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The Study of Potential Oxidation of Stainless Steel and Titanium alloys from Copper ions

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The Study of Potential Oxidation of Stainless Steel and Titanium alloys from Copper ions.

Renae Wollman
Sherri Adams, MS
Michaela Schock
Austin T. Morgen
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>PURPOSE OF EXPERIMENT</td>
<td>3</td>
</tr>
<tr>
<td>EXPERIMENTAL MATERIALS</td>
<td>4</td>
</tr>
<tr>
<td>PROCEDURE</td>
<td>5</td>
</tr>
<tr>
<td>Diagram</td>
<td>6</td>
</tr>
<tr>
<td>Images</td>
<td>7</td>
</tr>
<tr>
<td>COMPOSITION OF METALS</td>
<td>8</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>8</td>
</tr>
<tr>
<td>Titanium</td>
<td>8-9</td>
</tr>
<tr>
<td>$E_{cell}$ VALUES</td>
<td>10</td>
</tr>
<tr>
<td>Calculations</td>
<td>11</td>
</tr>
<tr>
<td>Possible Precipitates</td>
<td>12</td>
</tr>
<tr>
<td>DATA</td>
<td>13</td>
</tr>
<tr>
<td>Iron – Mass/Voltage/Graph</td>
<td>13-14</td>
</tr>
<tr>
<td>Stainless Steel – Mass/Voltage/Graph</td>
<td>15-16</td>
</tr>
<tr>
<td>Titanium – Mass/Voltage/Graph</td>
<td>17-21</td>
</tr>
<tr>
<td>ERRORS</td>
<td>22</td>
</tr>
<tr>
<td>CONCLUSION</td>
<td>23</td>
</tr>
<tr>
<td>WORKS CITED</td>
<td>24</td>
</tr>
</tbody>
</table>
Oxidization of Bio-metals

Purpose of Experiment

- To investigate the potential oxidation of surgical grade metals (SS and Ti) in the presence of Copper ions.

The human body utilizes various ions, including Copper, Zinc and Iron which are usually found attached to proteins, and free ions like Sodium, Potassium, Manganese, and Calcium. These ions are beneficial to the human body at trace amounts, but an increase in metal ions in the body have been recorded to have negative effects. An increase in Copper ions has been linked to Parkinson’s disease, Menkes disease, Wilson’s disease, Alzheimer’s disease and even cancer. Elevated Iron levels can be used by invading pathogens and also influence neurodegeneration (Wedd, A., & Maret, W., 2014). This illustrates the importance of researching the potential REDOX reactions between metal implants and bodily ions. Titanium alloys are widely used in medical applications due to a good mechanical strength and adequate corrosion resistance. However, Titanium tends to be a more expensive bio-metal. The application of Stainless steel alloys is substituted to decrease financial cost. These alloys maintain corrosion resistance by employing protective oxide surface layers (T.Hanawa, 2004). The purpose of this project is to evaluate REDOX reactions between ions in human bodily fluids and metals used in surgical implants. The effect of pH and temperature in Simulated Body Fluids (SBF) on the REDOX reaction rates will also be evaluated. The protective coating of oxide films, type of alloys, biocorrosion and wear behavior of bio-metals has to be taken into consideration (Matusiewicz, Henryk, 2014). The results of this research could benefit further research into biocompatibility of implants in the human body.
Experimental Materials

- Electrochemical cells
  - 565ml in 600ml beaker/300ml in 300ml beaker
  - 50ml in big porous cup (38mm X 75mm)
  - 35ml in small porous cup (32mm X 73mm)
  - 3-Finger clamps
  - Banana Clips
  - Buret Clamps
  - Iron Stand
  - 1M solutions except SBF (OA/RA)
    - NaCl//Cu(SO₄)
    - Na(NO₃)//Cu(NO₃)
    - SBF*/Cu(SO₄)
- Electrodes
  - Copper Electrodes (Flinn Scientific) 6” x 0.5”
  - Iron Electrodes (Flinn Scientific) 6” x ½” x 0.02”
  - Stainless Steel (McMaster) approx. 2” x ¾” x 1/8”
    - #1 316/316L
  - Titanium (McMaster) 6” x ¾” x 1/8”
    - Highly Corrosion Resistant Grade 2 Titanium
    - High-Strength Grade 5 Titanium
- Vernier LabQuest 2 Software and Technology
- Vernier Voltage Probe
- Denver Instrument™ Analytical Digital Scale
- Genesys™ 6 Spectrophotometer
- Battery for Titanium*

*SBF was mixed based on How to Prepare the Simulated Body Fluids (SBF) and its related solution, proposed by Kokubo and his colleagues. – Chikara Ohtsuki, Ph.D. One exception: Used autoclave to sterilize equipment vs. liquid sanitizer.

