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Effect of Shot Peening Parameters on Surface Properties and Corrosion Resistance of 316L Stainless Steel

Mariusz Walczak^{1*}, Jonas Matijošius², Doğuş Özkan³, Kamil Pasierbiewicz⁴

- ¹ Department of Materials Engineering, Faculty of Mechanical Engineering, Lublin University of Technology, ul. Nadbystrzycka 36, Lublin, 20–618 Lublin, Poland
- ² Mechanical Science Institute, Vilnius Gediminas Technical University, J. Basanavičiaus g. 28, LT-03224 Vilnius, Lithuania
- ³ Turkish Naval Academy, National Defence University, Tuzla 34942, Istanbul, Turkey
- ⁴ Faculty of Transport and Computer Science, WSEI University in Lublin, ul. Projektowa 4, 20-209 Lublin, Poland
- * Corresponding author's e-mail: m.walczak@pollub.pl

ABSTRACT

The present work deals with the enhancement of the surface characteristics of stainless steel 316L as a result of shot peening treatment using ceramic balls. In accordance with our own research and information available in literature, as a result of shot peening process, the shot balls can penetrate to the surface layer (permanently depositing) and modify the mechanical performance and the corrosion resistance in the products being treated in this way. Shot peening leads to a significant change to the surface hardness and topography, and consequently, to the change in corrosion behaviour dependent on the choice of processing parameters. Therefore, in this paper, steel samples were treated using two variable parameters of peening pressure (0.3 and 0.4 MPa) and peening time (30 and 60s). In the research, the reference surface were the samples subjected to mechanical polishing. The surface morphology of the samples was investigated by scanning electron microscopy (SEM). The potentiodynamic polarization tests were performed with 1 mV/s scan rate in 0.9% NalCl solution. The improved corrosion resistance (lowest current density $I_{corr} = 0.35 \ \mu\text{A/cm}^2$ and highest corrosion potential $E_{ram} = -0.164 \text{ V}$) was obtained for specimens with longer time (60 s) and higher pressure of shot peening treatment (0.4 MPa). Greater changes in surface roughness were observed with an increase in peening pressure than with an increase in the processing time. The treatment of the surface with ceramic shots results in an increase in the hardness of the treated surface by more than 110% (for sample 316L/0.4/60) compared to the reference surface. Moreover, an increase in average hardness values was recorded for all surfaces after shot peening (by more than 42% relative to reference samples).

Keywords: stainless steel 316L, shot peening, corrosion behaviour, surface morphology, surface roughness.

