Anodizing Titanium

WARNING: This is a high voltage procedure. Handle all electrical equipment with caution.

What is going on?

Titanium reacts to exposure to oxygen by forming an oxide layer on its surfaces. The anodizing process causes this reaction to continue further, which produces thicker layers of oxide. As the oxide layer thickens, it interacts with the light waves that pass through and reflect off of the titanium surface. Depending on the thickness of the oxide layer, the different wavelengths of light will either constructively or destructively interfere. Essentially, this allows some colors to be reflected off the surface while canceling others out.

Supplies

<table>
<thead>
<tr>
<th>Item</th>
<th>Substitute</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>☞ Titanium work piece</td>
<td>Niobium</td>
<td>Anode; work piece to be anodized</td>
</tr>
<tr>
<td>☞ Titanium sheet/wire</td>
<td>Niobium, stainless steel</td>
<td>Cathode. Note: Surface area should be greater than that of the anode, and it should be able to wrap around the inside perimeter of the container</td>
</tr>
<tr>
<td>☞ Trisodium Phosphate (TSP)</td>
<td>Borax, ammonium-phosphate</td>
<td>Provides the free ion content in the electrolyte solution</td>
</tr>
<tr>
<td>☞ Distilled water</td>
<td>Demineralized water</td>
<td>Note: tap water contains minerals that can cause defects in the oxide layer as it forms</td>
</tr>
<tr>
<td>☞ Power supply, &gt;100V</td>
<td>Batteries (e.g. 9V)</td>
<td>The color achieved is voltage dependent, and the power supply provides the voltage</td>
</tr>
<tr>
<td>☞ Plastic container</td>
<td>Glass or other non-conductive container</td>
<td>Holds the anodizing bath. Needs to be large enough to comfortably hold the both the work piece and cathode</td>
</tr>
<tr>
<td>☞ Beaker</td>
<td>non-conductive container</td>
<td>Holds distilled water to rinse work pieces after anodizing</td>
</tr>
<tr>
<td>☞ Alligator clips</td>
<td>Electrical wire</td>
<td>Connects to work piece and cathode to the power supply</td>
</tr>
<tr>
<td>☞ Plastic Mesh</td>
<td>–</td>
<td>Precautionary measure to further prevent work piece and cathode from coming into contact</td>
</tr>
<tr>
<td>☞ Safety Glasses</td>
<td>–</td>
<td>Eye protection</td>
</tr>
<tr>
<td>☞ Gloves</td>
<td>–</td>
<td>Skin protection</td>
</tr>
</tbody>
</table>

*** Observe all normal high-voltage handling guidelines
Directions

(1) Prepare the Titanium
   (a) Dirt, dust, oils, and other foreign materials interfere with the anodizing process, so the titanium must be cleaned before it can be anodized. Etching is the most effective. However, the best way to etch titanium requires the use of HF. The safest alternative to that is Multi-Etch® (link at end of document). Follow the manufacturer’s instructions carefully.
   (b) If you decide not to etch the titanium at, instead sand the piece, then use acetone or alcohol to clean it off.
   (c) Beyond the work piece, it is also helpful to have a titanium handling wire. This is used to fully submerge the work piece without the alligator clip coming into contact with the water. Depending on the nature of your work piece, you may consider making an assortment of hooks and cradles to suit your needs. While it is unnecessary to etch this wire, it should still be cleaned with acetone or alcohol to prevent foreign materials from being introduced into the electrolyte bath.

(2) Prepare the Anodizing Bath
   (a) Mix the TSP with distilled water and pour into the plastic container you’ll use for anodizing. Concentration: 5 g/L.
   (b) Put the cathode in the solution. In my setup, I used coiled titanium wire that wrapped around the perimeter of the bath. The titanium will anodize more evenly if the cathode wraps around the perimeter of the container, and is approximately equidistant from where the work piece will be. Also because this process does not have much throwing power, it is important that the cathode has a larger surface area than the anode.
   (c) Place the mesh inside the solution. The purpose of the mesh is to prevent the work piece from touching the cathode and shorting the circuit.

(3) Prepare the Power Supply
   (a) In my anodizing process, I used an 300V-3A power supply. Voltage and current this high is dangerous and can be fatal if not handled carefully. Before plugging in the leads, turn the power supply ON and make sure the voltage is 0. Then, with the power supply OFF, connect the black (negative) lead to the cathode where it rises out of the anodizing bath.
      (i) If using batteries, use a wire to connect the negative end of the battery to the cathode. As the color the titanium will anodize to is voltage dependent, reference the chart under 'references' to determine the number of batteries you require.
   (b) Note: The connections are the reverse of the electroplating process. This is because in the anodizing process, the current flows from the work piece instead of to the work piece.
   (c) IMPORTANT! The alligator clip on the lead must not touch the electrolyte bath. Any metal coming into contact with the solution other than titanium, niobium, or the cathode will disrupt the process from taking place.

(4) Anodize the Work Piece
   (a) Put on safety equipment (if not already done so). Specifically, rubber gloves and safety glasses.
   (b) Attach the work piece to a titanium handling wire (if being used). Then connect the red (positive), lead to either the handling wire or the work piece. Note: if the test lead is clipped directly to the work piece, the work piece will not be able to completely submerge in the bath and the entire piece will not be evenly anodized.
   (c) Turn the power supply ON, keeping the current and voltage set to 0.
   (d) Submerge the work piece into the anodizing bath.
   (e) Slowly, turn up the voltage until a predetermined setting is reached or until the desired color is achieved, then remove the work piece from the bath. Note: the current generally remains fairly low; however, if the reaction is too slow you can adjust the current up to quicken the reaction.
   (f) Turn the voltage down to 0 and turn the power supply OFF.
Notes and Precautions

- When the piece is anodizing, there will be bubbles in the solution. This is hydrogen bubbling off. Make sure the space has good ventilation to account for this explosion hazard, especially when anodizing large work pieces at high voltages.
- Always turn power OFF before connecting alligator clips to any circuit.
- Never use your finger to test a "hot" line. Use approved voltmeters or multimeters.
- Always be aware of the nearness of high-voltage lines or circuits.
- Always discharge the high voltage from components or terminals using a safety probe.
- Don’t allow any non-titanium (or niobium) parts to touch the solution. They can contaminate the solution and interfere with the anodizing process.
- Once a piece of titanium has been anodized to a certain color, it can only be anodized more to go up on the color scale. For instance, if you anodize a certain piece to yellow at 80V, you won’t be able to get the color purple it achieved at 35V.
  - That being said, all this anodizing does is form a thin oxide layer on the surface of the titanium. It is fairly easy to sand off. So, if you decide that you mistakenly anodized a piece too far or just want to start over, you can always sand it off.

References

http://www.riogrande.com/Content/Bath-Anodizing-Titanium-HT-psd
http://www.mrtitanium.com/interference.html
http://multietch.com/

DC Voltage (approximate)