Journal of Materials Processing Technology 212 (2012) 2012–2019

Contents lists available at SciVerse ScienceDirect

Journal of Materials Processing Technology

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

Effects of electrolyte on hardening in laser hole sealing of commercially pure grade 1 titanium

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ARTICLE INFO

Article history:
Received 24 November 2011
Received in revised form 17 April 2012
Accepted 1 May 2012
Available online 16 May 2012

Keywords:
Laser hole sealing
Titanium
Hermetic sealing
Electrolyte medium
Hardness
Interstitial elements
Microstructure

ABSTRACT

Following laser hole sealing the hardness of CP1 Ti welds was found to be directly related to the microstructure. It was observed that welds made with electrolyte present had a microstructure which consisted mostly of the acicular alpha phase compared to welds made without electrolyte which had a mixed structure of serrated alpha, platelet alpha and acicular alpha phases. The hardest regions of welds made in the presence of electrolyte consisted of a fine colonized acicular alpha phase. Results from energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) analysis further suggested that an increase in the composition of interstitial elements was the primary mechanism responsible for increased hardness when welding with electrolyte present.

1. Introduction

Laser microwelding (LMW) has been widely applied to manufacturing various medical devices. The extensive use of LMW in the medical device industry is a result of several advantages over competing joining technologies such as high precision, limited contamination of weld metal, small heat affected zone (HAZ) and highly consistent reliable joints, as reported by Xie and Safarevich (2003). Therefore, the LMW process is well suited for hermetically sealing titanium (Ti) medical device components. Sealing of commercially pure grade 1 (CP1) Ti capsules is often required for medical device components that are filled with an electrolyte medium. Commercially pure grade 1 Ti is extensively used because it provides excellent combinations of corrosion resistance, mechanical properties and biocompatibility.

The laser hole sealing process investigated in this study is a novel, quick and effective technique developed for the sealing of small holes (i.e. 150–225 μm in diameter) which at a previous process step were used for filling the component with electrolyte. In a previous investigation by Huang et al. (in press), it was suggested that differences in Ti weld metal properties will exist when hole sealing in the presence of an electrolyte. These changes in properties such as hardness imply changes in microstructure and more importantly weld metal composition. These changes can lead to differences in the performance of the Ti material such as yield strength, corrosion performance and biocompatibility, which are of particular importance in medical device applications.

Past literature has shown significant hardness increases in fusion zone of laser welded Ti. Roggensack et al. (1993) observed a 60 HV increase in hardness in laser welded Ti, which they attributed to interstitial impurities and a finer microstructure. Similarly Li et al. (2009) observed a hardness increase of 40 HV at the center of Ti laser welds and measured an increase in oxygen content in the fusion zone suggesting an interstitial hardening mechanism. Li et al. (2005) made a direct connection between the amount of oxygen in the shielding gas and the increase in hardness of the weld metal also suggesting that oxygen impurities are responsible for the increased laser weld metal hardness. Therefore, due to the sensitivity of the mechanical properties of Ti to interstitial and substitutional impurities and the highly reactive nature of Ti, laser welded Ti often has different properties compared to the base material. In the application of laser hole sealing where the weld was made in the presence of an electrolyte medium, the mechanisms behind changes in material properties such as hardness have not yet been investigated. A better understanding of these mechanisms will aid in the development of new laser hole sealing processes and improve on existing ones. In this study the effects of electrolyte on the laser hole sealing process and the weld metal microstructure

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http://dx.doi.org/10.1016/j.jmatprotec.2012.05.001

Table 1
Mechanical properties and chemical composition [wt.%] of CP1 Ti.

Tensile strength [MPa]	Yield strength [MPa]	N (Max)	C (Max)	H (Max)	O (Max)	Fe (Max)
240	170	0.03	0.1	0.015	0.18	0.2

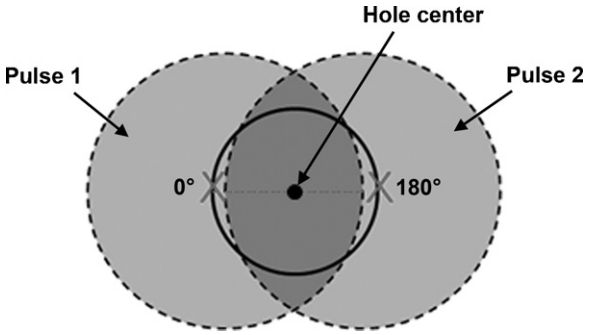

Fig. 1. Illustration of welding procedure with pulse 1 at 0° and pulse 2 at 180° relative to the hole center (not to scale).

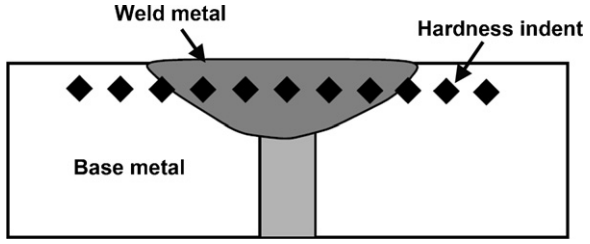

Fig. 2. Illustration of Vickers hardness indents made across the cross-sectioned Ti weld.

and hardness were investigated using optical microscopy, scanning electron microscopy (SEM), Vickers microhardness testing, energy dispersive X-ray spectroscopy (EDS) and micro X-ray diffraction (XRD) analysis.

