Contract No:

This document was prepared in conjunction with work accomplished under Contract No. 89303321CEM000080 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

Savannah River National Laboratory®

A U.S. DEPARTMENT OF ENERGY NATIONAL LABORATORY • SAVANNAH RIVER SITE • AIKEN, SC

Alternative All-Metal Scroll Pump Design

L. M. Angelette

R. G. Belliveau

September 15, 2021

SRNL-STI-2021-00463, Revision 0

SRNL.DOE.GOV

DISCLAIMER

Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2021-00463
Revision 0

Keywords: *pump, tritium, tritium processing*

Retention: *Permanent*

Alternative All-Metal Scroll Pump Design

L. M. Angelette
R. G. Belliveau

September 15, 2021

Savannah River National Laboratory is operated by Battelle Savannah River Alliance for the U.S. Department of Energy under Contract No. 89303321CEM000080.

REVIEWS AND APPROVALS

AUTHORS:

____

L. M. Angelette, Hydrogen Isotope Process Science Group Date

____

R. G. Belliveau, Hydrogen Isotope Process Science Group Date

TECHNICAL REVIEW:

____

J. E. Klein, Hydrogen Isotope Process Science Group Date

APPROVAL:

____

J. Cortes-Concepcion, Manager Date
Defense Programs Technology

Signatures on file

PREFACE OR ACKNOWLEDGEMENTS

The author gratefully acknowledges Anita Poore and James Klein for their support of this work. The author also would like to thank Kipplin Neikirk and the personnel at the High Pressure Lab for their help assisting with the SRNL pump functional testing rig, and John Wilson and Nathan Nicolas at Air Squared for providing the plated aluminum samples to analyze. The author appreciates NNSA NA-231 for funding this development.

EXECUTIVE SUMMARY

In tritium process systems, vacuum pumps are typically used to evacuate volumes and piping, as well as transfer gas to other parts of the process. This was done using the combination of an all-metal scroll pump with a metal bellows backing pump. The all-metal scroll pump, manufactured by Normetex, has been unavailable since 2012, and efforts continue to find a suitable replacement. The main obstacle is finding a pump that has no oils or polymer components, which degrade when exposed to tritium and introduce corrosive and/or hazardous impurities into the process.

There have been development efforts to design an all-metal scroll pump design alternative to the Normetex style. The first generation was fabricated out of machined stainless steel, but the thermal management and increased weight were not ideal. To mitigate the weight, an aluminum version was designed. The tritium holdup on untreated aluminum surfaces can be greater than stainless steel, so the wetted aluminum surfaces were electroless plated in nickel and then electroplated in gold. The development effort outlined in this report details the design of an improved version of the stainless steel version.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF ABBREVIATIONS

Ag	Silver
Al	Aluminum
ASTM	American Society for Testing and Materials
Au	Gold
cc	Cubic centimeters (volume)
CFM	Cubic feet per minute (volumetric flow)
EDS	Electron Dispersion X-Ray Spectroscopy
ISO	International Organization for Standardization
Met-Bel	Metal Bellows pump
Ni	Nickel
NTU	Nephelometric Turbidity Unit
s	Second (time)
scc	Standard cubic centimeters (volume)
SEM	Scanning Electron Microscope
SLPM	Standard liters per minute (flow)
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
std	Standard (760 torr and 0°C reference condition)
VAC	Volt AC (power)
VCR	Vacuum coupling radiation (fitting)

1.0 Introduction

The Normetex® Model 15 all-metal scroll pump, backed by a Senior Aerospace Metal Bellows pump, has been the standard pump used worldwide as the standard pump for tritium processing. However, in 2012, Normetex® halted production of the scroll pumps. Savannah River Site began searching for an alternative, which culminated in working with Air Squared to develop an all-metal scroll pump. There have been three generations of all-metal scroll pumps developed by Air Squared, all fabricated from machining blocks of stainless steel. One limitation of the stainless steel designs by Air Squared has been heat dissipation and weight due to the stainless steel.

In 2019, it was proposed by the Savannah River National Laboratory to fabricate a new design by Air Squared using from aluminum, as aluminum would decrease the weight while increasing the heat transfer from the heat of compression generated by the gas as it is compressed through the scrolls. However, aluminum has been shown to retain tritium in the surface due to the high level of hydroxyl groups bonded to the naturally oxidized surface.[1] In order to prevent a high activity of tritium on the surface of the aluminum, multiple tritium barrier coating options were evaluated and analyzed. The all-metal aluminum scroll pump, with the tritium barrier coating, was tested to evaluate the performance and compared to the original Normetex® pump curve.[2] This report details the results of the tritium barrier evaluation and pump performance testing.

2.0 Experimental Procedure

2.1 Tritium barrier coatings

Aluminum samples were prepared by Air Squared to test for durability testing of multiple chemical resistance coatings. The samples, fabricated from aluminum 6061, were supplied in a heat-fin style layout, as seen in Figure 2-1, and the fins were cut apart to allow manipulation and sectioning of the fins. The samples, shown as the cut fin sections in Figure 2-2, consisted of bare machined aluminum (Al), anodized Al, electroless nickel (Ni) coated Al, electroless Ni with electroplated silver (Ag) on top, and electroless Ni with electroplated gold (Au) on top, respectively. Also shown in Figure 2-2 is the small square coupons cut from one of the larger fins to fit on the Scanning Electron Microscope (SEM) stage. The small coupon was first cleaned with methanol and allowed to dry, then analyzed via SEM Electron Dispersive X-Ray Spectroscopy (EDS) for a qualitative surface elemental analysis. The SEM-EDS was performed using a Hitachi SU8200 Series Ultimate Cold Field Emission SEM. The small coupons were then loaded into a quartz sample tube with approximately 0.3 mL of quartz wool packing between samples. The sample tube was then loaded into a Micromeritics AutoChem II 2920 for hydrogen atmosphere thermal cycling. The hydrogen gas used was NexAir Research Grade hydrogen (99.9995%). The samples were loaded with 700 torr hydrogen and the temperature was cycled ten times between 40°C and ambient 21°C, with a 1 hour hold at each temperature. The small coupons were then analyzed via SEM-EDS a second time to determine if any chemical or physical differences could be observed.

2.2 Pump design specifications

The all-metal aluminum scroll pump fabrication was specified for use in tritium processing. The pump was detailed at a maximum flow rate of 8 cfm when backed by a Senior Aerospace Metal Bellows MB-601 pump with the heads piped in series discharging to 1 atmosphere, obtain an ultimate vacuum pressure of 0.01 torr when backed by an MB-601 pump, and maintain a helium leak tightness less than $1x10^{-7}$ standard cubic centimeters per second (scc/s) helium before and after testing. The inlet/outlet connections are ¾" Cajon vacuum coupling radiation (VCR) fittings. A forced-air cooling fan provides heat dissipation from the gas heat of compression. The pressure rating of the bellows was set to withstand ultimate vacuum to 30 psid without damaging the bellows, and up to 160 psig without breaching the pressure boundary. The pump was also specified to fit inside a 19.25" wide x 19.25" high x 25" long space envelope. The motor was specified as ½ horsepower at 60 Hz with a 3-phase 480 VAC power supply. Lifting lugs were included such that the lugs were to support at least 125% the weight of the pump.

