Foil Fabrication for the ROMANO Event

Joseph G. Romo Jr., John W. Weed, Glenn E. Griggs,
Theodore G. Brown, Patricia L. Tassano

INTRODUCTION

The Vacuum Processes Lab (VPL), of LLNL's M.E. Dept. - Material Fabrication Division (MFD), conducted various vacuum related support activities for the ROMANO nuclear physics experiment. This report focuses on the foil fabrication activities carried out between July and November 1983 for the ROMANO event. Other vacuum related activities for ROMANO, such as outgassing tests of materials, are covered in separate documentation.

VPL was asked to provide 270 coated Parylene foils for the ROMANO event. However, due to the developmental nature of some of the procedures, approximately 400 coated foils were processed. In addition, VPL interacted with MFD's Plastics Shop to help supply Parylene substrates to other organizations (i.e., LBL and commercial vendors) which had also been asked to provide coated foils for ROMANO. The purposes of this report are A) to document the processes developed and the techniques used to produce the foils, and B) to suggest future directions. The report is divided into four sections describing: 1) nuclear target foil fabrication, 2) Parylene substrate preparation and production, 3) calibration foil fabrication, and 4) foil and substrate inspections.

NUCLEAR TARGET FOIL FABRICATION

The principal VPL activity for the ROMANO event was the production of nuclear target foils. This activity required rapid process development and the use of coating materials (e.g., zinc and magnesium) that are normally considered undesirable in general purpose physical vapor deposition (PVD) systems. It was necessary to commit a major portion of VPL's PVD capabilities and to borrow both personnel and equipment from other LLNL groups. Over a four month period of time, approximately 400 target foils were produced for the ROMANO event. Of these, approximately 50% were required to backup expected losses during assembly, calibration and transportation. Approximately 70% of the foils produced were unusable due to various fabrication imperfections. (See Appendix -A- for a summary of the inspection results for coated and uncoated foils.)

The physics request called for the deposition of two materials: magnesium and zinc. However, because of the deposition properties of these materials, a nucleation layer needed to be deposited first.

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Foils were fabricated in six different combinations of four materials deposited in various thicknesses onto unbacked Parylene substrates. The four materials were: aluminum, magnesium, copper, and zinc. Programmatic specifications required that all coatings be pinhole free and light tight, within 10% of the nominal thickness, and within 5% in thickness uniformity. All of the metallic coatings were deposited by electron beam gun evaporation. The coating parameters for each combination are described in Table 1. Descriptions of each material/layer thickness combination with associated fabrication process considerations are provided in the following paragraphs.

Zn/Cu/Parylene/Cu/Zn (in 150/20/200/20/150 nm Layer Thicknesses) - Of the six combinations requested, these were the most difficult foils to fabricate. (Additionally, the largest quantity of foils, 144, were requested in this particular combination.) These foils also had the greatest ratio of mounting ring inner diameter (28.9 mm) to Parylene thickness (200 nm). The ratio of mounting ring I.D. to foil thickness provides a measure for comparing the delicacy of the foils and for estimating production run yields. Approximately 110 coating system runs, using six operators on two shifts, were accomplished with minimal coating variation between runs. This was made possible by the use of a deposition controller (Inficon IC-6000) having programmable closed-loop deposition rate control and monitoring features.

Zinc's poor nucleation properties on Parylene were overcome by first depositing a nucleating layer of copper. Copper had been used successfully in previous applications which revealed evidence of diffusion between copper and zinc providing adequate adhesion. (The rate of diffusion was clearly dependent on time and oxide thickness.) Previous attempts to use aluminum as a nucleating layer for zinc coatings were unsuccessful due to poor Al/Zn bonding. However, aluminum was used successfully for the magnesium coatings described later.

The radiant energy from the evaporation source and condensation energy from the arriving molecules cause physical changes in the Parylene substrates. During the deposition process the Parylene initially shrinks (becoming taut), then expands (slackens) as the deposition proceeds. The specific time of initiation and the magnitude of these changes are dependent, and to some extent adjustable, by changes of the following parameters: 1) evaporation rate, 2) source to Parylene distance, 3) source size and type, 4) chemical/physical characteristics of the deposit, 5) Parylene thickness and condition, and 6) Parylene temperature at the start of coating.

