Biological Performance of Ti-Ta-O Composite Coatings Prepared by Hydrothermal Method

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Abstract

This study investigates the effect of hydrothermal treatment on the biological performance of Ta-doped Ti-Ta-O composite coatings. The results show that as the concentration of $TaCl_5$ in the hydrothermal reaction increased, significant changes occurred in the surface color and hydrophilicity of the samples, with Ta_3 exhibiting superhydrophilicity and greatly enhancing cell adhesion. Hydrothermal treatment successfully introduced Ta elements onto the titanium-based material surface, and the Ta content increased progressively with the concentration of $TaCl_5$. The treatment led to the formation of micro-nanostructures on the surface, significantly increasing surface area and enhancing osteoblast adhesion, thus promoting bone integration. Cellular experiments demonstrated that the Ta3 sample exhibited optimal performance in terms of cell adhesion, spreading, and activity. In conclusion, the hydrothermally treated Ta-doped coatings offer notable improvements in biocompatibility and osteogenic properties, showing promising potential for future applications.

Keywords

Titanium; Acid Etching; Hydrothermal Method; Tantalum.

1. Introduction

With the aging population and the rising prevalence of orthopedic diseases, bone implant materials have become a focal point in biomedical materials research. Ideal bone implant materials should exhibit good biocompatibility, osteogenic properties, and be non-toxic, non-sensitizing, and non-carcinogenic to the human body [1]. Therefore, selecting appropriate materials and improving their surface properties to enhance bioactivity and osteogenic capacity is crucial for increasing surgical success rates. Tantalum demonstrates excellent biocompatibility, corrosion resistance, and chemical stability, making it widely used in dental implants, artificial joints, and spinal applications [2-3]. Studies have shown that tantalum's surface can support normal cell growth and survival [4-5], establishing it as an ideal material for bone implants. However, the challenges in processing and the high cost of tantalum limit its widespread application [6].

In recent years, with advancements in surface modification technologies, the application potential of tantalum has gradually expanded [7-8]. Research has shown that tantalum can effectively promote bone tissue integration and repair, demonstrating significant bone growth induction effects [9]. Therefore, tantalum holds broad potential as a bone implant material and warrants further investigation.

2. Material and Methods

In this study, commercially pure grade 4 titanium (TA4) was selected as the substrate material and processed into disk samples with a diameter of 13 mm and a thickness of 2 mm. The surface of the samples was sequentially polished using 800#, 1200#, 2000#, and 3000# sandpapers, followed by sandblasting and acid etching (SLA) treatment. After surface treatment, the samples were ultrasonically cleaned in anhydrous ethanol, acetone, and deionized water (for 30 minutes each), and finally dried at 40°C in a vacuum drying oven. The hydrothermal reaction system was prepared using high-purity tantalum pentachloride ($TaCl_5$, 99.9%), 20 wt% hydrogen peroxide (H_2O_2) solution, and citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) as raw materials, with the reaction mechanism shown in equation (1). By optimizing the hydrothermal process parameters, Ta-doped oxide coatings were fabricated on the SLA surface. The surface morphology of the samples was characterized using scanning electron microscopy (SEM, JSM-IT 500 HR), and elemental composition analysis was conducted using energy-dispersive spectroscopy (EDS, JSM-IT 500 HR). The samples were co-cultured with rat bone marrow mesenchymal stem cells (BMSCs), and the effect of tantalum on the surface bioactivity of the titanium-based material was analyzed by observing the cell morphology after 4 hours. The key chemical reagents and process parameters used in the experiments are detailed in Table 1.

$$TaCl_5 + H_2O_2 \longrightarrow [Ta(O2)4]^{3-} + C_6H_8O_7 \longrightarrow [Ta(O2)3(H2Cit)]^{3-} \quad (1)$$

Table 1. Process parameters for the preparation of Ta-doped oxide coatings on titanium-based SLA substrates using the hydrothermal method.

