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NEW MATERIALS AND TECHNOLOGY FOR SUPPRESSING MULTIPACTOR IN HIGH POWER MICROWAVE WINDOWS

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ABSTRACT

High power microwave window development work at the Stanford Linear Accelerator Center for the period June 1973 through January 1974 is given. Emphasis was on development and testing of window coating materials and procedures used to suppress multipactor in microwave windows. These windows were fabricated from $A\ell_2O_3$, type AL-300 ceramic. Coating materials tested included Cr_2O_3, oxides of titanium and TiN. The latter material was discovered and evaluated during this program, and was found to have excellent multipactor suppression properties. Also, a new vacuum coating procedure, featuring crossed field diode array sputtering, was developed[1] and successfully used to coat a number of windows. Windows were also evaluated which were coated with TiN using vacuum evaporation techniques. Windows were also coated in partial pressures of air, oxygen and argon. Windows featuring these coating materials and techniques were evaluated by testing in a high power microwave storage ring at a frequency of 2.856 GHz. The ring, windows permitting, will operate at peak power levels on the order of 100 MW at a duty cycle of $\sim 9 \times 10^{-4}$.

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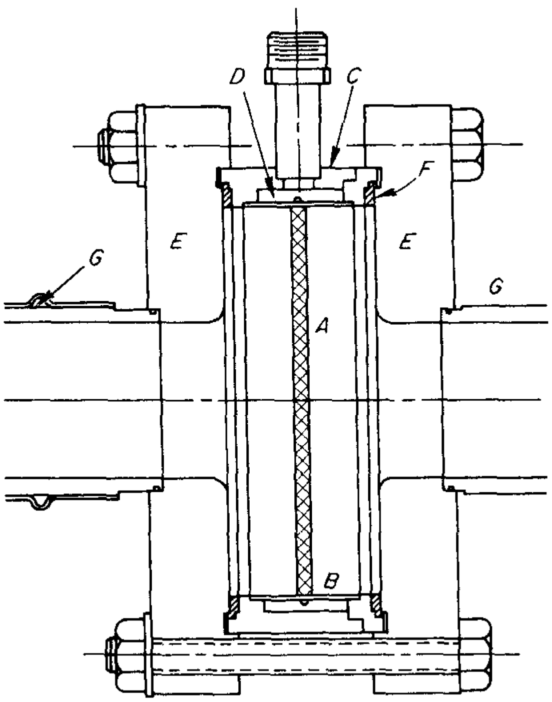
INTRODUCTION

High power microwave window development work has been an ongoing endeavor at SLAC for many years. The gradual upgrading of klystron efficiency and output power has made this essential. High dividends have resulted from this work as reflected in klystron manufacturing yield, operating reliability and extended tube life. Few SLAC klystrons have failed on the accelerator in recent years due to window problems.

High power microwave windows are particularly subject to multipactor problems.[2-7] Multipactor is an electron bombardment heating phenomenon unique to RF vacuum devices. It results from unfortunate combinations of optimum surface orientation with respect to the electric field, high secondary electron emission coefficient of material surface(s), electron energy, and RF frequency. For a number of technical reasons $A\ell_2O_3$ and BeO appear to be the best candidates for window material to date.[7] For reasons of safety $A\ell_2O_3$ is preferably used over the toxic BeO window material. On the other hand $A\ell_2O_3$ has a high secondary electron emission coefficient (on the order of 10 at the maximum of the energy-yield function). This high secondary emission coefficient and dimensional constraints imposed on RF window design compound the problem of multipactor. A typical SLAC high power klystron window is shown in Fig. 1. A transformation from the TE_{10} rectangular waveguide mode to a TE_{11} circular waveguide mode creates very complicated fringing fields in this structure. Problems of multipactor do not necessarily occur just at high power levels. Regions of multipactor shift in the window structure or completely disappear with changing power levels. This effect is illustrated in Fig. 2, which shows calorimetric power measured in the window water cooling jacket as a function of transmitted power in a microwave storage ring.

WINDOW COATING TO SUPPRESS MULTIPACTOR

In order to suppress multipactor, thin films of oxides of titanium have been used to coat windows for a number of years. This material has been shown to have a much lower secondary electron emission coefficient than $A\ell_2O_3$.[8] Both evaporation[6] and sputtering techniques[9,10] have been used in applying the material. Scientists at SLAC actively participated in much of this early work.[7,9,11,12]



A. High Purity Al_2O_3 Disk (~ 8.4 cm dia)
B. Cupronickel Sleeve, Brazed
C. Stainless Steel Jacket
D. Water Cooling Channel
E. Vacuum-RF Flange
F. Crushed Copper Gasket
G. Rectangular Waveguide (3.4 × 7.2 cm)

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FIG. 1--SLAC klystron microwave window.

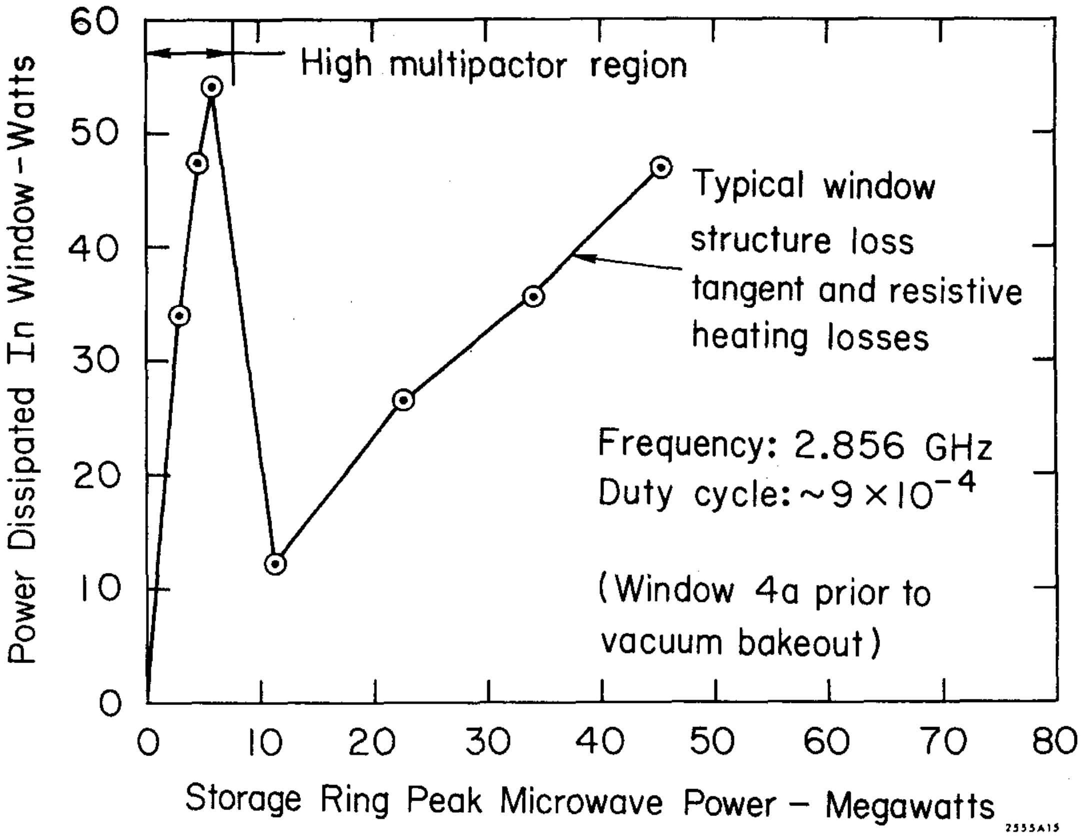


FIG. 2--Power dissipated in a typical Cr_2O_3 coated window as a function of RF storage ring power.

Subsequent success at SLAC has been largely due to rigorous control of procedures used in window coating. The procedures and associated equipment were the outgrowth of empirical work, and the technology did not readily translate to differing configurations. Careful control of film thickness was necessary, as films which were thick would result in resistive heating problems, and films which were too thin would not totally suppress multipactor. Also, there is some indication that properties of the reactively sputtered oxides of titanium may change with bakeout. For these reasons microwave window research work continues in the industry[13] and at SLAC.[1,14]

PROGRAM OBJECTIVES

Due to much higher power klystrons in the offing and the need to develop coating technology for a large ~~L-band~~ UHF klystron window in the early development stages, an independent window research program was commissioned to address the following tasks:

1. Refine RF ring testing procedures to yield quantitative data for "fine structure" comparison of window performance.
2. Test microwave windows offered by outside vendors and which feature promising new coating materials.
3. Investigate new possible multipactor suppression materials and application procedures.
4. Translate technology of conventional or newly developed materials and procedures for application to a generalized window configuration.

MICROWAVE RING TEST FACILITIES

Facilities did not exist to make calorimetric measurements of window losses during ring testing. Such measurements have been shown to be of value in evaluating window coatings.[13] The ring (microwave storage ring) was modified to include these provisions. Three possible water cooling configurations could be used to cool the window during tests. These are illustrated in Fig. 3. As shown, configuration "A" essentially involves cooling of the window assembly through conduction over long lengths of waveguide. Configuration "B" amounts to water cooling of the copper waveguide in close proximity to the window flanges. Lastly, configuration "C" amounts to direct water cooling of the window assembly as well as the nearby waveguide, as in "B". When using the "C" cooling configuration, power dissipation in the window water cooling jacket was measured

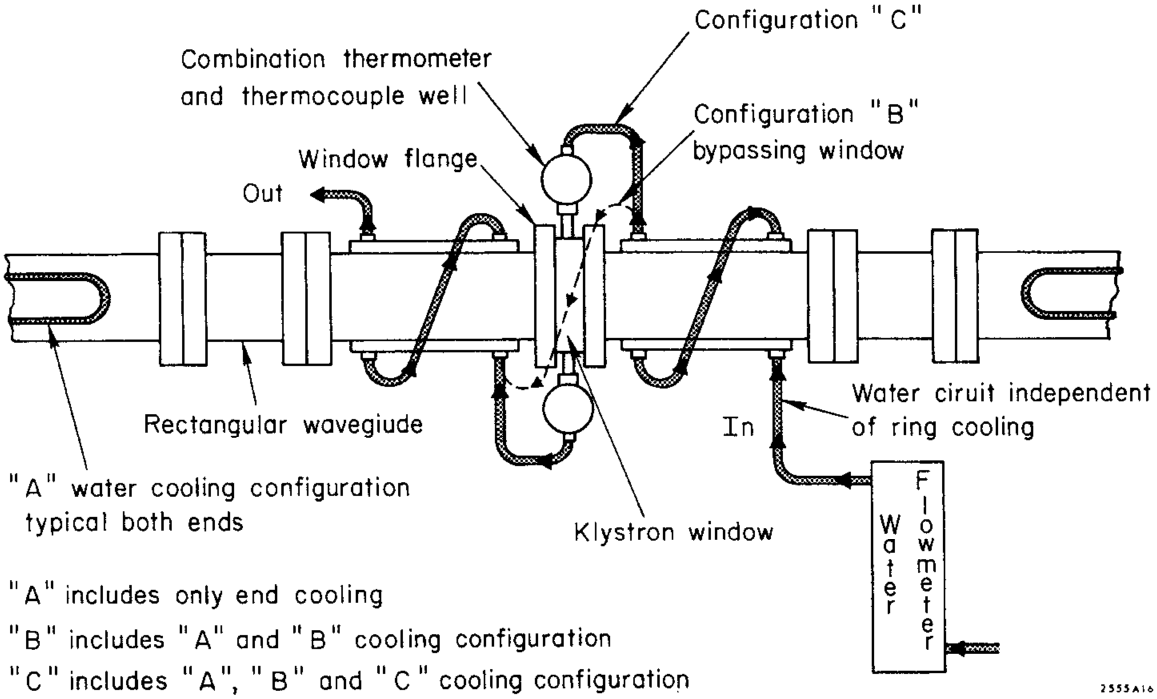


FIG. 3--RF test ring window cooling configurations.

