The effect of thermal treatment on the properties of SLM samples with a bionic design

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Abstract. This article shows the possibility of additive technology applied to medical implants. It's shown the difference of physical properties such as elastic module of bones, implants made of traditional technology and implants made of additive technology. Porous structure has been chosen for future implants through designing its and further calculation of its mechanical properties using software such as SolidWorks, Logos and Ansys. To confirm the right choice of design of structure we manufactured samples with using Rosatom 3D printer, made heat treatment and tested them.

1 Introduction

The use of titanium (GOST 5832-2) in medicine is gaining popularity, especially in the field of personalized prosthetics. Due to highly favorable qualities, such as inertness, hypoallergenic, biocompatible and firm structural characteristics, the use of titanium is permissible in a wide variety of clinical cases. Despite advantageous characteristics of the prosthetic material, traditionally prepared titanium implants do not provide necessary physiological compatibility and biointegration. These important properties can be achieved through personalized implant development, individual weight and size modification, physiological and anatomical osteointegration.

Recent studies have shown active integration of additive technologies in biomedical engineering. Also, this kind of manufacturing method of implants is energetically more effective than traditional method of metal machining. The advances in this field have allowed for the creation of complex geometrical titanium structures for implantation. Our study is based on the ability to design a structure with operating characteristics similar to osseous tissue. The process of modeling of these structures requires precise computerized design and extremely accurate production. An often-overseen variable in the production process of modern bionic structures is thermal treatment. This is one of the key steps in the preparation of high-quality personalized titanium biocompatible implants.

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2 Implant structural design

Titanium alloy implants are the most promising structures in bionic engineering. Comparison of physicochemical and structural properties of osseous tissue and VT1-00 titanium implant show excess mechanical characteristics of the later, which cause difficulties in biological osteointegration.

Table 1. Comparison of properties of bones and VT1-00

	Cast implant (VT1-00)	**Cortical bone**	**Cancellous bone**
Mass	4,5g	1,5g	0,2g
Elastic modulus	110 GPa	10 GPa	1-5 GPa
Tensile strength	200 MPa	50 MPa	5-10 MPa

Proper osteointegration requires the bioengineered implant to have a porous structure with microgeometrical configuration similar to that of osseous trabecular tissue. This requires the implant to have pore size varying from 100-1000μm, with over 70% porous consistency medium, as well as rough material surface quality [1-7]. Based on current understanding of material requirements for biointegration, we have developed a bionic structure with necessary functional and physical-structural characteristics for proper osteoinegration in vitro.

Given implant specifications. The developed titanium alloy 3D-printed bionic implant has an open cell pore size of 600μm. This network of spaces is divided by titanium trabeculae (300μm in diameter), similar to that of a cancellous bone. The open cells between artificial titanium trabeculae form tetragonal pores. This design provides the implantable material with significant advantages, including a highly stable and developed open cellular structure for complex osteointegration with trabecular osseous tissue. The high porous consistency (over 70%) and low elasticity coefficient (1.5 GPa - based on ANSYS analysis) with high porous consistency and biocompatible characteristics of titanium make this implantable material highly effective and suitable for bionic design (Table 1).

3 Biointegratable titanium implant production

The proposed titanium implant structure required a specially designed production algorithm. In order to acquire the desired implant characteristics, an experimental production program was developed, in which implant samples were prepared and later functionally tested. Implant preparation included selective laser melting (SLM) with subsequent annealing under different exposition protocols.

Heat treatment protocol. Selection of necessary heat treatment conditions was based on 6 different annealing protocols, which were carried out on different titanium implant samples after selective laser melting. 6 ductility samples with a 6mm working area (GOST 1497) and 6 cubic samples with a 10mm edge and a porous structure (ISO 13314) with previously listed specifications (see "Given implant specifications"). Samples were printed on a Russian-made 3D-printer for layered printing via selective laser melting (SLM) of metallic powdered materials (MeltMaster3D developed by Rosatom). Titanium VT1-00 metallic powder was used (Table 2).

Table 2. Chemical composition.

Alloy	Ti	O	N	H	Fe	Si	C	Other materials
VT1-00	base	0,11	0,02	0,001	0,02	-	0,02	-
GOST R ISO 5832-2 (Grade 1 ELI)	base	0,1	0,012	0,0125	0,1	-	0,03	-

As seen in Table 2, the titanium alloy chosen for the study has a high quality, similar to that of high purity Grade 1 ELI titanium. Requirements for mechanical properties in comparison with the properties according to GOST are given below (Table 3).

Table 3. Mechanical properties of bone and Titan.

Model	Treatment	$\sigma_{0,2}$, MPa (Flow stress)	σ_{B}, MPa (Ultimate tensile strength)	δ, % (Residual elongation)	ψ, % (Relative narrowing)	E, GPa (Young's modulus)
Requirements	not defined	210…700	150…530	12…40	20-30	1-7
Trabecular bone structure [8]	none		5-15			1-4
GOST R ISO 5832-2 (Grade 1 Eli)	annealed	200	140	30	-	110
GOST R ISO 5832-2 (Grade 4)	annealed	550	483	15	-	110

Selection of heat treatment conditions of unalloyed titanium after SLM. Evaluated temperature of heat treatment ranged between 625-725°C. This temperature range was chosen based on experience with undoped titanium and literature data. It should be noted that the increase in the annealing temperature was accompanied by a drop in strength for the entire investigated interval. An increase in the annealing temperature from 675°C to 725°C does not lead to an increase in plasticity. Based on our findings, the recommended heat treatment consists of 1 hour sample exposition at 675°C. This heat treatment allows the titanium implant sample to achieve required physical and mechanical properties and correspond to wrought titanium (GOST R ISO 5832-2, Grade 4).

