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Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Report on Performance of Prototype Dynatronix Power Supplies Developed Under a Phase I DOE SBIR

EW Hoppe
JH Merriman

March 2011

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Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

The purpose of this study is to evaluate the prototype power supplies fabricated by Dynatronix, Inc. This project supports the advancement of electroforming capabilities to produce ultra-high purity copper. Ultra-high purity copper is an essential material used for a range of current and future fundamental nuclear physics programs such as the MAJORANA DEMONSTRATOR.

The Mach 30 power supplies are a new design built to the specifications from the requirements of Pacific Northwest National Laboratory (PNNL) with regard to timing, voltage, current output, and the required tolerances. The parameters used in these tests were developed empirically over a number of years based on testing a combination of thermodynamic and kinetic responses on the electroplating process.

The power supplies were operated in a typical cleanroom environment for the production electroforming at PNNL. The units that were received by PNNL in July, 2010 have performed satisfactorily and have demonstrated short term durability.

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1. Purpose

The purpose of this study is to evaluate the prototype power supplies fabricated by Dynatronix, Inc. Dynatronix is an industry leader in the design and manufacture of Pulse, Pulse Reverse and DC power supplies for the world-wide metal finishing industry. The Mach 30 power supplies are a new design built to the specifications of Pacific Northwest National Laboratory (PNNL) with regard to timing, voltage, current output, and the required tolerances. Evaluation of a prototype power supply developed by the client under a phase I DOE SBIR needed to be performed in an electroforming laboratory within the normal operating constraints and environment. The primary assumption of this study is that the operable power supplies that have been purchased from the client are representative of those to be used on MAJORANA and other future project work. Iterative PNNL testing should be followed by subsequent modifications by the client. Finally the products/deliverables will be the assessment of the performance of the power supply under actual plating regimes. Results of any observed performance shortfalls will be communicated to the client.

The design parameters for the PNNL specific units were determined from the requirements of our waveform for plating. So in this investigation of the power supplies the requirements of our waveform were the primary standard used.

2. Background

This project supports the advancement of electroforming capabilities to produce ultra-pure copper and provides opportunities for further progress in material growth. Ultra-high purity copper is a fundamental material used for a range of current and future nuclear physics programs such as MAJORANA. The capabilities that are expected to be developed under this project will address significantly more challenging requirements including the unprecedented copper purity levels needed in neutrinoless double-beta decay and dark matter experiments.

Forefront research in neutrinoless double-beta decay and dark matter relies on developing detectors with extremely low backgrounds from naturally occurring radioactivity in order to reach the desired sensitivities which will allow observation of these very rare processes. For example, the MAJORANA experimental goal is a background of only 1 event in a 4 keV region of interest around the decay energy of 2039 keV per ton of active detector mass per year. This is a factor of 100 lower than previous generation experiments and represents an enormous challenge in production and assay of the materials used to construct and house the detector elements. For several decades, PNNL and its collaborators have led worldwide efforts to achieve the lowest measured radioactive backgrounds and achieve the greatest discovery potential for new physics.

The main requirement for materials used in these experiments is the ability to purify the material of ubiquitous naturally-occurring radioactivity from U and Th, as well as any cosmogenic radioisotopes present. Copper is easily purified and has additional desirable properties that make it the preferred ultra-low-background structural material for many types of experiments. It conducts heat very well and can be prepared with very low surface emissivity (important for cryogenic detectors), and has excellent mechanical and electrical properties.

Over the past two decades PNNL has continued to refine the copper electroforming process to improve material quality (grain size, strength, etc.) while also improving purity – two aspects that are generally at odds with one another. This work led to the world-leading limits on neutrinoless double-beta decay produced by the IGEX experiment and has enabled new methods in nuclear forensic and ultra-trace analysis for a range of applications.

