ELECTROCHEMICAL DEALLOYING OF TI-6AL-4V

3D 4 Health VIP Project

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Table of contents

Abstract

Complete

Introduction and Aim

The aim of this experiment was to increase and enhance the bioactivity of a printed titanium Ti-6Al-4V porous structure through the dealloying process, employing different combinations of variables such as varying electrolyte concentration and voltage. These surface modification techniques yield distinct microscopic morphologies, roughness, and wettability, potentially enhancing the biological inertness of the titanium alloy. The proven benefits of the porous structure include improved osteogenic differentiation, enhanced osteointegration ability, and promotion of cell function, rendering it suitable for applications such as spinal infusion.

Hypothesis

The combination of the highest concentration and highest voltage will do the most work on the structure creating the largest diameter for pore size. As per the study done on this, we can see their results which stated, "the pore size gradually increased, the grid structure became continuous and uniform as the electrolyte concentration increased"[1].

Materials

- Seven 5x5x5mm Ti6Al4V Gyroid Samples
- Sodium Hydroxide
- Distilled water
- Platinum (Cathode)

Equipment

- Potentiostat
- Three Electrode Electrochemical Cell
- Scanning Electron Microscope
- Safety glasses
- Lab coat

Method

1. Prepare a 1 Mol (40 g/L) and 5 Mol (80 g/L) solution of NaOH using distilled water.
2. Fill the electrochemical cell with 50mL of the 1 Mol solution
3. Place the platinum (cathode) and Ti6Al4V (anode) sample into the solution. Note only submerge the Ti6Al4V sample halfway into the solution.

4. Connect the electrochemical cell to the potentiostat and apply a 1 V potential difference for 30 minutes.
5. Repeat steps 2 to 4 with a new sample but increase the potential difference to 2V.
6. Repeat steps 2 to 4 with a new sample but increase the potential difference to 3V.
7. Repeat steps 2 to 6 using the 5 Mol solution.
8. Using the SEM machine collect images at magnifications 150x, 500x and 150 000x for each of the seven samples.

Results

The SEM images have been tabulated below into three columns of different image magnitude. It should be noted when viewing the 150,000x magnitude column, images vary in brightness. This is not significant, it simply occurs due to the method of acquiring SEM images.