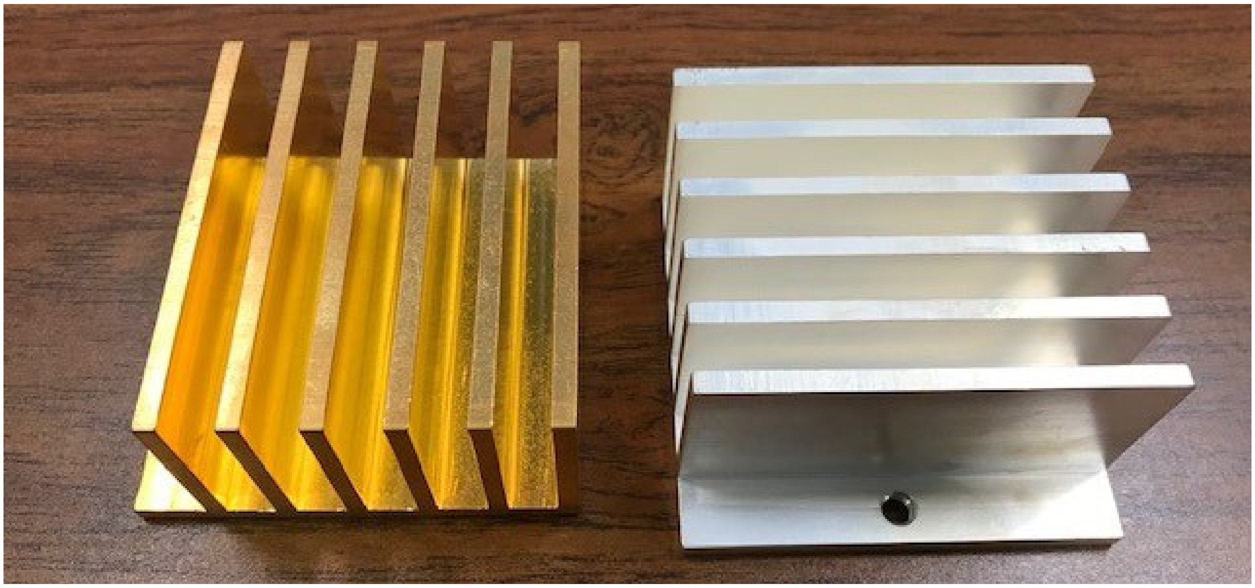

Figure 2-1: As-received plated aluminum samples showing the heat-fin style design. Left: Au plated sample. Right: Bare Al.

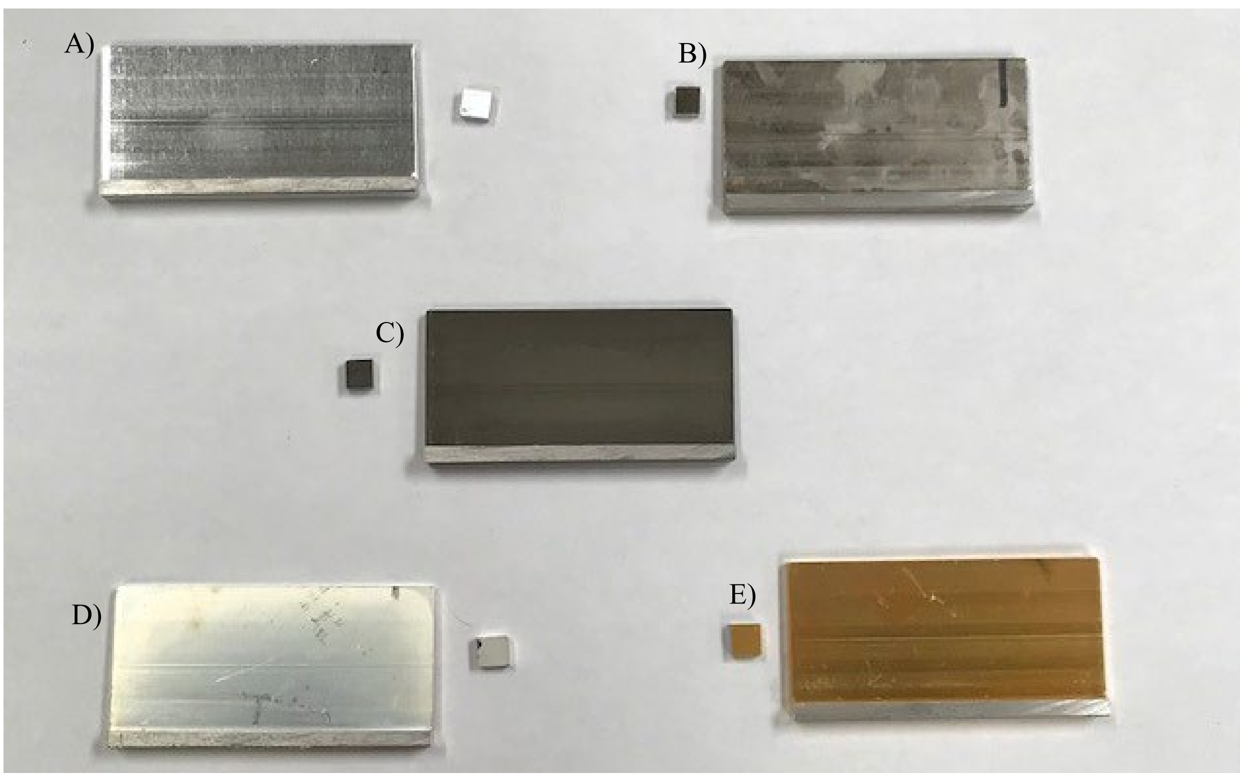

Figure 2-2: Sectioned fins with SEM stage sample coupons. A) Starting Al. B) Electroless Ni. C) Anodized Al. D) Electroless Ni with electroplated Ag. E) Electroless Ni with electroplated Au.

2.3 Pump Testing Plan

The aluminum scroll pump was tested to evaluate the performance of the pump at Air Squared and at SRNL to verify performance upon receipt of the pump. The testing at Air Squared consisted of: cleanliness test, initial helium leak test, lifting lug proof test, flow and compression ratio curve test, seventy-two (72) hour operating test, vibration test, proof pressure test, post-performance helium leak test, post-performance internal inspection, and reassembly performance verification test. The testing performed at SRNL consisted of a receipt helium leak test and flow/compression ratio curve test.

2.3.1 Initial Helium Leak Test

A helium leak test using the hood method per American Society for Testing and Materials (ASTM) E1603 was performed on the pump prior to the pump performance testing. The pump was connected to a Pfeiffer Adixen ASM 340 Helium Leak Detector with an internal 7.93×10^{-8} scc/s built-in leak. The pump was placed into a two-ply plastic bag and taped closed. A small hole was cut in the bag, a helium gas line was inserted, and the hole taped shut around the helium gas line. Ultra-high purity (99.99%) helium was introduced into the bag for three minutes such that the bag was noticeably inflated. The helium leak detector was started, the internal calibration leak was measured, the pump was pulled down to below 0.01 Torr, and the helium leak rate into the pump internals was measured for 10 minutes. The maximum permitted leak rate was set at 1×10^{-7} std cc/s.

2.3.2 Lifting Lug Test

The pump was connected to a chain hoist system via the two lifting anchors on top of the pump. The pump was then lifted off the table and two nominally 25 pound weights were added to the pump. The pump and additional weight was suspended for 10 minutes. The pump was then lowered and the weights removed. The two lifting lugs were removed, visually inspected for damage, the lugs cleaned with SKC-S Aerosol, sprayed with SKD-S2 Aerosol dye, and then sprayed with SKL-SP2 Aerosol developer to detect any stress cracks.

2.3.3 Compression Ratio Test

The compression ratio of the aluminum scroll pump was measured over a span of pressures. The system was evacuated down to 5 torr using an Air Squared V16H030A-AC scroll pump. Nitrogen was added to bring the system up to 50 torr, then vacuumed out again. The inlet to the aluminum pump was then blocked, and nitrogen was added to the pump outlet to stepwise increase the outlet pressure. The inlet pressure was measured by a Pfeiffer Vacuum PKR 361 Pirani-Cold Cathode vacuum gauge (PT01) and an MKS 100 torr Baratron Model 722B capacitance monometer (PT02), where the discharge pressure was measured by an MKS 1000 torr Baratron Model 722B capacitance monometer (PT03). For each step increase, the inlet pressure was measured via PT01 and the pressure was stepped up after the inlet pressure was stable for 2 minutes. The discharge pressure was measured with PT03. The pressure was increased stepwise from 10 torr up to 750 torr in the outlet.

