

STUDY ON CORROSION BEHAVIOUR OF A NANO COATING ON AZ91D MAGNESIUM ALLOY FOR ORTHOPEDIC APPLICATIONS

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Engineering degree in mechanical engineering

By

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SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC

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APRIL 2021



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Harish Prasanna (37150073)** and **Ravi Anirudhya (37150152)**, who carried out the project entitled “**Study on corrosion behavior of nano coating on AZ91D Magnesium alloy for orthopedic applications**” under my supervision from December 2020 to March 2021.

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We **HARISH PRASANNA (37150073)** and **RAVI ANIRUDHYA (37150152)** declare that the Project Report entitled “**Study on corrosion behaviour of a nano coating on AZ91D magnesium alloy for orthopedic applications**” done by us under the guidance of **Dr. M. Anish, M.E., Ph.D.**, is submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering degree in Mechanical engineering.

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ACKNOWLEDGEMENT

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

I would like to express my sincere and deep sense of gratitude to my Project Guide **Dr. M. Anish., M.E., Ph.D** for his valuable guidance, suggestions and constant encouragement 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 Mechanical engineering who were helpful in many ways for the completion of the project.

ABSTRACT

Magnesium (Mg) and its alloys have been extensively investigated as biodegradable implant materials for orthopedic applications. However, due to the low corrosion resistance of magnesium alloys, a wide application of these materials is limited. The present study focuses on the fabrication of a nano coating film on an AZ91D magnesium alloy via Electron beam physical vapor deposition (EB-PVD). The phase composition, microstructure, and surface morphology of the coatings will be investigated using Scanning electron microscopy (SEM), Energy-Dispersive X-ray (EDX) and Atomic force microscopy (AFM). Besides, tests in Artificial blood plasma.

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CHAPTER 1

INTRODUCTION

1.1. GENERAL

Magnesium (Mg) based biodegradable materials are new age strong install materials that are required to possess same mechanical properties as that of bone. Mg composites are considered as promising substitutes to interminable inserts in light of their biodegradability in the physiological environment. In any case quick disintegration rate is one of the huge goals of using Mg mixes in clinical applications notwithstanding their surprising biocompatibility. Approaches to manage out the obstructions consolidate the decision of agreeable alloying segments, suitable surface treatment, surface changes with covering to control the defilement rate. The review base on current advances on surface planning of Mg based biomaterials for biomedical applications.

1.2. BIOMEDICAL APPLICATION

Biomedical applications are by and large novel use for warm sprinkle cycles and coatings. Metallic coatings, particularly titanium and its blends, are showered onto metal prostheses in vacuum cycles to give nonoxidized titanium coatings. The coatings are bitoiner surfaces with satisfactory obnoxiousness and porosity allowing mechanical interlocking of the bone tissue. Bioactive Hap or fluorapatite coatings with bone-type phosphate structures have been extraordinary likeness and execution. These materials license improvement of bone tissue on a shallow level on the prosthesis. Various human additional parts for example hip, knee and tooth embeds are today covered by vacuum and climatic plasma measures.

1.3. IMPLANTS

A muscular embed is a clinical gadget fabricated to supplant a missing joint or bone or to help a harmed bone. The clinical embed is chiefly created utilizing tempered steel and titanium combinations for strength and the plastic covering that is done on its goes about as a counterfeit ligament. Interior obsession is an activity in muscular health that includes the careful execution of inserts to fix a bone. During the medical

procedure of broken bones through inner obsession the bone parts are initial diminished into their ordinary arrangement then they are held along with the assistance of inward fixators like plates, screws, nails, pins, and wires.

1.4. MAGNESIUM ALLOY

Magnesium amalgams are magnesium alloys containing other metals such as aluminium, zinc, manganese, silicon, copper, rare earths, and zirconium. The lightest underlying metal is magnesium. Magnesium composites also have a hexagonal cross section, which affects the important properties of these materials. Plastic disfigurement of hexagonal cross sections is more muddled than in cubic latticed metals such as aluminium, copper, and steel. Magnesium amalgams are commonly used as cast combinations in this way, but since 2003, there has been a greater emphasis on fashioned compounds. Fig 1.1 shows the Magnesium alloy.



Fig 1.1 Magnesium Alloy

Magnesium composites cover a wide cluster of uses, from auto and aviation applications to electronic and biomedical employments.

Car, aviation, mechanical, and business applications are instances of primary

applications. The upside of magnesium compound used to be utilized in such applications is their light weight, high solidarity to-weight proportion, high solidness to-weight proportion, castability, machinability, and incredible damping.

- 1) Automotive: support sections for brakes and grasp, lodging for transmission.
- 2) Aerospace: landing wheels, helicopter rotor fittings, gearbox lodgings.
- 3) Industrial: fast working apparatus, like material machines.
- 4) Commercial: baggage, hand apparatuses, PC lodgings, stepping stool.

Versatile clinical gear and wheelchairs that require light material utilize magnesium combinations. Additionally, cardiovascular stents and muscular gadgets are expected uses of some magnesium compounds because of magnesium's biocompatibility and bioabsorbability.

Magnesium composites will in general have a high solidarity to-weight proportion, excellent machinability and minimal effort. They have a low explicit gravity of 1.74g/cm and a responsively low youthful's modulus contrasted with other basic composites, for example, aluminum or steel amalgams.

1.5. PROBLEM

By utilizing Magnesium amalgam we have issue of brisk corrosion rate.

CHAPTER 2

LITERATURE SURVEY

T. M. Yue et al. (2011) while trying to expand the wear and erosion properties of AZ91D magnesium composite, blend of Zr coatings were applied on the combination by laser framing. In his work two layers of coatings were applied. It comprises of an undercoating of Zr + Mg powder having a 5:1 mass% having the Zr powder as top layer. The covering was exposed to wear test under dry sliding wear condition and it was seen that the wear obstruction of the covering was a lot of higher than that of the untreated AZ91D amalgam. Likewise it was seen that the untreated combination will in general have a weight reduction which is multiple times higher than that of covered amalgam. Another significant trademark which is noticed is that the covered amalgam shows better erosion opposition when coordinated with that of uncoated AZ91D composite. In wording current thickness applied for the consumption test, it was tracked down that the erosion current thickness estimated for the covered material was multiple times not exactly that of uncoated material.

YunchangXin et al. (2009) contemplated the erosion obstruction of ZrN/Zr bi-layered covering on AZ91D magnesium amalgam. He utilized cathodic plasma circular segment affidavit method and covering thickness was kept up at about 1.5 μ m. Primer investigations of the covering shows that the covering is fundamentally established of ZrN which is affirmed by utilizing XRD and EDS examines. While examining the erosion potential it has been seen that the covered alloy"s consumption potential is significantly more sure however the erosion current thickness of the covered amalgam is multiple times not exactly that of the uncoated combination. The yield from the EIS analyzer likewise uncovers some fascinating outcome that the erosion obstruction of the covered amalgam is far superior to that of the uncoated composite.

Hui-Long Wang et al. (2013) effectively arranged Ni-P/SiC angle coatings which is tried on AZ91D magnesium composite. In his exploration he utilized acidic ternary complexing specialists which depend on hypophosphite-diminished electro-less nickel shower and controlling dropping rate of concentrated plating shower containing SiC particles. To accomplish the most elevated erosion opposition, the SiC fixation in the

shower was kept up at 4g L-1 and the covering of Ni-P/SiC covering was kept up with 3.5% by weight of NaCl arrangement. At this level, the covering displayed the best outcomes.

AkashSingha, et al. (2012) contemplated the tribological conduct of titanium adjusted austentic treated steel (composite D-9) substrates by saving ZrN coatings. For applying these coatings the beat magnetron faltering method was utilized and the coatings were saved in the temperature scope of 300 - 873 K. From the result of the analyses it was derived that the testimony temperature and the sliding counter bodies profoundly affected the tribological properties. At higher testimony temperature and with steel ball, coefficient of contact was seen to be lower. At the point when the ZrN coatings were examined with steel ball, the coefficient of grating will in general be low which credited to the advancement of the tribo-layer on the covering.