An additional complication is introduced by the coefficient of thermal expansion (CTE) difference between the metallic deposit and the Parylene¹¹. In the early stages of the deposition process the CTE of the Parylene prevails. As the metallic deposit increases in thickness, it develops sufficient strength to dominate the Parylene behavior. When depositing 1500 nm or thicker coatings of zinc on Parylene foils of less than 300 nm, the Parylene is deformed. As the Parylene stretches, it progressively exposes additional surface area

to be coated. As the coating builds up on the newly exposed areas, it too increases in strength and contributes to the stretching of high stress or weak areas. The result is that imperfections (i.e., thin areas and pinholes) tend to develop, particularly at the perimeter where the Parylene is attached to a relatively unyielding mounting ring. Since the Parylene is sometimes captured between two rings, one possible means of alleviating this situation is for the ring that gets added after coating to have a smaller inner diameter than that of the ring on which the Parylene is mounted during coating. The inner portion of the added ring would then overlap the perimeter imperfections.

Zn/Cu/Parylene/Cu/Zn (750/10/500/10/750 nm) - Depositing thicker coatings on thicker Parylene produced higher quality foils than those in the above described combination. There were significantly fewer pinholes and the coatings had a better appearance. The thicker coatings also provided better coverage by overlapping poor nucleation sites. The additional strength of the thicker Parylene reduced the effects of the thermally induced forces. There were 13 foils fabricated in this combination.

Zn/Cu/Parylene/Cu/Zn (125/10/500/10/125 nm) - Though similar to the first combination described in this section, this combination was easier to fabricate because of the slightly thinner coatings and the significantly thicker Parylene specified. The 48 foils in this particular combination had originally been coated by a (commercial) vendor. However, after calibration, as the foils were being prepared for shipment to the test site, residual tensile forces caused progressive stretching and tearing failure of the Parylene substrates. There was insufficient time for the vendor to try again, so expeditious on-site fabrication was necessary. Applying the newly acquired experience with zinc on Parylene foils, high quality (i.e., minimal pinholes) foils were supplied on time.

Mg/Al/Parylene/Al/Mg (250/20/500/20/250 nm) - Fifty foils were fabricated in this combination. As with the zinc on Parylene foils described previously, the poor nucleating properties of magnesium on Parylene made the use of an interfacial nucleating layer necessary. Aluminum was chosen because it had been used successfully as an interface between magnesium and Parylene in prior work.

Because the vacuum evaporation systems in VPL were fully committed, a system located in the M.E./Weapons Engineering Division (WED)/Vacuum Support lab was used to accommodate programmatic scheduling. A team effort [by personnel from the WED/ Vacuum Support group, M.E./Materials Fabrication Division (MFD) groups (General Assembly, Tool Design, Machine Shops, and VPL), and E.E./ Machine Control Group] was conducted to convert a commercial RF-induction source evaporation coater into an electron beam evaporation coater. The conversion consisted of substituting evaporation sources, incorporating an Inficon IC-6000 process controller (to provide control comparable to that used in VPL systems), installing shielding to reduce magnesium contamination effects, and adapting fixturing to permit using the

system's planetary substrate assembly. The resulting system could handle more foils per run than the system being used in VPL (12 parts/run vs. 5 parts/run). This greatly helped to satisfy the tight schedule demands.

The significance of the physical response of the Parylene to the coating process became very apparent when the process controller program parameters established in the VPL systems were copied for the initial run in the modified WED system. Due to differences in source characteristics and source-to-substrate distance (even though these were taken into account through calibration runs), all the foils in the initial run self destructed due to excessive tautness. Based on the insight gained in coating the previously described combinations, the deposition rate was then increased to achieve the desired level of tautness.

Mg/Al/Parylene (12,000/20/500 nm) - An effort to produce foils in this combination was aborted due to an inadequate supply of magnesium source material. The commercial suppliers of this material required longer lead times than the Program's schedules permitted. However, no technical problems would be anticipated for the fabrication of these foils given an adequate supply of magnesium.

Al/Parylene/Al (150/850/150 nm) - No coating problems were encountered with the six foils requested in this combination. However, special fabrication considerations were required due to a programmatic restriction that, to prevent reflections, there be no coating deposited on one side of the foil holder. In order to accommodate this requirement the Parylene was mounted on a transfer ring, aluminum coated on both sides, and subsequently transferred to the final holder. The successful transfer to the holder required a float, trim and mount operation using the surface tension of distilled water as the foil support medium.

Technical Assistance to Other Target Foil Fabricators - In addition to fabricating nuclear target foils, technical assistance was given to other organizations engaged in supplying foils for the ROMANO event. This included examining magnesium on Parylene foils which were fabricated at the Lawrence Berkeley Laboratory (LBL) and were destroyed due apparently to the Parylene over-tightening during the coating process. As in instances noted above, a slight increase of the deposition rate also solved this problem. Thin Film Technology, a commercial supplier, experienced the same problem as LBL. However, in this case our interaction was too late to help them supply foils for ROMANO. We did provide information by phone that should enable them to satisfy future requests. We also contacted another commercial supplier, Industrial Vacuum Engineering, in an attempt to circumvent problems similar to those experienced by LBL and Thin Film Technology. However their approach involved magnetron sputtering, so our experience with electron beam evaporation did not apply. Substrates were sent to Industrial Vacuum Engineering for coating, but they did not produce coated foils in time for use on ROMANO.