Mechanical properties and annealing of porous implant samples. Annealing of the porous titanium VT1-00 implant samples after SLM was conducted similarly to that of unalloyed titanium samples. Mechanical qualities were evaluated via the ISO 13314 method for porous and cellular metals, the acquired results are presented in Table 4 and Figure 1, Figure 2. The test system ISO 13314 for mechanical, ductile, compression testing includes the following parameters: σpl - mean plateau stress at strain intervals between 20% and 30%, σmax – first maximum compressive strength during transition from elastic to plastic deformation on a stress-strain diagram for tension. In our opinion, the main characteristic of porous sample stability should be chosen from the minimum of the listed. σmax – can only be analyzed for

samples with 725°C annealing longways, annealing 725°C across, annealing 675°C across, and in this case the result is higher than the conditional yield strength. σpl for porous titanium VT1-00 samples was consistently >2 times greater than σ0.2. Therefore σ0.2 was used as a sample strength characteristic. The elasticity coefficient for all samples varied from 1.5±0.5 GPa. The authors conclude that the plasticity characteristic did not depend on annealing protocol, but the sample strength properties consequently decreased with the rise of annealing temperature.

Table 4. Physical and mechanical properties of samples depending on heat treatment conditions.

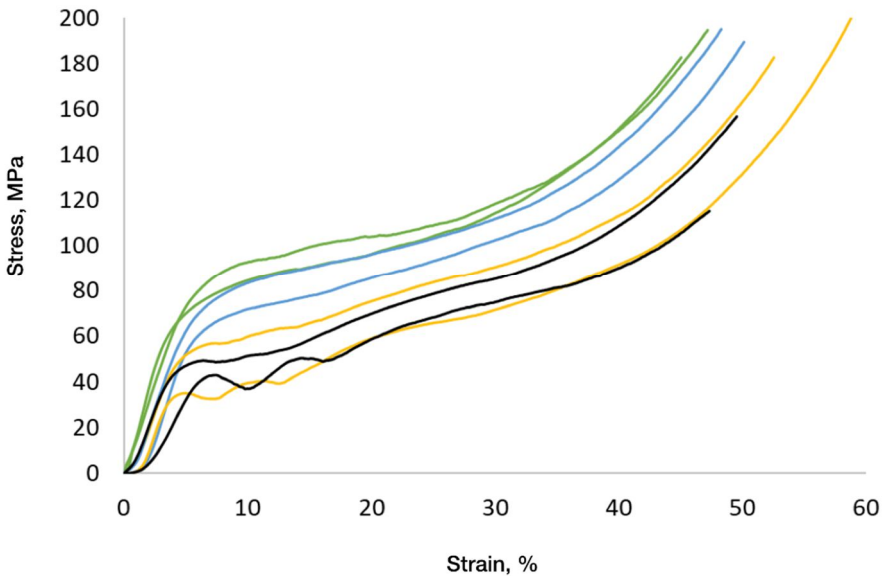| Sample preparation	Direction	$\sigma_{0,2}$, MPa	σ_{B}, MPa	σ_{max}, MPa	E, GPa	e_{ple}, %
SLM (without annealing)	longways	66	133	-	1,7	44
	across	60	131	-	1,9	43
Annealing at 625°C	longways	55	114	-	1,6	44
	across	57	119	-	1,6	43
Annealing at 675°C	longways	47	101	-	1,4	44
	across	30	81	40	1,7	44
Annealing at 725°C	longways	38	82	43	1,1	45
	across	41	97	49	1,1	44

Fig. 1. Stress-strain diagram to determine the characteristic values from compression testing of porous samples under different preparation protocol (after SLM only - green; annealing at 625°C - blue; annealing at 675°C - yellow, annealing at 725°C - black).
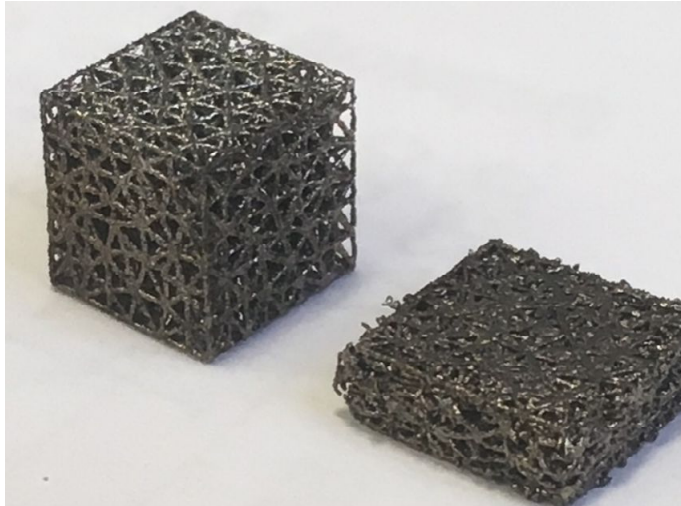
Fig. 2. Photo of porous samples before and after ISO 13314 testing.

Porous structures that underwent only SLM showed a tendency to deviate from original cubical form, due to weakness of unannealed titanium structural component and occurrence of local thermal stresses.

Conclusion

Personalized titanium VT1-00 implants in this study successfully adapted necessary characteristics for proper osteointegration with tuberous osseous tissue. The method of choice for bionic biointegratable implant production is SLM with consequent annealing at 675°C for 1 hour. Annealing should be performed until the implant is separated from the plate in order to exclude deformation of the implant.

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