

Research Article

Effect of Laser Emission Parameters on Mechanical and Physical Properties of Cast Pure Titanium

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The objective of this study was to investigate the effect of laser beam parameters on the mechanical and physical properties of cast commercially pure titanium. Dumbbell-shaped test specimens (ISO6871) were cast with ASTM grades 2 and 3 Ti. The cast surfaces were laser-treated with various laser emission parameters (current: 200, 220, and 240 A; spot diameter: 1.0, 1.6 mm; pulse duration: 10, 1 ms) under argon shielding. Tensile testing was conducted at a crosshead speed of 1.0 mm/min. Data of tensile strength and elongation were statistically analyzed using ANOVA/Tukey's test ($\alpha = 0.05$). Hardness depth profiles were made with cross sections of rod-shaped specimens after laser treatments. Control specimens without laser treatment were also prepared. The laser-treated specimens showed significantly higher tensile strengths than those of control specimen for both grades 2 and 3 Ti. The specimens treated with 1.0 mm spot diameter indicated higher tensile strengths than those treated with 1.6 mm spot diameter for both currents (200 A and 240 A). The laser treatment decreased the subsurface hardness and increased the hardness between 75 μm and 400 μm in depth when compared to the hardness of control specimens. Laser surface treatment for cast commercially pure titanium modified integrity of cast surface and significantly improved mechanical and physical property.

1. Introduction

Commercially pure (CP) titanium and its alloys have been widely applied for orthopedic and dental implants because of their excellent biocompatibility [1–3], high corrosion resistance [4, 5], inherent ability to osseointegrate, and low modulus of elasticity (Young's modulus) to more closely match to the elastic modulus of the bone [3, 6, 7]. The CP titanium is classified into four categories grades 1 through 4 depending on their varying degrees of tensile strength and hardness, as a function of oxygen content, with the grade 1 being the most ductile (lowest tensile strength and hardness with an oxygen content of 0.18%) and grade 4 the least ductile (highest tensile strength and hardness with an oxygen content of 0.40%) [8]. Even if the grade 4 pure titanium possesses highest strength and hardness, its strength and hardness are lower

than those of titanium alloys, in which alloying elements modify microstructures resulting in improvement of their mechanical and physical properties. In dentistry, the CP titanium and its alloys are cast to fabricate metal framework for individual patient's demand [9–15]. Since titanium has extremely high affinity with oxygen at high temperature, cast titanium surfaces are usually contaminated with refractory oxide contained in the investment material during casting. The contaminated surface layers reduce the integrity of cast surface and induce hard and brittle cast surfaces which lead to decrease in mechanical properties [16], fatigue [17], and wear resistances [18].

Metal peening is one of the methods to enhance mechanical property and fatigue resistance of metal materials and has been applied in industry [19–21]. The process of peening metal's surface, which is normally cold work, includes

mechanical means such as hammer blows and blasting with shot (shot peening). Mechanical peening expands the surface of the cold metal, introducing residual compressive stresses [19, 20] and relieving the tensile stresses that are already present, and also encourages strain hardening of the surface metal [21]. The residual compressive stresses in a peened surface and tensile stresses in the interior are induced by plastic deformation of the metal surfaces and increase fatigue and corrosion resistances of the peened materials. Another peening method to increase mechanical properties and fatigue resistance is laser peening. Laser beams are widely used for enhancement of metal surfaces by modifying the surface microstructure into specific phases such as metastable phase and nanocrystalline grain phase [22–29]. Laser surface melting of metallic materials should be undergone in controlled inert gas atmosphere of argon or helium, particularly for titanium, in order to shield it from contamination with atmospheric gasses such as oxygen, nitrogen, or hydrogen since the contamination of titanium surface reduces integrity of surface microstructure resulting in decrease of mechanical properties. Microstructure of titanium surface is also affected by heating temperature, holding time, and cooling rate of the substrates; therefore the laser melting parameters should be optimized in order to reduce defects and to optimize microstructure [30]. Morphology of the layers created and nature of the phases formed on surface mostly depend on the laser beam overlapping [31] and the intensity of laser beam [27, 32] and nature of surrounding gas [24, 25, 27, 29].

