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Effect of high repetition laser shock peening on biocompatibility and corrosion resistance of magnesium

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ABSTRACT

Magnesium, as a biomaterial has the potential to replace conventional implant materials owing to its numerous advantages. However, high corrosion rate is a major obstacle that has to be addressed for its implementation as implants. This study aims to evaluate the feasibility and effects of High Repetition Laser Shock Peening (HRLSP) on biocompatibility and corrosion resistance of Mg samples and as well as to analyze the effect of operational parameters such as peening with overlap on corrosion rate. From the results obtained using hydrogen evolution and mass loss methods, it was found that corrosion rates of both 0% overlap and 66% overlap peened samples reduced by more than 50% compared to that of unpeened sample and sample peened with 66% overlap exhibited least corrosion. The biocompatibility of peened Mg samples was also enhanced as there was neither rapid pH variation nor large hydrogen bubble formation around samples.

1. Introduction

Implants made of magnesium are considered for orthopedic applications due to its biocompatible nature and also mechanical properties of Mg are quite similar to that of human bone [1]. The use of conventional implants made of stainless steel, titanium and cobalt-chromium result in stress shielding effect, which retards the stimulation of new bone. Further, interaction of human bone with the permanent implant causes lot of complications due to the mismatch in the mechanical properties [2]. Huafang Li et.al stressed the advantage of using biodegradable magnesium implants as they would eliminate the need for another surgery required for removing the implant from the body after bone healing [3]. Also magnesium is essential for human metabolism and any excess Mg present in human body can be harmlessly excreted through urine [4]. Vormann had elucidated the influence of magnesium on body metabolisms and also stressed the importance of magnesium intake [5].

Despite having such advantages, the major obstacle is the corrosion rate of magnesium that hinders its use as implant material. Magnesium implants in the human body tend to corrode at a rapid rate [6]. This rapid corrosion of magnesium results in large amount of hydrogen generation causing complications. The evolved hydrogen forms gas pockets around the implant there by causing unfavorable conditions for the bone growth [7]. Many alternatives such as usage of FDA approved biodegradable polymers [8], alloying, coating with Hydroxyapatite (HA) [9], phosphate [10] and mechanical treatments such as burnishing [11] have been suggested. Zheng et al. mentioned that there was no proper control over degradation of polymers [12]. The drawback in the above mentioned processes is that they do not provide control over the parameters that determines the corrosion rate and hence predicting corrosion behavior becomes difficult. However, recent advancements have identified laser shock peening (LSP) as potential solution to overcome this limitation. Laser peening on ANSI 304 stainless steel resulted in better resistance to stress corrosion cracking [13] and pitting corrosion [14]. Also its corrosion rate varied when peened with different parameters [15]. Such behavior suggests that accurate control along with desired corrosion rate can be achieved when peened with appropriate parameters. In addition, its success in enhancing material performance in aerospace applications has resulted in extension of implementing laser shock peening in medical field [16,17].

LSP is a surface treatment technique that uses a high intensity laser beam to cause plastic deformation on the surface of the specimen. This results in inducing compressive residual stress (CRS) in the specimen, which is the major factor influencing the corrosion rate [18]. Also, using LSP the CRS can be distributed more uniformly across the target material surface [19]. The magnitude of CRS induced in the implant can be precisely controlled by controlling the laser power. Hence depending upon the nature and the seriousness of the injury the magnitude of CRS to be induced can be varied. But due to its expensive nature, the process was confined only to major industries.

Recently a method called high repetition rate laser shock peening (HRLSP) was developed by the authors of this paper with a view to

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make the process more economical, fast and efficient. Using this process, peening can be carried out with comparatively low energy lasers. Due to lower peak power of pulses, the number of pulses striking the target material should be significantly high in order to generate same kind of effect on the surface. Another main advantage in using HRLSP is that the compressive residual stress (CRS) is formed closer to the surface, which gives controlled corrosion rate from the start [20].

Guo et al. mentioned that laser shock peening reduced the corrosion rate of magnesium alloy by at least 100× and this varied considerably with change in overlap ratio [21]. Apart from the corrosion rate, pH distribution and pH shift caused by the corroding material should also be measured to determine its biocompatibility [22]. The most common in vitro experiments implemented to determine the corrosion rate of magnesium and its alloys are mass loss method, hydrogen evolution measurement and electro chemical experiments such as polarization resistance and tafel plot [23]. Over the years, these methods have been widely employed to understand and study the corrosion behavior of magnesium based implants. However, Song et al. stressed that estimating corrosion rate of magnesium from tafel plot alone would be misleading due to its corrosion mechanism and suggested that hydrogen evolution measurement and mass loss method to be more reliable for determination of corrosion rate [24]. In this paper hydrogen evolution measurement and mass loss method were implemented to determine and compare the corrosion rates of unpeened and high repetition laser shock peened magnesium.