PARYLENE SUBSTRATE PREPARATION AND PRODUCTION

A small stock of Parylene, left over from a previous event, allowed foil production to begin without a new supply of Parylene. Having this stock available allowed the start of the floating, mounting, and coating activities necessary to establish coating parameters and work flow. Discussions with Nuclear Test Program representatives are proposed to determine if projections justify establishing and maintaining an inventory of Parylene foils to ease the typically tight schedules encountered in fabricating diagnostic foils for nuclear events. Decisions would need to be made as to sizes and quantities, funding sources, storage location, and responsibility for maintenance of the inventory.

Estimates of the amount of Parylene needed to satisfy ROMANO foil requirements were based on past experience in obtaining Parylene for other events. In the past, failure to satisfy thickness specifications alone has accounted for a rejection rate of over 50% of delivered Parylene. To help assure meeting their schedule, the programmatic requesters placed duplicate orders with Union Carbide and MFD's Plastics Group for 800 Parylene coated, 127 x 127 x 3.18 mm, glass slides with release layers. The Program also purchased a second Parylene deposition system, from Union Carbide, which enhanced MFD's thin film Parylene fabrication capabilities for ROMANO and subsequent nuclear test needs.

To produce unbacked Parylene foils, Parylene is chemically vapor deposited onto glass flats which are first coated with a release agent. For these foils to be of high quality, the glass flats must be very clean prior to coating. Because in the past the Plastics Shop has been neither equipped nor staffed to do either the glass cleaning or the release layer application, VPL has been doing both of these processes for the Parylene foils deposited by the Plastics Shop. The procedure VPL uses for cleaning the glass slides is complex and requires 19 steps and several hours to complete. The negative impact of the glass cleaning operation on VPL's other coating activities was recognized and an effort to transfer this activity to the Plastics Shop has been initiated. Some duplicate equipment had to be acquired to accomplish this transfer. This was not accomplished in time to benefit the ROMANO effort, so the burden of this activity remained in VPL. However, in response to anticipated needs, the Plastics Shop is preparing to conduct this activity in the future.

The entire first shipment of Parylene coated slides received from Union Carbide was unusable. Inadequate packaging had resulted in physical damage to the Parylene films, the coated slides were heavily contaminated with particulate matter, and the Parylene films could not be released from the glass slides. Union Carbide was informed of these problems (while they were at LLNL installing MFD's new Parylene deposition system) and they committed to replacing the entire shipment. In response to Union Carbide's request, they were provided with our current glass-cleaning and release agent dip procedures. We

also provided examples of acceptable packaging containers. The second Union Carbide shipment arrived too late to be used for the ROMANO event. Due to the tight schedule aspects of foil fabrication activities for subsequent nuclear events, the second shipment has not yet been inspected for quality, thickness, and releaseability. However, we plan to do so soon and Union Carbide did indicate that they had been successful in producing releaseable films from samples of the second shipment.

All of the useable Parylene foil material for ROMANO was produced by the MFD Plastics Shop. However, to obtain diagnostic foils for ROMANO, compromises had to be made in requested thickness, uniformity, and overall quality of the Parylene (and the resultant diagnostic targets). The original programmatic specification for the 200 nm Parylene stipulated plus or minus 10% in thickness tolerance. In order to obtain sufficient foils, the tolerance had to be increased to plus 50%, minus 10%. Logistics also required that Parylene films be used even though they had defects (described in Appendix -B-) that adversely affected the overall quality of the coated foil.

Less than 50% of the MFD produced Parylene coated slides were usable. Significant improvements in all areas of Parylene production were made as work progressed. As experience developed there was a decrease in the frequency of defect occurrence and an increase in the ability to satisfy thickness requirements. The latter was aided by the introduction of an in-situ thickness monitoring technique based on laser interferometry. But, at the end of this activity the process yield still required considerable improvement.