In previous studies, laser treatment on cast titanium surfaces could produce reliable titanium metal frameworks for dental prostheses since significant enhancement of their mechanical properties can be obtained by laser treatment [33, 34]. Therefore, the objective of this study was to investigate the effect of laser beam parameters (out-put current, spot diameter, and pulse duration) on the mechanical and physical properties of cast commercially pure titanium.

2. Materials and Methods

2.1. Preparation of Specimens. Two types of commercially pure titanium (CP-Ti) were used in this study: ASTM grade 2 (T-alloy M, GC Corp., Tokyo, Japan) and grade 3 (T-alloy H, GC Corp.) pure titanium ingots. Dumbbell-shaped patterns (Figure 1: ISO6871, 18 mm for gauge length, 3 mm in diameter) and rod patterns (8 mm length and 3 mm in diameter) were prepared for casting of the CP-Ti. The CP-Ti was cast with a magnesia-based investment material (Selevest CB, Selec Co., Osaka, Japan) using an argon-arc melting/centrifugal titanium casting machine (Ticast Super R, Selec Co.). After casting, the molds were bench-cooled to room temperature and the cast specimens were retrieved. The surfaces of the cast specimens were air-abraded with 50 μm Al_2O_3 particles to uniform surface conditions. The specimens were then ultrasonically cleaned with acetone for 10 min.

2.2. Laser Surface Treatment. Laser surface treatment was applied using a dental Nd:YAG laser welding machine (Tanaka TLL-7000, Homburg, Germany) using the parameters as

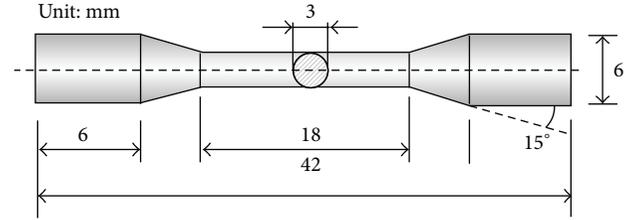


FIGURE 1: Schematic illustration of dumbbell-shaped tensile specimen (ISO6871).

TABLE 1: Laser emission parameters used in this study.

| Condition | Current (A) | Pulse duration (ms) | Spot diameter (mm) |
|-----------|-------------|---------------------|--------------------|
| 1 | 200 | 10 | 1.0 |
| 2 | 200 | 10 | 1.6 |
| 3 | 220 | 10 | 1.0 |
| 4 | 220 | 1 | 1.0 |
| 5 | 240 | 10 | 1.0 |
| 6 | 240 | 10 | 1.6 |

shown in Table 1. The parameters (current of 240 A, pulse duration of 10 ms, and spot diameter of 1.0 mm), which produce strongest laser pulse energy, were selected for laser to penetrate into cast and air-abraded titanium surface with 0.8–1.0 mm in depth [35]. During the laser surface treatment, argon gas shielding was applied from two nozzles set at a 45-degree angle on both sides above the treatment area of the specimen. Single-pulsed laser was shot perpendicular to the long axis of each specimen in order to avoid misshaping of the straight gauge due to the induction of compressive stresses induced by laser. The laser pulses were applied to whole gauge surfaces including up to 3 mm of the gripping surfaces and were 50% overlapped to ensure the entire testable surfaces as shown in Figure 2. The specimens without laser treatment were prepared as controls.

2.3. Tensile Testing and SEM Observation. Tensile testing was conducted using a universal testing machine (Model, Instron Corp., Canton, MA) at a crosshead speed of 1.0 mm/min. Tensile strength (MPa) and percent elongation (elongation with total extension at break) were recorded. The data ($n = 3$) were statistically analyzed using ANOVA and Tukey's test at a significant level of $\alpha = 0.05$. After tensile testing, the fracture surfaces were observed using a scanning electron microscope (SEM).

