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Laser hole sealing of commercially pure grade 1 titanium

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Hermetically sealed Ti capsules filled with electrolyte solutions are required in many medical device applications. The laser hole sealing process is well suited for this type of application. There is, however, a lack of understanding of the laser hole sealing mechanism, especially in the presence of an electrolyte medium. In this study, the mechanism of the laser hole sealing process was investigated by characterizing the surface morphology and cross-sections of welds made both with and without electrolyte. It was shown that the laser sealing mechanism transitions from (i) no sealing due to insufficient energy; (ii) the coalescence of the weld pool and the onset of sealing; (iii) increasing penetration depth up to full penetration; and (iv) laser ablation and drilling. Laser hole sealing in the presence of electrolyte decreases the size of the process window for suitable laser energies and affects the microstructure of the sealed hole. © *2012 Laser Institute of America.*

Key words: laser hole sealing, titanium, hermetic sealing, electrolyte medium, sealing mechanism, microstructure

I. INTRODUCTION

The laser hole sealing process is a novel, quick, and reliable technique developed for the sealing of small holes (i.e., 150–225 μm in diameter). Sealing of commercially pure grade 1 (CP1) titanium (Ti) capsules is often required for hermetically sealing medical device components that are filled with an electrolyte medium. In this application, commercially pure Ti is extensively used in medical device applications because it provides excellent combinations of corrosion resistance, mechanical properties, and biocompatibility.[1–4]

Laser microwelding (LMW) processes have been widely applied to manufacturing various medical devices. The extensive use of LMW in the medical device industry is a result of many advantages over competing joining technologies. Some of these advantages include high precision, no contact with work pieces (i.e., limited contamination), small heat affected zone (HAZ), and highly consistent reliable joints.[1,2] Although there is a substantial literature detailing the LMW processes, to the authors knowledge, no literature on the laser sealing of holes exists to date. The laser hole sealing process is very complex with many factors such as the energy input to the weld and forces that act on the weld pool determining whether a quality hermetic seal can be produced. A better understanding of the sealing mechanism will aid in the development of procedures that ensure sound hermetic welds are being produced. The objective of the current work was to characterize the surface morphology and cross-sections of welds made both with and without electrolyte using the laser hole sealing process. The effect of electrolyte on the final microstructure was of particular interest and will be introduced in this study.

II. EXPERIMENT

In this study, laser hole sealing was performed on CP1 Ti discs both with and without the presence of an electrolyte medium. The properties and chemical composition of CP1 Ti are given in Table I. The electrolyte used in this work was composed of a mixture of de-ionized water and organic compounds, containing carbon (C), hydrogen (H), oxygen (O), and nitrogen (N). Ti test disc were used in this study having a thickness of 200 μm and each containing 16 test holes of 150 μm diameter.

A welding fixture was designed 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 process. The reservoir in the fixture base was sealed to the Ti test disc via

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TABLE I. 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

an o-ring. This configuration allowed for the simulation of a hermetic sealing process. The 2 ml reservoir below the Ti disc was filled with electrolyte before sealing using a syringe. A consistent amount of electrolyte was injected into the reservoir until the electrolyte was observed to protrude from the 16 holes in the Ti disc, as illustrated in Fig. 1.

Laser hole sealing was performed using a Miyachi Unitek LW50 pulsed Nd:YAG microlaser 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 purpose of the first pulse is to seal the hole and evaporate any electrolyte on the surface. Following the first pulse, the repeatability of the joint is not acceptable and the joint geometry is not ideal, where a crater was often present at the pulse center and the penetration or sealing depth varies. Therefore, the purpose of the second pulse is to improve the weld surface condition and ensured a repeatable and quality seal is produced. It also was found in unpublished work that by firing the first pulse at 0° and the second at 180° relative to the hole center, as illustrated in Fig. 2, a more favorable surface condition is achieved where a flat weld surface is possible. Therefore, this double pulse offset hole sealing protocol was adopted in this study. The sealing 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 0.5 to 1.0 kW, and the pulse duration was varied from 2 to 5 ms. Each parameter set was repeated both with and without electrolyte. Due to the highly reactive nature of Ti a protective atmosphere was necessary during sealing. 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 found to be sufficient in this study; no signs of oxidation were observed under these shielding conditions.

After sealing, the Ti discs were cut into smaller pieces for observing 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 center of the sealed holes then polished in preparation for optical microscopy. A 9:1 mixture of colloidal silica and 30% diluted hydrogen peroxide 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.