There are numerous variables which influence the usability of electroformed copper in these types of low background experiments. Among the most critical is the potential applied during electrodeposition and its stability. During electrodeposition, the applied voltage determines which species in the plating bath will be reduced at the cathode according to classic thermodynamic models such as the Nernst equation. This allows copper to deposit while excluding impurities, such as thorium and uranium that require greater potential to be reduced. One may be tempted to casually apply classic thermodynamics to this problem and conclude that very close control of the voltage alone would perhaps dictate purity. For example, the Nernst equation predicts that thorium with a half cell potential (E^o) of -1.9 V, would have to be at a concentration over 150 orders of magnitude greater than copper before it would deposit when the half-cell potential of copper of 0.34 V is applied. One would expect to obtain copper of extreme purity from contaminants such as Th when electroplating at the voltages required for copper electroforming.

However, the presence of impurities in electroplated copper such as thorium indicates that the Nernst equation does not accurately model most real-world plating systems. At PNNL, where assays for thorium at very low concentrations in copper have been developed, it has been found that codeposition of Th occurs regardless of its concentration relative to that of copper in the bath. Obviously, mass transport and other factors play a major role in the behavior of contaminants at these concentrations.

In order to help overcome some of these additional factors, pulse reverse plating techniques are employed. In this process, a potential waveform is used rather than simple direct current. This allows the surface of the electroform to undergo multiple deposition, dissolution, and redeposition cycles, allowing the resuspension of contaminant species and reducing mass transport effects. Unfortunately, the use of pulse reverse waveforms introduces an added level of complexity that, if not performed under extremely well-controlled conditions, may only provide nominal structural or purity improvements. Paramount to defining the myriad of variables affecting the electroforming process will be the production of highly accurate and variable voltage waveforms which spend a minimal amount of time at non-optimal potentials (fast rise and fall times) and demonstrate minimal noise characteristics once proper potentials have been achieved. In addition, the ability to control and monitor these parameters at the electroforming bath will be invaluable.

Critical to measuring any performance improvements from a new power supply will be the ability to perform physical property and purity tests on the copper produced. Electroformed copper produced at PNNL is near current assay sensitivity limits. These levels, in the sub picogram of contaminant per gram of copper range, are still about a factor of 10 greater than that required for next-generation nuclear physics experiments. Demonstration of this level of purity has only recently been possible and has proven to be extremely challenging. The existing best assay technique uses dissolution and pre-concentration of thorium employing ion-exchange methodologies, with subsequent ICP-MS analysis. The sensitivity of this radiochemistry and ICP-MS method is currently limited by process blanks, i.e. extremely small levels of thorium leaching from the process materials. Work is ongoing to push assay limits ever lower, and such assays have allowed the purity of samples from the finished material to be checked. Ultimately, the purity of the large-scale electroformed cryostats and shielding requiring even greater purity will rely on tightly controlled electrodeposition, entailing unprecedented voltage regulation.

The waveform used in these tests was developed empirically based on a combination of thermodynamic and chemical kinetics of the electroplating process. It is believed that this form maximized the plating purity but is relatively uncommon for the plating industry. Within the industry, shorter time duration and constant current driven plating are the main parameters that are used to design power supplies. PNNL's needs were unique in that a low voltage driven power supply was desired that would be operated for months at a time, whereas usually a few days would be considered a long time for most industrial purposes. Operational parameters need to be varied at the milli-volt level and kept at less than 1 volt for several months of plating. The specifications provided to Dynatronix and the power supplies capability as delivered are summarized as follows in Table 1:

Project Requirement	**Existing 3 Volt Output Capability**	**As delivered**
Voltage Resolution as low as possible.	0.003V	0.000-3.000V, 0.001V
Voltage Accuracy as low as possible.	1.0%	<1.0%
System Noise as low as possible.	<0.050Vrms	<0.030Vrms
Amperage resolution as high as possible	0.01 Amps	0 – 2.4 Amps, 0.001 Amps 2.5 – 24.0 Amps, 0.01 Amps 24.1 – 240.0 Amps, 0.1 Amps
80 Amps maximum average current	10 Amps	80 Amps
240 Amps maximum peak current	30 Amps	240 Amps
Voltage and Current Rise Time as fast as possible.	<50 *u*sec	<20 *u*sec
Voltage Overshoot as low as possible.	<10%	<5%
Timing: Forward and reverse	On-Time: 0.0 – 99.9 ms Off-Time: 0.0 – 99.9 ms	On-Time: 0.0 – 99.9 ms Off-Time: 0.0 – 99.9 ms
Timing Step Size	0.1 ms	0.1 ms
Operating Range as wide as possible.	10-100% of maximum rated voltage.	1-100% of maximum rated voltage.
Remote Voltage Sense Capable	Capability has been implemented in electropolishing applications but not to this level of sensitivity.	Capability has been implemented
As long of operating time as possible	10+ days	Theoretically unlimited

