Date: 12-Dec-2011
Grant No.: DE-SC0001979
Company: Dynatronix, Inc.
Project Name: "Voltage And Waveform Control For Improved Selectivity In Electrodeposition In Low Background Species"

Final Report

I. Cover Page

Contractor	Dynatronix, Inc. Amery, WI
DOE Laboratory Partner	Pacific Northwest National Laboratory Richland, WA
Project Title	"Voltage And Waveform Control For Improved Selectivity In Electrodeposition In Low Background Species"
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DYNATRONIX

Since 1971, Dynatronix, Inc. has custom designed and manufactured pulse, pulse reverse, and DC power supplies used in electroplating applications for the semiconductor, defense, aerospace, medical, nanotechnology, and general metal finishing industries. Located in Amery, Wisconsin, Dynatronix employs 70 people including 35 engineers and technicians plus administrative, sales, and production personnel. Over 65% of our products are shipped globally, reaching 2000+ customers to date.

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II. Purpose

This project required a custom power supply capable of controlling a copper electroforming process to the extent that ultra-pure copper may be electrodeposited.

III. Background

The majority of the power supplies sold into the electroplating industry only have direct current (DC) output and most copper plating is for the printed circuit board or semiconductor industry. In the printed circuit board market, the manufacturers are typically concerned with throughput, or the control of the power supply's amperage (current) output to maintain consistent copper plating rates. Impurities in the deposit or uniform distribution are not a major concern. Semiconductor manufacturers focus on high throughput and uniformity of the deposit through precise control of the output amperage. In both industries, the process run times are typically in seconds or minutes. Most operate 24/7, making equipment reliability a critical factor.

Pulse and pulse reverse power supplies were developed for the electroplating industry in the early 1970's, but have only gained mainstream acceptance within the industry in the past 15 years. Manufacturers have historically designed standard power supplies and worked to adapt them to the end user's process. Very few companies have invested in the engineering resources required to design custom power supplies for a specific process.

High frequency pulsing power supplies are designed around two basic topologies: linear and switchmode. Linear power supplies provide a stable, low noise, high accuracy output throughout the entire output range but tend to weigh more for their output capabilities. Linear power supplies have been the design of choice for most pulse power supplies over the years. Switchmode power supplies also provide a stable, low noise, accurate output through the entire range and are lighter and more compact than similar linear designs.

Pulse and Periodic Pulse Reverse plating have many advantages over traditional DC plating, including:

- Periodic Reverse improves the uniformity of the deposit without adding leveling agents.
- Pulse plating improves control of grain size and hardness of the electroform without metallic or nonmetallic additives to the plating solution.

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Dynatronix designs both linear and switchmode pulse power supplies and, with a large engineering staff on hand, is accustomed to designing pulse and DC power supplies to meet each end-user's requirement. For this project, we utilized a linear power supply topology to meet the stringent noise requirements requested by Pacific Northwest National Lab <PNNL>. Our portfolio of pulse power supplies included a standard design with an operating range of 10-100% of the maximum current rating and 10-100% of the maximum voltage rating. Our patented Extended Range (XR) programmable power supplies are designed with three internal current ranges that give the power supply an operating range of 0.1% - 100% of the maximum current rating and 10-100% of the maximum voltage rating. The XR design essentially combines three different current rating power supplies into one design.

In this project, Dynatronix, Inc. and Pacific Northwest National Laboratory determined the following custom designed, single output, programmable pulse and pulse reverse power supply provided capabilities to meet project requirements.

- 3 Volts maximum
- 80 Amps maximum average or DC output
- 240 Amps maximum peak pulse output

III. Design Work

In order to achieve PNNL's requirements, output regulation and accuracy needed to improve 10-100 times across several variables. To achieve this, Dynatronix, Inc. utilized the following design approach:

- Leveraged our existing patented XR technology to the voltage output of the power supply over a wide range of load conditions.
- Applied very precise remote voltage control in DC, pulse, and pulse reverse mode to plate the purest copper.
- Minimize electrical noise in the control circuit.

