Examination of welded joint of titanium alloy used in oral surgery

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Abstract. The additive manufacturing of customized Ti-6Al-4V (Grade 23) implants, which in the present research concerns the dental and maxillofacial surgical field, may require complex manufacturing technology due to their complexity and assembly. In practice, additively manufactured titanium implant elements are supplemented by threaded sleeves that allow disassembly. These turned elements made from Grade 5 material quality rolled preform are joined by laser micro-welding with Grade 1 material. This special process for implant production is still not widespread in current manufacturing practice, and there is no technical recommendation for the manufacturing parameters of these implants. The aim of our research is to explore the possibilities and limitations of additive process in the manufacture of custom-made implants and to provide guidelines for optimal manufacturing and welding parameters. In the initial phase of the research, preliminary experiments were conducted with the conventional and additive manufacturing of cylindrical test specimens and with the production of combined products by micro-welding. The specimens were subjected to tensile testing, the weld was examined by CT, and the fracture surfaces were studied by optical microscope and scanning electron microscope.

1. Introduction
Various materials are used in dental applications, such as metals, ceramics and polymers. Among metallic materials, commercially pure titanium and titanium alloys are becoming more and more widespread due to their relatively simple handling and high strength, their excellent corrosion resistance and their biocompatibility [1]. The most widely used titanium alloy is Ti-6Al-4V, which is generally chosen for medical devices, prostheses and various implants, as well as the material for bone replacement. Until the 1980s, manufacturing technology was divided into two classes: metal forming through plastic deformation, without adding or removing material, and removal processes, and those technological solutions, in which the final product is obtained by removing excess metal from the stock, thus results in waste.

By the end of the 20th century, a new manufacturing process became available due to advances in digital technology. In this technology, the desired geometry is obtained from digital data and then builds the object layer by layer. These processes are referred to as "Additive Manufacturing (AM)" [2,3]. Due to its many advantageous features, not only products with special geometry (mould inserts with conformal cooling) can be manufactured, but also components with special mechanical properties can be produced by using materials with unique composition by mixing powders [4,5].
Computer-aided design and manufacturing (CAD-CAM) became widespread in dental applications in the 1980s [6]. Powder bed fusion technology allows the creation of porous-structured products based on digital models. Due to several advantages of this technology, not only fields, such as tooling and of These can be used as dental implants that fulfil both mechanical and physical requirements [7, 8]. Porous structures can improve osseointegration between the bone and implant. There is an increasing number of studies in this field [9-11].

Titanium, the main base material of the implant, requires great attention in all areas of processing, especially during welding [12]. Welding technology is complex for titanium as the material is highly reactive with atmospheric gases, such as oxygen and nitrogen in liquid phase and above 550 °C. Contamination can be caused by impurities in the shielding gas, poor protection of the welding zone, and improper preparation or cleaning of welding and filler materials before or during welding [13].

There are numerous welding processes available for pure titanium and titanium alloys, such as laser welding, gas tungsten arc welding, and brazing [14]. For metallic materials, there are currently three different techniques investigated based on welding materials: laser welding [15-17], shaped metal deposition [18-20], electron beam deposition [21]. Numerous studies have investigated welded zone microstructure in commercially pure titanium and titanium alloys and have especially focused on analysing the effect of contamination on mechanical properties [19]. Two different welding processes – solid state laser welding and gas tungsten arc welding – have been compared for additively manufactured Ti-6Al-4V alloys [20]. Welding tests were conducted on (Grade 2) titanium alloys and it was proved that discoloration at the weld zones was correlated to the oxygen and nitrogen content of the weld zone, the absence of protective atmosphere, and the mechanical properties of the weld joint [19].

In our present research we used Ti-6Al-4V alloy and produced cylindrical specimens by conventional way with machining and by powder bed fusion. We made pieces by joining the differently made parts with micro-welding. The specimens were subjected to rupture, the weld was examined by CT, and the fractograph were analysed by optical microscope and scanning electron microscope.

