# **Closed Feedback Loop Anodizing**

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This paper describes a closed loop feedback method applied to the anodization process. This simple and effective method uses a current source, and permits measurement of the bulk resistance of the work as it anodizes. By monitoring this resistance, it is practical to determine in real time; the time at which dissolution overtakes anodic growth, and the thickness/density of the anodic layer. Automatic compensation for other anodizing parameters; such as temperature effects, electrolyte dissolved Al content, and anode/cathode area ratio is available. This closed loop feedback method also shows an unusual tolerance for Al alloy composition. A "smart rectifier" to automatically execute this method will also be described. Also described are an efficient anode wire attachment method, and a means to measure the surface area of the work that has a complex shape.

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#### Scope

The perspective of this paper is Small Scale Anodizing. This differs from the commercial large scale practice only in the size of the setup. Tank sizes range from 11 L to 379 L (3 to 100 gal.), power sources range from as low as 150W up to about 3 kW. The usual applications for Small Scale Anodizing are research experiments, commercial prototype shops, small businesses and university laboratories. This should not be confused with "amateur" or "hobby" anodizing; it is not done with a battery charger in a plastic bucket. Small Scale Anodizing can produce results as good as or better than standard commercial practice. This is because better control over all anodizing parameters can be obtained in small scale and at lower cost. The actual temperature and temperature gradient of the electrolyte and the work can be more tightly controlled. Agitation methods are more effective in small scale. The power sources used provide much better current regulation, voltage compliance, and absolute accuracy than a traditional multi-kilo Amp rectifier. Small Scale Anodizing will not replace commercial practice, but occupies a niche where commercial practice is neither efficient nor cost effective. In spite of the small scale perspective, most of the material presented here can also apply to large scale commercial practice.

### The Constant Current Source

Constant current anodizing is hardly a new idea, but when anodizing is looked at from the electrical point of view, constant current is clearly the easiest and best method.

Constant current control is explained as follows. Power sources that are capable of this have a means to measure the current that they are providing; they also have a circuit that compares this measurement with a reference that is adjustable by the user. This control circuit adjusts the output voltage so that the output current is identical to current set by the user. As the load on the power source changes, the control circuit responds by further adjusting the output voltage so that the set current remains the same. With change in load, the voltage goes to whatever value is required to keep the current at the constant value. Hence the name of this control method, *constant current*. This relationship is maintained up to the maximum voltage the power source is capable of; this limit is called the power source's maximum *voltage compliance*.

Constant voltage control in contrast, adjusts the output voltage to conform to the reference set by the user, and allows the current to change in order to keep the output voltage constant. This will operate up to the maximum current capability of the power source, which is its *current limit*. This method is the most common in commercial power supplies in general and in anodizing rectifiers.

Laboratory type general purpose power supplies are commonly used in small scale anodizing; the majority of these will operate in either constant current or constant voltage mode. Premium professional units<sup>1</sup>; with excellent regulation, measurement capability and electrical protection, are available up to 30kW, and cost less than \$1 per Watt. The more recent professional anodizing rectifiers also have both control modes available, even for the very large models. Advances in industrial electronics during the last 50 years have made constant current control of even very large anodizing rectifiers practical and affordable.

### **The Anodizing Circuit**

Describing the general anodizing process in terms of a closed loop DC electrical circuit is useful in explaining the compensation functions that constant current operation provides. The term *closed loop* implies that the system operates with a feedback mechanism, and responds in a favorable way to electrical changes it can sense in the anodizing circuit. The DC circuit analogy described here also applies to pulsed current anodizing; because the pulse frequencies used are so low in electrical terms, AC effects such as impedance and reactance have little effect and can be conveniently ignored.

The anodizing circuit is so simple it doesn't warrant a schematic diagram, its nothing more than a number of resistances, all connected in series with the power source. The power source is capable of measuring both the voltage and the current it is providing. These resistances and what they represent are listed below:

## Power source to anode connection wiring resistance

This is either wire or a bus bar, if properly sized for the setup this resistance is stable and typically measures a few to tens of m $\Omega$  in magnitude. If racking is used, this resistance is represented by multiple resistances, connected in parallel to each other.

