Corrosion Science 82 (2014) 328–338

Contents lists available at ScienceDirect

Corrosion Science

journal homepage: www.elsevier.com/locate/corsci

SEM/EDS characterization of laser shock peening effect on localized corrosion of Al alloy in a near natural chloride environment

Uroš Trdan, Janez Grum *

Faculty of Mechanical Engineering, University of Ljubljana, Askerceva 6, 1000 Ljubljana, Slovenia

ARTICLE INFO

Article history:
Received 31 October 2013
Accepted 24 January 2014
Available online 31 January 2014

Keywords:
A. Aluminium
B. Polarization
B. EIS
B. SEM
C. Hardening
C. Pitting corrosion

ABSTRACT

This study examines the effect of laser shock peening without coating (LSPwC) on the corrosion behaviour of AA6082-T651 alloy in a near natural chloride environment. The results confirmed LSPwC as an effective method, yielding lower anodic dissolution ($E_{sw} - E_{corr}$), improved repassivation ($E_{corr} - E_{rp}$) as well as corrosion current reduction. Characterization of the corrosion attack was performed by SEM/SEI/BEI and EDS analysis, where results confirmed crystallographic pitting as the predominant effect due to local dissolution of the Al matrix around Fe rich precipitates. Moreover, it was found that LSPwC reduces crystallographic and surface-hemispherical pitting, as well as intergranular attack.

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1. Introduction

Nowadays aluminium alloys represent an important material category due to their good weight/strength ratio and high corrosion resistance. Peak-aged wrought Al alloys in particular have been widely used in the aircraft, automotive, marine, and construction industries [1–4]. However, despite the general high corrosion resistance of aluminium alloys in a chloride environment localized corrosion [5,6] and stress corrosion cracking [7] often occurs. In this aspect, additional surface treatment and protection is essential to prolonging the service life of machine parts [8]. In recent years, laser shock peening (LSP) has attracted global interest due to its ability to improve both mechanical properties [9–11] and corrosion resistance [5,6,12].

LSP surface treatment is a versatile, fast emerging, and promising technique that can be used to achieve surface enhancement without affecting the bulk material properties. The process involves underwater sample irradiation with laser pulses of short duration, i.e. nanoseconds, and power density of several GW/cm^2 with high numbers of repetition of laser pulses. LSP enables plasma generation at the moment of the interaction of laser light and propagation of shock impact waves in the material [8,13]. In practice, LSP has already been accepted as a substitute for conventional Shot Peening (SP). Higher fatigue strength [14,15], increased wear resistance [9], improved resistance to stress-corrosion cracking [16,17], and enhanced surface finish with an absence of micro-cracks [18] are just a few of the numerous benefits that make LSP promising for a variety of practical applications. In addition, Shen et al. [19] have confirmed the applicability of a new three-dimensional (3D) forming technique, i.e. laser shock embossing, where laser-generated shock wave are applied to shape the work-piece with micro-moulding.

Our preliminary studies on laser shock peening without ablative coating (LSPwC) on Al alloy 6012 and 6082 [6] have shown improved mechanical properties of the LSPwC treated material compared to the untreated one, with increased microhardness and generated compressive residual stresses (RS) of a high gradient. Lu et al. [20] confirmed that there was an obvious grain refinement mechanism at work after LSP treatment of LY2 alloy, with the grain size in the top surface in the range from 100 nm to 200 nm. Similar results showing an ultra-fine-grained surface layer on a Zr–1% Nb alloy induced by ultrasonic impact peening (UIP) due to severe plastic deformation were also reported by Mordyuk et al. [21]. Results suggested that the mechanical properties of a Zr–1% Nb alloy containing (in wt.%) 1 Nb, ~0.15 Fe, <0.16 O, and <0.01 Hf in the form of 10 mm thick strips (hot forged and rolled) can be tailored by additional alloying. However, the corrosion behaviour of Zr–Nb alloys in saline solutions can be improved more easily by means of proper superficial processing techniques leading to grain refinement. Furthermore, the authors [21] have pointed out that the strategy of purposeful grain refinement is undoubtedly useful in the preparation of materials needing

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* Corresponding author. Tel.: +386 1 477 1203; fax: +386 1 477 1225.
E-mail addresses: uros.trdan@fs.uni-lj.si (U. Trdan), janez.grum@fs.uni-lj.si (J. Grum).

http://dx.doi.org/10.1016/j.corsci.2014.01.032
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superior functional characteristics, such as those widely used in nuclear plant systems, and biomedicine.

Furthermore, comparison of the strain hardening effect of both SP and LSPwC [13] has confirmed the latter as the more effective method, with a lower increase in surface roughness and higher compressive residual stresses (RS) in the thin sub-surface layer of the treated material (−242 MPa vs. −113 MPa). Our recent study [8] has also confirmed the strong influence of LSPwC surface scan direction, i.e. the laser beam movement in the transverse direction yielding higher compressive RS regardless of the pulse density. Moreover, it has been shown [5] that corrosion resistance after LSPwC can be greatly improved, with the corrosion current reduced by factor of 12 compared to the untreated material.

