

Structural analysis of titanium alloys

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Abstract. The material of choice for dental implant devices is titanium alloy (Ti-6Al-4V). In this study, the two fundamental manufacturing technologies of dental implant prostheses are compared. Titanium alloy specimens and models were created with conventional precision casting and with modern, innovative additive manufacturing technologies. Hereupon, the microscopic analysis of specimen cut-offs was carried out. We were focusing on fundamental microstructural differences. The thus-created specimen models were subjected to tensile tests for comparative examination.

1. Introduction

In the field of dental applications, there are several different materials available such as metals, polymers, and ceramics. Metallic materials are even more widespread due to their high strength and good processability. Titanium and its alloys bear with outstanding corrosion resistance and biocompatibility [1]. The most common titanium alloy for prostheses, implants, and medical devices is Ti-6Al-4V alloy, which is the standard material for bone replacements [2]. Manufacturing technologies were classified into two groups until the end of the 1980s. One group was metal forming, during which adequate external forces were exerted to obtain the proper geometry of the workpiece. During the processing technology, material continuity was not broken, and the mass of the workpiece did not change. The other processing technology was subtractive manufacturing, during which the required geometry was created with cutting.

Because of the development in digital sciences, a new manufacturing technology emerged by the end of the 20th century. This method utilizes additive manufacturing instead of subtractive technologies to build up the required workpiece geometry. During this new process, the workpiece is built up layer-by-layer, and prototypes can be manufactured using digital data [3]. Nowadays, these processes are the so-called Additive Manufacturing (AM) technologies [4].

Different synonyms are in use, such as additive processes, additive manufacturing, etc. [5]. By the 1980s, computer-aided design and manufacturing (CAD/CAM) became widespread in dental fields [6].

Additive manufacturing enables a high-degree freedom of design of implants and prosthetic frames which meet all the mechanical and physical requirements [7]. 3D printing makes it able to create complex, hierarchic structures directly from a computer model, thus structured, porous dental implants can be manufactured [8]. More and more studies prove that porous structure helps tissue connection, thus enhances osseointegration between the implant and bone tissue [9-11].

Here we can mention that the corrosion resistance of the metal structure comes from the high affinity of oxygen to titanium. This helps osseointegration process by the formation of a passive oxide layer [12]. The oxide layer formed on titanium alloy is made up of TiO₂ (titanium-dioxide) [13]. The thickness of the formed oxide layer is approximately 10nm, and provides high resistance against several chemical effects. Thus, titanium-based prosthetic implant structure frames are highly corrosion resistant also in oral environments [14]. Conventional manufacturing technology of titanium structures is casting, which



can create the required geometric contours. Shaping and coating of titanium implant surfaces raise important questions [15].

The use of titanium casting machine developed for dental applications increased the widespread use of cast titanium. Titanium is heated in a vacuum atmosphere, so oxidation rate is kept low during the melting of the metal [16-19]. In everyday routine, professionals encounter several problems during lost-wax casting technology. Possible emerging problems can be the distortion of wax, casting defects that lead to imprecision of the product [20]. Together with casting technology, modern additive technologies also show an ever increasing development.

2. Materials and Methods

2.1. Casting of specimens

Cast specimens were created with a Dentaurum Universal 230 Autocast casting unit (Fig. 1). The first step of the manufacturing process was 3D-printing of specimens from polymeric material. A pre-designed model was made during manufacturing, then the wax pattern was embedded. With the help of silicone casting material, a wax copy was made. The necessary organic wax for lost-wax casting had to be gassing-free and had to be suitable for burning without sudge formation. Afterwards, spruing and ceramic embedding of the models took place. The open-pore embedding material and the pattern inside is burned out in a furnace. Then titanium is casted into the remaining places of the pattern with a vacuum-pressure casting machine. This process takes place in an argon atmosphere. After this, embedding material is removed from the cast and the testing of geometrical sizes starts (Fig. 1).