Simple Code	$TaCl_5$ (g/L)	20wt/%H_2O_2 (ml)	C6H8O7 (g/L)	Temperature (°C)	Time (h)
1	10	50	24	90	8
2	15	50	36	90	8
3	20	50	48	90	8

3. Results and Discussion

Figure 1 shows the color changes and hydrophilicity of the samples after hydrothermal treatment. As observed, the samples treated with conventional acid etching appear silver-gray, while the Ta_1 and Ta_2 samples after hydrothermal treatment exhibit a brown-yellow color, and the Ta_3 sample has a brownish-blue surface. The color change suggests that as the concentration of $TaCl_5$ increases, the extent of the hydrothermal reaction changes significantly. Further analysis revealed that, as the concentration of $TaCl_5$ increased, the hydrophilicity of the sample surface also changed accordingly. The hydrophilicity of the Ta_1 and Ta_5 samples is similar to that of the standard SLA surface, while the Ta_3 sample displays superhydrophilicity. This phenomenon may be linked to the presence of citrate ions remaining on the Ta_3 sample surface, which could enhance its hydrophilicity. The increased surface hydrophilicity helps promote cell adhesion, as verified in subsequent cell experiments.

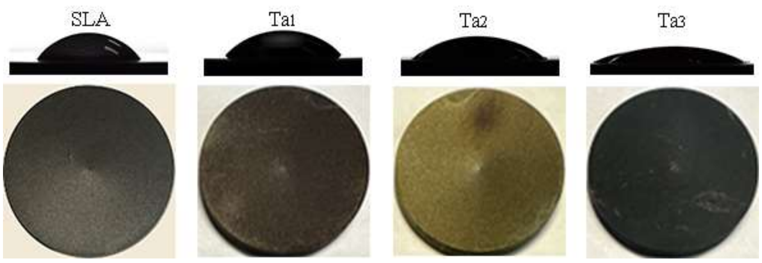

Figure 1. Color changes and hydrophilicity after acid etching and hydrothermal treatment.

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Figure 2 shows the surface morphology and elemental distribution of the samples. The scanning electron microscopy (SEM) images reveal that the acid-etched samples exhibit a rough morphology with micro-nanostructures on the surface, significantly increasing their surface area. This surface structure enhances the attachment of osteoblasts and provides favorable conditions for protein adsorption, promoting osseointegration between the implant surface and the surrounding bone tissue [10]. Additionally, energy dispersive spectroscopy (EDS) analysis indicates that hydrothermal treatment successfully introduced Ta elements onto the sample surface, with the Ta content gradually increasing as the $TaCl_5$ concentration increased. The EDS images also show small particles on the sample surface, which are Ta oxide particles formed during the hydrothermal reaction. It is noteworthy that the hydrothermal treatment did not significantly alter the surface morphology after acid etching.

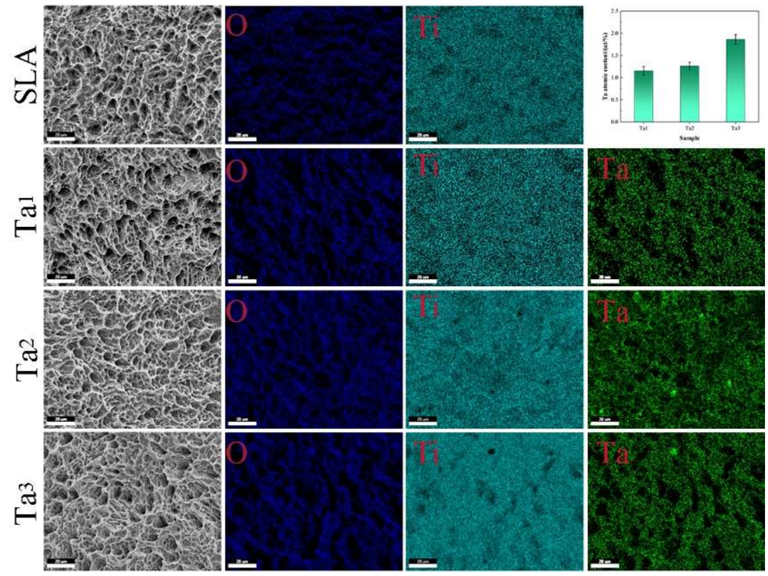

Figure 2. Surface morphology and elemental distribution images of the samples after acid etching and hydrothermal treatment.