Jerzy Smolik et al. (2012) built up a composite covering which was "intermetallic layer MgAl – PVD covering" saved on AZ91D Mg compound. This composite covering is explicitly intended for improving the counter erosion and hostile to wear properties. This kind of covering is acknowledged by the cross breed surface treatment innovation which essentially made out of dissemination treatment in Al powder prevailing with electron shaft testimony procedure. The test results showed that this mix of half breed surface treatment innovation, dispersion treatment in Al powder and electron pillar affidavit, which was exceptionally planned by the creators serves to extensively upgrade the erosion and tribological opposition of AZ91D magnesium composite.

Hai-Long Yao, Zhi-Hai Yi (2019) To improve the erosion obstruction and bioactivity of biodegradable Mg compound substrate, novel Zn-HA/Zn twofold layer coatings with various HA/Zn proportions were in weight were stored on AZ91D substrates by cool showering. Electrochemical consumption practices of both Zn-HA/Zn twofold layer coatings were explored in Hank's reenacted body liquid utilizing potentiodynamic polarization and electrochemical impedance spectroscopy (EIS).

Lokesh Choudhary, Jeremy Szmerling (2011) Mechanical honesty of biodegradable magnesium composites embed stays an issue in bio-compound and destructive climate of human body as a result of their unsuitably high corrosion rate. Thusly, comprehension of concurrent impact of pressure and destructive climate, stress destructive breaking (SCC) of these amalgams in physiological climate is fundamental before their genuine use.

Aydin Tahmasebifar, Said Murat Kayhan (2016) In this study, the impact of assembling conditions on physical, mechanical and electrochemical properties of Mg compound circles were explored. The fundamental inspiration of this investigation was to accomplish the assembling of permeable and miniature surface finished Mg-based biomedical inserts with great mechanical and electrochemical properties.

CHAPTER 3

AIM AND SCOPE

3.1. AIM

The aim of this project is

- To Coat Magnesium alloy with Zirconium and add a composite material.
- To do test/analysis to find, corrosion rate of coated magnesium alloy.

The main aim of this project is To Study the Corrosion behaviour of a nano coating on AZ91D Magnesium alloy for Orthopedic Applications.

3.2. SCOPE OF PRESENT INVESTIGATION

In orthopedic applications Magnesium is being used to repair human bone fracture, which has high corrosion rate. In order to reduce the corrosion rate we have come up with a solution by coating the magnesium with zirconium and add a composite material to it.

3.3. METHODOLOGY FLOWCHART

The following flowchart is a brief methodology of our project.

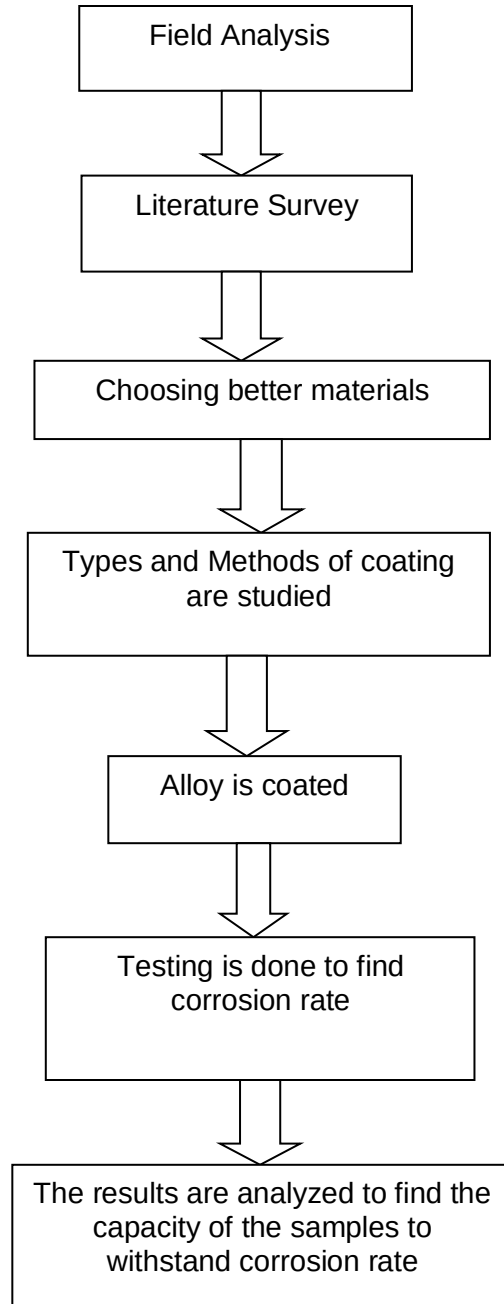


Fig 3.1 Methodology Flowchart

CHAPTER 4

EXPERIMENTAL OR MATERIALS AND METHODS

4.1. MATERIALS

AZ91D Magnesium alloy, Zirconium coating, and Artificial Blood Plasma were used in our research.

4.1.1. AZ91D MAGNESIUM ALLOY

The most commonly used magnesium die cast alloy, Alloy AZ91D, has a perfect combination of mechanical properties, corrosion resistance, and castability. Corrosion resistance is accomplished by imposing extremely stringent limits on three metallic impurities: iron, copper, and nickel. Tab 4.1 shows the property of AZ91D magnesium alloy.

Applications

Several parts are made of magnesium AZ91D alloy cast. The following are a few of them:

- Protective clothing
- Residences
- Tools that can be carried around
- Sports equipment
- Computer components
- Cellphones and mobile phones
- Appliances for the house
- Automobile elements

Table 4.1 Properties of AZ91D Magnesium alloy

PROPERTY	UNIT (METRIC)	VALUE
Density	g/cm	1.81
Melting Range	°C	421
Specific Heat	J/Kg.°C	1.047
Thermal Conductivity	W/m.K	72.7
Thermal expansion coefficient	/°C	26
Elongation	%	3-7
Tensile Strength	MPa	250
Elastic Modulus	GPa	45
Hardness	N/mm	63

4.1.2. ZIRCONIUM

Zirconium is a radiant silver-dim metal that is solid, moldable, and malleable. Its chemical and physical properties are identical to those of titanium. Zirconium is incredibly resistant to heat and corrosion. Zirconium is lighter than steel and has a copper-like hardness. When the metal is finely partitioned, it can light up in air almost instantly, particularly at high temperatures. Zirconium powder is a dark material that poses a fire hazard. In acids and salts, zirconium does not disintegrate.

Applications

Zirconium is utilized in compounds, for example, zircaloy, which is utilized in atomic applications since it doesn't promptly ingest neutrons. Likewise utilized in exhaust systems, percussion covers and heater blocks. Baddeleyite and polluted zirconium (zirconia) are utilized in lab pots.

The significant end employments of zircon (ZrSiO_4) are refractories, ceramic opacification and foundry sands. Zircon is additionally advertised as a characteristic

gemstone utilized in adornments. The metal additionally has numerous different uses, among them in photographic flashbulbs and careful instruments, to make the glass for TV, in the expulsion of lingering gases from electronic vacuum tubes, and as a solidifying specialist in compounds, particularly steel. The paper and bundling businesses are finding that zirconium intensifies make great surface coatings since they have phenomenal water obstruction and strength.

Health effects of Zirconium

Zirconium and its salts for the most part have low fundamental harmfulness. The assessed dietary admission is around 50 microg. Most goes through the gut without being adsorbed, and that which is adsorbed will in general amass marginally more in the skeleton than in tissue.

Zirconium 95 is one of the radionuclides associated with climatic testing of atomic weapons. It is among the extensive radionuclides that have delivered and will keep on creating expanded malignancies hazard for quite a long time to come. Fig 4.1 shows the Zirconium.



Fig 4.1 Zirconium

4.2. SAMPLE PREPARATION

4.2.1. WIRE CUTTING

A wire shaper (in any case called a wire EDM) is based off a comparable norm as a regular EDM. It uses a cathode, which is electrically charged, so when it moves toward a conductive material, the force needs to jump across to it. This causes a radiance, which lands on the conductive material and disintegrates it. The single differentiation is that because of wire cutting, the anode is a piece of copper wire.

