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POROUS BIOMATERIAL FOR ORTHOPAEDIC IMPLANTS BASED ON TITANIUM ALLOY

ABSTRACT

Titanium and its alloys are widely used as biomaterials for orthopaedic applications. Research connected with their best corrosion and wear resistance, biocompatibility and bioactivity are still being conducted. The current research is also focused on the design and manufacturing of the porous materials based on e.g. Ti-13Nb-13Zr alloy, which can be applied for implants. One of the most effective manufacturing methods of the porous materials are powder metallurgy techniques. The aim of the presented work was the design of powder preparation procedure and design a parameters of pressing and sintering processes in order to obtain the porous structure from Ti-13Nb-13Zr alloy. Investigation results of the microstructure morphology, pore size and porosity of the obtained porous material on the base Ti-13Nb-13Zr alloy in dependence of the pressing and sintering parameters are also shown and discussed.

Keywords: *porous material, implants, biomaterial, titanium alloy*

INTRODUCTION

Despite of a major progress in a field of orthopedic biomaterials the bone implants properties - e.g. their's ability to fix to a bones are still remain under development [1-3]. Orthopedic implants still creating an unsolved problem because their mechanical properties are too high in comparison to the properties of human bones, for example Young's modulus. Such a significant difference of Young's modulus of the bone and currently used solid implants makes the bone is over protected against physical loads and inadequately loaded - so in consequence it may lead to it's resorption [4]. Animal testing and clinical research showed the dependence between the implants elasticity and bone resorption and prooved the negative influence of too high Young's modulus of the implants materials on bone structure [5-10]. As a result, the current research is focused on the development of the new porous materials for orthopedic application, mainly based on the currently used metallic biomaterials. (e.g. Co-Cr and Ti alloys).

The first concept of the use of the porous metals in biomedicine (for osseointegration) was introduced by Weber and White in 1972 [12, 13]. In early 70's of

XX century a numerous investigations with use of the porous materials (ceramic, polymer and metallic) have been conducted [4,14-24].
Among many different metals used for orthopedic implants manufacturing, the tantalum and it's alloys is one of the less examined materials. Usage of the stainless steels or Ni-Ti alloys for implants can provoke allergic reactions in contact with human body, from the other hand cobalt and it's alloys show a lower corrosion resistance [4]. In conclusion, the titanium and it's alloys seems to be the most appropriate or even the sole candidate as a best orthopedic material.
Currently, there are many different fabrication methods for porous materials. The most common used methods are: powder metallurgy (conventional or with use of space holders - the space holders can be removed by temperature or chemical treatment), plasma spraying, laser remelting, rapid prototyping (by SLM/SLS methods), metallic foams manufacturing, heat and chemical treatment (hydrogenation) and the others.
The powder metallurgy (PM) technique is one of the cheapest and fastest method of manufacturing. It enables to obtain a porous structures with random pore size and porosity.
As is mentioned above, the PM technique can be performed without and with a use of space holders. The space holders method is used for obtaining the porous materials with higher porosity. Before or during sintering process the space holders can be removed by melting or by dissolution.
The SLM method is used in order to obtain: uniform porous structure, graded porosity throughout specimen body or graded porous structure with a solid (non-porous) center of part (specimen).

EXPERIMENTS AND RESULTS

The aim of the presented work was to obtain the porous structure made of the selected material. The properties of the obtained structure should be very close to the properties of the human bones - e.g. bioactivity and biocompatibility, appropriate static and fatigue resistance, durability, Young modulus, pore size and porosity. In order to select the base material for the research purposes the following criteria have been determined:

- high corrosion resistance,
- high wear resistance,
- high compatibility with human body,
- imperceptible or none allergic and toxic reactions with contact of human body,
- ability to carry the expected loads,
- light weight.

Following above assumptions, more than 30 Ti alloys have been considered. The Ti-13Nb-13Zr alloy has been finally selected as the most suitable material: it contains neither Al and V and it has one of the lowest Young modulus values and reasonable fatigue strength.
The chemical composition of a selected as a base material Ti alloy is given below, in Table 1.

Table 1. *Chemical composition of the investigated Ti alloy, in wt. %*

Nb	Zr	Fe	C	N	O	Ti
13	13	0.05	0.04	0.019	0.11	rem.

As a fabrication method of the porous structure the classic PM (without use of space holders) method has been chosen among other.
In this paper the results of attempts to obtain the porous structure from selected Ti alloy by PM method are presented.