using both mercury thermometers and thermocouples, while water flow was measured with a Brooks flowmeter (500 cc/min. full scale). Due to thermal losses from the system, calorimetrics serve only as a relative measure of window merit. However, we achieved excellent repeatability and were able to predict probable window failure and cause of same using this technique. In over 1000 simultaneous thermocouple and thermometer water temperature measurements the difference in window power dissipation indicated by the two temperature measuring methods amounted to 2.6 watts ($\sigma = \pm 2.5$ watts) and was random. Figure 4 shows this microwave ring test facility.

Provisions existed for measuring temperature of the center of the window through view ports in the ring using an IR detector. However, temperature calibration problems occur with changes in the emissivity of window materials, view port transmission, and instrument adjustments. Also, there are problems of interpretation of indicated temperatures for different water flow rates and cooling configurations. A method was established for normalizing "out" the effects of changes in emissivity of the various windows. This involved integrating the temperature function in the manner illustrated in Fig. 5. Coordinates are translated for the data of each scanning so that the temperature minimum at the window edge is taken as zero. The integral then represents only the area shown "hashed" in the above figure. The effects of change is spectral emissivity with temperature variations across the window are second order in this normalized integral. Also, changes in the value of the normalized integral with emissivity settings of the IR detector amount to only 16% over the range of 0.2 - 0.6. The normalized integral of the temperature function qualitatively describes the degree of "peakedness", or kurtosis of the function. It has been shown that relative kurtosis is an excellent indication of window quality.[14] When used in conjunction with calorimetric data, it is particularly useful in finding the cause of window heating. A kurtosis value $\leq 50\,^{\circ}$C-cm at 50 kW average power is acceptable. Kurtosis values ≥ 150 to $200\,^{\circ}$C-cm indicate serious window problems at 50 kW average power or less.

The storage ring was calibrated by direct insertion of a special flanged bimetal waveguide section.[15] The bimetal section was first calibrated up to an average power of ~ 50 kW by calorimetric measurements on insertion in the output waveguide of a high power klystron with a water load. It was discovered that

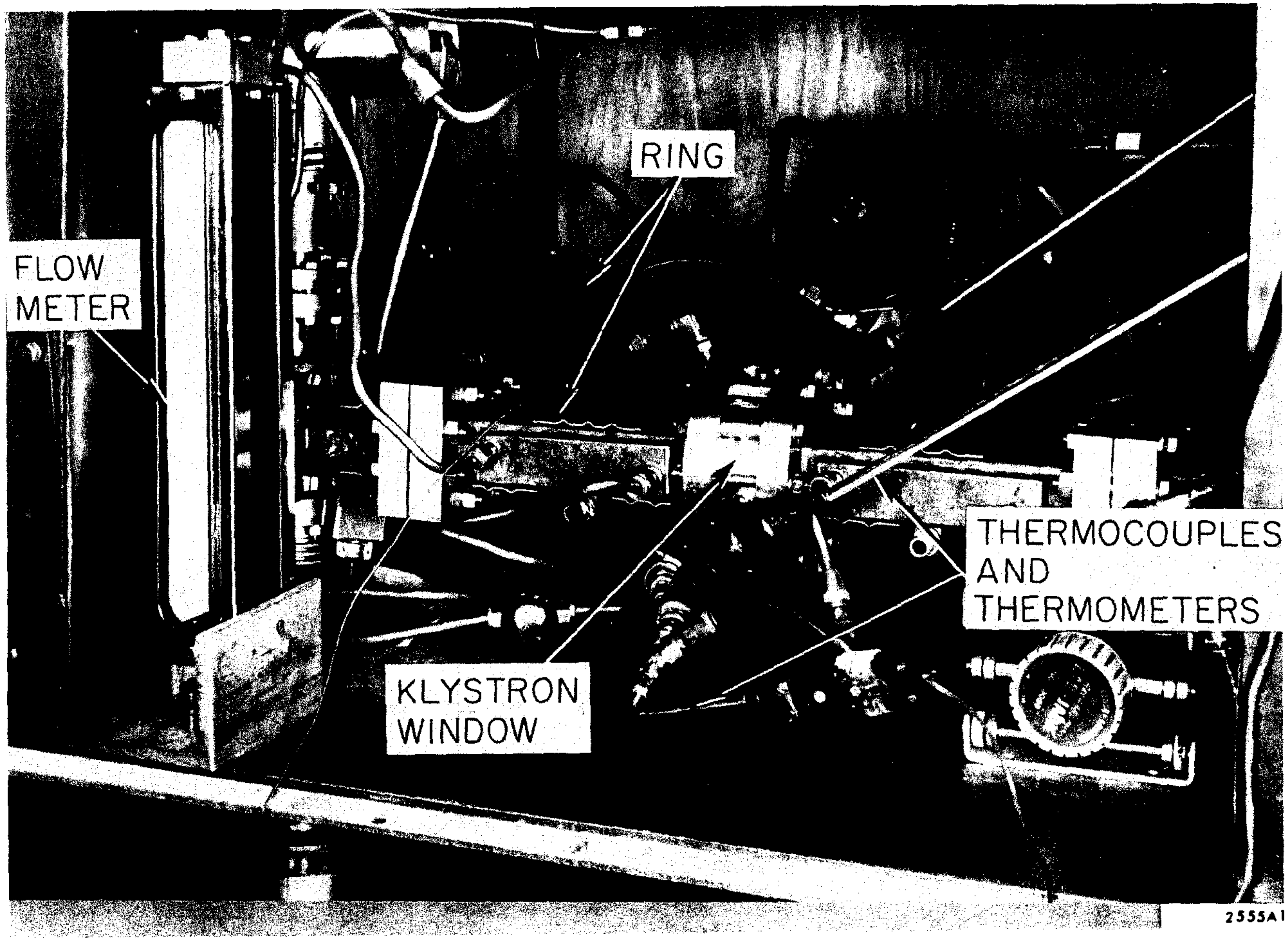


FIG. 4--High power microwave storage ring test facility.

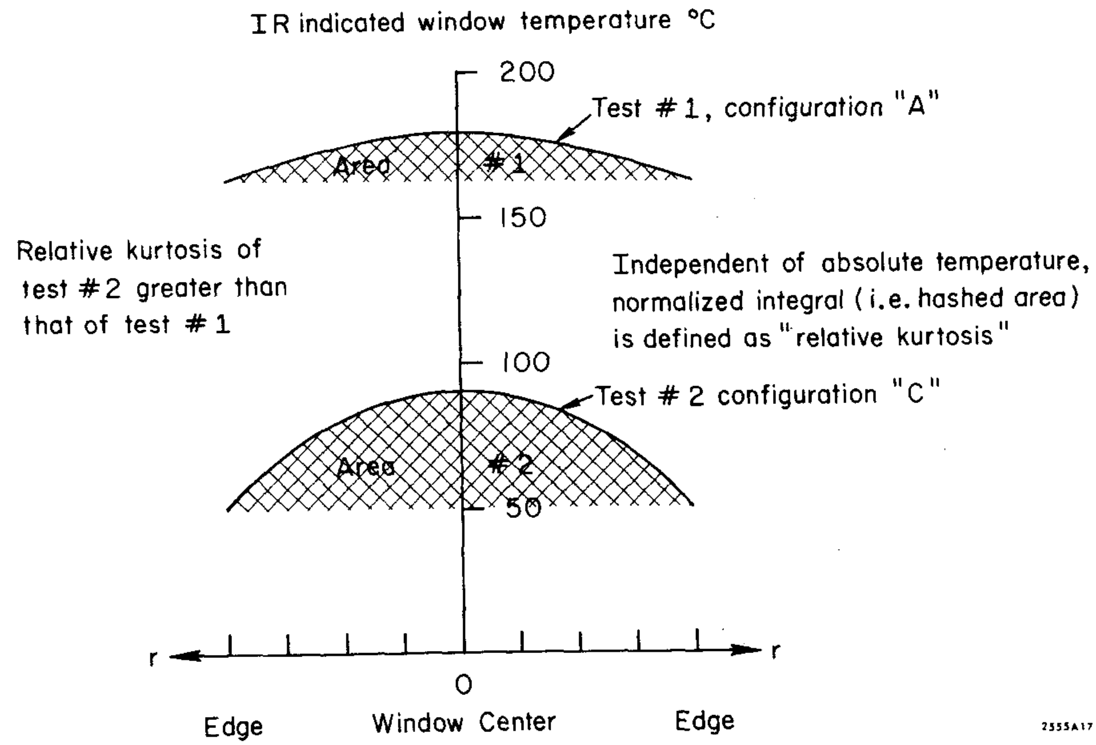


FIG. 5--Kurtosis as defined in report.

ring power levels were actually ~17% lower than originally thought. The new value is probably correct to within ±5%.

COMPARISON OF Cr_2O_3 COATED WINDOW WITH STANDARD SLAC WINDOWS

The first three months of the program were spent refining window testing methods, establishing "normal" window behavior as measured with these new testing methods, and evaluating a number of windows coated with Cr_2O_3 (submitted for evaluation by Litton Industries, Inc., San Carlos).

The statistics of "normal" standard SLAC coated windows, both before and after the typical 625°C vacuum bakeout, are shown in Fig. 6. This data represented an adequate statistical sample to serve as a "bench mark" in all future work. Window data taken during hot test of a klystron are also shown in this figure. Window degradation due to bakeout is suggested in the data.

Having established "normal" window behavior, we sought to quantify extremely "abnormal" window behavior with the tools at hand. Tests were therefore conducted on one window with no coating and a second window with standard coating only on the cupronickel sleeve (see Fig. 1). These test data are shown in Fig. 7. They graphically demonstrate the merit of window coating to suppress multipactor.