2.3.4 Flow Test

The system schematic used for the flow test is shown in Figure 2-3. Immediately following the compression ratio test, the aluminum scroll pump was tested for flow rates as a function of inlet pressure. The aluminum pump was evacuated using the evacuation pump. The outlet was then repressurized with N_2 to 30 torr in the discharge. The 30 torr discharge is equivalent to that of an MB-601 backing the aluminum scroll pump. The inlet to the aluminum scroll pump was initially blocked by closing $V_{Isolation}$ and $V_{Isolation}$ was slowly opened until flow rates of interest were reached. The flow, in standard liters per minute, was measured using an MKS Instruments GM50A thermal mass flow controller. The flow rate was allowed to equilibrate for 2 minutes before the flow rate was recorded and the flow rate increased. For each flow rate measured, the inlet pressure (PT01) for the aluminum scroll pump was recorded. The flow rate of the pump, Q, was converted into cubic feet per minute (CFM) using Equation 1.

$$Q = \frac{Q_O \times P_O \times T}{P \times T_O} \times \frac{1\ SCFM}{28.31685\ SLPM} \qquad \text{Equation 1}$$

where Q_o is the standard flow rate in standard liters per minute (SLPM), P_o is the flow meter standard pressure (760 torr), P is the inlet pressure, T is the pump temperature, and T_o is the flow meter standard temperature (298.15 K).

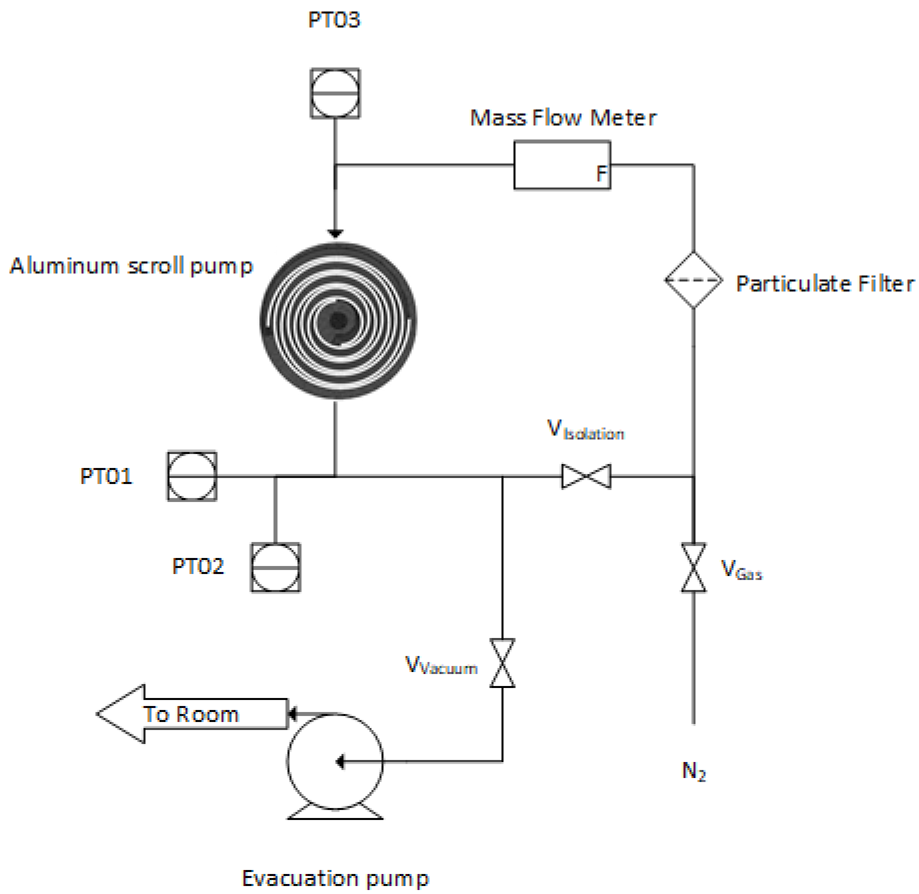

Figure 2-3: Schematic of aluminum scroll pump flow test at Air Squared.

2.3.5 Pressure Test

The pump was tested for maximum operating pressure without damaging the bellows or process fittings. The pressure rating for the process fittings were determined using bolt tensile stress areas from International Organization for Standardization (ISO) 898. Using the equations in ISO 965, the maximum allowable pressure could be determined, and the maximum working pressure for the fittings is 1/3 the maximum allowable pressure. The maximum pressure rating of the bellows was determined by burst testing a bellows, and the maximum working pressure was determined by slowly increasing the pressure inside the bellows until the bellows began to permanently deform.

2.3.6 Maintenance Inspection

Following the pressure test, the pump was placed into a clean hood. The aluminum casing was removed, the bellows unscrewed at one end, and the rotating scroll removed. The internals of the rotating and fixed scrolls were visually inspected for any indication of wear or particulates from the coating. The pump was then reassembled for final testing.

2.3.7 Final Helium Leak Tests

Two helium leak tests were performed following the maintenance inspection and reassembly. A helium in-leakage using the hood method was performed to measure for leaks through the sealing surfaces. Then the plastic bag was removed and the freed helium was allowed to diffuse over two hours. The helium leak detector was then switched to the sniffer probe for a helium out-leakage test. The pump was pressurized with 15 psig of helium and the sniffer probe was swept over all connection points for the pump including the bellows seals, bearings, and VCR fittings.

3.0 Results and Discussion

3.1 Tritium barrier coatings

The four aluminum samples with different coatings, along with the bare Al, were cut to expose an outside corner. During the cutting, it was noticed that the electroless nickel with electroplated silver had small flakes coming off the coating, with one area shown in Figure 3-1. The other four samples did not experience any damage to any coating present on the samples. As the samples were imaged via SEM, the silver plated sample coupon lost more of the electroplated silver, with portions of the silver layer adhering to the carbon tape on the SEM stage. For the gold plated sample coupon, it should be noted that very small areas of the underlying electroless nickel was observable during the SEM-EDS elemental mapping, shown in Figure 3-2.

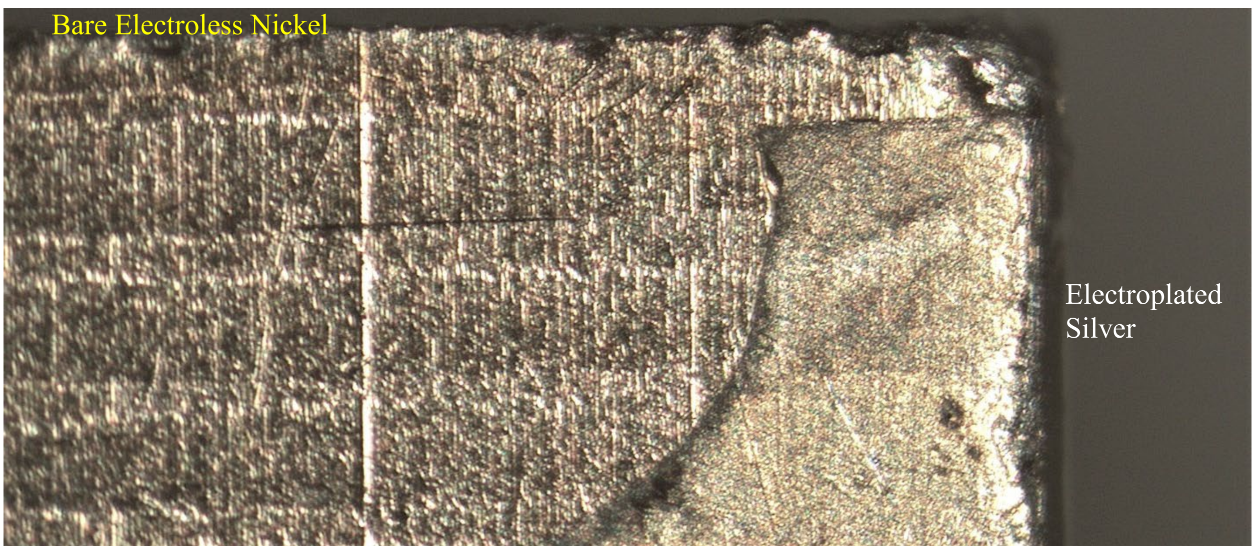

Figure 3-1: Optical microscopy image of the edge of the electroless nickel with electroplated silver SEM coupon. Noted are the areas where the silver is still present and where the silver has flaked off, exposing the electroless nickel.