Particularly like EDM, wire cutting is an extraordinarily definite cycle. Regardless, it is a drowsy connection and ends up being drastically more delayed as the material being cut extensions in thickness. To get something wire cut, you'll need a solid CAD model of the part, with the objective that the machine has a model to create a cut-route around. It has a sublime surface fulfillment and can make diverse or delicate models as well.

Wire EDM was first modernly made during the 1970s and has gotten logically renowned on account of it's reliable precision and low machine wear. It has that low machine wear and appropriately less machine upkeep as it's a non-contact technique. At the point when the anode is satisfactorily close, it starts and breaks up the metal. It's generally proper to high opposition parts, and incredibly hard plans, or other limit materials like carbide or titanium. Fig 4.2 shows the wire cutting machine.



Fig 4.2 Wire cutting machine

4.2.2. MIRROR POLISHING

Cleaning and cleaning are finishing cycles for smoothing a workpiece's surface using a grinding and a work wheel or a calfskin stop. Technically cleaning insinuates measures that usage an unpleasant that is adhered to the work wheel, while cleaning uses a free grinding applied to the work wheel. Cleaning is a more strong connection while cleaning is less coldblooded, which prompts a smoother, more splendid consummation. A regular disarray is that a cleaned surface has a mirror awe inspiring fruition, in any case most mirror splendid fulfillments are truly cleaned.

Cleaning is regularly used to improve the presence of a thing, hinder spoiling of instruments, kill oxidation, make a clever surface, or prevent utilization in pipes. In metallography and metallurgy, cleaning is used to make a level, deformation free surface for evaluation of a metal's microstructure under an amplifying focal point. Silicon-based cleaning pads or a gem grade plan can be used in the cleaning cycle. Cleaning tempered steel can moreover construct its sterile benefits. Fig 4.3 shows the double disk machine and fig 4.4 shows magnesium after polished.



Fig 4.3 Double disk polish machine



Fig 4.4 Magnesium alloy after polished

4.3. CORROSION

Consumption is a characteristic cycle. It is the steady obliteration of materials (generally metals/composites) by substance or potentially electrochemical response with their current circumstance. It is brought about by the kind of metal and the ecological conditions, especially gasses that are in contact with the metal, decide the structure and pace of decay. Erosion can likewise happen in materials other than metals, like ceramics or polymers. Erosion debases the valuable properties of materials and constructions including strength, appearance and porousness to fluids and gases. Numerous primary compounds erode simply from openness to dampness in air, yet the interaction can be firmly influenced by openness to specific substances. Erosion can be focused locally to shape a pit or break, or it can stretch out across a wide region pretty much consistently eroding the surface. It prompts the disappointment of the metal/amalgam.

4.3.1. ELECTROCHEMICAL CORROSION

Electrochemical corrosion occurs when a metal is electrochemically oxidised in reaction to an oxidant such as oxygen or sulphates. Rusting, or the formation of iron oxides, is a good example of electrochemical erosion. This form of damage often creates oxides or salts of the first metal, resulting in the metal's debasement. Finally, the metal deteriorates to the point of disappointment. In any case, it is considered a safe form of consumption because it is predictable, rational, and often avoidable.

Electrochemical corrosion occurs in the channel and blower of an aeroplane motor when contaminants or foreign substances in the air react with these components since they are the air admitting units.

4.3.2. HOT CORROSION

Hot consumption is a type of sped up oxidation that is created by the substance response between a metal or composite and liquid salts kept on its surface. The mix of high temperature with a metal that contains impurities like sodium, sulfur and vanadium over it brings about the wonder of hot consumption. Hot erosion involves a perplexing arrangement of compound responses, making consumption rates extremely hard to anticipate and it is at eccentrically fast rate. Sodium sulfate is

generally the essential segment of the store and corrosion turns out to be more serious with expanding focus levels of toxins like sodium, potassium, vanadium, sulfur, chlorine, fluorine, and lead. The rate and instrument of hot erosion assault is affected by temperature. The powerlessness to either absolutely forestall or if nothing else identify it in beginning phases prompts the disappointment of the material. In Aircraft motor, Hot Corrosion happens where there is high temperature. It for the most part happens in ignition chamber, turbine cutting edges and spout since it manages high temperature ($>500^{\circ}\text{C}$).

According to the temperature that the hot corrosion occurs, it is classified into two types. They are,

- Type I HTHC (High Temperature Hot Corrosion) – It occurs between Temperature $850 - 1000^{\circ}\text{C}$.
- Type II LTHC (Low Temperature Hot Corrosion) – It occurs between Temperature $550 - 800^{\circ}\text{C}$.

4.3.3. PREVENTION OF CORROSION

1) Alloy Selection

The block of super blends against the hot erosion depends on to the substance formation of the combination. Chromium is the best alloying part for improving check against hot consumption. This is a result of the reaction of Cr_2O_3 to shape a stable Cr_2O_4 solute. Appropriately hindering erosion. Cobalt and Nickel based super blends are in like manner more impenetrable to HTHM. Cerium, Lanthanum, Zirconium, Yttrium and Scandium have colossal resistance against hot utilization. Titanium and Niobium similarly have incredible resistance against Hot Corrosion.

2) Protective Coating

This is a supported procedure, regardless, when hot utilization safe composites like Aluminum, Copper, Zinc are used. They give more security from the mixtures. They structure a layer over the composite which gives guarantees against damaging attack. They moreover go probably as an encasing. Today, there are different varieties of high temperature is use.

3) Washing of compounds

A demonstrated way to deal with decrease hot erosion assault is to washing the combinations with plain water which manages high temperature. This takes into account dissolving and diverting of destructive impurities, along these lines forestalling hot erosion.

4.4. METHODS OF COATING

Thermal barrier coating is done for our magnesium alloy because TBC provide not only corrosive resistance, it also provide thermal resistance to the alloy due to its Ni-Cr bond coat. For thermal barrier coating there are three commonly used methods. They are

- Electron Beam Physical Vapor Deposition (EB-PVD)
- Electro Plasma Oxidation
- Plasma Spray Coating

4.4.1. ELECTRON BEAM PHYSICAL VAPOR DEPOSITION (EB-PVD)

EB-PVD, or electron-beam physical vapour deposition, is a form of physical vapour deposition in which a target is bombarded with an electron beam emitted by a charged tungsten filament in a high-vacuum setting. The electron beam causes the target to change from a solid to a gaseous state. These atoms solidify, leaving a thin layer of anode material on everything in the vacuum chamber (within line of sight). It's used to make things that need thin films for mechanical, optical, chemical, or electronic functions. Fig 4.5 shows the Electron beam physical vapor deposition (EB-PVD) machine.



Fig 4.5 Electron beam physical vapor deposition (EB-PVD)

4.4.2. ELECTROLYTIC PLASMA OXIDATION

Electrolytic plasma oxidation (EPO), likewise known as plasma electrolytic oxidation (PEO) or is an electrochemical surface treatment measure for generating oxide coatings on metals. It is like anodizing, yet it utilizes higher possibilities, so that discharges occur and the subsequent plasma alters the construction of the oxide layer. The covering is a compound transformation of the substrate metal into its oxide, and becomes both inwards and outwards from the first metal surface. Since it becomes internal into the substrate, it has fantastic grip to the substrate metal. This interaction can be utilized to develop thick (many micrometers), generally translucent, oxide coatings on metals such as aluminium, magnesium and titanium. They can introduce high hardness and a ceaseless hindrance, these coatings can offer assurance against wear, erosion or warmth just as electrical protection. A schematic representation of electro plasma oxidation coating is seen in fig. 4.6