Windows coated with Cr_2O_3 films of varying thickness ("fog", 0.025 mm, and 0.075 mm) were RF tested before and after bakeout. Detailed results of these extensive tests are reported elsewhere.[14] However, typical performance data before and after bakeout are shown in Figs. 8 and 9 respectively. These data do not agree with results reported elsewhere,[13] where, (1) direct measurements of the secondary electron emission coefficients indicated Cr_2O_3 and oxides of titanium were essentially identical, and (2) it was concluded that Cr_2O_3 "adequately suppressed multipactor", based on test data. Results of data in Figs. 8 and 9 would indicate to the contrary. This result may be largely due to differences in the geometry of the windows tested. This is further indicated by the comparitively small difference in performance of uncoated windows and Cr_2O_3 coated windows reported at that time.

Test results at SLAC on windows coated with this material indicated the following: (1) there is inadequate suppression of multipactor at low power levels independent of window coating thickness, (2) there is poor reliability or quality control in window coating as suggested by failure at the offing of one fogged, one

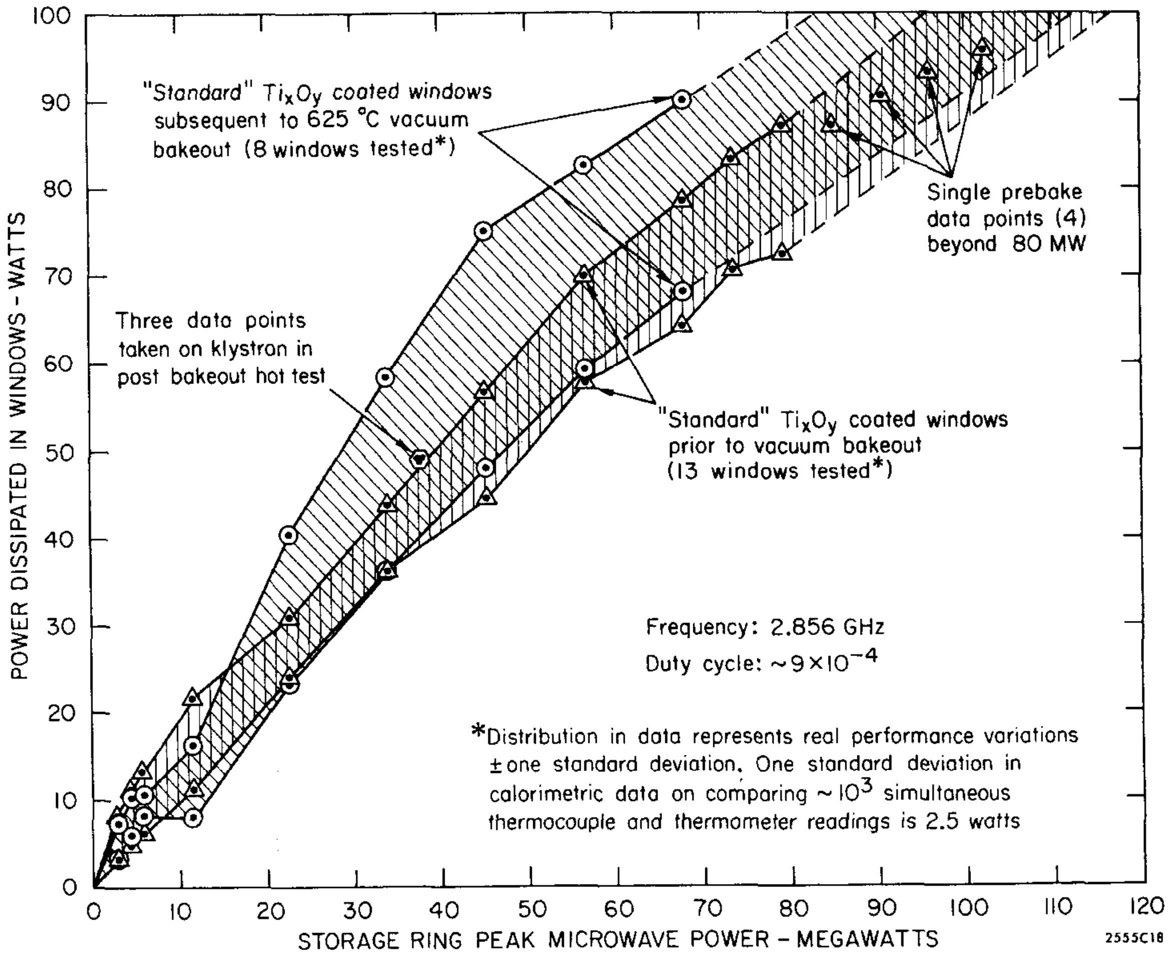


FIG. 6--Power dissipated in windows with standard Ti_xO_y ac sputtered coating before and after 625°C vacuum bakeout.

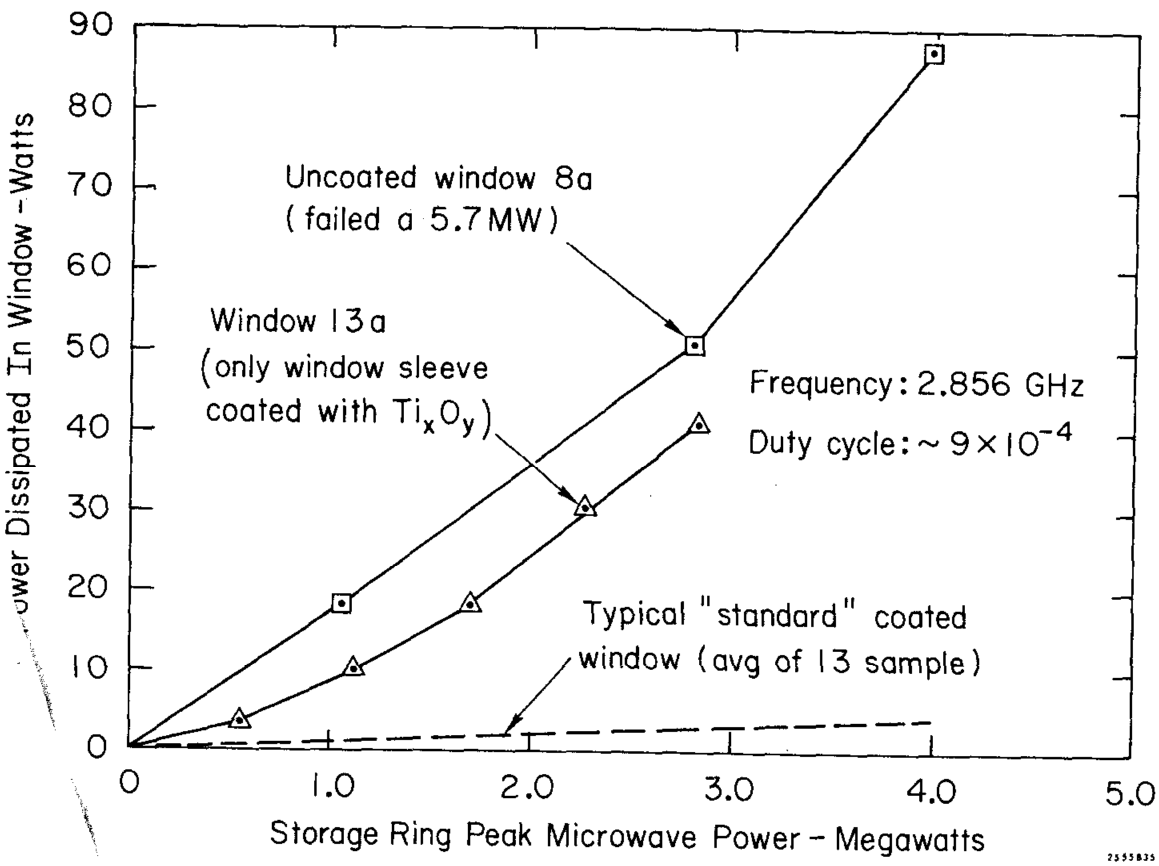


FIG. 7--Power dissipated in windows without coating.

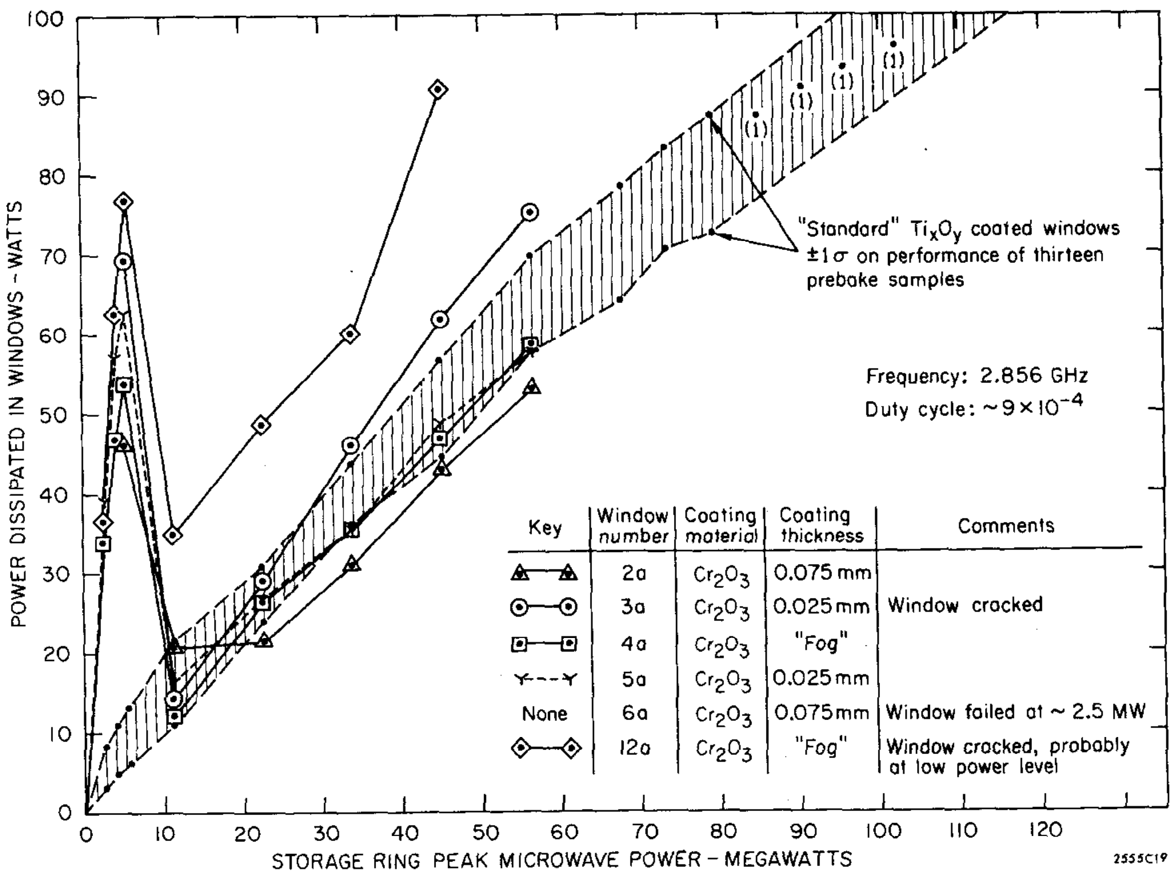


FIG. 8--Power dissipated in windows coated with Cr_2O_3 compared with standard Ti_xO_y coated windows prior to vacuum bakeout.

