

305. Characteristics of porous implant steel 316L for orthopedic applications

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Abstract. The porous materials are recently considered to be the prospective biomaterials for restorative medicine. The Young's modulus of porous metallic biomaterials is much lower than those of non-porous and comparable to modulus of hard tissues, which can improve the conditions of bone remodeling and healing. The porous structure is also interesting from tribological point of view. The usage of porous material for friction elements of joint endoprostheses changes the wear mechanisms and causes decrease of resistance to motion and material wear loss.

The aim of present research was to analyze the structure of porous 316L implant steel and its influence on main functional properties of sinters. Samples with porosity of 26, 33, and 41% were made of the 316 stainless steel by the powder metallurgy method. Microstructure was described using conventional image analysis techniques. The mechanical properties were determined in static compressing tests. Fatigue tests were conducted under the fully reversed strain controlled mode. A good correlation has been found between structural parameters and tribological as well as mechanical properties. The low-cyclic fatigue investigations confirm the steady properties of porous materials in the certain range of strain.

Keywords: porous biomaterials, microstructure analysis, mechanical properties, fatigue analysis

Introduction

The porous materials are recently considered to be the prospective biomaterials for restorative medicine. Although the initial focus has been on porous polymers and ceramics, porous metals are now commonly used in orthopedic and dental applications [1]. A number of authors have reported the favorable properties of such materials for biomedical devices [2], [3]. The Young's modulus of porous metallic biomaterials is much lower than those of non-porous and comparable to modulus of hard tissues (10-30 GPa) [4]. It allows to avoid the "stress-shielding", occurring when a bone is insufficiently loaded due the mismatch of properties between bone and metallic implant [4]. Consequently, the conditions of bone remodeling and healing improve. The porous structure is also demanded from implant osteointegration point of view. The roughened surface geometry promotes bone ingrowth into the pores and provides not only anchorage for the fixation, but also a system, which enables stresses to be transferred from the implant to the bone. The optimal porosity of implant material for ingrowths of new-bone tissues is in the range of 20-25 vol. % [5].

A conception of self-lubricating friction pair for hip joint endoprostheses made of porous implant alloys and saturated with simulated body fluid SBF has been proposed in the author's previous works [6]. Obtained results showed that the usage of porous material has quite changed the friction and wear mechanisms in the model system and caused decrease of resistance to motion and wear loss. Nevertheless, mechanical properties are strongly connected with material porosity. The production of porous implants with appropriate mechanical properties is currently an important issue in biomaterial research, because these have been perceived as the ideal bone substitute [7]-[8]. Many authors have reported the influence of porosity, in particular, the fraction, size, distribution, and morphology of pores on the decline of strength and fatigue behaviors [9], [10]. The high fatigue strength is attributed significantly to low overall porosity, pore clustering, and rounded pores [11]. On the other hand, the interconnected large pores are required from the point of view of good permeability, osteointegration, and tribological properties.

The aim of present research was to analyze the structure of porous 316L implant steel and its influence on main functional properties of sinters.

Materials and methods

The sintered compacts made of the 316L stainless steel powder (SANDVIK METNINOX STEEL LTD) have been investigated. Due to obtain the great value of open porosity there have been used powders with the particle size distribution of 125-250 μm . Materials were produced with the usage of powder metallurgy method. The powder was annealed in vacuum at the temperature of 950°C for 2 h before the process. After compaction at the pressure of 200, 400 and 600 MPa the specimens were sintered in vacuum at the temperature of 1230°C for 1 h.

The porosity of fabricated sinters was evaluated from the weight and the apparent volume of the specimen. The open porosity was measured by means of water penetration in accordance with ISO 2738:2001 and estimated with gas pycnometer Accu Pyc 1330. Microstructures of sintered compacts were observed with a scanning electron microscope (SEM) HITACHI S-3000N. The pore shape was determined using conventional image analysis techniques and characterized using a shape form factor: $F=4pA \cdot P^{-2}$, where A is the measured pore area and P the measured pore perimeter. A shape form factor of one denotes a perfectly round pore, and values that approach zero correspond to increasingly irregular pores. The permeability tests were performed using air according to EN ISO 4491-3:2006.

The mechanical properties were determined in static compressing tests in a universal testing machine INSTRON 8502 with a computer-control 8800 Fast Track 2. The static tests were conducted during the axial compression with the deformation velocity of 0.1mm s⁻¹. The fatigue tests were carried out under the fully reversed strain controlled mode. The frequency of cycles was 0.2Hz. The pattern of hysteresis loops evolution with the constant strain amplitude is presented in Figure 1. Tests were carried out at room temperature. The test was performed in an elastic range of deformation up to 1.7% of strain.

