water content, nitric acid, and hydrofluoric acid. After cleaning the etched sample with distilled water to get away from any harmful material, it was dried for 60 seconds using hot air. Cellophane tape was used to seal the prepared sample in the unetched region (Lee.,et.2012). Using a furnace, the sample had been annealed to 550 °C for each sample. After preparing the sample the material has been connected to anode terminal and the cathode terminal of Platinum wire has been connected. Both anode and cathode had been immersed in an electrolyte that included NH_4F (0.5 wt%) of ethyleneglycol and no water. The procedure was set up with an input potential of 30 V for 1 hour of anodization. After 5 minutes, 10% of water was injected to continue the experiment until it was finished. In another experiment, 20 vol. % water was added 5 minutes after the experiments started (Coha.,et.2021). To convert phases, the samples were heated to 600°C for one hour.



Fig: 4.5: Anodic TiO_2 Nanotubes Fabrication

4.2.1. Etching Process:

- Chemicals: Distilled water, Nitric acid, and Hydrofluoric acid.
- Using a 50 ml beaker to mix the chemicals in the appropriate ratios.
- Apply with ink filler to the sample.
- Immerse the prepared sample in the electrolyte solution.
- The electrolyte enhance the etching process by conducting.

4.2.2. Input potential for samples:

Sample 1: 25V for first 30 minutes and 30 Volt for next 30 minutes.

Sample 2: 30 Volt for first 30 minutes and 35 Volts for next 30 minutes.

Sample 3: 35 input voltage for first 30 minutes and 40V for next 30 minutes

Sample 4: 40 input voltage for first 30 minutes and 45V for next 30minutes

Table 4.1: Experimental condition of sample preparation

Sl. no.	Sample 1	Sample 2	Sample 3	Sample 4
Input voltage (V)	40	30	20	20
Water vol%	2.5	5	2.5	5

CHAPTER 5

RESULTS AND DISCUSSION

5.1. FABRICATED SAMPLES



Fig: 5.1: Fabrication of TiO_2 under - 40V



Fig: 5.2: Fabrication of TiO_2 under -30V



Fig: 5.3: Fabrication of TiO_2 under – 20V



Fig: 5.4: Fabrication of TiO_2 under – 20V

The experimental procedure involved the fabrication of TiO₂ nanotubes using commercially pure titanium plates, which were polished to a mirror-like finish and chemically etched before anodization. Anodization was carried out in an electrolyte containing NH₄F (0.5 wt%) in ethylene glycol, with different voltages Fig: 5.1: Sample1- 40v, Fig: 5.2: Sample 2- 30v, Fig: 5.3: Sample 3- 20v and Fig 5.3: Sample4- 20v shows voltage which is applied for 1 hour. The introduction of water during anodization, either 10% or 20 vol. %, after 5 minutes of the start, aimed to modulate the growth rate and morphology of the nanotubes. Post-anodization, samples were annealed at 550°C for one hour to convert phases. The results and discussion focus on the effects of applied voltage, water content, and annealing on the characteristics of TiO₂ nanotubes. Variations in voltage likely influence nanotube growth rate and morphology, while water content can affect formation kinetics and crystallinity. Annealing enhances crystallinity and structural stability. Comparative analysis of samples subjected to different voltages provides insights into optimal synthesis conditions for various applications, such as photocatalysis and energy storage. Further characterization is necessary to fully understand and optimize the synthesis process.

Upon analysis of the resulting nanotube structures, it was observed that the morphology and dimensions varied significantly with changes in voltage and the timing of water addition. Higher voltages generally led to larger nanotube diameters and lengths due to increased electric field strength, promoting faster growth. However, excessive voltage could lead to irregularities or damage to the nanotubes.

Furthermore, the timing of water addition appeared to influence the growth kinetics and the homogeneity of the nanotube array. Early introduction of water might alter the electrolyte composition and affect the formation kinetics, potentially resulting in variations in nanotube morphology.

Overall, these experiments has the significance of process parameters suchas voltage profile and electrolyte composition in controlling the morphology andproperties of TiO₂ nanotubes. Further optimization and understanding of these parameters could lead to the development of tailored nanotube structures with enhanced functionalities for various applications, including catalysis, sensing, and energy storage.