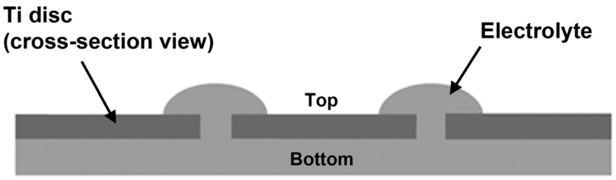

FIG. 1. Electrolyte protruding through holes in Ti disc prior to hole sealing (ideal situation with same amount of electrolyte protruding from each hole).

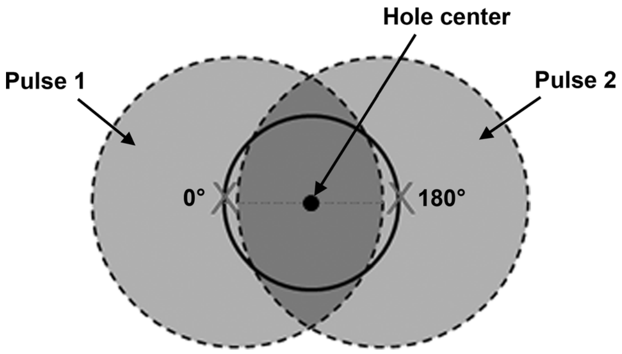

FIG. 2. Illustration of welding procedure with pulse 1 at 0° and pulse 2 at 180° relative to the hole center (not to scale).

III. RESULTS

A. Surface morphology of welds

SEM images of the top and bottom surfaces of representative welds are shown in Figs. 3–6 made both with and without electrolyte. In general, the weld quality was found to be excellent for all welds where sealing was found to be possible. Limited amounts of spatter or expulsion were observed on the surfaces of these welds.

For the welds made without electrolyte and the lowest peak power and pulse duration of 0.5 kW and 2 ms, respectively, the hole was sealed as shown in Fig. 3(a). However, when electrolyte was present using these same parameters, sealing was not possible, as shown in Fig. 3(c). Full penetration of the 200 μm thick Ti disc was found to be possible at higher energy levels, as shown in Figs. 4–6. At the highest energy setting with peak power and pulse duration of 1.0 kW and 5 ms, respectively, laser drilling begins to occur when electrolyte was present. Approximately, 50% of welds made with electrolyte were not sealed at these welding parameters, as shown in Fig. 6(e). Sealing was, therefore, determined not to be possible at this high energy parameter set when electrolyte was present.

A directional solidification structure was clearly observed in full penetration welds made without electrolyte as shown in Figs. 4(b), 5(b), and 6(b). This was not the case in welds made with electrolyte as shown in Figs. 4(d), 5(d), and 6(d).

B. Weld cross-sections

Optical micrographs of weld cross-sections are shown in Figs. 7–10. As observed in the SEM images of the weld surfaces from Sec. III A, consistent sealing was not possible when using either the low or high laser energy parameter sets with electrolyte [Figs. 7(b) and 10(c)]. Full penetration was clearly observed in the experiments where the hole has been completely melted, as shown in Figs. 8–10.

The weld microstructure can also be determined from the weld cross-sections providing more insight into the

