Schematic review of plasma arc oxidation process for Mg Alloy Bio Implants.

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Abstract. The biomaterials are having diverse applications in temporary implants. The implant performance and compatibility inside the human body element are enormous factors of research. The implant material requires excellent mechanical properties that can endure corrosion, higher stress, load-bearing capacity. The compatibility with local tissues of the human body is a crucial point of tolerance. This article is based on a review of papers in the area of Magnesium alloy bioimplants. The purpose of the review is to understand different methods and recent developments for Mg alloy material in the domain. Different methods for producing coatings and the effect of the addition of nanoparticles are discussed.

1. Introduction

The biomaterial plays a vital role in the implants. They either support or replace human tissue. Hence they should intend the same properties as that of bone and tissue. In most of the implants, the function desired is to give stability at the point of contact between the bones in the process of healing or replace the joints permanently. The metals are widely used for biomaterial as it can be configured with the same characteristics of bone by improving its material characterization. The review only focuses on Mg alloy and its compatibility to be used as a biomaterial.

The mg alloy has mechanical strength required for implants with the ability to control the corrosion rate by alloying with different elements. To improve the corrosion resistance of magnesium implants different surface modification methods of coatings are used. The important aspect of bio implant is an antibacterial surface treatment and different methods to produce such surface coatings. The coating is nothing but a surface modification technique ether by improving properties if the existing one or applying different material on the base metal.
Figure 1 The gradual decrease in stiffness of biodegradable magnesium implant and concurrent healing zone [1]

Table 1 shows different materials used for bio-implants as per various locations in the human body. Apart from metals Polymers are also widely used for implants however they have some limitations like post-implantation recoiling which is difficult in the case of cardiovascular implants. Table shows different methods of producing coatings on the implants.

| Material     | Application                        | Reference |
|--------------|------------------------------------|-----------|
| Ti alloy     | Used for producing pins, bolts, screws | [1]       |
| Mg alloys    | Orthopaedic implants               | [2]       |
| Stainless steel | Long term/ Permanent implants     | [3]       |
| Co–Cr alloy  | Total joint replacement            | [4]       |

2. Required Material properties for orthopedic implants

The requirement of the orthopedic implant is high stability considering various forces inside the body. As the implant has to sustain at least up to the healing process, the biocompatibility is required to be higher. Hence the tissue friendly biomaterials are preferred for implants. However, these implants face degradation due to a longer presence in the bone and due to tissue environment. The characteristics desired are mechanical properties similar to bone, biocompatibility with surrounding tissues, uniform degradation behavior.

3. Alloying elements in implants

The addition of Copper enhances corrosion rate when used in NaCl environment. Still, the concentration of copper needs to be limited as the excess amount can cause deleterious biological effects such as neurodegenerative disease and specially cytotoxicity. It is added to improve the corrosion resistance of the implant. The Mn is preferred because of its essential bio element in the human body. It plays many roles within a cellular system such as different co-factors for many metalloenzymes. On the other hand excess use of the Mn leads to a neurological disorder like Almizer. Aluminum is considered as the material with good mechanical properties and corrosion resistance. Due to low weight and passivating element, it's known with good corrosion resistance. On the demerit side, the use is limited since in high doses it causes neurotoxicity which alters functioning due to the brain-blood barrier. The addition of Zinc increases yield strength in the base material. Magnesium alloys are preferred as implants for high Young’s modulus 3-20 GPa which is similar to
Young’s modulus of Bone 20 Gpa. When Mg combined with Zinc it works effectively against the hydrogen gas evaluation. Study shows that the Zn rich Mg alloy rarely form hydrogen gas during simulation in body fluids[8]. It is less detrimental than other elements such as Mn, Al due to high absorbing ability in human body cell. On contrast the higher amount of Zn can damage to stomach lining cell[9]