5.2. CHARACTERIZATION

5.2.1. XRD Analysis

X-ray diffraction has been used to examine the crystalline phase change. Anodized samples that were immersed in water were heated to 600°C. Sample 1 showed a low degree of crystallinity during phase transformation, with an anodization setting of 30 V and 2.5 vol% of water. Sample 2 provided a phase transition to crystallinity at 550 °C. Sample 3 saw the greatest phase alteration process following the annealing process. Sample 4 showed a change of phase structure. For titanium phases, the diffraction peaks were found in major angles of 38.39, 40.16, 52.92, 62.88, and 70. 63°. The amorphous & rutile phases were found to be at 25.26°, 56.72°, 54.33°, and 27.46° were found. But the bare titanium sample shows that there are no rutile or amorphous phases present. The sample's phases showed that an amorphous peak was identified as (A) and that amorphous phases were fragile and weak after the sample had been anodized and heated to 450°C. The amorphous phases are transformed into crystalline structures by additional heat treatment. The samples showed mixed amorphous or crystalline phases at around 550°C. The first sample showed phase transitions, but the samples with the highest water content & input potential showed high crystallinity phase shifts.

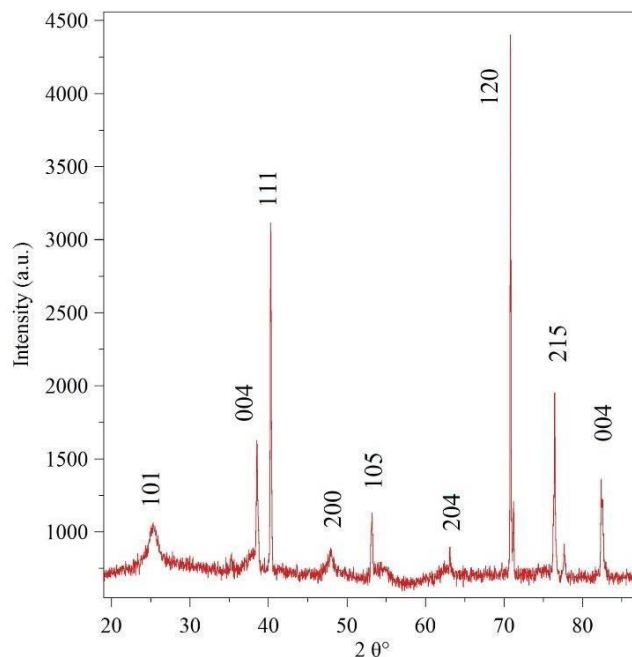


Fig: 5.5: X-ray diffraction of TiO₂ nanotubes

5.2.2. Surface Morphological Analysis

The TiO_2 nanotubes were fabricated using commercially pure titanium is studied with an HR-SEM, a high-resolution scanning electron microscope. When ethylene glycol and fluoride were included as an electrolyte, the nanotubes' produced smooth and cylindrical shape. The HR-SEM study revealed distinct surface morphological outcomes. Fig:5.6 exhibits the nanotubes that were created with less than 10% water content. Because of the amount of water level added to the electrolyte, nanotube surface morphology shows that the rib structure has formed in the first section. The sample that was created with 20% water content shows the same pattern. It might be possible to create rib structures covered in nanotubes. Figure:5.7 shows the tube top layers displaying the self-organized nanotubes. The growth of the tubes has fluctuated. Figures:5.8 and 5.9 have been used to identify the ups and downs layers. The change in even growth with the presence of water in it indicates that the water content has affected the way nanotubes form. Moreover, a few other conditions may potentially influence the creation of TiO_2 nanotubes. The formation of self-organized nanotubes on the surface of titanium can be facilitated by a variety of combinations of factors, including the electrolyte's temperature, anodization time, ammonium fluoride, and water content percentage.