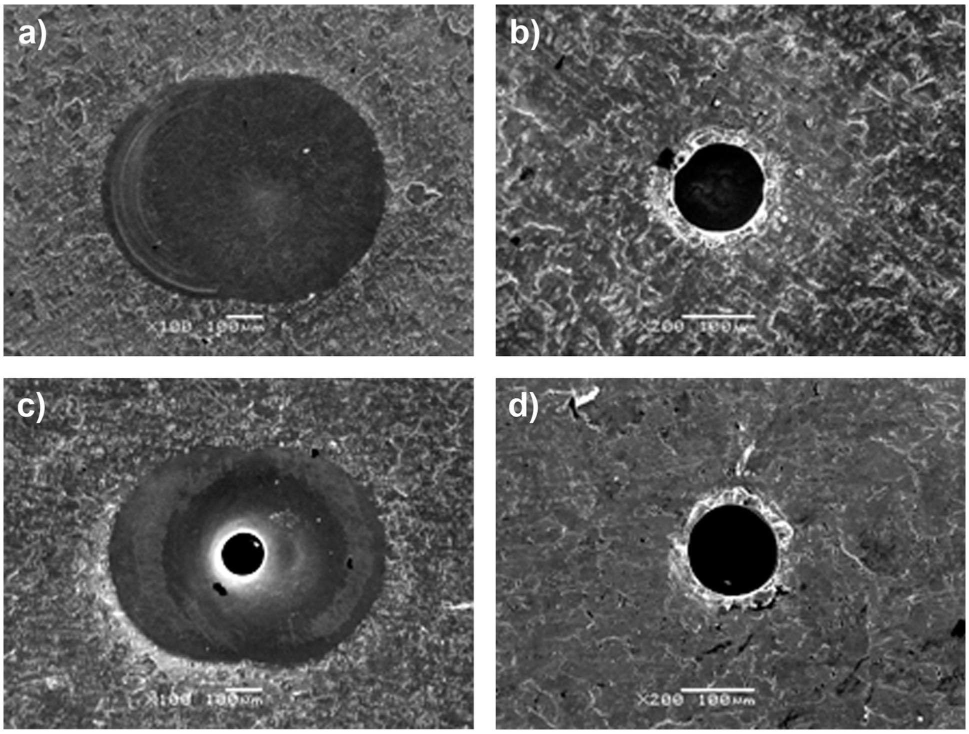

FIG. 3. Top (a) and bottom (b) surfaces of welds made with 0.5 kW peak power and 2 ms pulse without electrolyte. Top (c) and bottom (d) of welds made with electrolyte.

effects of electrolyte on weld metal properties. The CP1 Ti base metal microstructure consists of an equiaxed alpha phase as shown in Fig. 11(a). After sealing, however, the microstructure was observed to change. The weld metal produced with no electrolyte consists of a mixture of serrated alpha (S), platelet alpha (P), and acicular alpha (A) as shown in Fig. 11(b). In welds produced with electrolyte, the microstructure contains mostly acicular alpha with some of the platelet alpha phase mixed in, as shown in Fig. 11(c). It was also clearly observed that microstructure gets finer when electrolyte was present. In certain areas of welds made with electrolyte, the acicular alpha phase forms colonies which

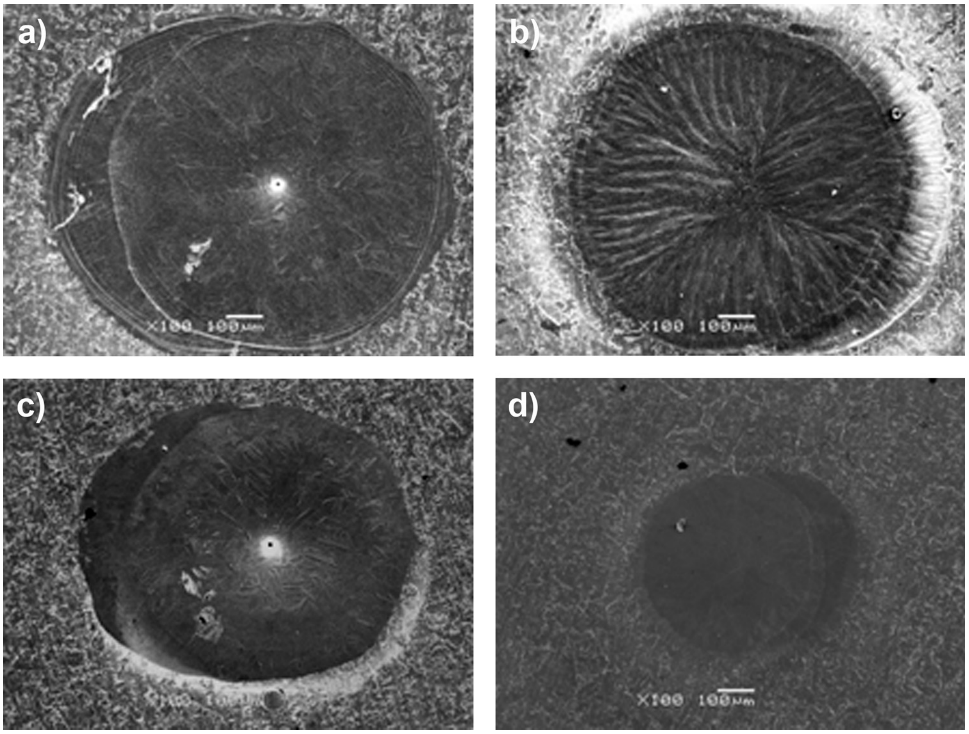

FIG. 4. Top (a) and bottom (b) surfaces of welds made with 0.5 kW peak power and 5 ms pulse without electrolyte. Top (c) and bottom (d) of welds made with electrolyte.