Table 2 Different metals used in orthopedic implants using various techniques of the coating

| Material      | Technique for processing | Properties                                      | Reference |
|---------------|--------------------------|-------------------------------------------------|-----------|
| Ti Alloy      | Anodizing                | High corrosion resistance                       | [1]       |
| Stainless steel | Phase reversion         | Superior corrosion resistance for wear and corrosion resistance for long stability of the implants | [5] |
| Co Cr Mn alloy | Heat treatment           | High wear and corrosion resistance, biocompatibility | [6] |
| HA            | Sol gel synthesis        | Improved densification and biocompatibility      | [6] |
| CaP           | Sol gel combustion method| Good viability, integration with enamel          | [7] |

4. Review of literature

The research is carried on the different classical biomaterials as well as non-metallic Nano phase materials. The study is mainly carried out on different base metals, different types of implants, different coating methods, surface characterization, additives and suspended particles and addition of nano particles. Researchers studied the effect of the surrounding medium like aggressive ion, chloride, phosphate using Potentiometric polarization, impedance spectroscopy for various characteristics. From results, it has been proved that chloride ions can induce pitting corrosion. The presence of phosphate reduces corrosion. Hydrogen carbonate ions increase the rate of corrosion but make the surface passivation[10]. Effect of the hydrothermal treatment with a different time in hours on MAO coated Mg alloy studied by the author [11]. Due to the hydrothermal treatment in Ca-EDTA solution and MAO coating, corrosion of the Mg alloy reduces boosting the healing process. Due to micro-arc oxidation, the layer of the magnesium alloy forms over the surface. This layer reacts with body fluid, prevents the peeling off due to the implant process. Hence the process is beneficial for orthopedic implants.

Song et. Al. [12] studied the compatibility of the Mg-Zn-Y-Nd-Zr alloys in a bone implant. Different in vitro tests were carried out to find the effect of the alloying on the implant. As the main objective is to find the cyclocompatibility of the implant, the samples were tested for cytotoxicity for degradation in implanting. The metallic irons released by alloy promote the ostogenetic differentiation. As the calcium
content in the body can be improved with the help of the degradable implant material along with the corrosion-resistant material. Hence achieving biodegradable material with a positive effect in the area where the implant is placed can be made. The material of the implant is taken as Mg-0.5Ca-xY, with a variation of Y content, which results in better compatibility, refining of microstructure, and reduces the corrosion. SEM images show that the improvement in the microstructure is due to the cubic structure of the Yttrium in Mg alloy. The alloy of Mg, Ca, and Y formed using the powder metallurgy by heating all elements 710-720°C. The researcher only discusses the formation of the alloying of Y in different compositions and improvement in structures with the help of SEM images [13]. The biocompatible material can also be enhanced by the plasma electrolyte oxidation method, where the coating formed on Mg alloy is corrosion resistant. The author discussed the experimental evaluations of such type e of coated material for in vivo testing results. As compared to uncoated material, the micro-arc oxidation coating optimizes the degradation rate and can be used as a safe biomaterial [15]. Few Mg alloys enhancement can be done by adding nanoparticles like Graphene. The research work shows that adding Graphene strengthens the alloy and reduces the wear rate. These types of implant materials can be used for the longer sustaining requirement of the part inside the body. Also, it helped to reduce the degradation of the implant material [16].

Most of the bio-implants formed using the powder metallurgy with heat treatment. The Mg alloy, Mg-Nd-Zn-Zr, developed by the heat treatment and then immersed in NaCl solution for a longer time. The effect of the soaking for the different periods discussed for the corrosion behavior of the alloy metals. The material formed with LSPO (Long stacking period ordered) structure shows a slower corrosion rate [17]. The alloy AZ31 and AZ91 of Mg was formed by a micro-arc oxidation method to study the coatings for anti-corrosive properties. From experimental results, AZ91 Mg alloy found to be more resistant to corrosion as compared to AZ31. The researcher also concluded that once the corrosion started on AZ 91 material, it spreads faster due to differences in morphologies of the two alloys [18]. Gao et al. worked on the addition of Graphene oxide coatings for the bio implant material. The tests taken in body fluid shows that GO (Graphene oxide) shows considerable improvement in the corrosion resistance when along with hydroxyapatite (HA). As per experiments conducted, this has been found that hybrid coating of the GO forms nucleation and crystallization of HA, creating a uniform coating [19].

