

## INFLUENCE OF SEVERE PLASTIC DEFORMATION ON Ti13Nb13Zr SURFACE MORPHOLOGY

*Asli G. BULUTSUZ*<sup>1</sup>, M. Emin YURCI<sup>1</sup>, Malgorzata LEWANDOWSKA<sup>2</sup>

<sup>1</sup> Yildiz Technical University, Mechanical Engineering Department İstanbul Turkey,  
[asligunaya@gmail.com](mailto:asligunaya@gmail.com), [meyurci@yahoo.com](mailto:meyurci@yahoo.com)

<sup>2</sup> Warsaw Technical University, Materials Science Engineering Department, Warsaw Poland,  
[malew@inmat.pw.edu.pl](mailto:malew@inmat.pw.edu.pl)

**Abstract** – In this study surface behaviour of sand blasted and acid etched ultra-fine grained Ti13Zr13Nb specimens were investigated. Surface characters are a crucial factor that effect osseointegration performance of implants. Ti13Nb13Zr specimens were processed by means of hydrostatic extrusion technique. The effect of the grain size and surface modification on the surface roughness and topography were systematically investigated with stylus-profilometer and scanning electron microscope.

**Keywords:** Surface characterization, Surface Roughness, SPD, Biomaterial.

### 1. INTRODUCTION

Titanium and its alloys are most common materials for biomedical and dental applications due to their close mechanical properties to human body, high specific strength with low elastic modulus and good corrosion resistance [1-3]. Among their mechanical properties, surface characteristics are also important for biomedical application. With the development of severe plastic deformation techniques, ultrafine-grain sized alloy metals become attractive because of its good properties [4-8]. An enhanced mechanical strength [4], good mechanical compability to the body with low young modulus [5-7] and good biocompatibility with non-toxic behaviour [8] on  $\beta$  type ultrafine-grained titanium were reported. In addition, it is reported that small grain size provides rapid cell formation on the implant surfaces [9, 10]. Decreasing cell sizes increases grain boundary lengths. According to the literature cells favoured to accumulate along the grain boundaries [11]. Besides, biomaterials still need some surface modification to biologically activate their bioinert surfaces [12].

In literature there are plenty reports for surface modification of biomaterials to provide a proper surface morphology and chemical composition for cell adhesion on surfaces. The cited studies have been performed on a wide variety of material surfaces produced through various processes, using different modification techniques including blasting [13, 14, 16], chemical etching [15,16], laser pulses [17], micro-arc oxidation [18], coating technique [19]. The results indicate that surface characteristics have a significant effect on the osseointegration success.

Severe plastic deformation techniques bring about grain size down to sub-micron level [20-25]. For titanium alloys some biologically toxic elements (Al, V) are being widely used to increase mechanical properties for implants [26-27]. This plastic deformation technique enables improving material strength without changing its chemical composition. Till now, a number of severe plastic deformation techniques have been developed, including hydrostatic extrusion [21, 22], high pressure torsion [23], equal-channel angular pressing [24, 25].

The purpose of the present work is to investigate surface morphology and roughness of ultrafine grained Ti13Nb13Zr for biomaterial usage. The specimen surface morphologies and roughness values are compared with coarse grained Ti13Nb13Zr and pure titanium (Grade 4). After sand blasting of modified sample discs surface characteristics were observed with SEM investigations and quantified with roughness measurements by means of stylus-profilometer.

### 2. MATERIALS METHOD

The experiments were conducted using Ti–13Nb–13Zr alloy with chemical composition given in Table 1. The material was supplied in the form of cylindrical bars from Timet, USA. The specimens were in roll geometry with 30 mm diameter. Hydrostatic extrusion applied ambient condition. For both extrusion steps the hydrostatic pressure was measured nearly 1 GPa. After each extrusion the materials were water quenched at the die exit.

Table 1. Ti13Nb13Zr Chemical Composition

Material	Wt(%)
Ti	Base
Nb	12,5
Zr	13,8
Fe	0,059
C	0,009
N	0,024
O	0,09

H	0,003
---	-------

For the surface morphology, three discs selected from each specimen group. Specimens were observed on a scanning electron microscope with energy dispersive spectroscopy (EDS) for qualitative chemical analysis.