The five SEM sample coupons were then loaded into a sample tube for a Micromeritics AutoChem II Chemisorption Analyzer. During the packing, thin layers of quartz wool were packed above and below each sample coupon to prevent the coupons from rubbing against another. The samples were then thermally cycled 10 times in a 700 torr hydrogen atmosphere, cycling between 21°C and 40°C, with a one hour dwell time at each temperature. 40°C was chosen as the upper limit because this is the highest temperature recorded in the operation of the aluminum scroll pump. Once the cycling was complete, the sample coupons were unloaded from the sample tube and placed onto an SEM stage for post-cycling analysis. The bare Al, anodized Al, Al with electroless Ni, and electroless Ni with electroplated gold were shown to have no detectable differences from the pre-cycling analysis. The electroless Ni with electroplated silver was observed to have lost more silver area around the cut edges.

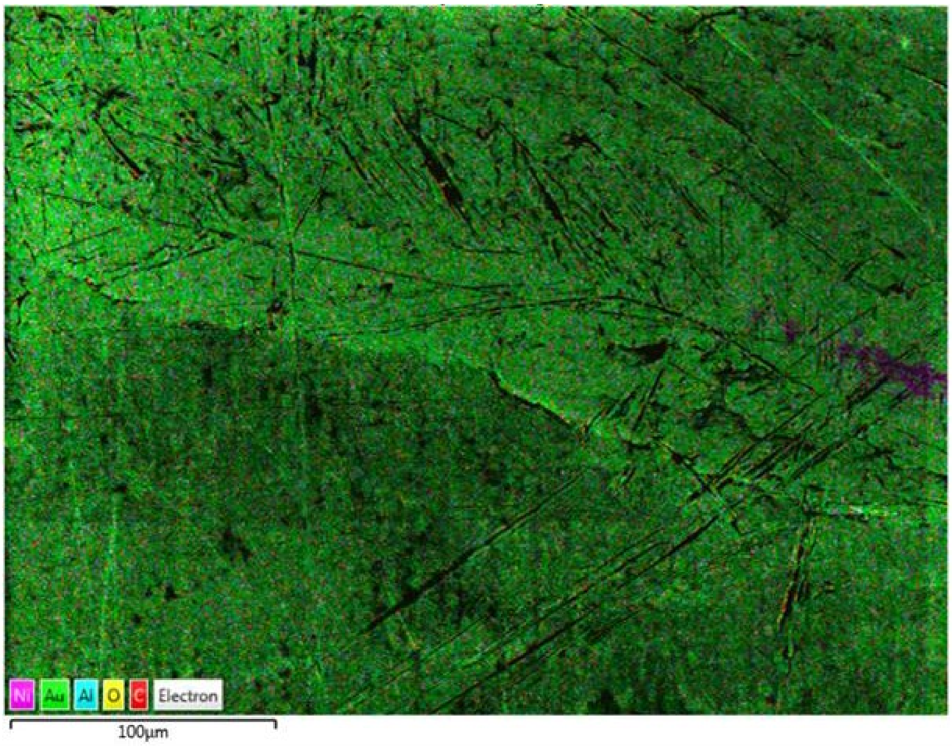

Figure 3-2: SEM-EDS elemental mapping of the electroplated gold with traces of the underlying nickel detectable. SEM-EDS mapping was performed at 347x magnification, 15 keV beam voltage, and a 15.2 mm working distance.

3.2 Pump Testing

3.2.1 Initial Helium Leak Test

The pump and process fittings were sealed inside a two-ply plastic bag before helium introduction. Included in this was a single KF-25 to ¾" VCR adapter where the leak detector connected to the pump. Prior to the introducing helium into the bag, a background measurement was collected. The background helium leak rate was measured to be 4.7×10^{-11} scc/s. Once helium was introduced to the bag, the helium leak rate after 10 minutes of measurement was 1.2×10^{-10} scc/s.

3.2.2 Lifting Lug Test

The pump was positioned underneath an electric lift and attached using hooks to the top two lifting lugs. Two 25 pound weights were added to the top of the pump, which added approximately 38% weight to the pump. This was above the minimum additional 25% added weight. The pump was lifted off of the supporting surface, suspended off the ground for 10 minutes, and then lowered. The additional weights were removed and the lifting lugs removed for analysis. The lifting lugs, following being cleaned, dyed, and developed (as shown in Figure 3-3, Figure 3-4, and Figure 3-5) showed no sharp red lines, indicating that there were no stress cracks formed from the lifting test.

Figure 3-3: Lifting lugs following post-lifting test cleaning.

Figure 3-4: Lifting lugs with stress crack dye indicator applied.

Figure 3-5: Lifting lugs with stress crack indication developer applied.

3.2.3 Compression Ratio Test

The inlet pressure of the pump was measured for outlet pressures ranging from 10 torr to 750 torr, shown in Figure 3-6. The pump has a very linear trend in the inlet vs discharge pressure in the 10 torr to 100 torr range. Above 100 torr in the discharge, the pump is less efficient in its ability to pump. As the discharge pressure increases, the inlet pressure increases closer to the discharge pressure. At a discharge of 750 torr, the inlet pressure is approximately equal to the discharge pressure. This follows a similar trend seen in the original Normetex pumps, except the Normetex had a linear semi-log pressure increase until 300 torr and then started to curve to end at a suction pressure of 400 torr with a 750 torr discharge pressure.[3] The compression ratio, shown in Figure 3-7, is calculated by dividing the discharge pressure by the inlet pressure.

3.2.4 Flow Test

The pump and test system were first evacuated. Then the pump isolation valve was closed and nitrogen was introduced into the discharge of the pump until the discharge pressure registered approximately 30 torr. The pump isolation valve was slowly opened, allowing for the gas flow to be gradually increased, shown in Figure 3-8. The pump was able to reach a gas flow of 2.35 CFM at an inlet pressure of 0.21 torr and a maximum of 7.71 CFM at 5.80 torr inlet. This also follows the trend seen by the original Normetex pump flow curve with simulated backing pump conditions.[3]