The coating of the Graphene oxide layer on Ti alloy was studied by researched to get the enhancement of the properties of the Ti alloy. From experimental results, it has been found that Graphene shows considerable improvement in the corrosion resistance [1]. The idea of bio absorbable stents were also proposed in 2006. Due to permanent joint the patient has to face the in stent restenosis. It causes due to reaction between the blood and stent surface and irritation of vascular tissue. These stents are first step towards the biodegradable temporary stents. The material get dissolved in long term. The experimental details of the Mg stents are mentioned by author [20]. The composition of the Mg alloy as biomaterial is studied by adding the alloying element such as Mg-Gd-Y, Mg-Gd-Zn. These materials were tested in saline environment in cast state. Due to alloying of these rare earth element the biomaterial shows uniform corrosion resistance and good corrosion resistance in case of Mg –Gd alloy [21].

Further to improve the orthopedic implants the addition of alloying element Y is studied. The material system of Mg-0.5Ca-xY were studied for different mechanical testing like indentation micro hardness, micro scratch and stiffness. Due to alloying the friction coefficient get reduced resulting in improvement in the mechanical properties. The researcher also proposed further scope of experimentation by heat treatment of the implant [2]. The orthopaedic biomaterial FeMnSi were studied for electrochemical behaviour of the coating of HA-ZrO₂ by pulsed laser deposition technique. The SEM testing was also carried out to find out the morphological properties. The SEM shows degradation of material during the polarization test. With heat treatment the phase change of the
alloying material was achieved to improve the characterization of the biomaterial like Mg-2Zn-0.5Nd-0.5Zr alloy. The increase in the solution temperature up to 500 °C changes the phase increase grain size 4-5μm. Due to homogeneous material structure the material shows uniform degradation behaviour [22]. Furthermore, the study on the addition of Gd in Mg alloy was done by Yao et.al. by varying percentage content of Gd in Mg-2 Zn-0.5 Zr-xGd for the study of microstructure and biocorrosion behavior. Gd is proven to be material adding good corrosion resistance. Hence the different proportion of material was tested for improvement in mechanical properties and anticorrosive performance. A particular weight percentage the nanoscale rod-like phases of (Mg Zn) 3Gd found that give better ultimate strength and yield strength. Also, the significant corrosion resistance with the uniform rate of corrosion was observed [14].

The addition of nanomaterial graphene on Ti alloy is also considered as the potential area. Instead of adding Graphene particle in suspended form Asgar et al. studied the effect of the formation of the graphene oxide layer on the base metal. The material characterization is analyzed using IR, X ray diffraction. It was observed that at a temperature around 200 OC, the reduction in GO coating occurs. This showed good corrosion resistance on potentiometer at the reduced contact angle[1]. Adhesion of coating is also an important factor in coating deposition. An increase in adhesion is effective in the chemical bonding and biodegradation behavior of the implant. The process of improvement in adhesion is called as surface activation. This is achieved by pretreatment methods. In the case of magnesium the increase in surface adhesion results in a reduction of the release of Mg ions. Surface treatment of CoCrMo also results in the release of harmful ions to the body in vivo testing. For Titanium alloy the surface treatment method improves the formation of the oxide layer, adhesion, and bonding of hydroxyapatite coating [8].

The Mg oxides are stronger ions formed during the formation of coating which enhances the mechanical properties. To increase its effect the metal phenolic networks are formed by deposition of nano-sized metal oxides using a chemical reaction between the metal substrate and phenolic ions. The researcher studied the formation of magnesium oxide films on Mg based substrate using, Mg phenolic network. The results showed that the concentration of Mg alloy is three times higher than the uncoated samples. In vitro tests showed that osteocompatibility significantly changes due to the increase in the Mg ions in the coating. The coating also reduces the formation of gas cavities around the implantation area increasing biocompatibility of the material [23].