One source of defects which are sometimes evident on mounted Parylene films was traced to interactions between the Parylene deposit and the detergent release layer applied to the glass substrates. The interactions appear to produce either composition or structural abnormalities in the Parylene. Additional defect characteristics observed during the production and inspection of MFD produced Parylene films indicate that the films did not meet all of Union Carbide's reported film properties [2] [3] [4] for Parylene-N (the type specified by the programmatic requesters). They failed to meet strength, uniformity, and thermal behavior characteristics. It is suspected that the detergent release layer used may have interacted with the Parylene and possibly modified and/or compromised its properties. The detergent was applied by dipping the glass substrates into a dilute solution of detergent and distilled water. Efforts to eliminate the defects by changing the release layer application procedures were only partially successful. Additional refinement of detergent release application procedures or alternative release materials (such as vapor deposited salts) need to be investigated for future needs.

One advantage of using detergent is that we are able to deposit it using batch processing techniques. Thus, the process is much faster than the deposition of other release agents which, because of equipment/technique limitations, have been previously applied to one

substrate at a time. However, the main reason detergent was chosen as the foil release layer was because previous attempts to explore the use of salt release agents were thwarted by the discovery that the salt layer on substrates got contaminated by vapors from the detergent release coatings used on the Parylene deposition system. The Plastics Shop previously had only one Parylene deposition system with which to service all programmatic needs. The majority of jobs processed in that system call for relatively thick deposits of Parylene. To facilitate removing excess Parylene buildup from the chamber and fixturing, the exposed surfaces were routinely coated with detergent. Now that the Plastics Shop has a Parylene deposition system which is dedicated to thin film foil fabrication for nuclear tests, release agent trials are assured a high priority in any Parylene foil fabrication process development.

Another source of defects was the contamination of the Parylene by particulates generated in handling the containers used to transport substrates and finished foils between VPL, Plastics shop, and other facilities involved in calibrating and fielding the foils. This problem should be alleviated by reducing the handling and shipping requirements once the glass substrate cleaning operation is transferred to the Plastics Shop and is adjacent to the Parylene deposition system.

An old problem was re-encountered. The normally dependable technique for securely mounting thin films relies on the fact that when thin films are brought into contact with smooth, clean metallic surfaces they cling with tenacity that far exceeds the strength of the foils. Thus, reliable bonds are formed without resorting to the use of adhesives (which often introduce new problems and complexities). Unfortunately, the foil support rings supplied for this event were made of stainless steel which had been electropolished as a final step in their fabrication. The exact failure mechanism has not been investigated, but electropolished surfaces somehow cause a failure of this bond. The problem was circumvented by simply resurfacing the rings using a very fine abrasive. A water break test was implemented as an acceptance test. In this test the rings are held vertically and water is poured across the mounting surface. To pass this test the water must flow in a thin continuous sheet rather than in beads. Unfortunately, before the bonding problem became apparent hundreds of man-hours and about 300 slides of Parylene were wasted in coating foils mounted on the electropolished rings.

Due to the large quantity of foils processed and to the poor reproduceability of the Parylene deposition processes, approximately 2.5 man-months were expended in measuring and documenting the thickness of Parylene films. Over 1500 thickness measurements were required for ROMAND. The Parylene deposited on the center substrate of each batch produced by the Plastics Shop and a strip adjacent to each Parylene film used for fabricating a target was measured. This was accomplished by separating scrap pieces from each side of the substrate during the foil release process, floating these pieces onto a glass slide, air drying the slide, and then measuring the thickness

using a profilometer. Our profilometer is old. After five years of continuous service, the large number of measurements required has left the profilometer worn and in need of replacement. Since profilometer measurements are essential for the conduct of many nuclear target fabrication processes done in VPL, the continued availability of this instrument's function is of critical importance. Because neither repair nor replacement can be accomplished quickly enough to accommodate typical nuclear test schedules, a replacement profilometer needs to be acquired before the present instrument breaks or wears out completely.

The magnitude of the preparation and production of Parylene substrates activities and the problems encountered emphasize the need to improve the MFD's overall thin film Parylene fabrication capability. To maximize the Parylene process yield and film quality, the following areas need to be investigated and acted upon: 1) clean-room facilities for all substrate and Parylene processing, 2) acquisition and implementation of an automated substrate cleaning station, 3) improved measurement capabilities (including in-situ methods as well as a backup profilometer), 4) transport containers with improved antistatic and low level particulate contamination features, and 5) Parylene foil deposition process development.

CALIBRATION FOIL FABRICATION

VPL was also requested to fabricate foils which were needed for reference purposes on a system being used to calibrate nuclear target foils for the ROMAND event. A foil of each of the following four listed materials was required: gold, iridium, lead, and a classified material. One or two thicknesses of each material were required. In some cases we were able to provide a back up foil, since it could be fabricated simultaneously with very little extra effort. The requester preferred that the foils be unbacked and required that they be pinhole free. We fabricated all the foils by electron beam evaporation. The coating parameters for foils of each material/thickness category are described in Table 2. Included below are descriptions of the fabrication process considerations for each category.