2. Experimental

The laser hole sealing was performed on CP1 Ti discs both with and without the presence of an electrolyte medium. The laser hole sealing and sample preparation procedures are similar to those used by Huang et al. (in press). The properties and chemical composition of CP1 Ti are given in Table 1. The electrolyte used in this study was composed of a mixture of de-ionized water and organic compounds containing carbon (C), hydrogen (H), oxygen (O) and fluorine (F). The Ti test discs used in this study had a thickness of 400 μm and each disc contained 16 test holes of 150 μm diameter.

A custom welding fixture was used to hold the Ti test discs in a manner that allows for the bottom side of the discs to be exposed to electrolyte during the laser sealing, simulating a hermetic sealing process, as was done by Huang et al. (in press). Before welding a consistent amount of electrolyte was injected into a reservoir that was sealed to the bottom of the disc until electrolyte was observed to protrude from all 16 holes.

Laser hole sealing was performed using a Miyachi Unitek LW50 pulsed Nd:YAG micro-laser welding system capable of 50 J pulse energy and 5 kW peak power. Two laser pulses fired at a rate of 1 pulse per second (pps) were used to seal the holes in the Ti discs. The first pulse was fired at 0° and the second at 180° relative to the hole center as illustrated in Fig. 1. The welding was performed with the laser in focus having a spot size of 600 μm and the step indexed (SI) fiber used provided a top hat spatial profile. The peak power and pulse duration welding parameters were varied in this study. The peak power parameters were varied from 0.8 kW to 1.0 kW and the pulse duration was varied from 2 ms to 5 ms. Each parameter set was repeated both with and without electrolyte present. Due to the highly reactive nature of Ti a protective atmosphere was necessary during welding. In this study a protective atmosphere was achieved by directing an argon (Ar) shielding gas at the hole to be sealed. A shielding gas flow rate of 14.2 l/min (30 CFH) was

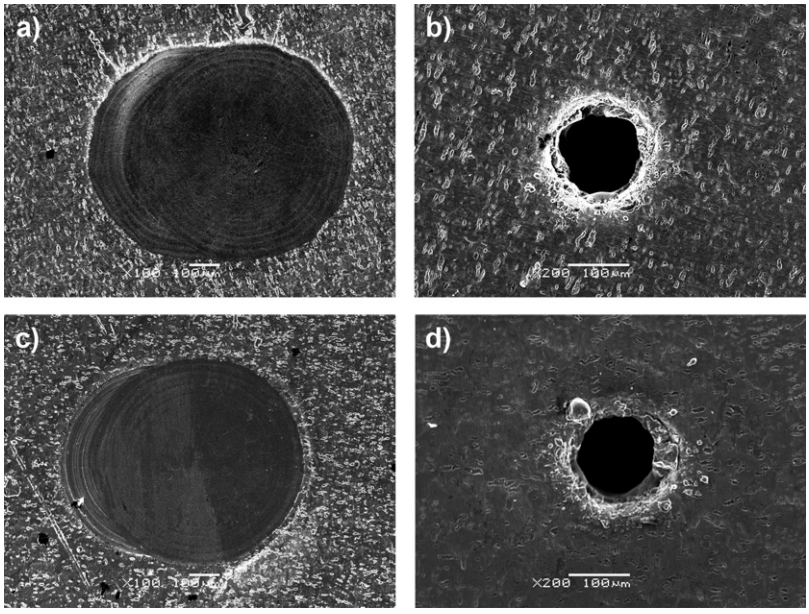

Fig. 3. Top (a) and bottom (b) surfaces of welds made with 0.8 kW peak power and 2 ms pulse without electrolyte. Top (c) and bottom (d) of weld made with electrolyte.

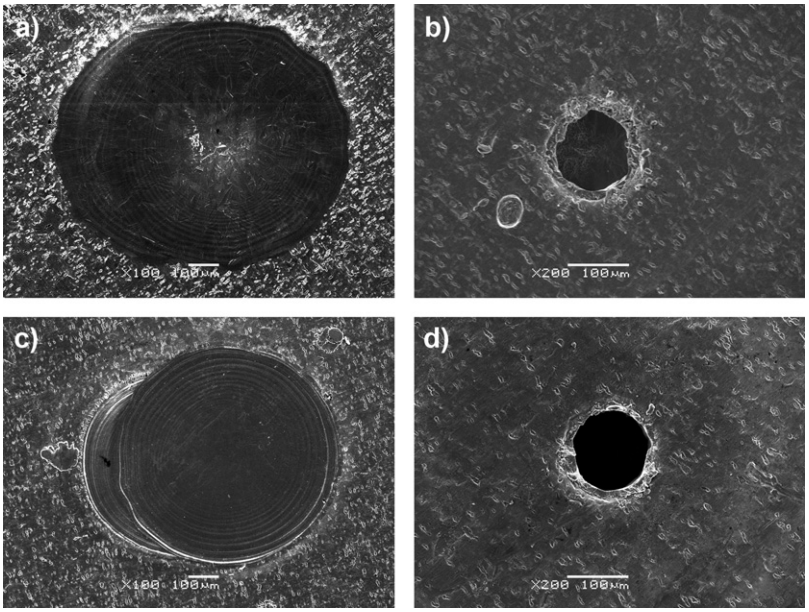

Fig. 4. Top (a) and bottom (b) surfaces of welds made with 0.8 kW peak power and 5 ms pulse without electrolyte. Top (c) and bottom (d) of weld made with electrolyte.

found to be acceptable in this study. No signs of excessive oxidation were observed under these shielding conditions.

