Nanoscale surface modification of biodegradable materials by severe shot peening

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Introduction

Metallic materials commonly used as bone implants have a notable mismatch of mechanical properties with those of natural bone. This incongruity induces stress shielding by not transferring the applied load to the surrounding tissue [1]. Another downside for these materials is their redundancy when used for temporary fixation purposes. Biodegradable metallic materials with mechanical characteristics comparable to those of mammal hard tissue that can stabilize damaged segments under relatively large amplitudes of static and dynamic loading over time, and then resorb without adverse tissue reactions, can be of significant importance to address the existing challenges in hard tissue engineering. They have high potential to overcome the aforementioned issues and eliminate the need for costly and complicated retrieval surgeries. However, regardless the long list of favorable characteristics of these materials with bioresorbable non-toxic degradation products, their low fatigue strength, uncontrolled corrosion rate and undesirable hydrogen gas production in physiological environment have considerably limited their application in biomedical field [2, 3].

Mg based alloys can be of high potential to be used for fixation plates in orthopedic, trauma and maxillofacial surgery, if their general mechanical and corrosion properties are improved. Many solutions have been suggested to enhance Mg based materials' corrosion resistance, including alloying [4] and surface coatings [5, 6]; however, some of these approaches can cause further complications for example compromising biocompatibility and mechanical properties. New Mg alloys have been recently developed for orthopedic implants [7], but their application particularly for load bearing cases still remains a challenge. Nanocrystallization has the potential to promote general mechanical characteristics. It also gives the prospect of decreasing the risk of prompt localized failure of biodegradable metallic biomaterials that is an issue particularly under cyclic loading. There are some studies on the effect of grain refinement on Mg based material obtained via severe plastic deformation (SPD) methods. SPD results in significant grain refinement by applying large plastic deformations at high strain rates [8-10]. Enhanced mechanical properties are reported through application of SPD methods to different Mg alloys [11-14]. Nonetheless, many opposing results have been reported about corrosion and degradation resistance of Mg alloys after SPD [15-18]. The electrochemical data are reported to be sensitive to a wide range of parameters including applied plastic strain, reduced grain size, crystallographic orientation and basal texturing [19, 20]. The broad scatter of the applied SPD treatments (equal-channel angular pressing (ECAP), surface mechanical attrition treatment (SMAT), high pressure torsion (HPT), etc.), various studied Mg alloys and the wide range of resultant grain size and orientation, in some way justify the contradicting results available in the literature.

Objectives

Herein, we applied severe shot peening (SSP), as a low cost and versatile severe plastic deformation technique aimed at surface grain refinement, on AZ31 alloy to evaluate its potential in enhancing mechanical characteristics. The parameters that distinguish SSP from the traditional shot peening are higher Almen intensity and surface coverage. SSP has been applied to a wide range of materials including low alloy steels, stainless steel, cast iron and Al alloys [21-28]. Application of SSP to AZ31 was motivated by reports on favorable effect of conventional shot peening in improving limited fatigue properties of Mg alloys typically used in automotive and aerospace industry [29-31]. Our previous studies also evidenced favorable effects of SSP on promoting cell-316L substrate interaction and reducing bacterial adhesion [32, 33]. Thus, in this study, we considered as-received not peened AZ31 specimens as well as specimens, which were shot peened with conventional parameters commonly used for this class of materials.

Methodology

Cold rolled and annealed AZ31B sheets of 6 mm in thickness (Alfa Aesar GmbH, GE) were shot peened using the parameters described in Table 1. The parameters of the applied conventional shot peening (CSP) were chosen to represent the treatment commonly used for this class of material SSP with much higher Almen intensity and surface coverage was considered to enhance the kinetic energy of the shot peening process in order to induce grain refinement on the top surface layer. As received not peened (NP) series was used as reference. A repeened severe shot peened (RSSP) series, which was SSP treated and subsequently subjected to a soft repeening using glass peening media, were also considered. After the surface treatments, each plate was cut into discs of 10 mm diameter using a milling machine. Structural and mechanical properties of the treated specimens were evaluated through microscopical observations, surface roughness and wettability as well as microhardness and residual stress distribution measurements. In-depth distribution of residual stresses was characterized by AST X-Stress 3000 portable X-ray diffractometer, removing a very thin layer of material by electro-polishing method at each step.