*In Mckayla’s readings a ±30V Voltage Probe was used. Due to the program accounting(x3) for a much larger voltage than our reading, the “noise” made it difficult to verify optimal results. She never measured readings for a positive voltage. Towards the end of her research only mass was studied.

*In Consideration of our negative voltage for the Titanium, resting conditions of all cells have an inside negativity of 60 to 90 mV. This arises from a tug of war between potassium ions in the body. The resting potential acts like a tiny battery, stored energy is used to power electrical impulses of our nerve and muscle fibers. (Ashcroft, Frances. The Spark of Life, 2012)
Procedure

The effect of Copper ions on surgical grade metals was assessed. The electrochemical half-cell solutions were mixed using standard lab procedures. Hard Strength, Resistant Titanium, and 316L Stainless Steel were evaluated using an Electrochemical cell. Each electrode was scrubbed using 3M 336U (60) Sandpaper, polished with Steel Wool, rinsed clean with deionized water, then acetone, and allowed to air dry. The sandpaper caused slight scratches in the metal electrodes, this was intentional to mimic natural wear and tear. The 316L electrode had jagged and rough finish from using a wet saw, curtesy of the Gillette Tech welding shop, to cut a bigger block into the desired sized electrodes.*The electrodes were wiped dry with Kim wipes. Each metal electrode mass was collected using the Denver Instrumental Analytical scale and recorded in the Student Lab Notebook. Gloves were always worn when measuring mass to prevent any mass differential from fingerprints.

The electrochemical cell consisted of a 600 ml beaker and a porous cup acting as a salt bridge. The porous cup was positioned in the middle of the beaker using a 3-finger clamp attached to an Iron Stand. The beaker was filled with 565ml of NaCl or SBF solution. Next, the porous up was filled with 40ml of CuSO₄. For smaller 300ml beaker, a solution volume of 300ml was used. For smaller porous cup, a solution volume of 35ml was used. The SBF solution electrochemical cells were set-up similar to the NaCl solutions, however a constant temperature of 36.5°C was maintained by housing the electrochemical cell in a water bath. The Iron, Stainless Steel or Titanium electrodes were placed into the solution in the beaker and the Copper electrode was placed into the porous cup. The electrodes were clipped into position using banana clips that were kept in place by a buret clamp. It was good practice to make sure the electrodes would clip in the correct position before adding any solutions. The voltage output was measured using a basic ±10V Vernier Voltage Probe*. The red clip (cathode) was always clipped to the copper electrode and the black clip (anode) was clipped to the Fe, SS, or Ti. The data for the Voltage output was recorded and saved using Vernier LabQuest2 Software. Each Stainless steel experiment was allowed to run for a max of 24 hours. The Titanium was allowed to run for 78 - 96 hours, this proved to be a sufficient amount of time to observe precipitants. After each run, the electrochemical cells were evaluated for any precipitates and photos were taken of each cell. The electrodes were carefully rinsed into a beaker and any precipitates were saved. The electrodes were allowed to dry in a descanter for a period of 24hours. The mass of each electrode was measured and compared to re-experiment masses. The voltage readings and mass measurements were recorded in the student lab Manuel.

A generalization of the voltage readings: A positive increase in potential indicates that an oxide layer is forming. A constant steady potential indicates that the protective surface oxide layer is intact and a negative voltage indicates that a break has occurred in the surface film (I.Gurrappa, 2003). For our data, if the Bio-metal electrodes were treated as cathodes (Clip red to Ti/SS electrode) and clip the black to the copper (anode), or reverse our negative and positive signs, ie. “flip” the graphs, our data could be interpreted with these generalizations.

*The smoother the surface of 316L the greater the corrosion resistance. (McMaster)
Diagrams

- **NaCl Solution**
  - Fe is anode
  - Cu is cathode
  - Voltage: 0.730V

- **SBF Solution**
  - Ti/SS is anode
  - Cu is cathode
  - Voltage: 1.60V
  - CuSO4 Solution
  - Ions
Image 1: Electrochemical set-up with porous cup. Vernier Voltage probe is clipped to electrodes and connected to Vernier Lab Quest Pro2 device.