INTRODUCTION

Corrosion processes are one of the key phenomena that determine the selection of metal alloys for the use as implants or medical instrumentation (medical devices) [1]. Medical grade 316L SS, despite some limitations (mainly related to the inability to undergo heat treatment, unsatisfactory resistance to pitting corrosion, fatigue and wear resistance), is still widely used for the production of biomaterials, such as prosthesis, stents, osteosynthesis plates as well as to a wide range of medical devices [2, 3]. The corrosion resistance of AISI 316L is closely related to its tendency to form a thin (several nanometers) layer of protective oxide Cr_2O_3 on its surface [3, 4]. It is well known that pitting corrosion is a consequence of the effects of the surrounding environment of body fluids on the state of the surface layer. The increasing demand for advanced properties surface layer under static and cyclic loading, in corrosive environments, have fueled the development of material processing techniques for a wide range of applications [5]. Literature data indicate that a very limited number of surface modification techniques can be applied to austenitic stainless steels without losing their favorable properties [3, 6]. Surface treatment technologies produce desired surface texture and establishing to improve energy conditions of the surface layer [7]. Despite the fact that, at present, surface engineering is mainly focused on the study of titanium for medical applications [8, 9], the topic of improving the surface properties of stainless steel biomaterials modified by shot peening treatment is still being addressed in the literature (it has not lost its relevance) [10, 11]. Shot peening is a cold treatment process during which the surface of the metal is bombarded with a large amount of medium (shots) shaped like spheroid. Under the influence of shot impacts on the surface, in addition to crushing, favorable compressive stresses are generated in the surface layer [11]. A key role in obtaining favorable surface properties is played by the parameters used in the treatment process, such as pressing time and pressure, angle, shot size and shot material. [2, 9]. The impact of shots on the surface causes plastic deformation, hardening of the surface as a result of which the surface morphology improves and recrystallization of the surface grains occurs [12]. In general, the fine-grained structure shows better corrosion resistance [13]. In addition, the literature indicates that the fragmentation of grains in the surface layer facilitates the formation of passive areas that provide protection against corrosion [14, 15]. As for the corrosion resistance of surfaces treated by the shot peening processes, the literature seems to be divided, i.e. one can find positions where this treatment causes an increase in corrosion resistance [15, 16] as well as a decrease [17, 18], which can be largely attributed to the induced transformation of austenite into martensite [19] and an increase in surface roughness. The results of study of Szala et al. [20] indicate that surface roughness of AISI 316L is important factor leading to corrosion. The increase in surface roughness caused by the shot peening treatment makes the surface more vulnerable to pitting corrosion [14], resulting in the destruction of passive areas on the surface of the sample [1]. The best combination for achieving both high strength and lowering the rate of corrosion processes should be the optimization of processing parameters. The authors of the papers [1, 14] observed an increase in surface hardness and roughness with increasing shot size and the intensity of the shot peening treatment. Matuszak [21] found that vibratory shot peening with steel balls as the

amplitude increased allows obtaining lower surface roughness. Champaine [22], on the other hand, justifies that the shot peening time is an important factor in achieving the desired surface coverage of a material. Sharma and Mubeen [23] studied the effect of shot size on the intensity of shot peening, guided by the selection of the right shot size to achieve the desired intensity of surface coverage. Suyitno et al. [24] have demonstrated that surface treatment with largediameter steel balls leads to an increase in surface microhardness and a decrease in current density, and thus, an increase in corrosion resistance. A model of the changes occurring in the surface layer after the shot peening process was presented by Kameyama and Komotori [25]. From this model, it appears that the surface layer transfer and the mechanical mixing of particle fragments of the pressing medium take place. As a result of the surface treatment, the shot fragments become more firmly penetrate into the surface structure of the material. This phenomenon leads to the formation of a lamellar structure characteristic of this type of treatment, with different properties from the core of the material. Most of the research studies on metal treatment for medical purposes describe the use of high impact energy shot associated with the use of steel shot, as this translates mainly into favorable mechanical properties, while marginalizing the corrosion behavior [26]. And yet, the choice of pressing medium can have a key impact on the cytotoxicity and, consequently, biocompatibility of treated parts for medical purposes [27-29]. It seems that more favorable results in terms of corrosion resistance (which also translates into biocompatibility) can be obtained by shot peening using bioinert ceramics. Therefore, the purpose of this article is to investigate the effects of selected shot peening parameters using ceramic balls on some important properties of AISI 316L steel, in particular, corrosion behavior, microhardness and surface roughness.

EXPERIMENTAL

Specimens preparation and treatments

This study used commercial 316L SS, with the chemical composition checked on a Magellan Q8 spark emission spectrometer (Bruker, Germany) the results of which are included in Table 1. The samples in the shape of disks with dimensions ø20 mm and 6 mm in height were cut from the rod. After that, all samples were wet ground with SiC papers from 200 to 1200 grit, and then

Table 1. Results of spectrometer analysis of the tested AISI 316L stainless steel (wt.%)

С	Cr	Ni	Мо	Mn	Cu	Si	Р	S	N	Fe
0.016	17.31	10.78	1.87	1.47	0.41	0.364	0.043	0.013	0.046	bal.

polished by 3 μ m diamond suspension. The samples subjected to polishing were reference surfaces. The shot peening process was carried out on the Peenmatic micro 750S device (IEPCO, Switzerland) conducting surface treatment perpendicular to the surface using two variable parameters: peening pressure (0.3 and 0.4 MPa) and peening time (30 and 60s). Ceramic beads (ZrO₂-based) from Kuhmichel Abrasiv GmbH of average size of 125÷250 μ m were used as the pressing medium (shot). The detailed parameters of ceramics beads are given in the paper [9]. The distance of the working nozzle from the modified surface was about 30 mm.