Figure 1. Manufacturing steps of titanium alloy specimens at casting processes

2.2. 3D printing of specimens

For the comparison of casting technology and 3D printing, specimens were also created with a SismaMysint 3D printer (Fig. 2). Hereby, printing was carried out with Laser Metal Fusion (LMF) technology. A programmable furnace was deployed for post-production heat treatments considering specific printing strategy and heat-diverting support materials. Different software and milling machine are available for precision post-processing. Additive manufacturing process is preceded by digital model design. 3D printing is an additive manufacturing process that builds up parts by layering and melting together metal powder particles.



Figure 2. 3D printing unit and an image showing the printing process

2.3. Heat treatment

After the preparation of models, the necessary time interval for 3D printing was approximately 4 hours. The specimens were separated from the building platform. The disc and the support material connecting the specimens were removed. Next, the workpieces were subjected to heat treatment with parameters determined by the manufacturer. Fig. 3 shows the process diagram for heat treatment.

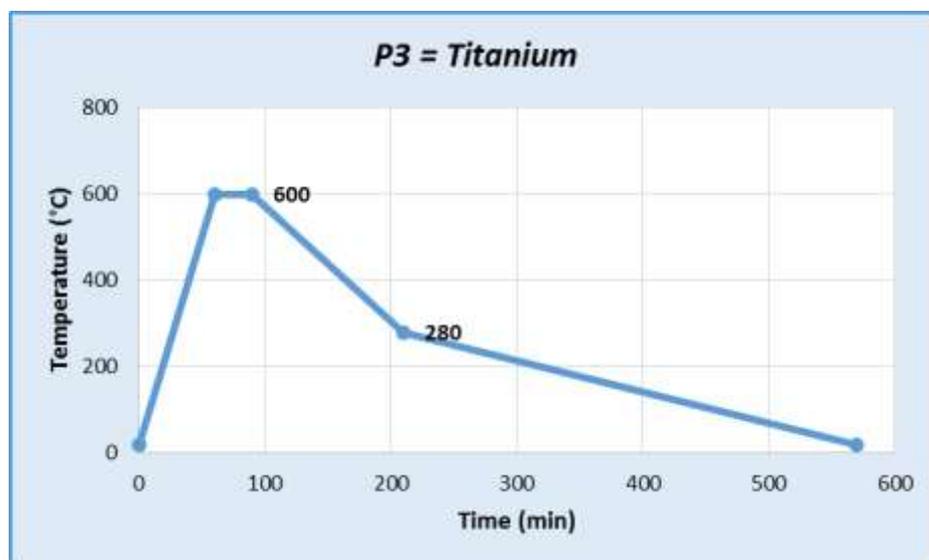


Figure 3. Heat treatment process diagram

2.4. Materials of choice

For the experiments, Ti-6Al-4V titanium alloy, and commercially pure titanium materials were chosen. Grade 5 ELI (marked "A") for casting, and Grade 23 (marked "B") for 3D printing were used for metallographic analysis. Grade 1 (marked "C") titanium was examined during tensile tests. The chemical composition of different titanium and titanium alloys are shown in Table 1.

Table 1. Chemical composition of different titanium alloys

Component	Chemical composition		
	Percentage (%)		
	„A”	„B”	„C”
Oxygen (O)	0.20	0.13	0.18
Nitrogen (N)	0.05	0.03	0.03
Carbon (C)	0.08	0.08	0.08
Hydrogen (H)	0.015	0.0125	0.015
Iron (Fe)	0.4	0.25	0.2
Aluminum (Al)	5.5-6.75	5.5-6.5	0
Vanadium (V)	3.5-4.5	3.5-4.5	0

3. Testing of specimens

Printed and casted specimens were subjected to microscopic metallographic analysis and mechanical tensile tests.

3.1. Metallographic analysis

Microscopic images were made in the material testing laboratory at the Department of Material Science and Technology in Szechenyi Istvan University. Microscope: multifunctional optical microscope; type: ZEISS Axio Imager M 1. The first test was the comparison of Ti-6Al-4V samples manufactured with casting and 3D printing.



