PRELIMINARY STUDIES OF NEW Ti ALLOYS WITH DIFFERENT Mo CONTENT

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Abstract

This work aims to investigate the mechanical characteristics and biocompatibility of two novel titanium alloys, Ti15Mo7Zr15Ta1Si and Ti20Mo7Zr15Ta0,75Si.These samples have previously undergone cutting, grinding, polishing, and chipping. The studied samples were subjected to electrochemical, metallographic and corrosion behavior. Ti15Mo7Zr15Ta1Si and Ti20Mo7Zr15Ta0.75Si, the study samples, have demonstrated high corrosion potentials, lower corrosion rates, and consequently higher corrosion resistance. In summary, this study's data indicates that both alloys exhibit good corrosion behavior.

Introduction

Biomaterials science has created new materials that can solve many of the medical issues of today and extend and improve human life because of the ongoing relevance of research in biology, chemistry, engineering, and medicine. But as life expectancy has grown in developed nations, so too has the number of elderly and obese people. The latter is a result of leading a more sedentary lifestyle, which increases their risk of developing chronic musculoskeletal conditions like osteoarthritis, which is especially common in the hips and knees, and necessitates more frequent surgical implant repairs.

The new Ti alloys with different Mo content were created by vacuum arc remelting, and this study examined their microstructure, microhardness and corrosion behavior.

Experimental

In order to perform various experiments and ascertain the effect of molybdenum addition in one of the samples, we have analyzed two different compositional alternatives of new alloys containing titanium, molybdenum, zirconium, tantalum and silicon. This has allowed us to obtain the properties of the alloys. Sample 1 composed of Ti15Mo7Zr15Ta1Si (62% Ti, 15% Mo, 7% Zr, 15% Ta, 1%Si) and Sample 2 composed of Ti20Mo7Zr15Ta0.75Si (57,25 % Ti, 20% Mo, 7% Zr, 15%Ta, 0.75% Si) are the chemical compositions of the alloys under study. High-quality elements, such as Zr (99% purity), Mo (99% purity) Ta (99% purity), and Ti (99% purity), were provided by Alfa Aesar through Thermo Fisher Scientific as raw materials. These alloys were produced at the Ghe. Asachi Technical Faculty of Materials Science and Engineering employing a vacuum arc remelting furnace (VAR), which used the heat generated by an electric arc between the electrode and the ingot to fuse a consumable electrode in a vacuum at a measured pace. The alloys were centrifuged, remelted six times (three times for each portion) in an inert argon environment in order to attain a sufficient level of homogeneity, and then solidified as an ingot. The new TiMoZrTaSi alloys were developed using arc melting because it yields the purest ingots possible. Las Palmas de Gran Canaria University received a portion of the ingots for testing and preparation.

Initially, a 4:1 ratio of epoxy resin was added to molds to prepare the surfaces of the two samples for embedding. That is to say, one drop of catalyst was added for every four drops of resin. The samples were then cut with a Buehler IsoMet 4000 precision saw (Buehler, Lake Bluff, IL, USA) longitudinally at a thickness of 1 to 1.5 mm.

Furthermore, the cutting tool was used to make vertical cuts that were roughly 0.5 mm thick. Next, utilizing the Struers TegraPol-11 polishing machine (Struers ApS, Ballerup, Denmark), grinding and polishing were done in two steps: first, with progressive grit silicon carbide papers from 280 to 1200, and then, for the final polish, with 0.1 microns of alpha alumina suspension to polish the surfaces to a mirror finish. The experimental procedures followed ASTM E3-11(2017) for the preparation of samples for metallography.

In order to eradicate any remaining dirt or contaminants, the samples were lastly submerged in a heated “Ultrasons-HD” ultrasonic device from J.P. Selecta (JPS, Barcelona, Spain) for ten minutes. For peer review, see Bioengineering 2022, 9, x. 4 of 23 surfaces with a mirror-like sheen using an alpha alumina suspension. The experimental procedures followed ASTM E3-11(2017) for the preparation of samples for metallography.

In metallography, the phases and compounds that comprise a metallic material are arranged spatially along with any impurities or possible mechanical flaws.

The Axio Vert.A1 MAT ZEISS optical metallographic microscope (Jena, Germany) was used to take surface images of each specimen in order to examine the microstructure.

The attacked surface of each sample was photographed after it was immersed in Kroll's reagent, which is composed of 20 mL glycerin, 30 mL hydrochloric acid, and 10 mL nitric acid, at intervals of roughly 15 s. Three attempts were made to complete the test.

For the electro-chemical tests, the samples were sequentially added to an electrochemical cell that had three electrodes: the samples functioned as the working electrodes, the reference electrode was a saturated calomel electrode, and the counter electrode was a platinum electrode.