Surface characterization

The analysis of the surface structure morphology after shot peening treatment was performed using a Phenom ProX SEM microscope (Phenom-World, Waltham, MA, USA) in topographic mode with a magnification of 500×. Surface roughness measurements were taken at 12 randomly selected locations using a Dektak 150 contact profilometer (Veeco Instruments, USA). The following parameters were used to analyze the surface changes after the shot peening tests: arithmetic average roughness Ra, maximum profile valley depth of the roughness profile Rv and maximum profile peak height of the roughness profile Rp. Hardness measurements were carried out on the surface of the samples using a Vickers micro-hardness FM-700 with an automatic ARS 900 system (Future-Tech Corp., Japan). Twenty indentations were made at 2.94 N load (HV0.3) for each of the surface types.

The evaluation of corrosion resistance was performed on the basis of potentiodynamic polarization tests using the Atlas 0531 corrosion test kit. The tests were carried out in 0.9% NaCl solution at 37 °C in a three-electrode electrochemical chamber. A platinum electrode was used as the auxiliary one, and the reference electrode was a saturated calomel electrode (SCE). The surface area of the electrode under test was 0.5 cm². Tafel polarization curves were recorded with an automatic potential shift of 1 mV/s from -700 mV to +1000 mV. The values of corrosion current density I_{corr} and potentials E_{corr} were determined from Tafel curves based on the analysis of potentiodynamic curves using AtlasLab software. The corrosion rate CR (mm/year) was calculated using according to ASTM G102 as indicated in Eq. (1) [30]:

$$CR (mpy) = K \frac{I_{corr} \cdot EW}{\rho}$$
(1)

where: $K = 0.1288 \text{ (g·}\mu\text{A}^{-1}\cdot\text{cm}^{-1})$, I_{corr} – is the corrosion current density ($\mu\text{A}\cdot\text{cm}^{-2}$), ρ – is the density of the metal (g·cm⁻³) and EW – is the alloy equivalent weight (calculations according to Table 1 and include only those components whose content in the alloy is not less than 1 wt.%).

RESULTS AND DISCUSSION

Surface morphology

The changes in the surface roughness parameters of the samples before and after the shot peening treatment are shown in Figure 1. The treatment of the surface with ceramic balls resulted in an increase in the roughness Ra both at 0.3 and 0.4 MPa pressures and when the treatment time was increased from 30s to 60s. However, slightly larger changes in the *R*a parameter were observed when peening pressure was increased rather than when treatment time was changed. A similar trend of changes was reported in the work of [2], with the difference that the reference surface was subjected only to grinding and in this study the reference samples were additionally subjected to a polishing procedure. Similar conclusions were drawn in the work [31] in the case of a twin grade of AISI 304 steel treated with a CrNi shot. In general, most papers [1, 31] devoted to the analysis of surfaces after shot peening confirm the fact that an increase in time or pressure translates into an increase in surface roughness, especially when the reference surface is relatively smooth. At the same time, the studies [1, 3, 19] suggest that every material has a certain threshold of strengthening, where, when exceeded, an increase in the



Figure 1. Change in surface roughness parameters of AISI 316L SS after shot peening treatment

intensity of surface treatment can lead to a decrease in roughness parameters. Therefore, when analyzing the average values of the Rv parameter especially when using a pressure of 0.4 MPa, smaller valleys for the surface become apparent when increasing the treatment time from 30 s to 60 s. In addition, small changes (not statistically significant) are observed in the analysis of the *Rp* parameter, especially in the comparison of 316L/0.3/30 vs. 316L/0.4/30 samples. Although in this type of research mainly the Ra or Rq parameters is considered as the most representative for the evaluation of changes in the shot peening process, it seems that the more crucial influence on the corrosion resistance will be the individual elevations and valleys [2].