The roughness parameters were measured two-dimensionally with a contact method using a stylus profilometer (Mitutoyo). Three discs from each group were used; the roughness parameters were determined in two directions in each specimen ( $n = 5$ ). The parameters for characterizing the roughness were: arithmetic mean of the absolute values of roughness (Ra), and the root square value of average roughness (Rq).

Before blasting procedure, samples were either abraded with P200 silicon carbide abrasive metallographically polished to a 1  $\mu\text{m}$  finish using diamond paste. Samples were grit-blasted at 80 psi using 60-100  $\mu\text{m}$  diameter particle sized titanium oxide abrasive. Samples were blasted from a 60 mm distance to provide a uniform surface topography.

### 3. CONCLUSIONS

Biomedical implants require a proper surface roughness and morphology for a successful osseointegration performance. Homogenous morphology enables proper fixation with equal cell adhesion on the implant surface. Ultrafine grained Ti13Nb13Zr material expected to have more homogenous surface owing to its refined grain structure.

### ACKNOWLEDGMENTS

This work was supported within the EU 7<sup>th</sup> framework programme FP7/2007-13 under Marie-Curie project Grant No. 264635 (BioTiNet-ITN).

### REFERENCES

- [1] M. Niinomi, Recent metallic materials for biomedical applications, *Metall. Mater. Trans. A*, 33, pp. 477–486, 2002.
- [2] E.T. Den Braber, J.E. de Ruijter, H.T. Smits, L.A. Ginsel, A.F. von Recum, J.A. Jansen, Effect of parallel surface microgrooves and surface energy on cell growth, *J Biomed Mater Res*, 29, pp. 511–518, 1995.
- [3] X. Liu, P.K. Chu, C. Ding, Surface modification of titanium, titanium alloys, and related materials for biomedical applications, *Mater. Sci. Eng. R*, 47, pp. 49–121, 2004.
- [4] L. Taekyung, H. Yoon-Uk, L.S., Microstructure tailoring to enhance strength and ductility in Ti–13Nb–13Zr for biomedical applications, *Scripta Materialia*, Volume 69, Issues 11–12, Pages 785–788, 2013.
- [5] M. Nakai, M. Niinomi, J. Hieda, H. Yilmazer, Y. Todaka, Heterogeneous grain refinement of biomedical Ti–29Nb–13Ta–4.6Zr alloy through high-pressure