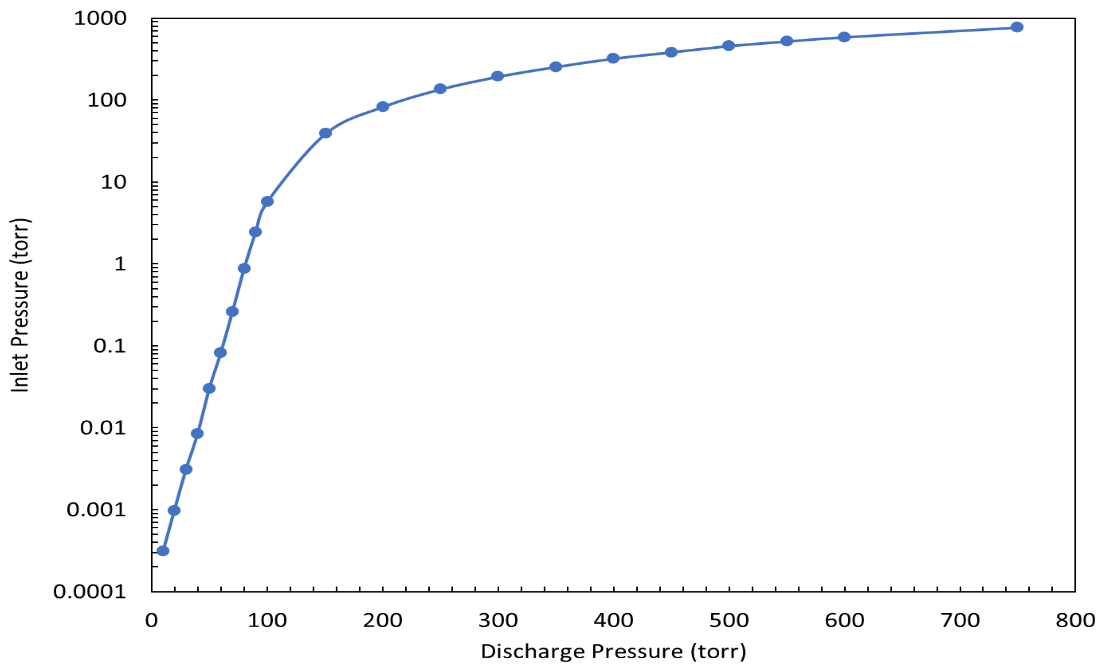

Figure 3-6: Inlet pressure over a range of discharge pressures.

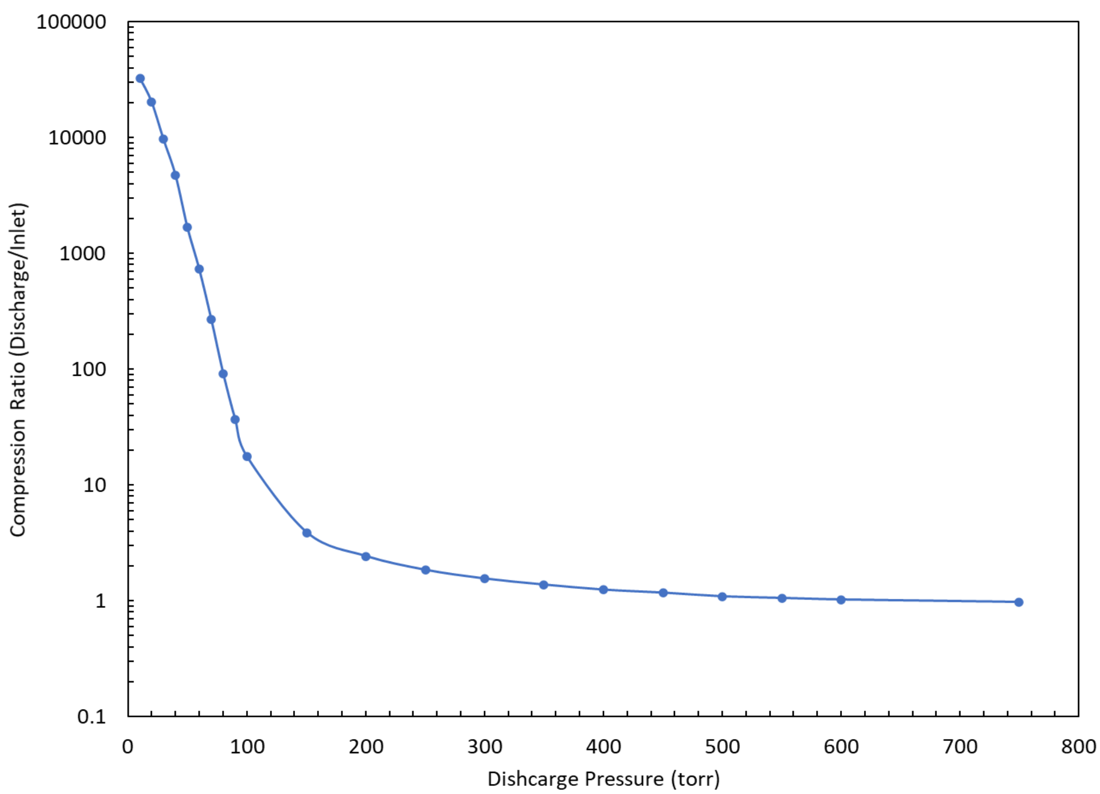

Figure 3-7: Compression ratio as a function of discharge pressure.

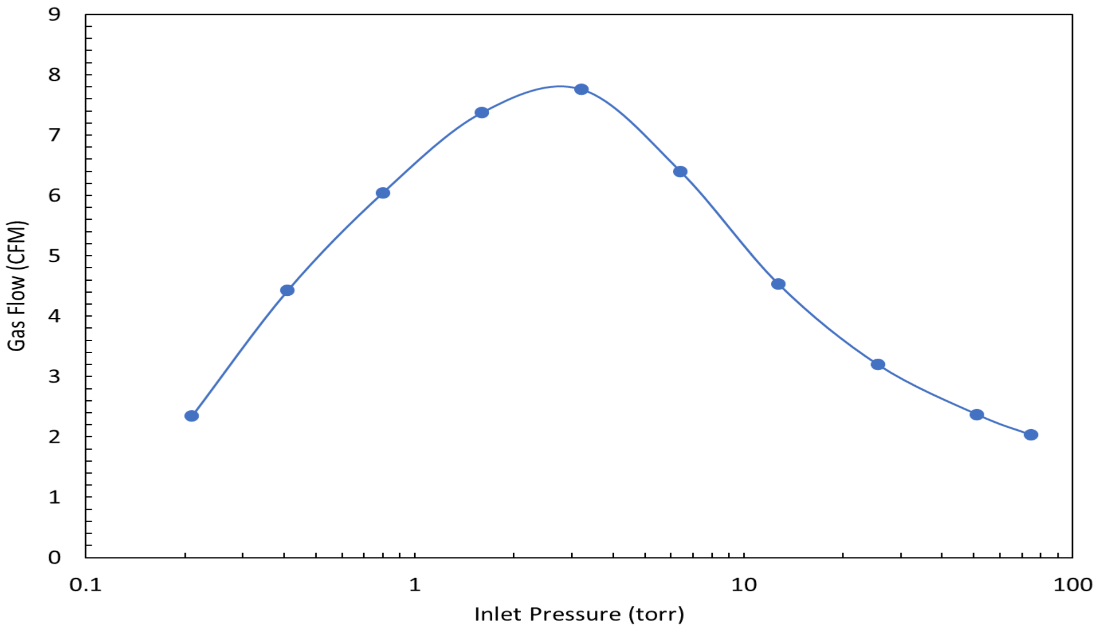

Figure 3-8: Gas flow over a range of inlet pressures.

