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PROGRESS REPORT

March-May 1984

**Metal Finishing and Vacuum Processes Groups
Materials Fabrication Division**

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L. A. JONES

July 11, 1984

Lawrence
Livermore
National
Laboratory

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SUBJECT: Progress Report - March-May 1984 - Metal Finishing and
Vacuum Processes Groups - Materials Fabrication Division

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WEAPONS PROGRAM

W-84 Aluminum Coatings for A. Copeland

In this report period we successfully coated two B0A parts and one mock full assembly coating. Two of these three coated parts are now waiting to be tested in the environmental test chambers in Bldg. 131. Early indications look very promising. One difference between these coatings and coatings done in the past on full mock assemblies is that we used two 10 kilowatt magnetron sources rather than six 1 kilowatt sources. Another difference is that the substrate is rotated in the center of the chamber and the sources are rotated relative to the substrate. The coating uniformity on this particular assembly was very good, $\pm 10\%$, and the overall thickness was 0.0762 mm.

Tom Collie and Chuck Egebert of Y-12 visited Tom Beat to discuss the status on this project at Y-12 as well as at Livermore. The Y-12 people have decided to set up a corrosion test of their own.

NUCLEAR TEST PROGRAM

Pinex Pinhole Fabrication and Development for R. E. Lear/C. H. Adams/A. D. Edge

For NORMANNA a research and development program was designed to produce integral units 4-1/2 inch long with a cylindrical section of 2, 3, and 4 mils diameter by 1-1/2 inches with a 1-1/2 long tapered section on each end. This has not been completed, and may or may not be possible. The major problem is that of removal of the mandrel. We did successfully dissolve the mandrel with the 4 mil central section, however, there were 3 cross-holes drilled through the central section. In addition to our mandrel dissolution problems difficulties were encountered in machining the 4-1/2 inch long, 1/8 inch OD part to the required tolerances. As stated in the March 1984 report, each phase of manufacture was a development project so we produced and delivered 1-1/2 inch long cylindrical sections with 2, 3, and 4 mil diameter holes as back-up parts.

Pinhole Mandrels for EGMONT for A. Edge

A pinhole mandrel, having a straight center section and tapering up to a larger diameter at the ends, was made by VPL for the E1-Pinex assembly of the EGMONT nuclear physics experiment. The overall length of the mandrel was 4.500". The diameter of the center section measured 8 mil over a length of 1.500" and tapered uniformly to a diameter of 0.024" at both ends.

The mandrel was fabricated by sputter coating copper on a 8 mil O.D. copper tube. A tube, instead of a wire, was used as the substrate to facilitate the later removal of the mandrel by acid leaching. The sputter source was a six inch diameter, ten kilowatt magnetron sputter gun. A fixture was made that rotated the tube and moved a mask between the sputter source and the rotating tube. The rotation preserved the circularity of the tube while the motion of the mask generated the required taper. The fabricated mandrel was delivered to Metal Finishing for gold electroplating and subsequent copper leaching.

Aluminum Coating on Vacuum Barrier for WEXFORD for J. Leppien

To create a special window for the WEXFORD event, a sheet of Mylar had to be coated in a concentric pattern with two different thicknesses of aluminum. The outer ring, of 15.24 cm O.D. x 10.16 cm I.D., required approximately 1000 nm of aluminum. The coating in the inner area had to be only 100 nm thick. The coating process, electron beam evaporation, required that the Mylar substrate be suspended over the evaporation source. To coat the outer ring required masking the inner area. This was accomplished by holding a 10.16 cm diameter disc in place by means of magnets placed on the opposite side of the Mylar. The 1000 nm thickness was deposited first. The mask was then removed and the entire surface was coated with the 100 nm thick deposit. For areal uniformity the substrate assembly was nutated above the evaporation source.

Outgassing Tests of Stainless Steel Velcro for C. Cochran

The requester is considering using Velcro in high vacuum applications as a convenient means of attaching parts. We were given sample strips of Hi-Garde 2000L/40H stainless steel Velcro to test. These were briefly degreased with Freon vapor and then tested under vacuum for 190 hours. Hydrogen and water were the main gas constituents that evolved during the first few hours of the test run. Throughout the remainder of the run the major constituents were CH_3 , CH_4 , N_2 , CO , and CO_2 . Significant amounts of oil fragments were evident throughout the run. The outgassing rate was similar to that of 1.6 mm thick Polyethylene sheet and about 5 times that of 6061-T6 aluminum.