Figure 4. Grain structure of cast titanium samples

Fig. 4 shows the grain structure of cast titanium samples at different magnifications. No anisotropy can be discovered in the grain structure, typical grain size is relatively large exceeding 50-100 μm . At higher magnification (middle and right images), the characteristic pattern of α - β titanium alloys can be observed. Alpha-phases (light-colored parts) have a relatively regular geometry represented in plate-like shapes. Between alpha phases, thin areas of beta-phase (dark-colored parts) appear. In the left and middle images, small black grains can be detected, which prove that a certain extent of porosity remain in the structure after the casting process.



Figure 5. Grain structure of 3D-printed titanium samples

Images in Fig. 5 show the grain structure after 3D printing. The left and middle images were shot with different expositions after etching. The right image shows the sample before etching. Grain structure can be characterized with the left and middle images. Grain structure is anisotropic. Grains are elongated, and the longitudinal direction is parallel with the direction of laser scanning. The typical thickness of columnar grains lies between 50-100 μm , which is in the same order of magnitude than that of cast specimens. Black grains observed in the right image are small porosities. Generally, the same extent of porosity is present both in cast and 3D-printed specimens based on visual inspection.

3.2. Tensile tests

During the testing procedure, the previously introduced material at 3D printing was compared to commercially pure titanium. Commercially pure titanium is widely used in dental practice to create cast parts. Its chemical composition is shown in the following.

The test-specimens were fixed into a tensile testing machine, and then were subjected to controlled tension until failure. Fig. 6 shows the state of the specimens after testing. In the right image, the fracture site can be observed, which shows signs of brittle fracture.



Figure 6. Photos of a broken specimen after loading test.

Fig. 7 shows load-extension curves of specimens. Numbers from 1 to 3 mark 0°, 45° and 90° manufacturing orientations, respectively. Cast titanium specimen is marked no. 4.