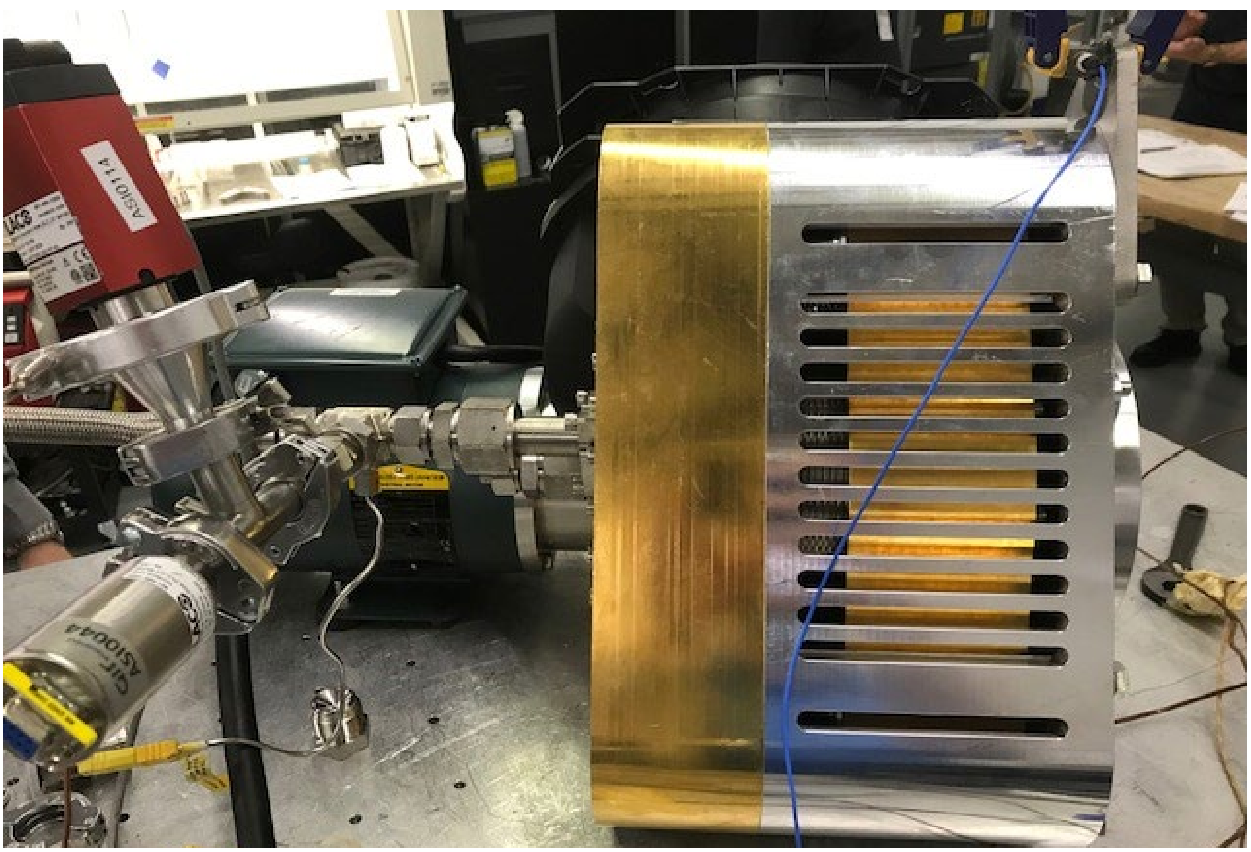

Figure 3-9: Exterior of the all-metal aluminum scroll pump. The non-gold region is the aluminum rotating equipment shield.

3.2.5 Maintenance Inspection

The pump, shown in Figure 3-9, was transferred to a clean hood for disassembly. The pump was disassembled starting at the face opposite the pump and process fittings, shown in Figure 3-10. The shroud bolts were removed, allowing for the main aluminum heat shield to be removed, shown in Figure 3-11 and Figure 3-12. The M60 bolts were then removed, disconnecting the rotating scroll from the containment bellows, shown in Figure 3-13. The bellows was left attached to the fixed scroll to minimize the number of seals that needed to be replaced, shown in Figure 3-14. No wear was observable, and no metal shavings were seen in the maintenance inspection. The pump was then reassembled for post-maintenance testing.

3.2.6 Final Helium Leak Test

The pump, reassembled following the maintenance inspection, was placed into a two-ply plastic bag. The pump inlet was plugged and the helium leak detector was connected to the outlet of the pump. The bag was taped closed, a helium line inserted, and the bag was inflated. The initial helium detector background taken before the test was measured at 8.5×10^{-11} scc/s of He. During the helium in-leakage hood test, the helium leak detector recorded a leak rate of 5.9×10^{-8} scc/s of He. After the bag was opened to release the helium, the helium was allowed to disperse over two hours. Once the helium background was at an acceptable rate of 1.7×10^{-6} scc/s of He using the sniffer probe, the pump was pressurized with 15 psig of helium and the sniffer probe was used to probe all boundary connection points. The highest helium leak rate detected with the sniffer probe was 4.8×10^{-6} scc/s of He. It should be noted that points towards the bottom of the pump could not be measured due to helium off-gassing from the rubber feet of the pump.

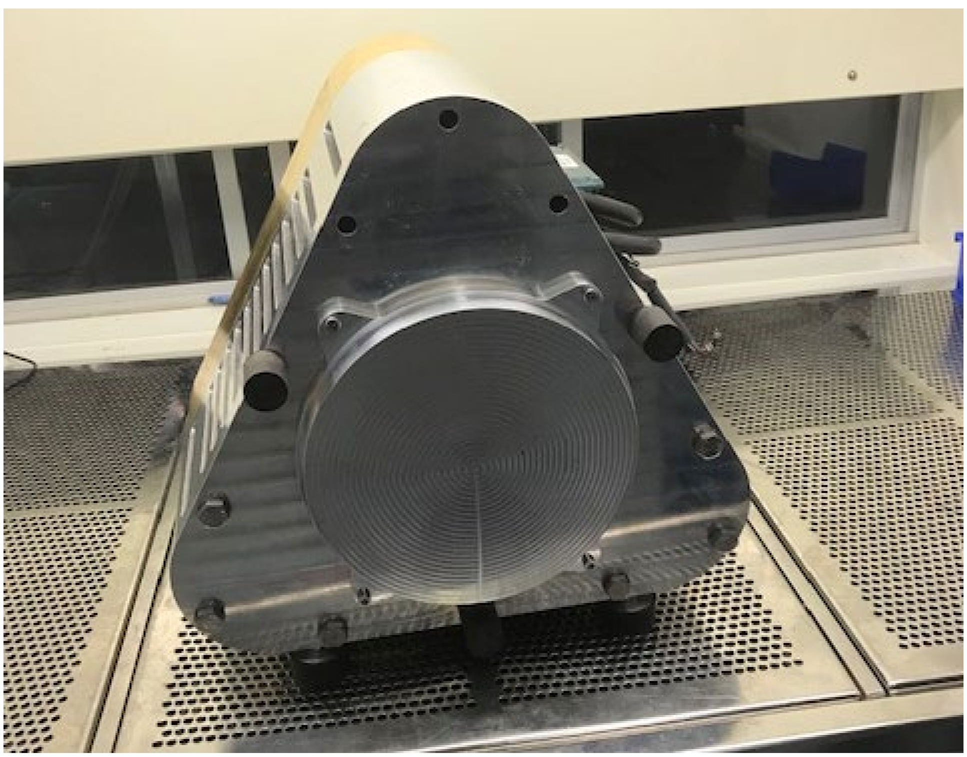

Figure 3-10: Rear of pump with bolts to remove the rotating equipment safety shroud.