However, making accurate comparisons is complicated by the intricate geometry of the samples. The surface area was calculated as if the Velcro were a solid block having a thickness equivalent to the thickness of the backing plus the height of the loops or hooks. This was done to simplify the calculation of the gas load for a given length of Velcro. The actual surface area is considerably greater and therefore the computed outgassing rate is higher than would be expected for stainless steel. The gas species detected indicate that the outgassing rate would have been improved by a more thorough removal of oil and solvents. For use in clean vacuum environments, we recommend rinsing the as-received Velcro in methyl alcohol or liquid Freon first, then Freon vapor, and finally vacuum baking (off line). We conclude that due to its relatively low outgassing rate, and its resistance to heat (427°C) and corrosive chemicals, stainless steel Velcro can be a useful fixturing aid in a high vacuum environment.

Microchannel Plate Coatings for COTTAGE for M. Eckart

Using electron beam evaporation, coatings of niobium and of gold were consecutively deposited on microchannel plates. The niobium was deposited first - approximately 20 nm thick. Its function was to provide adhesion between the gold coatings and the glass microchannel plates. The gold was subsequently deposited to a thickness of 800 nm. To avoid coating the internal surfaces of the microchannels, the coatings were done at a 15° angle of incidence on the front sides and 45° on the back sides.

Germanium Coatings on Beryllium for COTTAGE for W. Junk

We were asked to deposit 21.5 mg/cm^2 of germanium onto 0.005 cm thick beryllium substrates for the COTTAGE event. The relative thickness of the germanium (in the order of 1-1/2 mils) proved to be a problem for two reasons. First, not enough material could be loaded into the special tantalum crucible required to provide an adequate evaporation rate, and when the system was vented to add source material a thin oxide layer formed and remained as an interface between successive deposits of germanium. Second, it was necessary to have the beryllium hot at the time of deposition of the germanium to obtain adequate adhesion, and the large difference in coefficient of thermal expansion between the two materials induced high stresses upon cooling. As a result of the two problems, the coatings would delaminate. Negotiations with the applications physicist resulted in a compromise whereby only 0.01 mm (in the order of 1/2 mil), but on a larger substrate, was accepted. We anticipate being able to produce the reduced thickness coatings in the near future.

Parylene Substrates for COTTAGE for D. Bowen/W. Junk

We mounted and inspected 13 Parylene foils for use as substrates by a vendor engaged to provide routine coatings for the fabrication of nuclear targets for the COTTAGE event. Ten of the substrates were produced from Parylene films deposited by Union Carbide and three were produced from Parylene deposited by the MFD Plastics Fabrication shop. Of particular significance was the trial use of Dow Corning - VacSeal to glue the foils to their mounting rings. VacSeal's relatively low vapor pressure makes it a better choice than previously used adhesives for the fabrication of nuclear targets which are used in vacuum environments. It was comparable to previously used adhesives in ease of application, and barring any unforeseen problems we plan to use it in other similar cases.

Aluminum Coatings on Fluoroplastics for COTTAGE for D. Nelson

Aluminum coatings were provided on three sides of rectangular substrates, 0.762 mm x 0.762 mm x 25.4 mm, made of several types of fluoroplastic. In accomplishing this task for the COTTAGE event, a unique fixturing approach was used that will be the subject of future reporting.

Cesium Iodide Coatings for COTTAGE for D. Bowen

A continuing need for cesium iodide coatings has been demonstrated by ongoing requests from Nuclear Test Engineering. Prior requests have been met using considerable jury-rigging to permit processing these very hygroscopic coatings. Nuclear Test Engineering has allotted \$16K of expense money to provide a more permanent, safer-to-use enclosure joining two vacuum deposition systems (US-100 and US-101) used previously for this activity. A used glove box was acquired, refurbished, and is being installed to enclose the tops of the two small bell jar systems. For safety, a bell jar hoist was added to one of the systems that did not have a hoist. The resultant assembly will permit transferring, measuring, and packaging cesium iodide coatings without exposure to the ambient atmosphere. Completion of the installation is imminent, and will be used immediately for foils for the COTTAGE event.