Table 1: Summary of the power supply capabilities available before this project, the needs of the PNNL electroforming group, and the observed performance of power supplies provided by this project.

The power supplies which are proposed need to be operated in typical clean room and electroforming laboratory conditions. The PNNL electroforming lab is a class 1K clean room that is located 37 meters water equivalent underground. The facility contains a number of electroforming baths the largest of which are approximately 2 feet square and 3 feet deep containing 200L of electrolyte with 1200 lbs of copper nuggets that form the anode. A cylindrical cathode of stainless steel is placed at the center with a copper-acid sulfate electrolyte solution. A usual plating time frame for copper grown in these large baths lasts several months. The size of the baths and the duration of the plating present challenges that the proposed power supply parameters can overcome.

3. Investigation

The power supplies were operated in a typical environment at PNNL for the production electroforming (see Figures 1, 2 and 3).

Figure 1: Power supplies set up in the laboratory.

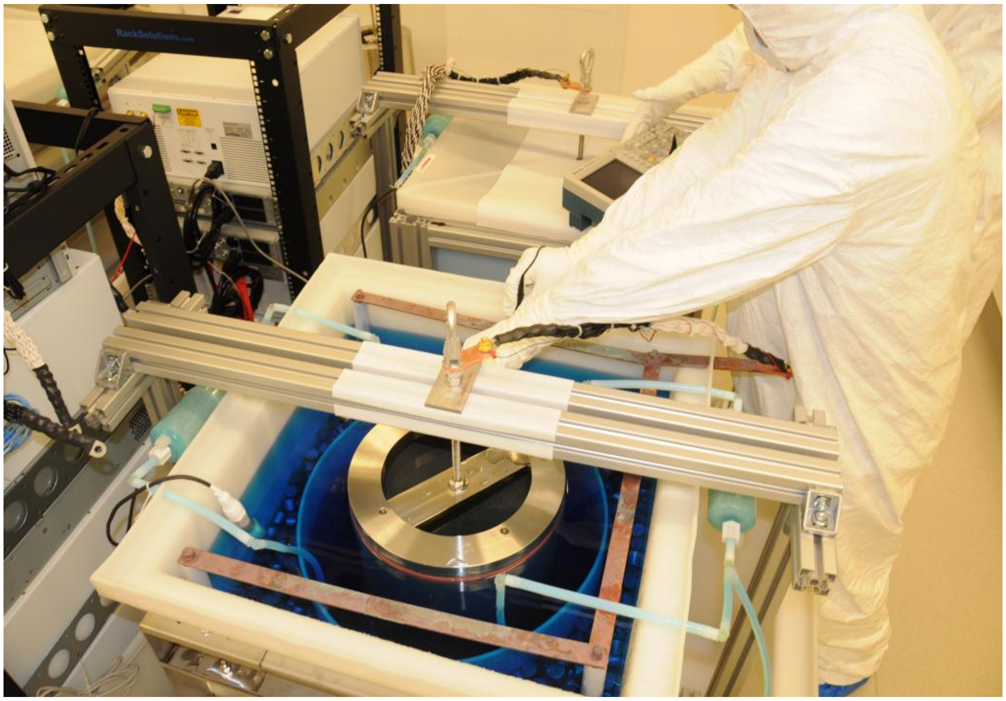

Figure 2: Typical bath set up and shows the checking of the waveform from the plating surface of the mandrel.