After sealing, the Ti discs were cut into smaller pieces for observation using the scanning electron microscope (SEM) or for mounting in epoxy for optical microscopy. The samples mounted in epoxy were carefully cross-sectioned to the weld center then polished. A 9:1 mixture of colloidal silica and hydrogen peroxide 30% was used for polishing. To reveal the microstructure of the cross-sectioned samples an etchant composed of 10% hydrofluoric acid, 45% nitric acid and 45% water was used.

The hardness of the Ti welds was measured using a Shimadzu HMV-2000 Vickers hardness tester. A suitable load and dwell time for these tests was found to be 100 gf and 15 s respectively. Hardness indents were made along the welded region as illustrated in Fig. 2. A modified procedure based on the American Society for Testing and Materials (ASTM) standard E384-10e2 for hardness testing was used in this study. In order to get a high enough resolution of hardness measurements on such a small sample, the indents were made closer together than the 2.5 indentation width specified in the standard.

In order to rule out the hardening effect of secondary phases produced by the segregation of interstitial elements, a micro XRD analysis was performed. A Rigaku ACF-8 XRD was used in this study with a Cu Kα X-ray element. A beam size of 300 μm, with a voltage and current of 50 kV and 40 mA, respectively, were used.

3. Results

3.1. Surface morphology

SEM images of the top and bottom surfaces of the welds are shown in Figs. 3–6 for welds made both with and without

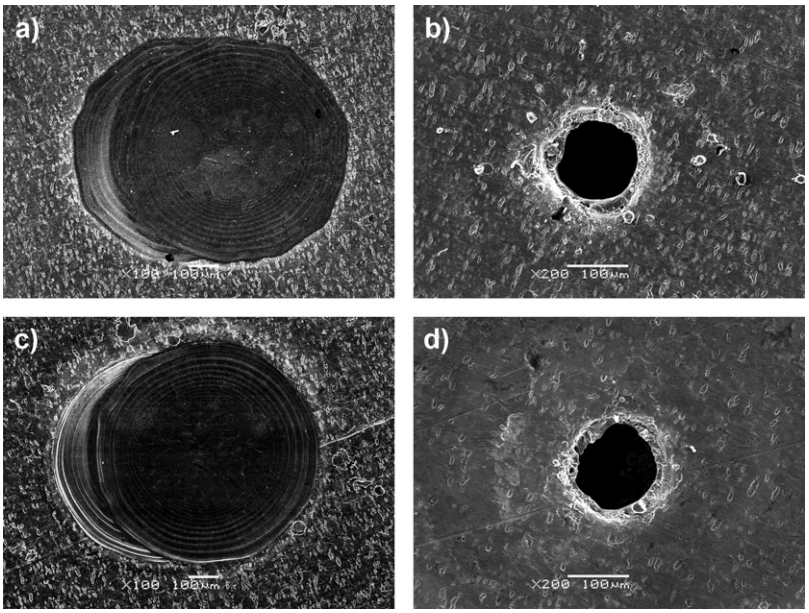

Fig. 5. Top (a) and bottom (b) surfaces of welds made with 1.0 kW peak power and 2 ms pulse without electrolyte. Top (c) and bottom (d) of weld made with electrolyte.

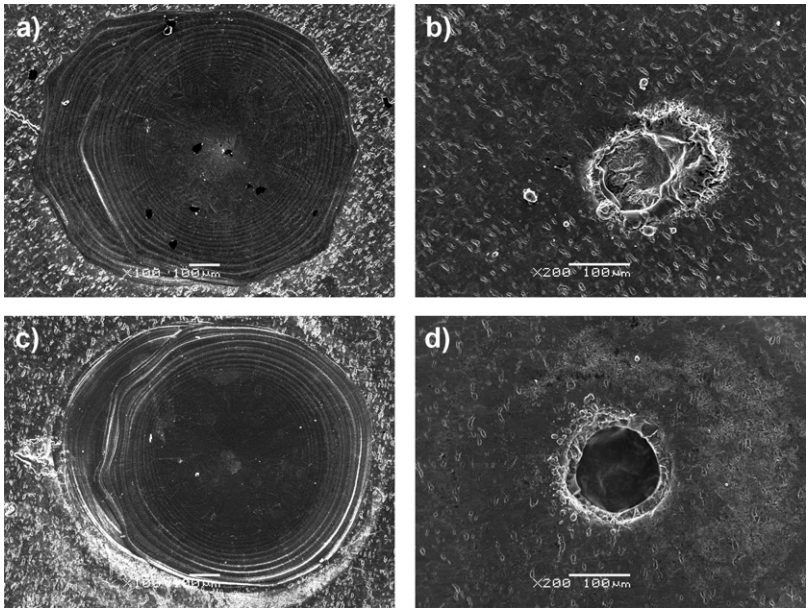

Fig. 6. Top (a) and bottom (b) surfaces of welds made with 1.0 kW peak power and 5 ms pulse without electrolyte. Top (c) and bottom (d) of weld made with electrolyte.