Powder preparation

For further research, the powder of the selected Ti-13Nb-13Zr alloy was prepared by a plasma spraying method.
By this method, the powder of the investigated alloy has been obtained by it's plasma melting and spraying over the cooled surface. Generally, the size of the obtained particles ranges between some micrometers up to 250 μm. Then the sieve analysis of the obtained powder was carried out. Powder particles were divided into four fractions: 0÷45, 45÷105, 105÷250 and above 250μm.
For the further studies connected with the obtaining porous structures by PM method the powder of particles size ranging from 106 to 250μm was choosen (Fig. 1).

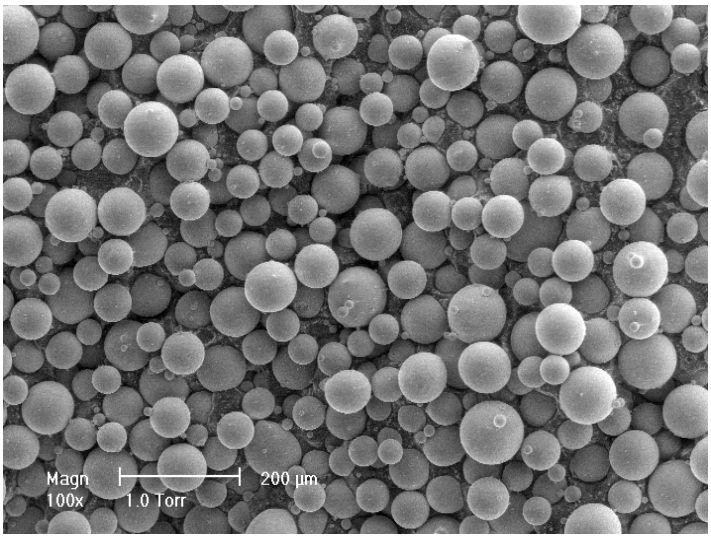


Fig. 1. A view of the powder of the investigated Ti alloy obtained by plasma spraying method

Compacting process

The process of powder compacting was performed using a die shown in Fig. 2. It was assumed that the compacting process will be performed as an uniaxial cold compaction process with use of the loads ranges from 795 MPa and 1200 MPa. There was no addition of any kind binder to the compacted powder.

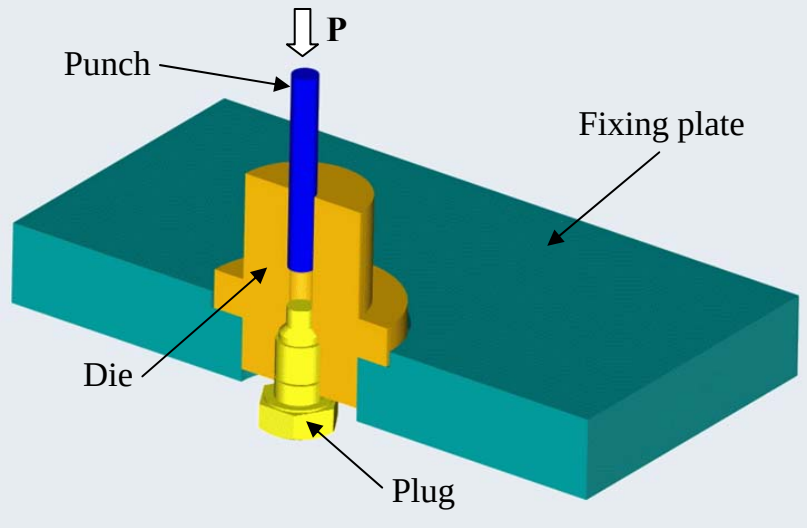


Fig. 2. View of a die for a metal powders cold compaction

In result of compacting process a porous round samples were obtained (so-called green bodies) of diameter and height of 8 mm.
In Fig. 3 the microstructure of the compacted samples (moldings) obtained from the spherical powder is presented.

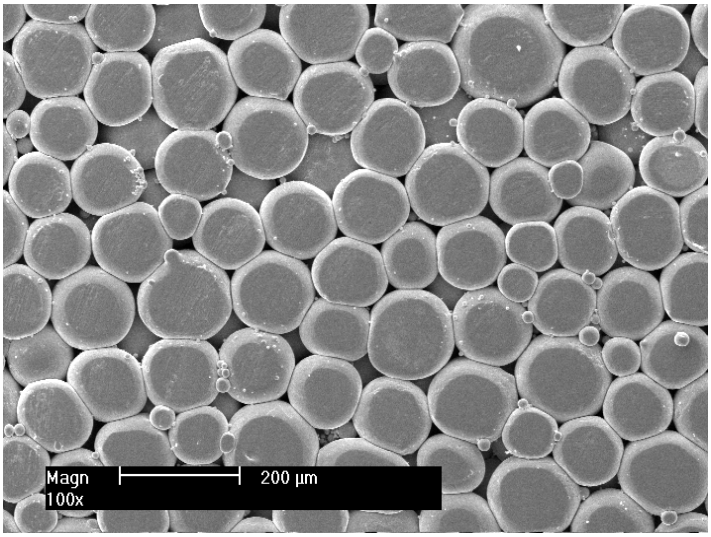


Fig. 3. Surface microstructure (SEM) of the sample after the compacting powder obtained by plasma spraying method

The mechanical compaction process of a Ti-13Nb-13Zr alloy particles in a powder form proved that is possible to obtain the material in which the grains are joined together.