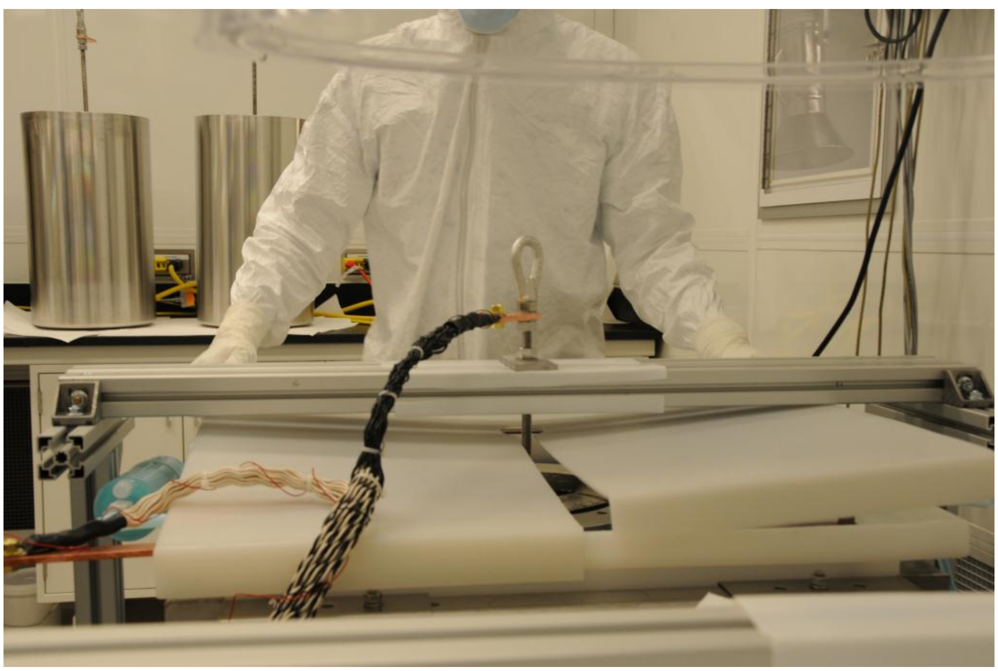

Figure 3: Configuration of the sensor wire and the braided cable for the baths at PNNL.

During setup the first item requirement was to give new IP addresses to each of the units. There were six units brought online and each could not utilize the same IP address. This was a simple process using the Mach10 configuration utility and an Ethernet connection.

The sensor wires did not allow for the spacing of the PNNL bath distance between the anode and cathode connection terminal. This problem was overcome by cutting the outer plastic covering to allow the two ends of the sensor wire to be spread apart. Two sets of these wires were provided at varying length. Accuracy of the waveform voltage is likely affected by the variation of impedance due to the length of this wire. Inductance or capacitance of this wire is likely affected by its physical orientation which may impact other parameters particularly at high waveform frequencies. The present configuration of the power cables as seen in Figure 3 are designed to minimize the vibration by using multiple 16 gauge wire pairs braided together. A single large gauge wire would pulse as the amperage changed but the finer wires allow the current to flow but limit the vibration from the pulsing. The braided pairs also served to reduce electronic noise at the low operating voltage and provided good cable flexibility.

Once the power supplies were set up they were attached to their respective baths and waveforms were captured on a Tektronix DPO2024 oscilloscope. Voltage settings were set based on the oscilloscope readings from the working surface of the bath electrodes. Five of the units performed nominally but one did experience communication problems.

Unit 1(S/N 0934805) was set at a voltage of 0.335 forward and 0.335 reverse. The amperage output was within nominal parameters. The waveform was slightly noisy but well within the existing capability of <0.050 volts and routinely met <0.030 volts.

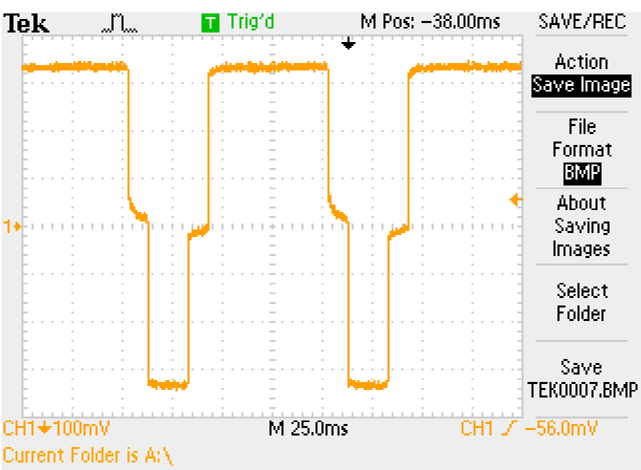

Figure 4: Waveform for Unit 1 as measured from the plating surface of the mandrel.