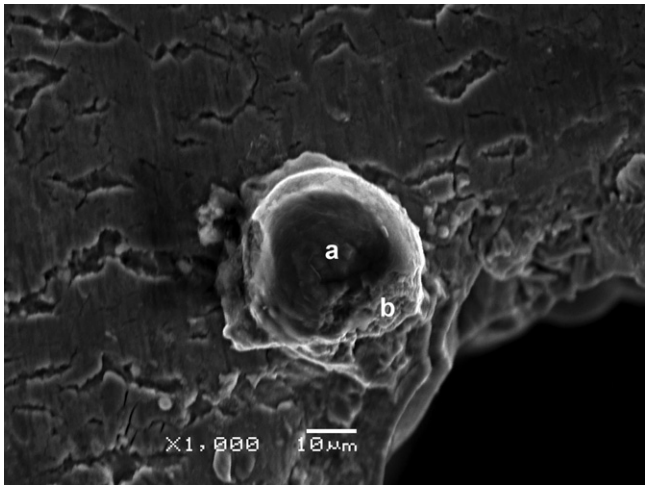

Fig. 7. Weld spatter on bottom of weld made with 0.8 kW peak power, 2 ms pulse, and electrolyte; EDS analysis at locations (a) and (b).

Table 2
EDS analysis results of weld spatter presented in Fig. 7 [wt.%].

Analyzed area	O	F	Ti
a	8.73	0.67	90.60
b	10.11	6.57	83.32

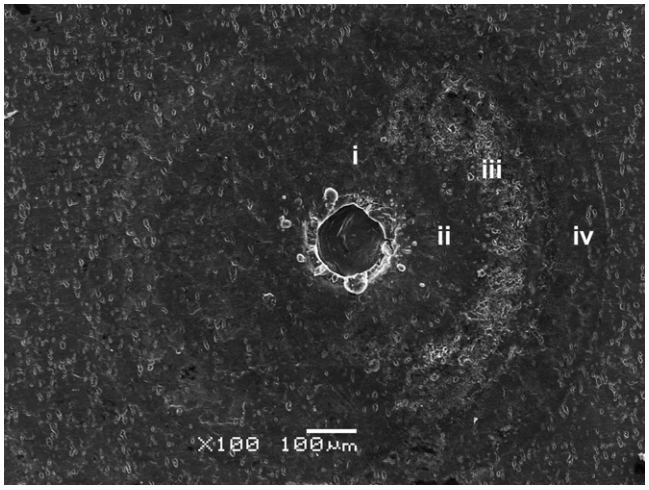

Fig. 8. Lower magnification image of bottom of weld (1.0 kW × 5 ms) showing ring around hole (i.e. SHAZ) after welding with electrolyte present; EDS analysis at locations (i)–(iv).

electrolyte present. Sealing was found to be possible with each of the parameter sets selected for this study. Full penetration, as observed by Huang et al. (in press), was never achieved due to the greater thickness of the Ti discs used in this study. The weld quality was found to be excellent for all welds having produced a hermetic seal with limited defects such as weld spatter. The limited amount of spatter that was observed on the bottom side of the welds was analyzed using EDS analysis. An EDS analysis of the spatter was performed as shown in Fig. 7. The EDS results are given in Table 2. This spatter was observed in both welds made with and without electrolyte as shown in Figs. 3–6. However, elevated levels of F along with higher levels of O were identified in samples welded in the presence of the electrolyte.

An interesting observation that was made from SEM images of the weld surfaces was an annular ring of different contrast surrounding the bottom of the sealed hole for samples welded in the presence of electrolyte. This ring was easily seen in a lower magnification image of the bottom side of the weld as shown in Fig. 8. An EDS analysis of this region compared to a region away from the weld showed that this ring was in fact a surface heat affected zone (SHAZ) where larger amounts of O and F were detected. The regions analyzed using EDS are shown in Fig. 8 and the EDS results are presented in Table 3.