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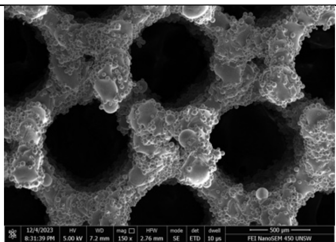
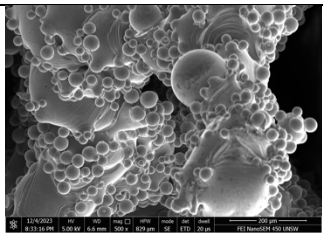
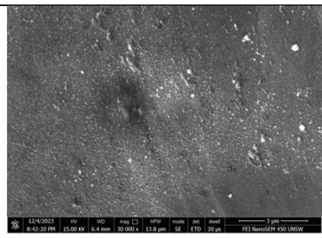
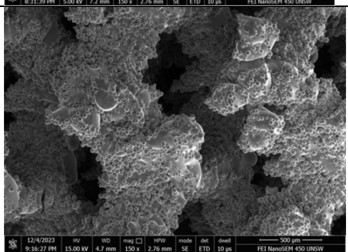
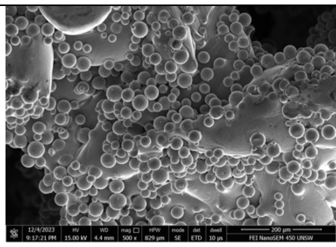
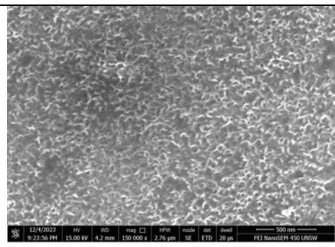
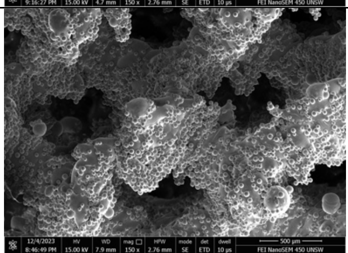
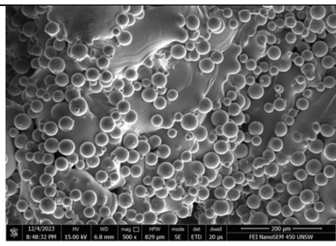
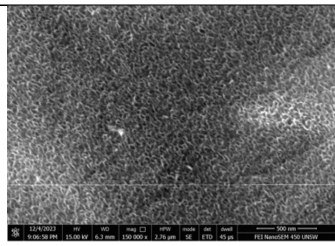
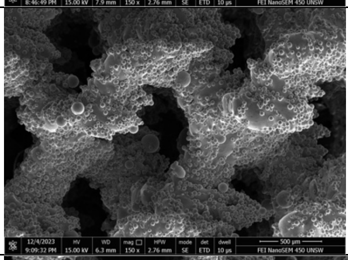
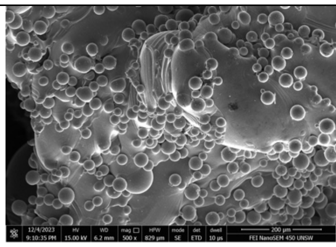
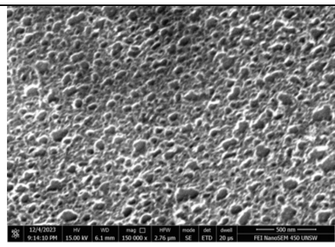
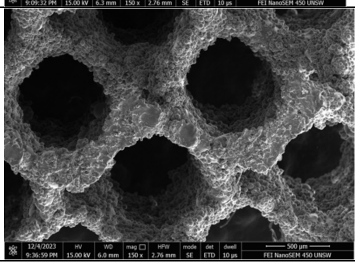
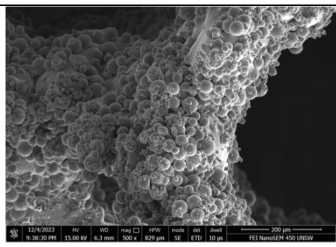
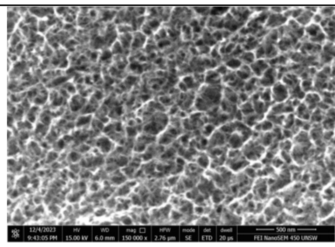
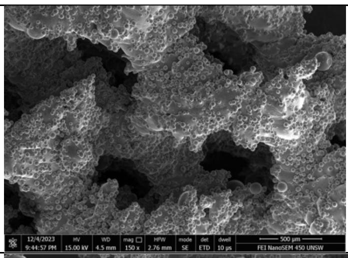
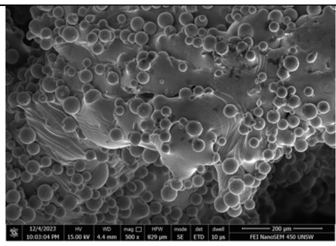
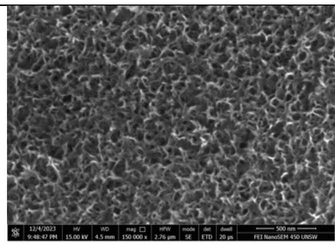
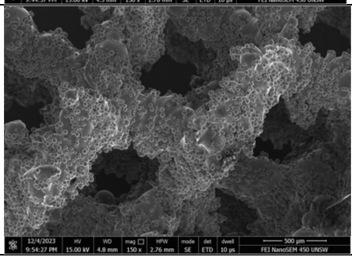
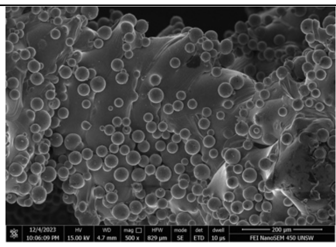
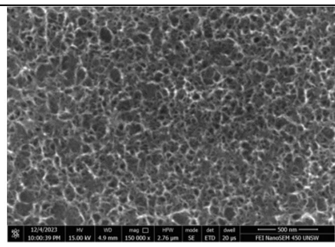
Reference			
1MOL 1V			
1MOL 2V			
1MOL 3V			
5MOL 1V			