- torsion, *Scientia Iranica* Volume 20, Issue 3, Pages 1067–1070, 2013.
- [6] H. Matsumoto, S. Watanabe, S. Hanada Beta TiNbSn alloys with low Young's modulus and high strength *Mater. Trans.*, 46 (5), pp. 1070–1078, 2005.
- [7] D. Kuroda, M. Niinomi, M. Morinaga, Y. Kato, T. Yashiro Design and mechanical properties of new beta type titanium alloys for implant materials *Mater. Sci. Eng. A*, 243 (1–2), pp. 244–249, 1998.
- [8] M. Geetha, A.K. Singh, K. Muraleedharan, A.K. Gogia, R. Asokamani, Effect of thermomechanical processing on microstructure of a Ti–13Nb–13Zr alloy, *J Alloys Compd*, 329 (1–2), pp. 264–271, 2001.
- [9] T.J. Webster, C. Ergun, R.H. Doremus, R.W. Siegel, R. Bizios, Enhanced functions of osteoblasts on nanophase ceramics, *Biomaterials*, 21, pp. 1803–1810, 2000.
- [10] Y. Estrin, C. Kasper, S. Diederichs, R. Lapovok, Accelerated growth of preosteoblastic cells on ultrafine grained titanium, *J Biomed Mater Res*, 90A, pp. 1239–1242, 2009.
- [11] T.J. Webster, J.U. Ejiogor, Increased osteoblast adhesion on nanophase metals: Ti, Ti6Al4V, and CoCrMo, *Biomaterials*, 25, pp. 4731–4739, 2004.
- [12] C.Y. Zheng, F.L. Nie, Y.F. Zheng, Y. Cheng, S.C. Wei, R.Z. Valiev, Enhanced in vitro biocompatibility of ultrafine-grained biomedical NiTi alloy with microporous surface, *Applied Surface Science*, Volume 257, Issue 21, Pages 9086–9093, 2011.
- [13] M. Kern, V.P. Thompson, Sandblasting and silica coating of a glass-infiltrated alumina ceramic-volume loss, morphology, and changes in the surface composition, *J. Prosth Dent*, 71, pp. 453–461, 1994.
- [14] E. Conforto B.-O. Aronsson, A. Salito, C. Crestou, D. Caillard, Rough surfaces of titanium and titanium alloys for implants and prostheses, *Materials Science and Engineering: C* Volume 24, Issue 5, Pages 611–618, 2004.
- [15] Laurent Le Guehennec, Marco-Antonio Lopez-Heredia, Benedicte Enkel, Pierre Weiss, Yves Amouriq, Pierre Layrolle, Osteoblastic cell behaviour on different titanium implant surfaces, *Acta Biomaterialia* Volume 4, Issue 3, Pages 535–543, 2008.
- [16] Y. Germanier, S. Tosatti, N. Brogginini, M. Textor, D. Buser Enhanced bone apposition around biofunctionalized sandblasted and acid-etched titanium implant surfaces. A histomorphometric study in miniature pigs, *Clin Oral Impl Res*, pp. 251–257, 2006.
- [17] G. Pető, A. Karacs, Z. Pászti, L. Guczi, T. Divinyi, A. Joób, Surface treatment of screw shaped titanium dental implants by high intensity laser pulses, *Applied Surface Science* Volume 186, Issues 1–4, Pages 7–13, 2002
- [18] I. Çelik, A. Alasaran, G. Purcek, Effect of different surface oxidation treatments on structural, mechanical and tribological properties of ultrafine-grained titanium, *Surface and Coatings Technology*, Volume 258, Pages 842–848, 2014
- [19] M. Catauro, F. Bollino, F. Papale, R. Giovanardi, P. Veronesi, Corrosion behavior and mechanical properties of bioactive sol-gel coatings on titanium implants, *Materials Science and Engineering: C* Volume 43, Pages 375–382, 2014
- [20] R.Z. Valiev, Producing bulk nanostructured metals and alloys by severe plastic deformation (SPD), *Nanostructured Metals and Alloys*, Pages 3–39, 2011
- [21] W. Chrominski, M. Kulczyk, M. Lewandowska, K.J. Kurzydowski, Precipitation strengthening of ultrafine-grained Al–Mg–Si alloy processed by hydrostatic

- extrusion, *Materials Science and Engineering: A*, Volume 609, Pages 80-87, 2014
- [22] M. Lewandowska, A. T. Krawczyńska, M. Kulczyk, Krzysztof J. Kurzydłowski, Structure and properties of nano-sized Eurofer 97 steel obtained by hydrostatic extrusion, *Journal of Nuclear Materials*, Volumes 386–388, Pages 499-5027, 2009.
- [23] P. Bazarnik, Y. Huang, M. Lewandowska, T. G. Langdon, Structural impact on the Hall–Petch relationship in an Al–5Mg alloy processed by high-pressure torsion *Materials Science and Engineering: A*, Volume 626, Pages 9-15, 2015
- [24] G. J. Raab, R. Z. Valiev, T. C. Lowe, Y. T. Zhu Continuous processing of ultrafine grained Al by ECAP–Conform, *Materials Science and Engineering: A*, Volume 382, Issues 1–2, Pages 30-34, 2004
- [25] V.V Stolyarov, Y.T Zhu, T.C Lowe, R.K Islamgaliev, R.Z Valiev, A two-step SPD processing of ultrafine-grained titanium, *Nanostructured Materials*, Volume 11, Issue 7, Pages 947-954, 1999
- [26] Daisuke Kuroda, Mitsuo Niinomi, Masahiko Morinaga, Yoshihisa Kato, Toshiaki Yashiro, Design and mechanical properties of new  $\beta$  type titanium alloys for implant materials, *Materials Science and Engineering: A*, Volume 243, Issues 1–2, Pages 244–249, 1998
- [27] N. J. Hallab, C. Vermes, C. Messina, K. A. Roebuck, Tibor T. Glant and J. J. Jacobs, Concentration- and composition-dependent effects of metal ions on human MG-63 osteoblasts, *Journal of Biomedical Materials Research*, Volume 60, Issue 3, pages 420–433, 2002.