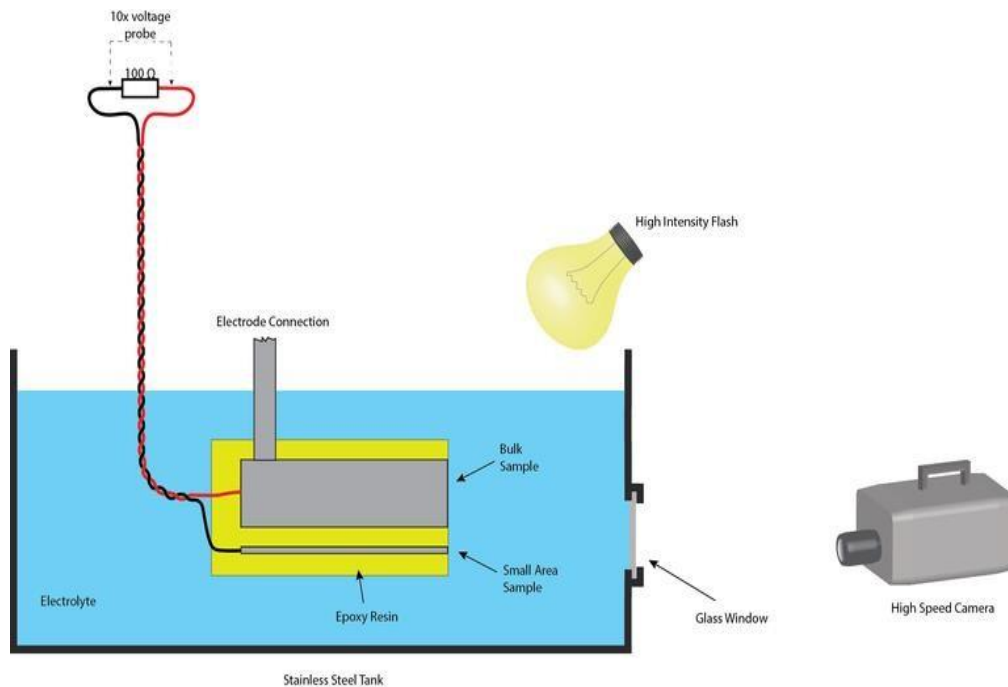


Fig 4.6 Electrolytic Plasma Oxidation

4.4.3. PLASMA SPRAY COATING

The Plasma Spray Process is essentially the showering of liquid or warmth mollified material onto a surface to give a covering. Material as powder is infused into a high temperature plasma fire, where it is quickly warmed and sped up to a high speed. The hot material effects on the substrate surface and quickly cools framing a covering. This plasma shower measure completed accurately is known as a "chilly interaction" (comparative with the substrate material being covered) as the substrate temperature can be kept low during handling staying away from harm, metallurgical changes and mutilation to the substrate material.

The plasma splash weapon contains a copper anode and tungsten cathode, the two of which are water cooled. Plasma gas (argon, nitrogen, hydrogen, helium) streams around the cathode and through the anode which is formed as a choking spout. The plasma is started by a high voltage release which causes limited ionization and a conductive way for a DC circular segment to shape among cathode and anode.

The opposition warming from the curve makes the gas arrive at outrageous temperatures, separate and ionize to shape a plasma. The plasma leaves the anode

spout as a free or nonpartisan plasma fire (plasma which doesn't convey electric flow) which is very extraordinary to the Plasma Transferred Arc covering measure where the curve stretches out to the surface to be covered. At the point when the plasma is settled prepared for splashing the electric curve stretches out down the spout, rather than shorting out to the closest edge of the anode spout. This extending of the bend is because of a warm squeeze impact. Cold gas around the outside of the water cooled anode spout being electrically non-conductive tightens the plasma bend, raising its temperature and speed. Powder is taken care of into the plasma fire most usually through an outside powder port mounted close to the anode spout exit. The powder is so quickly warmed and sped up that splash distances can be in the request for 25 to 150 mm. Fig 4.7 shows the process of plasma spray coating.

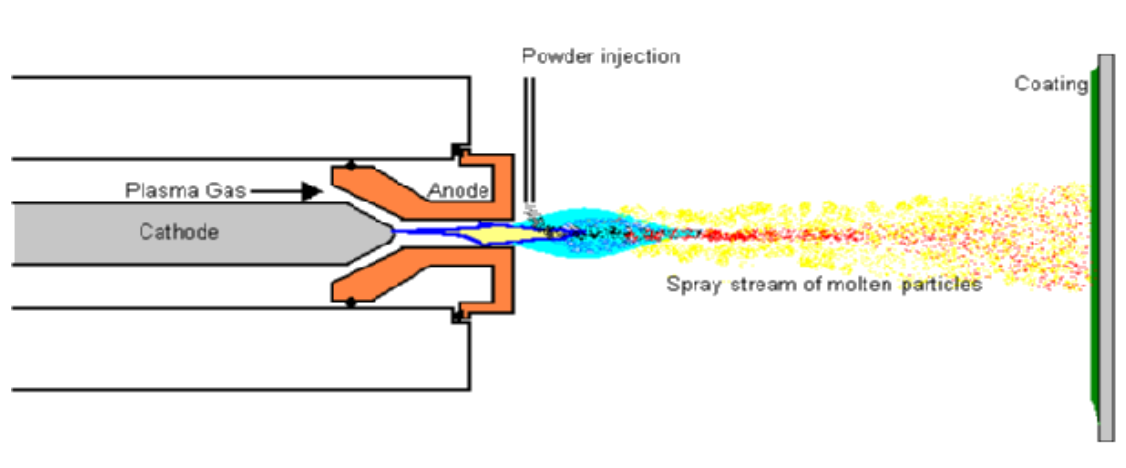


Fig 4.7 Process of Plasma Spray Coating

Advantages of Plasma Spray Coating are,

- Unlike combustion methods, it can spray products with exceptionally high melting points, such as tungsten and zirconia. Plasma-sprayed coatings are typically denser, heavier, and cleaner than those generated by other thermal spray methods.
- Plasma spray coatings are perhaps the most flexible of all thermal spray coatings and applications.
- As compared to other deposition processes, greater coating thickness (300 m) can be achieved.

4.5. EXPERIMENTAL TESTS

To find out the Corrosion Property of our coated Magnesium alloy, we did four analysis/tests. The tests are,

- SEM Analysis
- EDS Analysis
- AFM Analysis
- Corrosion Test

4.5.1. SEM ANALYSIS

SEM represents Scanning Electron Microscopy. It is a sort of electron magnifying lens that produces pictures of an example by examining the surface with an engaged light emission. The electrons cooperate with iotas in the example, creating different signs that contain data about the surface geology and structure of the example. The electron pillar is checked in a raster examine design, and the situation of the shaft is joined with the force of the recognized sign to create a picture. In the most widely recognized SEM mode, optional electrons radiated by iotas energized by the electron pillar are distinguished utilizing an Everhart-Thornley finder. The quantity of optional electrons that can be distinguished, and along these lines the sign power, depends, in addition to other things, on example geography. SEM can accomplish goal better than 1 nanometer. Fig 4.8 shows the Scanning electron microscopy (SEM) machine.

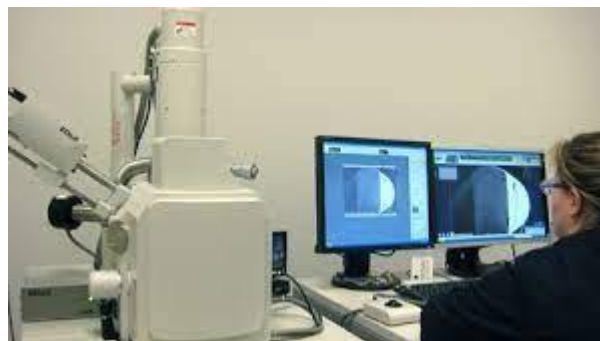


Fig 4.8 Scanning electron microscopy (SEM)

4.5.2. EDS Analysis

EDS stands for Energy-Dispersive X-Ray Spectroscopy.

The age of the X-beams in a SEM is a two-venture measure. In the initial step, the electron bar hits the example and moves part of its energy to the molecules of the example. This energy can be utilized by the electrons of the molecules to "hop" to an energy shell with higher energy or be knocked-off from the particle. On the off chance that such a progress happens, the electron gives up an opening. Openings have a positive charge and, in the second step of the cycle, draw in the contrarily charged electrons from higher-energy shells. At the point when an electron from a particularly higher-energy shell fills the opening of the lower-energy shell, the energy distinction of this change can be delivered as a X-beam.