2. Experimental

2.1. Materials

For the experiments different grades of titanium and alloys were used. Ti-6Al-4V titanium alloy, Grade 5 (Bibus Metals AG) was used to produce tensile test specimens by machining rods. Ti-6AL-4V ELI powder, or Grade 23 (LPW Technology Ltd), which is the higher purity version of Ti-6Al-4V (medical titanium grade) was used for powder bed fusion. We used wire (Ø 0.2 mm) made of commercially pure titanium Grade I for welding experiments. Table 1. shows the nominal chemical composition and density of applied materials.

| Titanium grade | Chemical composition (wt%) | Density (g/cm³) |
|----------------|---------------------------|----------------|
| Grade I        | -                        | res 4.51       |
| Grade 5 ELI    | 5.5-6.5 3.5-4.5 0.25 0.13 0.08 0.05 0.012 | res 4.47       |
| Grade 23       | 5.5-6.5 3.5-4.5 0.25 0.13 0.08 0.03 0.012 | res 2.49       |

2.2. Manufacturing of specimens

Cylindrical Ø6 mm tensile test specimens were produced by turning from rod and by laser metal fusion (LMF) using a SISMA mysint100. Specimens were produced by welding in the following steps:

1. Fixing of the conventionally produced and additively manufactured parts by spot welding in argon atmosphere.
2. Filling the seam with filler material based on the set parameters of the laser welding machine (Dentaurum Basel Laser Desktop).
3. Stress relief heat treatment process in argon (600°C)
The technical characteristics of welding machine are summarized in Table 2. Two different configurations (W1 and W2) were used when setting the parameters of the welding machine:

- W1: 295 V; 9.5 m/s; 2 focuses
- W2: 280 V; 7.5 m/s; 0 focus

| Table 2. Main technical characteristics of laser welding equipment |
|------------------------------------------|
| **Electrical connection** | 200 – 240 V, 50-60 Hz, 10 A |
| **Max. power** | 2.2 kW |
| **Average power** | 50 W |
| **Wavelength** | 1064 nm |
| **Pulse shape** | 4 pre-formed pulse shapes |
| **Pulse energy** | 50 J |
| **Pulse peak power** | 5 kW |
| **Pulse duration** | 0.5-20 ms |
| **Pulse frequency single pulse** | 25 Hz |

2.3. Test methods
For characterization of the parts tensile tests (3-3 pieces of homogenous specimens and 2-2 pieces of welded parts) according to MSZ EN ISO 6892-1:2016 method B were carried out at room temperature with Instron 5582 equipment (test speed 10 mm/min). Welded specimens were examined by computed tomography before and after the tensile test by YXILON Modular CT equipment. Fractographs of the specimens were studied by Zeiss Stereo Discovery V20 stereo-microscope and by HITACHI 3400 scanning electron microscope (SEM).

3. Results
The machined parts (M), additively manufactured specimens (AM) and their combinations joined by laser welding (W1, W2) were tested under tensile loading and the average values of tensile strengths can be seen in Figure 1.

![Figure 1. Tensile strength of Ti-6Al-4V parts made by machining (M), additively manufactured (AM) and their combination joined by laser welding (W1, W2)](image)

In the case of tensile strength values, much lower values were obtained for the welded pieces than for the homogenous specimens. Machined and AM parts showed different behaviour under tensile loading, which is indicated also by the percentage elongation values after fracture (Figure 2).
Figure 2. Percentage elongation values after fracture of Ti-6Al-4V parts made by machining (M), additively manufactured (AM) and their combination joined by laser welding (W1, W2)

Additively manufactured parts showed almost 40% less elongation after fracture under tensile loading than machined parts, where the neck formation can be clearly seen. Images made by stereo-microscope show the fractured surface of the tested parts (Figure 3).

Figure 3. Images (stereo-microscope) of typical fractured surface of tensile test specimens (a) machined, b) additively manufactured, c) W1 welded, d) W2 welded

As it can be seen on Figure 3.c the W1 test specimen fractured in the AM part, while W2 welded parts broke in the seam. The results show that the welding technology was not carefully carried out. The high porosity of the weld and the lack of fusion resulted in a weak connection.

Examination with high magnification of a fracture surface is critical of a failure investigation, because the topography and fracture features can help to determine the fracture mode.