Table 1. Parameters used for shot peening

Specimens	Shot type	Almen intensity (mm) and Surface coverage %	Repeening Shot and Surface coverage%
Not peened (NP)	-	-	-
Conventionally shot peened (CSP)	AZB100	0.15N-100	-
Severely shot peened (SSP)	AZB100	0.4N-1000	-
Repeened severely shot peened (RSSP)	AZB100	0.4N-1000	AGB6-100

Results and analysis

Optical microscopy observations of polished and chemically etched cross sections (Fig. 1) illustrate the microstructural evolution in the cross-section of all shot peened specimens. For all the shot peened series, a differently etched top surface layer, which corresponds to the highly deformed and grain refined zone, is observed. The thickness of this dense top layer varies between the shot peened specimens depending on the applied treatment. The average thickness of this affected surface layer, were determined to be about 35 μm for CSP specimen, and 65 μm for both SSP and RSSP series. The cross section of RSSP specimen was very similar to that of SSP specimens with comparable thickness of the affected top layer, as expected. The results obtained from lateral cross section microscopical observations confirm that increasing the kinetic energy of the process form CSP to SSP clearly increases the thickness of the affected surface layer.

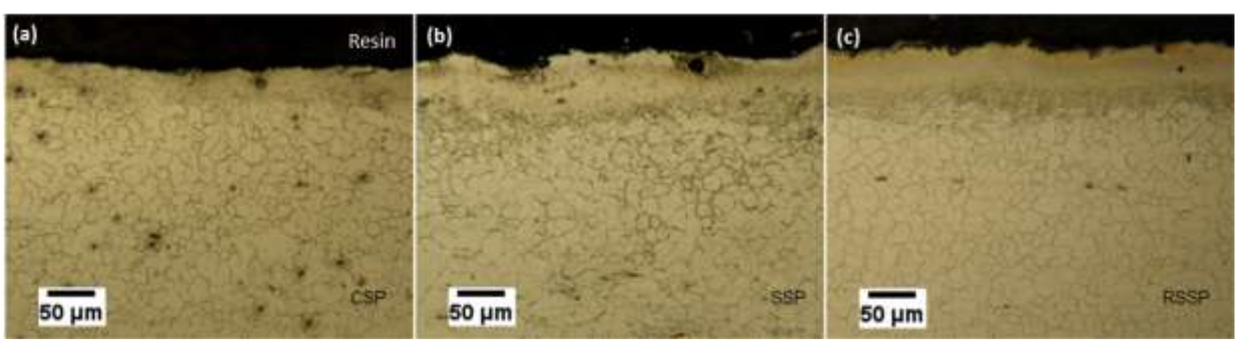


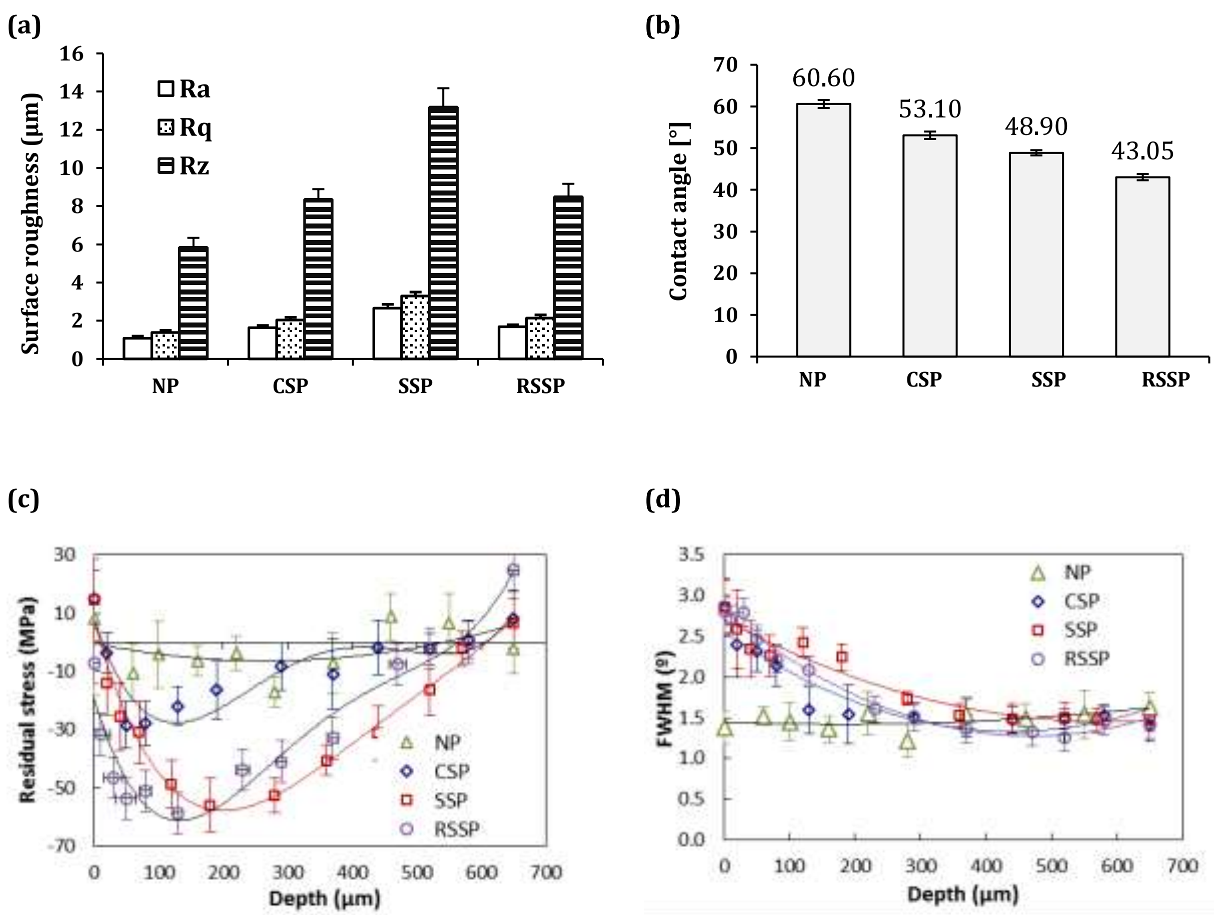
Fig.1. Representative cross section optical micrographs of (a) CSP, (b) SSP and (c) RSSP specimens.