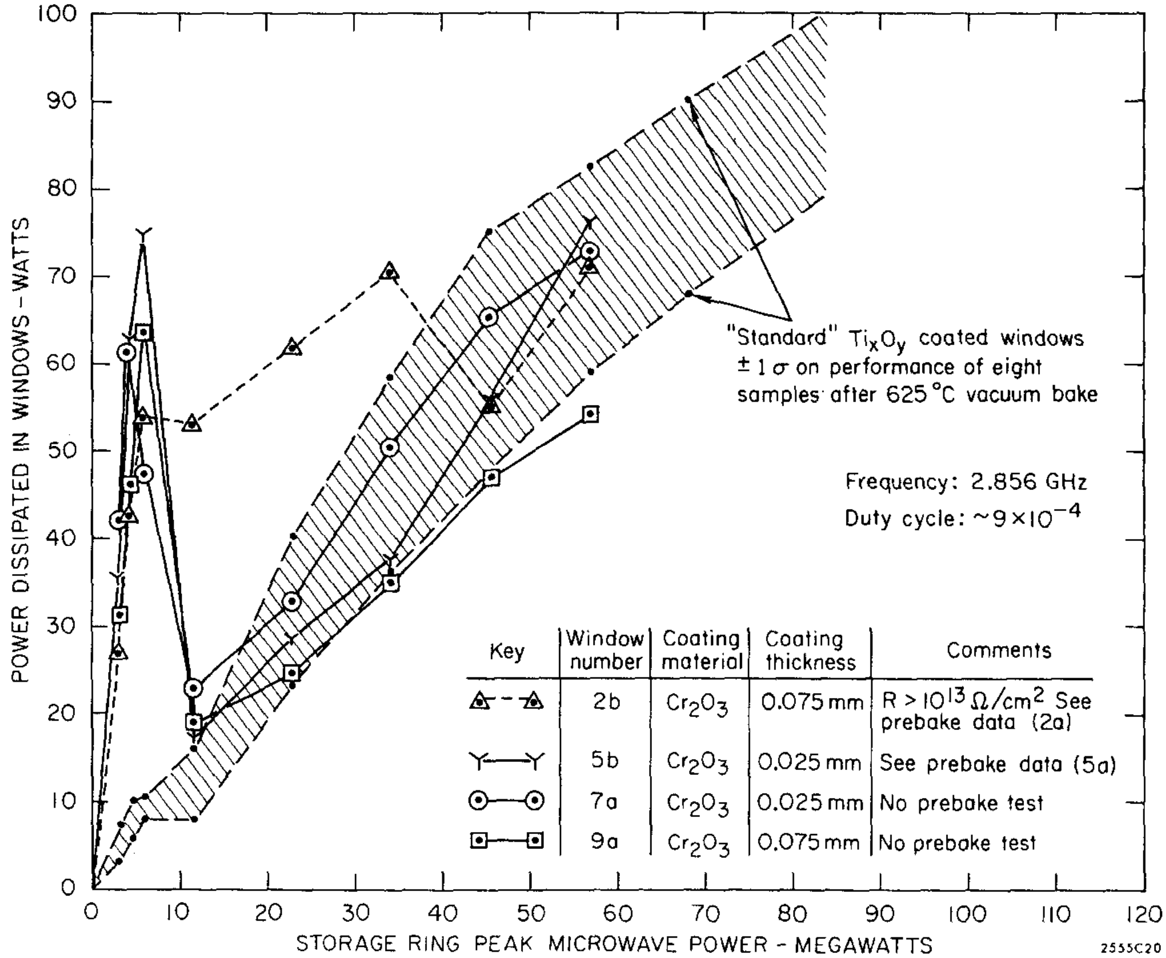


FIG. 9--Power dissipated in windows coated with Cr_2O_3 compared with standard Ti_xO_y coated windows after 625°C vacuum bakeout.

0.025 mm, and one 0.075 mm thick coating window, (3) there appears to be a deterioration in degree of multipactor suppression subsequent to bakeout, (4) the chromium oxide coating is not reduced to a partial metallic state as a consequence of high temperature vacuum baking, (5) the loss tangent properties of the material are excellent as evidenced by negligible differences in power dissipated in windows with three different film thicknesses (pre-bakeout data), and lastly (6) the dielectric strength of Cr_2O_3 is not adequate for high power window application as evidenced by craters and "worm-like" eruptions which existed to varying degrees on all of the windows tested.

A NEW APPROACH TO WINDOW COATING

It occurred to the writer that titanium nitride was thermally much more stable than oxides of titanium. Therefore, if it proved to have satisfactory secondary emission properties as well as thin film resistivity comparable to oxides of titanium, it might then prove to be a superior window coating material. Experiments really became "mixed" when it was decided to use a newly invented coating apparatus to deposit the TiN films as well as films of Ti_xO_y (i.e., complex oxides of titanium) on test windows. This coating apparatus was the outgrowth of research in low pressure crossed field electrical discharges at SLAC over a number of years,[1,16-19] and it was coined a crossed field diode sputtering array.

A double array of diodes was constructed for a "first try" at (1) use of a new crossed field sputtering scheme in deposition of thin films, and (2) reactive sputter coating of TiN films for the suppression of multipactor. A window was placed between these arrays for coating as shown in Fig. 10. The assembly, referred to as the first generation apparatus, was placed in a bell jar and evacuated to $\sim 10^{-7}$ Torr. The window was then coated using an anode potential of +3.5 kV and a magnetic field of $\sim$0.06 T for a period of 5 minutes at an indicated pressure of $\sim 5 \times 10^{-5}$ Torr. A gas mixture of 90% O_2, 10% A was used in this initial coating. The cathode plates were removed from the diode arrays, new plates installed (a precaution taken in all subsequent experiments), and a second window coated. During this coating operation a gas mixture of 90% N_2, 10% A was used.

Inspection of both windows indicated significant nonuniformity in thickness. The anode array pattern was transposed, to some degree, onto the surface of the

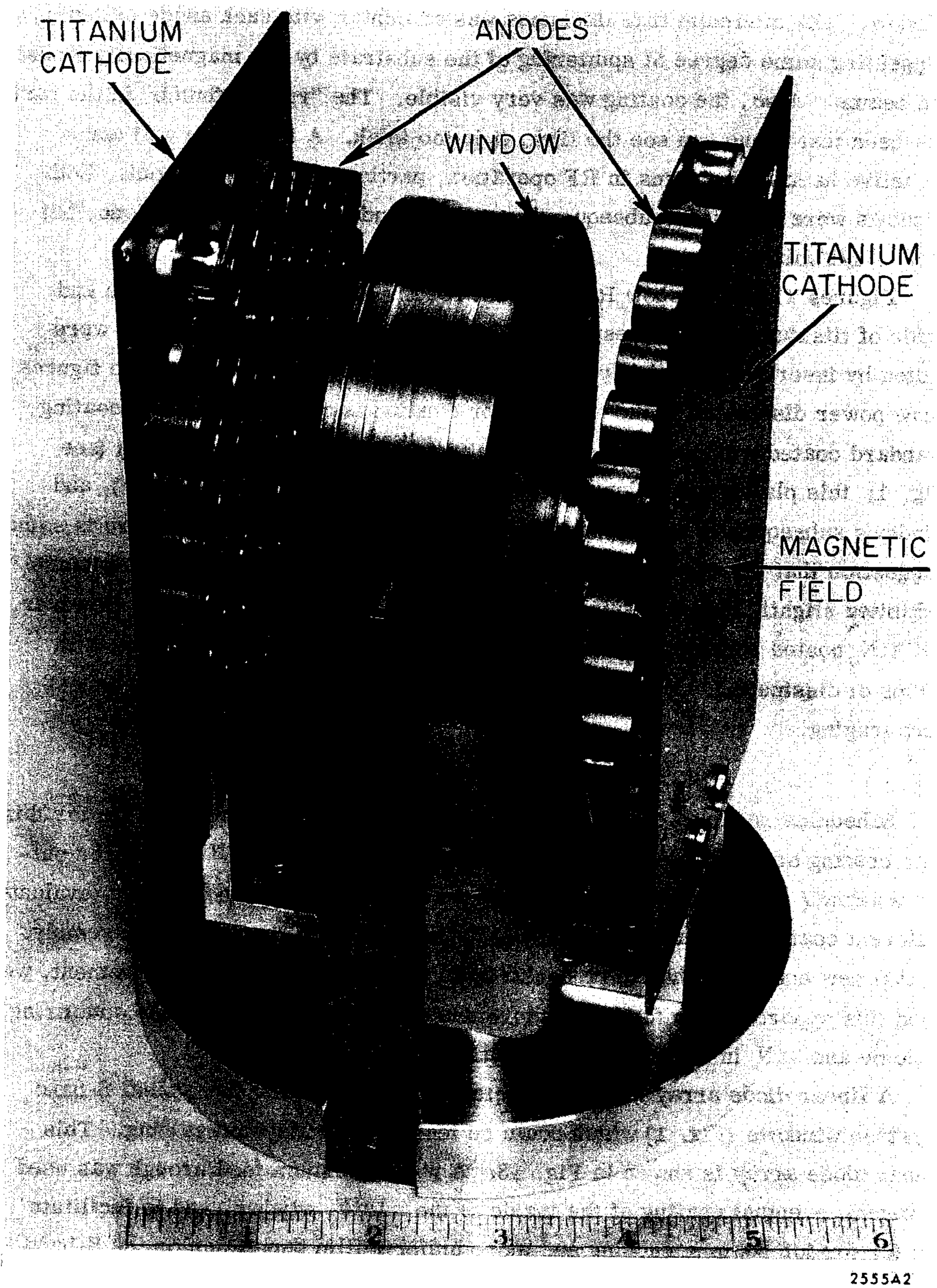


FIG. 10--Microwave window positioned in first generation sputtering apparatus.

window. The minimum film thickness was on center with each anode cylinder, suggesting some degree of sputtering of the substrate by the magnetically focused ion beams. Also, the coating was very visible. The "rule of thumb" in the past has been that if one can see the film, it is too thick. A thick film will cause resistive heating problems in RF operation, particularly after bakeout. Both windows were therefore subsequently recoated under the same conditions, but for a 1-1/2 minute period.