Unit 3 (S/N 0925301) was brought online next. The connection of the communication wire was much like that of unit 1 but after programming and operating, the following waveform resulted. Only the forward portion of the waveform was problematic.

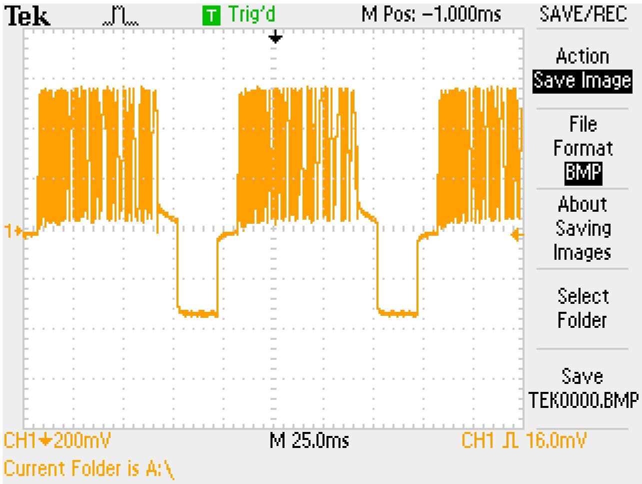

Figure 5: Waveform for Unit 2 with the noise of the forward pulse from the plating surface.

Several potential problem connections were investigated and ultimately the sensor wire was replaced several times. The sensor wires had to have a section of the outer most plastic insulator covering removed to allow for greater separation between the two ends, it was determined which lead us to believe that perhaps the inner plastic insulator was compromised and was the source of the noise.