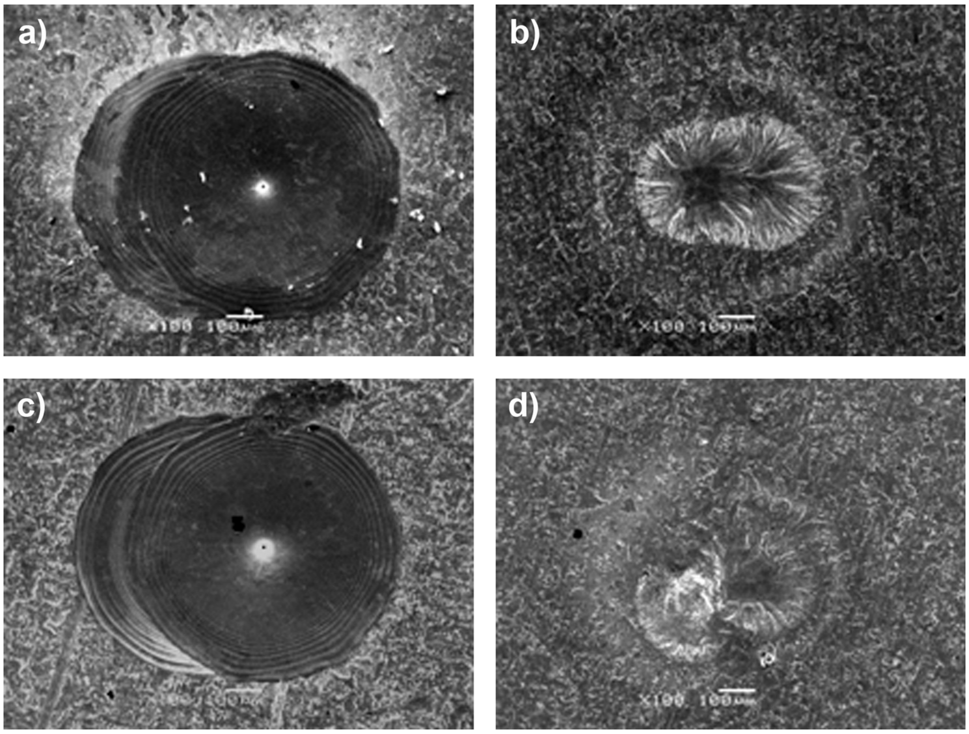

FIG. 5. Top (a) and bottom (b) surfaces of welds made with 1.0 kW peak power and 2 ms pulse without electrolyte. Top (c) and bottom (d) of welds made with electrolyte.

have a “basket weave” type appearance. In the finest regions, this colonized alpha structure became ultrafine and etches very dark, as shown in Fig. 11(d).

IV. DISCUSSIONS

A. Sealing mechanisms

The mechanisms behind laser hole sealing are complex. There are multiple factors that will affect laser sealing such as the energy input to the weld and forces that act on the weld pool. Some forces that act on the weld pool include gravity, surface tension, convection, and laser forces such as expanding gasses and plasma forces.[5] All of these factors influence whether sealing occurs and the quality of the weld produced. In this section, a physical model is developed through observing the evolution in weld surface conditions and cross-sections with increasing laser energy, providing a platform for further research into the laser hole sealing mechanisms. The illustrations shown in Fig. 12 will aid in this discussion.

As the energy was increased, the volume of molten metal also increased in the fusion zone, as illustrated in Figs. 12(a)–12(f). At both the upper and lower energy limits, no sealing was possible. At the lower energy limit (i.e., 0.5 kW × 2 ms) with electrolyte, insufficient melt volume prevented sealing, as shown in Figs. 3(c) and 7(b). Only the top edge of the hole was melted in this low energy condition [Fig. 12(a)]. There are several different phenomena that contribute to energy losses in the laser sealing process, each of which will reduce the amount of energy available for melting and can prevent sealing. These different energy losses include reflected laser energy, absorbed laser energy by electrolyte, heat conduction through electrolyte, and heat conducted through the Ti disc. Since the electrolyte contributes to energy losses through several different mechanisms, higher energy losses are expected. For example, evidence of the dissipation of energy through the electrolyte was observed in the SEM images of the weld surfaces. A directional dendritic solidification structure was clearly observed in full penetration welds made without electrolyte [Figs. 4(b), 5(b), and 6(b)]. Dendrites grow in the direction of highest temperature gradient, where in this case with no electrolyte, was perpendicular to the fusion boundary where heat was being extracted mainly by the Ti disc.[6] However, no directional solidification was observed in welds made with electrolyte because the electrolyte extracts a significant amount of heat changing the direction of the largest temperature gradient and dendrite growth direction. Therefore, these results confirm that the presence of electrolyte does affect energy losses where conduction of heat occurs through the electrolyte.