NUCLEAR DESIGN PROGRAM

L4 Program for G. Guenterberg

In this report period we built up and fielded successfully another load shot which was fired in Nevada. We learned a great deal about the vacuum aspects of this particular program from the L4 event that we had not learned in previous experiments. We've determined that we have definitely overcome a great number of the problems that we had experienced earlier, and we were able to identify a big problem that we didn't realize existed. The pressure level in the vessel at shot time was very good, an order of magnitude or so below what we've seen in the past. This is attributed to the way that this particular chamber was handled during precleaning and assembly. The problem that we identified in preparing for the L4 event is the fact that the O-rings that we used in the past to seal the experiment's enclosure were extremely dirty. The O-rings were literally impregnated with an estimated total of 10-20 cc of oil. We discovered this when we started baking O-rings in preparation for the downhole events. We had to cycle them many many times through a series of alternating Freon rinses and vacuum bake-outs.

MAGNETIC FUSION PROGRAM

RTNS-II for C. M. Logan/G. A. Harter

The electroforming of targets is on hold since it was recently discovered that the yield strength of the substrate material was below the accepted value. Twenty new targets are being processed through the phase of etching the cooling channels, and they will be ready for electroforming in the near future. During this shutdown we are manufacturing an additional 90° angle electroforming fixture.

We also started a program on use of electrodeposition to produce a dispersion hardened copper that could be used as substrate material for targets. In this work, attempts will be made to codeposit metals such as Cr, Mo, W, Mg and Sn with copper to provide a material that will retain high strength after heating. Because of a heavy workload in electrodeposition, we may look to Battelle Columbus Laboratories to provide help on this project.

RTNS Filament Chuck Braze for L. Scott

We were asked to assemble and braze a filament chuck for RTNS. The assembly consisted of 16 different parts, including two different kinds of (pre-brazed) ceramic-to-metal feedthroughs, various copper pieces, and two stainless steel fittings.

The number of joints, the variety of materials of construction, and the joint geometries encountered, necessitated applying a combination of techniques and devising a technical approach to accommodate the diverse requirements presented. Our basic approach was to conduct step brazes (i.e., sequentially performed brazes using alloys with progressively lower melting temperatures). Copper shims were inserted into some of the joints to reduce excessive joint gaps that would not have been bridged by Cusil, a eutectic alloy. Interim steps required induction brazing (which permits localized heating) to keep from melting the pre-brazed feedthrough joints. Radiant heating was used to provide uniformity in making the final series of braze joints. The job was successfully completed and delivered.

Vacuum Technology Support for Materials Testing for G. Cruz

Outgassing tests of 3M Brand ECP-2200 black (solar absorbing) paint were completed. The test data included outgassing and residual gas analysis as a function of time for painted aluminum substrates in the "as-received" condition, as well as for painted substrates which were vacuum baked.

Contrary to expectation, the results indicate that this type of paint is as vacuum compatible as electroplated black chrome or black electroless nickel coatings. The comparative outgassing data for these coatings is given below:

<u>Description</u>	<u>Outgassing Rate (T-l/s-cm²)</u>
o 3M ECP-2200 Black Paint	
Run #1 -	
+ As-received, after 20 hours	1 x 10 ⁻⁸
+ As-received, after 100 hours	2 x 10 ⁻⁹
Run #2 -	
+ During 24 hour, 100°C Bake	4 x 10 ⁻⁷ (maximum)
+ 76 hours after 24 hour bake (100 hours)	1 x 10 ⁻¹⁰
o Electroplated Black Chrome	
+ As-received, after 20 hours	5 x 10 ⁻⁸
+ As-received, after 100 hours	4 x 10 ⁻⁹
o Black Electroless Nickel	
+ As-received, after 20 hours	3 x 10 ⁻⁸
+ As-received, after 100 hours	4 x 10 ⁻⁹

ATA Vacuum System Maintenance and Troubleshooting for W. Elsholz

We're beginning to address some vacuum related problems on the ATA system at three different levels: "hands-on", education, and ongoing consulting. The "hands-on" approach includes the short-term (2 to 4 month) loan of technician support to help develop good work habits and to help solve the problems which crop up day-to-day. The education category included the suggestion of a topical outline of a short course on vacuum technology and finding an outside consultant to carry out the actual lectures (which are scheduled to begin in July). The ongoing consulting consists of informal regular visits to Site 300 by one or two VPL staff members to provide ongoing assistance and an easy path for technical communications.