To conduct the tests, the area of each sample was established. The Grifols Laboratories' Ringer solution (Barcelona, Spain) had the following mmol/L values: Na^+ 129.9, Cl^- 111.7, $C_3H_5O_3$ 27.2, K^+ 5.4, and Ca^{2+} 1.8.

The BioLogic Essential SP-150 potentiostat (Seyssinet-Pariset, France) was utilized to perform Corrosion Potential, Corrosion Rate, and Electrochemical Impedance Spectroscopy. The experiments were conducted in an aerated Ringer solution at 25 ◦C.

The corrosion potential was measured using the "Ecorr vs. time" technique over the course of 24 hours, with potential values of ±10 V and potential recordings made every 300 s or 200 mV apart. The acquired data were plotted as a potential vs. time graph, which could exhibit a trend toward passivation or corrosion or stay constant over time.

These experiments were conducted using the "Linear Polarization" approach, and the viability of the approach was confirmed by entering the sample surface area value and the 20-minute test period. The potential scanning, which was conducted with data collected every 0.50 seconds, showed a 0.167 mV/s time-variation relationship between −0.025 and 0.025 V against the open circuit potential (OCP) and an intensity that remained at 100% throughout the process. The corrosion rate estimates for each sample were then determined using EC-Lab's "Tafel Fit" method after these linear polarization curves were presented.

"Potential Electrochemical Impedance Spectroscopy" was chosen for the impedance measurement, and the surface value and the five-minute measurement period were input. For every sample, this measurement was done seven times at ± 300 mV vs. Ecorr in Ringer's solution, with ± 10 V for the maximum and lowest potential values. Equivalent circuits and Bode and Nyquist diagrams were used to illustrate these data.

In compliance with ISO 14577-1:2015, 10 measurements were made for each sample's applied load—in this case, 1, 5, and 10 gf—using the Future Tech FM-810 hardness tester (Kawasaki, Japan). As the stress increases, the mark may include pieces from several phases, giving an approximate measure of the material's overall hardness. It is possible that the mark will only be found in one phase when relatively light weights are positioned, making it possible to evaluate the hardness of that phase. Then, using the observed diagonal lengths, the Vickers microhardness values were automatically calculated by the iVicky software (v2.0, Sinowon, Dongguan, China). Plotting the quantity of indents made against the scan length was done.

Results and discussion

1) Potential (E_{corr})

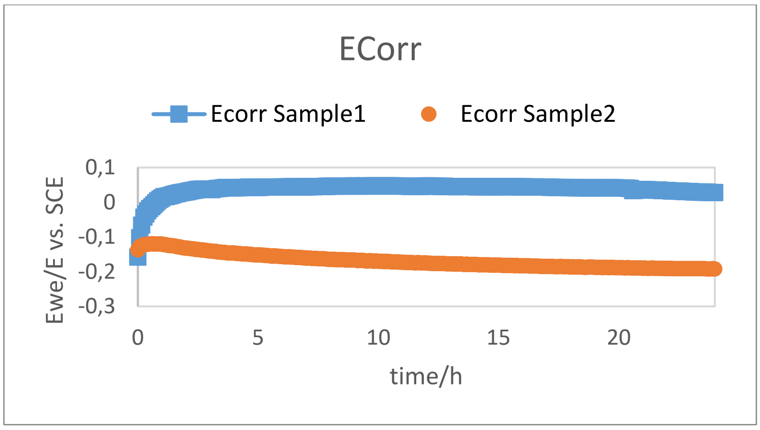

Figure 1. Corrosion potential curves for Ti15Mo7Zr15Ta1Si and Ti20Mo7Zr15Ta0,75Si

As the Molybdenum in the new Ti alloys increase, the corrosion potential becomes more negative. In Figure 1., we have the corrosion potentials for Sample1 Ti15Mo7Zr15Ta1Si and for Sample2 Ti20Mo7Zr15Ta0,75Si. It can be observed that Sample1 is passivating while Sample2 slowly corrodes.

1) Electrochemical impedance spectroscopy (EIS)