Gold - We produced 550 nm and 1900 nm unbacked gold foils that were of high purity and pinhole free. The 1900 nm foils were particularly noteworthy because they were thicker than foils normally produced using conventional electron beam evaporation processes. A 70 nm thick layer of sodium chloride was first vacuum deposited onto glass substrates to provide for the release of the subsequently deposited gold coating. After the deposition process, the gold coating was released by immersing the substrates in distilled water to dissolve the salt layer. The resultant foils, which were supported by the surface tension of the water, were then floated onto stainless steel mounting rings and bonded to the rings with Permabond-910 adhesive.

Iridium - Under tight schedule constraints we produced a single Mylar backed, 550 nm thick iridium foil. We first attempted to produce an

unbacked foil, but the deposit was highly stressed and it curled and cracked. Unfortunately, there were some pinholes in the iridium foil we delivered. We attribute the pinholes to imperfections in the Mylar (supplied by the requester) and to subsequent particulate contamination.

Lead - We fabricated unbacked lead foils of two thicknesses, 830 nm and 2900 nm. Initially we used sodium chloride as the release layer for both thicknesses. This worked well with the 830 nm foil, but we could not release the 2900 nm deposit and had to change to detergent as the release layer in order to produce the 2900 nm lead foil.

Other - Foils of the classified material were required in two different thicknesses, 960 nm and 3300 nm. We successfully fabricated the 960 nm unbacked foils using the same techniques as were used to produce the gold foils. However, we were unable to successfully fabricate an unbacked 3300 nm foil. It ruptured during the release/float operation, apparently due to its low intrinsic strength. To circumvent this problem, we deposited the classified material onto a thin Mylar backing foil.

In producing the lead and the classified material foils, the evaporation process initially proved to be too unstable to take full advantage of the closed loop deposition rate control features of a programmable process controller. This type of monitor/controller can usually provide source performance and control not attainable by a human operator using only a deposition monitor. It can also provide source performance data not otherwise obtainable. Its monitoring features emphasized the poor performance of the electron beam gun source with these two materials.

The specific evaporation problems encountered were: gas bursts, spatter, poor evaporation rate control, gun contamination, and the requirement for a higher gun power input than should be necessary for such low temperature source materials. One means of alleviating these problems is to use a crucible liner. Experience had shown that commercially available liners would only partially solve these problems. A crucible of unusual design was fabricated and installed for evaluation. An immediate improvement in the source and source material's performance was apparent during the first runs with this new design (enabling the full use of the programmable controller).

The new crucible is made of tantalum sheet metal which is spun into an "M" shaped cross section. It was shaped to fit in the hopper portion of an Airco Temescal Model SFIH-270-2 electron beam gun evaporation source, but the design could be adapted to other commercially available guns. The new design provides a larger volume for evaporant than the gun's crucible and a longer thermal path to the water cooled hearth. This latter feature promotes more uniform heating, more complete melting, and better outgassing of the source material. The longer thermal path also reduces the amount of electron beam power required to achieve and maintain evaporation temperature, thereby reducing the cooling/heating lag and allowing stable, closed-loop

control of the evaporation rate. As is the case when using liners, an additional benefit is derived from the avoidance of contact between the source material and the copper hearth; which can result in undesirable alloying that causes hearth erosion and contamination of the deposited coating.

FOIL AND SUBSTRATE INSPECTION

For ROMANO, we implemented procedures for the inspection and documentation of both uncoated Parylene substrates and coated target foils produced by LLNL, LBL, Thin Film Technology, and Union Carbide. A major consideration was that the programmatic application of the target foil assemblies required that there be no light leaks either through or around the foils. This meant that there could be no perforations in the Parylene substrates, pinholes or scratches in the coatings, or gaps between foils and their mounting rings. The inspection and documentation procedures served two main purposes: 1) to identify defects/processing problems, thereby permitting corrections to be made in the immediate fabrication procedures, and 2) to record the condition and characteristics of each foil prior to delivery to our programmatic requesters, thereby providing the potential for using the correlation between foil quality and programmatic test results to guide future efforts.

