

Osseo-integration Improvement of Additive Manufactured Dental Alloys

Elena-Manuela Stanciu¹, Nestor Florido-Suarez², Pedro Socorro-Perdomo² and Julia Mirza-Rosca²

¹Transilvania University of Brasov, United States, ²University of Las Palmas de Gran Canaria, United States

Introduction

Titanium and titanium alloys, being biologically inert materials, are not chemically bonded to bone tissue. Consequently, in clinical use they must be fixed to the bone by mechanical interlocking, and the potentially loosening over an extended period can be a serious challenge (Zhang and Chen 2019; Kaur and Singh 2019). It has been found that biologically active materials, like hydroxyapatite and some glasses, were found that form strong chemical links with bone tissue and are being used extensively to fill bone defects. However, due to their brittleness, they are only suitable in situations where mechanical loads are absent or principally compressive. Titanium and its alloys are the most suitable to exhibit bioactivity in case of load-bearing components but must be previously subjected to a special surface treatment, involving changes in the passive film properties. In this paper, the improvement of osseointegration of two titanium alloys was studied.

Experimental

Ti-6Al-4V and Ti-6Al-4Fe alloys were obtained by Electron Beam Melting (Arcam AB, Sweden), built layer-by-layer on a titanium commercially pure grade 2 as substrate. The samples obtained in the form of flat discs (approx. 0.5 cm in diameter) were initially polished on a Struers TegraPol-11 polishing machine (López Ríos et al. 2020) with emery discs of various sizes (800 to 3000) and then with diamond paste (0.1 µm) on a polishing cloth. The surface of each sample has been left as a mirror surface to ensure reproducibility of results (Pałka and Pokrowiecki 2018). The samples were immersed for one day in a 10M NaOH aqueous solution at a temperature of 60°C and then immersed for 3 months in Carter-Brugirard artificial saliva. For comparison, samples with the same composition but without chemical treatment were immersed in the same conditions to differentiate the effects of the treatment.

Data on mechanical and electrochemical behavior are reported. SEM observations of the surface film and EDX measurements were carried out. Elemental distribution by EDX maps were employed to establish the composition of the oxide layer, the alloy morphology and to detect any contamination particles. Optical metallography, Vickers microhardness and tensile tests were used to analyse the mechanical behavior. The electrochemical performance of these alloys was determined in artificial saliva by electrochemical polarization and electrochemical impedance spectroscopy.

Results and discussion

During the study of the mechanism of bone union of biologically active ceramics, it has been shown that the fundamental requirement for synthetic materials to be bonded to a living bone is the development of a bone-like apatite film on their surface.

The resulting apatite is very close in composition and structure to the bone material. Therefore, osteoblasts favourably regenerate and differentiate to form apatite and collagen in this apatite film and, consequently, the adjacent bone can come into proximal contact with the apatite film from the surface. After this happens, a vigorous chemical link is created between the bone minerals and the surface of the apatite layer to decrease the energy of the interface between both (Zhenhuan et al. 2020).

Titanium and its alloys are generally covered with a thin TiO₂ passive layer, and hence is chemically durable. However, even this stable TiO₂ layer reacts with NaOH solution to form a sodium titanate gel, and this layer can be stabilized as an amorphous sodium titanate by a suitable heat treatment (Kokubo et al. 2004). This sodium titanate layer forms many Ti-OH groups on its surface in the living body via the exchange of its Na⁺ ions from the surface with H₃O⁺ ions in the surrounding body fluid.

The apatite layer is so tightly bonded to the substrates that a fracture occurs not at the apatite-substrate interface but in the glue or the apatite layer when a tensile stress is applied normally to the substrate at a crosshead speed of 1 mm.min⁻¹ (see Fig.1 and Fig.2). The estimated adhesive strengths between the apatite layer and the substrate ranged from 9.8 to 11.5 MPa.

The observation that apatite deposition occurred better on the contact substrates indicates that the increase of pH in the simulated body fluid could play a decisive role in the formation of apatite on NaOH-treated alloys. Evidently, a much higher pH is predicted to accumulate in the gap under the contact substrates due to the narrow pathway for ion diffusion, while a higher pH, necessary for apatite nucleation, could not be achieved in the proximity of the open surface due to the greater ease of ion diffusion. Thus, the decisive role of ion diffusion in the bioactivity of sodium titanate gel can be demonstrated. It also suggests that the biological activity of sodium titanate gel decreases with contact with aqueous solution because of the dissolution of the sodium component of the gel.

Conclusions

It turned out that the oxide films on the metal surface showed a tendency to passivation and very high stability and no evidence of any form of local corrosion was noted. The experimental electrochemical performance data of such layers were fitted by an equivalent circuit having two-time constants. The mechanical and EDX results support the existence of a porous outer passive film with a high oxygen concentration and a dense and protective inner passive film where titanium dioxide is the predominant compound. EIS results confirmed the mechanistic findings. The outcomes show that the employment of a surface processing improves the adherence of the passive film to the alloy surface and enhances the biocompatibility of the medical implants.

