

## Ti-13Nb-13Zr SCAFFOLD PRODUCTION USING HYDROGENATED ALLOY POWDER AND SPACE-HOLDER TECHNIQUE

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## SUMMARY

When the local bone defect requires mechanical support, porous metal has been applied [1–3]. Although the Ti-13Nb-13Zr alloy was developed to reduce the elastic modulus, compared to other metal materials applied in the orthopedic area, it is still higher than the bones [4]. In the present work we decided to use the hydrogenated Ti-13Nb-13Zr powder and the space-holder technique to develop a cheaper and suitable metallic scaffold. [5–8]. First, we obtained the Ti-13Nb-13Zr alloy rod by arc-melt, solubilization heat treatment at 1000°C for 1 h followed by quench and forging in a rotary swage. Then we machined it to obtain chips, which were washed, etched, dried, and hydrogenated at 950°C and 0,31MPa of hydrogen gas atmosphere by 7h, followed by quench. The chips were then milled for 2 h in a Fritsh Pulverisette mill, using a jar of Ti, Nb balls, a ball-to-powder ratio of 5:1 and a 216 rpm mill speed. The powder was analyzed by SEM+EDS, XRD, XRF, and TGA, and the particle size was calculated by SEM image analyzes. The hydrogenated alloy powder was mixed with naphthalene (previously sieved in the range 500µm to 1mm), and PVAl solution, and pressed in a tester machine using pressure of

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200MPa, maintained for 2 minutes, with stearine lubrificated 6mm diameter cylindric matrix and punchs. One sample was sintered in vaccum better than 10<sup>-5</sup> mbar in four steps: heating using 5°C/min until 115°C, soaking for 30 minutes; heating using 5°C/min until 390°C, soaking for 24h; heating using 10°C/min until 900°C, soaking for 5h; heating using 10°C/min until 1000°C, soaking for 1h; and the sample was called Ti13Nb13ZrH2009. Another sample was prepared changing the 3<sup>rd</sup> step, heating until 900°C for 4h, and the 4<sup>th</sup> step, heating until 1000°C for 3.5h, and called Ti13Nb13ZrH4002. The samples were furnace cooled and analyzed by Archimedes test, helium picnometry, microtomography, mercury porosimeter, SEM+EDS, XRD, and Vickers hardness. The hydrogenated powder showed  $TiH_{0.85-1}$  and  $TiH_{1.91-2}$  (Ti- $\delta$ ) phases, particle size  $D_{50}$  near 2µm, and small quantity of impurities from hydrogenation process. The samples presented equiaxied  $\alpha$ -phase distributed in  $\beta$ , with some areas with higher concentration of Nb and Zr in the microstructure. The Ti13Nb13ZrH4002 sample also showed needle shaped  $\alpha$ ' and  $\alpha$ '', as well as areas of unreacted Nb. The scaffolds showed around 60% porosity, with homogeneous macropores distributed through the material, interconnected with the surface and with other pores inside the material. Ti13Nb13ZrH2009 and Ti13Nb13ZrH4002 had 45% and 48% of pores with size in the range of 100µm to 900µm, 31% and 17% of pores with size in the range of 10µm to 100µm, and 24% and 35% of pores with size in the range of 1µm to 10µm, respectively. Although more investigation must be conducted, the results showed that the process is promising for the production of Ti-13Nb-13Zr alloy scaffolds.

## REFERENCES

[1] G. Tang, Z. Liu, Y. Liu, J. Yu, X. Wang, Z. Tan, X. Ye, Recent Trends in the Development of Bone Regenerative Biomaterials, Front. Cell Dev. Biol. 9 (2021) 1–18. https://doi.org/10.3389/fcell.2021.665813.

[2] A.K. Aggarwal, V. Baburaj, Managing bone defects in primary total knee arthroplasty: options and current trends, Musculoskelet. Surg. 105 (2021) 31–38. https://doi.org/10.1007/s12306-020-00683-7.

[3] H. Migaud, H. Common, J. Girard, D. Huten, S. Putman, Acetabular reconstruction using porous metallic material in complex revision total hip arthroplasty: A systematic review, Orthop. Traumatol. Surg. Res. 105 (2019) S53–S61. https://doi.org/10.1016/j.otsr.2018.04.030.



[4] M. Long, H.J. Rack, Titanium alloys in total joint replacement—a materials science perspective, Biomaterials. 8 (1994) 597–611. https://doi.org/10.1016/S0142-9612(97)00146-4.

[5] A. Bigham, F. Foroughi, E. Rezvani Ghomi, M. Rafienia, R.E. Neisiany, S. Ramakrishna, The journey of multifunctional bone scaffolds fabricated from traditional toward modern techniques, Bio-Design Manuf. 3 (2020) 281–306. https://doi.org/10.1007/s42242-020-00094-4.

[6] A. Rodriguez-Contreras, M. Punset, J.A. Calero, F.J. Gil, E. Ruperez, J.M. Manero, Powder metallurgy with space holder for porous titanium implants: A review, J. Mater. Sci. Technol. 76 (2021) 129–149. https://doi.org/10.1016/j.jmst.2020.11.005.

[7] B. Arifvianto, J. Zhou, Fabrication of Metallic Biomedical Scaffolds with the Space Holder Method: A Review, Materials (Basel). 7 (2014) 3588–3622. https://doi.org/10.3390/ma7053588.

[8] O. Ivasishin, V. Moxson, Low-cost titanium hydride powder metallurgy, in: Titan. Powder Metall., Elsevier, 2015: pp. 117–148. https://doi.org/10.1016/B978-0-12-800054-0.00008-3.