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REVIEW ARTICLE

Surface modification techniques of magnesium-based alloys for implant applications

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Abstract Biodegradable materials like magnesium-based alloys are widely employed for making implants. Mg-based alloys show good biocompatibility, biodegradability, and mechanical properties similar to bone material unlike other commonly used implant materials, i.e., stainless steel, titanium, and Co–Cr alloy, which have negligible degradation rates and require revision surgery for the removal of the implant. Magnesium alloy-based implant has high degradation rates and avoids the need for a second surgery. But high degradation rates and poor mechanical properties in magnesium alloys also pose a challenge to their use as implant material. In the absence of controlled degradation, they can degrade completely before serving their intended purpose in the human body. Several surface modification techniques are used to control the degradation rates. Surface coating is one of the methods of surface modification. This paper discusses various types of surface coating techniques for magnesium-based alloys. This paper also discusses the future scope of surface technology of magnesium-based implant materials.

Keywords Biomaterials, Magnesium alloys, Implant applications, Surface modification, Conversion and non-conversion coatings

Abbreviations

PEO	Plasma electrolytic oxidation
MAO	Micro-arc oxidation
HA	Hydroxyapatite
SEM	Scanning electron microscopy
PLA	Polylactic acid
DCPD	Dicyclopentadiene
DCPA	Dicalcium phosphate anhydrous
MAO/TiN	Micro-arc oxidation/titanium nitrate
MAO/Al	Micro-arc oxidation/aluminum
CS	Chitosan
RF-MS	Radio-frequency magnetron sputtering
BG	Bioglass
SBF	Simulated body fluid
EPD	Electrophoretic deposition
MAO/BAG	Micro-arc oxidation/bio-active glass
CGDS	Cold gas dynamic spraying
PED	Pulsed electrodeposition
EIS	Electrochemical impedance spectroscopy
PLGA	Poly(lactic-co-glycolic acid)
nHAp	Nanostructured hydroxyapatite
Rsbf	Revised simulated body fluid

Introduction

Medical implants are used to support a fractured bone or replace a missing biological part in the body. Various materials like zinc, magnesium, titanium, stainless steel, polymers, and others are widely used for making implants.[1] The selection of a material for implant applications depends on requirements. In a minor fracture, biodegradable material can be an ideal choice as implant material. In case of severe fracture or replacing a missing part of the body, an implant with high strength and durability should be preferred. Materials like stainless steel, Ti alloys, and cobalt-chromium alloy have good corrosion resistance and

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mechanical strength but poor biodegradability. The implants made of these materials remain present for a longer duration in the body and cause endothelial dysfunction and other health-related problems.[2] This necessitates the removal of the implant by a second surgery, which is costly and painful.[3]

In contrast, biodegradable materials like magnesium have favorable properties for implant applications. They have excellent biodegradability and biocompatibility. Biodegradable implant materials have mechanical properties like density and modulus of elasticity, similar to human bone. Stress shielding is caused due to differences in modulus of elasticity of the implant material and bone.[4] In magnesium, the impact of stress shielding is negligible.[5]

Magnesium-based alloys also possess excellent biodegradability and biocompatibility.[6] The implant made of magnesium-based alloys need not be removed through revision surgery as it fully degrades with time and mixes with body fluid to work as a nutrient in the body.[7] The main problem associated with magnesium-based implants is the uncontrolled degradation rate in the body environment.[8] These implants get corroded before completing their role as an implant to support the bone. Hence, to control higher degradation rates and to enhance mechanical properties, the surfaces of these implant materials must be modified. Various surface modification techniques have been developed by researchers, like alloying, protective coating, and chemical treatment.[9]

Surface coating is one of the techniques of surface modification to make implants corrosion resistant.[10] Substrate surfaces are deposited with single or multiple layers of protective coating. While surface coating, the focus is not only on corrosion protection but also on other parameters like biocompatibility, bioactivity, adhesion, strength, hardness, and other properties.

Surface coating techniques in magnesium-based alloys include plasma electrolytic oxidation, anodization, radio-frequency magnetron sputtering, hydrothermal process, electrophoretic deposition, pulsed electrodeposition, physical vapor deposition, dip coating, and spin coating. Other used techniques include electrospinning, cold gas dynamic spraying, and surface cladding.

However, despite extensive work and the availability of multiple surface modification approaches, more dedicated research is needed to overcome the problems associated with magnesium-based implants. Improving the corrosion rate without sacrificing the biocompatibility and improving strength without stress shielding effects have become essential for a successful implant.

This review paper aims to give an account of various types of surface modification techniques being used for magnesium-based alloys and related aspects. Typical examples taken from recently published works in this area have been presented in this paper. We have also discussed the factors necessary for selecting an implant material and future scope.

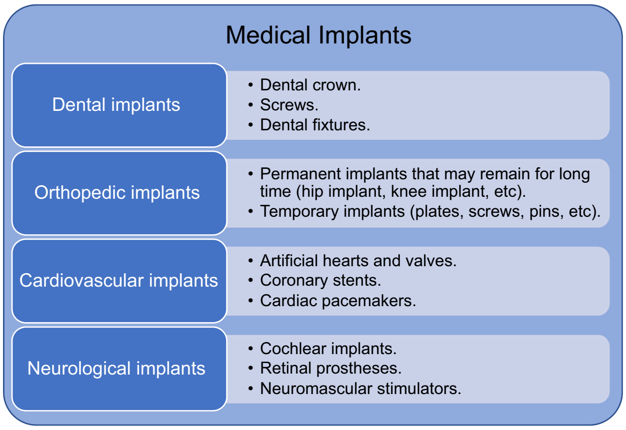


Fig. 1: Types of medical implants

Types of medical implants

An implant is a prosthetic device fabricated from biomaterials used to support a fractured bone or replace a missing part of the body. Implants can be classified on the basis of their applications as dental implants,[11] orthopedic implants,[12] cardiovascular implants,[13] and neurological implants.[14] Figure 1 shows types of medical implants with their applications.

Basic requirements of a suitable implant material

The important criteria on which the properties of the implant depend are grouped into four categories: (1) Physical, (2) Mechanical, (3) Chemical, and (4) Biological, as shown in Fig. 2.

The basic requirements for selecting a material for implant applications depend on the material properties. In Table 1, important properties related to implant material are given.

Commonly used materials for implant applications

Various types of materials are used for making implants for medical applications.[31] Material selection for implants depends on the type of application. In some applications, implants are intended to stay inside the body for longer periods, then non-biodegradable materials are used. When implants are intended to remain inside the body for a limited time and are required to degrade completely inside the body, biodegradable materials are used. The most commonly used materials for implant applications are given in Table 2.

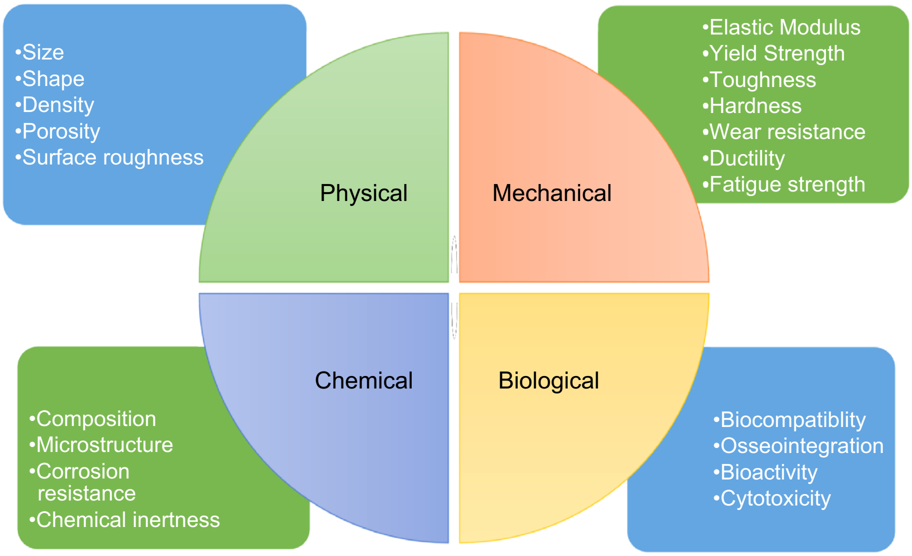


Fig. 2: Basic criteria for the selection of an implant material

Table 1: Important properties of implant material

Properties	Description
Density	Implant density should be close to the bone material[15]
Porosity	Porous implants improve osseointegration and bone fixation[16]
Strength	Should have adequate strength to prevent fracture and improve stability[17]
Surface roughness	Surface roughness affects implant stability and osteointegration[18]
Modulus of elasticity	It should be close to the bone material[19]
Toughness	It prevents bone fracture due to impact loading[20]
Hardness	An increase in hardness reduces implant wear[21]
Ductility	Ductility imparts shaping and contouring to the implants[22]
Fatigue strength	Implant should have enough strength to withstand cyclic loading[23]
Wear resistance	Excess wear causes debris which is not acceptable in some uses[24]
Chemical inertness	Implants should be stable in the body environment[25]
Biocompatibility	Should show favorable biological response[26]
Biodegradability	Should be vanished inside the body after serving the purpose[27]
Osseointegration	Good osseointegration ensures proper contact between bone and implant[28]
Corrosion resistance	Should have adequate corrosion resistance[29]
Cytotoxicity	Implant material should not damage body cells[30]

Table 2: Commonly used materials for implant applications

Materials	Major applications
Stainless steels[32]	Stents, plates, pins, nails, and other medical instruments
Co-Cr alloy[33]	Dental applications, joint replacements, cardiovascular valves, etc
Titanium-based alloys[34]	Dental implants, screws, nails, cardiac pacemakers, etc
Tantalum-based alloys[35]	Joints, bone fixators, dental implants, etc
Magnesium-based alloys[36]	Stents, screws, pins, plates, etc
Zinc based alloys[37]	Plates, screws, stents, wound closure devices
Fe-based alloys[38]	Stents, plates, pins, etc
High entropy alloys[39]	Stents, hip joints, cardiovascular devices, etc
Polymers[40]	Dental implants, cardiovascular devices, cochlear implants, hip implants, orbital implants, etc
Ceramics-based[41]	Prosthetic hip, knees, bone grafts, dental applications, etc

Importance of magnesium alloy for implant applications

Magnesium is a revolutionary material for implant applications. Mg has several properties that overcome the limitations of other implant materials. Magnesium has low density. Its strength-to-weight ratio is quite high.[42] The elastic modulus of magnesium is 45 GPa, close to that of human bones (40–57 GPa).[43] At the same time, other implant materials have a much higher modulus of elasticity. Commercially pure titanium has a modulus of elasticity of 106 GPa.[44] Due to this favorable property, the stress shielding effect in magnesium-based implants is much lower than other metallic implants. Magnesium also has high degradation rates, and if degradation rates are controlled, implant material will degrade at the desired speed and degrade completely inside the body. This avoids the need for revision surgery for implant removal, thereby reducing health risks, costs, and scarring.[45] Magnesium acts as a nutrient in human body. Hence, the remains of magnesium are not toxic and harmful to the body. Magnesium is also helpful in the prevention of many diseases.[46] The daily requirement of Mg ions in a human body is between 250 and 500 mg depending on age and other factors.[47] About 20 g of Mg remains present in a healthy human body of an average of 70 kg. Magnesium has various metabolic and biological functions in our body, and the degraded products of magnesium can aid in those functions.[48]

Designation of magnesium alloys

Magnesium alloys are designated based on the amount of principal alloying elements. The abbreviations used for the designation of magnesium alloy are given in Table 3 and an example of alphanumeric designation of magnesium alloy is given in Table 4.