Sealing of the hole occurs when the molten material coalesces at the center of the hole, as illustrated in Fig. 12(b). No literature on laser hole sealing is available, but topics in literature investigating the effects of part fit-up or “gap” in laser welding are valuable references in researching laser hole sealing.[7] From literature, it has been found that a single pool is formed between two sheets when the two pools coalesce due to sufficient growth of the molten metal via thermal expansion or by the rounding of square corners. In situations where there is an appreciable gap, coalescing of pools may occur by pool motion. Decoalescence, however, will occur before solidification begins if two separate molten pools are more thermodynamically favorable.[8] Thermodynamic stability of a single pool depends on heat transfer, mass transfer, recoil pressure, and capillarity; the conditions

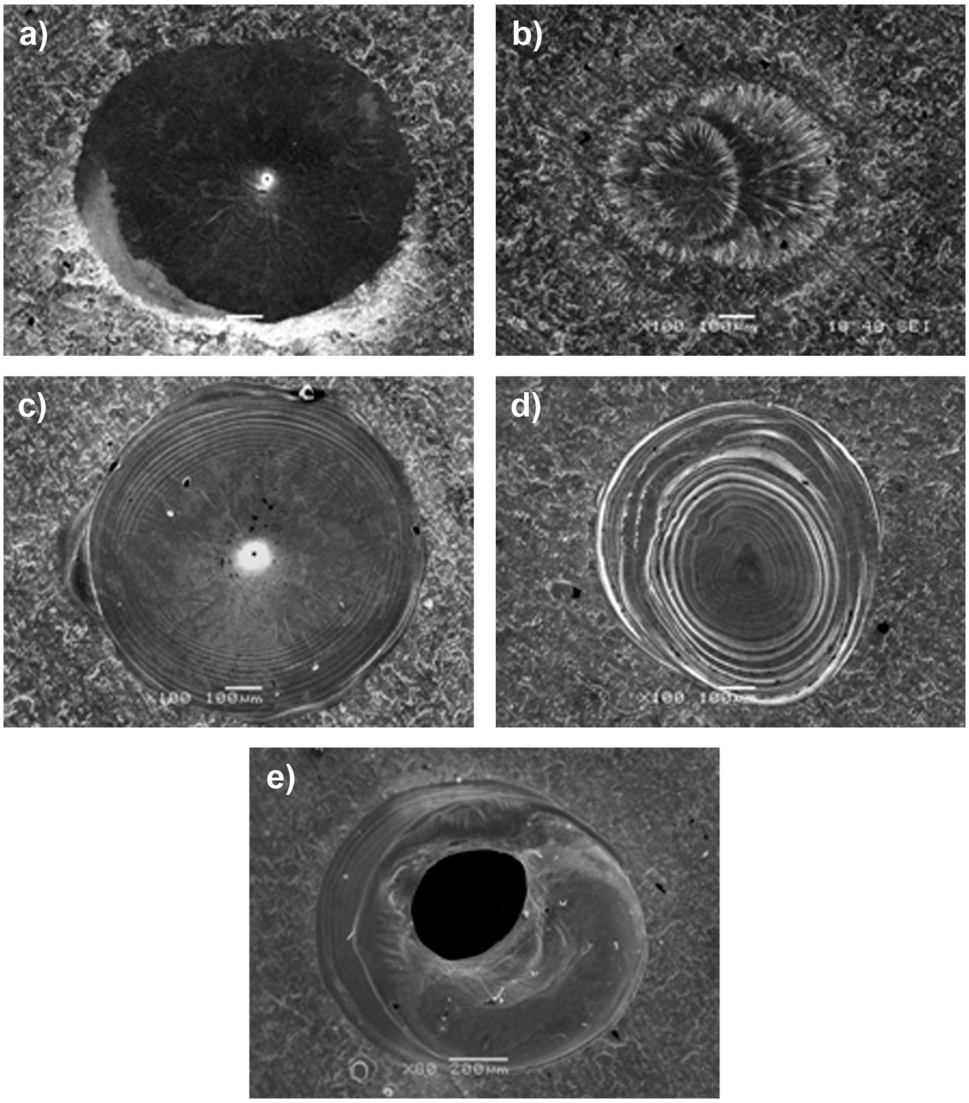

FIG. 6. Top (a) and bottom (b) surfaces of welds made with 1.0 kW peak power and 5 ms pulse without electrolyte. Top (c) and bottom (d) of welds made with electrolyte. Top (e) of weld made with electrolyte where sealing did not occur.

are found to be extremely dynamic.[8] In general, whether a hole or a sound weld is attained is a function of the fraction of material lost from spatter or ablation and the aspect ratio (i.e., material thickness divided by radius of molten pool).[8–10]