Uncoated Substrate Inspection - Defects and other distinguishing features were recorded in one of two logs, depending on whether the bare Parylene films were to be coated by VPL or by other coating labs. The characteristics of approximately 500 Parylene films to be coated in VPL were logged in Nuclear Test Engineering's "Parylene Information Sheet". To assure that the other labs involved in coating foils for ROMANO received substrates of acceptable quality we inspected an additional 214 uncoated Parylene films, rejected those having obvious defects, and recorded any observed anomalies (to permit their later correlation to coating quality). The characteristics of those to be sent to other coating labs were recorded in VPL's "VPL Uncoated Foil Inspection Log" (see sample blank form, VPL document 0746g, in Appendix -C-). A coded, tabular method for noting 17 distinguishing features was devised to ease the effort involved in identifying salient features, recording them, and subsequently analyzing the logged data. These features and their codes are described in a companion document, "Definitions of Uncoated Foil Phenomena Codes" (See VPL document 0748g, Appendix -B-). The frequency of occurrence of each type of defect is presented in chart form in Appendix -A-.

Coated Foil Inspection - An intense back light was used to inspect the approximately 400 completed foils. The light intensity was adjustable and a ground glass plate was used to diffuse the light. The back light was fitted with a light tight fixture to hold the individual foils during inspection. The assembly was used in a dark room to prevent ambient light from interfering with the inspection. Each foil was inspected for rips, punctures, size and quantity of pinholes in the coatings, and light leaks at the perimeter between the

foil and its mounting ring. Early in the program a nine level grading system was developed to rate each foil based on these characteristics and a document titled "Explanation of Post Coating Foil Grading System" was prepared describing the criteria used for establishing each grade (see Appendix -D-). Each foil's grades and other pertinent features, along with the coating parameters used in its production, were recorded in a log created for this purpose titled "Vacuum Processes Lab Coated Foil Inspection Log" (see sample blank form, document 0751g, in Appendix -E-). The logs indicate that of the 400 coated foils produced for ROMANO, approximately 20% were of a quality suitable for programmatic use. The distribution of coated foil grades are graphically represented in Appendix -A-.

SUMMARY AND RECOMMENDATIONS

With very little lead time, we were requested to fabricate 270 detector targets and calibration foils for the ROMANO event. Fabrication of some of the foils, particularly those involving the zinc coating of Parylene, required that process development and midstream process tuning be conducted rapidly to meet the tight programmatic schedule constraints. The effort also included producing the Parylene substrate foils that Union Carbide was unable to deliver in time for the event. We accomplished this latter requirement working in concert with the Plastics Shop. The preparation of substrates for Parylene deposition and the inspection of Parylene foils represented a significant portion of the overall nuclear target fabrication activities for the ROMANO event.

Innovations, acquired knowledge, and products resulting from this effort that are particularly worth highlighting include: 1) the fabrication of relatively thick (for PVD processes) unbacked gold foils, 2) the fabrication of zinc on Parylene nuclear target foils, 3) the design and application of a unique crucible for improved electron beam evaporation of low evaporation temperature materials, 4) a better understanding of Parylene's properties and effects on the quality of foils and, 5) a standardized inspection and documentation procedure that allowed process corrections, future data analysis, and better communications between the requesters, vendors, and fabricators.

For future applications we are exploring the implementation of higher energy vacuum deposition processes, such as magnetron or ion gun sputtering, because they appear to offer a strong probability of significantly improving the quality and yield of our target fabrication processes. Among other benefits, these processes offer significant advantages for depositing materials having high vapor pressures (e.g., zinc and magnesium) onto thin films of hydrocarbons (such as Parylene).

The issue of nuclear target foil fabrication is also being addressed by a special committee within Nuclear Test Engineering Division with the goal of improving quality and schedule factors. We have met with this committee and discussed a number of related factors including:

establishing a unified means of providing priorities for nuclear test foils, improved Parylene production, improved metallic foils and coatings, the use of VPL personnel on a continuous basis to improve production capabilities, clean room facility requirements, and equipment needs. These factors are described in reference [5].

DISCLAIMER

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TABLE 1 - NUCLEAR TARGET FOILS

Coating Parameters

	<u>Foil 1</u>	<u>Foil 1</u>	<u>Foil 2</u>	<u>Foil 2</u>	<u>Foil 3</u>	<u>Foil 3</u>	<u>Foil 3</u>	<u>Foil 3</u>	<u>Foil 4</u>	<u>Foil 4</u>	<u>Foil 5</u>
Material	Zn	Cu	Zn	Cu	Zn	Cu	Zn	Cu	Mg	Al	Al
Layer Thickness (nm)	150	23	750	10	125	10	125	10	250	20	150
Electron Beam Source Voltage (KV)	10	10	10	10	10	10	8	8	10	10	10
Average Emission Current (A)	.02	.24	.02	.24	.02	.24	G	G	.03	.4	.55
Deposition Rate (A/S)	1.9	2.0	1.9	2.0	1.9	2.0	1.9	2.0	10.0	1.0	20.0
Chamber Pressure Prior to Coating (Torr)	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷
Average Pressure During Coating (Torr)	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶	10 ⁻⁶
Type of Gun	E	E	E	E	E	E	D	D	D	D	D
Substrate Motion	C	C	C	C	C	C	B	B	B	B	C
Average Source to Substrate Distance (cm)	34	31	34	34	34	34	50	50	50	50	65

