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ELECTROCHEMICAL AND MECHANICAL PROPERTIES OF COBALT-CHROMIUM DENTAL ALLOY JOINTS

ELEKTROKEMIJSKE IN MEHANSKE LASTNOSTI RAZLIČNIH SPOJEV STELITNE DENTALNE ZLITINE

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In dentistry cobalt-chromium alloys are frequently used for partial denture frameworks. For fabrication of some complex frameworks, separate metal segments have to be joined. The longevity of these restorations is limited due to the mechanical or corrosive failure of the joints. The purpose of this study was to determine which joining method offers the best properties to cobalt-chromium alloy frameworks. Intact specimens, brazed and two types of laser-welded joints were compared for their electrochemical and mechanical characteristics. Electrochemical impedance spectroscopy and potentiodynamic polarization in two artificial saliva solutions were used to assess basic corrosion parameters and tensile strength of brazed and laser welded specimens was measured. The fracture surfaces and corrosion defects were examined in a scanning electron microscope. The average tensile strength of brazed joints was significantly greater than the tensile strength of both types of laser-welded joints. When laser welding was used, successful joining was limited to the peripheral aspects of the weld. The welding technique did not affect significantly the joint tensile strength. Electrochemical measurements indicated that primarily due to differences in pasivation ability, the corrosion resistance of the laser-welded joints was better than that of the brazed.

Key words: brazing, laser welding, dental alloys, corrosion, strength

V zobni protetiki pogosto izdelamo ogrodja fiksnih in snemnih protetičnih izdelkov iz stelitnih (kobalt-kromovih) zlitin. Pri kompleksnejših konstrukcijah je treba spojiti posamezne dele ogrodja. Trajnost protetičnega izdelka pogosto omejujejo mehanske in korozijske poškodbe spojev. Namen te raziskave je bil ugotoviti, s katerim načinom spajanja dobimo korozijsko in mehansko najbolj odporne spoje. Primerjali smo lotanje in dva načina laserskega varjenja. Osnovne korozijske parametre smo ugotovili z elektrokemijsko potenciodinamsko polarizacijo in elektrokemijsko impedančno spektroskopijo v dveh različnih raztopinah umetne sline. Pri lotanih in lasersko varjenih vzorcih smo izmerili natezno trdnost. Lomne ploskve in korozijske poškodbe smo pregledali z vrstičnim elektronskim mikroskopom. Povprečna natezna trdnost lotanih spojev je bila značilno višja od trdnosti obeh skupin lasersko varjenih spojev. Z laserskim varjenjem smo uspešno spojili le površinsko plast vzorcev. Način laserskega varjenja ni značilno vplival na natezno trdnost spojev. Elektrokemijske lastnosti spojev kažejo, da so, predvsem zaradi razlike v sposobnosti pasivacije, lasersko varjeni spoji korozijsko obstojnejši od lotanih.

Ključne besede: lotanje, lasersko varjenje, dentalne zlitine, korozija, trdnost

1 INTRODUCTION

Cobalt-chromium (Co-Cr) alloys are frequently used for fixed and removable partial denture frameworks.[1–3] In the fabrication of some complex frameworks and in repairs or additions, separate metal segments of the framework have to be joined.[2] The longevity of these restorations is limited due to the mechanical or corrosive failure of the joints.[4–6] Several joining techniques are available but brazing and laser welding are most commonly used.

Brazing is a process in which a molten filler metal wets and fills the gap between the parent metal surfaces.[3] The filler metal has a lower melting point than the parent metal.[3] In welding, the parent metals fuse and form the joint with or without a filler alloy.[2] Conventional heat sources tend to produce excessive thermal damage to prosthodontic restorations and are therefore not used in dentistry.[2] Laser welding has recently gained popularity, mostly because it is simpler and less time consuming than brazing.[7] For brazing Co-Cr alloys, a Co-Cr alloy with a lower melting point or a noble alloy serves as a filler.[5–8] Gold-based filler alloys are often used because their melting points are well below those of Co-Cr alloys.[5–8] Research has shown that these joints have poor mechanical properties and corrosion resistance[5,7,9] and exhibit a significantly lower tensile strength than laser welded joints.[7,9] Angelini et al [5] compared the corrosion resistance of Co-Cr alloy brazing done with a gold-based filler and a Co-Cr filler and concluded that Co-Cr filler is more appropriate than gold-based filler.