The scanning electron microscope is very important in the proper evaluation and classification of a fracture surface. The greater depth of field provided by the SEM allows to make high resolution images of surfaces after fracture at higher magnifications. Fractured surface of broken parts in case of machined, additively manufactured and welded (W2) showed quite different structure (Figure 4.).
Figure 4. SEM images of fractured surfaces of tensile specimens, made by machining (a, c), additively manufactured (b, e) and W2 welded parts (c, f) 
(scale bar: 2.00 mm (top) and 50.0 µm (bottom))

Ductile fractures are characterized by extensive plastic deformation just prior to fracture compared to brittle fractures. Ductile fractures often initiate at voids, or inclusions, forming distinct elongated dimples such as shown both in case of homogeneous parts, shown in Figure 4. In case of welded parts, there are several areas when the lack of fusion (red arrows) can be easily explored (Figure 5). Compared the SEM images with the CT tomographs (Figure 5) the same patterns can be observed which confirms the lack of fusion.

Figure 5. Tomographs of welded parts in the welded region, left: W1 part, right: W2 part (scale: defect volume (mm$^3$))
4. Conclusions
In our present experiments cylindrical tensile test specimens were prepared from Ti6Al4V alloy by conventional technology (cutting) and by powder bed fusion, and the differently made parts were joint by micro-welding with two parameter settings. The welding quality was evaluated by tensile test, CT examination, and the fractographs were studied by stereo-microscope and scanning electron microscope.

Based on the results, the following conclusions were made:

- The conventionally manufactured specimens have higher deformation capacity (based on the percentage elongation after fracture) and lower tensile strength than the additively manufactured parts.
- The mechanical properties of the welded specimens could not reach those of base materials, so welding parameters should be chosen more carefully with further experiments.
- Both specimens produced by different technologies and welded parts exhibit tough behaviour based on SEM images.

A further aim of the research is to map the effect of laser micro-welding parameters on the properties of combined specimens from conventionally made and additively manufactured pieces and to provide recommendations for the application of optimal technological parameters in the implant production.

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References
[1] Brunette DM et al (Eds.) 2001 Titanium in medicine, Material Science, Surface Science, Engineering, Biological Responses and Medical Applications, (Springer, Berlin, Heidelberg) pp. 25-51
[2] Hatos I and Zsoldos I 2012 Proceedings of Factory Automation (University of Pannonia (Eds.) 42-45
[3] Gibson I et al 2009 Additive Manufacturing Technologies (Springer-Verlag New York) pp. 19-42
[4] Hatos I et al 2018 Stroj Vestn-J Mech E 64(2) 121-129
[5] Koecsis B et al 2020 J Magn Magn Mater 501 166425
[6] Al-Mesmar HS et al 1999 J Prostheth Dent 82 (1) 15-21
[7] Arvidsson A et al 2015 J Biomed Mater Res B Appl Biomater 103 12-20
[8] Yang F et al 2017 Sci Rep 7 45360
[9] Maniatopoulos C et al 1986 J Biomed Mater Res 20 1309-1333
[10] Unger AS et al 2005 J Arthroplasty 20 (8) 1002-1009
[11] Wu S et al 2013 Artif Organs 37 (12) 191-201
[12] Saresh N 2007 J Mater Process Technol 192-193 83-88
[13] Klas W 2003 Welding Processes Handbook (Woodhead Publishing Ltd) pp. 148-170
[14] Xie J and Safarevich S 2003 Proceedings of the Materials and Processes for Medical Devices Conference (Anaheim, CA, ASM International) 25-30
[15] Kelly SM and Kampe SL 2004 Metall Mater Trans A 35(6) 1869-1879
[16] Qian Let al 2005 Mater Sci Tech 21(5) 597-605
[17] Dinda GP et al 2008 Metall Mater Trans A 39(12) 2914-2922
[18] Balasubramanian M et al 2008 Mater Design 29(1) 92-97
[19] Li X et al 2005 J Mater Sci 40(13) 3437-3443
[20] Brandl E et al 2010 Phys Procedia 5 Part B 595-606
[21] Nowotny S et al 2007 J Therm Spray Tech 16(3) 344-348