Image 2: Same electrochemical cell set-up as Image 1, except the cell is housed in a hot water bath and glass thermometers are used to measure temperature.
## COMPOSITION OF METALS

### STAINLESS STEEL – 316/316L

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.036</td>
</tr>
<tr>
<td>Manganese</td>
<td>2.00</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.045</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.030</td>
</tr>
<tr>
<td>Silicon</td>
<td>1.00</td>
</tr>
<tr>
<td>Chromium</td>
<td>16.00 - 18.00</td>
</tr>
<tr>
<td>Nickel</td>
<td>10.00 – 14.00</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>2.00 – 3.00</td>
</tr>
</tbody>
</table>

*based on A276-06(ASTM.org)*
The surface oxide film on Austenitic Stainless Steel consists of iron, chromium and a small amount of molybdenum. The surface film when polished mechanically in de-ionized water is high in OH- and Iron. Nickel, molybdenum and manganese are just under the surface oxide film. The film is approximately 3.6nm. Austenitic SS does not contain nickel in air or chloride solutions. (T.Hanawa, 2004)

TITANIUM - Ti6Al4V

Table 2. The composition of High Strength Ti6Al4V Grade 5. (azom.com)

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>&lt;0.08</td>
</tr>
<tr>
<td>Iron</td>
<td>&lt;0.25</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Oxygen</td>
<td>&lt;0.2</td>
</tr>
<tr>
<td>Aluminum</td>
<td>5.5 – 6.76</td>
</tr>
<tr>
<td>Vanadium</td>
<td>3.5 – 4.5</td>
</tr>
<tr>
<td>Hydrogen(bar)</td>
<td>&lt;0.0125</td>
</tr>
<tr>
<td>Titanium</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The surface oxide film of Ti6Al4V alloy is similar to Ti, with Ti+4, Ti+3 and Ti+2 oxides, but differs slightly in having small amounts of aluminum oxides. The other most layer consists mostly of TiO₂ and is highly resistant against chloride ions (T.Hanawa, 2004).

Table 2. The composition of Grade 2 unalloyed Highly Corrosion Resistant Titanium. (asm.matweb.com)

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>&lt;0.10</td>
</tr>
<tr>
<td>Iron</td>
<td>&lt;0.30</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>&lt;0.03</td>
</tr>
<tr>
<td>Oxygen</td>
<td>&lt;0.25</td>
</tr>
<tr>
<td>Hydrogen(bar)</td>
<td>&lt;0.015</td>
</tr>
<tr>
<td>Titanium</td>
<td>Balance</td>
</tr>
<tr>
<td>Residuals</td>
<td>&lt;0.10 each, 0.40 max</td>
</tr>
</tbody>
</table>

The Grade 2 nonalloy Titanium has a surface film with Ti+4, Ti+3 and Ti+2 oxides.
Half cell Equations were pulled from Chemistry and Chemical Equations, 7th Edition, Kotz, Treichel, Townsend and chemeddl.org.
\( E_{\text{cell}} \) Calculations

\[ E_{\text{cell}} = E_{\text{cathode}} - E_{\text{anode}} \]

**Standard Fe/Fe²⁺/Cu²⁺/Cu**

Reduction

\[ \text{Cu}^{+2} (\text{aq}) // \text{Cu}(s) \quad E^0 = +0.337 \text{ V} \]

Oxidation

\[ \text{Fe}(s) //\text{Fe}^{+2} (\text{aq}) \quad E^0 = -0.440 \text{ V} \]

\[ E_{\text{cell}} = 0.342 - (-0.447) = 0.777 \text{V} \]

**Possible Stainless Steel \( E_{\text{cell}} \) Calculations**

\[ E_{\text{cell}} = (\text{Fe}(s) + 20\text{H} \leftrightarrow \text{Fe(OH)}_2(s) + 2\text{e}) + (\text{Cu}(s) \leftrightarrow \text{Cu}^{2+} + 2\text{e}) = \]

\[ E_{\text{cell}} = (0.877 \text{V}) + (-0.337 \text{V}) = 0.54 \text{V} \]

\[ E_{\text{cell}} = (\text{Cr}(s) + 3 \text{OH}^-(\text{aq}) \leftrightarrow \text{Cr(OH)}_3(s) + 3\text{e}) - (\text{Cu}(s) \leftrightarrow \text{Cu}^{2+} + 2\text{e}) = \]

\[ E_{\text{cell}} = 2(1.3 \text{V}) + 3(-0.337 \text{V}) = 1.589 \text{V} \]

Any Iron Oxides, including Chromium oxides and possibly Nickel and Molybdenum.