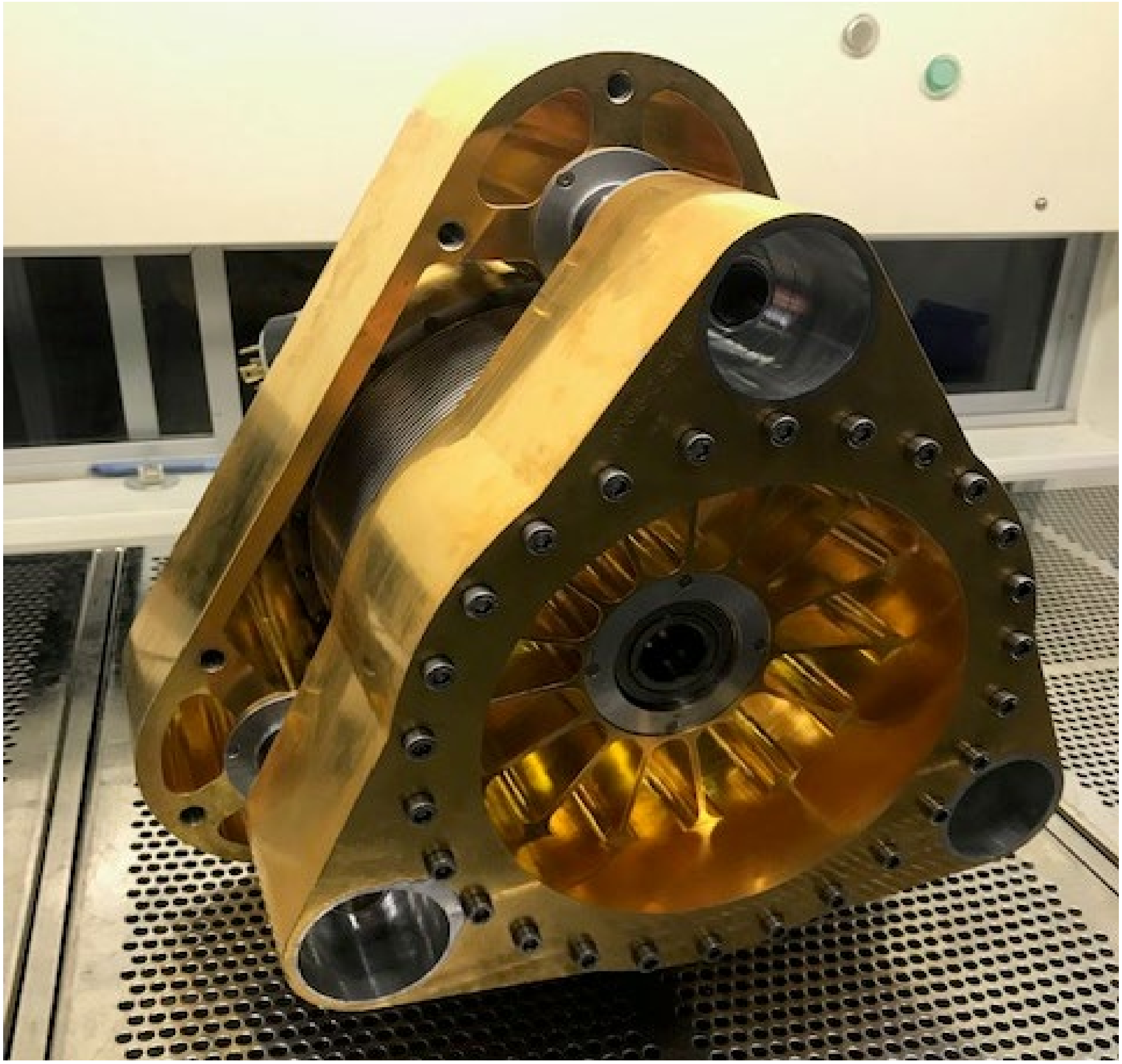

Figure 3-11: Pump with the rotating equipment safety shroud removed.

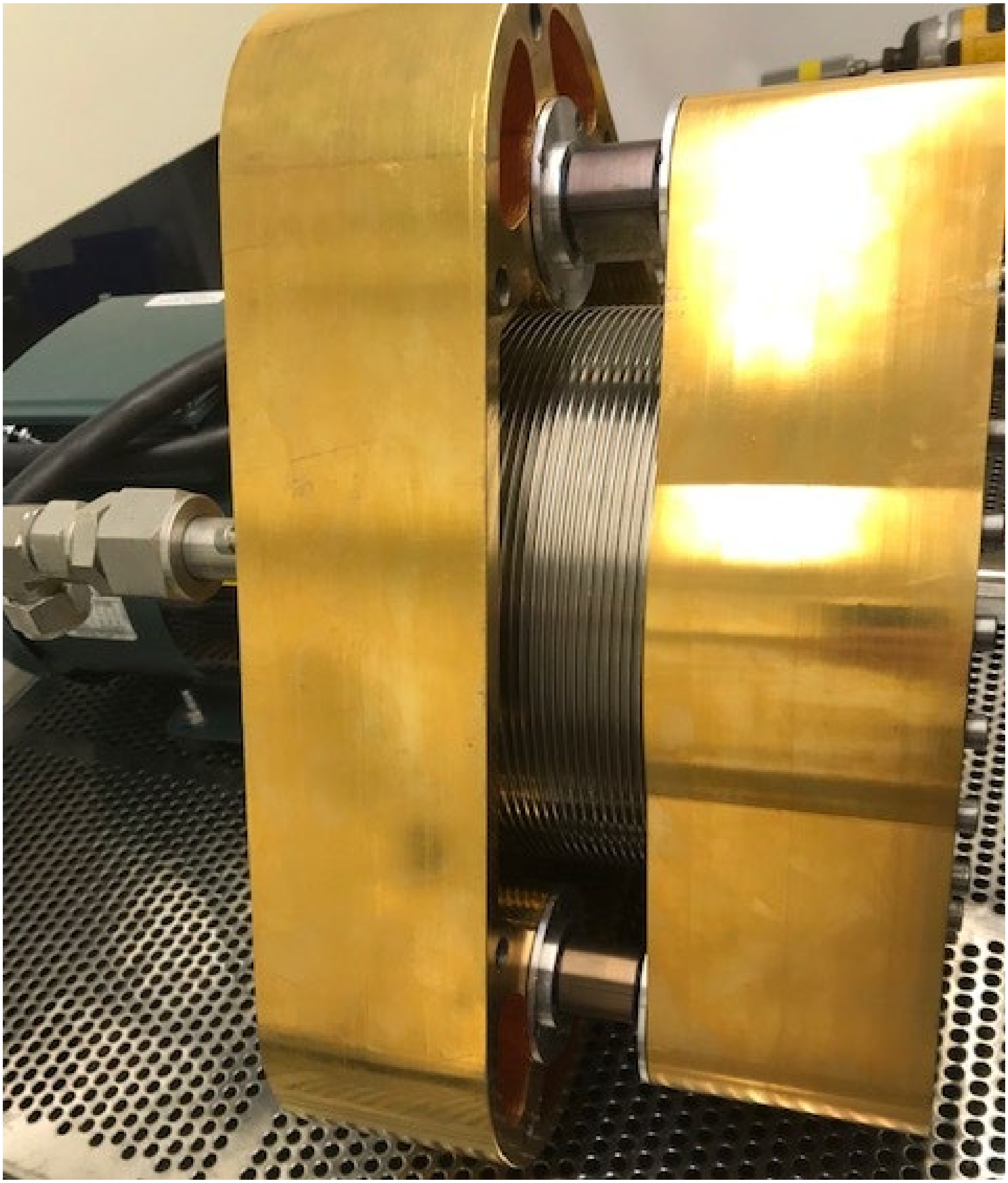

Figure 3-12: Side view of the pump with the rotating equipment safety shroud removed.