2. Materials and methods

2.1. Materials and properties

Peening was done on cylindrical samples of dimension 34 mm in diameter and 5 mm in thickness. These samples were cut from a commercial purity Mg rod having a composition of 99.8% Mg and 0.2% Ca. Hank's balanced salt solution (HBSS) from Sigma Aldrich was used as the testing medium for corrosion experiments. The composition of the HBSS is indicated in Table 1. Hanks solution was chosen as the corrosion medium because of its composition similar to blood plasma.

2.2. Sample preparation

Prior to peening, the cut samples were ground to 1200 grit using SiC paper. During the grinding process ethanol was used as the coolant and after each step the sample was washed with DI water and dried using high pressure air. The ground samples were then ultrasonically cleaned using acetone and deionised water and finally dried using pressurized air. The peening was confined to an area of 10 mm×10 mm and hence the remaining material was cut in order to expose only the peened surface for the corrosion experiments. The specimen dimensions used throughout the corrosion experiments are 10 mm×10 mm×5 mm with sides of the specimen coated with epoxy resin.

2.3. High repetition rate laser shock peening (HRLSP)

High repetition rate laser shock peening was performed using PRISMA TM 1064-16-V diode pumped, solid-state laser with frequency 10 kHz and pulse duration of 14 ns on Mg alloy. The peening was confined to an area of 1 cm^2 on a 34 mm diameter cylindrical Mg specimen and the laser spot size used for peening was 40 µm. For peening to occur, the shock pressure generated by each pulse should exceed the Hugoniot elastic limit (HEL) of the material [25], indicated by Eq. (1).

$$HEL=\frac{(1-\upsilon)\sigma_y^{Dyn}}{1-2\upsilon} \tag{1}$$

where σ_y^{Dyn} is the dynamic yield stress, υ is the Poisson's ratio. Considering the above said requirement the peening was processed with laser power of 2.5 W and its corresponding laser power density was 1.4 GW/cm^2 which is well above the Hugoniot elastic limit (HEL) of Mg alloy. Once the resulting pressure due to the laser pulse exceeds the dynamic yield stress, plastic deformation of the material occurs resulting in micro-dent formation. The shock pressure produces very high strain rates and under such conditions the material behaves much different than under static conditions, exhibiting increased yield strength. Hence for plastic deformation to occur, the resulting pressure should be greater than dynamic yield stress. The experiments have been conducted without any protective coating on the target material and quartz as the confining medium. Quartz has been chosen as the confining medium because of its high impedance compared to Mg alloy. Using a confining medium with higher impedance results in enhancement of shock pressure. A detailed explanation regarding HRLSP is given in the earlier research [20]. Fig. 1 shows the experimental setup used to perform HRLSP.

2.4. Scanning system

In HRLSP, due to low energy of pulses the spot size is reduced in the order of microns. As a result the frequency of pulses is increased by 1000 times. Hence a high speed scanning system is vital to perform HRLSP and also to avoid multiple pulses striking at the same place which may lead to material damage or melting.

The high speed scanning system used in this research is THORLABS GVS002 galvo-scanners. This arrangement consists of two galvoscanner mirrors (X and Y axis) attached to DC servo motors and are placed at the focal length of the lens. A NI6211 DAQ was used to control the motors. The position of the motors can be adjusted as a function of voltage which inturn set the galvo-scanners at a proportional angle. The operational range of the galvoscanner is between −12° and +12° and the voltage is between −10 and +10. A lab view program was developed to control the scanning system. The required scanning speed for peening can be obtained using Eq. (2).

$$S_s=D_S*\left[1+\left(1-O_P\right)\cdot\left(f-1\right)\right] \tag{2}$$

The speed of scanning (S_s) is influenced by laser spot size (D_s), percentage of overlap (O_p) and the frequency (f). The corrosion experiments were carried out on samples peened with 0% overlap (no overlap) and 66% overlap as shown in Fig. 2. The objective of this research is to validate the effectiveness of HRLSP by determining the corrosion rate of peened magnesium samples and also to understand the effect of process parameters such as overlap on corrosion rate.

2.5. pH variation and mass loss method

The prepared samples were dried in desiccator for a day and then weighed. Prior to the start of the experiment the sides of the specimen were covered with Teflon tape to ensure only the top surface (area 1 cm^2) is exposed to the hank's solution. The samples were then

Table 1
Chemical composition of Hank's solution in g/L.

Composition	NaCl	KCl	$MgSO_4$ (anhyd.)	$CaCl_2\cdot 2H_2O$	Na_2HPO_4	KH_2PO_4	$NaHCO_3$	D-Glucose
Hank's solution	8	0.4	0.09767	0.185	0.04788	0.06	0.35	1.0