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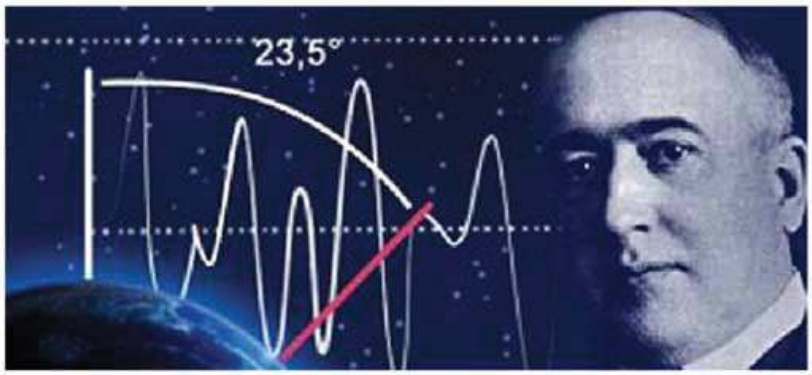

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CORROSION CHARACTERISTICS OF LASER CLEANED BRASS SURFACES

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Abstract: *Different types of weapons and military equipment, made of brass, are often exposed to extreme atmospheric conditions, to chemical agents, erosion and wear. It is important to evaluate the corrosion characteristics of brass in different corrosive environments, and especially after laser cleaning of corrosion products, various deposits and other undesirable surface layers. The brass corrosion resistance changes, after surface laser cleaning, were examined in this paper. Nd:YAG laser with λ=1064 nm was used for laser cleaning process. SEM analysis was applied for investigation of the brass surface micro-morphology before and after laser cleaning. Electrochemical techniques such as linear polarization resistance, electrochemical impedance spectroscopy, linear sweep voltammetry and electrochemical frequency modification were used to study the corrosion characteristics of laser treated and mechanically prepared brass surface. The results of electrochemical tests have shown that the corrosion rate of laser treated brass surface is approximately same as the corrosion rate of brass surface prepared by the standard grinding and degreasing process. This indicates that the brass surface was efficiently cleaned by laser treatment with applied operating parameters without lowering its corrosion resistance. The abilities of different electrochemical methods for determination of corrosion rate were compared.*

Keywords: *laser cleaning, brass, corrosion resistance, electrochemical methods.*

1. INTRODUCTION

Defense industry requires specific metals and alloys not only for weaponry and defense equipment, but also for communications equipment and infrastructure. Copper and copper-based alloys are used for the aircraft elements, munitions, gas pipes, naval equipment, electronics and other highly specialized products. These materials are often exposed to high or low temperatures, humidity, chemical agents, wear and so on. They have to withstand the corrosion, temperature extremes and wear.

The main problem in metal equipment is metal corrosion. Cleaning is a required on the almost all items: planes must be de-painted; ground equipment, electronics, ships or any other piece that operate or they are stored in the depot, have to be cleaned.

Laser technology provides a safe, environmentally acceptable and effective cleaning method for a wide range of materials [1-5]. Cleaning by the lasers is versatile, controllable, selective, precise and reliable. With appropriate parameters it is possible to remove unwanted layers without removing the original material. The portable lasers are suitable for items that are too large or located far away from the depot. On that way cleaning flexibility and processing times can be improved.

Besides the fact that lasers are widely used in metal surface cleaning, laser-surface interaction, during cleaning, can affects surface properties, especially corrosion properties. Study of the structural, chemical and mechanical properties changes, corrosion resistance and others similar characteristics are of interesting to get an insight in the efficiency of applied laser treatment. On that way, the laser beam parameters that produce desired changes on the object surface can be chosen [1-5].

The interaction of a laser beam with a material surface is complex and shows the multi-physics nature of the phenomenon: thermal, mechanical and optical. The kind of interaction depends mainly on the material properties and also on the laser parameters [6, 7].

The solid surface laser removal is based on the ablation phenomena. Because of short pulses of high peak power laser irradiation, thin surface layers rapidly heats and

vaporizes, which is used to clean a surface. The ablation phenomenon is characterized by ablation threshold fluence.