Co-Cr dental alloys have an excellent corrosion resistance, which is provided by a thin adherent layer of chromium-based oxides on the surface.[5,6,10] Considerable defects caused by corrosion tend to appear primarily in the joints. Beside specific problems related to crevice forming, joining of 2 metals with different corrosion potentials can form a galvanic cell. [11] In such a case, the less noble metal acts as a main anode, and it could exhibit a relatively high dissolution rate.[12,13] Such problems with brazed joints, caused by the dissimilar composition of the filler and the parent metals are reported.[14–16] Corrosion not only results in a poor esthetic outcome, but can also compromise physical properties and induce biological irritation in form of an allergic reaction, lichen planus, or some other soft tissue inflammation.[5,12,17,18]

Corrosion properties are commonly assessed by various types of electrochemical measurements.[13,19] Electrochemical potentiodynamic polarization (EPP) techniques are often used, providing general information about the corrosion resistance and susceptibility, such as the general corrosion rate, the range of passivation, and the break-down potential.[12,20] These results should be carefully considered since with EPP the information is not obtained in stationary conditions.[20] In rather passive systems with relatively low corrosion rates (application of noble metals or systems with protective coatings) more reliable information can usually be gained from electrochemical impedance spectroscopy (EIS) measurements.[12,20] EIS applies sinusoidal voltage signal of relatively small amplitudes (usually a few tens of mV) and the conditions of the electrodes are only slightly disturbed.[12,21] Beside the general corrosion properties of an investigated system, specific information about underlying electrochemical mechanisms can also be obtained from the measured impedance spectra.[20,21]

These electrochemical methods have been successfully implemented in several investigations of various corrosion problems in dentistry.[21–25] Electrolyte solutions, such as artificial saliva, are often used as the corrosion medium because their electrochemical properties are similar to those of the natural saliva.[26] The level of corrosion resistance of brazed and laser-welded Co-Cr dental alloy joints has so far been determined merely with microscopic assessment of corrosion defects. Electrochemical properties of different joints have not yet been compared quantitatively.

Most studies investigating the mechanical resistance of various joints showed that laser welds have higher tensile strength than soldered or brazed joints using noble solders.[7,9,26,27] However, laser welds are more prone to fatigue damage than brazed joints.[9]

Short laser pulses which heat metal beyond the melting point are used for laser welding. The amount of energy released in each laser pulse is controlled by setting the welding parameters (voltage, pulse duration, and focus diameter).[4] Spots where laser pulses are applied cool rapidly and the welding depth is sometimes relatively shallow in comparison with the diameter of the welded object.[28,29] More powerful laser pulses not only deepen the weld penetration, but also increase porosity.[2,28,29] In an attempt to overcome this problem, different joint designs have been proposed.[2] If adjacent joint-forming surfaces are ground so as to form the shape of the letter X, they can be laser-welded starting from the center and the joint is built towards the surface of the object.[2] In the process, metal is added to the joint by a filler wire with a composition equal, or very similar to the parent metal.

The aim of this study was to determine which method of joining Co-Cr alloy framework segments produces joints with the best strength and corrosion resistance. The joining methods used were brazing with a Co-Cr filler and laser welding with 2 different joint designs.

2 MATERIAL AND METHODS

52 cylindrical specimens with a diameter of 2 mm and the length of 35 mm were cast in a Co-Cr alloy (Remanium GM 380; Dentaurum), following the procedures recommended by the alloy manufacturer. The alloy composition is shown in **Table I**. 16 specimens were selected for electrochemical measurement, and divided into 4 groups of 4 specimens each. In the intact group, the specimens were left as cast. The specimens of the remaining 3 groups were sectioned at the center, perpendicular to their long-axis, using a 0.6 mm separating disk and subsequently re-joined by brazing (brazing group) and laser welding, using an X- or I-shaped joint design (X laser and I laser groups, respectively).

36 specimens were selected for tensile strength testing, and divided into 3 groups in which specimen pairs (n = 6) were to be joined by brazing or laser welding, using an X- or I-shaped joint design.

To achieve a standard gap for the brazing group, a 0.3 mm metal shim was placed between the two halves of specimens. The shim was removed once the specimens were positioned and invested in the phosphate-bonded investment. Flux was applied, and a Co-Cr solder was used as the filler metal. Its composition is shown in **Table I**. The assembly was pre-heated to 750 °C and torch brazed at 1180 °C, following the procedures recommended by the manufacturer of the solder.