With increasing the energy further, the penetration depth increased until full penetration was obtained as illustrated in Figs. 12(b)–12(d). After full penetration was achieved the weld pool grows outward from the hole center with increasing laser energy [Fig. 12(e)]. It was also observed that the thickness of the weld cross-section also decreases slightly, as illustrated in Fig. 12(e). This loss of weld metal volume at high energy levels was attributed to laser ablation, where the Ti material is heated by the absorbed laser energy to a point

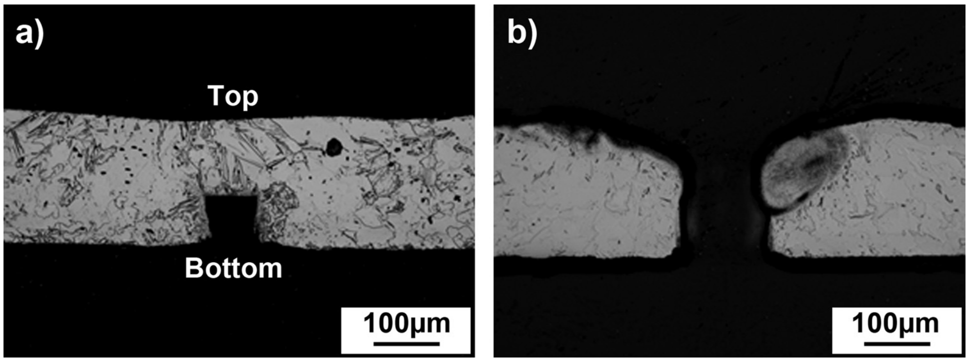

FIG. 7. Cross-section of weld made with 0.5 kW peak power and 2 ms pulse duration without electrolyte (a) and with electrolyte (b).

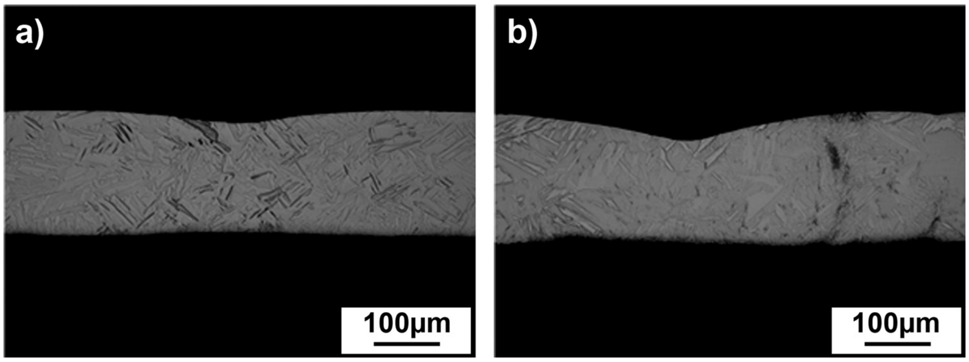

FIG. 8. Cross-section of weld made with 0.5 kW peak power and 5 ms pulse duration without electrolyte (a) and with electrolyte (b).

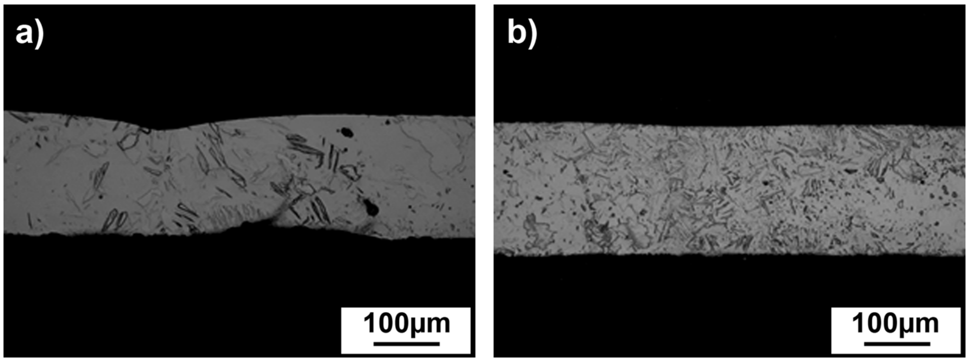

FIG. 9. Cross-section of weld made with 1.0 kW peak power and 2 ms pulse duration without electrolyte (a) and with electrolyte (b).