## Anode connection

This is the actual electrical connection to the work, and usually the most troublesome connection. We have all experienced unacceptable anodizing due to this connection being inadequate or degrading to inadequate. Since this connection is subject to anodization, if it is not liquid tight the resistance of this connection will increase substantially during the anodization. The typical resistance ranges from a few  $\mu\Omega$  for a fusion welded connection to 1-50 m $\Omega$  for mechanical connections. The welded connection is the most stable because the electrolyte cannot penetrate it at all. Mechanical connections can be nearly as stable in the case of bolted connections, or those that have had enough mechanical force applied to cold flow one or both of the metals. Connections relying on spring force or the weight of the work on a contact point are the least stable. The resistance stability of these connections will vary widely; 10's of m $\Omega$  in the best case to an open circuit in the worst case.

## Coating equivalent resistance

This is composed of the electrical conductance of the electrolyte when it is restricted by the hollow columnar shape of the pores. All pores that can make any electrical contact with the base metal and the electrolyte in the tank contribute to this. Its magnitude is affected by the coating density, coating thickness, and surface area. This equivalent resistance has dimensions of Ohms per unit area. It is recognized that representing the coating as a resistance per unit area is an over simplification, but this serves the purpose here.

## Electrolyte conductivity

Electrolyte conductivity is the reciprocal of electrolyte resistance; it is affected by the electrolyte composition, temperature, the surface areas of the anode and cathode in contact with it, and the anode/cathode separation. Dissolved aluminum in the electrolyte reduces the electrolyte conductance<sup>2</sup>.

### Cathode to power source return wiring resistance

This is similar to the anode wiring resistance.

#### The Operation of the Anodizing Circuit

When power is applied to the circuit, Ohm's Law requires that the current passing through each resistance must be the same, thus the voltage dropped across each resistance will depend on the value of each resistance. Kirchhoff's Law of Circuits states that the sum of the voltages dropped by the resistances must equal the power source voltage.

# Constant Voltage Operation

If the power source is regulated by constant voltage, any change in any resistance will cause a change in the current flowing in the circuit. This causes the current density to change. The equivalent resistance of the coating will increase as it gets thicker, reducing the current density. The change in electrolyte conductance with temperature will also change the current density. Connection resistance; especially the anode connection, can change the current density drastically. The current density change can be compensated for by adjusting the power source voltage manually.

## Constant Current Operation

If the power source is regulated by constant current, the current will remain the same regardless of resistance changes. This provides a uniform current density throughout the anodization. This will be the case so long as the constant current source has sufficient voltage compliance. This operation is automatic, and requires no intervention by the operator.

#### **Voltage Ramping**

At the start of the anodizing process, the equivalent resistance of the coating will be very low, because there is no coating yet. With constant voltage, this very low resistance causes a very large current to flow, limited only by the power source's current limit. This will start to subside after a few minutes when some coating is formed and provides some resistance. Since higher current densities provide faster coating formation, this over-current condition lasts for a shorter time with higher current densities. This over-current condition can quickly damage electrical connections, and should be avoided. In constant voltage operation, the usual remedy for this is to start the process with a low voltage, and to increase it with time up to the desired current. It is difficult to correct the change in current density accurately with this method.

Unless ramping is done to provide a reduction in current density at the beginning of the process for another process reason, it is not necessary or desirable when using constant current. The current regulation is functional and linear all the way down to zero in a properly designed constant current source. The preset current is accurately maintained even at the start of the process.

#### A Coating Thickness/Density Assessment Method

The simple Ohm's Law based electrical analogy described above was applied in an attempt to gain insight in the workings of several groups of anodization experiments.