5MOL 2V			
5MOL 3V			

Discussion

Compare the 1M
Compare the 5M

From the images provided above, it's evident that the 150x and 500x magnifications don't offer substantial insights into the actual structure of the titanium alloy. However, a more detailed examination at 150,000x, in comparison with our reference, reveals significant disparities. Notably, as both concentration and voltage increased, a porous structure emerged, indicating the success of our experiment. This outcome aligns with our hypothesis and the research paper ''Improvement on biosafety and bioactivity of Ti–6Al–4V alloys by construction the three-dimensional grid structure though electrochemical dealloying,''[1] .

Given additional time and resources, an exploration of higher parameters, such as 10MOL and 5V, could provide valuable insights. This might elucidate whether it is more important to increase voltage or concentration to successfully create large porous diameters.

Further analysis of the images suggests that the sample with 5MOL and 3V exhibited a noticeably deeper porous structure than the one with 1MOL and 3V. However, caution is required when increasing the mol and voltage variables to avoid potential damage to the titanium alloy in the process.

(Copy and paste)
3.2. Surface morphology and characterization
The effect of voltage on the surface morphology after dealloying was shown in Fig. 4. The samples were undergone 0.8 V, 1.4 V, 2 V, and 3 V voltages for 1 h in the 1 M concentration of electrolyte. It could be observed from the SEM that the degree of the dealloying process gradually increased with the rose of voltage. The plate structure was formatted at 0.8 V, which indicated the spontaneous oxide film was broken down. When the voltage was reached at 1.4 V, the formation of a three-dimensional grid structure arose in the partial

area. It is obvious that the entire surface of the specimen turned to be the homogeneous three-dimensional grid structure at 2 V. After the voltage increasing to 3 V, however, the surface morphology had a height difference.

Fig. 5 exhibited the surface morphology, three-dimensional morphology, roughness values, and contact angle values of samples after electrochemical dealloying. According to Fig. 4, the voltage at 2 V was selected to prepare dealloyed samples in 1 M, 2 M, and 10 M concentrations of electrolyte. The nano-scale three-dimensional grid structure was formed in all concentration of electrolyte. The pore size of dealloyed sample in 1 M electrolyte was about 22.98 ± 1.30 nm (measured from over 40 pores using Image J software), and the pore size of dealloyed samples was 40.63 ± 3.80 nm and 55.71 ± 7.30 nm in 2 M and 10 M electrolyte, respectively. It is worth noting that the pore size gradually increased, the grid structure became continuous and uniform as the electrolyte concentration increased.

To tallk about
Compare all the 1M
Compare all the 5M
Comapre 1M vs 5M

We conclude what is the best combination

Conclusion

In conclusion, the microscopic analysis of the titanium alloy, Ti6Al4V, at different magnifications revealed that lower levels of magnification, such as 150x and 500x, provided limited insights into the actual structure. However, a more detailed examination at 150,000x, coupled with a comparison to the reference, showed significant variations. The increase in concentration and voltage demonstrated a clear correlation with the emergence of a porous structure, indicating the success of our experimental approach. Moving forward, the potential for further exploration, particularly at higher parameters like 10MOL and 5V, holds promise for understanding potential enhancements in porous diameter.

References

[1] L. Wang *et al.*, “Improvement on biosafety and bioactivity of Ti–6Al–4V alloys by construction the three-dimensional grid structure though electrochemical dealloying,” *J. Mater. Res. Technol.*, vol. 17, pp. 546–559, Mar. 2022, doi: 10.1016/j.jmrt.2022.01.002.