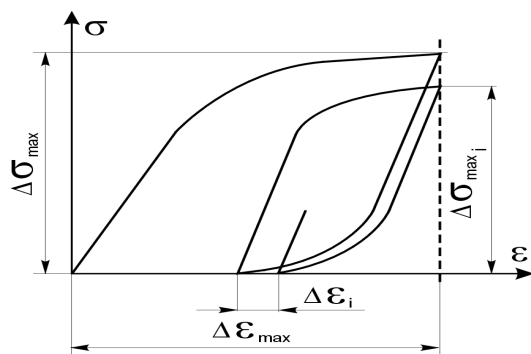


Fig. 1. Pattern of hysteresis loops evolution with the constant strain amplitude

Results and discussion

The specimens with porosity of 26, 33, and 41% were obtained (Fig. 2) as a result of chosen technological parameters. The share of open porosity, which participates in fluid circulation as well as in bone ingrowth processes,

amounted about 88% of the whole porosity. The detailed characteristics of investigated structures are presented in Table 1.

Quantitative analysis of the pore distribution for the examined sinters, shown in Fig. 3, indicated the insignificant effect of pressing pressure on the share of large pores. Pore shape analysis proved that all structures had similar pore parameters. The lowest value of shape factor for sinters characterized by higher porosity indicated that the most expanded pores surface in this case. The influence of increasing density due to higher pressure applied resulted in slight increase of average shape factor. This parameter is very important from corrosion resistance point of view because the large metal surface contacting with body fluids might cause increasing of corrosion current and intensify process.

As it should be expected, the permeability was strongly depended on materials porosity (Table 1). The value of permeability coefficient significantly increased with increasing porosity and pores size. This good correlated with results of tribological tests, which were detail reported in previous author's works [6], [12]. Analysis confirmed that the using of porous material has quite changed the friction and wear mechanism and caused decrease of resistance to motion and wear loss. It could be declared that the larger porosity is the larger effect.

The stress-strain dependence acquired during static compression proved good plastic properties of porous sinters (Fig. 4). In the whole accepted range of load (up to 40% of relative strain), plastic deformation of material ensued without any signs of cracking. It allowed defining only the value of yield strength $R_{0.2}$ (Table 2).

The variable loads caused changes in mechanical properties of materials, especially porous. Exemplary changes of strain character are readily seen on the graph of static compression performed with repeated unloading and reloading (Fig. 5). The AB segment presented the features of linear permanent deformation of porous specimen. However, the CD segment testified the elastic properties and allowed to evaluate the modulus of elasticity (Young's modulus). The value of porous sinters coefficient of elasticity was keeping on a constant level during the following strain cycles, which testifies the stable properties of these materials. It can be noticed, that the Young's modulus values of obtained porous sinters are comparable to the modulus of cortical bone.

As it can be expected, the values of examined properties are strongly depended on the sinters porosity (Table 2). The complex fatigue processes took place in porous material as result of variable loads. The typical static characteristics gave in the fatigue features. In order to further quantify the damage evolution during fatigue of the porous steel, stress-strain hysteresis experiments were conducted with the constant strain amplitude $\Delta\epsilon_{\text{max}}$ (Fig.1).

The graphs $\Delta\sigma_{\text{max}} = f(N)$ were prepared on the basis of measured loads according to the determined strain range during particular cycle (Fig.6). As the number of cycles increased the decrease of stress range was observed during

certain amount of cycles, which implied the phenomenon of the fatigue life decrease. Than the $\Delta\sigma_{max}$ value stabilized and was keeping at a constant level σ_n . This phase of curve testified the fatigue stability of material in the examined range (up to 1000 loading cycles). All tested samples showed similar behaviors. The σ_n was estimated for two levels of $\Delta\epsilon_{max}$. Comparison between the values of stabilized stress σ_n and static compression results showed

the significant effect of the strain range on the σ_n values. The location of fatigue points below the static strain-stress curve indicated the weakening of porous material due to fatigue. However, the behavior of porous sinter during following cycles proved the stabilization of mechanical properties. The further high-cyclic fatigue investigations confirm the steady properties of porous materials in the certain range of loading.

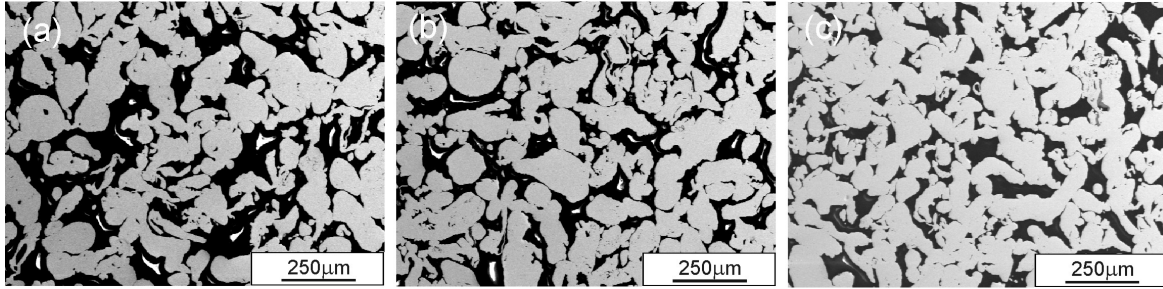


Fig. 2. BSE images of microstructure of 316L steel sinters with porosity of: (a) 41%, (b) 33%, (c) 26%