2.4. Hardness Depth Profiling. Hardness depth profiles were made on the cross-sectioned surfaces of the rod-shaped specimens after the cast rods underwent the same laser treatments. Vickers' hardness depth-profiles include measurements at 25 μm from cast surface to 1,000 μm in depth with 25 μm increments up to 100 μm and 50 μm increments for 100–1,000 μm . Three measurements were averaged for each depth. The data were statistically analyzed by ANOVA and Tukey's test ($P < 0.05$).

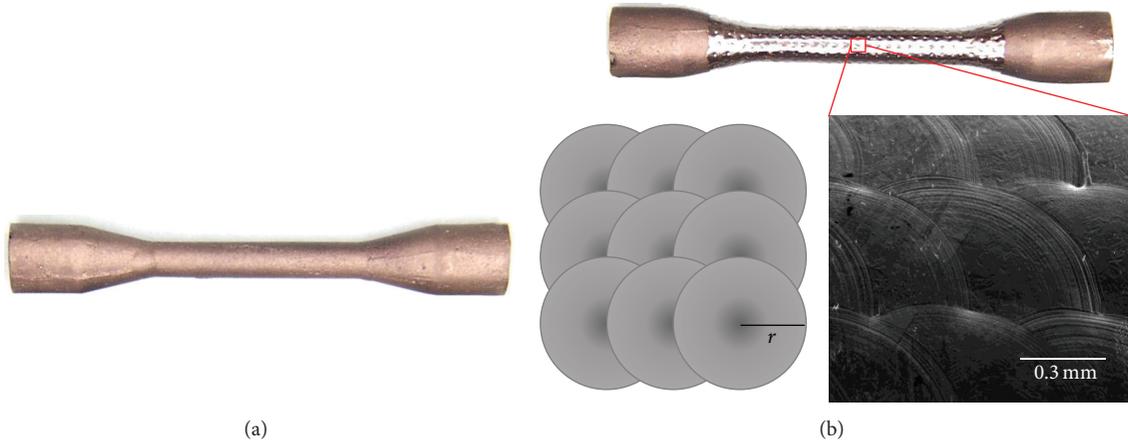


FIGURE 2: Schematic illustration of overlapped laser surface treatment: (a) control specimen after air-abrasion with $50 \mu\text{m Al}_2\text{O}_3$ and (b) laser-treated specimen; schematic illustration (bottom left, r : radius of spot diameter) and SEM image (bottom right) of a specimen surface after laser treatment.

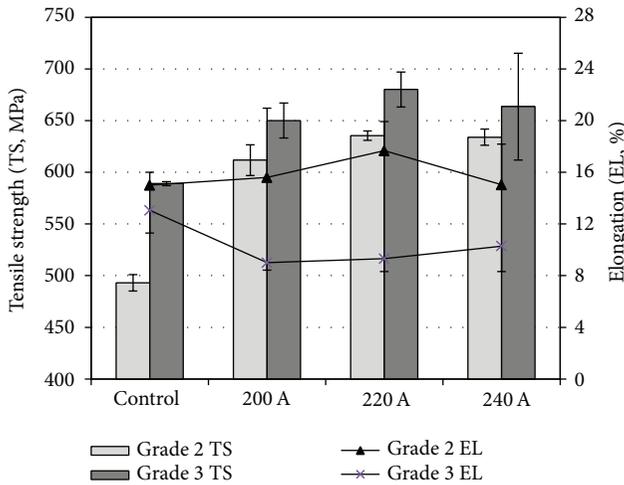


FIGURE 3: Tensile strengths and elongations of control specimens (grades 2 and 3) and the specimens laser-treated with several emission currents (200 A, 220 A, and 240 A) at a constant pulse duration (10 ms) and a spot diameter (1.0 mm).