The SEM topography of the AISI 316L surface after shot peening is shown in Figure 2. The reference surface is reasonably smooth with minor scratches created by the surface polishing process. The surfaces subjected to shot peening treatment bear traces of plastic deformation and indentations from the shot medium. The observed increased number of pits per unit area is related to the intensity (time, pressure) of the treatment process carried out, and the above surface observations are consistent with the results of roughness (see Fig. 1). The choice of shot peening parameters affects the obtained surface roughness. In the same unit of time we have a greater number of blows/shots per unit of surface area and the situation is similar when increasing the pressure. Higher values of peening pressure lead to greater surface deformation and the generation of an irregular surface structure. More craters and higher roughness values are then observed. A similar finding can be found in the paper by Ahmed et al. [1].

Surface hardness

The results of the surface hardness measurements in Figure 3 show that the average hardness of all post-shot peening treatments increased compared to the reference samples. Compared to the reference samples, the largest increase in hardness of nearly \sim 110% is observed for the surface samples of 316L/0.4/60. In addition, the analysis of the hardness results shows that the longer the time treatment and the greater the peening treatment, the greater the strengthening of the modified material. At the same time, higher values of



Figure 2. Surface topography of SS 316L before and after shot peening



Figure 3. Change in the Vickers hardness

average hardness were obtained when the time treatment was increased from 30 s to 60 s (changes in the range of $9.27\div12.24\%$) than when the peening treatment was increased from 0.3 MPa to 0.4 MPa (changes in the range of $8.17\div11.11\%$). A similar trend in hardness changes was obtained for AIS 304 austenitic steel which was peened with a CrNi shot [31].

The observed increase in surface hardness in the literature is explained, among other things, by an increase in the dislocation density of the surface layer and the formation of a nanocrystalline structure associated with grain fragmentation [14, 32]. The depth of the reinforced layer, depending on the peening treatment conditions, can reach to a depth of 150 μ m [32] and, according to the study of Chen et al. [4], up to 200 μ m. In addition, the literature indicates that an induced martensitic transformation can occur in the gradient layers [16, 17, 32], although there is also no shortage of papers where no martensitic phase was observed [4, 12, 33]. The increase in surface hardness can be attributed to the small fragments of shots' particles that are formed when they strike the surface of the sample. During a strike, the high kinetic energy of the particles can break these materials into many small fragments [34]. Such small fragments, which are irregularly shaped, are then deposited on the surface by successive impacts of the ceramic medium, leading to a significant local

increase in hardness. Despite the ultrasonic cleaning procedure used in this study, traces of elements such as Zr, Si, Al, among others, were observed in the EDS analysis (Fig. 4). Similarly, Arifvianto et al. [34] indicated an increased Si component in the surface layer coming from the shots material.