This X-beam has energy which is normal for the energy distinction between these two shells. It relies upon the nuclear number, which is an interesting property of each element. The information that is produced by EDX investigation comprises of spectra with tops comparing to every one of the various components that are available in the sample. Every component has trademark pinnacles of extraordinary energy, all broadly recorded.

EDX can be utilized for subjective (the kind of components) just as quantitative (the level of the centralization of every component of the example) investigation. In many SEMs, committed programming empowers auto-distinguishing proof of the pinnacles and figuring of the nuclear level of every component that is recognized.

4.5.3. ATOMIC FORCE MICROSCOPY (AFM)

An AFM generates images by scanning a small cantilever over the surface of a sample. The sharp tip on the end of the cantilever contacts the surface, bending the cantilever and changing the amount of laser light reflected into the photodiode. The height of the cantilever is then adjusted to restore the response signal resulting in the measured cantilever height tracing the surface. Fig 4.9 shows the Atomic force microscopy (AFM) operation.

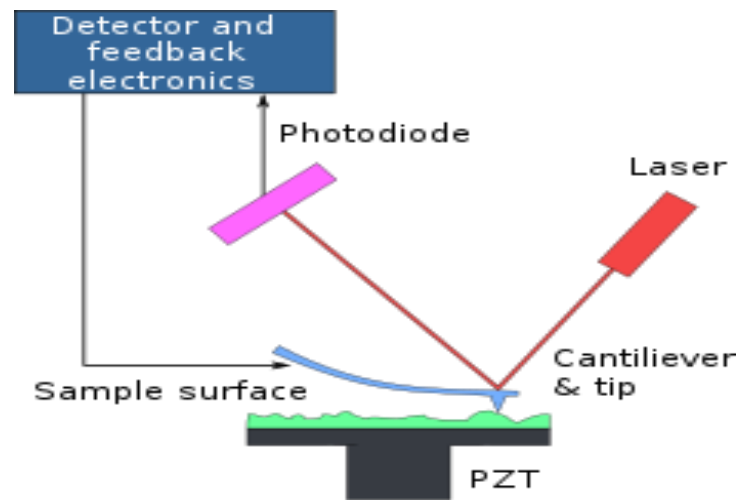


Fig 4.9 Atomic force microscopy (AFM)

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1. SEM ANALYSIS

5.1.1. PURE MAGNESIUM BEFORE CORROSION

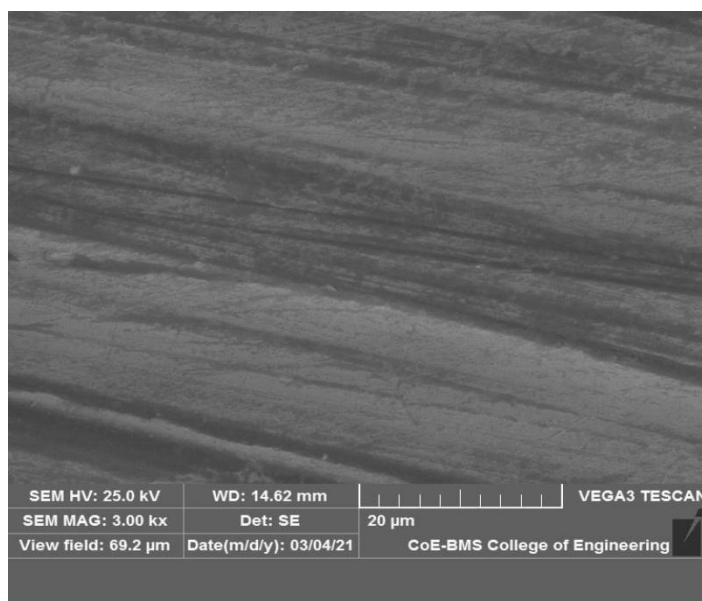


Fig 5.1 SEM of Sample 1

The EB-PVD coating is magnified to have the vision over the imperfection in the coating surface, such as having many porous. This porous surface can be responsible for deformation of the surface structure. Fig 5.1 shows the SEM of sample 1 Surface image of magnesium.

5.1.2. PURE MAGNESIUM AFTER CORROSION

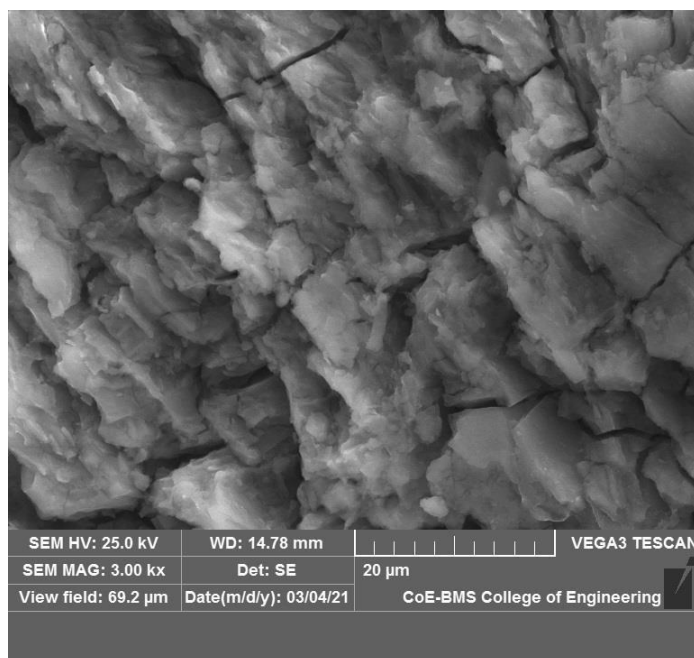


Fig 5.2 SEM of Sample 2

From Fig 5.2. We can say that there lot of imperfections in the coating. Pores are also seen in the Mg and Zr particles are completely merged. This may be least corrosive resistant.

5.1.3. YTTRIA BEFORE CORROSION

In SEM of sample 3 imperfections are seen from Fig 5.3. Ups and downs are seen in the figure. Some of the larger in size may be Zr particles.

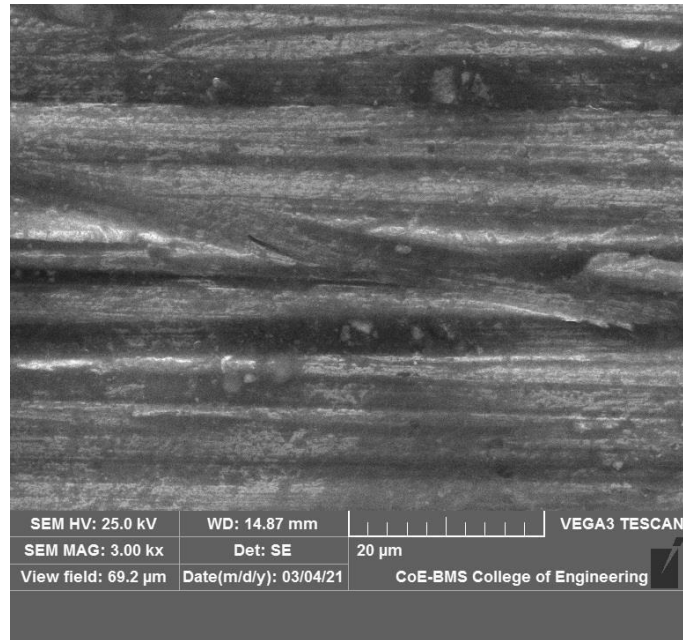


Fig 5.3 SEM of Sample 3

5.1.4. YTTRIA AFTER CORROSION

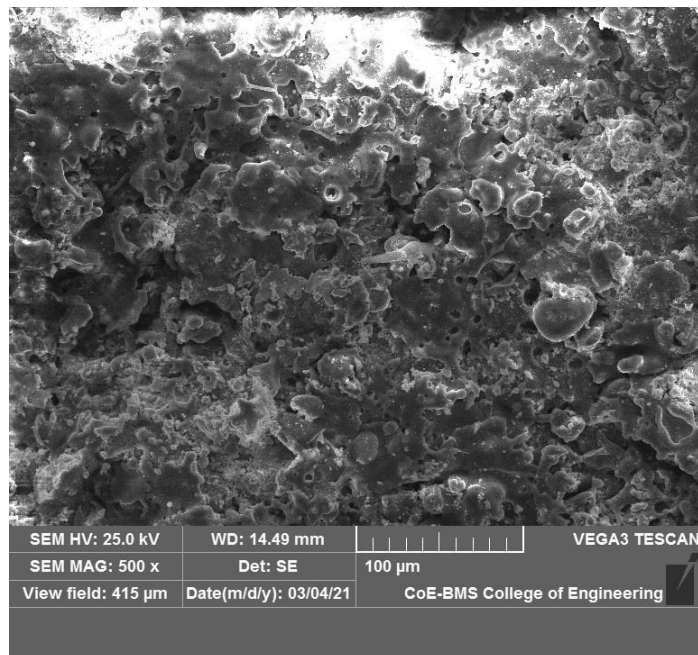


Fig 5.4 SEM of Sample 4

From Fig 5.4 SEM of Sample 4 shows that Corrosion resistance of yttria after corrosion is higher than the pure magnesium after corrosion. Lots of clusters and pores are seen in figure.