Foil Description

Codes

Foil 1 - Zn/Cu/Parylene/Cu/Zn

A No Motion

Foil 2 - Zn/Cu/Parylene/Cu/Zn

B Planetary Substrate Motion

Foil 3 - Zn/Cu/Parylene/Cu/Zn^[1]

C Rotating Substrate Motion

Foil 4 - Mg/Al/Parylene/Al/Mg

D 270° Bent Beam, 15 cc Pocket, 4 Hearth Turret

Foil 5 - Al/Parylene/Al

E 270° Bent Beam, 7 cc Pocket, 4 Hearth Turret

F 270° Bent Beam, 40 cc Pocket, Single Hearth
(With Newly Devised Crucible)

G Data Not Available

Notes

[1] These Foils Were Fabricated in Two Different Systems

JWA/kmb/10179

TABLE 2 - CALIBRATION FOILS

Coating Parameters

	<u>Foil 1</u>	<u>Foil 2</u>	<u>Foil 3</u> ^[1]	<u>Foil 4</u>	<u>Foil 5</u>	<u>Foil 6</u>	<u>Foil 7</u> ^[2]
Material	Au	Au	Ir	Pb	Pb	(H)	(H)
Layer Thickness (nm)	550	1900	550	2900	830	960	3300
Electron Beam Source Voltage (KV)	10	10	10	10	10	10	10
Average Emission Current (A)	.34	.47	.7	.02	.02	.01	.01
Deposition Rate (A/S)	7.0	7.0	11.0	5.7	5.7	5.0	5.0
Chamber Pressure Prior to Coating (Torr)	10 ⁻⁸	10 ⁻⁸	10 ⁻⁶	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷
Average Pressure During Coating (Torr)	10 ⁻⁷	10 ⁻⁷	10 ⁻⁶	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷	10 ⁻⁷
Type of Source	D	D	D	F	F	F	F
Substrate Motion	C	C	C	C	C	C	C
Average Source to Substrate Distance (cm)	65	65	54	56	56	56	56

Codes

- A No Motion
 B Planetary Substrate Motion
 C Rotating Substrate Motion
 D 270° Bent Beam, 15 cc Pocket, 4 Hearth Turret
 E 270° Bent Beam, 7 cc Pocket, 4 Hearth Turret
 F 270° Bent Beam, 40 cc Pocket, Single Hearth (With Newly Devised Tantalum Crucible)
 G Data Not Available
 H Classified Material

Notes

- [1] Mylar Substrate, Heated In-Situ to 100°C Prior to Deposition
 [2] Mylar Substrate

APPENDIX A

DEFECTS IN UNCOATED PARYLENE FOILS

(LOT SIZE: 214 SAMPLES)

DISTRIBUTION OF COATED FOIL GRADES

(LOT SIZE: 334 SAMPLES)

APPENDIX B
DEFINITIONS OF UNCOATED FOIL PHENOMENA CODES

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- A. Star effects-localized areas of the foil material where lines emanate from a center spot to form a "starburst" . The center spot appears to be a thickness spot (see definition below).
- B. Orange peel - a texturing of the material that gives it the appearance of an orange's skin.
- C. H₂O/glue stain on frame - when water and glue mix they can cause a white powdery residue that can adhere to, and stain the frame supporting the foil.
- D. Single-point edge bunching - refers to the condition where the foil material has "bunched up" at its interface with the frame. It is observed as small creases emanating from the edge of the frame.

- E. Multi-point edge bunching - same as single-point except at more than one place around the frame.

- F. Indentations - deformations of the foil material. Not a thickness change, but a change in form or shape. Usually occurs in lines, wavy lines or small patches.

- G. Thickness spots - small to large spots in the material that are a different color than the rest of the material. These spots are thicker than the rest of the material.
- H. Holes or tears - self-explanatory.
- I. Color shift - a change in color over the length of the material indicating a change of thickness.
- J. Particulates - dust, etc., on the surface of the material.
- K. Impeded holes in mounting frame or ring - glue or material in the holes of the mounting frame.
- L. Crazing - similar to orange peel except on a larger scale. Not as much a texture difference, but a pattern like cracked glass.

- M. Fold or crease in material - self-explanatory.