Table 3: Abbreviation used for alphanumeric designation of various alloys of magnesium

Abbreviation	Principal alloying elements
M	Mn
AM	Al–Mn
AZ	Zn–Mn–Al
K	Zr
ZK	Zr–Zn
ZE	Zn–Zr with rare earth
EZ	Zr-rare earth metal
QE	Ag–Zr-rare earth metal
WE	Y–Zr-rare earth metal
ZC	Zn–Cu–Mn
AS	Al–Si–Mn
AJ	Al–Sr

Mechanical properties analysis of Mg with other metallic implants

Metallic materials like titanium alloys, cobalt-chromium alloys, and stainless steel have been extensively employed for making implants for a long time. These metallic materials have good strength, toughness, and low susceptibility to corrosion. Due to these properties, they remain for a longer time in the body and support the cracked or damaged bone until there is a full recovery. But, in case of minor damages in bones and tissues, these implants, which are less susceptible to corrosion, are not preferred. These metallic implants also differ from the properties of human bones. Biodegradable materials like zinc,[50] biopolymers,[51] and magnesium alloys nowadays replace conventional metallic implants. They possess good biodegradability and biocompatibility. Their properties are also comparable to bone material. The mechanical properties of various implant materials are given in Table 5.

Based on mechanical properties, we can compare magnesium alloys with other implant material. For implant applications, magnesium alloys have certain advantages and disadvantages which are mentioned in Tables 6 and 7, respectively.

Advantages and disadvantages of Mg alloys

See Tables 6 and 7.

Need of surface modification in magnesium-based implant materials

In the biological environment, magnesium degrades rapidly and even before the complete healing of the fractured bone.[69] During degradation of the implant material, hydrogen evolution takes place, which further accelerates the corrosion rates. Also, there are high chances of alkalization at the implant-tissue interface. There will be premature implant failure, reduced bioactivity, and severe inflammatory reactions in these circumstances.[70] Therefore, surface modification of the magnesium alloys is required to reduce or control the degradation rate to match the healing rate of the bone. The surface modification also improves the biocompatibility, bioactivity, and mechanical properties of the implant.[71]

Various surface modification techniques of magnesium-based alloys

Surface modification means modifying the implant surface by altering its microstructure, composition, or both. Surface modification can be achieved in the following ways:

Table 4: Alphanumeric designation of magnesium alloys (example of AZ31B in parentheses)[49]

First part (AZ)	Second part (31)	Third part (B)
The first and second letters indicate two major alloying elements	Indicates percentage of the two major alloying elements	It differentiates one alloy from another, having the same amount of alloying elements. Letters of the alphabet are used except I and O
In AZ31B, AZ-shows Al and Zn as two major alloys	In AZ31B, aluminum is 3%, and zinc is 1%	

Table 5: Mechanical properties of implant materials

Implant material	Density (g/cm^3)	Elastic modulus (GPa)	Ultimate tensile strength (MPa)	Yield strength (MPa)	Percentage elongation (%)
316L stainless steel[52]	8	193	490	190	40
Co-Cr alloy[53]	9.2	210	860	310	20
Ti6Al4V titanium alloy[54]	4.43	114	950	880	14
Tantalum[55]	16.6	185	207	138	–
Pure iron[56]	7.87	200	210	150	40
ZnCl4Cu1 zinc alloy[57]	5	130	210	171	1
WE43 Mg alloy[58]	1.84	44	220	170	2

Table 6: Advantages of Mg-based alloys

Advantages	Description
Low density	The density of Mg is low and comparable to human bone[59]
High specific strength	The strength-to-weight ratio is comparatively high[60]
Good machinability	Easy to machine into various shapes and sizes[61]
Good	biocompatibility
Mg alloys promote bone formation[62]	
Low-stress shielding	Modulus of elasticity of Mg comparable to human bone avoids stress shielding[63]
Non toxic	Corrosion products are non-toxic in nature[64]
Good	biodegradability
Complete degradation of the implant inside the body avoids revision surgery[65]	

Table 7: Disadvantages of Mg-based alloys

Disadvantages	Description
Rapid degradation	Due to rapid degradation rates, implants unable to support bone recovery[66]
High hydrogen evolution	H_2 gas evolves at the surrounding soft tissues and damages them[67]
Low elastic modulus	Lower elastic modulus is good for avoiding stress shielding but it tends to have poor load-bearing capacity[68]

(1) Deposition: Protective layer is deposited on the substrate surface to prevent corrosion.[72]

(2) Alloying: Two or more metals are mixed to form an alloy that is corrosion resistant.[73]

(3) Microstructural modification: Techniques like severe plastic deformation[74] or ultrasonic nanocrystalline surface modification.[75]

Surface modification based on deposition or surface coating techniques can be classified into two major categories: (1) conversion coatings and (2) non-conversion coatings. Figure 3 shows classification of surface coating techniques of magnesium-based alloy.

Conversion coatings

Conversion coatings of magnesium alloys consist of coating oxide or hydroxide of magnesium. The oxidation of the substrate surface results in the formation of oxides or hydroxides layers which act as barriers against corrosion. Various techniques come under conversion coatings like plasma electrolytic oxidation (PEO), anodization, or hydrothermal treatment.

Plasma electrolytic oxidation is the most versatile, simple, and eco-friendly coating technique in all conversion coating processes.[76]

Plasma electrolytic oxidation

Micro-arc oxidation or plasma electrolytic oxidation is the most commonly used surface coating technique to produce thick and dense oxide layers.[77] In this method, plasma is generated and discharged using an external power supply in a low concentration electrolyte near the workpiece, which acts as anode of the system. A high-temperature plasma is generated between the electrodes. The generated plasma melts the workpiece surface. An inert and highly adhering oxide layer is formed over the substrate surface.[78] The oxide layer formed on the surface of the implant material can have variables.[79] This makes the process favorable for biomedical applications. The basic setup of the plasma electrolyte oxidation technique is shown in Fig. 4.

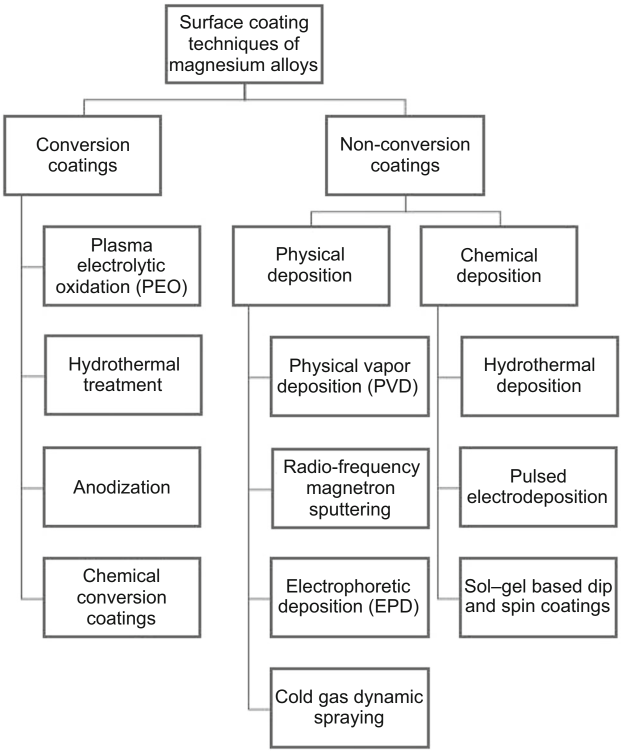


Fig. 3: Classification of surface coating techniques for magnesium alloys

Tang et al. coated hydroxyapatite on AZ31 Mg alloy by plasma electrolyte oxidation.[80] They studied the variation in HA-coated alloy properties with the variation in applied voltage. The corrosion studies of the substrates were done by placing them in the Hanks' solution.[81] SEM images of the substrates are shown in Fig. 5. The bonding strength of coatings decreased on increasing applied voltage. Both surface roughness and contact angles were increasing with the voltage. Hydrogen evolution was minimum at 400 V, and thus best corrosion resistance was observed at this voltage.

Hussein et al.[79] studied the effects of process parameters on corrosion resistance of AJ62. The impact of current mode and discharge was studied. The unipolar current mode caused significant porosity and microcracks. The bipolar current mode caused a thick inner part, and a thin outer layer with a porous structure. The bipolar mode had better corrosion resistance than the unipolar mode.

Zhuang et al.[82] investigated the effects of current density on PEO-coated magnesium alloy. From the SEM images taken (Fig. 6), after coating, pores were found on the surface. With the increase in current density, pore size increased, and the number of pores decreased. Coatings produced at higher current densities were thicker and thus had more corrosion resistance.