The interaction between the laser beam and oxide layer may generate sufficient thermal effects to induce additional thermal oxidation or even melting a thin layer of the surface. Such a thermal effect is dependent on several factors, including laser beam wavelength, pulse width, laser processing parameters, thickness of oxide layers, and nature of oxide layers as well as substrate materials [1-3, 8].

Mottner and coauthors [1] observed that the interaction between surfaces and laser energy for the tested brass sample was greater for lower lasers wavelength.

The absorbance of oxidized alloys is higher than for ideal pure polished metals [9]. Generally, UV lasers clean oxide surfaces most efficiently since the UV radiation is easily absorbed by the oxide.

Zhang and coautors [8] showed that the laser-cleaned surface exhibited higher corrosion resistance than as-received hot-rolled alloy.

The aim of this paper was to examine the changes in the corrosion resistance of the brass plate after laser cleaning by Nd:YAG laser ($\lambda \approx 1064$ nm). The results of the corrosion resistance testing of the mechanically prepared brass surface are also presented, in the purpose of comparison. The corrosion tests were performed using various electrochemical techniques [10]: electrochemical frequency modulation (EFM), linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS) and linear sweep voltammetry (LSV). As a result of the electrochemical measurements, the charge transfer resistance and the corrosion current density were obtained, which can be expressed as the corrosion rate. The corrosion rate was determined in a neutral solution containing chlorides and sulphates.

2. EXPERIMENTAL PART

Nd:YAG laser, Thunder Art Laser, produced by Quanta System (with wavelengths λ = 1064 nm, optical pulse duration < 8 ns, and output pulse energy up to 1000 mJ) was used in the presented experiments. The repetition rate is up to 20 Hz, with a beam diameter of 10 mm and 70 % fit to Gaussian energy distribution. All experiments were performed on a 1 mm thick brass plate. The brass sample, used in this test, was covered with corrosion products.

The laser parameters used for two cleaning zones were: λ = 1064 nm, the energy E = 750 mJ (fluence 6 J/cm^2), the number of pulses n = 25 and the laser spot diameter d = 4 mm. The specimen (76 x 30 x 1 mm brass plate) was placed in front of the laser head so that the laser beam was directed perpendicularly to the specimen. A dry cleaning method was used. The experiment was performed in atmospheric conditions.

A scanning electron microscope (SEM) JEOL JSM-6610LV, which operates at 20 kV, equipped for energy dispersive spectroscopy (EDS) measurements, was used to analyze the morphology of the laser treated brass surface before and after cleaning. The same SEM/EDS device was used for the analysis of the chemical composition of the brass surfaces (CuZn27).

The corrosion resistance of the mechanically prepared brass sample as well as that of the laser cleaned samples was studied applying four electrochemical techniques.

2.1. Electrochemical techniques

The following electrochemical techniques were applied: electrochemical frequency modulation (EFM), linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS), and linear sweep voltammetry (LSV).

2.2. Electrochemical Frequency Modulation (EFM)

This is a new nondestructive corrosion-measurement technique that can directly give values of the corrosion current without *a priori* knowledge of Tafel constants. Like Electrochemical Impedance Spectroscopy (EIS), EFM is a technique using small AC signals (in this case ± 10 mV vs. E_{corr}). Unlike EIS, however, two sine waves at different frequencies are applied to the cell simultaneously. Because current is a non-linear function of potential (Butler-Volmer equation), the system responds in a non-linear way to the potential excitation. The current response contains not only the input frequencies, but also contains frequency components which are the sum, difference, and multiples of the two input frequencies. The theory of this technique is very complex [10]. However, this non-linear response has been shown to contain enough information about the corroding system so that the corrosion current can be calculated directly.

The two frequencies may not be chosen at random. They must both be small, integer-multiples of a "base frequency" that determines the length of the experiment. In this experiment the two input frequencies are 2 Hz and 5 Hz. The base frequency was 1 Hz.