Table 1. Characteristic of porous 316L steel sinters

No series	Pressing pressure [MPa]	Porosity [%]			Specific surface Sv [μm^{-1}]	Average pore area A [μm^2]	Average pore perimeter P [μm]	Form factor $F=4\pi A \cdot P^{-2}$	Coefficient of permeability α
		Total	Open ¹	Open ²					
I	200	41	36.15	37.42	0.030	2.05	248	0.42	$9.53 \cdot 10^{-13}$
II	400	33	28,37	29.01	0.028	1.89	226	0.46	$4.77 \cdot 10^{-13}$
III	600	26	22.71	23.93	0.025	1.62	205	0.49	$2.33 \cdot 10^{-13}$

¹ measured by means of water penetration in accordance with ISO 2738:2001,

² estimated with gas pycnometer Accu Pyc 1330.

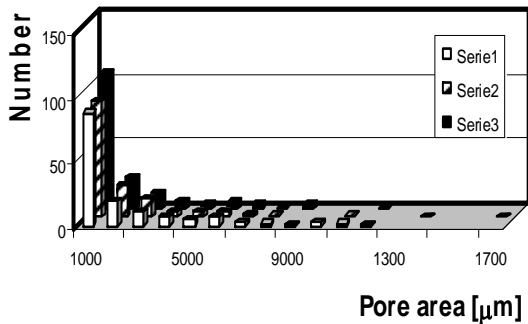


Fig. 3. Influence of compacting pressure on pore size distribution of 316L steel sinters

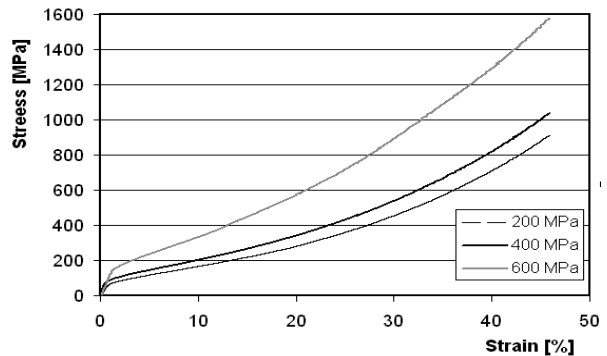


Fig. 4. Stress-strain curve under static compression of porous steel 316L

Table 2. Results of fatigue tests of porous steel 316L

Pressing pressure MPa	Porosity [%]	Static properties			$\Delta\epsilon_{max}$ [%]		σ_n [MPa]	
		E ₀ [MPa]	E ₁ [MPa]	R _{e02} [MPa]	1	2	1	2
200	41	4890.1	22340.0	65.50	1.035	1.671	38.2	41.7
400	33	6860.1	25478.0	71.81	0.663	0.833	36.8	53.3
600	26	11774.8	41469.6	88.04	0.774	1.098	62.8	82.5

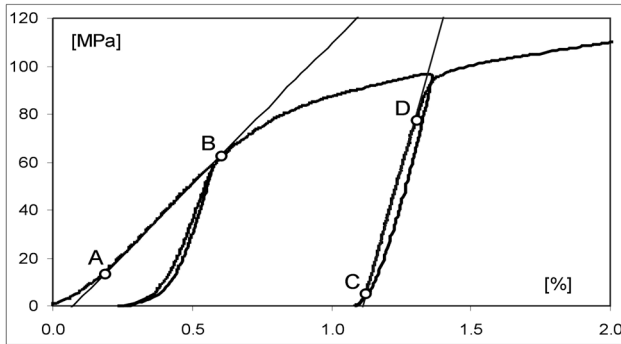


Fig. 5. Stress-strain curve of porous sintered 316L steel during repeated load

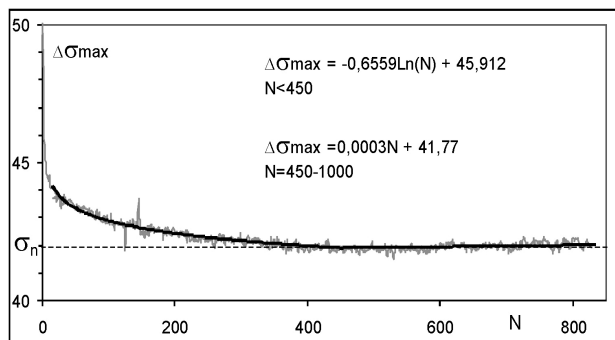


Fig. 6. Changes of dependence $\Delta\sigma_{\max}=f(N)$ during fatigue tests of porous steel 316L (specimen with porosity of 41%)

Conclusions

- The porous sinters made of 316L steel are characterized by good plastic properties during static compression tests.
- The low-cyclic fatigue tests showed the initial weakness of porous material and then stabilization of mechanical properties.
- The Young's modulus values of obtained porous sinters are comparable to the modulus of cortical bone.
- Porosity and mechanical properties of metallic sinters strongly depend on the compacting pressure.

Acknowledgements

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