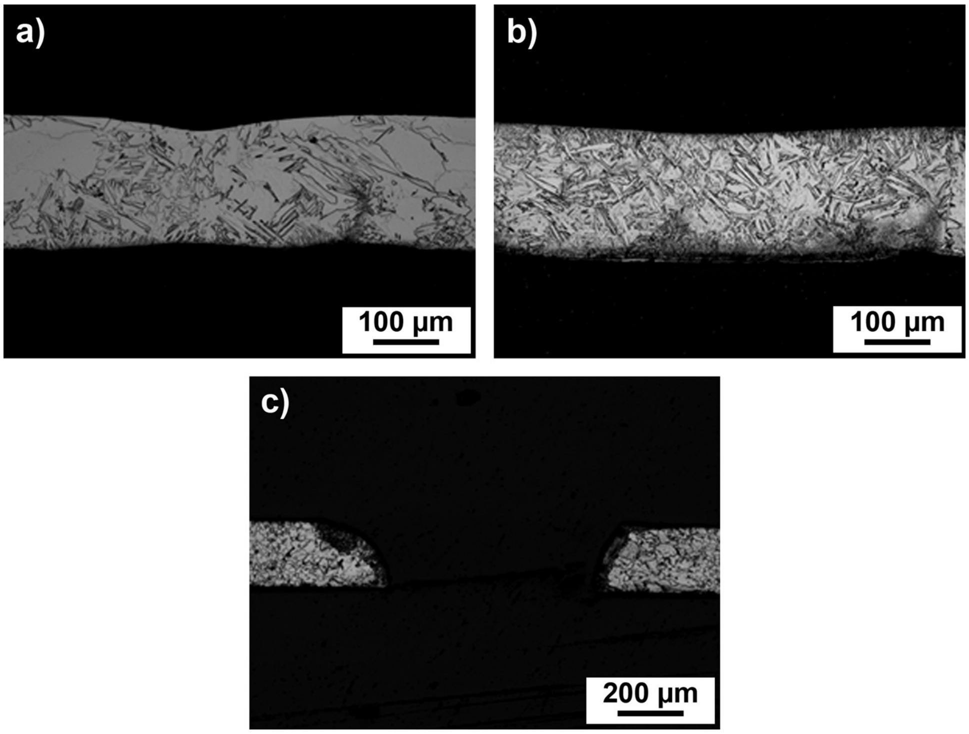

FIG. 10. Cross-section of weld made with 1.0 kW peak power and 5 ms pulse duration without electrolyte (a), with electrolyte sealed (b), and with electrolyte unsealed (c).

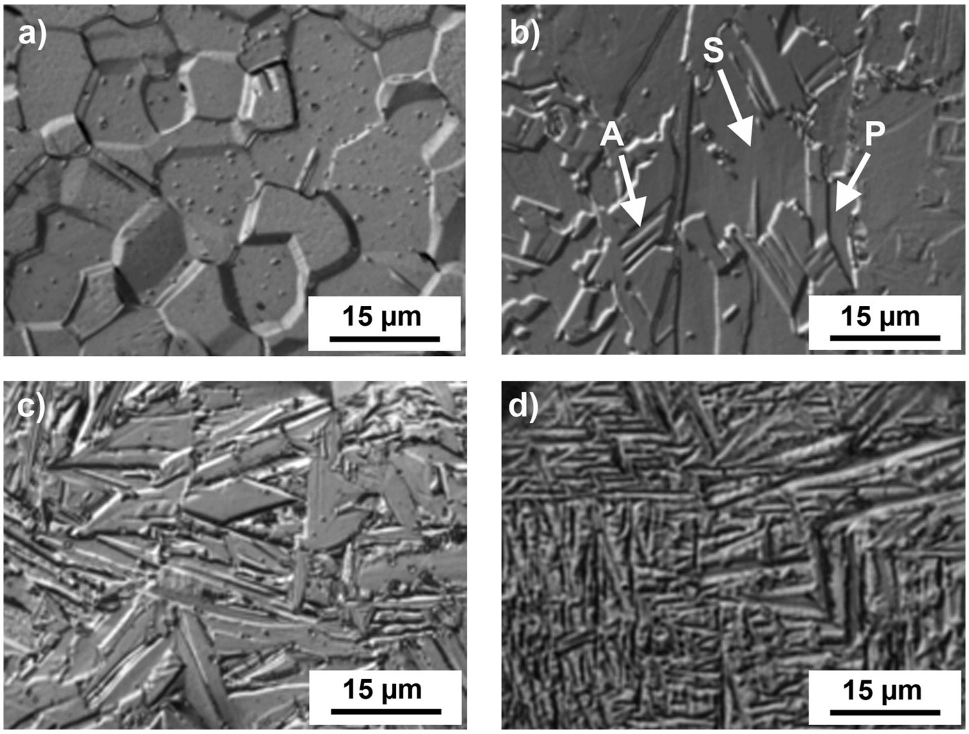

FIG. 11. Cross-section of base metal (a), 1.0 kW peak power and 5 ms pulse duration without electrolyte (b), 1.0 kW peak power and 5 ms pulse duration with electrolyte (c), and high magnification image of colonized acicular alpha from lower region (i.e., near bottom of test disc) of weld made with 1.0 kW peak power and 5 ms pulse duration with electrolyte (d).