Toulabifard et al.[83] investigated the effects of electrolytes on coating. Coatings were developed on

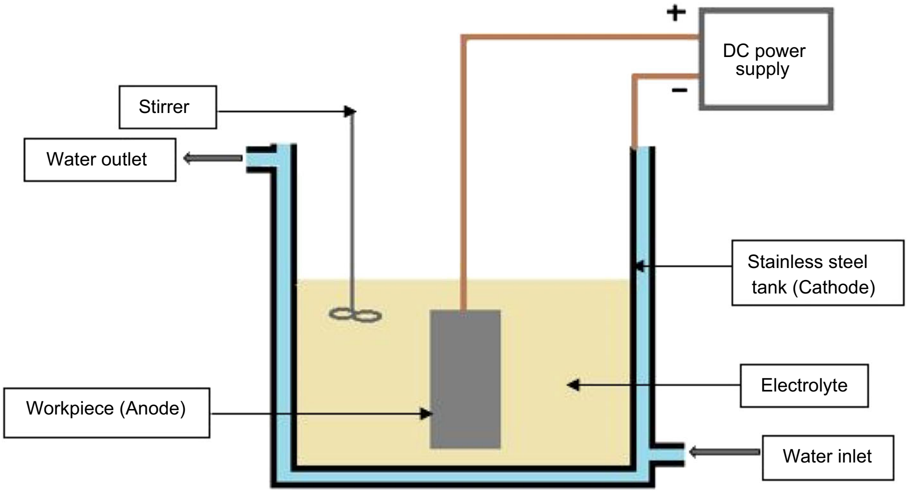


Fig. 4: Setup diagram of DC plasma electrolytic oxidation technique

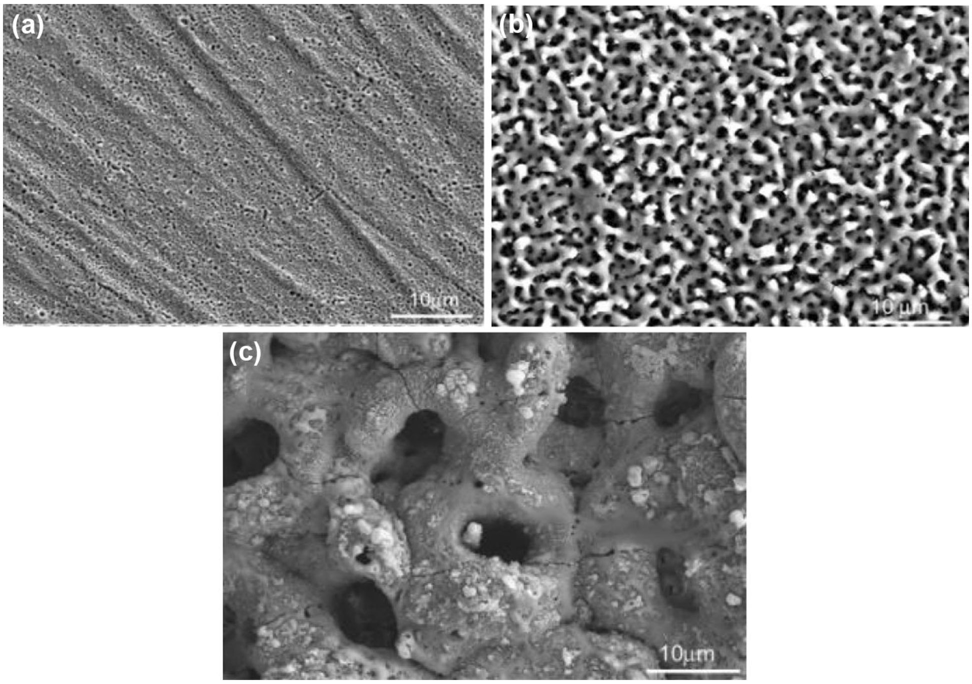


Fig. 5: Surface morphologies of the PEO coatings produced at (a) 250 V, (b) 400 V, and (c) 500 V[80]

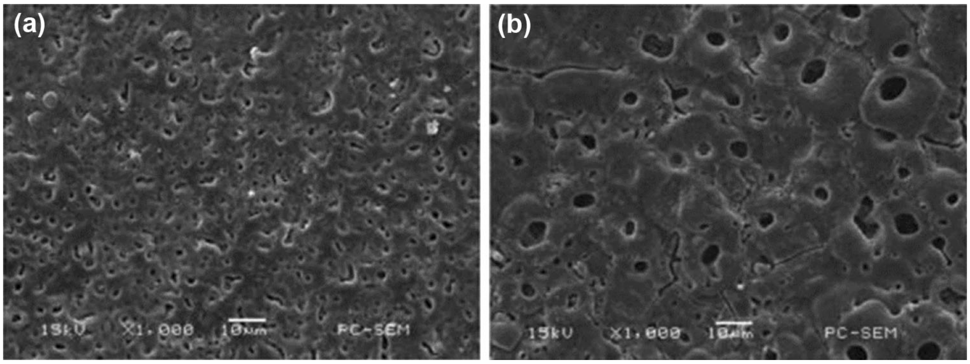


Fig. 6: SEM images of the surface morphologies produce at current densities of (a) 5 A/dm^2, and (b) 20 A/dm^2[282]

AZ31. Electrolytes used were silicate, phosphate, and aluminate-based. From the surface morphology of the substrates, a volcano type of structure with craters was observed on the aluminate-based coating. Non-uniformly distributed micropores with sintered craters were obtained on phosphate-based coating and a highly porous structure with a network of micropores was obtained on silicate-based coating. During two days immersion test, the highest corrosion resistance was observed in silicate-based coatings.

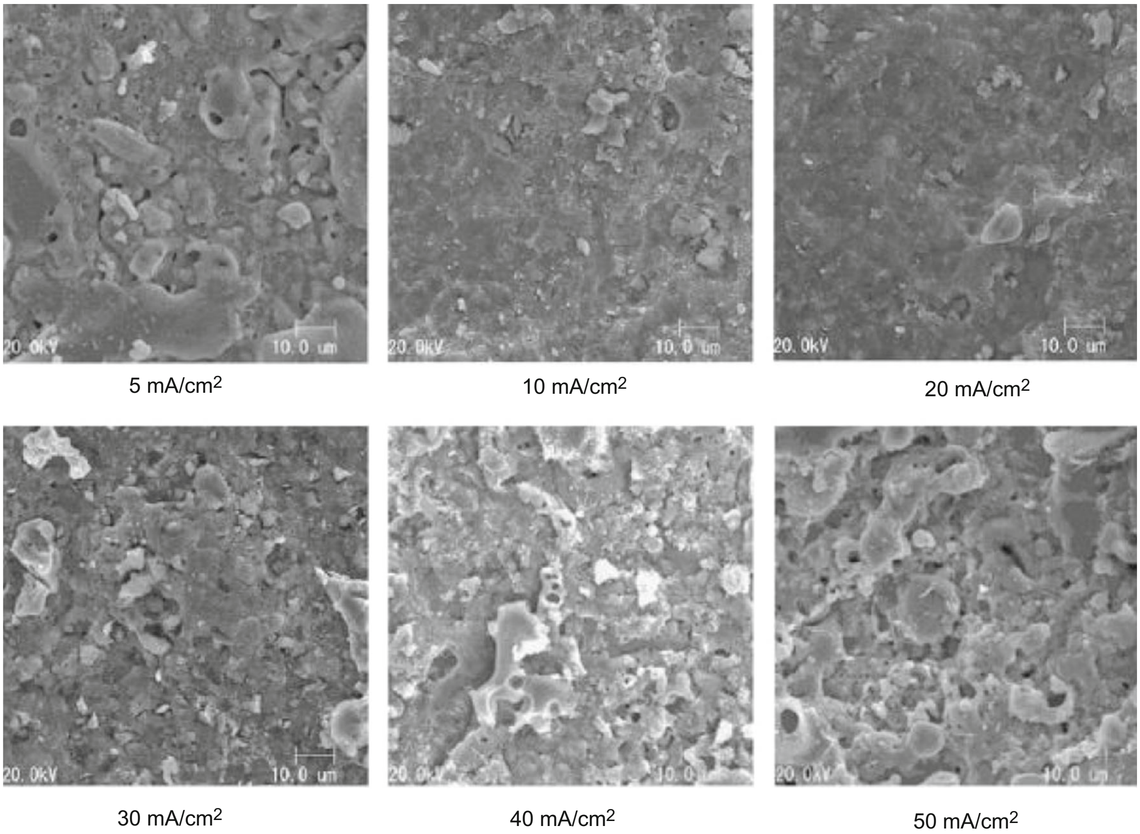
Parichehr et al.[84] deposited a composite coating of PEO/silane on AZ31 Mg alloy. The pores generated during PEO coating were filled by silane. The silane coating did not allow the electrolyte to enter into the substrate. This had improved corrosive strength of the alloy.

Wang et al.[85] deposited MgO and ZrO_2 and compared their corrosion behavior and cytotoxicity. From the polarization tests, the corrosion potential of ZrO_2-based and MgO-based coating was – 1.3911 V and – 1.4174 V, respectively. Thus, ZrO_2-based coating had better corrosion resistance than MgO-based coating. ZrO_2-based coating did not cause any toxicity and promoted the growth of the cells.

da Silva Rodrigues et al.[86] performed PEO coating on ZK30 alloy and investigated their corrosion resistance. The PEO-coated ZK30 exhibited increased corrosion resistance and decreased hydrogen evolution rates compared to the uncoated ZK30 alloy.

Anodization

Anodization is a simple technique of surface coating of magnesium alloys to control the degradation rate.[87] This is an eco-friendly process. It employs low voltage


5 mA/cm2

10 mA/cm2

20 mA/cm2

30 mA/cm2

40 mA/cm2

50 mA/cm2

Fig. 7: SEM images of the surface anodized at different current densities[90]

and low energy. The coating thickness between 5 and 200 μm can be achieved by this process.[88]

Xue et al.[89] deposited coatings pure Mg and AZ91D alloy from an electrolyte containing sodium hydroxide and sodium silicate showing increased corrosion resistance. Anodization time had substantial effects on corrosion resistance. Variation in the anodization parameters, like electrolytic concentration, time, and current density, greatly affected the porosity and quality of coatings.