## First Group of Anodizations

In the first group alloy was the selected variable. The anodizing conditions were as follows:

- 1. Electrolyte; 9.4% (vol.) sulfuric acid.
- 2. Electrolyte dissolved aluminum content; near zero, new mixture.
- 3. Electrolyte temperature 21.33°C ±0.17°C (70.4°F ± 0.3°F). Substantial aeration was also provided.
- 4. Current density held constant ( $\pm 0.015\%$ ) at 1.67 A/dm<sup>2</sup> (18 A/ft<sup>2</sup>).
- 5. Anodizing time 90 minutes.
- 6. Four alloy samples; T-1100, T-6061, T-2024, and T-7075. All were commercial bar stock, average surface area 93 cm<sup>2</sup> (0.10 ft<sup>2</sup>) identical surface preparation.
- 7. Anode connection; 14 AWG T-1100 wire, fusion welded to the sample.
- 8. Each sample was dyed black for 30 minutes at 60°C (140°F) and sealed in boiling water for 10 minutes.
- 9. Coating thickness measurements were made with an eddy current electronic coating thickness gauge. The average of 10 measurements made 48 hrs. after sealing results in the thickness figures provided. The span was calibrated against a commercial 100 μm calibration shim, and the zero was set with an unanodized sample with the same surface treatment as the other samples.

Data was collected by sampling and recording the applied voltage at 1 minute intervals. The data was processed by conversion into its equivalent resistance. All curves actually start at zero seconds and near zero Ohms. The truncation was done to improve Y axis resolution. Figure 1. shows the plotted data.



Figure 1. 18 A/ft<sup>2</sup> Anodizations of Four Alloys

My interpretation of Figure 1. is the following:

1. The curves are in good agreement with published literature<sup>3</sup> regarding which alloys are more readily anodized, and which aren't.

- 2. Each alloy has its own characteristic bulk resistance values.
- 3. The "difficult" alloys (2024, 7075) do not show in increase in bulk resistance with time. These alloys also had the thinner coatings. The downward slopes are due to the slower rate of coating formation being overtaken by the effect of dissolution widening the pores, which lowers the bulk resistance.
- 4. The "easy" alloys (1100, 6061) show an increase in bulk resistance with time. This indicates that the rate of coating formation is faster than the rate of dissolution, resulting in thicker coatings and smaller pore size.

After the samples were sealed, a powder residue was wiped off of the "difficult" alloys. This was assumed to be the remnants of the coating undermined by dissolution and broken off by the wiping action. This powder was not evident on the "easy" alloys.

## Second Group of Anodizations

In the second group current density was the selected variable. The anodizing conditions were the same as the 1<sup>st</sup>. group except for the following:

- 1. The alloy was 6061 exclusively.
- 2. Current densities of 0.59, 1.12, 1.40, 1.67, and 1.95  $A/dm^2$  (6, 12, 15, 18, and 21  $A/ft^2$ ) were used. Each was held accurate to three decimal places.
- 3. The electrolyte temperature was held less tightly to 21.1-22.2°C (70-72°F).
- 4. One anodization  $(15 \text{ A/ft}^2)$  was terminated at 60 minutes.

5.

Figure 2. shows the plotted data.



Figure 2. T-6061 Anodizations at Various Current Densities

My interpretation of Figure 2. is the following:

- 1. For a given alloy, the bulk resistance is higher with lower current density.
- 2. The thickness of the 18  $A/ft^2$  sample is less than the 6061 sample in Figure 1. but its bulk resistance is in agreement with Figure 1. for this thickness.

- 3. The 6,12, and possibly 15 A/ft<sup>2</sup> samples slope downward. This indicates that the coating growth rate should be faster to stay ahead of the dissolution rate existing here; in other words, the current density should be higher.
- 4. The 18 and 21 A/ft<sup>2</sup> curves slope upward, indicating adequate current density for the dissolution rate.
- 5. The 21 A/ft<sup>2</sup> curve looks too radical compared to the others. This could have been caused by a degrading connection. There are no other symptoms of a degraded connection; when the wire was broken off after sealing there was no sign of electrolyte intrusion, and the high coating thickness reasonably agrees with the high bulk resistance.

# Third Group of Anodizations

In the third group an electrolyte concentration of 5% (vol.) was used in place of the traditional 9.4% (vol.). These were done under the same conditions as the previous groups except for the following:

- 1. Low current densities were used for some of the anodizations.
- 2. The electrolyte temperature was held to 21.1-21.7°C (70-71°F).
- 3. Current density accuracy and regulation was to within 2% due to the electrical equipment differences in this group.