For laser welding the I-shaped joint design, the joint surfaces of specimens were straight and placed in tight contact with each other. An Nd:YAG laser with the wavelength of 1064 nm was used with the following settings: voltage of 290 V, pulse duration of 10 ms, and weld spot diameter of 0.7 mm. A weld spot overlap of approximately 75 % was used, so that the joint was

Table I: Composition (w/%) of Remanium GM 380 alloy, Co-Cr solder, and filler wire
Tabela I: Sestava zlitine Remanium GM 380, Co-Cr lota in varilne žice v masnih deležih (w/%)

	Co	Cr	Mo	Mn	Ni	C	Fe	Si	N	B	Nb
GM 380 alloy	64.6	29	4.5	<1	–	<1	–	<1	<1	–	–
Co-Cr solder	61	28.5	3.5	–	–	<1	1.5	4	–	1	–
filler wire	–	22.1	9.1	–	63.8	–	1	–	–	–	3

formed with 25 pulses per specimen. These laser conditions were chosen to simulate typical laboratory procedures for welding of Co-Cr dental alloys.[2,7,27]

With the X-shaped laser welding design the joint surfaces were ground to form the shape of the letter X if viewed from the side. During grinding, the surfaces were cooled with 75 % ethanol. The tip was rounded with a hand instrument to facilitate the alignment of the halves. Laser welding was performed using pulses of lower energy (settings: 255 V, 4.5 ms, 0.9 mm) because no deep weld penetration was required for this joint design. A Co-Cr filler wire was used to complete the joints. The composition of the wire is shown in Table I. Specimens were polished using conventional laboratory procedures[2] for removable partial denture frameworks with silicone polishers and polishing paste for Co-Cr alloys. The final joint diameter varied slightly due to the custom finishing.

The corrosion parameters were assessed by electrochemical potentiodynamic polarization (EPP) and electrochemical impedance spectroscopy (EIS). Since it was expected that the investigated electrochemical systems would exhibit relatively low corrosion activities, the polarization potential in EPP measurements were changed in a wide interval from cathodic to anodic region (–0.5 V vs *E*corr to +2 V vs *E*corr). From measured potentiodynamic curves, the corrosion current densities and break-down potentials were determined. In EIS measurements, the sinusoidal voltage signal with an amplitude of 10 mV in the frequency interval between 10 mHz and 5 kHz was applied. Significant parameters, as the total impedance ($|Z_0|$) and the polarization charge-transfer resistance (R_{CT}), were estimated from the measured spectra. The working electrode was always the specimen with a saturated calomel electrode serving as a reference electrode and a graphite electrode as a counter electrode. Fusayama type artificial saliva [30] was used as the corrosion medium. Its pH value is 4.65 and it has the following composition: NaCl 0.4 g/L, KCl 0.4 g/L, $CaCl_2 \cdot 2H_2O$ 0.795 g/L, NaH_2PO_4 0.69 g/L and urea 1 g/L. A potentiostat (PC3/750; Gamry Instruments Inc) with the appropriate software (CMS 100 and CMS 300) was used for the experiment.

The joined specimens were tested in tension. The total length of the specimens was 7 cm, which allowed for secure fixation in the testing machine (Z 030; Zwick GmbH & Co). The joint diameter was measured with each specimen using an electronic caliper having an accuracy of 0.01 mm. The determined value was used to compute the tensile strength of the joints by the formula $\sigma = F/\pi r^2$, where σ is the tensile strength, F is the load at fracture and r is the half-diameter of the joint.[3] Tensile tests were performed under constant extension rate of 0.008 s^{-1} (relative extension) according to the standard EN 10002-1.[31] A 30kN load cell was used (KAP-TC, class 0.05; Zwick GmbH & Co) with the software (testXpert V10.11) provided by the manufacturer.

The data were statistically analyzed using a 1-way analysis of variance (ANOVA) and Scheffé post hoc tests. Differences between groups were regarded as significant at $\alpha = 0.05$. After mechanical and electrochemical testing the fracture surfaces and corrosion defects were analyzed by scanning electron microscopy (JSM–5500; JEOL), whereas the longitudinal sections were studied under a metallographic optical microscope (Neophot 22, Carl Zeiss AG).