Chai et al.[90] studied the impact of various process parameters in anodized AZ31 Mg alloy. They studied the effect of sodium silicate in the electrolyte, corrosion current density, and temperature. The addition of sodium silicate resulted in sparking and anodic film formation. The film increased the corrosion resistance of the coating. The amount of sodium silicate also had a significant impact on coating. The optimum concentration was 90 g/L. The use of high current density increased porosity and corrosion resistance. The surface morphologies of the substrates at different current densities are shown in Fig. 7.

Forero López et al. studied the corrosion resistance of the AZ91D alloy-coated samples produced in molybdate solution. The corrosion current density obtained was 0.0165 ± 0.005 mA/cm^2.[91] The coating thickness was approximately 70 μm. The coating had shown good corrosion resistance. Mousa et al. investigated the corrosion resistance of AZ31B.[92] They had also investigated the cytocompatibility of the coating. The addition of ZrO_2 nanoparticles enhanced bioactivity and biocompatibility.

Chemical conversion coating

In this technique, magnesium alloy substrates are immersed into solutions containing calcium phosphate. Some organic constituents like tannic acid, epigallocatechin gallate,[93] PLA,[94] or phytic acid[95] may also contain in the solutions. Calcium phosphates, such as HA or brushite, are the most common coating constituents while $Mg(OH)_2$ being a minor component.

Zhu et al.[96] deposited AZ31 alloy with HA/tannic acid. The surface morphologies of the bare and coated alloys are shown in Fig. 8. A uniform microstructure was obtained due to the inclusion of tannic acid, which promotes the increment of nucleation sites of hydroxyapatite. Corrosion current density decreased considerably and corrosion potential increased by about 160 mV. The coated alloy also promoted cell growth. SEM images of the substrates after the immersion tests for 7 days are shown in Fig. 9.

A bilayer coating of DCPD/PLA was developed on Mg–Nd–Zn–Zr alloy by Zhang et al.[94] The value of i_{corr} for the coated alloy was 0.95 μA/cm^2. The coated

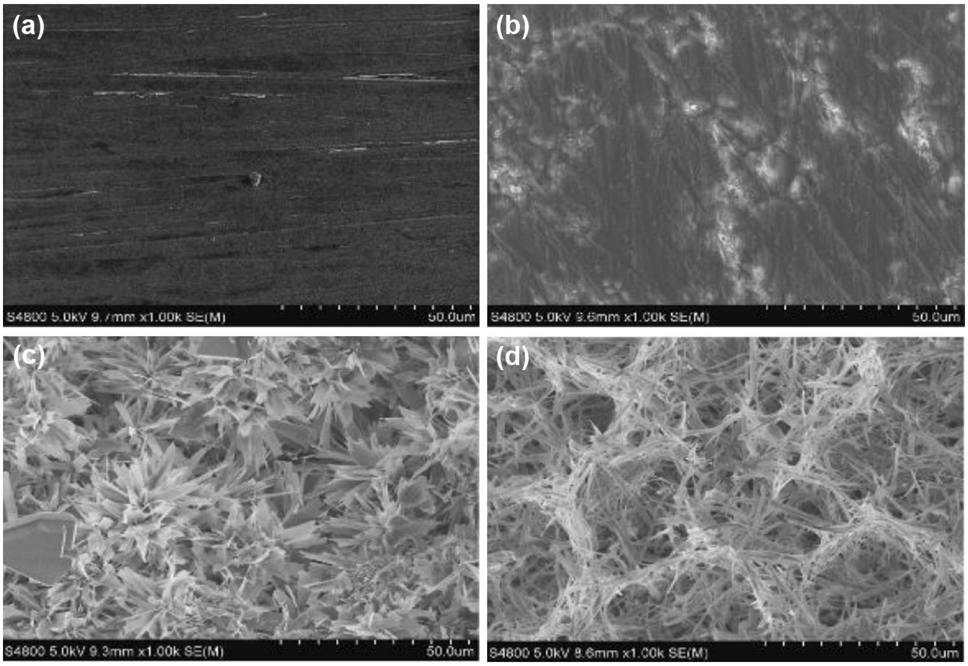


Fig. 8: SEM images of (a) AZ31, (b) TA/AZ31, (c) HA/AZ31, and (d) TA/HA/AZ31[96]

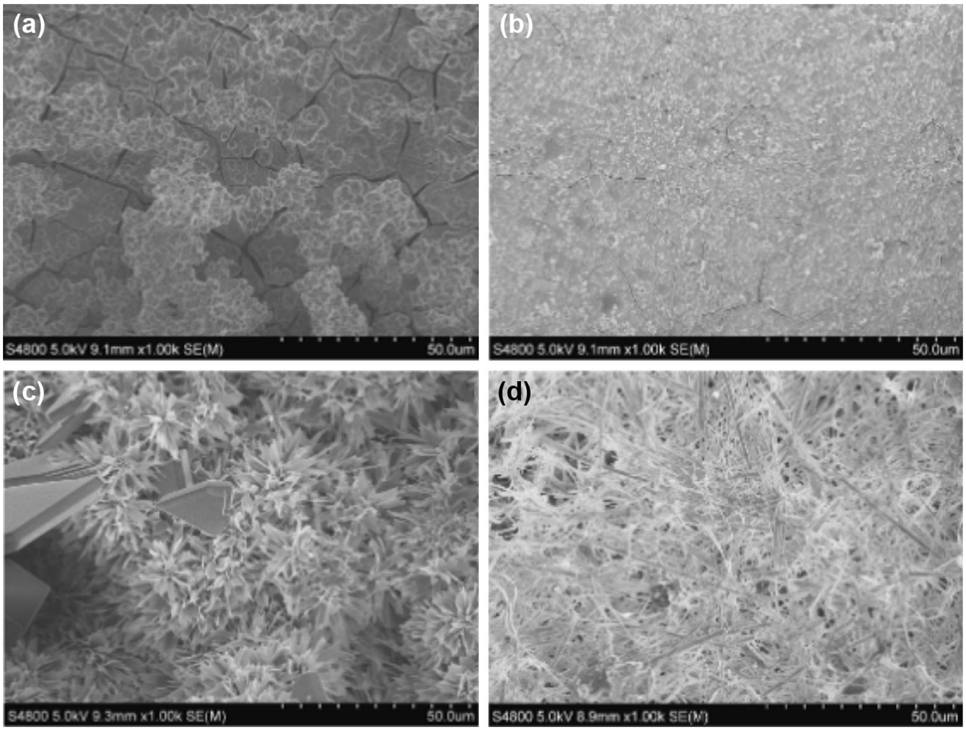


Fig. 9: SEM images of the surface after immersion test for 7 days (a) uncoated AZ31 (b) TA/AZ31, (c) HA/AZ31, and (d) TA/HA/AZ31[96]

alloy showed good corrosion resistance biocompatibility and promoted cell growth.

Zeng et al.[97] deposited chemical conversion coatings on AZ31 Mg alloy. Coatings of zinc calcium phosphate were developed. Coated samples improved corrosion resistance. But the presence of micro cracks and voids affected the corrosion resistance.

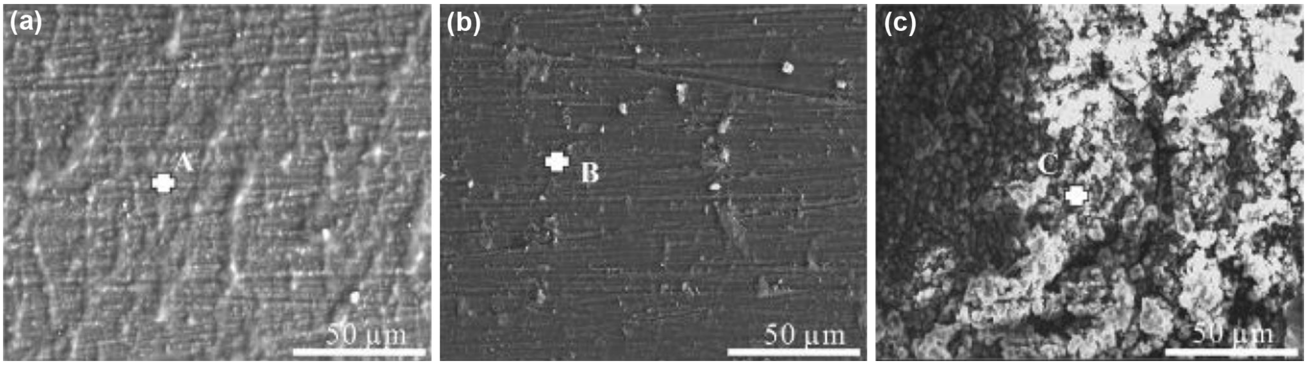


Fig. 10: SEM images of immersion test of $Mg(OH)_2$/PMTMS/ CeO_2-coated samples after: (a) 3 days, (b) 7 days, and (c) 14 days[103]

Liu et al.[98] deposited calcium-phosphate chemical conversion coatings on AZ91D Mg alloy. Coating consisted of $CaPO_4{\cdot}2H_2O$ protected from corrosion.

Hydrothermal treatment

Conversion coating by hydrothermal treatment is used to deposit magnesium hydroxide layers on the magnesium alloys surface.[99] By hydrothermal treatment, corrosion resistance of the substrate can be improved, and hydrogen evolution can be controlled.

Xu et al.[100] treated ZK60 Mg alloy hydrothermally with time and temperature variation. After the treatment, the formation of $Mg(OH)_2$ layers improved the corrosion resistance. A sample of 1.78 μm thickness of magnesium hydroxide film treated at 120°C for 24 h shows the best corrosion resistance.

Wang et al.[101] deposited a coating of $Mg(OH)_2$ on AZ31 substrates with hydrothermal treatment. The coating formed was dense and uniform, and coating thickness is directly proportional to time. But if treatment time became more than 6 h, coating quality deteriorated.

Peng et al.[102] deposited Mg–Al double hydroxide layer into $Mg(OH)_2$ coating on Mg-Nd-Zn-Zr alloy. The coating produced was corrosion-resistant and biocompatible. A compact film of $MgCO_3$ was generated if the exposure was for more than 3 h. The corrosion current density of the coated alloy was lesser than that of the bare alloy. Therefore, better corrosion resistance in the case of a coated alloy compared to the bare alloy.