Figure 3. shows the plotted data.

My interpretation of Figure 3. is the following:

- 1. As in Figure 2. lower current densities show higher bulk resistances.
- 2. The lower electrolyte concentration has lower conductivity than the traditional concentration, this is evident in the higher resistances observed.
- 3. The apparent dissolution rate is much lower with the lower concentration electrolyte. The coating growth rate can stay ahead of the dissolution rate down to current densities as low as  $4.5 \text{ A/ft}^2$ .
- 4. There was no powder residue after sealing observed in any sample, this indicates no excessive dissolution down to 3 A/ft<sup>2</sup> inclusive.
- 5. All samples dyed as easily and as well as those done previously with 9.4% (vol.) electrolyte. There was no evidence of insufficient pore size in either the dyeing or the apparent "hardness" of the coatings.



Figure 3. T-6061 Anodizations in 5% (vol.) Electrolyte

## The Benefits of Lower Concentration Electrolyte

A number of small scale anodizers, including myself, have been experimenting with 5% (vol.) electrolyte for quite some time. Although to my knowledge there have been no detailed examinations of the resulting pore structure, we see no detrimental effects. One effect that would be of negative consequence in a large scale setup is the lower conductivity of the electrolyte. This will cause more power dissipation than a higher conductivity electrolyte. The conductivity of 5% (vol.) electrolyte is about 15-25% lower than 9.4% electrolyte. The positive effects of the lower concentration electrolyte are the advantages of a lower dissolution rate, and the less corrosive, safer nature of the lower acid concentration. This provides lower electrolyte cost, and less degradation of electrolyte agitation and cooling equipment by acid attack. Table 1. shows for 90 minute anodizations, the increase in coating thickness when the losses due to higher dissolution are considered. These advantages may offset the higher power dissipation disadvantage in some large setups.

Current Density	Coating Thickness		Difference
	5% (vol.)	9.4% (vol.)	Difference
$6 \text{ A/ft}^2$	18 µm	15 µm	3 µm
$12 \text{ A/ft}^2$	41 µm	35 µm	6 µm
18 A/ft <sup>2</sup>	64 µm	56 µm	8 µm

# Table 1. Coating Thickness vs. Electrolyte Concentration

## **Other Items of Interest**

#### Electrolyte Dissolved Aluminum Content

Some anodizing literature contains recommendations of a minimum dissolved aluminum content in the electrolyte for good results. One source<sup>4</sup> recommends a minimum aluminum content of 2 grams per liter of electrolyte. The stated reason for this is to prevent "burning" during the initial application of "voltage" to the load. This implies constant voltage regulation is being used. The dissolved aluminum will have the effect of lowering the electrolyte conductivity, thus providing some inaccurate at best, and usually inadequate amount of current limiting. This is an unnecessary complication when constant current regulation is used; there is no need for additional current limiting. When constant current anodizing is used, a minimum aluminum content requirement does not apply.

## Welded Anode Connections

This method was used to attach the anode wires to the work for most of the anodizations presented here. In terms of electrical performance and immunity to the anodization process, this method is far superior to any mechanical attachment method.

Welded connections should be particularly valuable in Hardcoat (Type III) anodizing, where the very high current densities demand the lowest possible Ohmic connections to be reliable. Only some racking methods can exceed it in the speed at which connections can be made. After the entire anodization process is completed, the welded wires are broken or sheared off flush with the work. A weld pit is left on the work and an unanodized area the size of the wire diameter is evident as a flaw. In anodizing situations where this can be tolerated, welding will outperform and is faster and cheaper to apply than mechanical methods. There is no need for a mounting hole in the work to make the connection.