Achieving these specifications required Dynatronix to research the following manufacturing and test processes for noise measurement:

- How to accurately measure extremely low levels without background interference from an outside source.
- Determine equipment detection capabilities.

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Table 1: Summary of Technical Challenges and Research Required

Project Requirement	Existing 3 Volt Output Capability	RESEARCH CONDUCTED	RESULTS
Voltage resolution as low as possible.	0.003V	Reviewed voltage control of existing circuitry.	Increased the gain of the error amplifier in the control loop and used a 16 bit D/A and A/D converters.
Voltage accuracy as low as possible.	1.0%	Reviewed the circuit design and found the error amplifier gain is the biggest contributor to the output accuracy. Other signal conditioning amplifiers and their associated gains and offsets also contribute to the overall voltage accuracy	Selected error amplifiers and signal conditioning amplifiers to minimize offset errors while maximizing gain and bandwidth.
System noise as low as possible.	<0.050Vrms	Evaluated different noise sources in system.	Found that high frequency switching transients from switch-mode based DC/DC converters contributed to the noise spectrum in the system. Decided to implement transformer isolation and linear derived isolated power for internal control power. Also applied filtering in the system to reduce levels of conducted noise between subassemblies.
Voltage and current rise time as fast as	<50 *u*sec	Simulated several control schemes for linear output stage of the converter. Altered the drive	Altered our standard linear power output stage based on simulation results. Further improvements may be possible by further

possible.		circuit for the output stage to give more flexibility in compensating the control loop to provide speed and stability	modifications to the circuitry. More research would be required
Voltage overshoot as low as possible.	<10%	Simulation and experimentation with various load conditions and various control loop compensation networks.	Needed to sacrifice rise time of output pulse to ensure overshoot specification was not exceeded under various load conditions. This was a compromise solution since minimizing overshoot was given higher priority than rise time of waveform.
Operating range as wide as possible.	10-100% of maximum rated voltage.	Investigated implementing multi-range voltage control.	Found that work involved to significantly expand operating range would cause significant delays in the project. Given the intended application and the other higher priority requirements, the recommended operating range for voltage control was not significantly improved.
Remote voltage sense capable	Capability has been implemented in electropolishing applications but not to this level of sensitivity.	Reviewed remote sensing options	Found that use of shielded, twisted pair of sense wires was sufficient for this application. Included provisions in the design to power a remote preamplifier assembly if necessary in future applications.
Operation time of 10+ days	Already exits	Thermal analysis to optimize cooling.	Found that heat distribution on the internal heatsink was not sufficient and led to hot spots. Redistributed thermal load and thermally

			linked discrete heat sinks to provide lower operating temperatures for the power transistors.

IV. Summary

The power supply met all requirements of PNNL, see included report from PNNL.

Knowledge gained allowed us to improve power supply accuracy and to reduce overshoot on the output pulses without a significant impact on rise times. It also revealed other opportunities for product improvement. Future research could improve rise times under broader load conditions and enhance output waveform capabilities. The power output stage could still be improved upon to provide enhanced waveform capabilities. The ability to achieve true four quadrant operation from the power supply would be desirable in some applications. Also, higher input impedance for the remote voltage sensing amplifier will eventually be required. The ability to remotely monitor the output voltage and current waveforms would be desirable in some applications (remote oscilloscope functionality). The existing PC based control system used with this system is no longer compatible with newer versions of the Windows OS. A significant amount of work would be required to update this software program to allow for waveform editing and remote output monitoring.

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

Completed by Pacific Northwest National Laboratory (PNNL)
February 2011
Eric Hoppe
Jason Merriman

I. Purpose

The purpose of this study is to evaluate the prototype power supplies fabricated by Dynatronix. Dynatronix, Inc. 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 voltage, current output, and the required tolerances. The evaluation of a prototype power supply developed by the client under a Phase I Department of Energy Small Business Innovative Research Grant (Phase I DOE SBIR) needed to be performed in an electroforming laboratory within the normal operating 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.