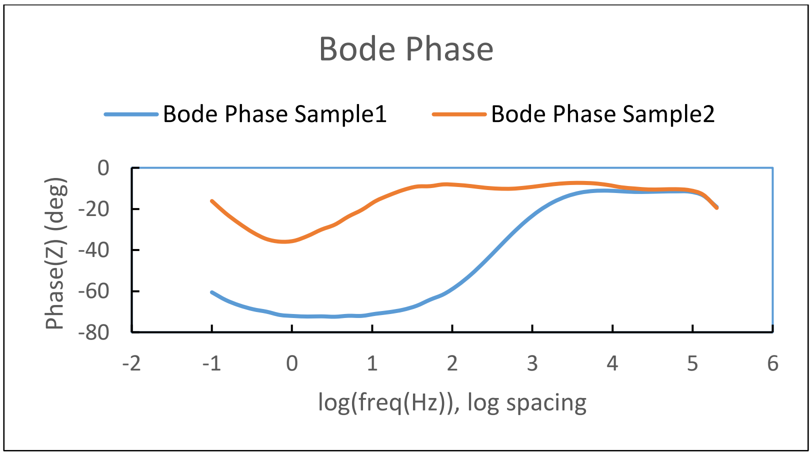

Figure 2. Bode Phase curves for Ti15Mo7Zr15Ta1Si and Ti20Mo7Zr15Ta0,75Si

The Bode Phase of Sample1(Ti15Mo7Zr15Ta1Si) and of Sample2 (Ti20Mo7Zr15Ta1Si) has a difference between the phases (see Figure 2). Sample1 have a low angle beside Sample2 which it´s angle is a little bigger. Because of the increase of the Mo, Sample2 tends to loose corrosion resistance.

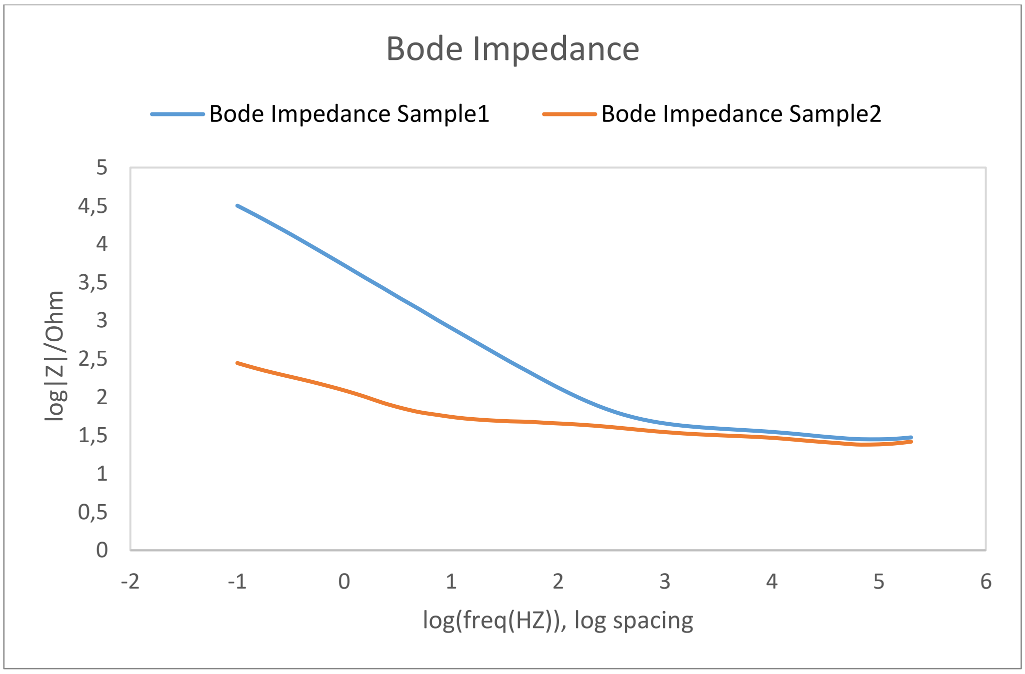

Figure 3. Bode Impedance curves for Ti15Mo7Zr15Ta1Si and Ti20Mo7Zr15Ta0,75Si

Using Figure 3 we can verify what it is said above that sample1 has a higher impedance level that means it is much more resistant to corrosion than Sample2.

Conclusion

From our analysis we can confirm that Sample1 with only 15% Mo is presenting good properties like good corrosion resistance and passivating capability. If we increase the percent to 20% Mo these properties diminish and from that Sample1 can be without fault the future material for biomedical applications.

Acknowledgements

I am very grateful to University of Las Palmas de Gran Canaria and The University Transilvania Brasov Erasmus+ for the occasion to work on this project.

References

[1] Cristina Jimenez-Marcos, Julia Claudia Mirza-Rosca, Madalina Simona Baltatu and Petrica Vizureanu in: „Experimental Research on New Developed Titanium Alloys for Biomedical Applications" Bioengineering 2022, 9, 686. https://doi.org/10.3390/bioengineering9110686

[2] Baltatu, M.S.; Vizureanu, P.; Sandu, A.V.; Florido-Suarez, N.; Saceleanu, M.V.; Mirza-Rosca, J.C. in : New Titanium Alloys, Promising Materials for Medical Devices." Materials 2021, 14, 5934. https://doi.org/10.3390/ma14205934