Sintering process

Samples obtained by the compaction process were then sintered. Sintering process was carried out by means of the tubular oven, in high vacuum, at temperature of 1150ºC over 3.5 hour. Microstructure cross-sections (LM) of samples after compaction and sintering is presented in Fig. 4.

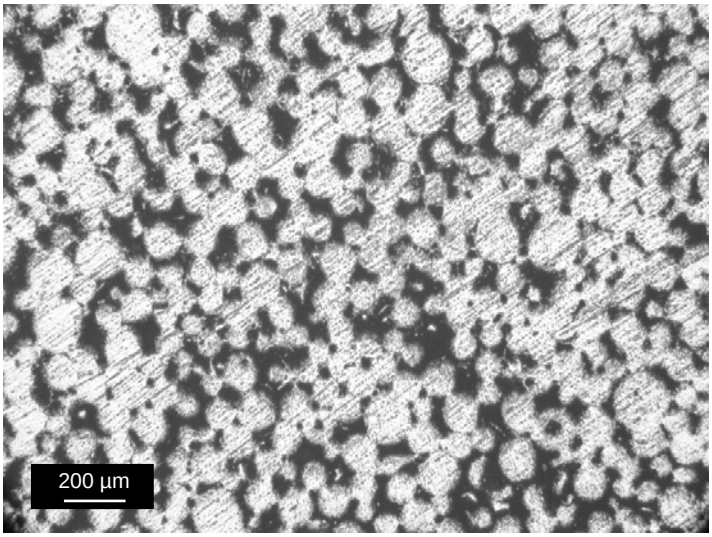


Fig. 4. Microstructure of sample after compacting and sintering. Powder obtained by plasma spraying method

Porosity of the samples after compacting and sintering processes was determined by the binary image analysis of microstructure shown in Fig. 4.
The average porosity of samples fabricated from powder obtained by plasma spraying method was approximately 36%.

Study of sinters microstructure morphology

The microstructure morphology of the obtained porous sinters made from the spherical powder (Fig. 5) have been also investigated, especially in bond (neck) area between sintered particles.

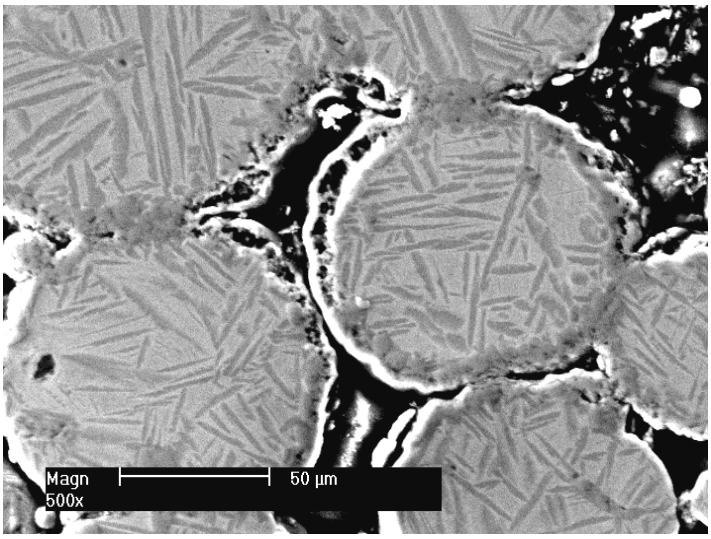
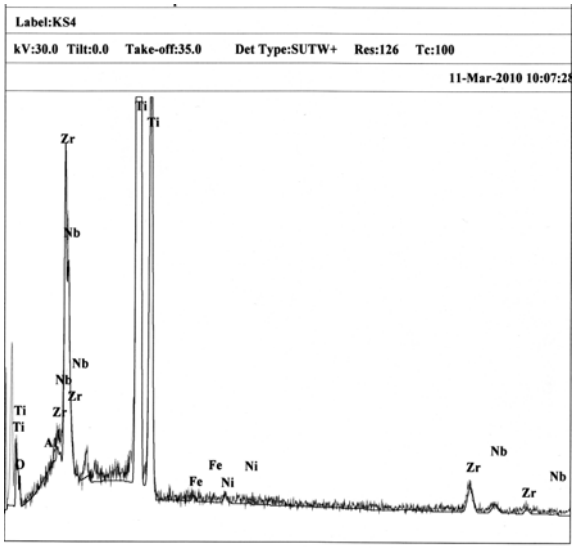


Fig. 5. Cross-section (SEM) of the obtained open-cell porous material that was fabricated by sintering of a Ti-13Nb-13Zr alloy powder

For this purpose, the EDS analysis of a structural bond between particles of a Ti-13Nb-13Zr alloy after compacting and sintering was carried out. Results are presented in Fig. 6 and 7. In Fig. 6 a chemical analysis of the sintered spherical particles in neck area is presented.



