Investigating Formations on Titanium Surfaces for Enhanced Fluid-Surface Interactions

LIBERTY UNIVERSITY

Noah Kohl[a], Sebastian Bernaschina[b], Dr. Michael Korn[b], Dr. Hector Medina[a]*

[a]School of Engineering and [b]Department of Biology & Chemistry

Abstract

The research project is aimed at investigating crystalized formations observed on titanium (Ti) surfaces via Scanning Electron Microscopy (SEM), which were etched with sulfuric acid (H2SO4) at temperatures between 40 to 90 degrees Celsius. According to the literature, similar formations obtained with different materials or processes have found important applications such as water purification, hydrogen storage, photocatalysis, and others. In general, these formations are desirable in fluid-surface interactions where surface composition and enhanced surface roughness can play a key role to promote such reactions. Various processes will be utilized to pursue the creation of both the observed and other similar formations. For example, Ti surfaces will be exposed to oxygen-rich and hydrogen-rich environments under temperature-controlled environments. In this study, we propose to investigate the factors that influence the nucleation and growth of this novel crystallized formations and their potential uses in aforementioned industrial chemical processes. Full understanding of both etching off (ground-down) as well as building up (ground-up) processes could lead to further understanding of the engineering science behind these formations. This knowledge could lead to applications such as: improved bone-implant devices; microbead-based local therapy for bone-deficient medical conditions; advance knowledge in corrosion science; and development of various improved devices involving enhance fluid-surface chemical reactions.

Objective

Primarily, the project seeks to reproduce rosette-like structures formed when Ti is reacted with H_2SO_4. The structures are believed to be small formations of titanyl sulfate $TiOSO_4$. The surfaces of the rosette-like structures will be investigated to be reacted with H_2O_2 to form hydrogen disulfatoperoxotitanate(IV) **(Figure 1)**. Additionally, the titanyl sulfate could be hydrolysis to form TiO_2 **(Figure 1)**. The challenge of the project lies in determining if the rosettes can be reproduced and if they will maintain their structural integrity once chemically modified.

Methods

Specimen Preparation

- Plates will be cut from 0.007" thin sheets and cleansed with chloroform.
- Plates will be submerged in different concentrations of sulfuric acid at 60°C or 90°C for 30 or more minutes.
- Plates will be rinsed in de-ionized (DI) water and then air dried.

Specimen Analysis

- Specimen mass will be recorded before and after etching for comparison.
- Scanning electron microscopy analysis will be used to visualize the specimens after treatment.