3 RESULTS

Potentiodynamic curves obtained for intact alloy and different joints are shown in **Figure 1**. The curves of intact alloy and laser-welded joints are similar as all of them contain rather steep and nearly straight sections up to approximately 800 mV versus saturated calomel electrode. Very low dependence of electrochemical current on the applied potential in this region indicates a nearly passive state. Beyond these potentials, the so-called break-down potentials, there are distinct transitions to active corrosion, where measured currents start to increase rapidly. The potentiodynamic curve corresponding to the brazed joint shows that the measured current increased continuously and no distinct passive region was observed. The differences in break-down potentials between groups were statistically analyzed and found to be significant ($P = 0.036$), with the brazed joint having much lower average break-down potential (544 mV) than other specimens (pooled

Table II: Mean values and standard deviations (SD) of corrosion potentials (E_{corr}), break-down potentials (E_{bd}), corrosion current densities (I_{cor}), total impedances at lowest frequency ($|Z_0|$) and estimated polarization charge-transfer resistances (R_{CT})

Tabela II: Povprečne vrednosti in standardne deviacije (SD) korozijskih potencialov (E_{corr}), porušitvenih potencialov (E_{bd}), gostote korozijskega toka (I_{cor}), impedance pri najnižji frekvenci ($|Z_0|$) in ocene polarizacijske upornosti (R_{CT})

	mean E_{corr} (mV)	SD E_{corr} (mV)	mean E_{bd} (mV)	SD E_{bd} (mV)	mean I_{corr} ($\mu A/cm^2$)	SD I_{corr} ($\mu A/cm^2$)	mean $\lvert Z_0\rvert$ (kΩ)	SD $\lvert Z_0\rvert$ (kΩ)	mean R_{CT} (kΩ)	SD R_{CT} (kΩ)
intact	–462.3	111.7	758.3	73.6	0.10	0.05	182.9	38.0	75.1	10.8
brazing	–409.9	32.0	543.8	298.0	0.07	0.04	80.9	37.3	7.1	5.1
I laser	–560.2	9.7	875.5	32.4	0.22	0.14	140.1	30.3	21.5	4.0
X laser	–555.0	29.5	885.0	84.4	0.16	0.06	173.6	16.2	29.5	6.0

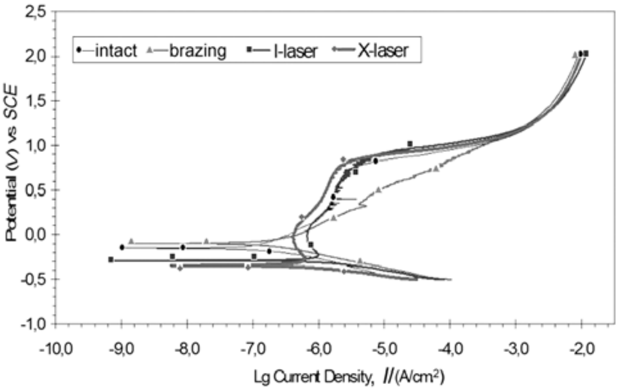

Figure 1: Potentiodynamic curves for different joints and intact alloy
Slika 1: Potenciodinamske krivulje za različne spoje in intaktno zlitino

average 840 mV). All corrosion current densities were very low (from 10^{-7} to 10^{-6} A/cm^2). Exact values of corrosion current densities were calculated by the extrapolation of the Tafel slopes[19] however, they are generally represented as the intersections of anodic and cathodic curves (**Figure 1**). Estimated values of the corrosion potentials, corrosion current density, and break-down potentials are shown in **Table II**.

The electrochemical impedance spectra generally confirmed the observation drawn from the electrochemical potentiodynamic measurements. Impedance spectra are presented as Nyquist plots in **Figure 2**. Total impedance at the low frequency range is generally related to the corrosion resistance. The impedances at the lowest frequency $|Z_0|$ and estimated values of the polarization charge-transfer resistance R_{CT} are shown in **Table II**.

The results obtained from EPP measurements and those from EIS measurements were in agreement. Both of them clearly expressed higher corrosion resistance of the laser-welded joints compared to the corrosion resistance of the brazed joints. SEM analysis of the specimens confirmed the results of the electrochemical measurements. The most pronounced corrosion damage was at the brazed joints, located primarily at the parent metal. Corrosion damage of the intact alloy and the laser-welded joints was minor and mostly located at certain defects on surfaces, such as inclusions or scratches.