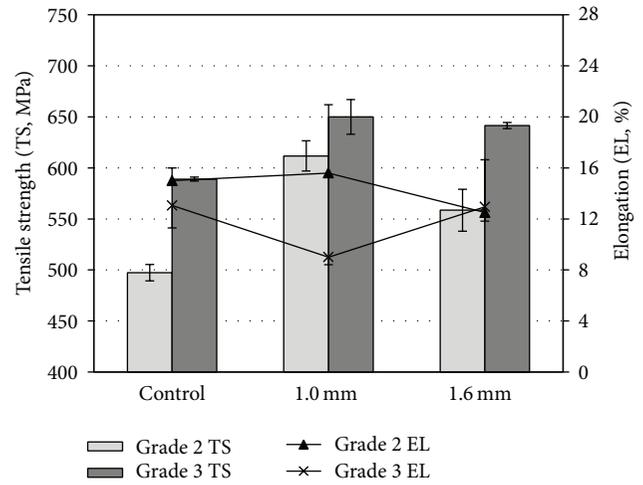


FIGURE 4: Tensile strengths and elongations of control specimens (grades 2 and 3) and specimens laser-treated with two different spot diameters (1.0 mm and 1.6 mm) at a constant emission current of 200 A and a pulse duration (10 ms).

3. Results

3.1. Tensile Test Results. Tensile strengths and elongations of control specimens (grades 2 and 3) and the specimens laser-treated with various parameters (current, spot diameter, and pulse duration) for laser emission are presented in Figure 3 through Figure 6. When the specimens are laser-treated with three different currents (Figure 3), the laser treated specimens showed significantly higher tensile strengths than those of control specimen for both grades 2 and 3 pure titanium (Ti). The tensile strength increasing rate was much greater for grade 2 Ti compared to grade 3 Ti. Increasing of current from 200 A to 220 A increased the tensile strengths of treated specimens for both Ti grades, thereafter the strength of the specimens treated at 240 A slightly decreased. The laser

treatment decreased the total elongation for grade 3 Ti, whereas it slightly increased the elongation of grade 2 Ti. The specimens laser-treated with two different spot diameters (1.0 mm and 1.6 mm) also increased their tensile strengths for both Ti grades at a constant pulse duration (10 ms) with a current at either 200 A (Figure 4) or 240 A (Figure 5). The specimens laser-treated with 1.0 mm spot diameter indicated higher tensile strengths than those laser-treated with 1.6 mm spot diameter for both currents (200 A and 240 A). The elongation was similar between control and 1.0 mm spot diameter for grade 2 Ti and decreased at 1.6 mm spot diameter for both currents (Figures 4 and 5). The grade 3 Ti decreased their elongations in order of control, 1.0 mm and 1.6 mm spot diameter except for 1.6 mm spot diameter at 200 A (Figure 4) which showed elongation similar to the control specimens.

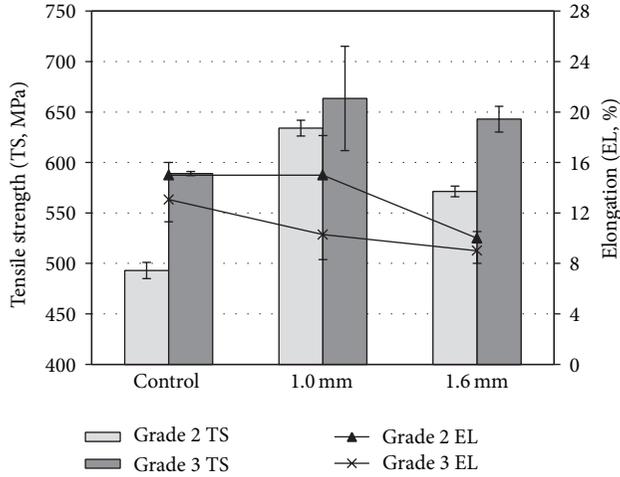


FIGURE 5: Tensile strengths and elongations of control specimens (grades 2 and 3) and specimens laser-treated with two different spot diameters (1.0 mm and 1.6 mm) at a constant emission current of 240 A and a pulse duration (10 ms).