3.2. Weld cross-sections

Optical micrographs of weld cross-sections are shown in Fig. 9. Sealing was found to be possible with all selected parameters both with and without electrolyte. Penetration increased with laser energy but full penetration was never achieved where, less penetration was observed in the samples welded in the presence of electrolyte. A fine acicular alpha microstructure (dark etching) was

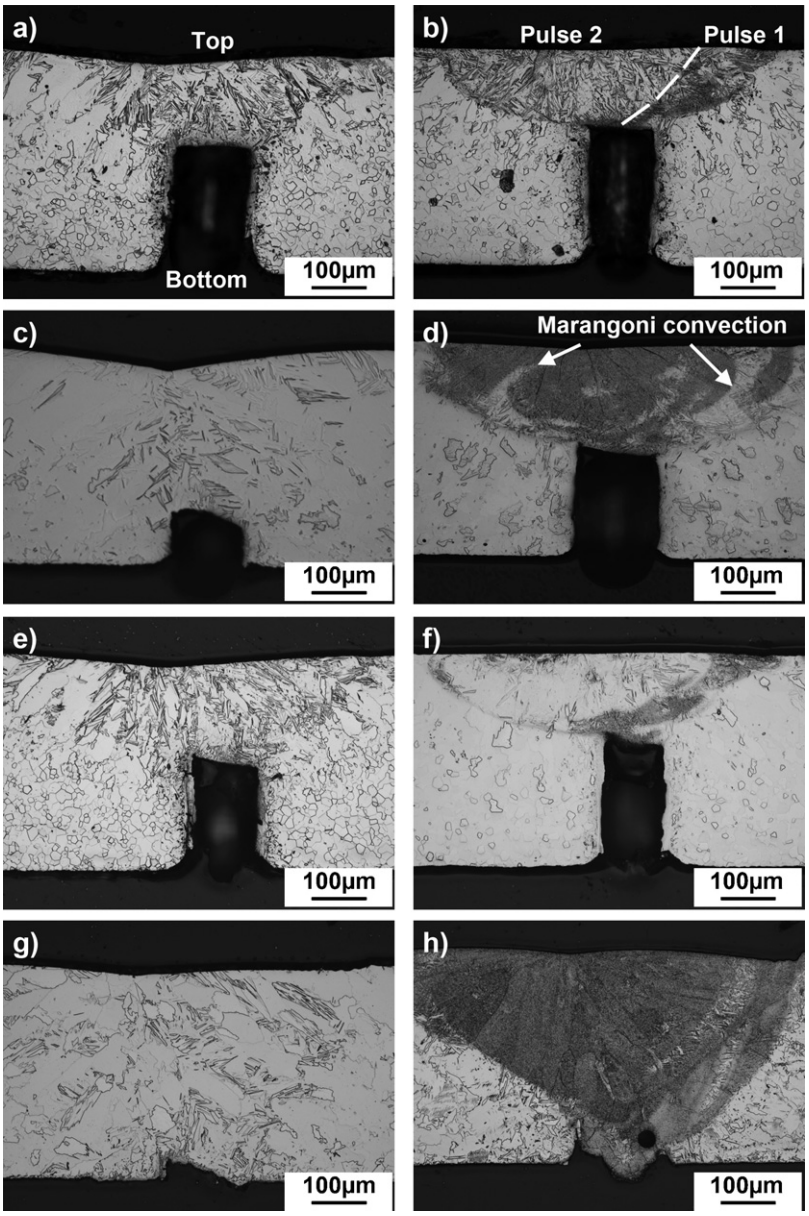

Fig. 9. Cross-sections of laser sealed holes. 0.8 kW peak power, 2 ms pulse duration (a and b), 0.8 kW peak power, 5 ms pulse duration (c and d), 1.0 kW peak power, 2 ms pulse duration (e and f), and 1.0 kW peak power, 5 ms pulse duration (g and h). Welds made without electrolyte (a, c, e, and g) and with electrolyte (b, d, f, and h).

Table 3
EDS analysis results of SHAZ on the back side of the specimen presented in Fig. 8 [wt.%].

Analyzed area	O	F	Ti
i	22.67	3.57	73.76
ii	17.05	3.95	79.0
iii	23.55	4.48	71.97
iv	22.97	3.04	73.99
Away from weld	2.27	0.80	96.95

identified in all samples welded with electrolyte present. This structure was identified in the first pulse of the 2 ms pulse samples (Fig. 9(b) and (f)) and was present throughout the weld when using the 5 ms pulse conditions (Fig. 9(d) and (h)).

3.3. Microhardness

The Vickers hardness results for welds made with and without electrolyte are shown in Fig. 10(a)–(d). A significant increase in hardness was found for all welds when produced with the presence of electrolyte. The hardness of the samples welded with electrolyte was found to vary across the weld. This was directly correlated with the heterogeneous microstructure in the weld metal observed in the previous section. This made calculating an average hardness of a sample ineffective and as a result comparison with other welds was difficult.

4. Discussion

Since the material used in this study was CP1 Ti with an equiaxed alpha microstructure as shown in Fig. 11, possible hardening mechanisms in samples welded in the presence of electrolyte include: (i) increasing the composition of interstitial elements; (ii) introduction of secondary phases; and (iii) refining the microstructure. Similar hardening mechanisms have been described by Chai and Chou (1998), in the laser welding of cast CP Ti.