Guo et al.[103] deposited a coating hydrothermally on AZ31 Mg alloy. The coated layer was sealed with polymethyltrimethoxysilane (PMTMS) and CeO_2. A 13-μm-thick coating was obtained in the process. The sealed surface did not allow Cl^- ions and H_2O molecules to get into the substrate. This had increased the corrosion resistance. From the polarization tests, i_{corr} for bare substrate was $1.51 \pm 0.08 \times 10^{-5} A{\cdot}cm^{-2}$. For coated substrates, i_{corr} was significantly reduced. SEM images of the substrate surfaces of $Mg(OH)_2$/ PMTMS/ CeO_2 were taken after the immersion for the periods of 3, 7, and 14 days (shown in Fig. 10). Microcracks were also sealed by coating.

Zhang et al.[104] used one-step hydrothermal to deposit a superhydrophobic coating on AZ31 Mg alloy. Corrosion tests of the coated and bare substrates were performed in NaCl solution. The coated alloy substrate had electric impedance 16 times more than bare alloy.

Cui et al.[105] deposited SnO_2-doped dicalcium phosphate using the hydrothermal treatment on AZ31. A thick coating of 40 μm was obtained. The surface smoothness of the coating increased by increasing the amount of SnO_2. The addition of SnO_2 increased corrosion resistance in the coated alloy.

Non-conversion coatings

In case of conversion coating, magnesium base metal is converted to get a coating of oxide or hydroxide of magnesium. While, in non-conversion coating, the coating material is not obtained from the base metal. Properties and composition of coating material may differ from base metal. Coating of calcium phosphates, predominantly hydroxyapatite, was done on the Mg alloy substrate. Both chemical and physical methods were used for the deposition of the coatings.

Physical deposition methods

Various methods of physical deposition have been used for the coating of magnesium alloys. These methods are physical vapor deposition, electrophoretic, magnetron sputtering, and cold gas dynamic spraying.

PHYSICAL VAPOR DEPOSITION: In physical vapor deposition, the material to be coated is first converted into vapor phase and then deposited onto the magnesium alloy substrate. This method is widely used for depositing thin coatings on magnesium alloys.[106, 107] A duplex coating of MAO/TiN[108] and MAO/Al[109] was applied on AZ31 using multi-arc ion

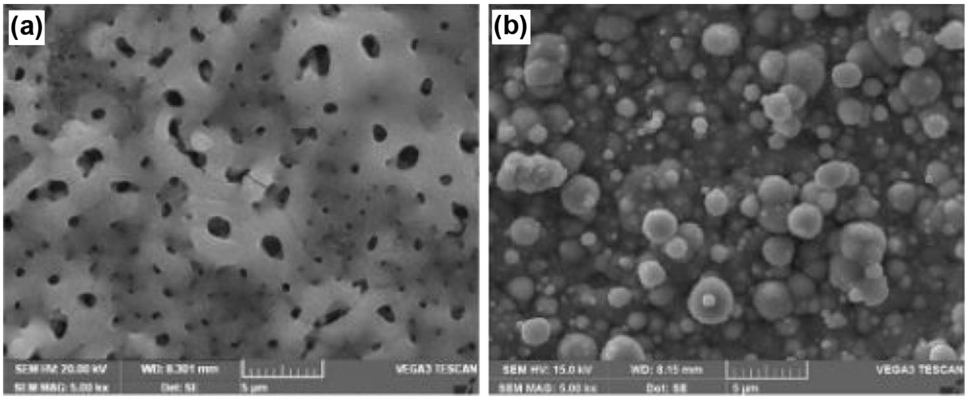


Fig. 11: SEM images of the surface of (a) MAO coated, (b) MAO/TiN coated[108]

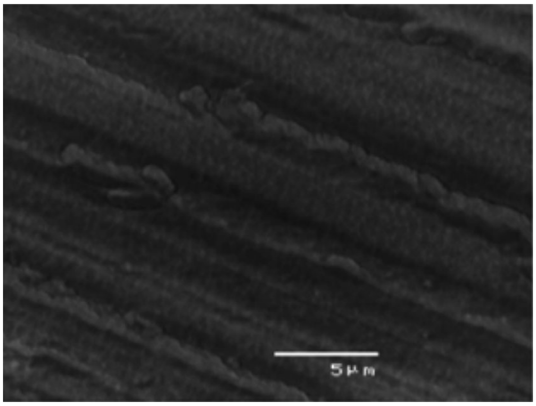


Fig. 12: Surface morphology of HA-coated Mg–Ca alloy[110]

plating. The SEM images of the coated surfaces are shown in Fig. 11. The coating films tend to seal the porous oxide layers and enhance the corrosion resistance properties of the alloy. However, droplets and cavities within the surface films may cause corrosion.

RADIO-FREQUENCY MAGNETRON SPUTTERING: Magnetron sputtering is a physical coating technique used for depositing thin films on the substrate by applying a magnetic field. It allows faster deposition at a lower pressure than other coating techniques.

Surmeneva et al.[110] had deposited an ultrathin layer of coating of HA on Mg-Ca alloy. Coating thickness was between 550 and 750 nm. SEM images of the coated substrate surface are shown in Fig. 12. The Tafel plots for uncoated and coated alloy are shown in Fig. 13. The corrosion current density, i_{corr} of the bare electrode, was 90 ± 14.1 $\mu A/cm^2$, while for coated alloy, i_{corr} was 1.8 ± 1.8 $\mu A/cm^2$. The addition of HA decreased corrosion current density by 98% and hence enhanced corrosion resistance significantly. In this study, researchers found that applying an ultrathin layer of HA on the substrate can cause a significant reduction in corrosion. This behavior can be utilized to develop long-lasting coatings on magnesium alloy-based implants.

Bita et al.[111] deposited hydroxyapatite (HA) and bio-glass (BG) on Mg-Ca alloy substrates by RF-MS and evaluated the adhesion of the coatings. The mean adhesion values obtained from adhesion pull-out tests for both HA and BG were higher than required (15 MPa).

A thin nanostructured HA was coated on AZ91 alloy.[112] SEM images uncoated and HA-coated alloy substrates are shown in Fig. 14. The coating was dense, smooth, and pore-free. Polarization tests were performed in which corrosion current density, i_{corr}, was obtained lower in the coated alloy (0.35 $\mu A/cm^2$) than the bare alloy (80.88 $\mu A/cm^2$). SEM images after electrochemical corrosion tests are shown in Fig. 15. Corrosion pits were found in large numbers in uncoated alloy substrates. Immersion tests were conducted in the SBF solution. During immersion tests in the SBF fluid, a bone-like apatite was formed.

Yoshimura[113] deposited a thin coating of palladium on magnesium substrates using RF-magnetron sputtering process. The deposited layer acts as hydrogen absorbent which further protected the surface from corrosion.

ELECTROPHORETIC DEPOSITION: Electrophoretic deposition is a versatile technique for magnesium-based alloy coatings. Various biomaterials like hydroxyapatite, polymeric material, calcium magnesium silicate, graphene, and carbon nanotubes have been used for coating using the EPD process.[114–116] This process has many advantages, including simple equipment, tight microstructural features, and orthopedic applications.

Manoj Kumar et al.[117] applied hydroxyapatite on Mg-3Zn alloy. From morphological studies, at low surface roughness and high annealing temperature, coatings formed were uniform and homogeneous with the minimum number of cracks. At 400°C, when surface roughness decreased, fracture toughness elastic modulus, elastic modulus, and microhardness of the coating increased by 73%, 26%, and 30%, respectively. The corrosion resistance of HA coating deposited by the EPD process increased about 25 times. Cell adhesion and viability also improved in HA-coated Mg alloys. SEM images of the coated surface after immersion tests are shown in Fig. 16.

Singh et al.[118] deposited HA/Fe_3O_4/CS composite coating on AZ91 alloy. The potentiodynamic polarization tests revealed that i_{corr} for uncoated AZ91 substrate was higher than other samples. HA-1Fe substrate had the lowest value of i_{corr} and thus had better corrosion resistance.

Cordero-Arias et al.[119] deposited nanostructured TiO_2/alginate coating on AZ91D alloy and studied the corrosion behavior of the samples. Corrosion potential for coated alloy substrate was more than bare alloy.

Amiri et al.[120] deposited nano-zirconia on AZ91D Mg alloy by electrophoretic deposition. The value of i_{corr} for uncoated alloy was 18.4 μA/cm^2, while for the coated substrate was 12 μA/cm^2. This showed an increase in corrosion resistance after coating.

Rojaee et al.[121] deposited MAO/BAG coating on AZ91 alloy. During immersion tests, apatite-like material was generated on the surface of coated alloy and thus promoting bioactivity. From the polarization curves obtained, i_{corr} for uncoated AZ91 substrate was 22.14 ± 3.214 μA/cm^2, for MAO-coated substrate 0.58 ± 0.010 μA/cm^2 and for MAO/BAG substrate was 0.02 ± 0.006 μA/cm^2. This showed improvement in corrosion resistance.

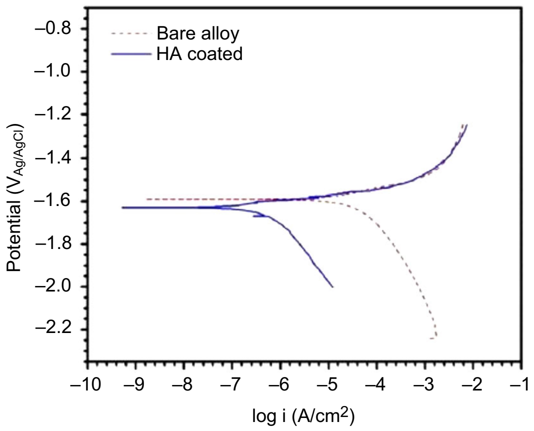


Fig. 13: Tafel plots for uncoated and HA-coated alloy[110]

Cold gas dynamic spraying: In this technique, coatings were produced by spraying gas at lower temperatures and applying high pressure to deposit the coating material particles on substrates. A CGDS coating was deposited to AZ51 Mg alloy to obtain a thickness of 20–30 μm.[122] Substrates were first preheated to 400°C. HA particles were then sprayed to get a coating thickness of 20–30 μm. The surface morphology of the HA-coated substrate is shown in Fig. 17. The surface morphologies during immersion tests for 4, 10, and 14 days are shown in Fig. 18. During immersion tests, coating started to dissolve initially but regenerated after 10 days. Degradation of HA coating caused the deposition of various calcium phosphate compounds. Thus, HA-coated samples reduced the degradation rates with the dissolution and re-precipitation of apatite.