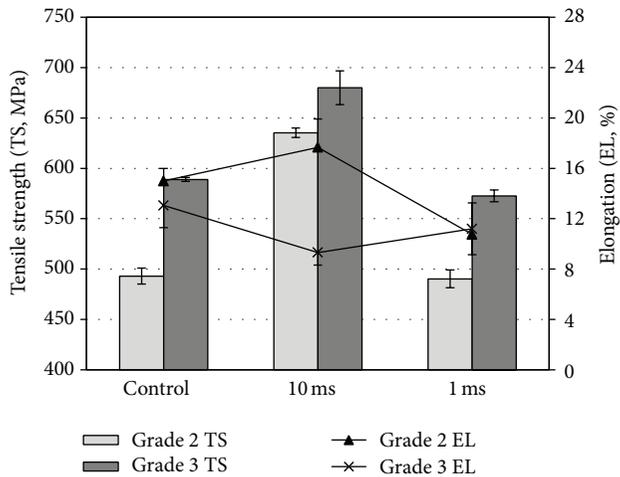


FIGURE 6: Tensile strengths and elongations of control specimens (grades 2 and 3) and specimens laser-treated with two different pulse durations (10 ms and 1 ms) at a constant emission current (220 A) and a spot diameter (1.0 mm).

When two different pulse durations (10 ms and 1 ms) are applied to treat the pure titanium at a constant current of 220 A and a spot diameter of 1.0 mm, the specimens treated with 10 ms pulse duration showed increased tensile strength compared to the control specimens, whereas the specimens laser-treated with 1 ms pulse duration showed lower tensile strength than the control specimens. The results of elongation demonstrated the results similar to those obtained in Figure 4 for testing of different spot diameters at a constant current (200 A) and pulse duration (10 ms).

3.2. Hardness Depth Profile. The hardness depth profiles obtained from cross-sections of control specimens and specimens laser-treated with various parameters are shown in

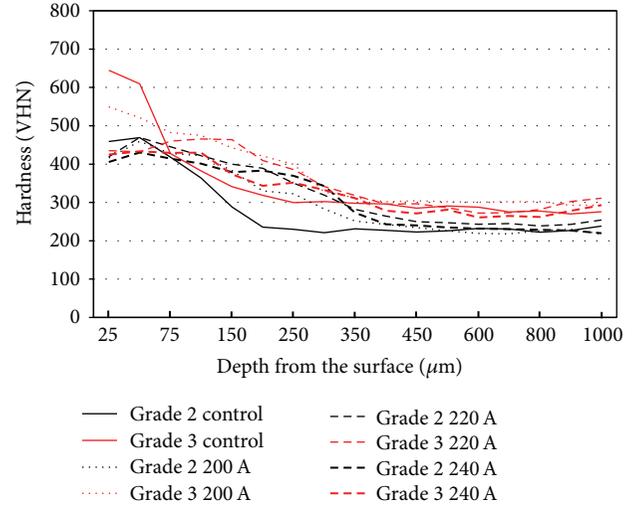


FIGURE 7: Hardness depth profiles from cast surface for control specimens (grade 2: black solid line, grade 3: red solid line) and specimens laser-treated with several emission currents (200 A, 220 A, and 240 A) at a constant pulse duration (10 ms) and a spot diameter (1.0 mm).