Microstructure of welds made in the absence of electrolyte was found to be a mixture of serrated alpha, platelet alpha, and

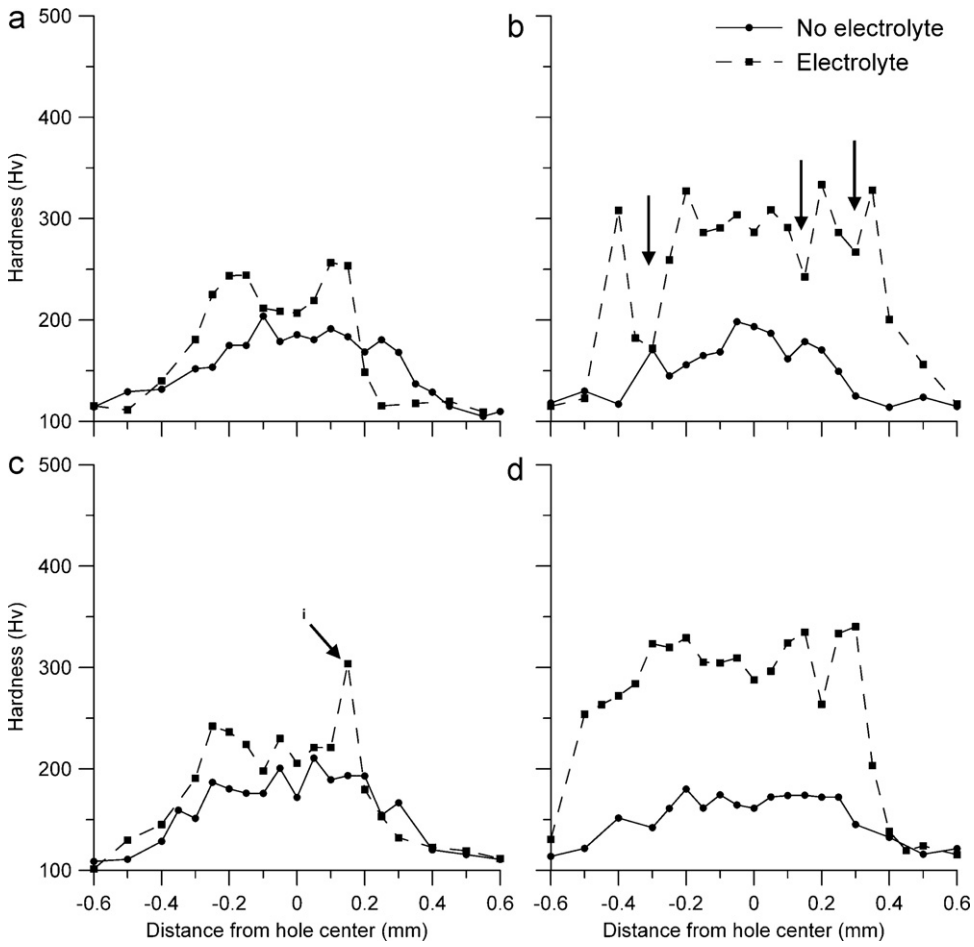

Fig. 10. Vickers hardness test results for weld without electrolyte and with electrolyte in 0.8 kW peak power and 2 ms pulse duration (a), 0.8 kW and 5 ms (b), 1.0 kW and 2 ms (c), and 1.0 kW and 5 ms (d).

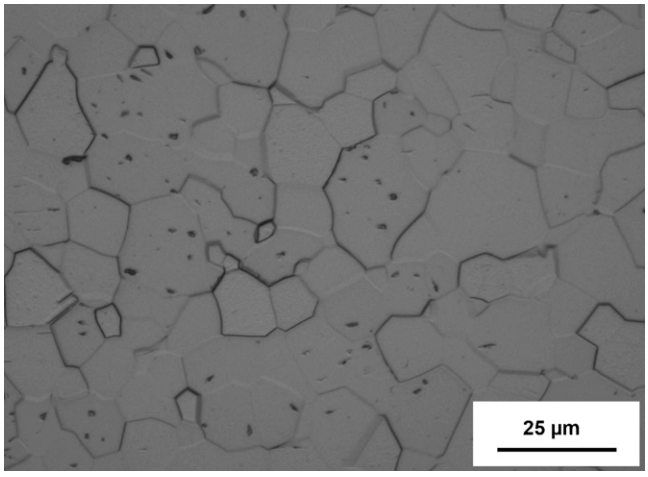

Fig. 11. Cross-section of base metal showing equiaxed alpha structure.

acicular alpha phases as shown in Fig. 12(a). In contrast, welds made in the presence of electrolyte contained noticeably finer microstructure and consisted of more acicular alpha as shown in Fig. 12(b). Though variations in weld metal hardness made comparisons between welds difficult, correlations between weld microstructure and hardness were easily made. For example when considering the valleys in the hardness trend indicated by arrows in Fig. 10(b) and the respective microstructure at the location where hardness indents were made from Fig. 9(d), the microstructures is clearly quite different. These regions of lower hardness have a light etching coarser microstructure. Indent (i) from Fig. 10(c) was made in the fine microstructure of the first pulse observed in Fig. 9(f), consisting mainly of the dark etching colonized acicular alpha phase. Therefore, a finer microstructure, or a microstructure that consists of a larger amount of the acicular alpha phase, was found to have higher hardness. Similar phenomenon was observed in a study conducted by Li et al. (2005), on the pulsed Nd:YAG laser welding of CP Ti, where a change in microstructure occurred with the increased O content in the shielding gas. The increasing amount of fine acicular alpha in the microstructure with increasing O content was also found to correlate well with the increased hardness of the weld metal.