where excessive evaporation and sublimation occur.[11,12] Eventually, the upper limit in energy input was reached where laser drilling begins, as illustrated in Fig. 12(f). Laser drilling was observed to occur only in the welds made with electrolyte using the highest welding parameters [Figs. 6(e) and 10(c)]. This result is consistent with literature,[13] where water was found to assist the drilling process. In laser drilling evaporation and high-pressures due to expanding gases and plasma contribute to the explosive removal of material with high enough energy input. However, in water assisted laser ablation process, it was found that the impact induced by the plasma is four times higher and the shock-wave is two–three times longer.[13] Cavitation will also occur in the liquid which will also assist in the removal of material and prevent laser sealing.[13] Therefore, the upper energy limit in the laser hole sealing process with electrolyte is lower than when no electrolyte is present.

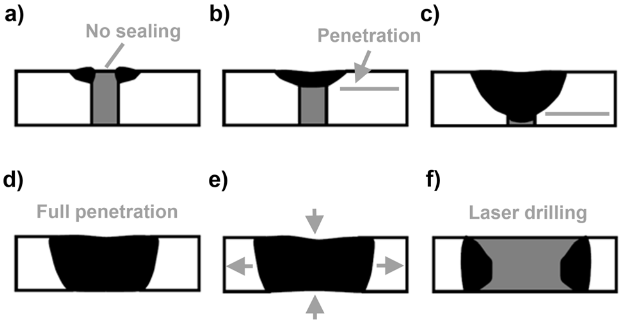

FIG. 12. Illustration of weld cross-section with increasing laser energy from (a) to (e). (a) No sealing due to insufficient melted volume; (b) onset of sealing; (c) increased weld penetration; (d) full penetration; (e) weld pool growth with full penetration; (f) laser ablation and drilling (not to scale).

In summary, with increasing energy the condition changes from (i) no sealing due to insufficient energy for melting; (ii) the coalescence of the weld pool where sealing begins; (iii) increasing penetration depth until full penetration is achieved; and (iv) laser ablation, which at high enough energy inputs leads to drilling and again sealing is not possible [Fig. 12].

B. Effects of electrolyte

In addition to narrowing the process window of the hole sealing process, the microstructure of the weld metal was also found to be affected by the presence of the electrolyte. A mixture of serrated alpha, platelet alpha, and acicular alpha phases were observed in samples produced without electrolyte. In welds produced with electrolyte, the microstructure was significantly finer and consisted mainly of acicular alpha. Similar microstructures were reported by Li *et al.*[4] in a study of laser sealing commercially pure Ti where increasing amounts of oxygen were supplied to the weld via the shielding gas. Therefore, it is possible that the electrolyte interacts with the weld metal during the sealing process providing interstitial contaminants. This difference in microstructure suggests differences in properties such as ductility, hardness, or even corrosion performance. The effects of electrolyte on the physical properties of the Ti weld metal are investigated in a second study.

V. CONCLUSIONS

The laser hole sealing process developed to hermetically seal Ti medical device components filled with an electrolyte medium was investigated thoroughly in this study. The knowledge obtained provides valuable insight into the laser hole sealing mechanism and serves as a platform for further research in this area. High quality repeatable welds were produced at all parameters where sealing was possible.

The process window in which hermetically sealing occurs was clearly identified, and valuable knowledge on the role of electrolyte in the sealing process was obtained. The presence of electrolyte was found to decrease the process window of the laser hole sealing process from both the upper and lower energy limits. The upper and lower limits of the sealing process with electrolyte were found to be 1.0 kW peak power and 5 ms pulse duration and 0.5 kW peak power and 2 ms pulse duration, respectively.

Differences in weld microstructure were also observed between welds made with and without electrolyte. A finer structure consisting predominantly of acicular alpha was found in weld metal of samples sealed with electrolyte. However, when no electrolyte was present, the microstructure was found to consist of a mixture of course serrated, platelet, and acicular alpha phases. This change in microstructure will affect the performance of the weld metal, and therefore, more work investigating the weld metal properties is necessary.

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