**Possible Titanium \( E_{\text{cell}} \) Calculations**

\[ E_{\text{cell}} = (\text{Ti} + 2\text{H}^2\text{O} \leftrightarrow \text{TiO}^2+ + 4\text{H} + 4\text{e}) - (\text{Cu}(s) \leftrightarrow \text{Cu}^{2+} + 2\text{e}) = \]

\[ E_{\text{cell}} = (0.860 \text{V}) + 2(-0.337 \text{V}) = 0.186 \text{V} \]

\[ E_{\text{cell}} = (\text{Ti}(s) + \text{H}2\text{O} \rightleftharpoons \text{TiO}(s) + 2\text{H} + 2\text{e}) - (\text{Cu}(s) \leftrightarrow \text{Cu}^{2+} + 2\text{e}) = \]

\[ E_{\text{cell}} = (1.31 \text{V}) + (-0.337 \text{V}) = 0.973 \text{V} \]

Any Titanium Oxides, focusing more on Ti+2, including Vanadium and Aluminum oxides.
**Possible Precipitates for Iron/Copper**

FeCl₃ is a yellow solid

FeCl₂ is light green to colorless

Fe(SO₄) is light green

Fe(OH)₃ is reddish brown

Fe(0) is black

Cu(O) is black

Cu₂(0) forms a brick red crystalline powder.

Iron(3) solutions appear yellow or brown-yellow, It turns blood red when bonding with thiocyanate.

**Possible Precipitates for Titanium/316L/Copper**

NiSO₄ is a yellow solid, blue crystal

NiCl₂ is a yellow solid, green hydrate

MnCl₂ is pink

Mn(SO₄)₃ is a white solid, pale pink when hydrated

Cr₂O is black

Cr₃O₂ is light to dark green

Cr0₂ is red solid, bright orange when hydrated
Experimental Data

Iron

- Fe/Fe\(^{2+}\)/Cu\(^{2+}\)/Cu (NaCl/CuSO\(_{4}\))
  - Max Voltage readings of 0.780V
  - Mass
    - Fe: 9.4543g \(\rightarrow\) 7.7401g
    - Cu: 4.2715g \(\rightarrow\) 6.0325g

Graph 1: Fe/Cu Voltage in NaCl
• Fe/Fe²+/Cu²+/Cu (SBF/CuSO₄)
  - Max Voltage readings of -0.780
  - Mass
    - Fe:
    - Cu:

Graph #2: Fe//Cu Voltage in SBF

Optimal results with Positive Voltage of 0.780V. Comparable to calculated voltage of 0.777V. This electrochemical cell was set-up as a reference and to make sure our Vernier probes worked properly.

The Precipitates in the Fe/NaCl//Cu/CuSO₄ cell consisted of Fe₂Cl and Fe₃Cl. Obviously there was some “Rust” in the solution as Iron undergoes oxidation as soon as it is exposed to Water, and the process is expedited in the presence of NaCl. It should be acknowledged that copper undergoes a similar reaction leading to Cu₂Cl, more commonly known as patina. The Presence of Fe₂Cl was verified using spectrophotometry and comparing a known Fe₂Cl sample to an unknown sample. The presence of Fe₃Cl was verified by mixing solid potassium thiocyanate into the unknown of the electrochemical cell solution which led to a blood red appearance, which results from Fe₃ bonding with thiocyanate. The reasoning for testing for different Iron ions was the possibility that they would appear in the Stainless steel electrochemical cell solution.
Stainless Steel

- 316L/Cu (NaCl/CuSO₄)
  - Max Voltage Readings* = -0.25
  - Mass:
    - #1SS: 15.7935g --> 15.7933g Loss of 0.0002g
    - Cu: 3.4072g --> 3.3813g Loss of 0.0259g