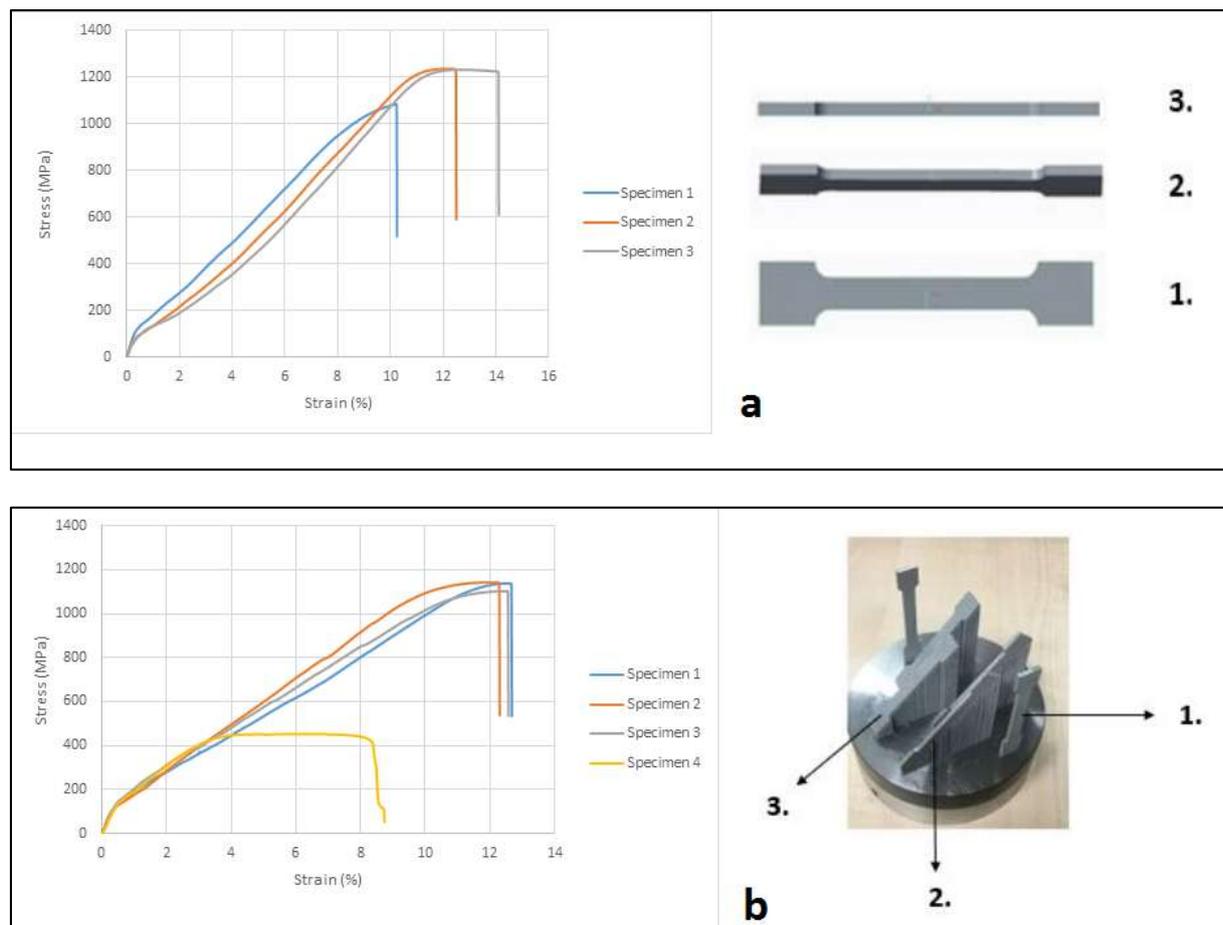


Figure 7. Load-extension curves and different printing orientations of specimens created with additive manufacturing. a: loading diagram for various orientation around the specimen axis. b: loading curves according to various specimen orientation angles in the workplace of the 3D printer

Table 2 and 3 detail the material properties determined by tensile testing.

Table 2. Material properties of specimens shown in Fig. 7a determined by tensile testing

	Tensile stress at Tensile Strength (MPa)	Modulus (E-modulus) (GPa)	Tensile strain at Tensile Strength (%)
1	1138	29.03	12.49
2	1142	31.48	12.06
3	1103	28.92	12.44
4	453	27.10	5.68

Table 3. Material properties of specimens shown in Fig. 7a determined by tensile testing

	Tensile stress at Tensile Strength (MPa)	Modulus (E-modulus) (GPa)	Tensile strain at Tensile Strength (%)
1	1081	29.68	10.12
2	1235	19.83	12.12
3	1231	15.79	12.64

The differences in strength values originate from anisotropic grain structure. The elongated grains in the laid specimen are parallel to the load, which is favorable in a mechanical point of view. However, these elongated grains in the standing specimen are directed perpendicularly to the load. There are more grain boundaries present in the direction of the stress, and this orientation is mechanically unfavorable. The orientation of 45 degrees is a transition between these two cases. It is noticeable that higher values are present in the second group. The distinctness of values raises other issues about the structured melting of layers and the orientation of the specimen.

Table 4 contains material properties for commercially pure titanium obtained from literature, and data determined from our cast specimens.

Table 4. Properties after the casting process

	Ultimate Strength (MPa)	Modulus of elasticity (Gpa)
Commercially pure titanium (literature)	345	103
Commercially pure titanium (cast)	453	27

Table 5 contains the comparison of mechanical properties of Ti-6Al-4V titanium alloy obtained from literature and from testing of specimens after additive manufacturing and heat treatment. The values from literature apply to rolled, grinded fiber material. It is not subjected to heat treatment before machining.

Table 5. Data for fiber and 3D-printed material

	Ultimate Strength (MPa)	Modulus of elasticity (Gpa)
Ti-6Al-4V (literature)	895	114
Ti-6Al-4V (3D-printed)	1138	29

4. Summary

In conclusion, assume that laser sintering – 3D printing technology – is going to replace the role of casting in terms of titanium processing in dental applications. The homogeneity of products created with additive manufacturing is more uniform. The geometry to be realized is going to be determined. Both in terms of microstructure and tensile tests, laser sintered specimens outperformed casted ones. During casting process, both internal and external inclusions are formed. A special and expensive casting material is needed. Human expertise is necessary for post-processing that guarantees the precision of the finished product. Laser sintering provides more precise, and faster processing. As a result of this, 3D printing of titanium is becoming even more widespread in medical technology.

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