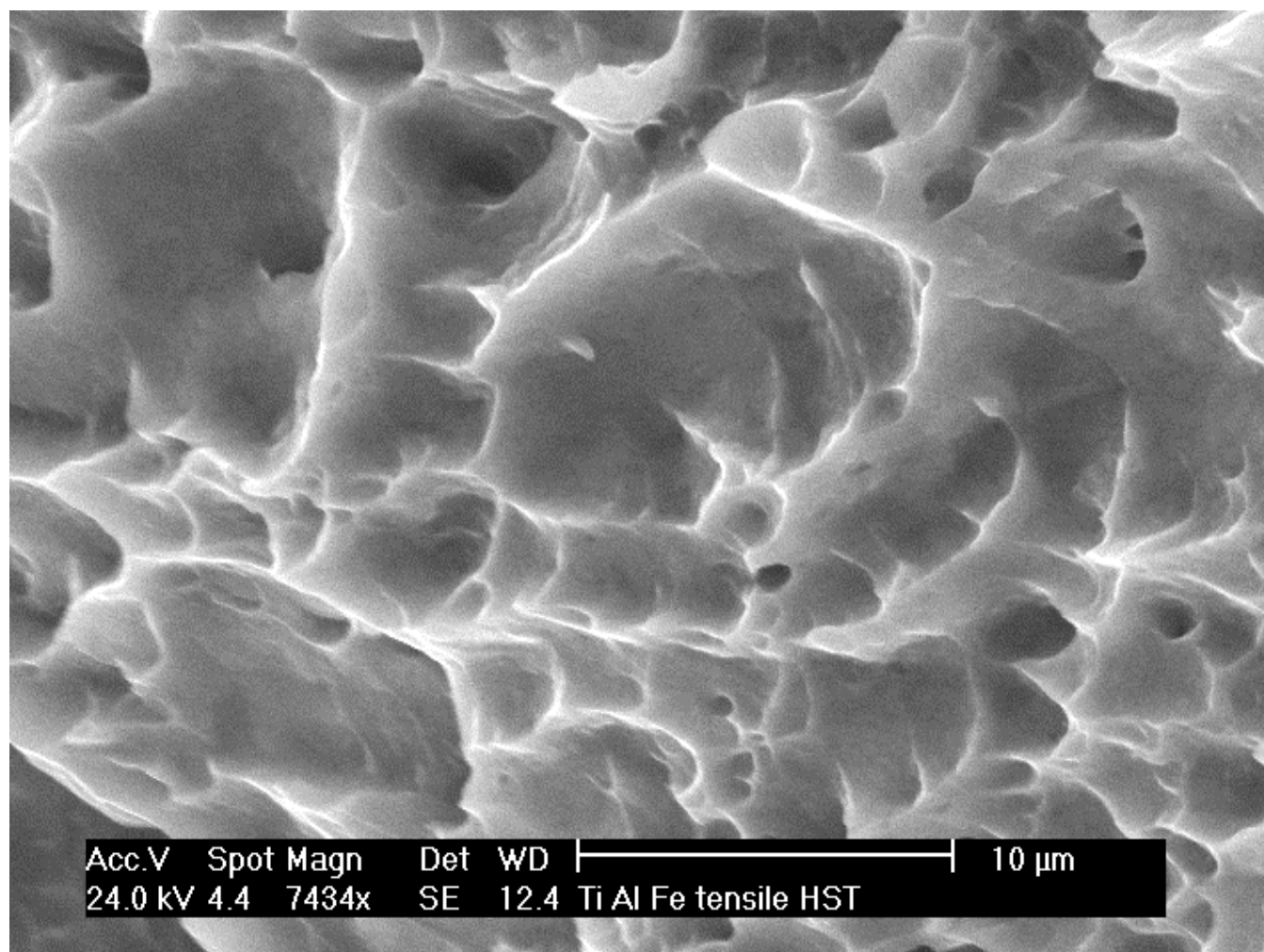


Figure 1. Fig.1 Ti6Al4Fe tensile test after NaOH treatment

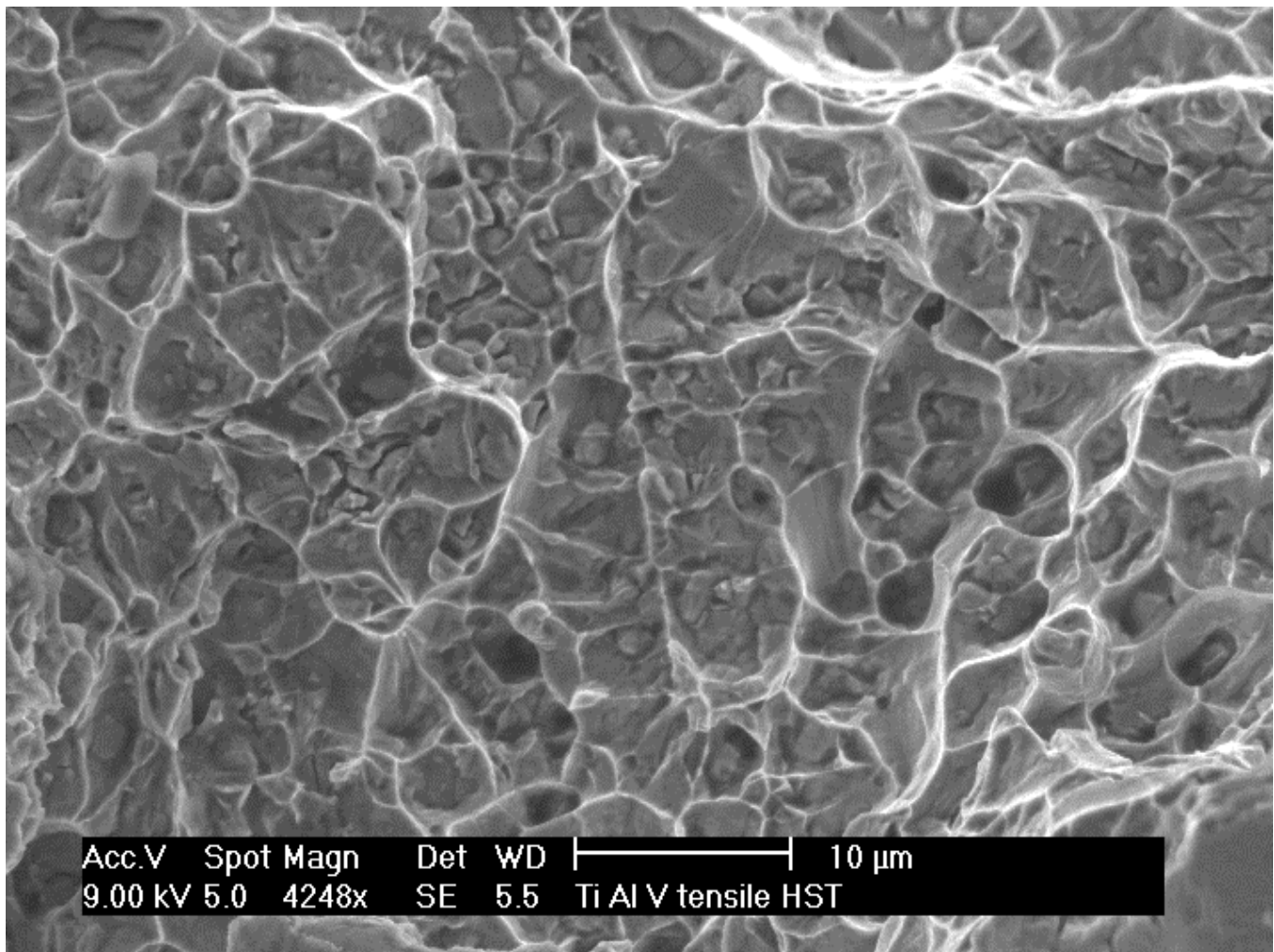


Figure 2. Fig.2 Ti6Al4V tensile test after NaOH treatment

References

- Kaur, Manmeet, and K. Singh. 2019. "Review on Titanium and Titanium Based Alloys as Biomaterials for Orthopaedic Applications." *Materials Science and Engineering C*. <https://doi.org/10.1016/j.msec.2019.04.064>.
- Kokubo, T., H.-M. Kim, M. Kawashita, and T. Nakamura. 2004. "Bioactive Metals: Preparation and Properties." *Journal of Materials Science: Materials in Medicine* 15 (2): 99–107. <https://doi.org/10.1023/B:JMSM.0000011809.36275.0c>.
- López Ríos, M., P. P. Socorro Perdomo, I. Voiculescu, V. Geanta, V. Crăciun, I. Boerasu, and J. C. Mirza Rosca. 2020. "Effects of Nickel Content on the Microstructure, Microhardness and Corrosion Behavior of High-Entropy AlCoCrFeNi_x Alloys." *Scientific Reports*. <https://doi.org/10.1038/s41598-020-78108-5>.
- Pałka, Krzysztof, and Rafał Pokrowiecki. 2018. "Porous Titanium Implants: A Review." *Advanced Engineering Materials*. <https://doi.org/10.1002/adem.201700648>.
- Zhang, Lai Chang, and Liang Yu Chen. 2019. "A Review on Biomedical Titanium Alloys: Recent Progress and Prospect." *Advanced Engineering Materials* 21 (4): 1–29. <https://doi.org/10.1002/adem.201801215>.
- Zhenhuan, Wu, Dai Yu, Luo Junsu, Ji Xiaowei, Xie Zongyu, Li Li, and Xie Xiaoli. 2020. "Physiochemical and Biological Evaluation of SLM-Manufactured Ti-10Ta-2Nb-2Zr Alloy for Biomedical Implant Applications." *Biomedical Materials (Bristol)* 15 (4). <https://doi.org/10.1088/1748-605X/ab7ff4>.