Table #3: SS//Cu in NaCl

The data for the 316L showed erratic readings indicating that the protective oxide layer that forms on the Stainless steel undergoes constant interruption. Longer runs would be necessary to determine how long the 316L fights corrosion. The precipitates for this electrochemical cell was determined to be Cu²Cl through spectrophotometry.
• #1SS/Cu (SBF/CuSO₄)
  ▪ Max Voltage Readings \(-0.199\)V
  ▪ Mass
    ▪ #1SS: 15.7880g \(\rightarrow\) 15.7880g exact mass
    ▪ Cu: 4.2872g \(\rightarrow\) 4.2814g loss of .0058g

Table #4: SS//Cu in SBF

Negative Voltage indicates high corrosion resistance. Similar to Titanium. No loss in mass of SS. However, a loss in mass of Copper, this is expected with a negative voltage. The jumps in voltage also indicate interruptions in the protective biofilm forms on 316L, the readings indicate a greater risk in corrosion, especially around 20 hours. Precipitates: light blue/green, there was also a definite blue color to the NaCl cell solution. The light blue precipitates are most likely Cu₂Cl, this was confirmed by running a spectrophotometry test comparing the unknown sample to a known Cu₂Cl sample. Given that the copper electrode was oxidized, it would increase the copper ions in the solution giving it a blue color and causing a formation of Cu₂Cl.
Titanium

- **Resistant Titanium/Cu**
  - (NaCl/CuSO₄) 72hr
    - Max Voltage Readings \(-0.32V \text{ After 72 hrs.}\)
    - Mass
      - R Ti: \(45.4633g \rightarrow 45.4629g\) __ Loss of .00101g
      - Cu: \(4.3881g \rightarrow 4.3033g\) __ Loss of .0848g
  - (NaCl/CuSO₄) 96hr
    - Max Voltage Readings \(-0.403V \rightarrow -0.320V \rightarrow -0.403V \text{ After 96 hrs.}\)
    - Mass
      - R Ti: \(46.0241g \rightarrow 46.0236g\) __ Loss of .0005g
      - Cu: \(4.3025g \rightarrow 4.1958g\) __ Loss of 0.1067g

*Table #5: R-Ti/Cu in NaCl*

These results are as expected when one considers the high resistance of titanium due to protective Ti oxide films that Ti forms spontaneously when in contact with air or water. It is notable that a spike in voltage around the 28 hour indicates a break in the TiO surface film. Precipitates were light blue/green, there was also a definite blue color to the NaCl cell solution. The light blue precipitates are most likely Cu₂Cl, this was confirmed by running a spectrophotometry test comparing the unknown sample to a known Cu₂Cl sample. Given that the copper electrode was oxidized, it would increase the copper ions in the solution giving it a blue color and causing a formation of Cu₂Cl.
• R-Ti/Cu (SBF/CuSO₄)
  ○ Max Voltage Readings -0.140V
  ○ Mass
    - R-Ti: 43.6996g → 43.6991g
    - Cu: 4.3270g → 4.3537g

Table #6: R-Ti/Cu in SBF

The data reads as a decreasing voltage until the 20 hour, (lost data during a battery outage) were it finally appears to become constant. An experiment running for a longer period would be optimal. (still reviewing precipitates)
• **Hard Strength Titanium/Cu**
  
  - (NaCl/CuSO₄) 96 hr.
    - Max Voltage Readings **0.381V**
    - Mass
      - H-S Ti: 29.8423g → 29.8465g Gain of .0042g
      - Cu: 4.3738g → 4.2786g Loss of .0952g

  
  **Graph #7: H-S Ti/Cu Voltage in NaCl**

The results from the hard strength Ti (NaCl) were surprisingly similar to the Resistant Ti (NaCl) in voltage readings. The graphs are very similar except the increase in voltage around the 25th hour is less noticeable than that of the Resistant Ti at the 28th hour. This would suggest that the Resistant Ti held a greater resistance to a break in the surface oxide film. As a reminder: A break in constant voltage indicates a break in the surface oxide film.
- HS-Ti/Cu (SBF/CuSO₄)
  - Max Voltage Readings: -0.255V
  - Mass
    - HS-Ti: 29.8201g → 29.8199g
    - Cu: 4.3654g → 4.3246g

Table #8: H-S Ti/Cu in SBF

Again, the graphs of the Hard Strength (SBF) is similar to the graph of the Resistant Ti(SBF). The Resistant Ti graph has a constant decrease, where the Hard Strength Ti appears to be slightly more erratic, indicating greater interruptions in the surface oxide film.
Battery