ATA Beam Dump Analyzer for D. Milani

Four graphite parts 6.0 inches in diameter and 6.0 inches in length required 25 mil thick electrodeposited copper on the outside diameter and nothing on the ends. The copper provides a more conductive surface to transfer the heat absorbed in the graphite from stopping the ATA beam to a solution cooled heat exchanger. The graphite alone will not transfer the heat rapidly enough and would vaporize from the beam's intensity.

Vacuum System Consulting for H. Galles

The ongoing consultive support which has been provided by the VPL staff over the last year has been recognized and we've been encouraged to extend that support to the engineering and design staff.

Arrangements have already been made with C. Jackson (MFED) to present an informal discussion of the engineering and design requirements for a mobile pumping system which would be used on an ATA beam dump. Because the system needs to operate in an unusual environment, the specification of such a pumping system is not straight forward, and we look forward helping to find a cost effective solution.

MISCELLANEOUSPenetrator Program for Picatinny Arsenal, N.J. for L. Gruss

Development work is being performed for the penetrator program to provide a corrosion barrier for uranium. In this case the coating process has to be compatible with mass production of sheets of uranium measuring approximately 0.6 x 1.2 meters. In this particular project we don't have the temperature constraints that we have had in some earlier coating projects. To date we have developed a matrix experiment containing four runs to identify the precleaning steps and four runs to establish coating parameters. For this application we need to avoid having to do any wet chemistry cleaning, so we are limiting ourselves strictly to cleaning that can be done as part of the vacuum process. Initial tests showed that we can clean uranium in the highly oxidized, as-received state by means of extensive sputter etching. It will take about 30 minutes to clean uranium at a power level of 1 watt/cm² on the substrate. We have also determined that we can reach the maximum substrate temperature of 400°C with just bias power. Progress was recently reviewed at LLNL with Len Gruss from Picatinny.

X-Ray Calibration and Standards Facility for T. Brown

Work continues on the preparation of procedures for the cleaning, welding, testing and handling of components for the ultra-high vacuum system (<10⁻⁹ Torr) required by the XCSF project. We're presently exchanging facility tours between SLAC/SSRL and LLNL in order to gain further information as well as to develop good working relationships. Extensive liaison is expected to continue throughout the 2 year life of this project, and the current progress is satisfactory.

The initial cost and time estimates have been spot checked and we've only found about 10% disparity in the assembly drawings which were sampled. The original manpower estimates that were provided to MFD in March of this year (about 3000 man-hours of cleaning and about 5,500 man-hours of IIG welding) are still the best available information concerning this future workload.

Alpha Program for TRW & DARPAA. Ion Plating

During the past quarter we finished all of the PVD development work planned for VPL on this project. We've completed the following studies: uniformity versus pressure, uniformity versus bias, and uniformity versus manipulation. We are still awaiting data on adhesion versus bias. We are also testing adhesion on non-anodic etched samples with and without chromium.

This information was presented at the Alpha quarterly review on May 23 at TRW. We have successfully transferred the bulk of this technology to OCLI at this point and they are gearing up for subsequent production runs on the actual geometries. Future plans call for us to be filling a consultant role for Alpha from now through the end of the program. We'll be spending a fair amount of time at OCLI through the first coating production runs on the full scale demonstrators as well as a lot of effort prior to that just debugging the process.

Alpha Program for TRW & DARPAB. Metal Finishing

We have completed the installation in Building 322 of the nine very large copper plating and related processing tanks, the push-pull exhaust system, the clean wall with HEPA filtered air, and the related chemical pumps, heaters, and controls. We are currently in a start-up and check-out mode. We completed a parametric study on acid activators for PVD copper coating and molybdenum compatibility and electroplated three stainless steel cleaning study cones and thirty 1-1/2-inch diameter stainless steel flats for machining and other process verification studies.