The current response is measured and processed. The result is a spectrum of current response as a function of frequency. The spectrum is called the "intermodulation spectrum" (see results below). The two large peaks are the response to the 2 Hz and 5 Hz excitation frequencies The Electrochemical Frequency Modulation analysis script uses these peaks to calculate the corrosion current and the Tafel constants. Between the peaks the current response is very small. Applying this analysis the value of j_{corr} and values of b_a and b_c Tafel constant can be calculated. The corrosion rate can be obtained directly from the value of j_{corr}.

The great strength of the Electrochemical Frequency Modulation technique is that, according to EFM theory, the ratio of the constant b to d (which appears during appropriate calculation) should not depend on corrosion rate or Tafel constants. The ratio d/b is given the name Causality Factor 2 because that ratio is 2.0. Likewise, the ratio of other two constants e/c should be equal to 3.0, and is given the name Causality Factor 3. These Causality Factors serve as an internal check on the validity of the EFM measurement.

2.3 Linear polarization resistance technique

This technique was used to determine the polarization resistance (or the charge-transfer resistance R_{ct}) of the tested

iron sample in the corrosion environment (test solution). The value of R_{ct} is inversely proportional to the corrosion current density j_{corr} and to the corrosion rate v_{corr} [10,11]. The iron sample was polarized in a narrow potential range ($E = \pm$ 10 mV) with respect to the corrosion potential E_{corr}, starting from the cathodic region to the anodic one, and then the corresponding current j was recorded. The potential sweep rate was 0.166 mV s^{-1}. The value of R_{ct} was determined as the slope of the experimental E - j curve at the corrosion potential E_{corr}. The corrosion current density j_{corr} was calculated using the experimentally determined value of R_{ct}.

2.4 Electrochemical impedance spectroscopy

The value of R_{ct} was also determined using the EIS technique [12]. Alternating potential of small amplitude (± 10 mV) was imposed on the working electrode (the tested iron sample) in the test solution. The applied frequencies f were from 100 000 Hz to 0.01 Hz. The value of R_{ct} was determined on the basis of the electrochemical impedance values at very high and very low frequencies. The corrosion current density and the corrosion rate were then calculated using the experimental value of R_{ct}. The corrosion current density j_{corr} was calculated using the Stern-Geary equation:

$$j_{corr} = B/R_{ct} \quad (1)$$

where B is a constant that depends on the values of the Tafel slopes (b_a and b_c):

$$B = b_a \cdot b_c / 2.303 \cdot (b_a + b_c) \quad (2)$$

The value of the constant B for the tested iron sample in $NaCl + Na_2SO_4$ (pH7) solution is calculated using b_a and b_c Tafel constants, obtained from results of previously performed EFM tests.

The corrosion rate v_{corr} can be calculated using the Faraday law, based on the corrosion current density j_{corr}, in accordance with ASTM G102 [13].

2.5. Linear sweep voltammetry

This technique was used to obtain the Tafel polarisation diagrams [14]. The iron sample was polarized in the potential range $E = \pm$ 0.200 V relative to E_{corr} and the corresponding current density j was recorded. The applied potential sweep rate was 1 mV s^{-1}. The corrosion current density j_{corr} was directly determined from the obtained Tafel diagrams by extrapolating the linear parts of the anodic and cathodic polarization curves to the corrosion potential E_{corr}.

The electrochemical tests were carried out using a potentiostat/galvanostat Gamry Interface 1010E, in a solution containing sulphates and chlorides (0.3 mol dm^{-3} NaCl + 0.1 mol dm^{-3} Na_2SO_4). The tests were performed at room temperature, in the presence of atmospheric oxygen.

The tests were carried out in an electrochemical cell with a saturated calomel electrode (SCE) as a reference electrode and a Pt mesh as an auxiliary electrode. The working electrode was the mechanically prepared brass sample and laser-cleaned brass sample. Before the beginning of the polarization measurements, the sample was kept for a certain time period at open circuit potential, to establish a stable corrosion potential E_{corr}.