5.2. EDS ANALYSIS

5.2.1 MAGNESIUM BEFORE COATING

The weight and atomic percentage of elements which are found in the sample are found from these spectra. These spectra of sample 1 and 2 can be seen in Fig 5.5 respectively. The analysis shows the elements from the surface to the base material.

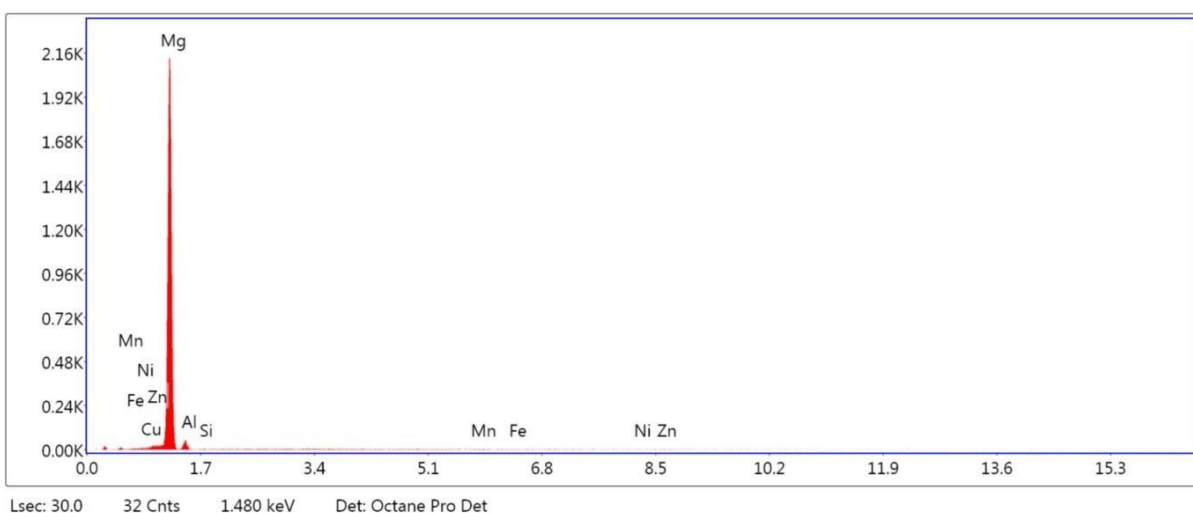


Fig 5.5 EDS of Sample 1

Table 5.1 Weight and Atomic percentage of Sample 1

Element	Weight %	Atomic %
MgK	88.40	90.44
AlK	9.80	8.37
SiK	0.29	0.25
MnK	0.50	0.23
FeK	0.29	0.13

NiK	0.46	0.19
CuK	0.38	0.15
ZnK	0.60	0.23

The amount and weight percentage of elements that are present in sample 1 obtained from the spectra are in the following table 5.1.

5.2.1 ZIRCONIUM OXIDE

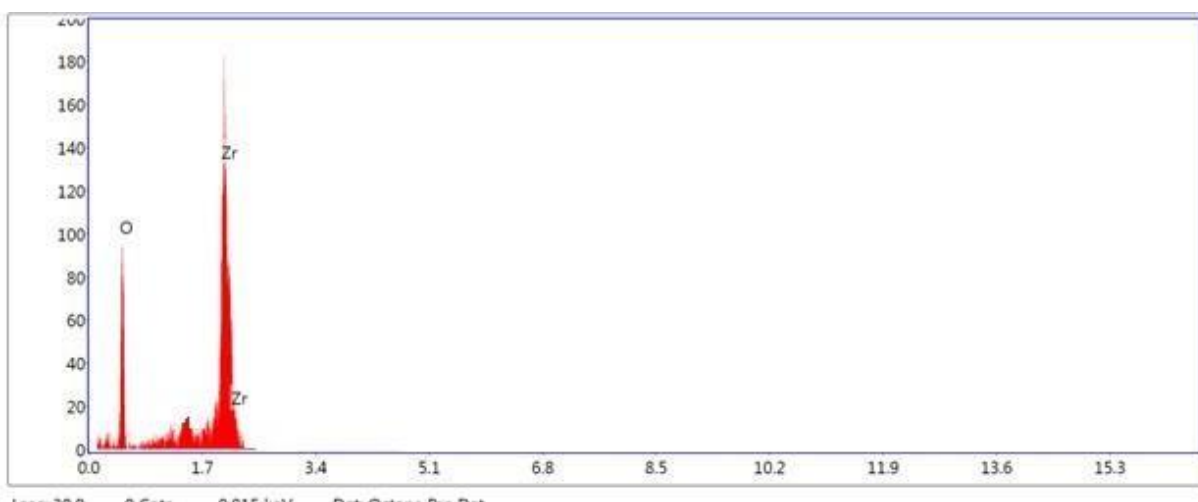


Fig 5.6 EDS of Sample 2

The weight and atomic percentage of elements which are found in the sample are found from these spectra. These spectra of sample 1 and 2 can be seen in Fig 5.6 respectively. The analysis shows the elements from the surface to the base material.

Table 5.2 Weight and Atomic percentage of Sample 2

Element	Weight %	Atomic %
ZrK	79.98	82.94
Oxide	21.02	23.04

The amount and weight percentage of elements that are present in sample 2 obtained from the spectra are in the following table 5.2.

5.3. AFM ANALYSIS

5.3.1 5X5 μm

Table 5.3 Surface Roughness of magnesium (5X5 mm)

Roughness Analysis	
Amount of sampling	46225
Max	80.8253
Min	0 nm
Peak-to-peak, S_y	80.8253
Ten point height, S_z	41.3143
Average	32.2244
Average Roughness, S_a	6.75975
Root Mean Square, S_q	8.62262
Second moment	1315.11
Surface skewness, S_{sk}	0.389365
Coefficient of kurtosis, S_{ka}	0.564933
Entropy	8.36176
Redundance	-0.32332

The table 5.3 represents the surface roughness for Zirconium coated magnesium for the amount of samples and maximum and minimum thickness of coating.

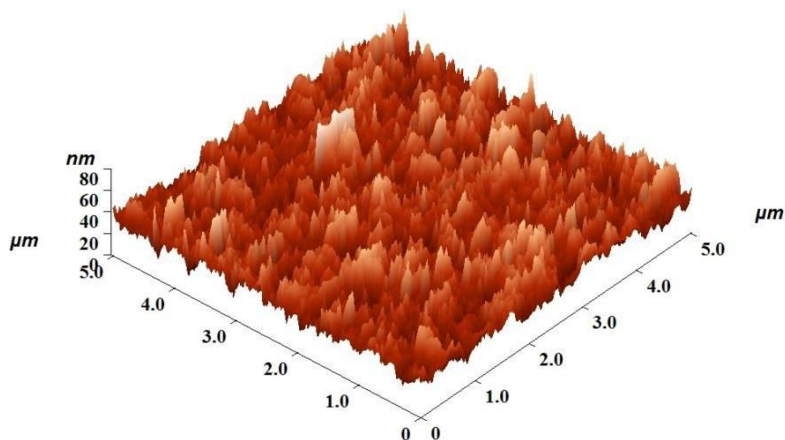


Fig 5.7 Surface Roughness of magnesium (5X5 mm)

The 3D view of Zirconium coated magnesium are scanned in atomic force microscopy to get surface roughness of the material. The Fig 5.7 shows the surface roughness of magnesium 5X5 mm.