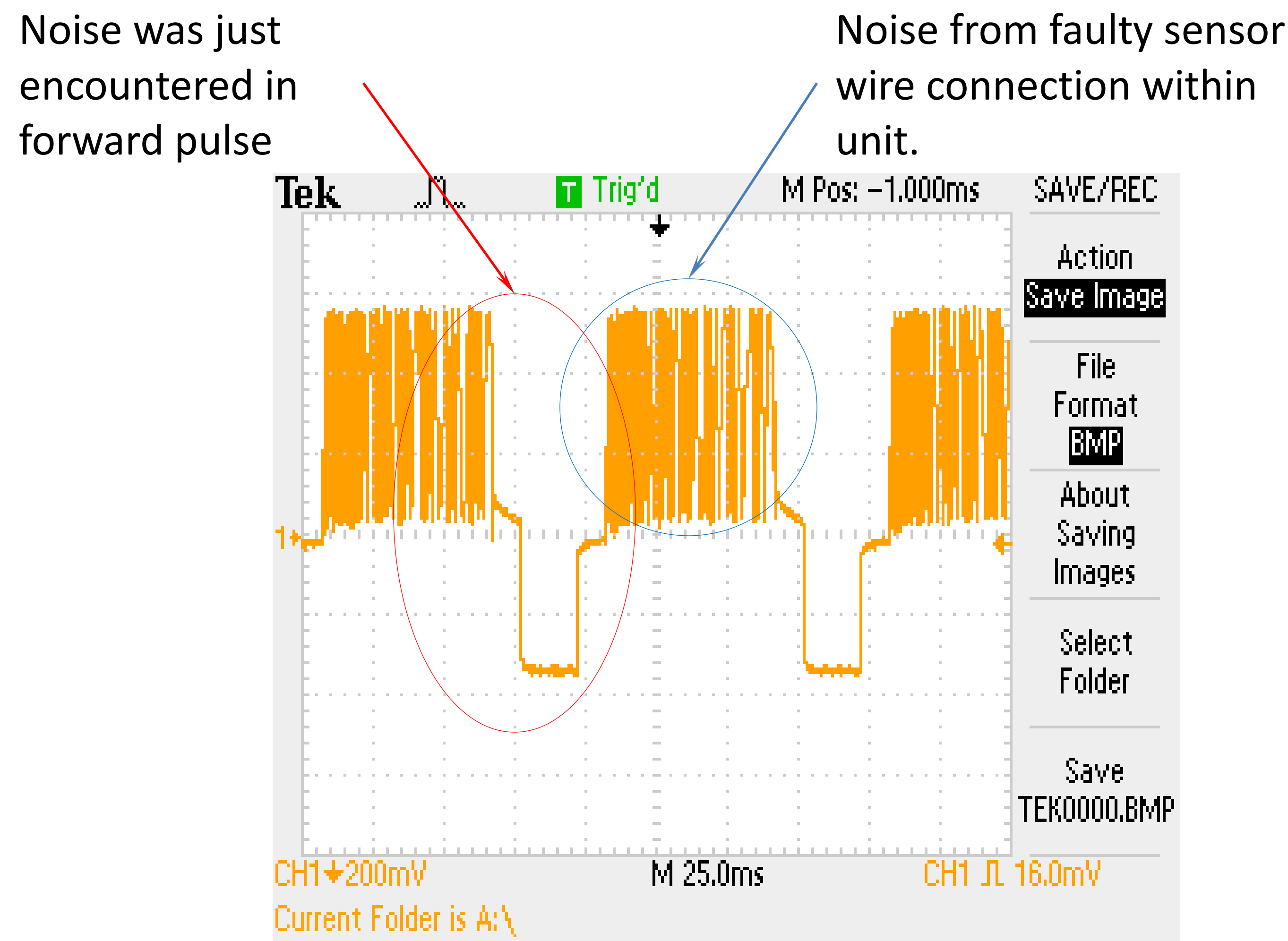

Figure 6: Forward pulse of the waveform.

When the sensor wire was pinched near the contact point of the electrode terminals the waveform was closer to what was programmed. The resulting waveform began to alternate between relatively normal and noisy. The forward pulse had a rather severe clipping of the forward corner. But this did not resolve the problem.

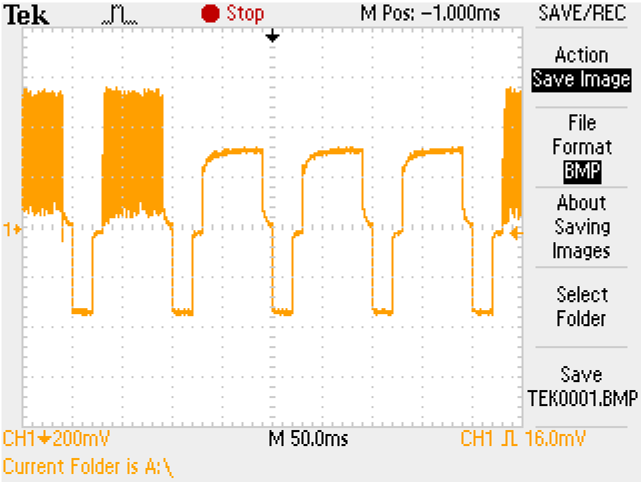

Figure 7: An example of the waveform as the sensor wire was pinched. It can be seen that the waveform oscillated between noisy and clipped forward pulse.

The unit was pulled and replaced (with S/N 0934801). This resolved the problem from a production stand point but the sensor problem still remains for one of the units. Once the problem was resolved the waveform was nominal. This also indicated that the slicing of the sensor wire was not the cause of the noise. It was unclear why in all instances only the forward pulse portion of the waveform was affected.