However, the micro-structural effects, i.e. the galvanic effect of the laser treated corroded material, have not been studied in detail. Therefore, the goal of this study is to understand in detail where the initiation of corrosion of both the untreated and LSPwC treated material is taking place. Specific attention was therefore focused on characterizing the pitting attack initiated on the untreated and LSPwC treated AA6082 Al alloy during cyclic polarization in aggressive 0.6 M NaCl solution. Surface pits were characterized using scanning electron microscopy (SEM/SEI, SEM/BEI), whereas the chemical composition of the corroded surface after CP was determined by energy dispersive X-ray spectroscopy (EDS). Electrochemical impedance spectroscopy (EIS) after a longer immersion time of 24 h was also carried out.

2. Material and methods

For this study, a commercial alloy AlSi1MgMn (AA6082-T651), supplied in the form 10 mm thick rolled plates, was used. The chemical composition of the investigated Al alloy is given in Table 1. Prior to LSPwC, specimens were cut along the longitudinal (L) and long-transverse (LT) directions. Prior to laser treatment, the specimens were thoroughly degreased with acetone then rinsed with deionised water.

LSPwC surface treatment (a schematic presentation is given in Fig. 1) was performed using a Q-switched Nd:YAG laser operated at an irradiation wavelength of 1064 nm. The focused laser beam was 2.5 mm in diameter, whereas the pulse duration (FWHM) and frequency were fixed to 10 ns and 10 Hz, respectively. The maximum pulse energy was 2.8 J, thus peak power density was equal to 5.7 GW/cm^2. Pulse density during LSPwC treatment was set to 900 pulses/cm^2.

Topography measurements prior to corrosion experiments were carried out using a ZEISS LSM700 laser confocal scanning microscope with a resolution of 20 nm. The selected analysed area for 3D topography for both untreated and LSPwC treated specimens was 2328.98 μm × 2328.98 μm. The surface roughness parameter S_a was calculated using a unified cut-off wavelength filter, i.e. $\lambda_c = 200$ μm. In addition, the cross-sectional microstructure of untreated and LSPwC treated specimen was studied by optical microscope (OM) Meiji MT-7100.

All electrochemical tests were performed using a PAR Versastat-4 potentiostat/galvanostat/ZRA. A classic three-electrode electrochemical cell was used, where a saturated calomel electrode (SCE) was employed as the reference electrode. A water jet process was applied in order to obtain disc samples of diameter for the electrochemical investigations.

Table 1
Chemical composition of the investigated AA6082-T651 alloy (in wt.%).

Elements	Composition in wt.%
Si	0.87
Mg	0.72
Mn	0.42
Fe	0.35
Others (Cu + Cr + Ni + Zn + Ti)	0.15
Al	Balance

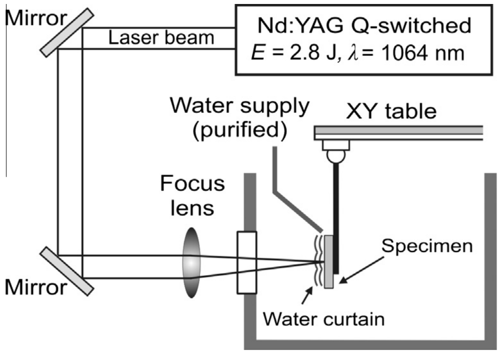

Fig. 1. Schematic presentation of LSPwC setup.

Prior to each experiment, the untreated specimen surfaces were successively abraded using 220, 320, 600, 800 and 1200 grit SiC paper; LSPwC treated specimens were not abraded due to a possible loss of information (ablation, shock waves, etc.). Immediately prior to each experiment, specimens were cleaned and degreased in acetone, alcohol, and deionised water for 3 min each, respectively. Afterwards, the specimens were embedded in a PAR Teflon holder, which served as the working electrode with an exposed surface of 1 cm^2. Specimens were then immediately transferred to the electrolytic cell. All the electrochemical experiments were carried out in naturally aerated, near natural (pH = 6.9 ± 0.2; $T = 21 \pm 1$ °C) 0.6 M NaCl, prepared with deionised water. Each experiment was conducted in the same manner and was carried out at least two times, using freshly prepared solution each time, afterwards the average of the data was taken in order to obtain reproducibility of the results.

Cyclic potentiodynamic polarization (CP) scans were performed using an initial delay time at the equilibrium state of 60 min at steady open-circuit potential (OCP) in order to stabilize the surface. The polarization scan started at 100 mV below the OCP at a rate of 1 mV/s. In order not to cause too much material dissolution or to destroy the sample surface by using the CP technique too expressively, we chose a limited threshold value according to previous studies [21–24]. Thus, the sweep direction was reversed to the cathodic direction at a chosen limited threshold of 1 mA/cm^2. The surface condition and chemical composition of the surface after CP were examined using a JEOL JSM-5610 scanning electromicroscope equipped with EDS microanalysis hardware.

The impedance EIS measurements were recorded during a 24 h immersion in the test medium. EIS spectra were recorded at OCP, where a sinusoidal 10 mV$_{RMS}$ perturbation signal through a frequency domain from 100 kHz down to 100 mHz was used. Experimental EIS data were determined using an equivalent electrical circuit with ZSimpWin software. The impedance data were additionally fitted and simulated with different models in order to obtain the most reasonable values for the EIS parameters.

3. Results and discussion

3.1. Surface morphology

Fig. 2 shows 3D topographic images of the untreated and the LSPwC treated specimens' surface, where the distinct influence of preliminary laser treatment, where more pronounced waviness is achieved compared to the untreated specimen. Surface roughening

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