II. Background

This project supports the advancement of electroforming capabilities to produce ultra-pure copper and provides opportunities for further progress in material growth science. Ultra-high purity copper is a fundamental material used for a range of current and future fundamental 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 uranium (U) and thorium (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. 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 pg/g 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. 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 they needed a low voltage driven power supply 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	0.01 Amps	0 – 2.4 Amps, 0.001 Amps

as high as possible		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.

III. Investigation

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

Figure 1: The top picture is the power supplies set up in the laboratory. The bottom illustrates a typical bath set up and shows the checking of the waveform from the plating surface of the mandrel.

Figure 2: In the picture above are the sensor wire and the braided cable for the baths at PNNL.

During setup the first item that was necessary was to give new IP address to each of the units. There were six units brought online and each could not be on 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.

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 preformed 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 out put 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.

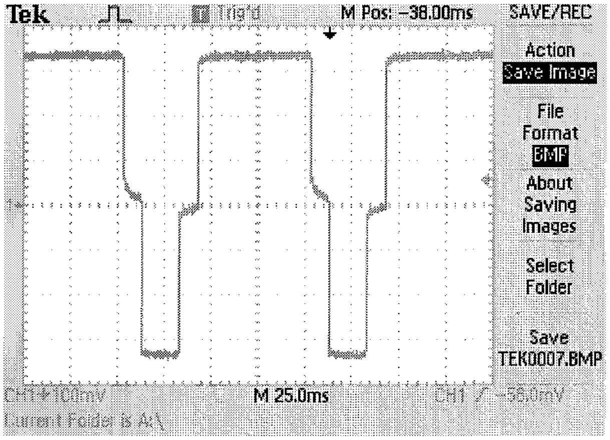


Figure 3: The above is the waveform for Unit 1 as measured from the plating surface of the mandrel.

Unit 3 (S/N 0925301) was brought online next. The connection of the communication wire was much like that of unit 1 but once we programmed the unit and began operations the following waveform resulted. Only the forward portion of the waveform was problematic.

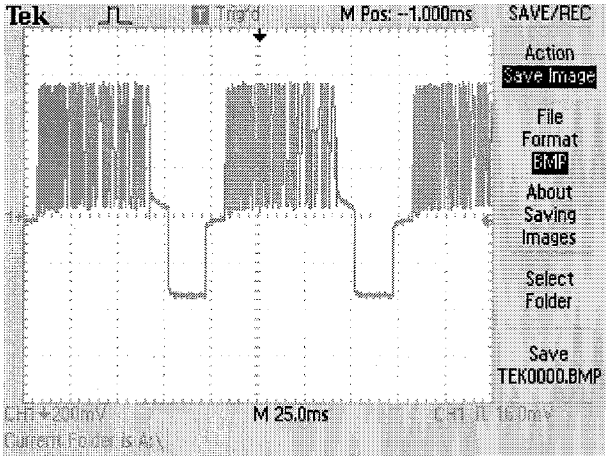
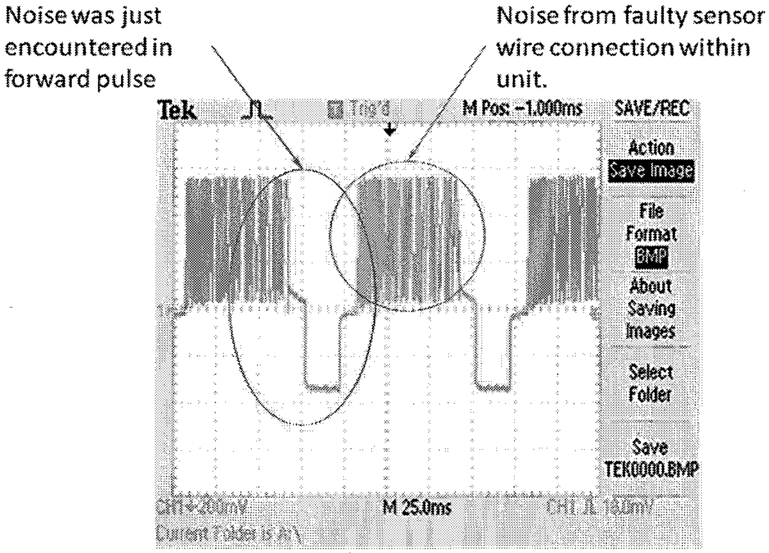