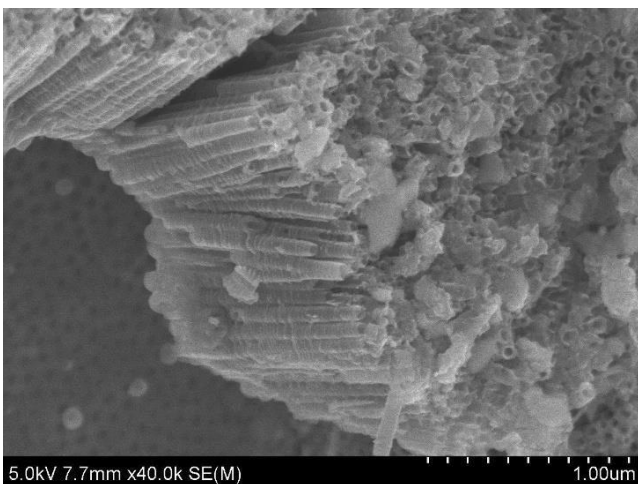


Fig:5.6: Cross sectional view of TiO_2 nanotubes under 30V

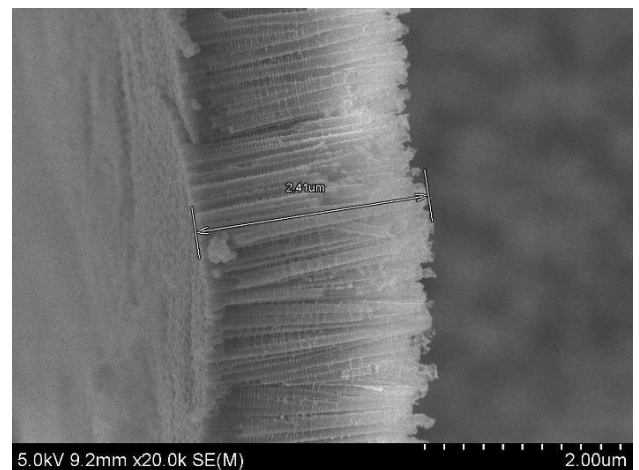


Fig:5.7: Cross sectional view of TiO_2 nanotubes under 40V

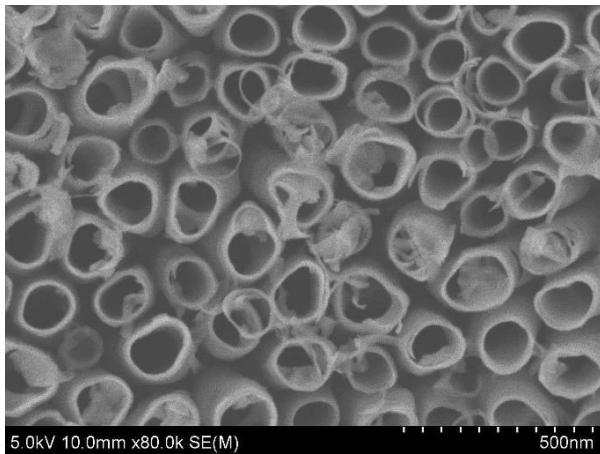


Fig:5.8: Top view of TiO_2 nanotubes under 30V

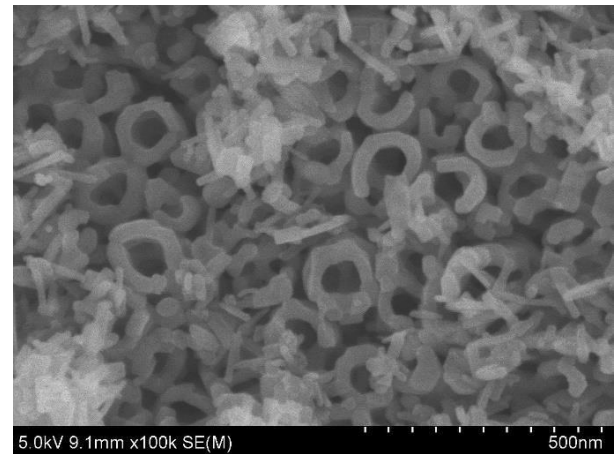


Fig:5.9: Top view of TiO_2 nanotubes under 40V

5.2.3. Corrosion Analysis

This section analyzed the TiO_2 nanotubes' corrosion behavior after they formed using the water addition method. SBF medium was used to investigate and explain the behavior. For this investigation, a new SBF was created each time. The platinum wire was attached to the negative terminal, anodized sample were connected to the positive terminal, and calomel was utilized as the reference electrode. It shows the use of computerized potentiostats to characterize the anodized rapid input current zone in the electrochemical polarization process. The exposed area of the working sample measured 1.0 cm^2 . The area of balance was glued shut with adhesive tape. The corrosion analysis was characterized using the SBF solution. At a scan rate of 1 mV/s , the potentiodynamic polarization was started between 1500 and 2000 mV . Prior to the analysis of polarization, to improve the corrosion results, the sample was left in the SBF solution for 30 minutes. A new solution was used for each experiment, and the temperature of the solution was continuously observed. The explanations for the change in crystallinity following heat treatment were the low corrosion rate and the shift in the high corrosion rate. After heat treatment at 550°C , the peak intensity increased in relation to the crystallinity. When assessing the greatest length of a nanotubes sample for nano hardness, the deformation of nanoindentation may improve the sample's resistance to corrosion. Using a gamry device, TiO_2 nanotubes were investigated to quantify their electrochemical impedance and their impedance spectrum when compared with an identical electrical circuit model. Both structural flaws and the insulating property are assessed in this analysis. Nyquist plot result that.

the EIS study produced. The graphical representation for samples 1, 2, 3 shows a semi-circle at the front of the semicircles form represents the material's resistivity within the SBF solution. Fig:5.10 to 5.12 shows Surface wetting behaviour of TiO_2 .