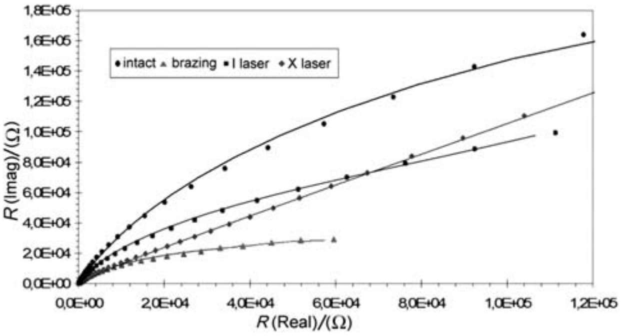

Figure 2: Nyquist plots for different joints and intact alloy
Slika 2: Nyquistovi diagrami za različne spoje in intaktno zlitino

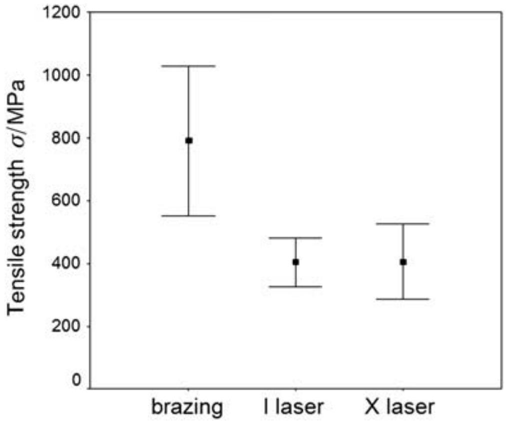

Figure 3: Means and standard deviations of tensile strength of the different joint types
Slika 3: Povprečne vrednosti in standardne deviacije natezne trdnosti različnih spojev

The mean value (standard deviation) for the tensile strength of brazed joints was 792 (238.5) MPa. This was significantly ($P = 0.004$) greater than the tensile strength of both types of laser-welded joints. The mean values for I-shaped and X-shaped joint designs were 404 (76.7) MPa and 405 (120.4) MPa, respectively. These are shown in **Figure 3**. All specimens, regardless of the joining technique, fractured in the joints. The strength of the laser-welded joints did not depend significantly on the joint design used. SEM examination, however, revealed differences in the effective cross-sections joined. The fracture surfaces of brazed joints were relatively smooth and they exhibited a fine grained partially ductile nature (**Figure 4**). Fracture surfaces of the I-shaped laser-welded joints showed that only peripheral aspects of these specimens were successfully joined, since under the surface there were some voids

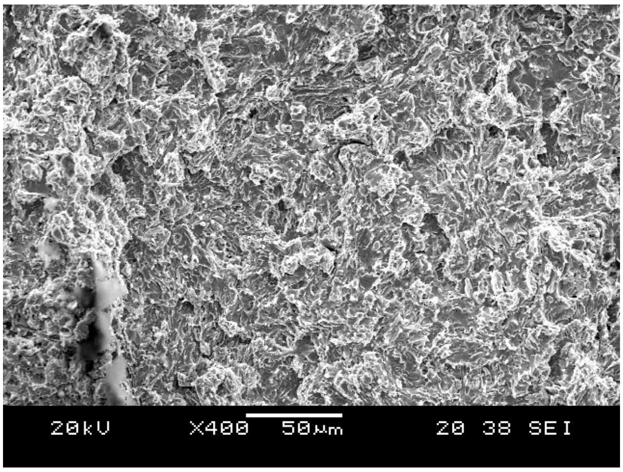

Figure 4: Fracture surface of brazed joint at X 400 magnification
Slika 4: Lomna ploskev lotanega vzorca pri 400-kratni povečavi

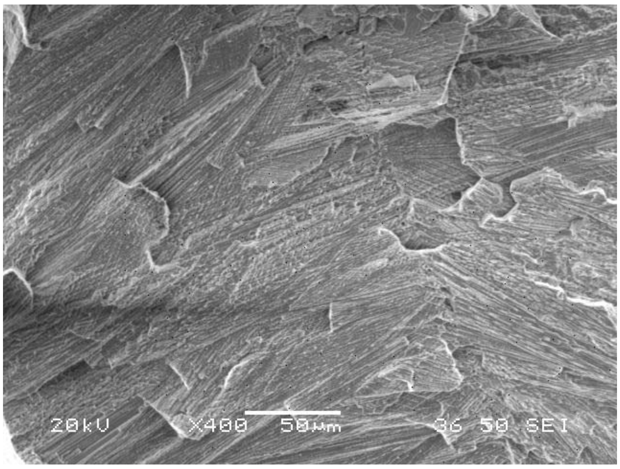

Figure 5: Fracture surface of laser welded joint at X 400 magnification
Slika 5: Lomna ploskev lasersko varjenega vzorca pri 400-kratni povečavi

and the central area remained unwelded. Even after X-shaped laser welding, the central area remained partly unwelded, but the joined effective cross-section was larger than in the I-shaped laser-welded specimens. However, there was no significant difference in the tensile strength of the two groups. In the laser welded joints the fracture surfaces were coarse grained and brittle (**Figure 5**).