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- N. H₂O/glue stain on material - same as C except on foil material.
- O. Scratch - a line in material similar to an indentation except on a much finer scale.
- P. Pock marks - small crater like indentations in foil material, usually occurring in groups.
- Q. Particulate or lint/hair in material - a condition where the particulate or lint is fused in and or/through the foil material.
- R. Reject - obvious gross defect.

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VPL UNCOATED FOIL INSPECTION LOG

W. Weed

8/8/83

Rev. 12/07/83

Material & Supplier

Date _____

Desired Thickness

Bonding Technique

Tolerance

Bonding Substance _____

Misc. _____

Inspector _____

Phenomena Codes

Frame #	Material #	Thickness	Grade
1	1	1/2"	A
2	2	1/2"	A
3	3	1/2"	A
4	4	1/2"	A
5	5	1/2"	A
6	6	1/2"	A
7	7	1/2"	A
8	8	1/2"	A
9	9	1/2"	A
10	10	1/2"	A
11	11	1/2"	A
12	12	1/2"	A
13	13	1/2"	A
14	14	1/2"	A
15	15	1/2"	A
16	16	1/2"	A
17	17	1/2"	A
18	18	1/2"	A
19	19	1/2"	A
20	20	1/2"	A
21	21	1/2"	A
22	22	1/2"	A
23	23	1/2"	A
24	24	1/2"	A
25	25	1/2"	A
26	26	1/2"	A
27	27	1/2"	A
28	28	1/2"	A
29	29	1/2"	A
30	30	1/2"	A
31	31	1/2"	A
32	32	1/2"	A
33	33	1/2"	A
34	34	1/2"	A
35	35	1/2"	A
36	36	1/2"	A
37	37	1/2"	A
38	38	1/2"	A
39	39	1/2"	A
40	40	1/2"	A
41	41	1/2"	A
42	42	1/2"	A
43	43	1/2"	A
44	44	1/2"	A
45	45	1/2"	A
46	46	1/2"	A
47	47	1/2"	A
48	48	1/2"	A
49	49	1/2"	A
50	50	1/2"	A
51	51	1/2"	A
52	52	1/2"	A
53	53	1/2"	A
54	54	1/2"	A
55	55	1/2"	A
56	56	1/2"	A
57	57	1/2"	A
58	58	1/2"	A
59	59	1/2"	A
60	60	1/2"	A
61	61	1/2"	A
62	62	1/2"	A
63	63	1/2"	A
64	64	1/2"	A
65	65	1/2"	A
66	66	1/2"	A
67	67	1/2"	A
68	68	1/2"	A
69	69	1/2"	A
70	70	1/2"	A
71	71	1/2"	A
72	72	1/2"	A
73	73	1/2"	A
74	74	1/2"	A
75	75	1/2"	A
76	76	1/2"	A
77	77	1/2"	A
78	78	1/2"	A
79	79	1/2"	A
80	80	1/2"	A
81	81	1/2"	A
82	82	1/2"	A
83	83	1/2"	A
84	84	1/2"	A
85	85	1/2"	A
86	86	1/2"	A
87	87	1/2"	A
88	88	1/2"	A
89	89	1/2"	A
90	90	1/2"	A
91	91	1/2"	A
92	92	1/2"	A
93	93	1/2"	A
94	94	1/2"	A
95	95	1/2"	A
96	96	1/2"	A
97	97	1/2"	A
98	98	1/2"	A
99	99	1/2"	A
100	100	1/2"	A

Comments

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APPENDIX C

0746g

APPENDIX D

EXPLANATION OF POST COATING FOIL GRADING SYSTEM

- 0+ No light leaks[1]
- 1 3 or less small light leaks
- 1- 10 or less small light leaks
- 2+ 1 or 2 medium light leaks, some small leaks
- 2 3-5 medium light leaks, some small leaks
- 2- Numerous medium light leaks, some small leaks
- 3+ 1 or 2 large light leaks, some small and medium leaks
- 3 3 or more large light leaks, some small and medium leaks
- 3- Through holes[2] or small tears and/or perimeter leaks[3]
- R Reject - Obvious gross defects such as large tears in foil

NOTES

- [1] A light leak is a hole (area of no nucleation) in the coating, but not in the Parylene.
- [2] A through hole is a hole through the coating and Parylene. This is typically observed as a brighter, scattered point of light through the foil.
- [3] Perimeter leaks are areas at the frame/foil interface where there is no coating.

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VACUUM PROCESSES LAB COATED FOIL INSPECTION LOG

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08/27/83
Rev. 12/07/83

Substrate Material _____ Coating Material _____ * Coating Tolerance _____
Date _____ Coating Thickness _____ Inspector _____
Per Side _____ Misc. _____
Total Coating Thickness _____

[illegible]

APPENDIX E