Chemical deposition techniques

Chemical deposition methods produce coatings by exposing the substrate surface to one or more volatile precursors. These precursors react or decompose on the substrate surface to produce the desired deposit of the coating.

Pulsed electrodeposition: Pulsed electrodeposition is more advantageous as compared to conventional electrodeposition. In the conventional electrodeposition, hydrogen bubbles get accumulated at the cathode. Hydrogen evolution disturbs the precipitation of the coating. In the case of pulsed electrodeposition, the use of pulsed current, the current gap allows the hydrogen bubbles to get desorbed from the substrate surface. This allows the use of higher current densities. However, even using higher current densities, a stoichiometric HA layer is

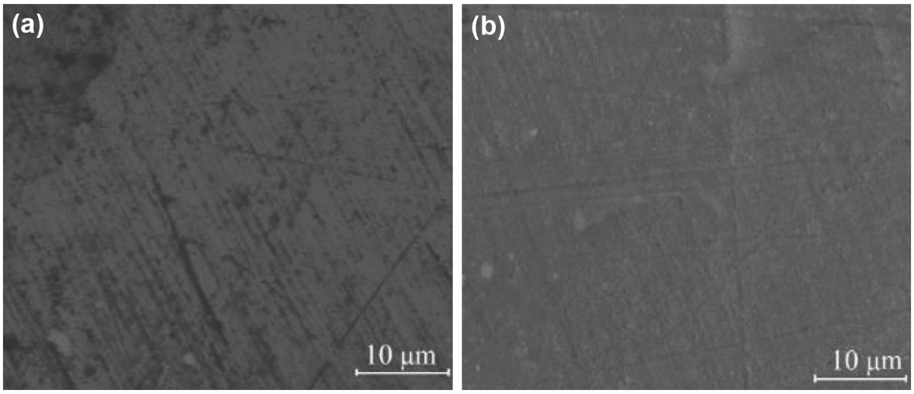


Fig. 14: SEM images of AZ91 alloy (a) bare substrate and (b) coated substrate[112]

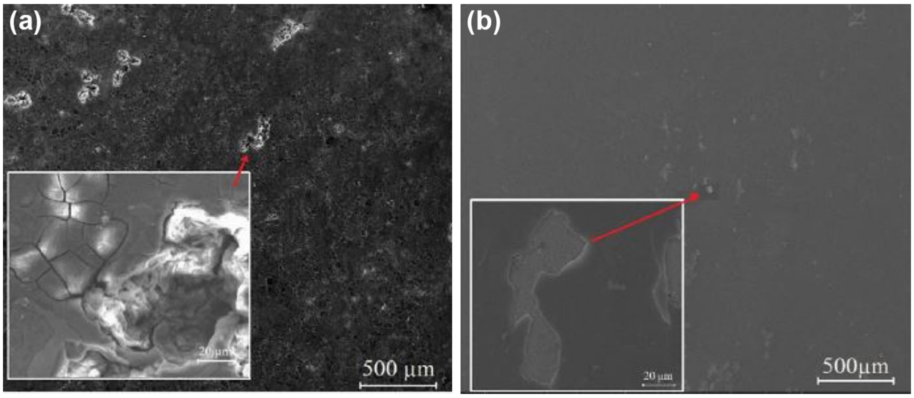


Fig. 15: SEM images after the corrosion test (a) bare alloy and (b) HA-coated alloy[112]

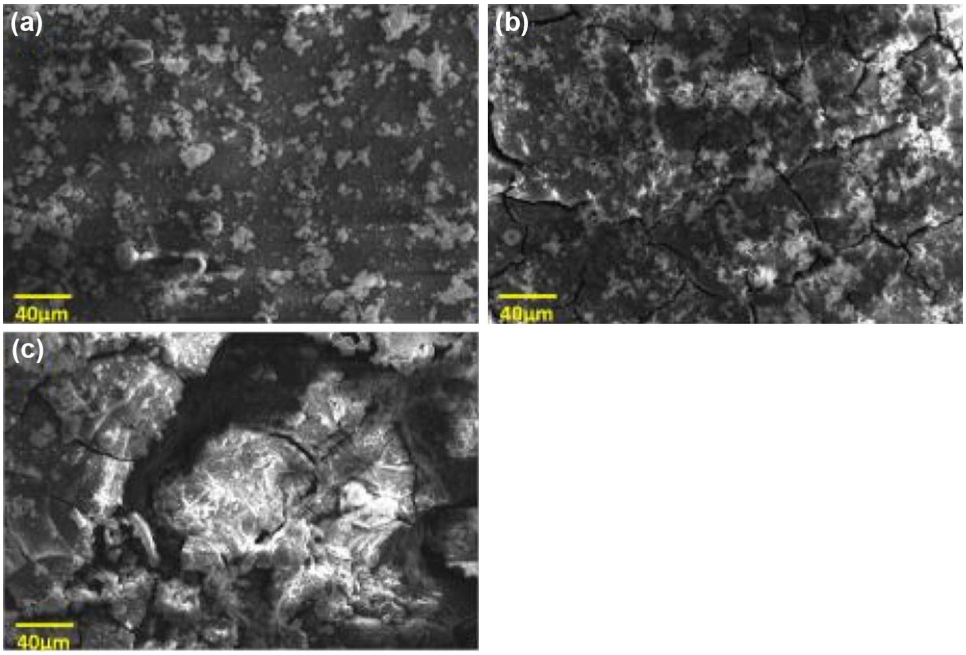


Fig. 16: SEM images of the surface of HA-coated Mg-3Zn substrate after immersion for (a) 3 days, (b) 7 days, (c) 14 days[117]

difficult to obtain.[123] Hence, hydrogen peroxide is used with the pulsed electrodeposition process to form HA.[124]

Wan et al.[125] investigated the effects of pulsed electrodeposition in Si-doped calcium phosphate on AZ31 alloy. Both time and temperature affected morphology, composition, and homogeneity of Ca–P coating. Denser coatings were produced with the time at the same deposition temperature. At a deposition temperature of 40°C and deposition time of 40 min, a compact and uniform coating was obtained with improved corrosion resistance. Shangguan et al.[126] studied the corrosion behavior of the inner corrosion layer formed on Mg–Sr alloy by pulsed electrodeposition. Figure 19 shows SEM images of the substrates. A corrosion layer was observed between the substrate and the outer surface of the PED coating. From corrosion test results, it was observed that the corro-

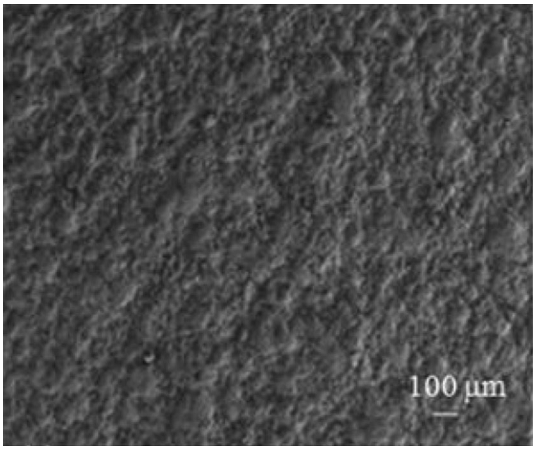


Fig. 17: Surface morphology of the HA-coated AZ51 substrate[122]

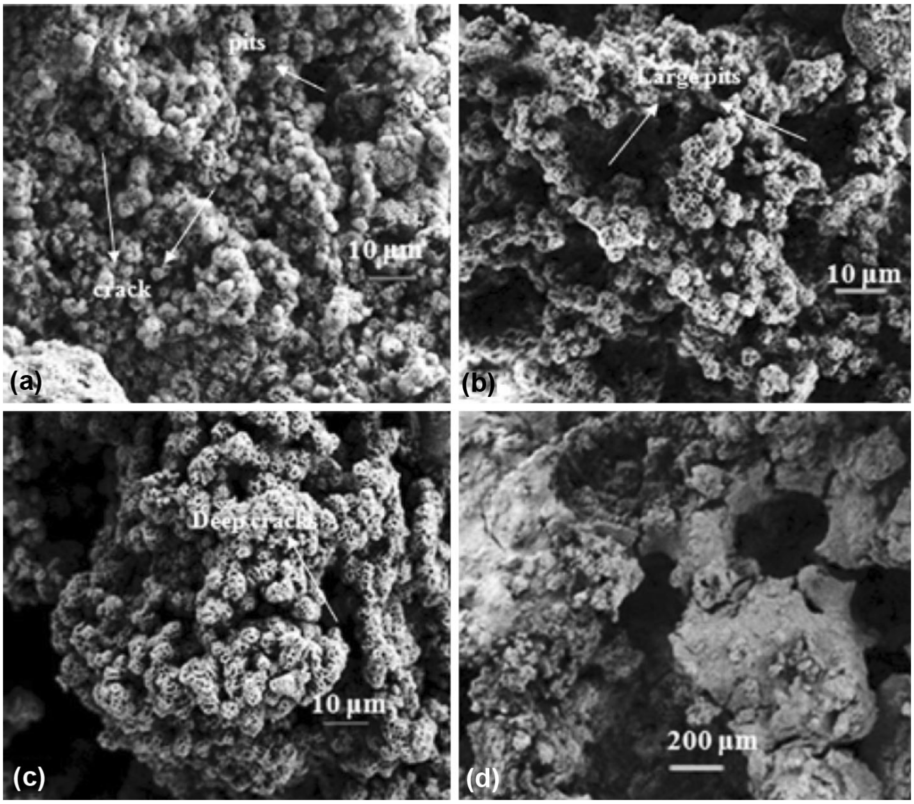


Fig. 18: Surface morphologies of the substrate during immersion tests in the SBF solution after (a) 1 day, (b) 4 days, (c) 10 days, and (d) 14 days[122]

sion resistance of the coated alloy was lower than the uncoated alloy. From this observation, it can be concluded that the pulsed electrodeposition process is not suitable for materials with poor corrosion resistance like the Mg–Sr alloy used in this study.