Figure 4: The above is the waveform for Unit 2 with the noise of the forward pulse from the plating surface.

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 which lead us to believe that perhaps the inner plastic insulator was compromised and was the source of the noise although it would have affected the waveform in both forward and reverse voltages.


Noise was just encountered in forward pulse

Noise from faulty sensor wire connection within unit.

Figure 5: The reasoning of our only isolating the forward pulse of the waveform as problematic is shown in this figure.

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.

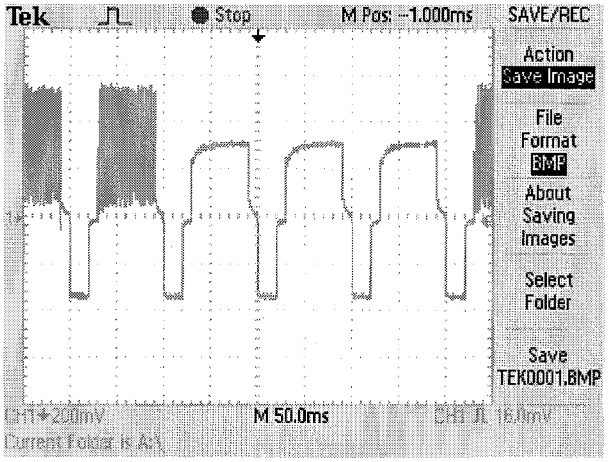
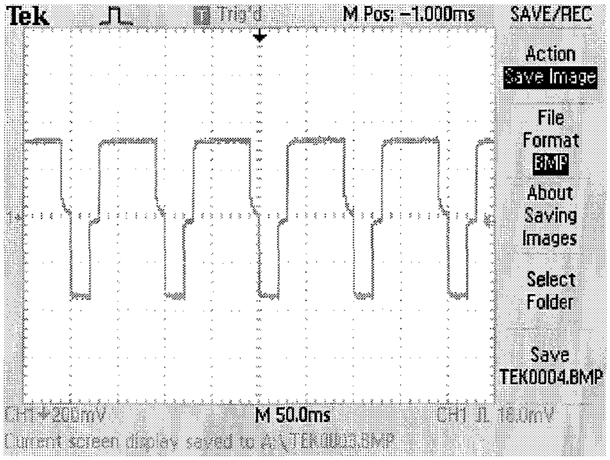


Figure 6: Here is an example of the waveform as the sensor wire was pinched. It can be seen that the waveform oscillated between noisy and clipped forward pulse.

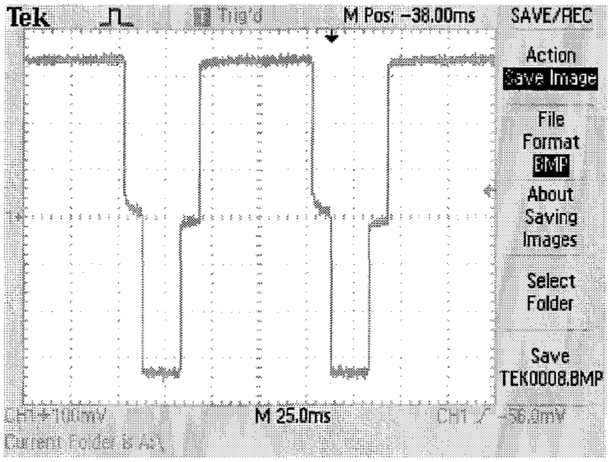
The unit was pulled and replaced (with S/N 0934801). This resolved the problem for us from a production stand point but the sensor problem still remains for one of the units. This also indicated that the slicing of the sensor wire was not the cause of the noise.