C. Germanium Coatings on Molybdenum

To verify a technique for detecting unbonded areas in copper coatings on molybdenum substrates, we were asked to deposit a precise pattern of germanium dots on molybdenum discs. The germanium was evaporated by electron beam and deposited 100 nm thick through a mask having an array of pinholes ranging from 0.005 to 0.030 cm in diameter. The dotted discs were subsequently overcoated with copper by ion plating. The germanium provided unbonded areas. The detection method proved to be successful and will be used for the Alpha Cladding project.

AXAF Program for J. Bryan

The goal in this program is to provide a thick adherent copper layer on glass for subsequent machining by diamond turning for an X-ray telescope application. The purpose of the coating is to allow the necessary critical alignment of these tubes. In the initial trial runs we produced a 0.64 mm thick coating on glass, but this deposit had some cracks which we believe are due to temperature related stress. This coating was produced on a flat plate and the temperature was much higher than would be the case in the actual application. We will next coat the ends of a 25.4 cm diameter tube, which is closer to the actual geometry of the final part. With this configuration we'll use heat sinking to reduce the substrate temperature. Other work on this project will involve the use of electrodeposition to build up to the final coating thickness after an initial, adherent coating is applied by physical vapor deposition.

Penetrometer Foil Coating for C. Anderson

We fabricated penetrometer foils of two different classified materials. In both cases the materials were electron beam evaporated and deposited through a mask onto .076 mm thick Kapton. The mask was used to define specific areas used for gravimetrically establishing specified areal densities. The foils produced are used in an instrument called a penetrometer, which measures the penetrating power of X-ray beams.

The requester specified that the part-to-part variation in areal density be less and $\pm .01 \text{ mg/cm}^2$ and that the total areal density tolerance be less than $\pm .05 \text{ mg/cm}^2$. These tolerances were extremely difficult to achieve because Kapton absorbs water readily. The weight gain/loss of the substrate due to absorbed/desorbed water under normal ambient conditions was greater than the weight of the deposited material. To overcome this phenomenon required that equivalent humidity conditions be achieved for comparative measurements. We determined that the majority of weight gain occurs within the first hour of exposing Kapton to the ambient atmosphere.

Another problem encountered was that the accuracy of the microbalance was adversely affected by static charging of the Kapton when the Kapton piece was larger than the microbalance pan. This problem was averted by limiting the size of the Kapton. A beta backscatter instrument was used to confirm that part-to-part deviation was well within tolerance.

Black Coating on Aluminum for C. Smith

An adherent coating with black body characteristics was required on aluminum. This coating had to have an absorptance value of 0.8 to 1.0 and survive a rapid ramp up in temperature from 27 to 538C and not peel or fade. A number of coatings were tried: electrodeposited black chrome, chemically blackened copper, carbon black paint and anodized and dyed aluminum. The only coating that maintained a good black finish after temperature cycling to 538C was the black chromium. Black anodized aluminum turned straw color after heating at 538C, chemically blackened copper flaked and peeled from the substrate and carbon black paint also peeled. Based on these results, the black chromium was used for this application.

Electroformed Gold Penetron for the BEVATRON at LBL for H. Heckman

The penetron is a precision diagnostic tool used to measure heavy ion penetration. Our Metrology Group produced a copper mandrel with single point diamond turning (S.P.D.T.) that was flat within 10 millionths of an inch. We designed a masking fixture with O-ring seals to protect the datum reference surfaces during the electroforming operation which required deposition of 55 mils of gold.

The gold electroform was then S.P.D.T. machined to the final geometry which was 3.0 inches in diameter with a 0.516 inch diameter center hole. The final thickness was 50 mils with one surface tapering to a feather edge of 0.5 mils in thickness at the center hole. The critical requirement was uniform wall thickness at any linear radial plane.

Electroformed Gold Penetron for the BEVATRON at LBL for H. Heckman (Cont'd.)