Chemical Reaction

$$TiOSO_4 + H_2O_2 \longrightarrow H_2[Ti(O_2)(SO_4)_2] + H_2O$$

$$TiOSO_4 + H_2O \longrightarrow TiO_2 + H_2SO_4$$

Concepts

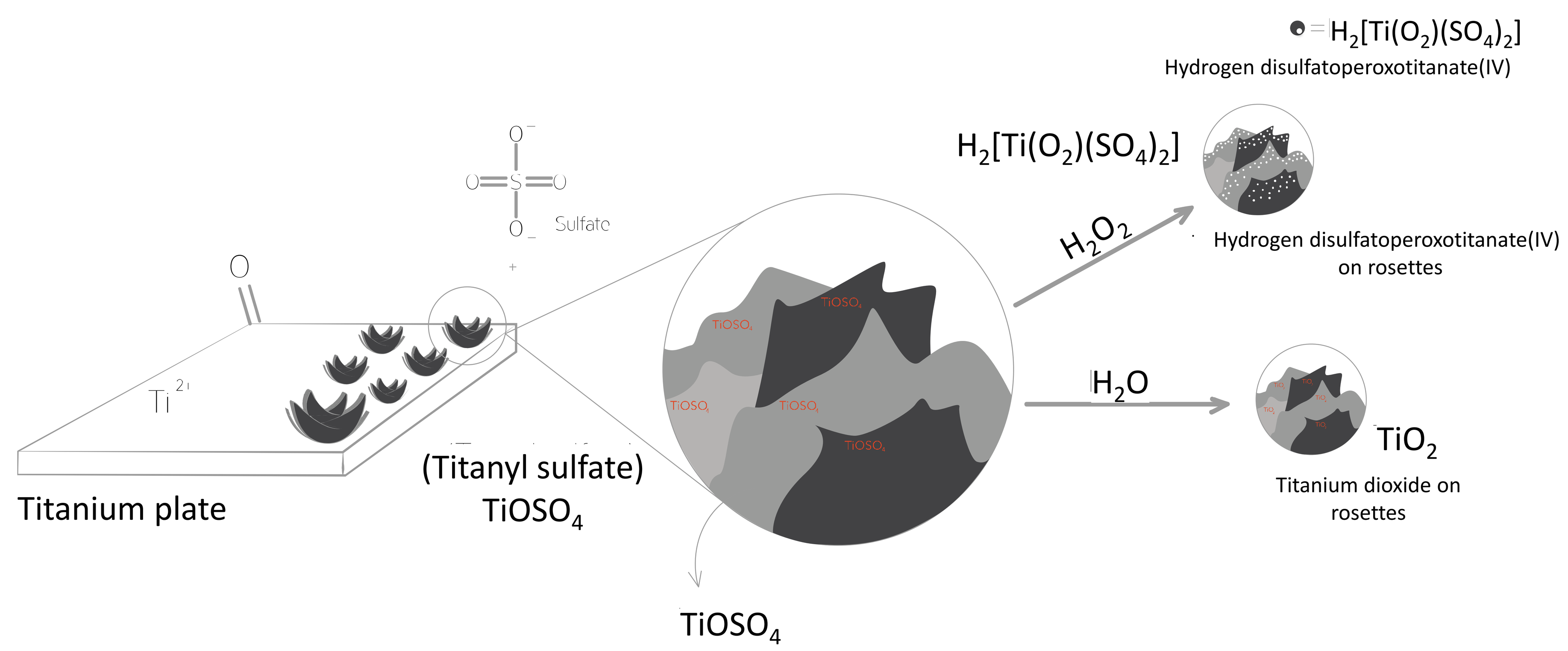

Figure 1. Illustration of the titanyl sulfate plate with rosette-like structures. The edges of the structures are reacted with H_2O_2 producing $H_2[Ti(O_2)(SO_4)_2]$ and treated to form TiO_2. (Villalba, 2019)

Initial Discovery

On-going research identified the presence of rosette-like structures on the surfaces of titanium plates. However, a systematic procedure has not yet been established. Titanium plates have been treated with a variety of concentrations of sulfuric acid in different temperatures and "resting" periods. Plenty of research [4][5] and SEM has shown that the surfaces of the titanium plates become etched when treated with concentrated sulfuric acid **(Figure 2)**. Energy-dispersive x-ray spectroscopy (EDS) analysis indicated that the composition of the rosettes is based on Ti, O_2, and S atoms; whereas EDS of regularly etched Ti reveal no S atoms.

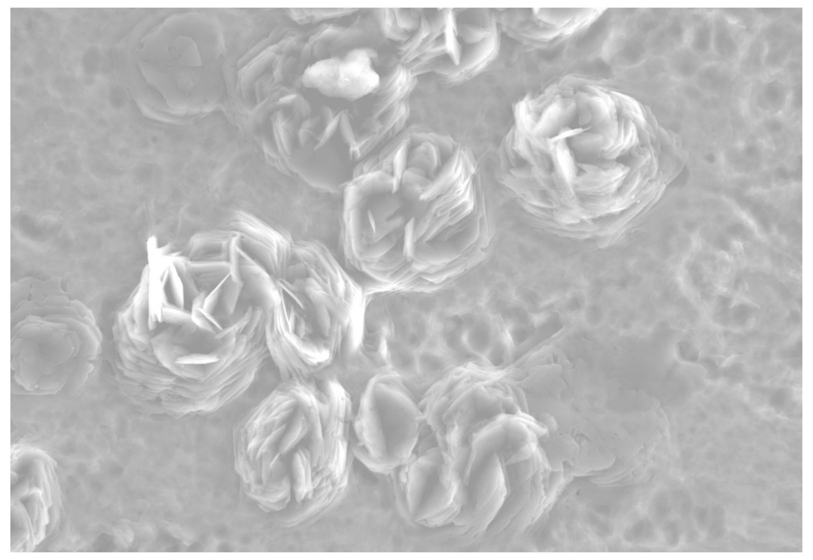

Figure 2. EDS image of the surface of titanium plate after being treated with sulfuric acid. [3]

The rosettes were about 15-30 μm in diameter and were discovered near specimen edges. These crystals are composed of many thin sheets resembling 'petals' that form in a roughly hexagonal arrangement, which resemble the rosette-like formation. These rosette structures exhibit a large surface area on titanium plates, which can be explored in a variation of surface-based interactions **(Figure 3)**.