Current screen display saved to A:\TEK0003.BMP

Figure 7: 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.



Current Folder is A:\

Figure 8: The 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.

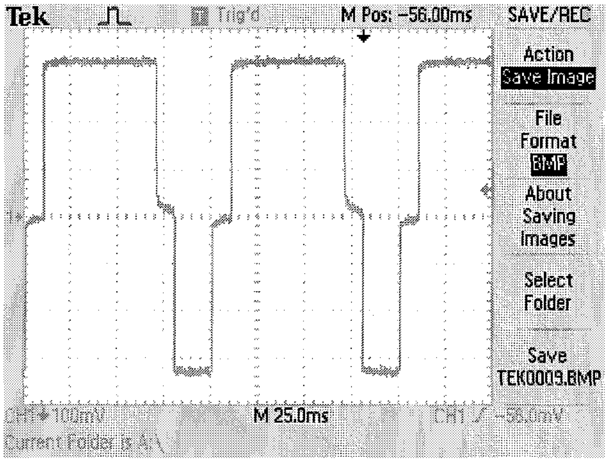


Figure 9: The programmed waveform from unit 4 from the plating surface.

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

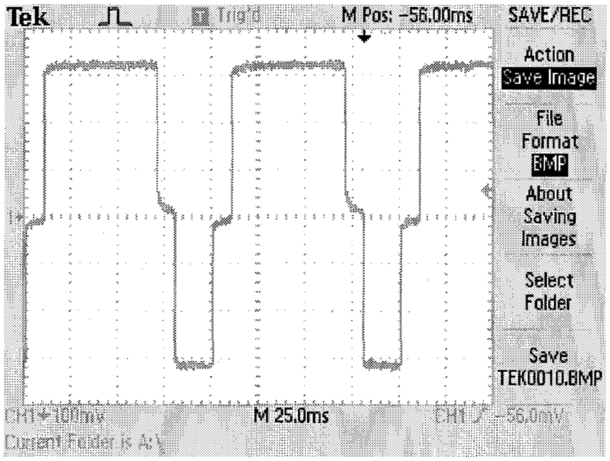


Figure 10: The waveform from unit 6 from the plating surface.

Unit 2 (S/N 0934804) was the last unit brought up. The start up was uneventful and operated within parameters.

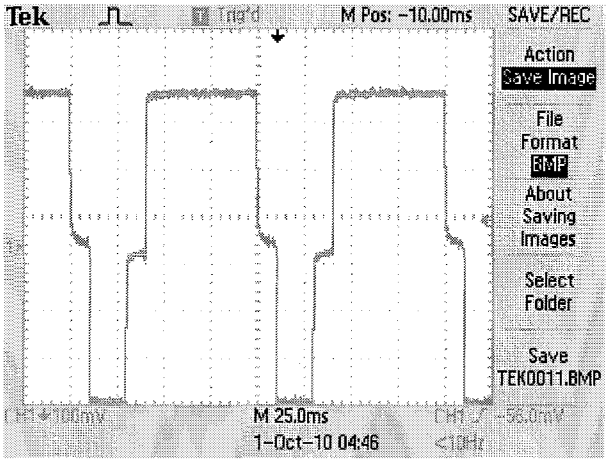


Figure 11: The above is the waveform for Unit 2 as measured from the plating surface of the mandrel.

IV. Discussion

The main problem encountered was with the feedback sensor 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 indicates 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. We thought this was an innovative method 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 to a limited degree by its physical orientation which may impact other parameters particularly at high waveform frequencies.

In conclusion, the units that were received by PNNL in July have performed satisfactorily and have short term durability. The long term performance and durability still needs to be determined as the unit in service for the Majorana prototype bath did demonstrate an issue with the feedback sensor system.