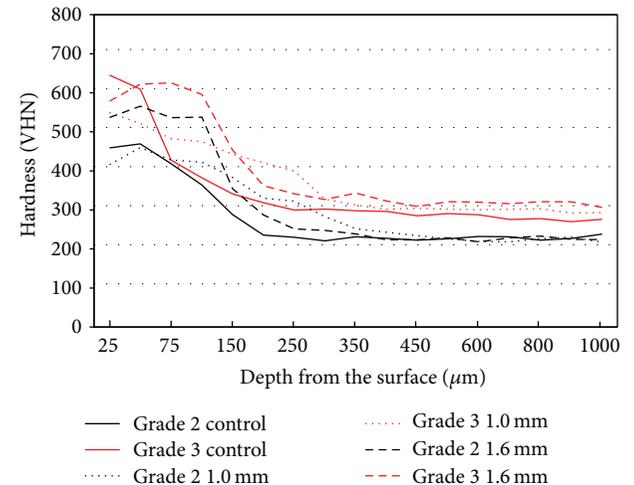


FIGURE 8: Hardness depth profiles from cast surface for control specimens (grade 2: black solid line, grade 3: red solid line) and specimens laser-treated with two different spot diameters (1.0 mm and 1.6 mm) at a constant emission current of 200 A and a pulse duration (10 ms).

Figures 7, 8, 9, and 10. Figure 7 demonstrates the hardness depth profiles for the specimens laser-treated with three different currents. The control specimens possess very high hardness at subsurface (25 μm and 50 μm) area on cross section. The laser treatment decreased the subsurface hardness and increased the hardness between 75 μm and 400 μm in depth when compared to the hardness of control specimens. Bulk hardness (>400 μm in depth) is similar for all conditions (control specimens and specimens treated in all currents) in each grade of Ti. Note that the bulk hardness of grade 3 (red lines) is higher than those of grade 2 (black lines) at area

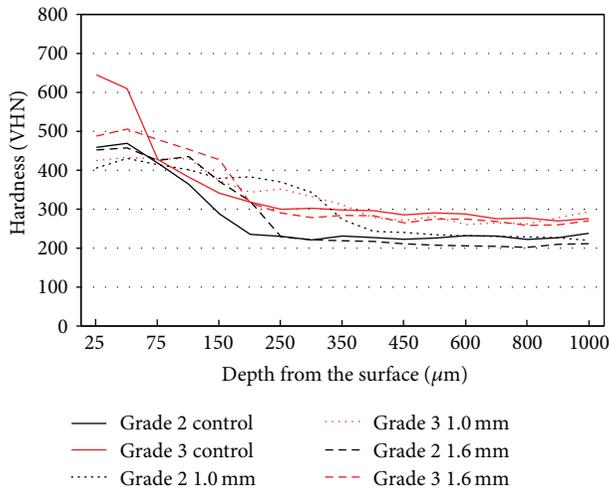


FIGURE 9: Hardness depth profiles from cast surface for control specimens (grade 2: black solid line, grade 3: red solid line) and specimens laser-treated with two different spot diameters (1.0 mm and 1.6 mm) at a constant emission current of 240 A and a pulse duration (10 ms).

deeper than 400 μm . Hardness depth profiles of the specimens treated with two different spot diameters and constant pulse duration (10 ms) are presented in Figure 8 (constant current at 200 A) and Figure 9 (constant current at 240 A), respectively. Higher subsurface hardness between 25 and 150 μm was obtained with lower current (200 A) and larger spot diameter (1.6 mm). The depth of the laser-affected area with high hardness increased as the spot diameter decreased and as the current increased (Figures 8 and 9). Figure 10 presents the hardness depth profiles of specimens treated with two different pulse durations with constant current (220 A) and spot diameter (1.0 mm). Subsurface hardness at 25 μm of specimens treated with 1 ms pulse duration was higher than those of control specimens for both Ti grades, and the higher hardness was maintained until 150 μm in depth. When the 10 ms of pulse was used to treat both grades of Ti, subsurface hardness at 25 μm of treated specimens was lower than those of control specimens. However the subsurface hardness with high hardness was maintained until 350 μm in depth.

3.3. SEM Observation of Fracture Surfaces. The fracture surfaces observed after tensile testing are demonstrated in Figure 11. The subsurface area (a2) of control specimen (a1) had the cleavage acicular fractures which are typical microstructure of fractured cast pure titanium due to the surface reaction layer between the investment and molten titanium during casting. On the other hand, the laser-treated specimen (b1) showed a flat fracture surface at subsurface (laser-treated) area (b2).