The use of a conventional aluminum welder for this purpose is hazardous to the work; they are much too powerful, which will cause large weld pits and can burn holes in thin work. We are developing a small welding machine, an early prototype of which was used in these experiments. This machine uses a Capacitive Discharge Welding method, and is optimized for welding 8 AWG to 18 AWG aluminum wire to aluminum. The machine incorporates a mechanism to control the weld force, which minimizes the weld pit and shields the user from the welding arc. It can be safely used on work as thin as 0.76 mm (0.03") and can be used on work over 25.4 mm (1") thick. It generates no perceivable heat in the work or the wire. No welding skills are required to use the machine optimally. Figure 4. is a photograph of this prototype. We hope to present a paper on this technology and its application at a future date.



Figure 4. Prototype CDW Welder

## Surface Area Measurement

To obtain good and consistent results when anodizing; it is necessary to know the total surface area of the work, in order to set the required current density. The calculation of surface area becomes complicated and laborious when the work has a complex shape. Since the measured conductance or resistance of an electrolyte will be largely related to the total surface area of the electrodes in contact with the electrolyte, this method should provide adequate accuracy in determining the area of the work, which is used as one of the electrodes.

The container or tank holding the electrolyte is sized to allow at least several inches of clearance between the work and any container surface. Sufficient electrolyte is added to the tank to totally submerge the work. The tank is made of or lined with an electrically conductive material and serves as one electrode. The work is provided with an electrical connection and is the other electrode. With work measuring 93 cm<sup>2</sup> (0.1 ft<sup>2</sup>) in area, a resistance of about 4 k $\Omega$  was measured when plain water was used as the electrolyte. This was conveniently measured with an ordinary digital multimeter. If the work is replaced with an electrode of known surface area, the area of the work can be derived by comparison of the resistance between the known area and the work. If the area of the work is large or the conductance of the electrolyte is high, an electrical conductance measurement may be used instead of a resistance measurement.

Early experiments showed that if the tank was used as one electrode, the effect on measured resistance or conductance by electrode separation was largely cancelled. This is because as the distance from one electrode to the tank increases, the distance on the opposing side decreases. It may be advantageous for the bottom of the tank to not be conductive since adding an electrode surface on the opposing surface (the top) would complicate matters. A cylindrical tank may work better than a square one.

The reference electrode of known area should be made of the same material as the work, or be close to it in electrical conductivity. The surface textures and degree of preparation should also be similar to reduce measurement error. An AC current may be used to make the measurement should polarization or ionization effects become a problem. This did not appear to

be an issue with the low DC current the multimeter applied to make the resistance measurement. A four wire (bridge) arrangement could also be used if an increase in measurement sensitivity is needed. Any suitable conductive liquid or possibly a dense conductive gas could be used as the electrolyte. Anodizing electrolyte could be used, but this has a very high conductance, which produces resistances too low to conveniently measure. Conductance measurement must be used in this case.

#### Conclusion

This method shows promise as a simple technique for monitoring the coating thickness/density while the process is operating. I don't see a way to separate coating thickness from pore structure (density) that will operate in real time. The equipment required to use this method is minimal and probably is already on hand for many anodizers. The 1 kW laboratory power supply I used has provisions to be monitored and controlled remotely either in a digital or analog mode. The digital mode will interface directly to a personal computer, thus the data collection and bulk resistance calculations can be easily automated.

It may be possible within limits to automatically apply a correction to sloping anodization curves, such that the coating growth / dissolution relationship is kept favorable during the anodization process. This additional feedback loop would adjust the current density and/or adjust the electrolyte temperature to provide the desired slope. Adjusting the electrolyte concentration in real time may not be practical.

Some may question the added step of converting the applied voltage data to bulk resistance; you will get similar curves by just plotting the voltage. In my opinion, doing this is tracking the symptom of an effect, and not the effect itself, which does not promote an understanding of what is actually going on. The calculation of bulk resistance is simply dividing the applied voltage by the current density. Like all things electrical, the work in anodizing is done by current, not voltage<sup>3</sup>. Voltage serves to overcome the resistance, so that the required current can flow. The resistance is the bulk resistance that has been discussed here. The chemical reactions and effects characteristic in anodizing manifest themselves in the anodizing circuit as changes to the bulk resistance, and have nothing to do with voltage in the anodizing circuit. This is Ohm's Law.

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