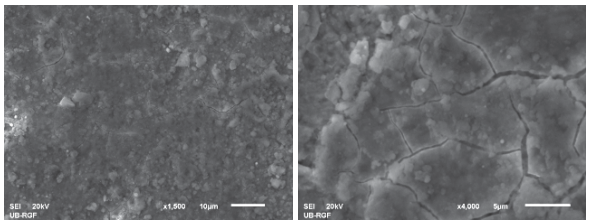
3. RESULTS AND DISCUSSION

3.1. Laser cleaning results

The laser beam can induce chemical, structural and morphological modifications on the sample surface, leading to a change in the surface characteristics.

The laser irradiation results were observed and analysed by SEM. Figures 1a) and b) present the SEM images of the uncleaned brass surface at different magnifications. It can be seen that the sample is covered with a layer of corrosion products. The corrosion products cover the entire sample surface.

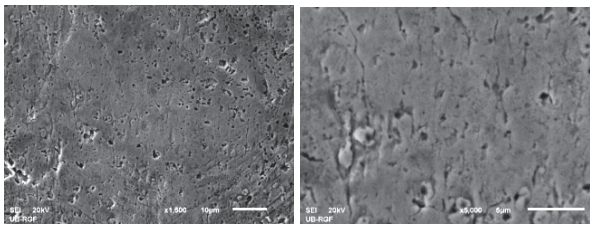
Figure 2 illustrates the topography of the zone on the brass sample surface after laser irradiation. It can be seen that all corrosion products have been removed in the central part of the laser-cleaned zone.



a) b)

Figure 1. SEM micrographs of the uncleaned brass sample, covered with corrosion products

The macroscopic analysis of the tested sample (Figure 2) shows that the applied laser fluence caused visible changes. The chosen parameters of corrosion products cleaning are below the damage threshold of brass. The laser beam caused the removal of corrosion products without damaging the base material.



a) b)

Figure 2. SEM micrographs of the laser-cleaned zone of the brass sample at different magnifications.

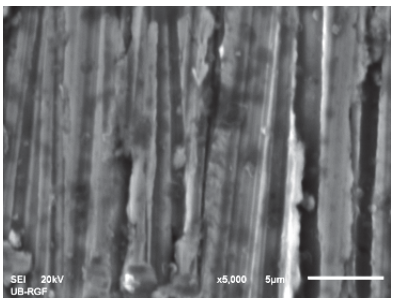


Figure 3. SEM micrographs of the mechanically prepared brass surface.

Electrochemical corrosion testing was also performed on the mechanically prepared brass surface with the aim of comparing its corrosion resistance with the corrosion resistance of the laser-cleaned iron zone. Figure 3 shows the brass surface after mechanical cleaning and degreasing. The traces of the previous mechanical grinding can be seen.

3.2. Results of electrochemical tests

3.2.1. Mechanically prepared brass sample and laser-cleaned brass sample

Corrosion on brass in neutral solutions in the presence of chlorides and sulphates is a complex electrochemical process that consists of several anodic and cathodic reactions.

In these solutions, the main anodic reaction is oxidation of copper and zinc with the release of electrons. Due to the presence of atmospheric oxygen, the main cathodic reaction is oxygen reduction:

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \quad (3)$$

Figure 4 shows the test results of the mechanically and laser treated brass sample by electrochemically frequency modulation technique: a) *E-t* dependence for mechanically prepared brass sample, b) *j-t* dependence for mechanically prepared brass sample c) *E-t* dependence for laser cleaned brass sample, and d) *j-t* dependence for laser cleaned brass sample.