4. Discussion

The laser surface treatment applied in this study apparently modified surface quality and resulted in increased mechanical strength (Figures 3 through 7). Cast pure titanium

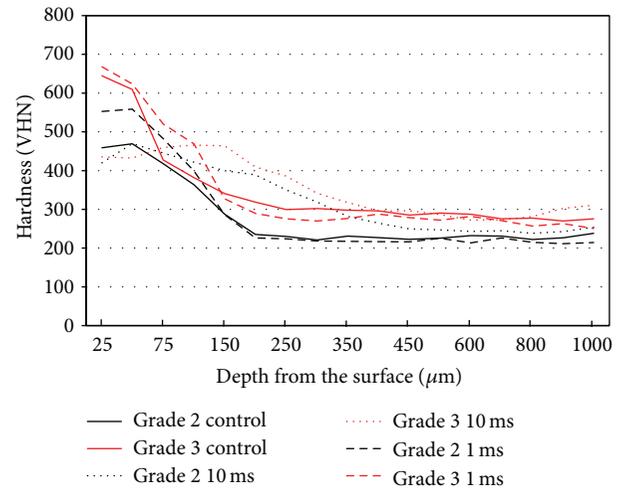


FIGURE 10: Hardness depth profiles from cast surface for control specimens (grade 2: black solid line, grade 3: red solid line) and specimens laser-treated with two different pulse durations (10 ms and 1 ms) at a constant emission current (220 A) and a spot diameter (1.0 mm).

normally produces hard and brittle cast surfaces generated by the reaction between investment material and molten titanium during casting as observed in Figure 7 and Figure 11. Note that the control specimens showed very high hardness at subsurface (25 μm and 50 μm) area on cross section (Figure 7) and fractured in brittle manner with cleavage acicular fracture (Figure 11(a2)). The thickness of surface reaction layer depends on the investment material used and casting conditions including atmosphere in casting unit and casting method (pressure type of machine, centrifugal machine). The thickness of cast metal frameworks also affects the thickness of surface reaction layer (thicker framework tends to thicken the reaction layer). In this study, the thickness of reaction layer could be estimated approximately 150–200 μm in depth from the hardness depth profiles of control specimens (Figure 7). The laser surface treatment decreased subsurface hardness of the reaction layer and decreased hardness maintained beyond the reaction layer up to 350–400 μm in depth (bulk hardness starts around this point). These hardness changes along with modification of subsurface microstructures (Figure 11) by laser treatment (Figure 11) resulted in improvement of mechanical properties. Another reason for improvement of mechanical properties might be due to the residual compressive stress of specimen surface induced by laser treatment. The laser can quickly melt and solidify the surface and the quick solidification introduced the residual stress on the surfaces.

Increasing of current up to 220 A significantly increased the tensile strength of laser-treated specimens for both Ti grades (Figure 3). However there are no statistical differences in tensile strength between 220 A and 240 A for both Ti grades. The reason might be the similar laser penetrations depth into the specimens between currents 220 A and 240 A as hardness depth profiles for grade 2 Ti show similar behavior for these conditions (Figure 7). When the specimens