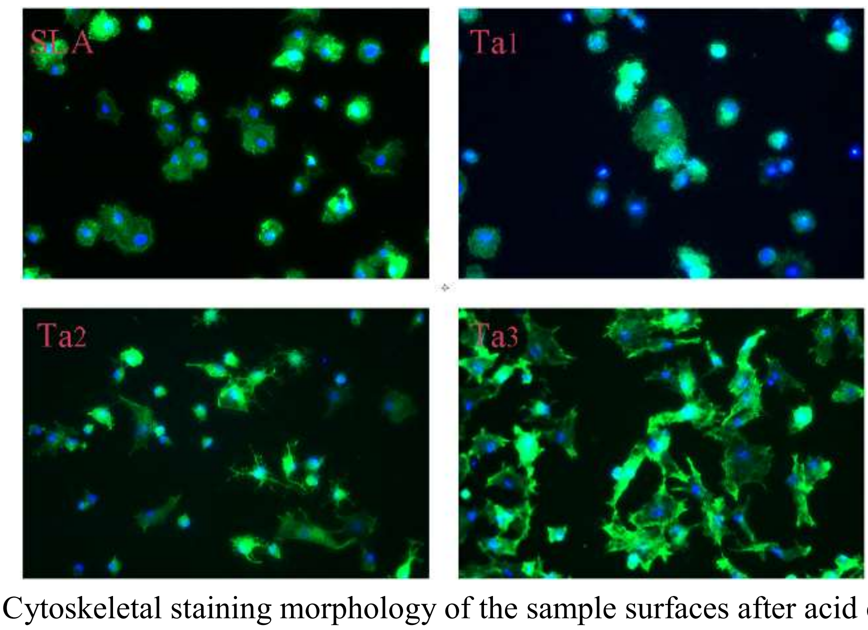

Figure 3. Cytoskeletal staining morphology of the sample surfaces after acid etching and hydrothermal treatment: 4 hours.

Figure 3 shows the cytoskeleton observed using fluorescence staining under a laser confocal microscope. Blue represents the cell nucleus, green represents actin filaments, and the culture time was 4 hours. As shown in the figure, the cells on the SLA and Ta_1 samples did not fully extend and exhibited a clustered morphology. In contrast, the cells on the Ta_2 sample showed good adhesion and some filopodial extension, demonstrating better cell activity. The cells on the Ta_3 sample exhibited superior adhesion and spreading compared to the other samples, with more extensive filopodial extension. Osteoblasts showed higher activity. This phenomenon may be attributed to, on one hand, the higher hydrophilicity of the Ta_3 sample, which favors cell adhesion, and on the other hand, the increased Ta content, which enhances the osteogenic potential of the sample, effectively promoting cell extension.

4. Conclusion

The hydrothermally treated Ta-doped Ti-Ta-O composite coating demonstrated significant improvements in biological performance. As the concentration of $TaCl_5$ increased, the color and surface hydrophilicity of the samples changed notably, particularly the Ta_3 sample, which exhibited superhydrophilicity and enhanced cell adhesion. The hydrothermal treatment successfully introduced Ta elements onto the sample surface, and with increasing $TaCl_5$ concentration, the Ta content gradually increased. The micro-nanostructures formed on the surface significantly enhanced surface area, which benefits osteoblast adhesion and osseointegration. Additionally, Ta doping improved the osteogenic potential of the samples, with the Ta_3 sample showing the best performance in terms of cell adhesion, spreading, and activity. Therefore, the hydrothermally treated Ta-doped coatings exhibit excellent biocompatibility and osteogenic performance, with broad application potential.

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