Element	Wt %	At %
O K	4.44	13.52
AlK	0.00	0.00
TiK	73.09	74.34
FeK	0.23	0.20
NiK	0.43	0.35
ZrK	14.86	7.94
NbK	6.95	3.65
Total	100.00	100.00

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	1.43	0.44	7.52	3.25
AlK	0.00	3.71	0.00	0.00
TiK	384.26	3.98	0.36	96.44
FeK	0.72	2.02	21.31	0.36
NiK	1.17	1.88	13.43	0.62
ZrK	5.03	0.84	3.64	5.99
NbK	1.83	0.64	6.81	2.84

Fig. 6. Chemical composition of a sintered region (SR) between two particles (neck region)

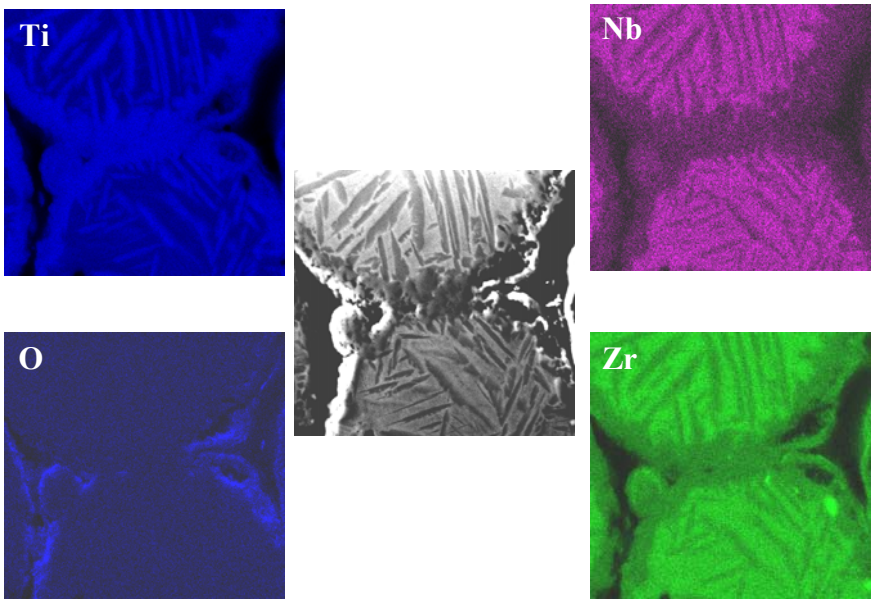


Fig. 7. Distribution of the elements in a structural bond between particles of a Ti-13Nb-13Zr alloy after compacting and sintering

As a result of the sintering process, in the contact area between the powder particles, the establishment of the characteristic necks and extension the plane of their connection are observed. Dominant processes during the particles sintering (joining) are the processes of diffusion – processes of mass transportation. The qualitative and quantitative elements distribution analysis in the bond area between particles (shown in Fig. 6 and 7) proved that at the sintering temperature (1150 ºC), the largest participation in the process of mass transfer have a titanium (Ti) and zirconium (Zr) elements. Titanium and zirconium elements (melting at 1649 °C and 1854,85 ºC accordingly) in the sintering temperature show a greater mobility in comparison with the atoms of niobium (2476,85 ºC). Thus, they dominate in the process of creating an interface connection (neck) between the sintered powder particles. In the necks area (Fig. 7) an increased content of Ti (73,09 %) and Zr (14,86 %) is observed in comparison with the amount of those elements inside the grains of investigated titanium alloy. The presence of oxygen on the surface of powder particles proves a partial oxidation during the sintering process.

CONCLUSIONS

As a result of the uniaxial compacting (P=40kN/30s) and sintering (T=1150ºC/3,5h) processes of the spherical particles obtained a porous material with a open structure and porosity up to ~ 36%.
Sintering enables bonding powder particles via element's diffusion processes - mainly titanium and zirconium.
The powder metallurgy method (PM) is useful in manufacturing porous material made of a Ti-13Nb-13Zr alloy used for orthopedic implants.

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