5.3.2 10X10 μm

Table 5.4 Surface Roughness of magnesium (10X10 mm)

Roughness Analysis	
Amount of sampling	54522
Max	44.8676 nm
Min	0 nm
Peak-to-peak, S_y	44.8676 nm
Ten point height, S_z	22.7944 nm
Average	21.9924 nm
Average Roughness, S_a	5.31011 nm
Root Mean Square, S_q	6.58095 nm
Second moment	526.973
Surface skewness, S_{sk}	-0.0410517
Coefficient of kurtosis, S_{ka}	-0.268393
Entropy	7.99224
Redundance	-0.465099

The table 5.4 represents the surface roughness for Zirconium coated magnesium for the amount of samples and maximum and minimum thickness of coating.

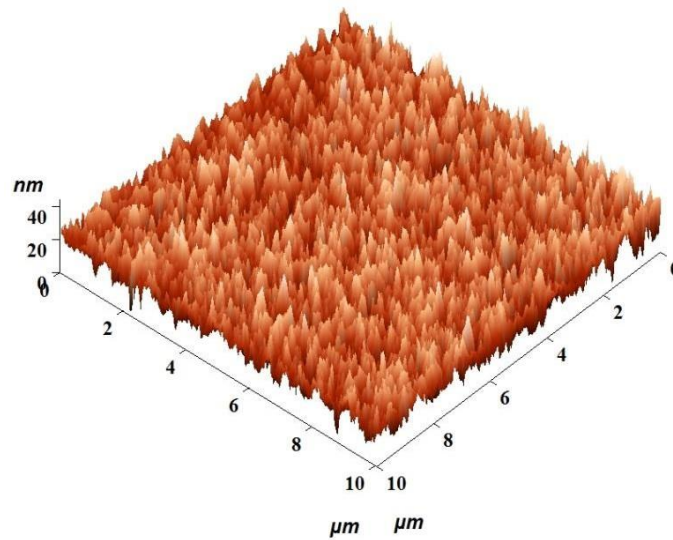


Fig 5.8 Surface Roughness of magnesium (10X10 mm)

The 3D view of Zirconium coated magnesium are scanned in atomic force microscopy to get surface roughness of the material. The Fig 5.8 shows the Surface roughness of magnesium 10X10 mm.

5.4 CORROSION TEST

5.4.1 CORROSION RATE OF MAGNESIUM

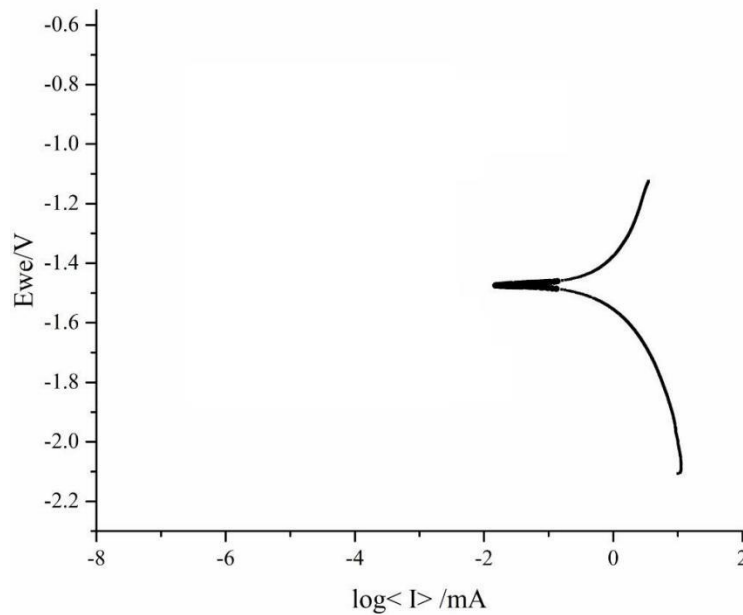
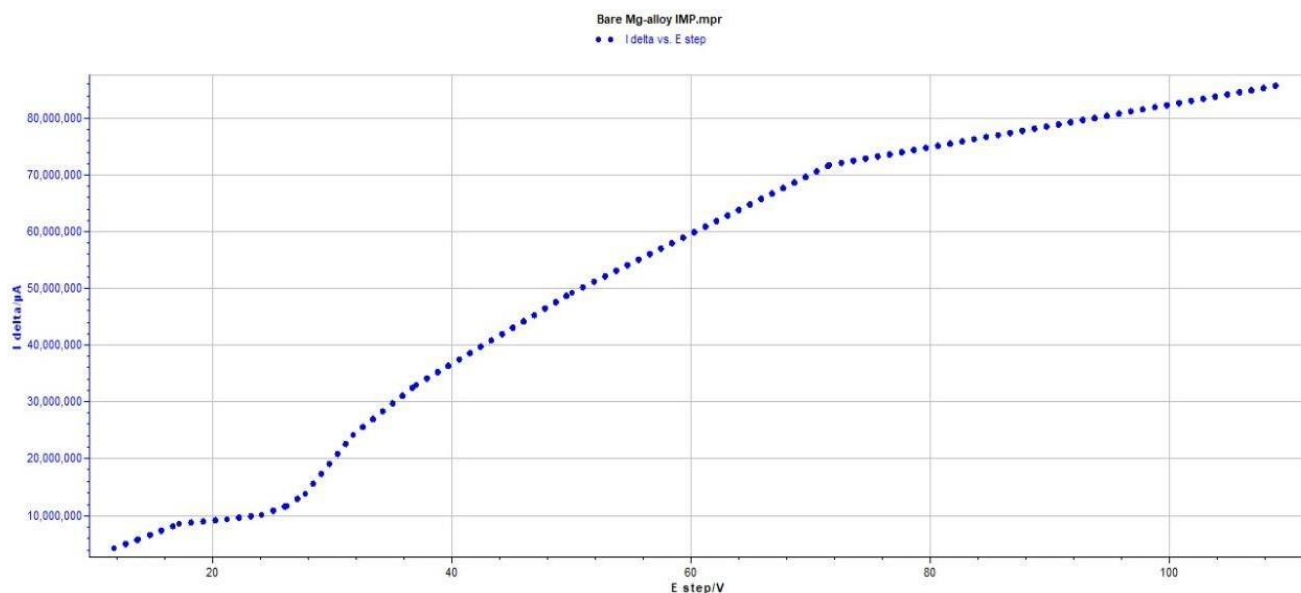


Fig 5.9 Corrosion result graph magnesium



From the graph we can conclude that Fig 5.9 has low corrosion resistance compared to zirconium. The corrosion resistance is determined by amount of weight the sample is gained when it is present in the furnace.

Table 5.5 Corrosion rate of magnesium

Re(Z)/Ohm	-Im(Z)/Ohm
11.710446357727051	4.311163425445557
13.740640640258789	5.899717330932617
17.176998138427734	8.509567260742188
21.140911102294922	9.418942451477051
24.027524948120117	10.031784057617188
26.214454650878906	11.659025192260742
27.731592178344727	13.874509811401367
31.739065170288086	24.161417007446289
37.009292602539063	33.088100433349609
49.994834899902344	49.262001037597656
71.571174621582031	71.735054016113281

109.745468139648438	85.964591979980469
151.160812377929688	84.640281677246094

The table 5.5 represents corrosion rate of magnesium that are tested using stimulated body fluid and this tables shows that magnesium has low and high corrosion resistance.