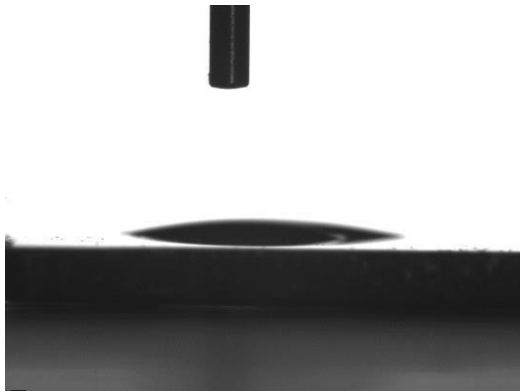


Fig:5.10: Corrosion analysis under 30V

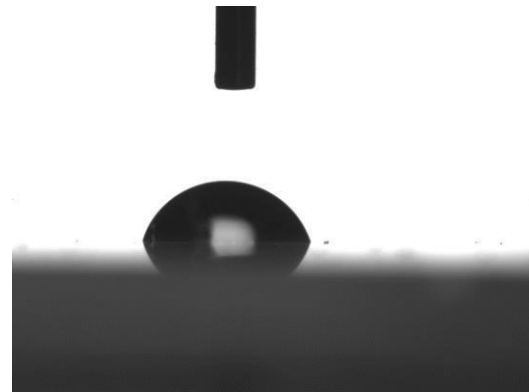


Fig:5.11: Corrosion analysis under 35V

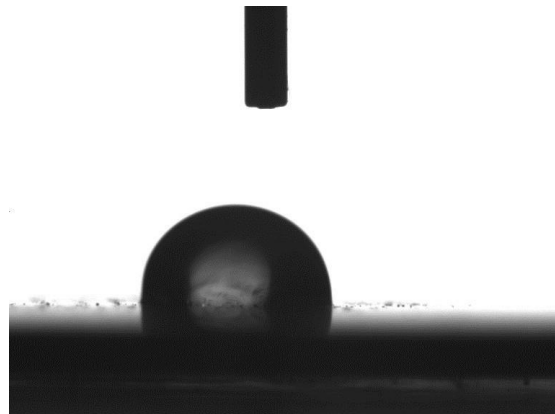


Fig:5.12: Corrosion analysis under 40V

5.3. DISCUSSION

The comprehensive study of anodized TiO₂ nanotubes using high-resolution scanning electron microscopy (HRSEM), X-ray diffraction (XRD), and corrosion analysis provides valuable insights into their structural, morphological, and corrosion properties. The HRSEM analysis revealed the formation of distinct nanotube types, including top-open and bottom-closed cylindrical structures, which are essential for various applications. XRD analysis elucidated the crystalline phase transformation, shedding light on the nanotubes' structural evolution during anodization. The corrosion studies conducted using the Gamry instrument 1010 interface, coupled with immersion in simulated body fluid (SBF) solution, allowed for the assessment of the nanotubes' corrosion resistance and kinetic behavior. The rapid preparation of the SBF solution within 24 hours ensured timely experimentation. This comprehensive approach contributes to a deeper understanding of TiO₂ nanotubes' properties, guiding their potential applications in areas such as biomedical implants, sensors, and corrosion-resistant coatings. Further investigations could focus on optimizing fabrication techniques and exploring novel applications based on the observed structural and corrosion characteristics.

CHAPTER 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

The utilization of titanium and its alloys for biomedical applications is increasingly favored, yet the formation of a passive layer over these materials remains a challenge. In its plain form, titanium lacks the necessary properties for effective bio implantation due to poor adhesion of biological elements such as blood cells, tissues, and bones. However, surface modification techniques, particularly through electrochemical methods, offer a simple and cost-effective solution. By adding water to the ethylene-based electrolyte, the oxidation process is enhanced, leading to the elongation of titanium nanotubes. Surface morphological analysis confirms the increased length of these nanotubes, while nanoindentation analysis demonstrates a corresponding rise in hardness levels. Moreover, corrosion analysis reveals that the elongated nanotubes contribute to improved corrosion resistance. Finally, cell viability assays using NIH-3T3 cells conducted with inverted microscopy analyzer underscore the biocompatibility and potential suitability of the surface-modified titanium for biomedical applications.

6.2 CONCLUSION

In conclusion, the surface modification of titanium through electrochemical methods, particularly with the addition of water to the electrolyte, presents a promising avenue for enhancing its properties for bio implantation, offering improved adhesion, hardness, corrosion resistance, and biocompatibility. Under 10 and 20 vol.% of water content injected five minutes after the experiment started, TiO_2 nanotubes were successfully produced. A high-resolution scanning electron microscope was used to identify and create two distinct surfaces of the rib structures. The mechanical strength of the nanotubes has been greatly enhanced by the ribs that are present throughout. Corrosion analysis revealed that oxide film deposition on the nanotubes had promoted corrosion resistance. The sample's resistivity on the electrolyte medium may have grown because of the anodization process's addition of water, as indicated by the Nyquist plot's lengthy semicircle. Furthermore, following a 72-hour incubation period, the titanium material's cell survival has been strongly stimulated.

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