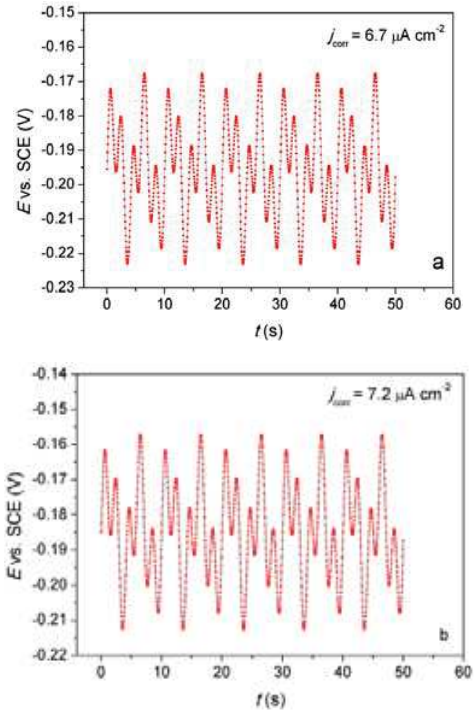


Figure 4. Results of EFM testing.

EFM technique is nondestructive technique that can directly give values of the corrosion current density and b_a and b_c Tafel slopes.

The internal check on the validity of the EFM measurement is performed by Causality Factor 2 and Causality Factor 3. Causality Factor 2 for mechanically prepared sample is 2.004, while for laser cleaned sample is 1.979. Causality Factor 3 for mechanically prepared sample is 3.043, while for laser cleaned sample is 2.817. Obtained Causality Factors show that the EFM experiments were performed correctly.

Values b_a and b_c for Tafel slope were 45 mV dec^{-1} and 118 mV dec^{-1} for mechanically prepared brass sample, while b_a and b_c for laser cleaned brass sample were 51 mV dec^{-1} and 164 mV dec^{-1}. These values were used for calculation corrosion current density in LPR and EIS tests.

The value of the corrosion current density j_{corr} obtained by EFM technique for the laser-cleaned brass sample (j_{corr} = 7.2 μA cm^{-2}) is approximately same as for the mechanically prepared brass sample (j_{corr} = 6.7 μA cm^{-2}). The value of the corrosion rate v_{corr} is directly proportional to the value of the corrosion current density j_{corr} [13].

Figure 5 shows the results obtained using the linear polarization resistance method on the mechanically prepared brass sample (Figure 5a) and on the laser-cleaned brass sample (Figure 5b). The slope of the experimental potential-current density *E - j* curve on the corrosion potential E_{corr} represents the value of the charge transfer resistance R_{ct}. The R_{ct} value for the mechanically prepared brass sample is 3500 Ω cm^2, which is approximately same value as for the laser-cleaned brass sample (R_{ct} = 3750 Ω cm^2).

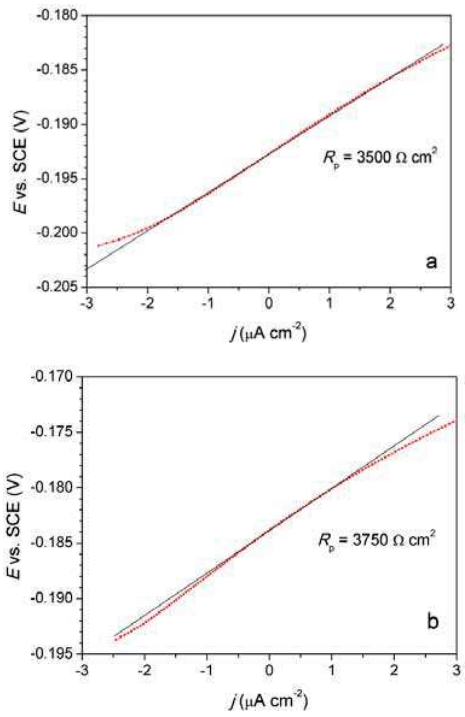


Figure 5. Results of the LPR tests: a) mechanically prepared brass and b) laser-cleaned brass.

Based on the R_{ct} values, the corrosion current density j_{corr} (Equation 3) and the corrosion rate v_{corr} of the brass samples were calculated using ASTM G102 standard [13]. The corrosion rate v_{corr} of the laser-cleaned brass sample is also approximately same as the corrosion rate v_{corr} of the mechanically treated brass sample.