As observed in Fig. 9(b) and (d) the ultra-fine colonized acicular alpha structure (dark etching) was formed in either the first pulse or mixed throughout the whole weld. This finer structure in the first pulse of poorly mixed samples was a result of a greater interaction of the first pulse weld metal with electrolyte. Before the initial laser pulse, an electrolyte pool was present on the top surface of the Ti disc. Also, since the hole was not sealed, weld metal directly interacts with the electrolyte. Prior to the second pulse, electrolyte is no longer on the top surface of the Ti disc since it vaporized during the first pulse and the hole was already sealed. Mixing of the weld metal can however lead to the finer structure occurring across the whole weld after both pulses. Weld pool mixing was attributed to Marangoni convection which is known to promote mixing with longer pulse durations, as observed by the more

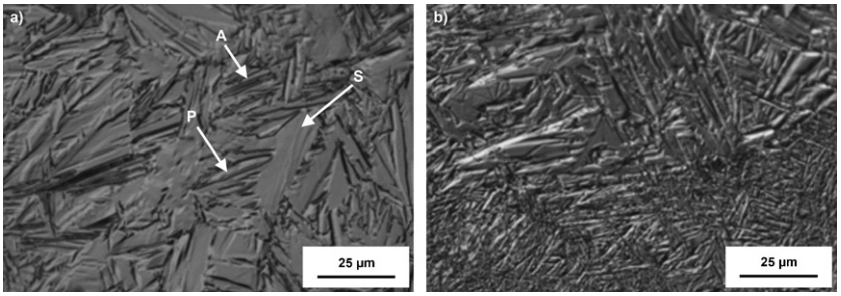

Fig. 12. Microstructure of weld made with 1.0 kW peak power and 2 ms pulse duration without electrolyte (a) and with electrolyte in the dark etching region (b).

uniform microstructures. The flow of the molten pool was identified by observing mixing of the dark and light etching structures in the weld cross-sections shown in Fig. 9(d) and (h).

Different interstitial elements interact with Ti on various degrees which include alpha/beta phase stabilization, grain refinement, and introduction of lattice strain. Simbi and Scully (1996) reported that the interstitial elements O, N, and C as well as the addition of Fe increase the tensile strength and hardness of CP Ti by beta stabilization, grain refinement, introducing lattice strain and solid solution hardening. However, as discussed by Lutjering and Williams (2007), some alloying elements are utilized more than others due to the solubility limits, density and other factors. Oxygen is the most common interstitial element cited for influencing the microstructure and hardness of Ti welds. Oxygen is also added as an alloying element to CP Ti to increase the yield strength.

Even with shielding the top side of the weld with Ar and having electrolyte below, a significant increase in hardness was observed along with a relatively finer microstructure compared to that of the welds made without electrolyte. The electrolyte mediums used in this study contain C, H, O, and F which can all occupy interstitial sites in the Ti lattice as discussed above. In the EDX analysis (Table 3) of the SHAZ on the bottom side of the welds, an increased level of O and small amount of F were found. A SHAZ was not found in the samples welded without electrolyte. The identification of increased levels of O, and F and the change in weld microstructure to ultrafine colonized acicular alpha in the samples made with electrolyte present suggests that the electrolyte does in fact react with the weld metal to some degree. EDS analysis of weld cross-sections was unable to detect significant differences in interstitial elements such as O and F in the samples welded with or without electrolyte. This was likely due to the inability of EDS to accurately quantify light element composition and the small (i.e. <0.1 wt.%) change in interstitial elements required to cause significant increases in the hardness of CP Ti alloys.

Understanding the chemical stability of a liquid is critical when studying the reaction between the liquid and metal such as in laser hole sealing or in the more common water-assisted laser processes. The molecules in a liquid at high temperature become unstable and undergo chemical reactions that may influence the targeted site. During laser processing, constituents in a liquid that are stable at room temperature can heat up to thousands of degrees and can be excited, ionized, or dissociated and thereby become chemically active. For example, a reaction observed during laser ablation in different media such as water, benzene, n-hexane, carbon tetrachloride and in Ar–H atmospheres leads to oxidation of metallic surfaces as reported by Kruusing (2004). Also, Dupont et al. (1995) reported that the dissociation of water molecules supplies the free oxygen radicals that can be interstitially embed into the weld metal and thereby increase the hardness. Additionally, Ashokkumar (1998) has identified TiO_2 as an active photocatalysts of water dissociation, which may translate to issues in the laser