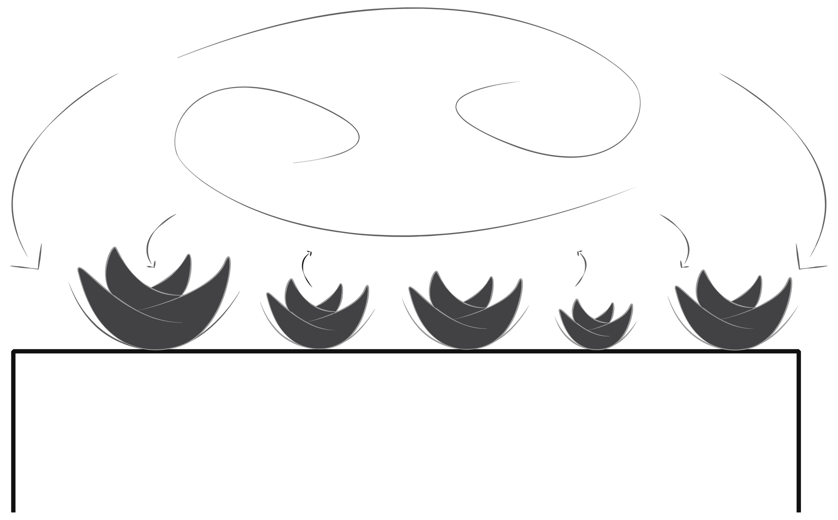

Figure 3. Interaction of the rosette-like structures with fluids. (Villalba, 2019)

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Research Plan

- Part 1 of our research plan consists of recreating the rosettes and changing variables to recreate rosettes under different conditions on the titanium plates **(Figure 4)**:
  - Acid concentrations: 4M, 9M and 18M.
  - Temperature: 60°C and 90°C.
  - Reaction time: 30 min. to 3 hours.
  - After heating, some samples will be analyzed, and some will remain in their tubes for longer time periods at room temperature.

Our control group will be commercial $TiSO_4$ reacted with H_2O_2, producing TiO_2; analysis by SEM and EDS.

Part 2, once the rosettes form, we will test how well the rosette structures maintain their geometry with and without exposure to chemicals. If they maintain their structure, we will chemically modify the edges of these rosettes and determine if the edges are hydrogen disulfatoperoxotitanate(IV) or TiO_2.

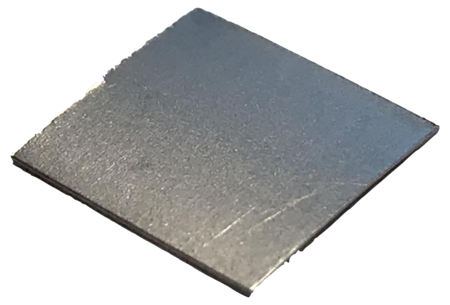

Figure 4. Commercially pure titanium plates. (Kohl, 2019)

Potential Applications and Broader Impacts

Potential applications for the rosettes will be investigated in the creation of a titanium-based device containing pre-made titanyl sulfate rosettes on its surface to use in detecting H_2O_2, forming a yellow compound and in providing micrometer size TiO_2 clusters on titanium, useful for subsequent Ti-based catalysis.

The goal of the current research project is to advance knowledge that can conduce to creating new technology aimed at helping society. For example, the creation of advanced titanium surfaces that can enhance biological interactions will lead to the development of improved bone implants. Simultaneously, the knowledge will lead to the development local therapy aimed at helping patient with bone tissue deficiencies. Furthermore, the enhanced surfaces can also be used in other applications such as water purification (i.e., the removal of ionic contaminants via surface-water interactions), the storage of hydrogen, and other processes. All these applications will lead to the development of advanced devices aimed at benefiting our society.

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