Figures 11 and 12 show RF ring test results for the titanium nitride and oxide of titanium coatings respectively. The windows herein discussed were tested by insertion in a high-power microwave storage ring. The above figures show power dissipated in the windows for conditions of standard SLAC coating, standard coated windows with copper plating on the cupronickel sleeves (see Fig. 1; this plating operation was a simultaneous development activity), and windows subsequently coated with the first generation apparatus. There is some suggestion that the window coated with $Ti_x O_y$ in the first generation apparatus exhibited slightly higher dissipation losses than the standard window. However, the TiN coated window appeared equivalent to the standard coated window in terms of dissipation. Initial results, though far from conclusive, were most encouraging.

SECOND GENERATION APPARATUS

Schedules required that an apparatus be developed in the near future for thin film coating of the inside surface of a cylindrical, $A\ell_2O_3$, UHF klystron output window (~ 15 cm dia. × 15 cm long). Test facilities did not exist to evaluate different coatings prior to commitment of the window to the klystron assembly. In that new equipment had to be constructed to satisfy the above requirement, we used this opportunity to further explore use of the crossed field diode sputtering scheme and TiN in suppression of multipactor.

A linear diode array was constructed, and used to coat the typical S-band klystron windows (Fig. 1) which could be tested in the RF storage ring. This linear diode array is shown in Fig. 13. A rotary vacuum feedthrough was used to permit eventual coating of the inside of the UHF window, and to facilitate motion for ion beam scrubbing and more uniform film deposition on the S-band windows. When coating the disk windows, the diode array was spaced from the surface of the window a distance equivalent to that spacing which would exist on

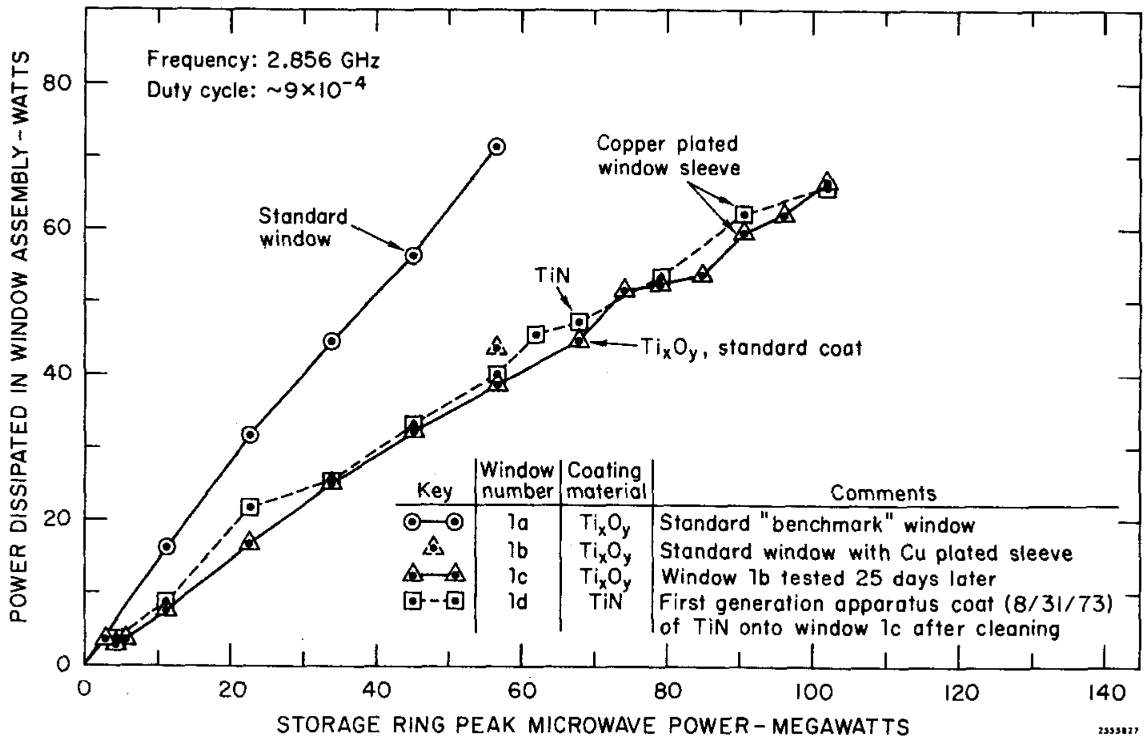


FIG. 11--Power dissipated in the same window with standard Ti_xO_y coat, with and without Cu plate on window sleeve and with first TiN coating.

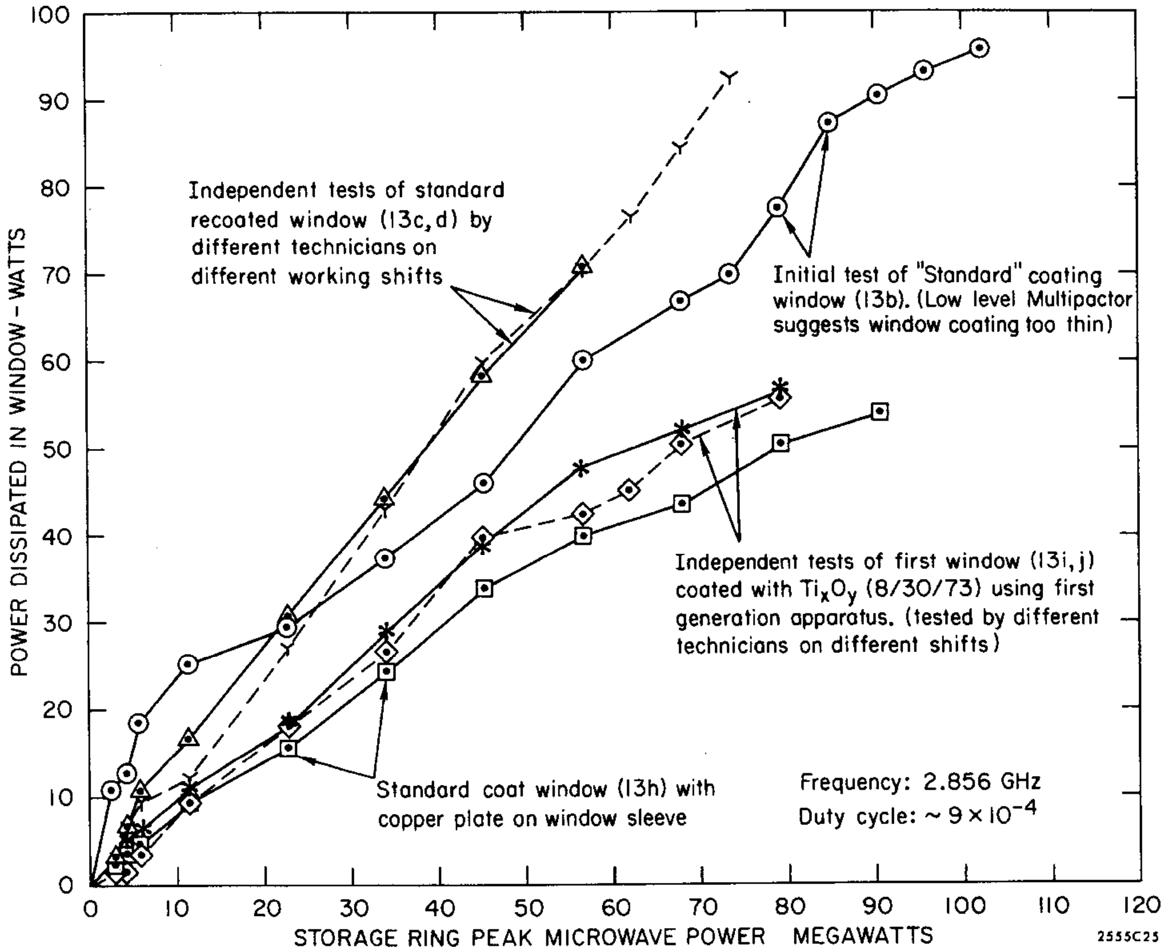


FIG. 12--Power dissipated in window with and without copper plating on sleeve and with standard coat and first Ti_xO_y coating using first generation apparatus.