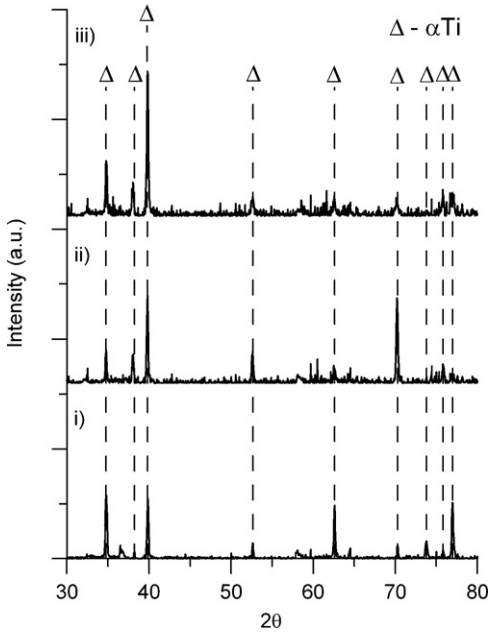

Fig. 13. XRD analysis results showing only alpha Ti in base metal (i) as well as in welds made with (iii) and without electrolyte (ii) present.

sealing of Ti components. Another reported reaction identified by Ageev et al. (1997) was the formation of titanium nitride when exposing Ti to ammonia and hitting it with a 2 ms Nd:YAG laser pulse.

In addition to interstitial hardening, the segregation of interstitials and the formation of a solid solution was also a possible hardening mechanism. Micro X-ray diffraction analysis was used to determine whether secondary phases were formed in the weld microstructure. Samples welded with 1.0 kW peak power and 5 ms pulse duration, shown in Fig. 9(g) and (h), were analyzed in the XRD analysis. These samples were chosen due to the large difference in microstructure and significant increase in hardness of the sample welded with electrolyte. The XRD results for the base metal and welds made both with and without electrolyte present are shown in Fig. 13. Only alpha Ti was found in this analysis which suggests that significant volume fractions of secondary phases such as oxides, nitrides, carbides, hydrides and fluorides are not present in the weld metal. Therefore, secondary phases do not significantly contribute to the increased hardness of the welds made with electrolyte.

A certain degree of hardening in the welds made with electrolyte can simply be a result of the electrolyte aiding in the dissipation of heat through conduction. The presence of electrolyte can change the cooling rate having a large effect on the microstructure of the solidified weld pool. Higher cooling rates leads to a finer structure

and higher residual stresses as discussed by Kou (2003). In laser welding the cooling rates are extremely high (i.e. $\sim 10^3$–10^5 °C/s) as indicated in the ASM Handbook (1993). These cooling rates are comparable to those in water quenching, as identified by Baeslack and Mullins (1982). Donachie (2000) showed that water quenching CP Ti produces an irregular alpha microstructure which is similar to what was observed in the laser welds made without electrolyte in this study. The welds made with electrolyte present are expected to have a higher cooling rate and therefore a finer and harder microstructure following the Hall–Petch relationship. Evidence of heat dissipation through the electrolyte was observed in full penetration welds by Huang et al. (in press), where changes in the direction of dendrite growth was observed during laser hole sealing in the presence of electrolyte. It is difficult to distinguish between the grain refinement caused by higher cooling rates to that caused by an increased amount of interstitial elements. However, higher hardness was measured in welds made with longer pulse times which implied higher peak temperatures and lower cooling rates. Therefore, higher cooling rates cannot be the primary mechanism responsible for the increasing hardness. In addition, the higher peak temperatures and longer pulse times facilitate reaction between the electrolyte and the molten weld pool further suggesting an interstitial hardening mechanism.

5. Conclusions

In this study it was found that the electrolyte has a significant effect on the properties of the CP1 Ti weld metal following the laser hole sealing process. It was suggested that the weld metal interacts with the different components of the electrolyte subsequently changing the composition and microstructure of the Ti weld metal. The main conclusions include the following:

1. Following laser hole sealing the hardness of the weld was found to be significantly higher in samples made in the presence of an electrolyte medium. The hardness within the weld region was found to correlate well with the weld microstructure where, the hardest regions consisted of an ultra-fine colonized acicular alpha structure.
2. In the welds produced with the presence of electrolyte the hardest regions, consisting of ultra-fine colonized acicular alpha microstructure, were either found in only the first pulse or mixed throughout the weld. At longer pulse durations better mixing in the weld pool was identified by a more uniform microstructure.
3. A SHAZ was identified on the bottom side of welds made in the presence of electrolyte. Greater compositions of O and F elements were identified in the SHAZ compared to the base material. The reaction of the CP1 Ti material with the elemental species derived from the electrolyte during laser hole sealing was suspected to produce the SHAZ.
4. No secondary phases were identified in the welded region when laser hole sealing with or without electrolyte present. The weld regions were found to consist of only alpha Ti. Therefore, the presence of secondary phases was determined not to have a significantly affect the hardness of the welds.
5. Increased cooling rates caused by thermal conduction through the electrolyte were suspected to influence the microstructure and hardness. However, changes in the cooling rate were not the primary mechanism behind the increased hardness.

Acknowledgements

This work has been funded by Program of Canada Research Chairs (CRC) in Microjoining (www.crc.gc.ca). A special thanks to Billy Tam for helping with the initial set-up and fixturing required for this work. The guidance and technical support received from Michael Lybarger, Pankaj Mohan, Shawn Johnson, and Paul Ray was also greatly appreciated.

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