Gel Oxidation of Titanium at Low Concentration Sodium Hydroxide (NaOH)

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Abstract
Gel oxidation is an effective thermochemical method for the preparation of bioactive titanium surfaces. This study aims to investigate the effect of low NaOH concentration on gel oxidation of titanium subjected to various oxidation temperatures. Titanium foils were soaked in NaOH aqueous solutions with the concentration of 0.5 M or 1.0 M, followed by oxidation in the range of 400°C to 800°C. The crystallinity of the film was determined using glancing angle X-ray diffraction (GAXRD). At low NaOH concentration, the amount of sodium titanate hydrogel, if present, was too low to detect. Subsequent oxidation at 400°C was insufficient to form sufficient anatase and/or rutile to be detectable by GAXRD. Oxidation at 600°C and 800°C resulted in the detection of rutile.

Keywords: Gel oxidation, sodium hydroxide, titanium, surface modification

Introduction

Gel oxidation is a combination of alkali and heat treatment which is introduced by Kim *et al.* to form a bioactive surface layer on a bioinert titanium surface [1]. Gelation process forms sodium titanate hydrogel on the titanium surface by immersing titanium sample into sodium hydroxide, NaOH aqueous solution. Oxidation is a process whereby the titanium sample with sodium titanate hydrogel on its surface is heat-treated at various temperatures, dehydrating and densifying the gel to form amorphous sodium titanate (<600°C) or a mixture of crystalline sodium titanate and TiO_2 (≥600°C) [2]. Different NaOH concentration and heat treatment temperature can control the physical and chemical properties of the surface layer [3, 4].

Experimental procedure

High purity titanium samples (25 mm x 10 mm x 0.05 mm) were wet hand-polished using 1200 grit (~1 μm) abrasive paper and subsequently cleaned with acetone and distilled water. Gelation was performed by immersing the titanium samples in ~5 mL of 0.5 M/1.0 M NaOH solution at 60°C for 24 hours. The titanium samples were placed on alumina refractory, subjected to oxidation at 400°C, 600°C and 800°C (heating rate 300°C/h) in air in an electrical furnace. Glancing angle X-ray diffraction (GAXRD) was used to determine the crystallinity of the films. The microstructures were examined using field emission scanning electron microscope (FESEM). Focused ion beam (FIB, FEI x P200) milling was used for cross-sectional imaging.

Results and discussion

Fig. 1 shows the GAXRD patterns of surfaces of Ti treated in 0.5 M NaOH after being subjected to oxidation at various temperatures.

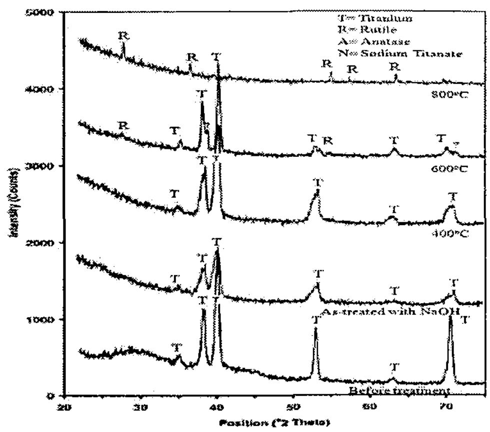

Fig.1: GAXRD patterns of the surfaces of Ti treated in 0.5 M NaOH after being subjected to oxidation at various temperatures.

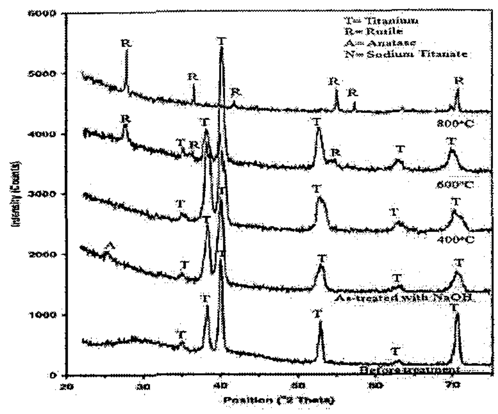

Fig. 2: GAXRD patterns of the surfaces of Ti treated in 1.0 M NaOH after being subjected to oxidation at various temperatures.

i) 0.5 M NaOH treatment

There was no peak observed for sodium titanate hydrogel on the surface of the titanium samples.

ii) Oxidation at 400°C, 600°C and 800°C

Subsequent oxidation at 400°C resulted in no peak of anatase and/or rutile. Peaks of rutile started to appear at ~27° (2θ) and ~53° (2θ) after oxidation at 600°C. More peaks of rutile can be observed after oxidation at 800°C with titanium peaks diminishing.

Fig. 2 shows the GAXRD pattern of surfaces of Ti treated in 1.0 M NaOH after being subjected to oxidation at various temperatures.

i) 1.0 M NaOH treatment

There was no peak observed for sodium titanate hydrogel on the surface of the titanium samples. Instead, a low intensity of anatase peak can be observed.

ii) Oxidation at 400°C, 600°C and 800°C

Subsequent oxidation at 400°C resulted in no peak of anatase and/or rutile. Peaks of rutile started to appear at ~27° (2θ), ~35° (2θ) and ~53° (2θ) after oxidation at 600°C. Higher intensity and number of peaks of rutile, compared to those shown in Fig. 1, can be observed after oxidation at 800°C with titanium peaks diminishing.

Conclusion

The films produced in 0.5 M and 1.0 M NaOH were too thin to identify if the sodium titanate hydrogel was present. The GAXRD patterns showed that rutile was present on the surface only after oxidation at 600°C and 800°C. Oxidation at 400°C was insufficient to form sufficient anatase and/or rutile to be detected by GAXRD.

References

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