Handling a part of this geometry during and after the mandrel removal process is a touchy process. We threaded four teflon screws into the flange area of the penetron and placed the unit in a container of 30% by volume nitric acid at 27 degrees centigrade to etch away the copper. We siphoned away the acid and the several rinse water cycles so as not to put undue stress on the 0.5 feather edge of the gold. The penetron has been delivered to LBL for testing.

Miscellaneous Non-Routine Vacuum Processes Lab AccomplishmentsOutgassing Testing:

Al/Polyethylene/Al foil sandwich for WEXFORD for C. Brooks.
Poly-R6000 and Poly-R6000/50% alumina for WEXFORD for C. Brooks.
II2987 epoxy for WEXFORD for C. Brooks.
101-C10 black paint on stainless steel for WEXFORD for V. Karpenko.
Gas analysis of sample bottle for S. Martin.
Fiber optics cable for COTTAGE for D. Nelson.

Deposition:

Bi & Se on Cu for X-ray measurements for C. Bruns.
Ge on Bi for X-ray measurements for C. Bruns.
Ni on polypropylene film for COTTAGE for D. Bowen.
Cr/Au/MgFl on microchannel plates for Janus and KMS laser experiments for M. Eckart and M. Gerassimenko.
Au & CsI on Parylene for KMS laser studies for M. Gerassimenko.
Au/Nb on microchannel plates for KMS laser studies for M. Gerassimenko.
Ni on Acrylic light guide for WEXFORD for B. Johannes.
Au on streak camera cathode Be foil for Phoenix laser system/Novette X-Ray laser experiment for H. Medeckl.
Al on Mylar for H₂/vacuum isolation COTTAGE foil for T. Stack.
MgFl on glass for Raman spectroscopy thin film characterization for N. Thomas.
Al on Parylene for COTTAGE - Sharacs fluor array test and calibration for A. Toor.

Brazing:

SST flange/bellows for High Field Test Facility for J. Aldridge.
W wire to W/glass feedthroughs for epitaxial plasma etcher for V. Healy.
IC and heater leads for LIS-ESCA for W. McClean.
Assembly for steady state vapor bubble cryostat for F. Allen.
W to Glass feedthroughs for epitaxial plasma etcher for V. Healy.
25 AuPd tubes/SST flange for H₂ purifier for A. Tullis.

Other:

Density comparisons of Au/Kapton specimens by beta backscatter for C. Anderson.
Vacuum-technology field-support for WEXFORD.

Miscellaneous Non-Routine Metal Finishing Accomplishments

Etching a groove 1 mm wide by .06 mm deep by 5 cm long in stainless steel for D. Selay.
Chemical etching Zerodur glass for J. Bryan.
Blackening mirror mount assemblies of aluminum and steel for B. Holloway.
Electropolishing of large parts for NORMANNA for B. Motta.
Electropolishing of six foot long stainless steel pipes for Sandia.
Black chromium plating of stainless steel straps for G. Henderson.

Presentations

1. H. L. Draper, A. DiGiallonardo, D. Walmsley and M. Benapfl, "Optimizing the Hot Hollow Cathode Process for Automated Production," International Conference on Metallurgical Coatings, San Diego, CA, April 12, 1984.
2. J. W. Dini, "Electroplating and Vacuum Deposition, Complementary Coating Processes," American Vacuum Society, New Mexico Chapter, 20th Annual Symposium, April 19, 1984.

Publications

1. E. D. Erikson, T. G. Jeat, D. D. Berger and B. A. Frazier, "Vacuum Outgassing of Various Materials," J. Vacuum Science and Technology, A2, (2), 206 (Apr-June 1984).
2. G. E. Overturf, W. K. Kelley, J. W. Dini and D. P. Atkinson, "Joining a Large Brass Ring to Stainless Steel with the Aid of Electroplating," Plating and Surface Finishing, 71, 64 (March 1984).
3. J. W. Dini and H. R. Johnson, "Plating on Titanium and Zirconium," Industrial Applications of Titanium and Zirconium, Third Conference, ASTM STP 830, Webster/Young, Editors.
4. J. W. Dini and H. R. Johnson, "Plating on Some Difficult to Plate Metals and Alloys," UCRL-90466, Feb. 21 1984.

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