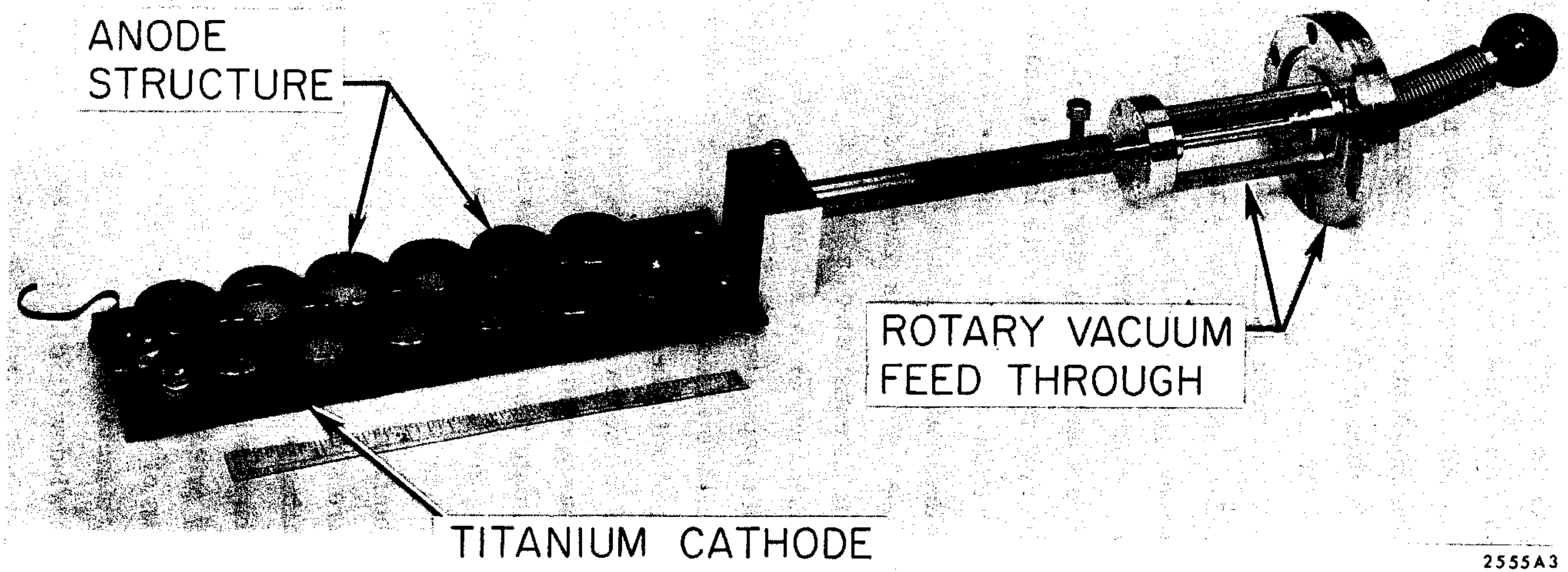


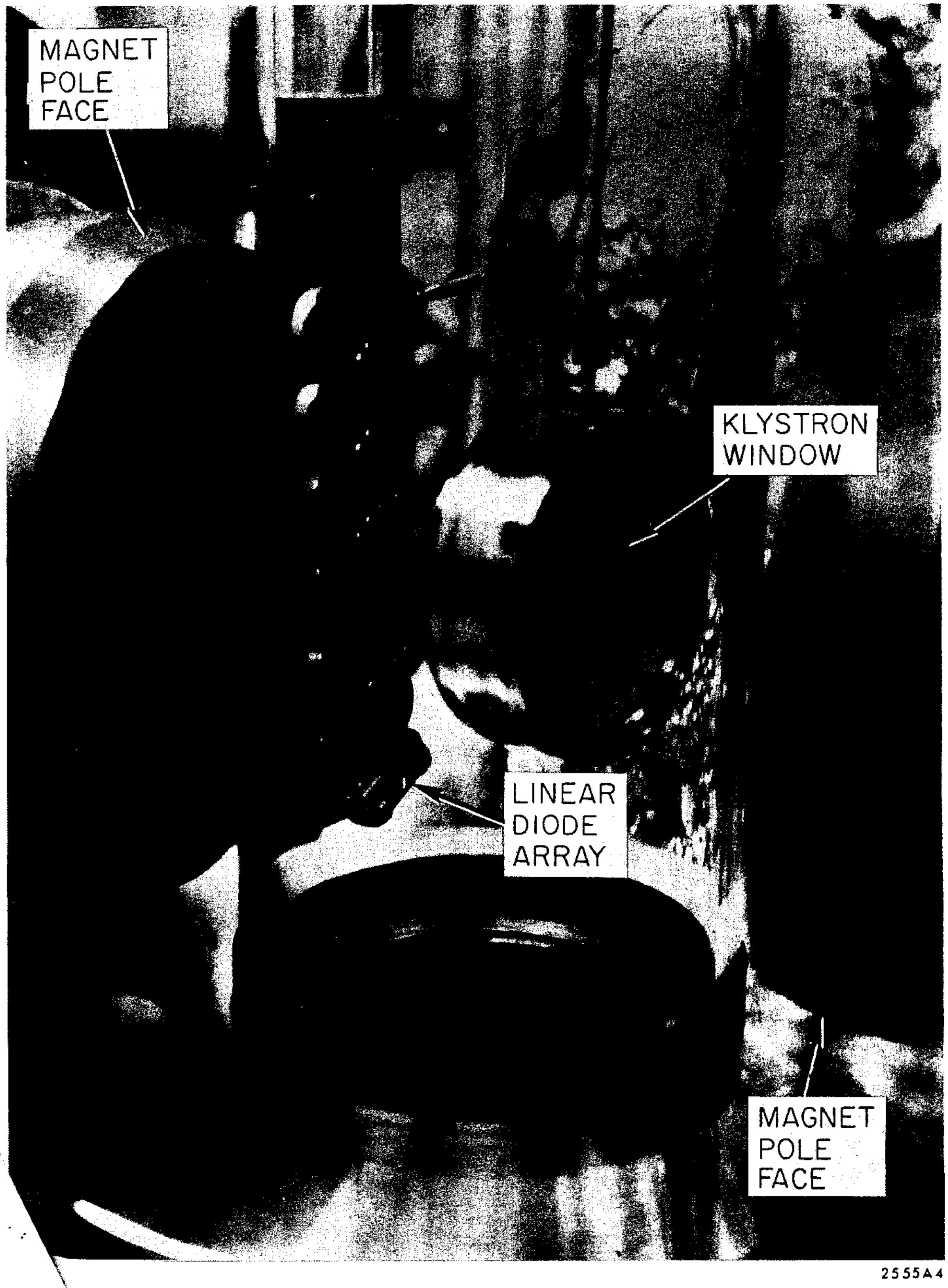
FIG. 13--Linear "half" diode sputtering array.

insertion of the array in the cylindrical output window. Figure 14 shows the smaller klystron window and linear diode array in a vacuum bell jar. The windows were also attached to a rotary feedthrough to permit sequential coating of each side without venting of the vacuum system.

A second bell jar sealing plate was also constructed to facilitate evaporation coating of thin films of TiN and Ti_xO_y onto S-band windows, and with the same flexibility for possible subsequent use in coating the UHF windows. This apparatus is shown in Fig. 15. Numerous window coating experiments were conducted using the above apparatus with the previously mentioned gas mixtures, as well as laboratory air and pure argon.

During early RF tests on the many standard SLAC windows, an aging effect was noted. It was observed that there were large variations in power dissipation in any particular window from hour to hour and even day to day. We subsequently determined that stability could be achieved in window performance by aging for one hour at ~50-60 MW peak power and with water cooling configuration "B". This effect is demonstrated in Fig. 16. We thereafter performed this aging operation on all windows on initial testing. The Cr_2O_3 windows previously discussed, and statistics generated on the standard windows reflect data taken after this aging ritual where window performance permitted. We did not determine if this was required with the TiN coated windows. Data in Figs. 11 and 16 are good indications of the repeatibility we achieved in our measuring technique. Repeated tests of the same window by different technicians were done without their knowledge to evaluate the overall statistics of our measurement technique. Results were gratifying and a tribute to the care exercised by these technicians in measurement technique.

Test results of the first four windows coated using the linear diode sputter array, and nitrogen are shown in Fig. 17. Calculations were made in an effort to predict window coating thickness. These calculations predicted a coating thickness of several hundred angstroms on the first window which was coated using the linear array (window No. 14b). In an effort to qualitatively evaluate loss tangent properties of the material, we increased the coating thickness of the subsequent window by a factor of ~3.7. As shown in Fig. 17, losses were slightly higher in this window (17c). To make sure that this variation was not due to intervening variables, window 17c was cleaned and recoated with slightly



G. 14--Linear sputtering array apparatus for L-band window coating feasibility studies.

FIG. 15--Filament evaporation coating apparatus.

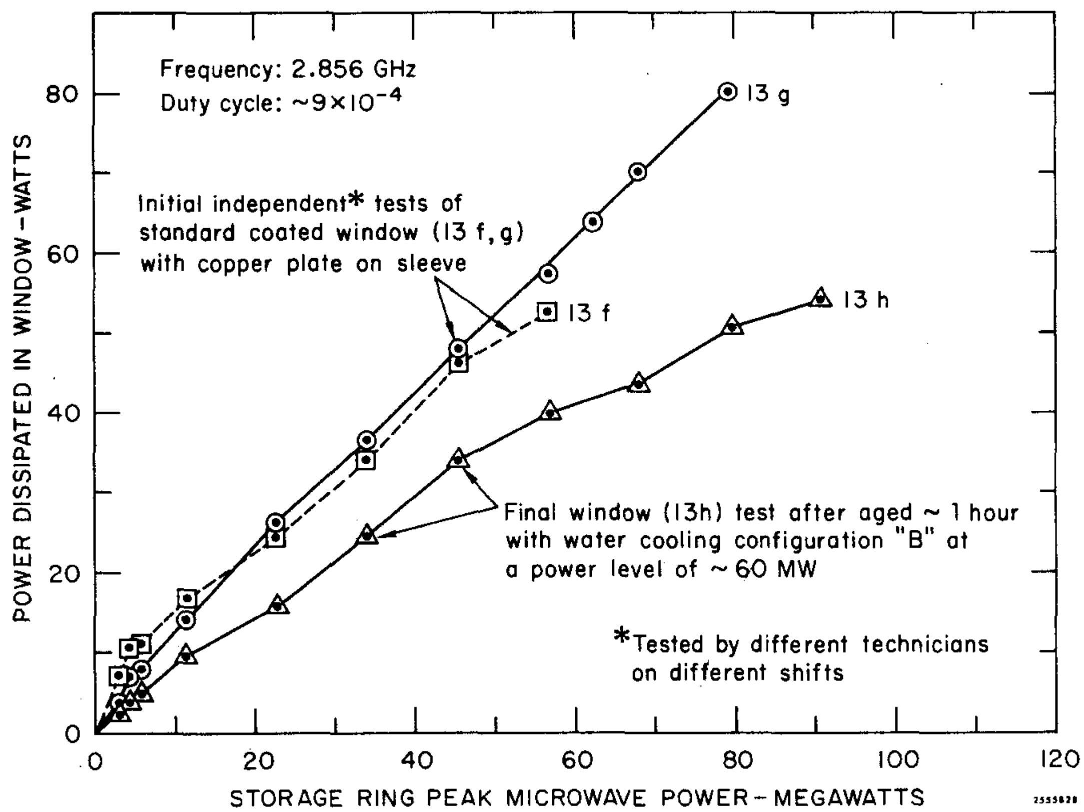


FIG. 16--Window aging effects during RF testing.

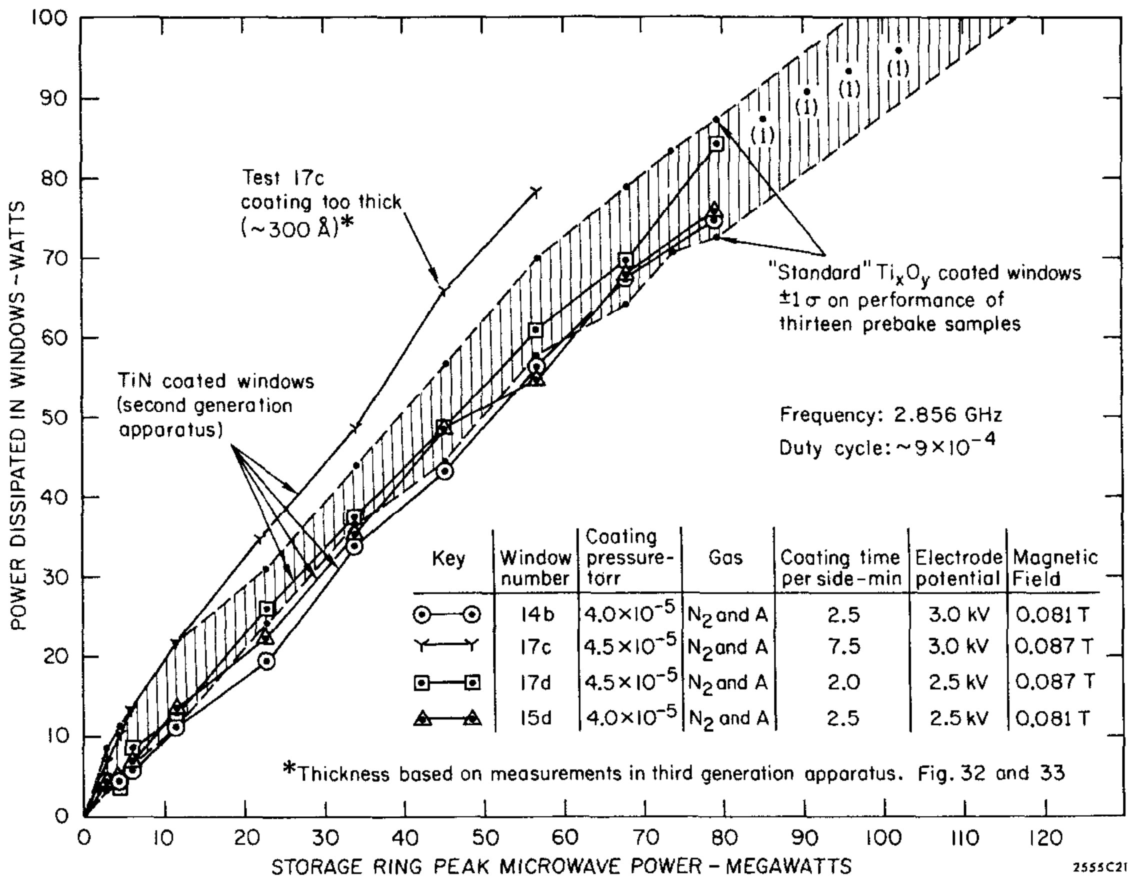


FIG. 17--Power dissipated in windows coated with titanium nitride in second generation apparatus compared with standard Ti_xO_y coated windows prior to vacuum bakeout.