The surface roughness parameters, shown in Fig. 2 (a), vary significantly between different series, apart from CSP and RSSP that present very similar data. The trend of parameters R_a, R_q and R_z are similar showing roughness increase from CSP to SSP series. Slight repeening after SSP treatment (RSSP) resulted in a similar morphology and comparable roughness to that of CSP series, despite the notable difference in Almen intensity.

The results of WCA measurements, presented in Fig.2 (b), indicate that shot peening treatment in general promotes hydrophilicity of the AZ31B specimens, showing the highest wettability for the RSSP series. The RSSP series have the nanocrystallized surface layer and lower surface roughness and a more regular morphology compared to the SSP series. This combination seems to have promoted the interaction of the water droplets with the substrate.

In-depth distribution of residual stresses and FWHM parameter, are shown in Fig.2 (c) and (d) respectively. Stresses for NP series are negligible; whereas, the two SSP and RSSP specimens show similar distribution of compressive residual stresses. The measurements revealed higher compressive residual stresses and higher depth of material affected by these stresses for the severely treated series (SSP and RSSP specimens), highlighting the positive effect of increased kinetic energy in enhancing the compressive residual stresses. In terms of FWHM parameter, considering NP specimen as reference, the results presented in Fig. 2 (d) confirm that all the shot peening treatments introduce substantial increase in FWHM close to the surface. The important difference in the evolution of FWHM parameter is the depth at which the value reaches that of the base material.

Microhardness profiles measured on specimens' cross-sections are presented in Fig.2 (e). The trends for all shot peened series depict maximum microhardness close to the treated surface and gradually decreasing to reach that of the base material (NP) at higher depths. SSP series represent higher surface hardness with respect of CSP samples.



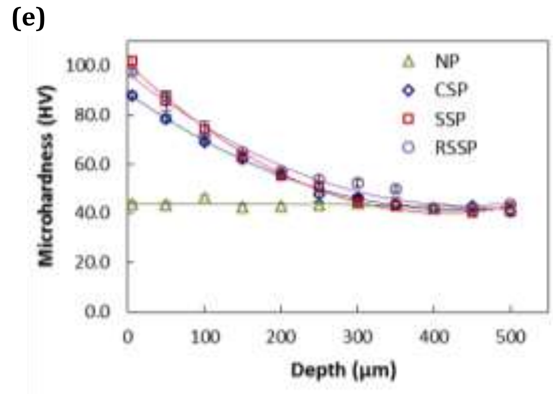


Fig.2. (a) Surface roughness parameters (b) WCA data; In-depth distribution of (c) residual stresses (d) FWHM; (e) and microhardness distribution on the specimens' cross section.

Conclusions

AZ31B Mg alloy was shot peened with different sets of parameters ranging from conventional treatment to severe shot peening. The results indicated that regardless the limited deformability of Mg alloy at room temperature, increasing the kinetic energy of the shot peening process induces notable grain refinement on the top surface layer; in addition, severe shot peening treatment increased surface roughness, enhanced microhardness and surface wettability and induced compressive residual stresses in a deep surface layer. Repeening treatment after severe shot peening was found to be effective in reconfiguring the surface morphology and roughness with minimal changes on the other studied characteristics.

Further studies are still on course to investigate the effect of different shot peening treatments on biocompatibility and corrosion properties of AZ31 as well as to investigating other biodegradable materials.

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