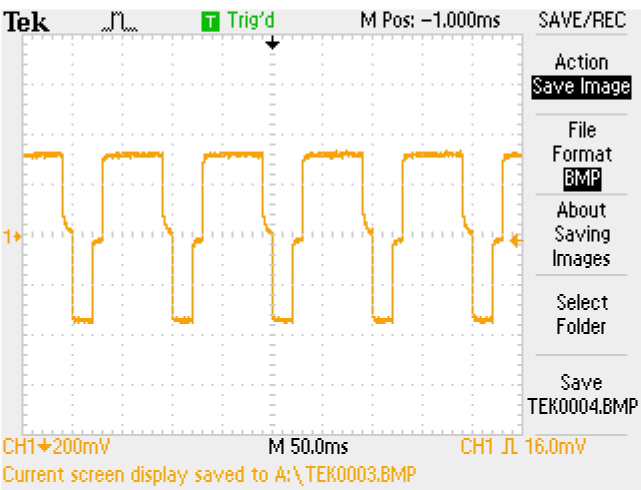

Figure 8: After replacing the unit the waveform can be seen to match what was programmed for the plating surface.

Unit 5 (S/N 0934807) was brought online after unit 3. The waveform was programmed and operated within normal parameters.

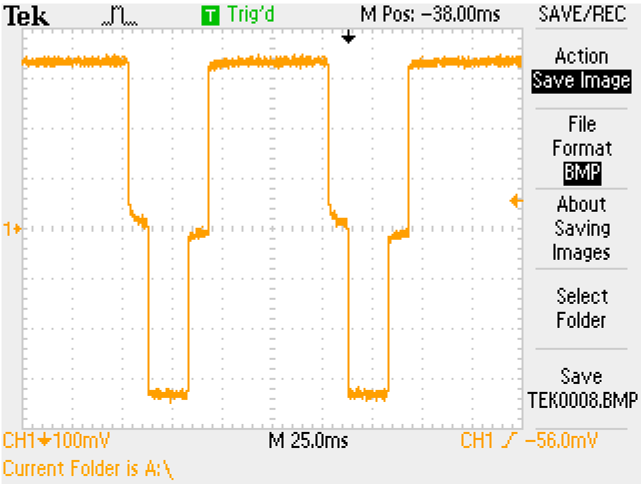

Figure 9: Waveform for unit 5 from the plating surface.

Unit 4 (S/N 0934802) was also brought online and performed as expected. The waveform was within operational parameters.

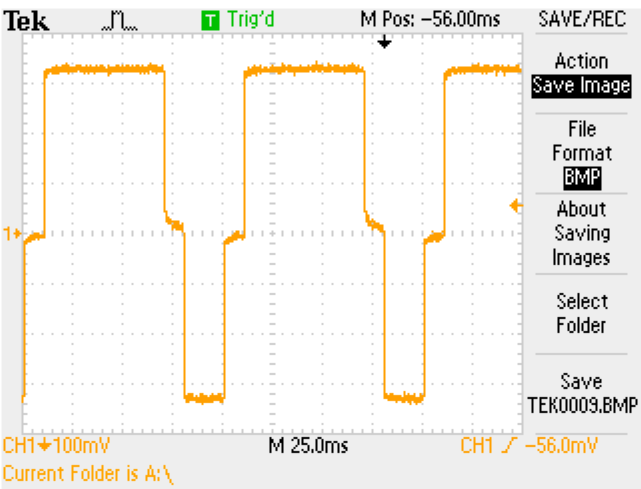

Figure 10: Programmed waveform from unit 4 from the plating surface.

Unit 6 (S/N 0901901) was also brought online as expected. The waveform was within operational parameters.

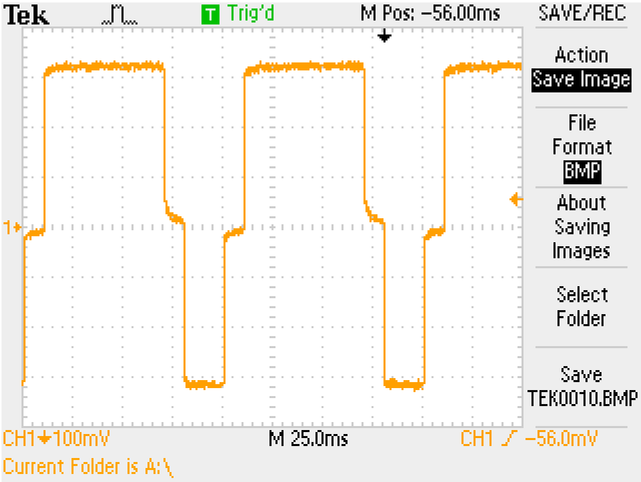

Figure 11: Waveform from unit 6 from the plating surface.