less coating (3% by appropriate scaling factors) than used with window 14b. The recoated window is numbered 17d, and performed similar to 14b, as also shown in Fig. 17. A third window was coated and verified the repeatibility and merit of the initial coating parameters (15d).

Three things were made evident by the above tests: (1) we were very lucky in our initial choice of coating parameters; (2) window performance does depend on TiN coating thickness; and (3) the coating procedure suggested good repeatibility.

Test data on these windows subsequent to bakeout are shown in Fig. 18. It was determined that window 15d had been contaminated with diffusion pump oil during bakeout. However, data are included for the value of this lesson alone.

Experimental results of windows coated with TiN and using evaporation techniques are shown in Fig. 19 (prebakeout) and Fig. 20 (post bakeout). These windows appear to run hotter both before and after bakeout than the windows coated with TiN and using the diode array system. It is much more difficult to control film thickness when using evaporation techniques, and one might therefore suspect that the coating was too thick. Results of window No. 16 in Fig. 19 suggest to the contrary. The low power multipactor peak in window 16c suggests the coating is too thin. On subsequent recoating the low level peak disappeared (window No. 16d, of Fig. 19) until after bakeout (window No. 16e, of Fig. 20). Therefore, in all of the above cases, there is some suggestion that the window coating was not sufficiently thick.

Table I lists the various combinations subsequently used in coating windows with the linear diode array system and evaporation filament. Subsequent sputtering with the linear diode array system and the O_2-A gas mixture gave very unsatisfactory results — to the point where calorimetric data could not be taken due to ring matching problems. This was inexplicable considering initial performance with the first generation apparatus. Windows were not coated by sputtering in argon with the linear array system but, all other coating combinations are included in Table I, with figures containing corresponding data.

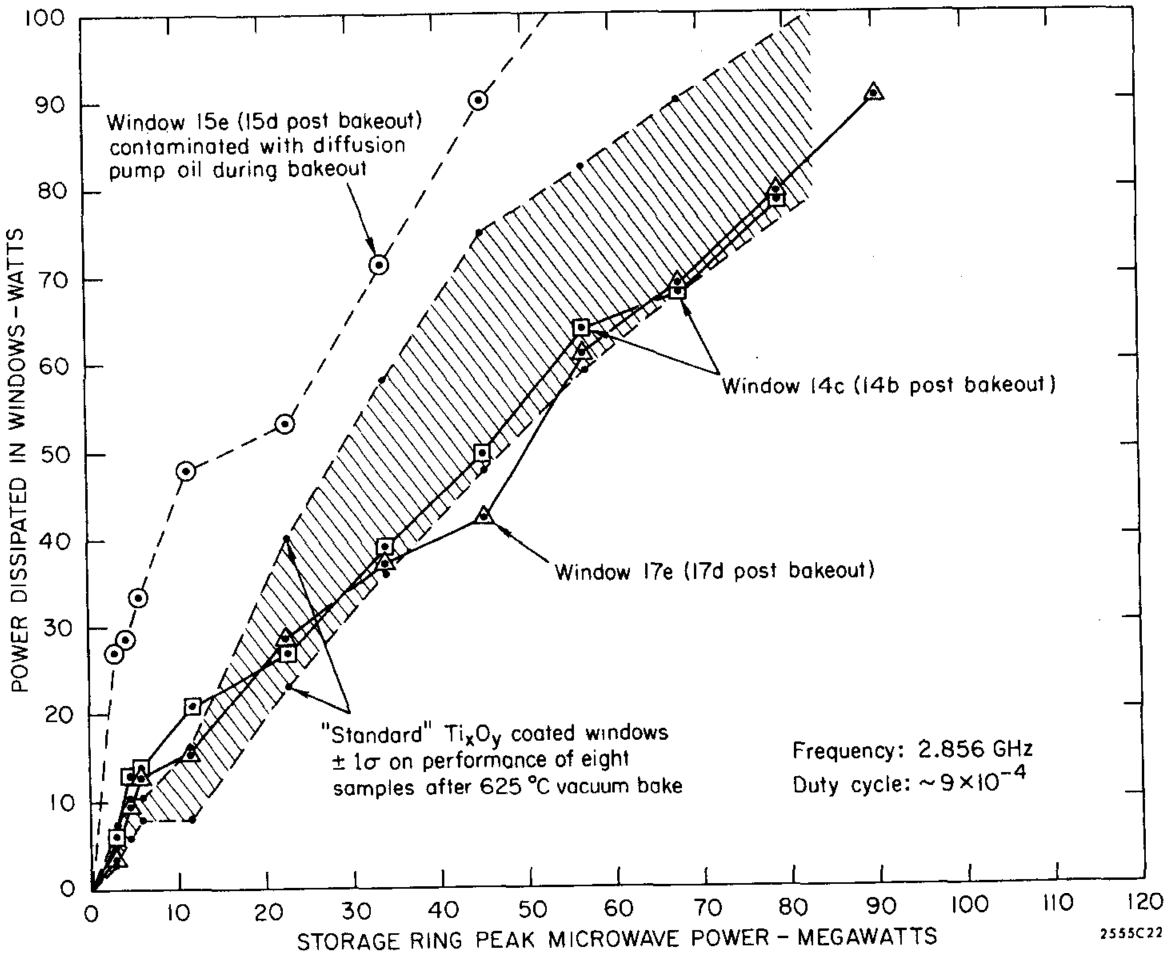


FIG. 18--Power dissipated in windows coated with titanium nitride in second generation apparatus compared with standard Ti_xO_y coated windows after 625°C vacuum bakeout.

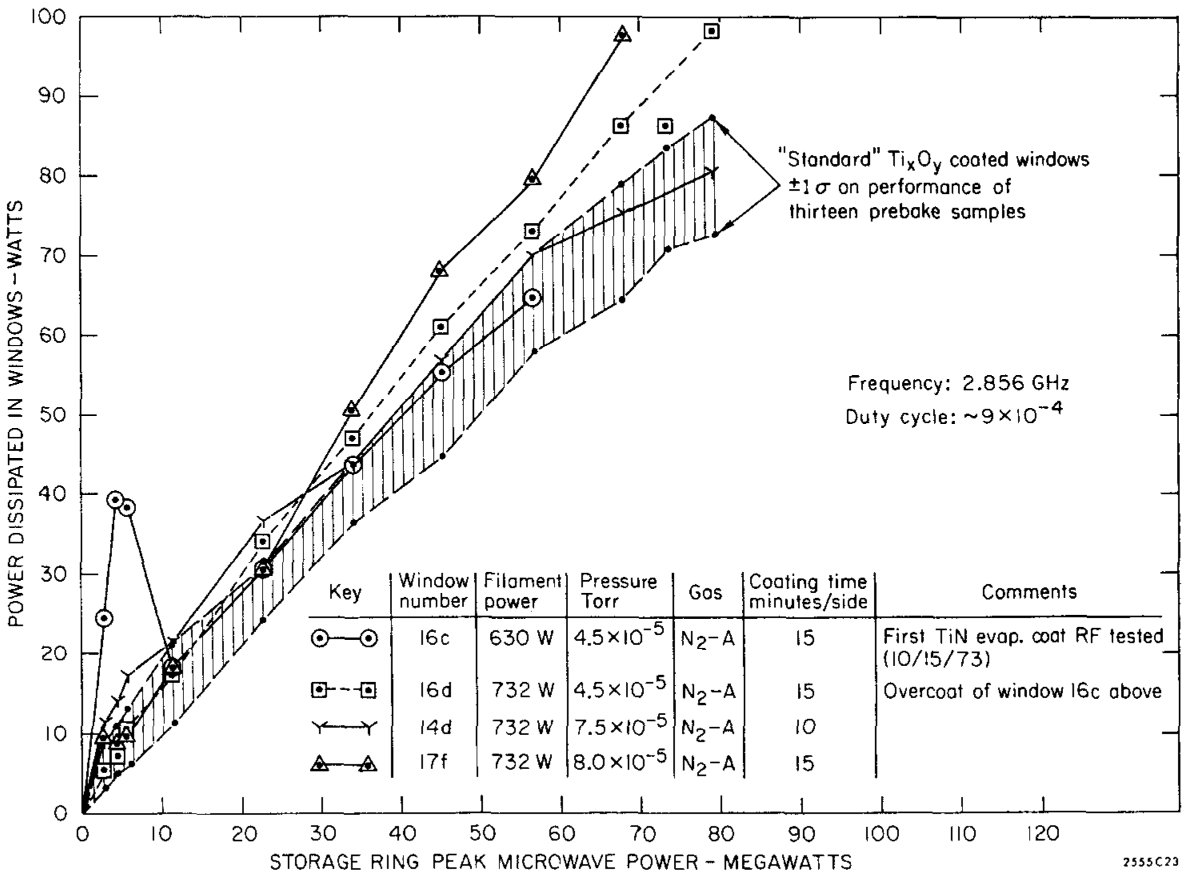


FIG. 19--Prebakeout comparison of power dissipated in standard Ti_xO_y coated windows with windows coated with titanium nitride by evaporation filament.