Figures 6 and 7 show the results obtained by the method of electrochemical impedance spectroscopy on the mechanically prepared brass sample (a) and the laser-cleaned brass sample (b). Figure 6 shows the obtained Bode plots (the Bode modulus and the Bode phase diagram), while Figure 7 shows the Nyquist plots. The charge transfer resistance R_{ct} value is obtained as the difference of impedance values at low and high frequencies. The R_{ct} value for the laser-cleaned brass sample is 3100 Ω cm^2, while the R_{ct} value for the mechanically prepared brass sample is 2500 Ω cm^2. The results of the EIS measurements show that the value of the charge transfer resistance R_{ct} for the laser-cleaned sample is somewhat higher than the value of R_{ct} for the mechanically prepared brass sample. The values of the corrosion current density j_{corr} and the corrosion rate v_{corr} were calculated using the obtained R_{ct} values.

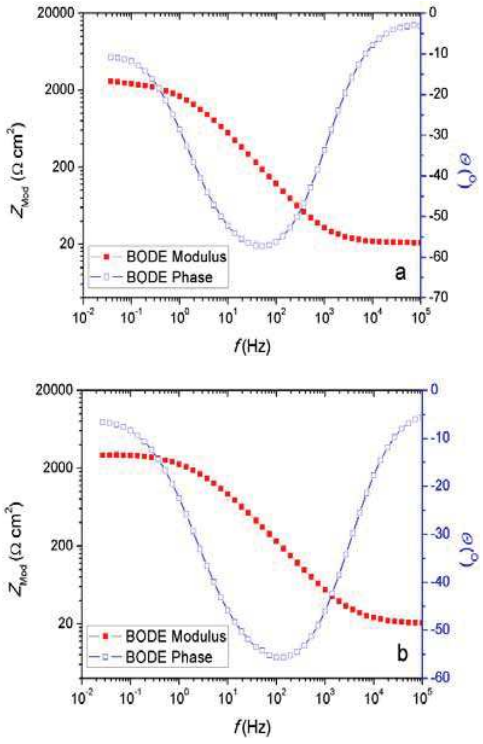


Figure 6. Results of the EIS tests: a) mechanically prepared brass and b) laser-cleaned brass.

Figure 8 shows the results obtained using the linear sweep voltammetry method (the Tafel diagrams). The results for the mechanically prepared brass sample and the laser-cleaned brass sample are shown in this Figure.

The value of the corrosion current density j_{corr} is determined by the extrapolation of the linear parts of the anodic and cathodic polarisation curves to the corrosion potential E_{corr}. The value of the corrosion current density j_{corr} is somewhat lesser for the laser-cleaned brass sample than for the mechanically prepared brass sample. The value of the corrosion rate v_{corr} is directly proportional to the value of the corrosion current density j_{corr} [13].

The results presented show that all four electrochemical methods give the same results (Figures 4-8).

The corrosion rate of the laser-cleaned brass sample is same or somewhat lesser than the corrosion rate of the mechanically prepared brass sample. This some unusual behavior of brass can be explained as follows: laser treatment (cleaning) causes the local surface heating of a thin metal layer, which leads to the melting and decomposition of the previous brass structure.

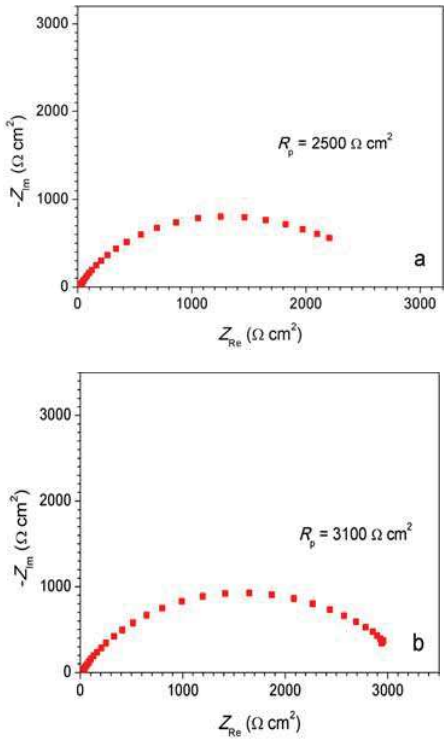


Figure 7. Results of the EIS tests: a) mechanically prepared brass and b) laser-cleaned brass.