Unit 2 replacement (S/N 0934804) was the last unit put into operation. The startup was uneventful and operated within parameters.

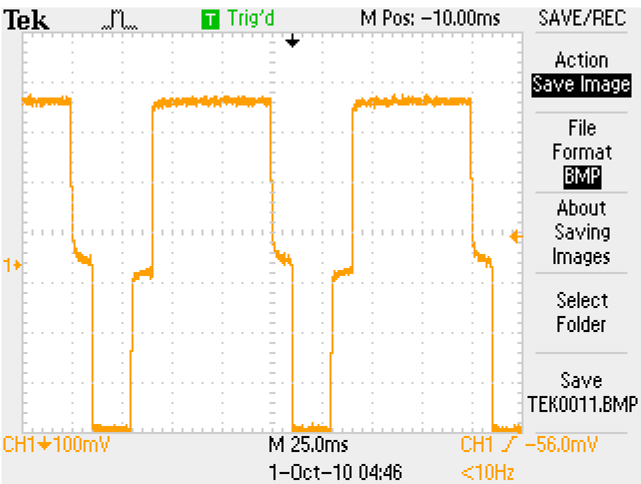

Figure 12: Above is the waveform for Unit 2 replacement as measured from the plating surface of the mandrel.

4. Discussion

The main problem encountered was with the communication of the sensor wire on Unit 3. This problem was significant due to its nature. It was determined that the issue was an internal problem with the unit and was beyond the scope of this effort. The unit that encountered this problem had been operated for several months previously which indicate that the current units will have to be observed for a much longer time scale to determine longevity.

Two sets of the sensor wires and output power cables were provided at two different lengths. Accuracy of the waveform voltage is likely affected by the variation of impedance due to the length of the sensor wire. Inductance or capacitance of this wire is likely affected by its physical orientation which may impact other parameters particularly at high waveform frequencies.

The output cable is a multi-stranded twisted pair arrangement of sixteen gauge wire. This method was utilized to solve potential noise issues while allowing the cable to carry a large current. However, as mentioned for the sensor wire, inductance or capacitance of this wire is likely affected by its physical orientation which may impact other parameters particularly at high waveform frequencies.

An overheating problem occurred early in the use of the first power supply. The unit was tested in an electropolishing configuration and ran at 80 amps for approximately 30 minutes. The unit over heated and shut down. It was discovered that a thermal protection setting was inappropriately low. The unit was returned to Dynatronix and the temperature alarm threshold increased. In subsequent units this was not a problem.

The current power supplies offer control of the waveform settings by driving the voltage to its set points and this allows for compensation for some of the capacitance of the system. The power supply does not drive the potential to zero. The roll-off, noted on the waveform as the power supply allows the system to return to rest shows the capacitance primarily from the bath and presents a possibility for further development. Driving the voltage to zero may be of interest is some cases so it must be examined in greater detail.

In conclusion, the units that were received by PNNL in July have performed satisfactorily and have demonstrated short term durability. The long term performance and durability still needs to be determined as the unit in service for 20 months did demonstrate an issue with the feedback sensor system and an overheating problem was discovered early in the use of the first power supply.

The following is a partial list of recommendations that have been made to Dynatronix for future improvements:

- An upgrade of the operating software should be developed to be compatible with current OS platforms such as Windows 7; the current version of DynaComm is only compatible with Windows XP.
- Isolatable reference probe configuration should be considered for greater control of plating voltages
- In more complex plating regimes the ability to produce an arbitrary waveform should be developed
- An ability to drive the waveform to zero in order to overcome the capacitance of the bath
- An elimination of particulate generation from the cooling fan requires an alternative cooling system such as a heat sink

If further developments are considered a poll of end users should be done to expand the list of potential improvements and/or capabilities to be added to these units.

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902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
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