- Resistant Titanium 0.5 Voltage (NaCl)
  - Max Voltage Readings __Input of 0.5V w/ battery__
  - Mass
    - R Ti: 46.0276g → 46.0260g
    - Cu: 4.2968g → 4.2314g

- Hard Strength Titanium 0.5 Voltage
  - Max Voltage Readings __________
  - Mass
    - H-S Ti: __________
    - Cu: __________

- 316L/Cu (NaCl/CuSO₄) 0.5V
  - Max Voltage Readings* __Input of 0.5V w/battery__
  - Mass:
    - #1SS: 23.1610g → 23.1331g Loss of .0279 g
    - Cu: 4.2192g → 4.2471g Gain of .0279 g

The results of this run were highly optimal since the loss of the SS electrode was equivalent to the gain of the Cu electrode.
Errors

- Salt Bridge vs. Porous cup
- Order delay and wrong items delivered
- Human error
  - Mixing Solutions
  - Cutting the 316L
- Mechanical error
  - Difficulty maintain constant temperature
- Not certified Metal electrodes
- Accurate simulation with CuSo4?
- Longer runs would yield better results
Conclusion/Results:

The Data shows that the corrosion resistance of the surgical metals is ranked as follows: 316L < Hard Strength Titanium < Resistant Titanium. According McMaster’s description of 316L and Ti, these rankings were to be expected. The initial drive of this research was to verify if these metals spontaneously undergo redox reactions in the presence of copper. The compositions of these metals evaluated along with the half-cell potentials, shows that, for example, Vanadium or Aluminum found in Titanium should be oxidized by copper. However, empirical data shows that both 316L, Ti and alloyed Ti undergo a spontaneous reaction with surrounding OH- to form a passive protective film around the metals. This is exactly what our data indicated; if copper did indeed oxidize Ti, or 316L, there would have been a positive voltage. Unfortunately, given this reasoning, all the experiments were set-up in such a way that the data read negative voltages. (Negative voltage only indicates that the reaction was going in the opposite direction). It was determined that 316L, Ti and the alloyed Ti do not undergo spontaneous redox, at least not under the environment studied in this experiment. Due to this conclusion, an alternative concept was adopted: Resting Potentials in the Human Body. The cells in our bodies have a resting potential across the membrane of 0.06V to 0.09V due to the tug and pull of potassium ions in our bodies. Could this voltage have an effect on metal implants? If a 0.05V is applied to the electrochemical cell of a SS electrode and a copper electrode (see pg. 21) the SS electrode does have a loss in mass, indicating corrosion.

The 316L electrode is oxidizing the copper electrode giving a negative voltage reading. The negative voltage reading is best explained by “flipping” the graph, (see pg.5, paragraph 3) the protective oxide layer is forming, this is known from empirical data (I.Gurrappa, 2003). The Fe and OH- oxide layer that forms on the surface of the 316L electrode causes a “positive” voltage (see pg5, paragraph 3).

The Titanium appears to be oxidizing the copper electrode, this means the electrochemical cell is going in the opposite direction of the typical Fe//Cu cell and the voltage reading is negative. When considering the composition of the titanium studied (Ti6Al4V) and the E-cell potentials of Vanadium, Aluminum and Titanium, the E-cell numbers and that copper is more likely to oxidize V, Al and Ti. These is the opposite of what the data showed. The best possible explanation is the formation of the TiO₂ layer, along with other oxides that form on the Titanium electrode when it is exposed to air or water. The Ti can also be treated to have a thicker layer of this oxide offering a greater corrosion resistance (Anan’in, N.S., & Polyanskii, V. M., 1998). An attempt to remove the oxide layers was made by soaking the Resistant Titanium in an Etching solution (Regis Aqua). The data gave the same results as a Ti electrode tested without Acid Etching. Since the oxides layers provide a resistance protection, it would be in the best interest of bio-metal manufacturing companies to treat Ti implants. For the purpose of our experiment, further manipulation to rid the Ti electrode of the oxide layers is unnecessary.
Works Cited


Ohtsuki, Chikara, Ph.D, (n.d.) How to prepare the simulated body fluid (SBF) and its related solutions, proposed by Kokubo and his colleagues. Retrieved from http://mswebs.naist.jp/LABS/tanihara/ohtsuki/SBF/