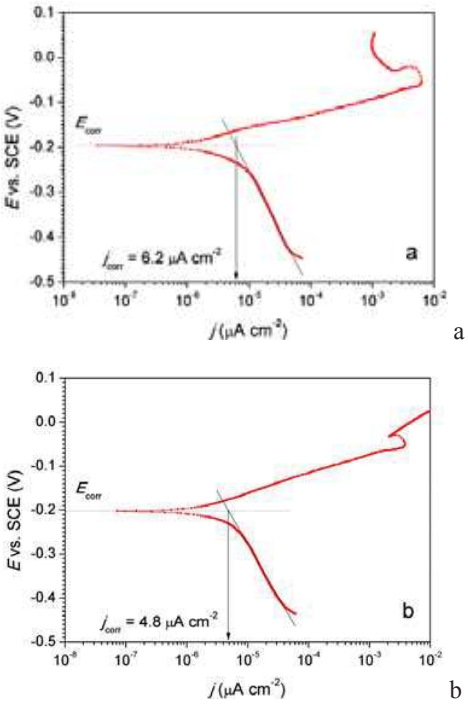


Figure 8. Results of the LSV tests (the Tafel plots): a) mechanically prepared iron and b) laser-cleaned iron.

Rapid cooling leads to some structural transformations as recrystallization. The formation of a fine recrystallized structure can occur in the brass surface layer. It is known that the presence of a fine recrystallized structure results in higher strength and hardening of the brass matrix and better stress corrosion resistance [14]. On the other hand, the rate of corrosion reactions (the rate of the cathodic reaction in particular) can be expected to decrease due to the formation of a fine recrystallized structure. Influence of grain size on corrosion resistance of various metal and alloys was reviewed in [15].

Table 1. summarizes the results of the electrochemical testing of mechanically treated and laser cleaned brass.

Table 1. Results of electrochemical testing

	LPR		EIS		EMF			Tafel		
	R_p, Ω cm^2	j_{corr}, μA cm^{-2}	R_p, Ω cm^2	j_{corr}, μA cm^{-2}	b_a, mV dec^{-1}	b_c, mV dec^{-1}	j_{corr}, μA cm^{-2}	b_a mV dec^{-1}	b_c mV dec^{-1}	j_{corr} μA cm^{-2}
Ms m	3500	4.1	2500	5.6	45	118	6.8	36	252	6.2
Ms l	3750	4.5	3100	5.5	51	164	7.2	41	199	4.8

4. CONCLUSION

Nd:YAG laser used for cleaning the brass surface covered with corrosion layers (fluences below 6 J/cm^2), has removed corrosion products without damaging the base material. The micro-morphological changes on laser treated zones are investigated by SEM. The result shows that laser cleaning is very safe and efficient, if the proper parameters are chosen in cleaning process.

The corrosion resistance of mechanically prepared brass and laser-cleaned brass was tested using four different electrochemical techniques (electrochemical frequency modulation, linear polarization resistance, electrochemical impedance spectroscopy and linear sweep voltammetry). All experiments were performed in a solution containing chlorides and sulphates.

The obtained results have shown that the corrosion rate of the laser-cleaned brass sample is approximately same or somewhat less than the corrosion rate of the mechanically prepared brass sample. This is probably caused by the structural transformation of the laser-cleaned brass sample, which led to the formation of a fine recrystallized brass structure. The corrosion rate is expected to be lower in the presence of this fine recrystallized structure.

The value of the corrosion current density j_{corr} determined by Tafel extrapolation is in accordance with the value of the charge-transfer resistance R_{ct}, determined by the EIS technique. In general, all applied electrochemical methods gave similar results.

The results of this study show that it is necessary to examine all the changes occurring in the laser treated surfaces in order to ensure successful application of lasers in corrosion cleaning.

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