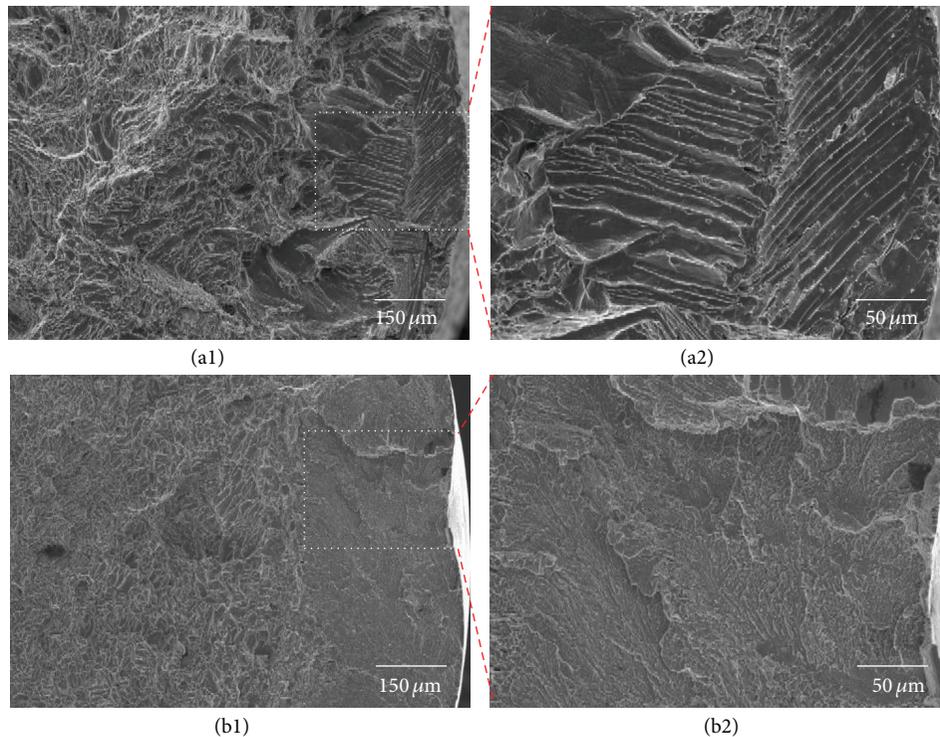


FIGURE 11: Fracture surfaces after tensile testing. (a1) Grade 2 control specimen and (a2) magnified image from dotted square (subsurface area) in (a1). (b1) Laser-treated grade 2 Ti specimen (current: 220 A, pulse: 10 ms, and spot diameter: 1.0 mm) and (b2) magnified image from dotted square (laser-treated area) in (b1).

were laser-treated with two different spot diameters (1.0 mm and 1.6 mm), the results of tensile test were similar between 200 A (Figure 4) and 240 A (Figure 5); that is, higher tensile strengths were obtained for specimens treated with spot diameter of 1.0 mm than those treated with spot diameter of 1.6 mm. The tensile strengths of the specimens laser-treated with 240 A (Figure 5) were slightly higher than those laser-treated with 200 A (Figure 4) in each corresponding condition (spot diameter and Ti grade). A laser with parameters such as small spot diameter and high current (high laser energy) can penetrate into metal surface deeper than a laser with large spot diameter and low current (low laser energy). The statement above can be confirmed by hardness depth profiles for both currents of 200 A (Figure 8) and 240 A (Figure 9). The tensile strengths of the specimens treated with small spot diameter (1mm) were higher than those laser-treated with large spot diameter (1.6 mm). Another reason for this can be the number of laser treatment spots on the surface which simply increased to cover all interacted area. A large number of laser spots increase residual stress on the surface and resulted in improved mechanical strength. Figure 6 demonstrates the effect of pulse duration on tensile strength of surface treated Ti specimens. The specimens laser-treated with 1 ms pulse duration had lower tensile strength than those of the control specimens for both Ti grades. It is interesting that the subsurface hardness of specimens

laser-treated with 1 ms pulse duration was higher than those of control specimens for both Ti grades. The high subsurface hardness indicates that the laser-surface treatment with 1 ms pulse duration increased brittleness of the surfaces and resulted in reduction of tensile strength for laser-treated specimens.

5. Conclusions

Laser surface treatment for cast commercially pure titanium modified integrity of cast surface and significantly improved mechanical property. From the results obtained in this study, the following conclusions can be drawn.

- (1) Surface treatment by laser with higher currents (high laser energy) increased the tensile strength of the laser-treated specimens up to 220 A for both Ti grades.
- (2) Tensile strengths of the specimens laser-treated with small spot diameter were higher than those laser-treated with large spot diameter for laser treatment for both Ti grades.
- (3) The specimens laser-treated with 1 ms pulse duration had lower tensile strength than those of the control specimens for both Ti grades due to the top surface thinner hardening compared to 10 ms pulse duration.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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