Corrosion behaviour

The potentiodynamic polarization curves for surfaces with different degrees of modification are shown in Figure 5 and the results of key electrochemical parameters are provided in Table 2. The test curves showed a typical for austenitic steels anodic polarization behavior. The analyses of Tafel polarization curves showed a decrease in corrosion resistance for the surface after the shot peening (decrease in corrosion current density I_{corr}). The exception is the 316L/0.4/60 sample with the most favorable electrochemical parameters in terms of corrosion resistance, i.e. high potential value and low current density. In this respect, the electrochemical parameters are shaped more favorably even if compared to the reference (untreated) sample, where significantly more favorable (higher) E_{corr} values were obtained with a slightly lower I_{corr} value. For all treated surfaces, a shift of the E_{corr} potential towards positive values is observed. Chen et al. [4] showed that increasing the pressing time improves the electrochemical parameters, i.e. a shift in the potential towards positive values and a decrease in I_{corr} were also observed. For almost all samples tested, the E_{corr} potential was in the range of approximately -300÷300 mV, which, in the case of corrosion tests in simulated body fluids, is considered safe when assessing biocompatibility for products to be used in the manufacture of implants [14]. Generally in the literature, the decrease in corrosion resistance of samples after shot peening treatment is attributed to high roughness. In addition to roughness, the effect on corrosion may be due to the grain fragmentation in the surface



Figure 4. SEM microphotographs with EDS analyses of shot peened surface



Figure 5. Potentiodynamic polarization curves of shot peened 316L SS with various parameters treatment

layer occurring after the shot peening process, which improves the ability to form a passive film [14, 31]. In addition, Chen et al. [4] indicate that the nano-grains and twins accelerate the nucleation process and increase the growth rate of the passive film, considerably improving the stability of the passive film. On the other hand, Qiao et al. [15] indicate that the grain boundary density increased sharply. Such a phenomenon leads to the fact that passive films nucleates instantaneously at these interfaces and grows uniformly in all directions along the surface plane. Therefore, it can be assumed that for the surface of the 316L/0.4/60 sample, the



Figure 6. Surface after the potentiodynamic polarization in the 0.9 wt.% NaCl solution for: a) and b) untreated 316L SS, c) and d) sample 316L/0.3/30.

Specimen	Current density, I _{corr} (µA/cm²)	Potential, <i>E_{corr}</i> (V)	Potential, <i>E_{Pit}</i> (V)	Corrosion rate, CR (mpy)
316L	0.44	-0.307	0.385	0.18
316L/0.3/30	2.43	-0.276	0.458	0.99
316L/0.3/60	2.41	-0.249	0.383	0.98
316L/0.4/30	1.45	-0.231	0.375	0.59
316L/0.4/60	0.35	-0.164	0.475	0.14

Table 2. Results of electrochemical parameters at different surface conditions

above phenomena have a much stronger/more powerful effect and outweigh the negative effect associated with high roughness. The analysis of the surface after corrosion tests (Fig. 6) showed corrosion pits (black areas). The formation of corrosion pits is associated with the occurrence of a puncture potential. Then, a passivity plateau is observed on the anodic curves, followed by (at a potential $E_{p_{it}}$ of about $0.38 \div 0.48$ V vs. SCE) a sudden increase in current density (passivity puncture) and the development of pits on the tested surfaces of the samples.

CONCLUSIONS

Based on the results of the studies and observations, the following conclusions can be drawn:

- EDS analysis of the chemical composition of the surface of the samples subjected to shot peening revealed small fragments of the peening particles in the surface layer, which can locally cause an increase in surface hardness.
- The treatment of the surface with ceramic balls results in an increase in the hardness of the treated surface by more than 110% (for sample 316L/0.4/60) compared to the reference surface. At the same time, higher values of average hardness are obtained when the pressing time is

increased from 30 s to 60 s, than when the pressure is increased from 0.3 to 0.4 MPa.

- The shot peening treatment of 316L steel resulted in an increase in surface roughness. At the same time, greater changes in the Ra parameter were obtained when the peening pressure was increased rather than when the treatment time was changed.
- Among the samples tested in 0.9% NaCl environment, the 316L/0.4/60 surfaces are the most favorable (lowest current density and highest corrosion potential). Moreover, the electrochemical parameters obtained in the corrosion test for all the tested surfaces can be considered safe in the context of assessing the corrosion resistance of products to be used as medical devices.

Considering the above observations, it can be concluded that the most favorable performance properties were obtained for the surface treated with ceramic balls at a pressure of 0.4 MPa and a time of 60 s.

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