4 DISCUSSION

The average tensile strength of the laser-welded joints was significantly lower than that of the brazed joints, mainly due to the smaller cross-section of the welded joints and partly due to the relatively strong brazed joints. For the purpose of comparing brazing with laser welding of Co-Cr alloys, most authors used a noble filler metal.[5–7] Average tensile strengths of these brazings ranged from 357 MPa to 519 MPa.[7,9] In this study, the average tensile strength of brazed specimens was 792 MPa. The average tensile strength of laser-welded Co-Cr dental alloy joints investigated thus far has ranged from 480 MPa to 751 MPa,[7,28, 29] which exceeds the average strength of laser welds in this study (404 MPa and 405 MPa).

An important reason for the relative weakness of laser-welded joints in this study is a small effective cross-section of specimens that was actually joined. This is a problem associated with low weld penetration depth.[28,29] In laser welding using the I-shaped joint design, laser pulses were not powerful enough to reach the central parts of the specimens, although peripherally the metal was overheated with a resulting porosity. Optimizing laser parameters could, to some extent, improve the quality of these welds.[29] The composition of the alloy greatly affects its weldability and in this respect, the carbon content is critical.[28] For laser welding, the manufacturer recommends an alloy with no carbon (Remanium 900). Yet, according to clinical experience, this alloy is not stiff enough for partial denture frameworks and was therefore not used in this study.

Nevertheless, laser welding of Co-Cr denture frameworks has been used with considerable success in clinical practice. A plausible explanation is that during mastication prostheses are never subjected to isolated tensile loads. In bending, most of the load is placed on the peripheral parts of the framework; one side is subjected to tension and the other one to compression, while central portions are less involved. Since the examination of fracture surfaces in this study showed that the laser welding technique was much more effective in the peripheral than in the central parts of the specimens, it is possible that the technique would prove satisfactory in most clinical situations.

The results of electrochemical measurements, which are in agreement with previously published data,[6,12,17,18] showed excellent corrosion resistance of the intact Co-Cr dental alloy. On the basis of previous qualitative observations, it was expected that laser-welded joints would exhibit better resistance to corrosion than brazed joints. This was confirmed by the results of both electrochemical methods used. With the potentiodynamic curves of the welded joints, a characteristic passive region was observed, whereas in the case of the brazed joints, measured current increased rapidly and continuously. The more noble potential of the filler additionally polarized the parent metal and consequently almost no passivation took place. Similar conclusions could be drawn from the measured impedance spectra, where all characteristic impedance values of the brazed joints were lower than those of the intact alloy and the welded joints.

However, the corrosion processes, which were investigated by EPP and EIS, occurred at the surface of the test specimen and thus were not influenced by bulk defects. Compared to brazed joints, laser-welded joints had more porosity and similar defects likely to initiate corrosion in a clinical situation. From EPP and EIS measurements it was not possible to determine weather the corrosion process was localized or generalized, which is a limitation of this study. SEM analysis revealed localized phenomena, such as pitting near the joints and pronounced corrosion in some defects on surfaces. In case of localized corrosion, and over longer periods of time, the process could become autocatalytic and more pronounced than in the study.

The laser welding process could, to some extent, be improved by increasing the weld penetration depth. Preparation of the areas to be welded, such as marking them black with a black felt-tipped pen or airborne-particle-abrading can reduce laser beam reflection and could probably improve the welding efficiency.[2] In situations where surfaces to be laser-welded can be designed in advance, large joint surfaces might ensure sufficient strength and limited thickness might enable complete joining with minimum porosity. These questions remain to be addressed in the future.

5 CONCLUSION

Laser-welded Co-Cr alloy joints exhibit excellent corrosion resistance, but their tensile strength is limited due to the shallow weld penetration. Using laser welding mainly the peripheral parts of the joints can be successfully welded. Brazed joints are less resistant to corrosion but have significantly higher tensile strength.

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