5.4.2 CORROSION RATE OF ZIRCONIUM COATED MAGNESIUM

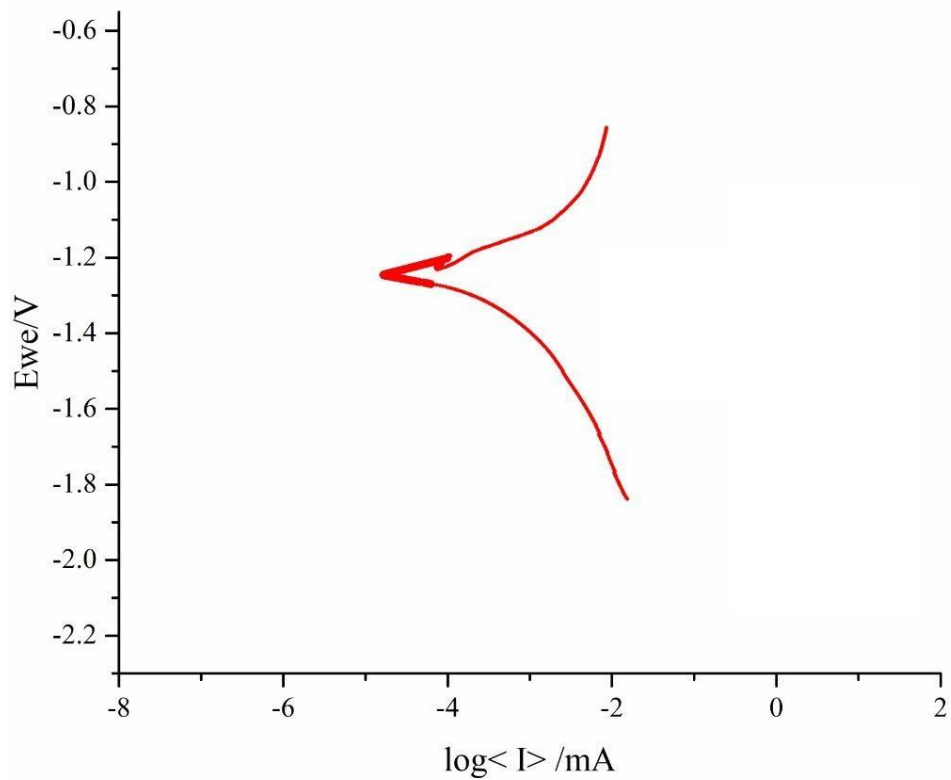
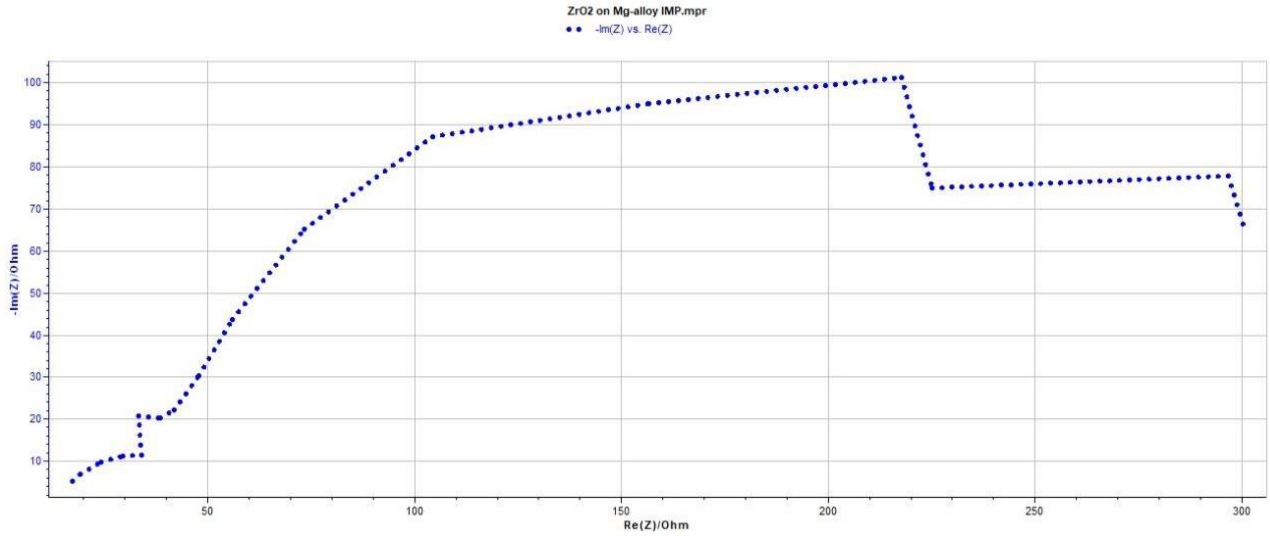


Fig 5.10 Corrosion result graph for Zirconium coated Magnesium



From the graph we can conclude that Fig 5.10 has high corrosion resistance compared to zirconium. The corrosion resistance is determined by amount of weight the sample is gained when it is present in the furnace.

Table 5.6 Corrosion rate of Zirconium coated magnesium

Re(Z)/Ohm	-Im(Z)/Ohm
17.182769775390625	5.269567012786865
19.229497909545898	7.027135848999023
24.160133361816406	9.908784866333008
29.351453781127930	11.236279487609863
33.923267364501953	11.548502922058106
33.349380493164063	20.870248794555664
38.386577606201172	20.312767028808594
41.912502288818359	22.233886718750000
47.698276519775391	30.375183105468750
55.856369018554688	43.652534484863281
73.288291931152344	65.136009216308594

104.260940551757812	87.228065490722656
156.684555053710938	95.063613891601563
217.744888305664062	101.252464294433594
225.170547485351562	75.059989929199219
296.833221435546875	77.945922851562500
300.404846191406250	66.349731445312500

The table 5.6 represents corrosion rate of magnesium that are tested using stimulated body fluid and this tables shows that magnesium has low and high corrosion resistance.

5.4.3 CALCULATION

Bare Mg-alloy Corrosion

(x) Ecorr = -1 470.805 mV

(x) Icorr = 500.805 uA

(x) beta c = 249.2 mV

(x) beta a = 311.0 mV

ZrO2 on Mg-alloy Corrosion

(x) Ecorr = -1248.068 mV

(x) Icorr = 114.826 uA

(x) beta c = 190.1 mV

(x) beta a = 130.7 mV

Corrosion rate of Magnesium

$$\begin{aligned}
 CR &= \frac{3.27 \times 10^{-3} \times i_{corr} \times E_w}{p} \\
 &= \frac{3.27 \times 10^{-3} \times 500.805 \times 24.36}{1.75} \\
 &= \frac{39.8927}{1.75} \\
 &= 22.7958 \text{ mmpy}
 \end{aligned}$$

Corrosion rate of Zirconium

$$\begin{aligned}
 CR &= \frac{3.27 \times 10^{-3} \times i_{corr} \times E_w}{p} \\
 &= \frac{3.27 \times 10^{-3} \times 114.826 \times 24.36}{1.75} \\
 &= \frac{9.1467}{1.75} \\
 &= 5.2266 \text{ mmpy}
 \end{aligned}$$

CHAPTER 6

CONCLUSION

The concept of coating Yttria Zirconium over the magnesium alloy have been achieved through Electron beam physical vapor deposition (EB-PVD). The artificial blood plasma causes the corrosion AZ91D Magnesium alloy and coated surface. The SEM and AFM revealed the formation of Magnesium coated Zirconium. Corrosion rate of coated samples is greater than the magnesium alloy. EB-PVD coating has improved the corrosion resistance of AZ91D magnesium alloy.

This Mg-Zr alloy has been developed as a biodegradable Mg-based alloy for bone implant applications. It achieves enhanced mechanical properties and antibacterial ability through alloying Mg with Zr. Thermomechanical processes of Magnesium were performed on the Yttria based Zirconium to further boost the mechanical properties.

Overall, based on the testing results for mechanical properties, corrosion and corrosive-wear property, degradation behavior, biocompatibility, and antibacterial ability, the Mg-Zr alloy is a promising Candidate for bone fracture fixation applications such as bone screws and plates of biodegradable implants due to its markedly enhanced mechanical properties, wear resistance, antibacterial ability and suitable degradation rate.

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