Heimann deposited a Si–HA layer on magnesium alloy.[127] The effect of the inclusion of Si in the HA structure was studied. Four different electrolytes were taken having different molar ratios of SiO_3^{2-}. The corrosion resistance of the coated samples prepared in 0.005 mol/L SiO_3^{2-} electrolytic solution was higher than undoped HA-coated alloy.

Fluorine-doped hydroxyapatite (FHA) and brushite were deposited on Ca-doped magnesium alloy.[128] The corrosion rate for bare alloy was 7.17 mm/year, for brushite-coated substrate was 0.14 mm/year and for FHA-coated substrate was 0.13 mm/year.

HYDROTHERMAL DEPOSITION: Kaabi Falahieh Asl et al.[129] deposited calcium phosphate on AZ31 alloy using hydrothermal deposition. The corrosion behavior and the biocompatibility of the coated alloy were studied. The deposited coating consisted of tricalcium phosphate and monetite. The corrosion protection of the coatings was dependent on deposition time and temperature. This had been confirmed by the corrosion studies. The mass loss in the simulated body fluid was higher in case of uncoated substrate. The corrosion current density of the coated magnesium decreased significantly.

Kaabi Falahieh Asl et al.[130] used one-step hydrothermal process to deposit composite coatings of calcium phosphate/polymer on AZ31 Mg alloy. The elastic modulus of the coating was found comparable to that of human bones and hereby reducing the stress shielding effects. This was due to the inclusion of polymer in the coatings. The cracking tendency was also reduced, which was revealed by nanoindentation tests. The corrosion resistance increased significantly, which is necessary for implant applications. Cell adhesion capability was also improved as per the cell proliferation studies.

Kavitha et al.[131] deposited strontium phosphate (Sr–P) coatings on magnesium by hydrothermal treatment process in the range of temperatures of 80–200°C. This process was performed in a short duration of 30 min. The effect of treatment temperature was significant on coatings. The surface obtained was homogenous and compact. As the treatment temperature was increased, parameters such as density and surface roughness also increased. The deposited coatings were extremely crystalline, with major constituents like magnesium phosphate and Sr phosphate and minor constituents like $Mg(OH)_2$ and $Sr(OH)_2$. The immersion tests were conducted in the simulated body fluid (SBF) solution

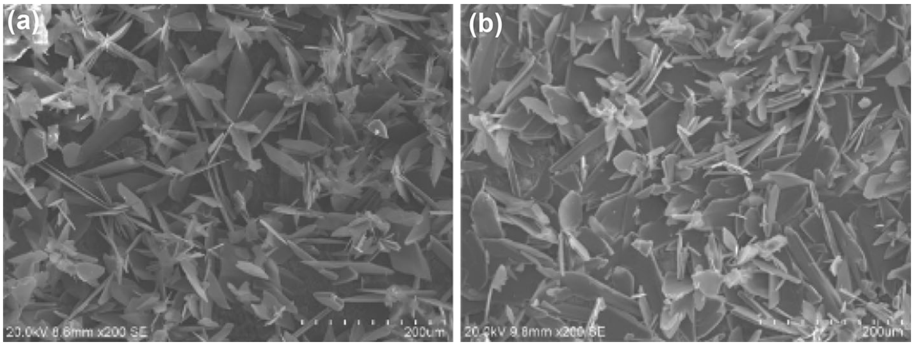


Fig. 19: SEM images of the surface of PEO coating on (a) as-cast Mg–Sr alloy and (b) as-extruded Mg–Sr alloy[126]

for 240 h. Polarization curves are shown in Fig. 20. The values of i_{corr} for uncoated alloy was 22.06 ± 2.7 μA/cm^2 and for Sr–P-coated alloy treated at 200°C was 0.985 ± 0.3 μA/cm^2. The coated magnesium was more corrosion resistant than the uncoated magnesium alloy in the SBF solution. During immersion, a bone like apatite was formed confirming its bioactivity.

Sol gel-based dip and spin coatings: The sol–gel process is a wet chemical method. It is commonly used to produce ceramic coatings by inorganic precursors or hydrolysis of organometallic compounds (alkoxides), producing a gel of hydrous oxides. A dense ceramic coating can be attained after the subsequent sintering.[127]

The dip-coating technique is similar to the sol–gel technique. A volatile medium is utilized in the dip-coating technique, which evaporates fast when the substrate is removed from the liquid coating medium.[132, 133] Dip coating is a faster than sol–gel technique. In dip coating, the liquid gel transforms to solid surface film in few minutes. It depends on rate of evaporation of the solvent.[134]

In spin coating, viscosity and spin velocity are the two main parameters. The thickness of the coating deposited decreases with the increase in spin velocity. Coating thickness also varies with time.[135] Sol–gel-based coatings were also generated using biopolymers.[136] Figure 21 shows the steps in sol–gel-based spin coatings.

Widati et al.[137] produced coatings of hydrophobic glass. They used silicon dioxide, titanium dioxide and methyltrimethoxysilane. Addition of titanium oxide improved surface roughness and hydrophobicity. Wang et al.[138] deposited silane/Mg(OH)$_2$ on AZ31. From the EIS and immersion tests, the coated samples were more corrosion resistant than uncoated samples. Coatings were also bioactive and promote cell growth.

Patil et al.[139] deposited n-decyltriethoxysilane and tetramethoxysilane. Coated samples were corrosion resistant and non-toxic. Coatings were bioactive and promotes cell growth. In cell culture, no calcium phosphate was observed on the surface.

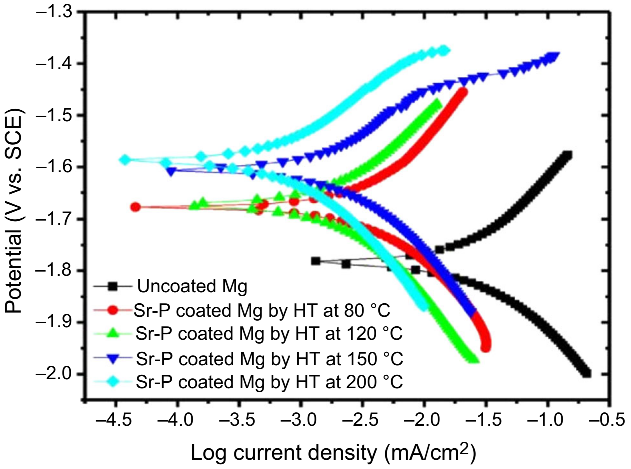


Fig. 20: Tafel curves of uncoated and Sr–P-coated Mg hydrothermally treated at different temperatures[131]

Ren et al.[140] deposited nanostructured magnesium phosphate/PLA coatings on AZ31. Coating thickness was 500 nm. SEM images of the substrates are shown in Fig. 22. Corrosion resistance of the samples was increased after coating. During the immersion in the SBF solution, apatite precipitation observed on coated sample surface. This showed higher biomineralization capacity indicating its potential to be used in orthopedic applications.

Córdoba et al.[141] developed a hybrid collagen/chitosan and TiO$_2$/silane coatings on AZ31 and ZE41 magnesium alloys. Due to biopolymers collagen and chitosan, CaCO$_3$ and MgCO$_3$ were produced as corrosion products. They provided corrosion protection to the magnesium substrate.

Upadhyay et al.[142] deposited silica-based sol–gel coatings on AZ31B. Coatings were doped with dopamine hydrochloride, diazolidinyl urea, quinaldic acid, and betaine.

Johnson et al.[143] deposited nanostructured hydroxyapatite(nHAp)/PLGA coatings to control magnesium-based alloy corrosion. Results of the characterization of the coated alloy substrate. Compared to uncoated Mg, the coated Mg increased the corrosion potential and decreased the corrosion current in the revised simulated body fluid. Deposition of calcium phosphate was observed on the surface of the

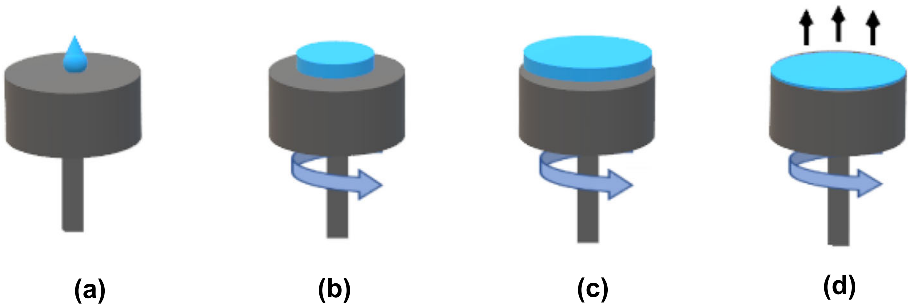


Fig. 21: Steps in spin coating process: (a) Deposition, (b) Spin up, (c) Spin off, and (d) Evaporation

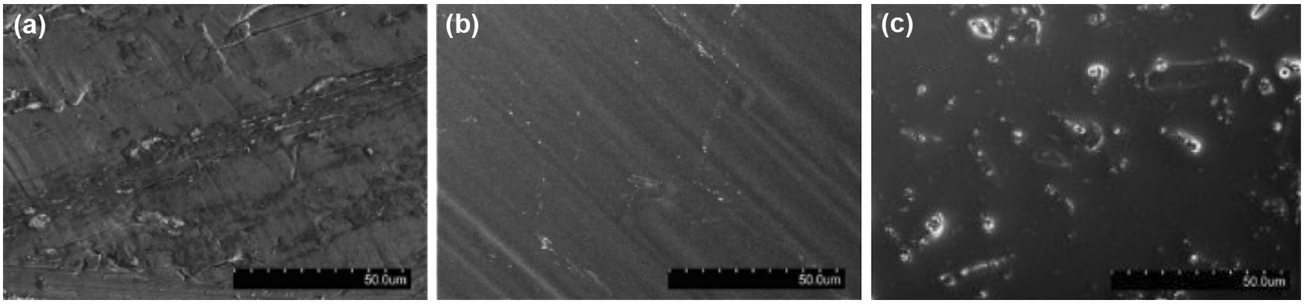


Fig. 22: SEM images of the surface of (a) uncoated AZ31, (b) PLA-coated AZ31, (c) nAMP/PLA-coated AZ31[140]

nHAp/ PLGA-coated alloy after 24 h. of immersion in Rsbf, ensuring its potential bioactivity. While in the case of only PGLA-coated Mg alloy, no significant amount of calcium phosphate was observed.

