Direct laser deposition as a method of biodegradable magnesium implant manufacturing

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Abstract. Magnesium can be used as a new biodegradable material for orthopaedic applications due to its outstanding properties including high biocompatibility, proper mechanical stiffness and biodegradability. Direct laser deposition (DLD) as a way of biomedical implant production has been used to make bulk pure Mg. Surface morphology, microstructure and mechanical properties of DLD sample have been studied. The relative porosity of DLD Mg sample has been determined.

1. Introduction

Nowadays, metals such as titanium, stainless steel, chromium–cobalt and its alloys are widely used as materials for orthopaedic applications [1]. At the same time, these implant materials have some shortcomings: difference in mechanical properties (microhardness, elastic modulus) between these biomaterials and natural bones can sufficiently damage the bone tissue [2]; the release of toxic elements may leads to the negative effect such as allergy or even cancer [3]. Currently, Mg has been proposed as a promising biomaterial, which possesses a good biocompatibility [4] and appropriate mechanical performance similar to the natural bone as compared to other metallic implant [5]. In addition, magnesium can continuously degrade in situ, which eliminates the necessity of secondary surgery as a result of Mg implant resorption in the human body [6].

Direct laser deposition (DLD) can be used to obtain implant materials. Polymers, ceramics, metals and their composites can be processed by DLD method. The DLD allows the layers of a powder to be successively melted by means of a laser beam heat. Each melted layer connects with the previous one until the final sample is formed.

In the field of personalized medicine, the magnesium based materials are very attractive to be used in the process of implant formation by means of such additive technique as DLD [7].

At the same time, Mg has some shortcomings such as high corrosion activity in the saline solutions, which leads to its too fast degradation [8]. Thereby, many scientific groups try to obtain new Mg alloys as well as to develop a new ways for treating and producing the personalized implant materials [9,10] as well as to protect Mg samples against intensive corrosion destruction [11-24]. Nevertheless, fast magnesium dissolution is the urgent problem in the implant surgery. Some researchers use laser treatment to reduce the magnesium alloy dissolution by means of Mg α-phase refinement and
homogeneous element distribution [25-30]. It should be noted that DLD technique is basically used to obtain titanium alloys, steels, but not magnesium alloys. Usually, scientists investigated the process of selective laser melting of pure Mg by single layer production [7]. There are a few studies dealt with the DLD of bulk Mg [31].

2. Experimental
Mg powder MPF-4 GOST 6001-79 (RusRedMet Research and Production Association) with following chemical composition: 0.05 wt. % Fe; 0.005 wt. % Cl; 0.1 wt. % H₂O; balance – Mg was used. The shape of particles of Mg powder is presented in figure 1a. The average size of Mg particles is 60–80 µm.

DLD process of Mg particles was realized by means of a laboratory robotic technological complex with KUKA robot KR-30 HA and a laser with 2.5 mm diameter beam. The Mg specimen was obtained in the isolated chamber suitable to work with inflammable materials, with strict control of the protective environment from process gases. Twenty-five layers were applied by DLD process to obtain Mg sample. The size of the samples was 30 mm × 15 mm × 6 mm.

X-ray diffraction (XRD) (Bruker D8ADVANCE) with Cu Kα radiation was applied to investigate the phase composition of the DLD Mg sample.

The optical microscope (Carl Zeiss Axiovert 40 MAT) was used to study the microstructure of the DLD specimen. Surface analysis was made using scanning electron microscopy (Carl Zeiss EVO 40) and energy dispersive spectroscopy (Shimadzu EDX-800).

Mechanical properties of the samples were determined by means of Shimadzu DUH-W201 ultramicrohardness tester at a load of 100 mN and a holding time of 5 s. Archimedean method was used to determine the density of the DLD sample.

3. Results and discussion
Figure 1b depicts the DLD Mg sample morphology. The specimen surface is rough with distinguished boundaries between DLD Mg layers. The presence of unmelted spheroidised particles can be found in the structure of the material.

![Figure 1. The images of Mg particles used for DLD (a) and the pure Mg specimen obtained by DLD process (b)]](image_url)

Analysis of the XRD measurements of the DLD Mg specimen indicates the presence of the only Mg phase (hexagonal structure) in the material composition.

Morphology of the specimen cross-section was studied by SEM analysis (figure 2a). These data indicate the presence of small cracks at the edge of each laser track. At the same time, the DLD sample
has a well compactness structure. There are some micropores in the material structure but neighbor layers are well connected to each other, which is a result of a good metallurgical bond created by DLD. Caused by the Gaussian laser energy distribution, the molten pools with an elliptical bottom are aligned in layers, which reflects the overlapping layered peculiarity inherent in DLD method (figure 2a).

Analysis of the SEM image shows that the width track is equal to about \(1.12 \pm 0.09\) mm and the penetration depth of molten pools is \(0.37 \pm 0.08\) mm. Thereby, remelting process of the part of each layer of the specimen is realized. Multiple remelting processes are very important to reinforce the metallurgical bond between layers, as well as to determine the microstructure of DLD specimen. From figure 2a, where the detail information about laser scanning tracks is presented, the heterogeneous character of the sample microstructure can be seen. It should be emphasized that to obtain the high strength metallurgical bond, the length between two adjacent tracks must be shorter than the width of track [32]. Figure 2b shows some unmelted particles at the boundary of the track.

![Figure 2. SEM (a) and optical (b) images of the cross-section of DLD Mg substrate](image)

The sample microstructure can be described with fine grains because of fast solidification [33], which lead to the high relative density (93.3%) as well as to high mechanical properties of the DLD pure magnesium. The microhardness and elastic modulus of the DLD specimen is equal to about \((0.49 \pm 0.09)\) GPa and \((30 \pm 4)\) GPa, respectively. The specimen density is equal to 1.622 g/cm\(^3\).

4. Conclusion
Analysis of the obtained results leads to the following conclusion.

DLD sample has a well compactness structure. There are some micropores in the material structure but neighbor layers are well connected to each other, which is a result of a good metallurgical bond created by DLD.

The DLD sample microstructure consists of \(\alpha\)-phase of Mg.

The DLD specimen possesses a high mechanical properties and high relative density. The main strengthening mechanisms for DLD magnesium sample are solid solution strengthening and grain refinement.

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