The synthesis of the double-layered fluorine-doped hydroxyapatite or polycaprolactone coating on Mg-2Zn-3Ce was done by electro deposition and a dip-coating method. The result showed the uniform layer of nanostructured. This composite coating gives significant protection for Mg against corrosion. The immersion test carried out in body fluid for 10 days showed higher compressive strength improving the degradation resistance in body fluid. The bond strength also improved [24]. The addition of nanoparticles in the alloying base material requires consideration of the success of implant, antibacterial activity, cell adhesion. Hence the nanomaterial design should be cell adhesive and wettability. The existing implants are costly and require a special facility to produce the material eg. 3D printing. Also, the implants’ life can be improved along with reducing the pain and inflammation at the location in the body. The production of smart material also in the stage of development[25]. Different review of the Mg alloy as the orthopaedic implants are done by researchers to identify potential area on improvement for the Mg alloy[17], [26]–[28]. Researchers developed different experimental and numerical methods of improving characterization and surface topology [29],[30]. With experimentation the efforts were also made to conduct the finite element analysis of implants in body fluid. This helps to determine the various stresses implant has to withstand and loading conditions [31]. Few researchers also worked on the method of producing coatings by plasma arc electrolytic oxidation on Mg alloy[32]. The different parameters like electrolyte compositions,
different input requirement of current, voltage and power supply. Also depending upon the composition of the electrolyte used and additives in different composition, the material characterization changes.[11], [18], [32]–[34]. The oxidation coatings were developed by researchers to optimize characteristics of the surface modification.[35][36]. These coatings were analyzed using SEM analysis, XRD analysis to identify the distribution of alloying element and uniform coating distribution.[36],[37],[11], [33], [38]. Another type of biological testing is immersion testing in which samples are immersed in solution similar as body fluid.[39] This helps to study the effect of the environment similar to body fluid on coating degradation. As the implant has to withstand up to healing of the joint it should not corrode easily up to specific time of healing. This gives the corrosion resistance of the material[40]. As study conducted by researchers for AZ31 mg alloy [33], AZ91D [30], MG-6Zn-Ca[38] which shows improvement in degradation behaviour due to alloying. The additives were added in base Mg alloy to strengthen the mechanical characteristics. The material like graphene was tested on Mg alloy as it forms the oxide layer on the coating as it promotes crystallization and nuclear growth of layer. Due to the mineralization of hydroxyapatite in modified SBF forms the dense structure of HA/GO hybrid coating. This promotes the corrosion resistance in the m- SBF[43].

5. Conclusion-

As per the literature review on the use of nanoparticles in the producing biomaterials, the few nanoparticles show significant improvement in the mechanical properties. The use of nanomaterial is increasing for the biomaterial due to properties enhancement. Different methods of producing coating also help to improve the properties regarding the application of biomaterial as per application location. Still the use of nanoparticles needs to be analyzed further to investigate the effect on the tissues of the body. When the implant is exposed to the body for a longer period, the nanoparticle has to be biocompatible enough to remain unaffected on tissues and the surrounding muscles of the body. Hence work can be done in finding out the biocompatibility of the materials like fullerene and graphene with alloys of Mg and Ti. Along with this, the material can also be tested for producing an oxide layer on the base material for improvement in corrosion resistance, wear-resistance, and other mechanical properties. To define the optimum value of the coating layer the parameters like electrode type, the distance between anode and cathode, nanoparticles, electrolytes, current and voltage requirements can be configured for a different set of combinations. This may help in producing a better oxide layer.

The main advantage of the magnesium and its alloy, if employed as an orthopedic implant, is its biocompatibility and the biodegradability properties, which ultimately can be used to avoid the need for a second surgery for the affected patients after implantation. But, the main drawback of these alloys is the rapid and rigorous corrosion, which is an inherent property of the magnesium and its alloys when they are in contact with the fluids with chloride content [16]. The human body fluids, such as blood plasma, consists of chlorides in it [17]. The magnesium orthopedic implants, when getting in contact with the bloodstream, they react with the chlorides and result in an alkaline environment and hydrogen liberation around the region. This will ultimately result in delayed healing of the tissues around the region and may lead to poisoning of the tissues also [18]. Concerning the mechanical integrity of the orthopedic implants, the corrosion also is responsible for the deterioration of mechanical properties of the implant leading to fracture and failure of the same before the complete healing has been achieved. Also, a comparison of Ti and Mg alloy at optimum configuration can be identified before in vivo testing of biomaterial

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