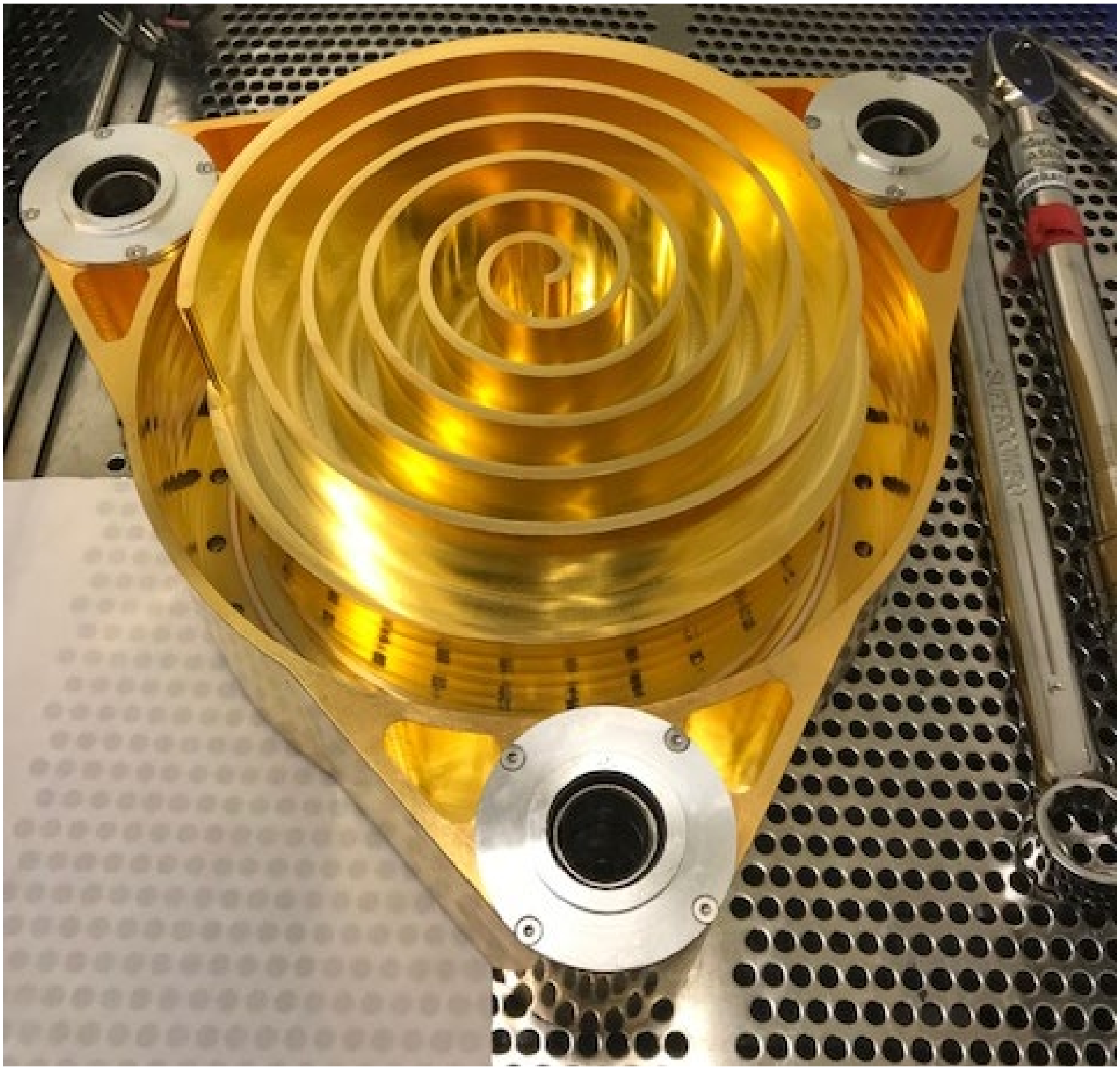

Figure 3-13: Rotating scroll after it was removed from the containment bellows.

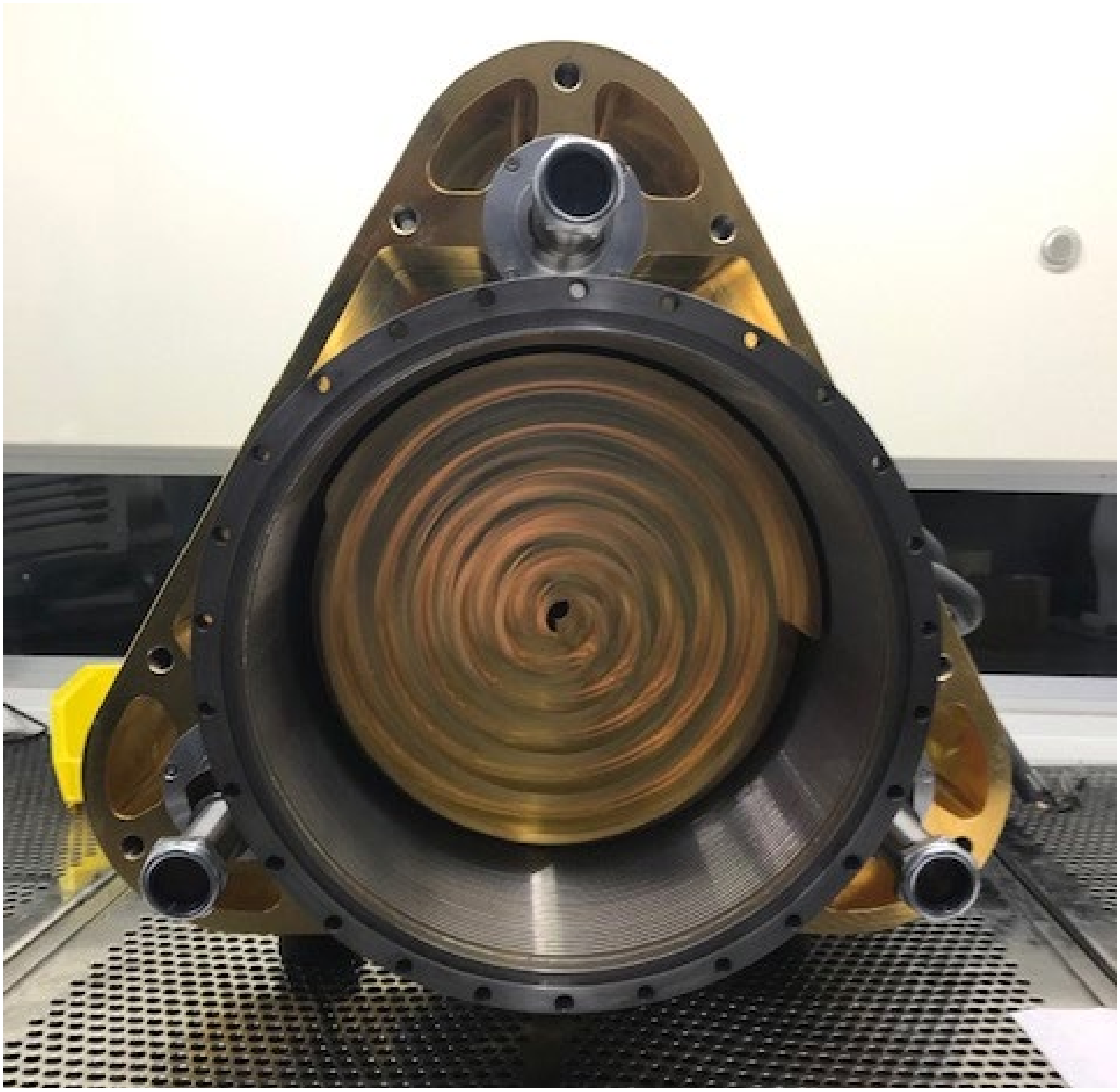

Figure 3-14: Internal fixed scroll with containment bellows.

4.0 Conclusions

An all-metal scroll pump was successfully fabricated using the alternative base metal of aluminum. Multiple coatings were considered for the aluminum to reduce the surface tritium contamination. Electroless nickel followed by electroplating gold was chosen to plate the rotating and fixed scrolls. The pump was tested for helium leak tightness, compression ratio, and flow rate as a function of inlet pressure. The pump successfully passed all requirements for the testing. The pump did not show any evidence of wear or metal shavings from the coating during the maintenance inspection. The pump also passed the reassembly helium leak tests.

5.0 Recommendations, Path Forward or Future Work

The pump will be tested for ultimate vacuum, compression ratio, and flow rates for various pure gases, included hydrogen, deuterium, helium, argon, and krypton. It is planned that these will be performed with a Metal Bellows MB-601 backing pump. A system with a larger internal volume will be used for the tests to prevent the need for gas to be added to the discharge to reach higher flow rates, keeping the system at constant discharge pressure.

6.0 References

1. Nishikawa, M., et al. "Tritium trapping capacity on metal surface." *J. Nuc. Mat.* **277**, (2000) 99.
2. Sharpe, C. L. *Tritium Gas Transfer Pump Development*. DP-MS-85-108, Savannah River Plant, Aiken, SC: E. I. du Pont de Nemours and Company, 1985.
3. Sharpe, C. L. "Tritium gas transfer pump development." 1985. Web, https://www.osti.gov/biblio/6079916.