Conclusions and future scope

In this literature, various surface modification techniques of magnesium-based alloys have been discussed for implant applications. The selection of a coating method depends on the requirements of the implant for a particular application. The coated surface obtained by various coating techniques was corrosion-resistant and biocompatible.

The critical factors to consider before introducing biodegradable coatings are discussed below:

Surface morphology

The surface morphology of coatings has impacts on the corrosion behavior and cell adhesion.[144, 145] In most surface observations, surface topography was studied using SEM.[146] The surface morphology mainly depends upon the type of alloy, principal alloying elements, and the type of biomaterial used for coating. In most of the studies, the biomaterials used for coating had increased the corrosion resistance of the base material. The coating thickness obtained was in the range of micron to nanometer, depending on the type of process and application. Other parameters like surface roughness, porosity, and surface wear also affect the performance of the implant.

Corrosion rate

Corrosion studies were done in most of the reviewed papers. The corrosion rates were found by immersion tests, EIS tests, and Tafel plots. The composition, concentration, time, and other related parameters of the electrolyte or the SBF solution were varied. The effect of these variations on the corrosion resistance of the coatings was studied.

The most commonly used technique for determining corrosion rates was the potentiodynamic polarization method.[147] It is performed in the SBF or NaCl solution to find corrosion current density, i_{corr}, and corrosion potential E_{corr}.[148] Higher E_{corr} and lower i_{corr} will have better corrosion protection.[149]

Materials used for coating

Various organic or inorganic materials are used to deposit a coating on the substrate surface of the magnesium alloy.[150] Materials for coating magnesium alloys include calcium phosphates, titanium dioxide, and biopolymers like PLA, PLGA, polypropylene, etc. Various materials used for coating magnesium alloys reviewed in this paper are presented in Table 8.

Table 8: Various coating materials reviewed in this paper

Reference	Coating material	Substrate	Process
80	HA	AZ31	Plasma electrolytic oxidation
84	PEO/Silane	AZ31	Plasma electrolytic oxidation
85	MgO/ZrO_2	AZ31	Plasma electrolytic oxidation
103	$Mg(OH)_2/PMMTS/CeO_2$	AZ31	Hydrothermal treatment
105	$SnO_2/DCPA$	AZ31	Hydrothermal treatment
96	HA/tannic acid	AZ31	Chemical Conversion
109	PEO/TiN	AZ31B	Physical vapor deposition
110	HA	Mg-Ca alloy	Radio-frequency magnetron sputtering
112	HA	AZ91	Radio-frequency magnetron sputtering
117	HA	Mg-3Zn	Electrophoretic deposition
122	HA	AZ51	Cold dynamic gas spraying
130	Ca-P/polymer	AZ31	Hydrothermal
131	Sr-P	Commercially pure Mg	Hydrothermal
138	Si doped Ca-P	AZ31	Pulsed electrodeposition
140	Magnesium phosphate/PLA	AZ31	Spin coating

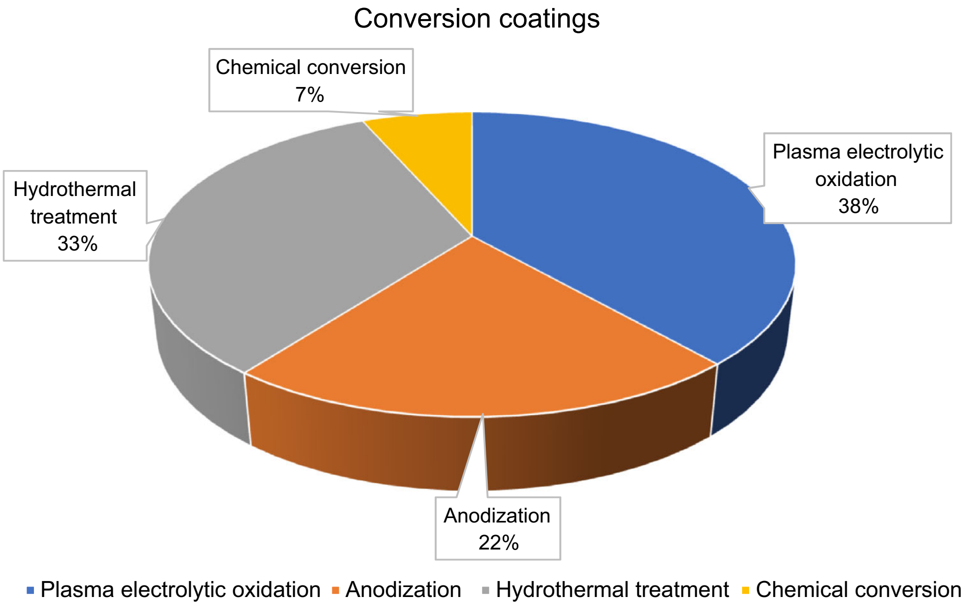


Fig. 23: Research articles reviewed in this paper of various conversion coating techniques

Mechanical behavior

The success of an implant material depends not only on its superior corrosion resistance but also on its mechanical behavior. Mechanical properties like microhardness, compressive strength, and fatigue strength affect the performance of an implant. Implants used in the hip[151] and knee[152] require high load-bearing capacity, and the implants used in dental applications require good fracture strength[153] and microhardness. By applying biomaterials coating on the substrate surface, mechanical properties had improved.

Type of method used for coating

The selection of a particular method of coating is also important. It depends upon various factors like the type of substrate material, the thickness of coating required, surface roughness, porosity, and others. The surface coating methods of magnesium-based alloys are mainly classified into two categories: conversion and non-conversion coatings. In Figs. 23 and 24, pie charts show the percentage of research articles related to conversion coating and non-conversion coating techniques, respectively (Table 9).

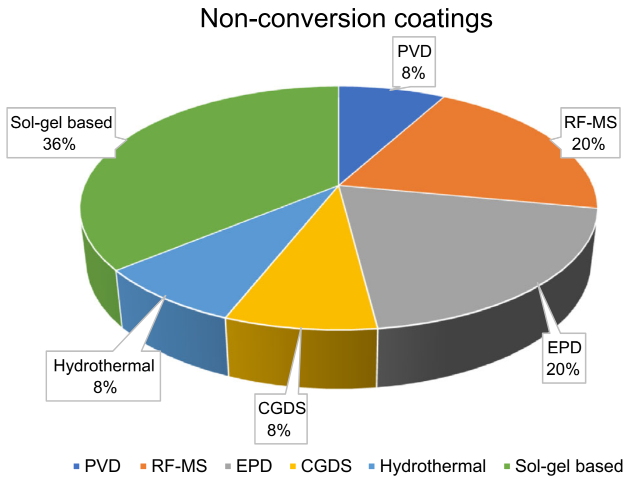


Fig. 24: Research articles reviewed in this paper of various non-conversion techniques

Although a lot of research has been done in the field of surface modification of Mg alloys for implant applications, there is still a lot of work to be done to modify the surface of the magnesium-based implant. Controlling the degradation rate without compromising the biocompatibility and bioactivity is necessary for a successful implant. This paper gives an overview of magnesium-based alloys in medical applications and compares their properties with other commonly used biomaterials. This comparison can help researchers to develop a basic understanding of material selection for implants. This paper gives an insight into surface coating techniques used for magnesium-based alloys and related works done previously by different researchers. The study of corrosion behavior and other properties of a specific coating process can help researchers in future studies of the process.

Table 9: Different coating processes, substrate materials, coating materials, related advantages, and disadvantages

Processes	Substrate material	Coating materials used	Advantages	Disadvantages
Plasma electrolytic oxidation	AZ31, AJ62, ZK30	HA, silane, MgO, ZrO_2, etc	Thick and dense coatings, greater control of porosity, corrosion resistance, biocompatible	High energy consuming, requires conductive material
Anodization	AZ91D, AZ31B	ZrO_2, etc	Low energy consuming, more control of coating thickness	Dense coating is difficult
Conversion coating by hydrothermal treatment	ZK60, AZ31, Mg-Nd-Zn-Zr	$Mg(OH)_2$, PMTMS, CeO_2, SnO_2, etc	Simple, easy and low-cost process	Reaction takes long time
Chemical Conversion	AZ31, pure Mg, AZ91D	HA, tannic acid, etc	Irregular shapes, porous structure, good adhesion, easy to operate, quick process	Low toughness and wear resistance
Physical vapor deposition	AZ31B	TiN, Al, etc	Thin film can be deposited, good corrosion and wear resistance	Requires vacuum
Radio-frequency magnetron sputtering	Mg-Ca, AZ91	HA, bioglass, palladium, etc	Better film quality and efficiency, works at low pressure, any type of material can be sputtered	Low deposition rates, complex set up, difficult to deposit on complex geometries
Electrophoretic deposition	Mg-3Zn, AZ91, AZ91D	HA, TiO_2, zirconia, etc	Porous structure, wear resistance	Requires conductive substrates
Cold gas dynamic spraying	AZ51	HA, etc	Simple and cheap	Limited use, low efficiency
Hydrothermal treatment	AZ31	Ca-P, Sr-P, polymer, $Mg(OH)_2$, etc	Good control of parameters, simple and easy process	Difficult to achieve uniformity
Pulsed		electrodeposition	AZ31, Mg-Sr alloy, Ca-doped Mg alloy	Ca-P, HA, brushite, etc
Higher current densities can be used, thick coatings possible	Costly process			

Table 9 Continued

Processes	Substrate material	Coating materials used	Advantages	Disadvantages
Sol–gel-based dip/spin coating	AZ31, WE43	Silane, $Mg(OH)_2$, collagen, chitosan, magnesium phosphate, TiO_2, PLA, etc	Cost effective, good adhesion, thick coating	Difficult control of thickness, slow process

Conflict of interest The authors have no conflicts of interest to declare. All authors have seen and agree with the contents of the manuscript and there is no financial interest to report. We certify that the submission is original work and is not under review at any other publication.

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