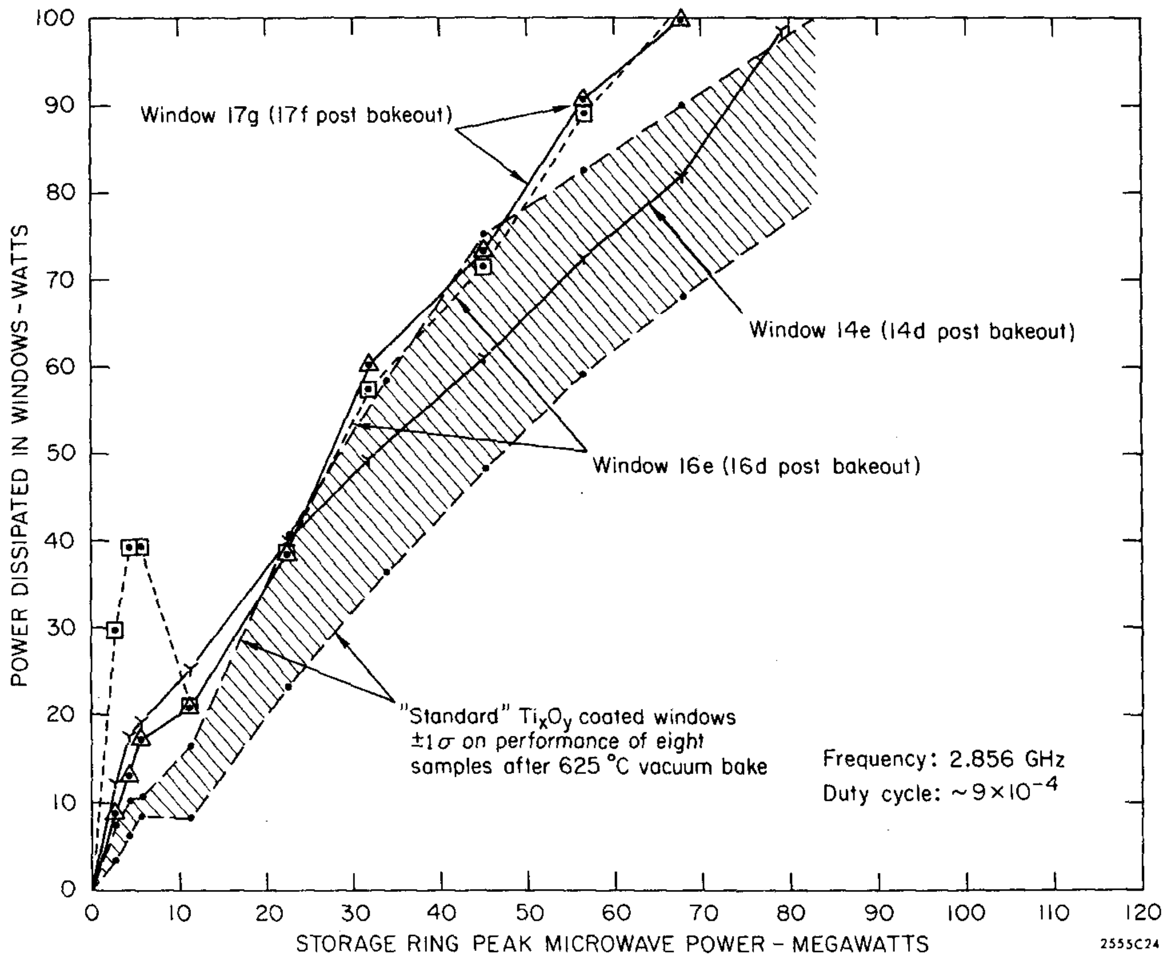


FIG. 20--Post bakeout test results of windows evaporation coated with titanium nitride compared with standard Ti_xO_y coated windows.

TABLE I

Window No.	Coating Medium			Apparatus		Bakeout		Figure
	N_2-A	A	Air	Sputter	Evap.	Before	After	
15a		X		(Standard)		X		21
15c			X		X	X		21
15d	X			X		X		21
17a		X		(Standard)		X		22
17b		X		(Standard)			X	23
17d	X			X		X		22
17e	X			X			X	23
17f	X				X	X		22
17g	X				X		X	23
19a		X		(Standard)		X		24
19b		X		(Standard)			X	25
19c			X	X		X		24
19d			X	X			X	25
19e		X			X	X		24
19f		X			X		X	25

Based on data thus far given, indications are that windows coated with TiN and the linear diode array gave the best results. There is some suggestion that a window sputter coated in a partial pressure of air would perform very satisfactorily both before and after bakeout. However, this was based on tests of only one window (19c and 19d). It was proposed by the writer that sputtering of titanium nitride be used in future applications due to the high control evidenced by the data, and overall satisfactory window performance.

The linear diode array was subsequently used to coat the cylindrical UHF window with TiN. A swivel magnet was constructed to permit proper orientation of the magnetic field with the diode array when coating the inner surface of the cylindrical window. The apparatus is shown in Fig. 26 with the UHF window removed from the system. This same apparatus is shown with the window installed in Fig. 27. The window served as the vacuum bottle during coating. Windows were also subsequently coated with the evaporation filament shown in Fig. 15.

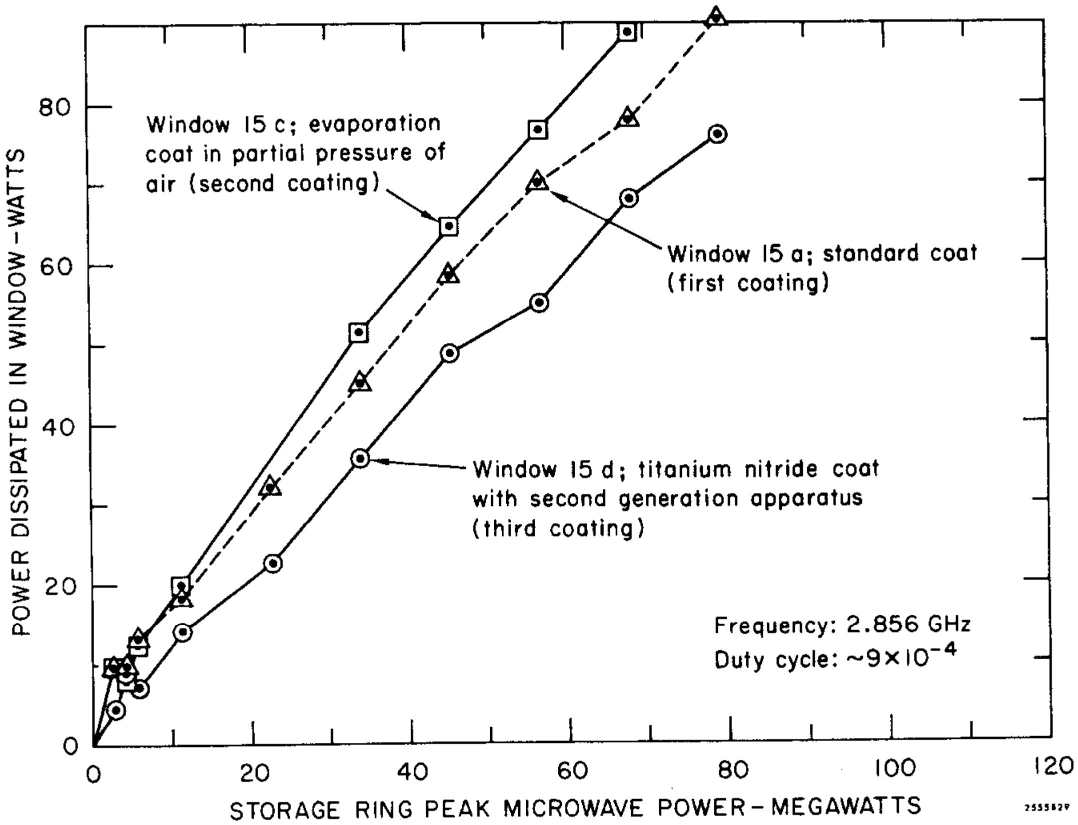


FIG. 21--Power dissipated in window number 15 as a function of sequential coating with different materials.

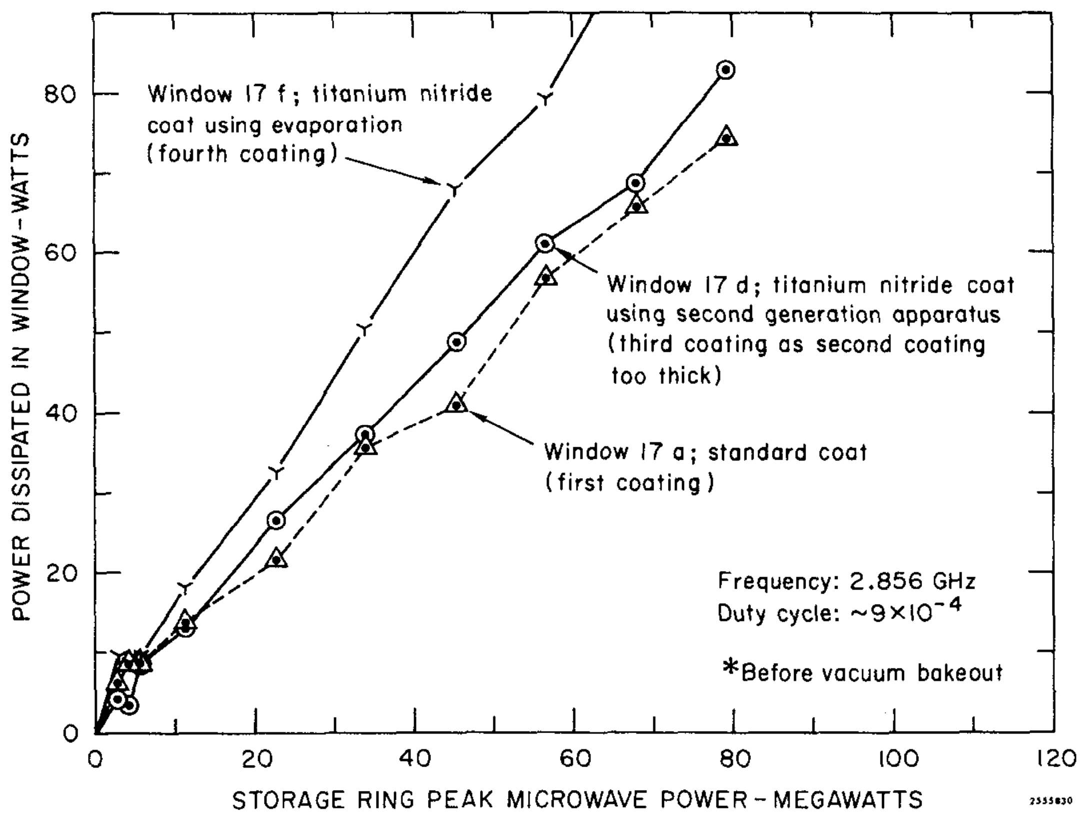


FIG. 22--Power dissipated in window number 17 as a function of sequential coating with different materials.

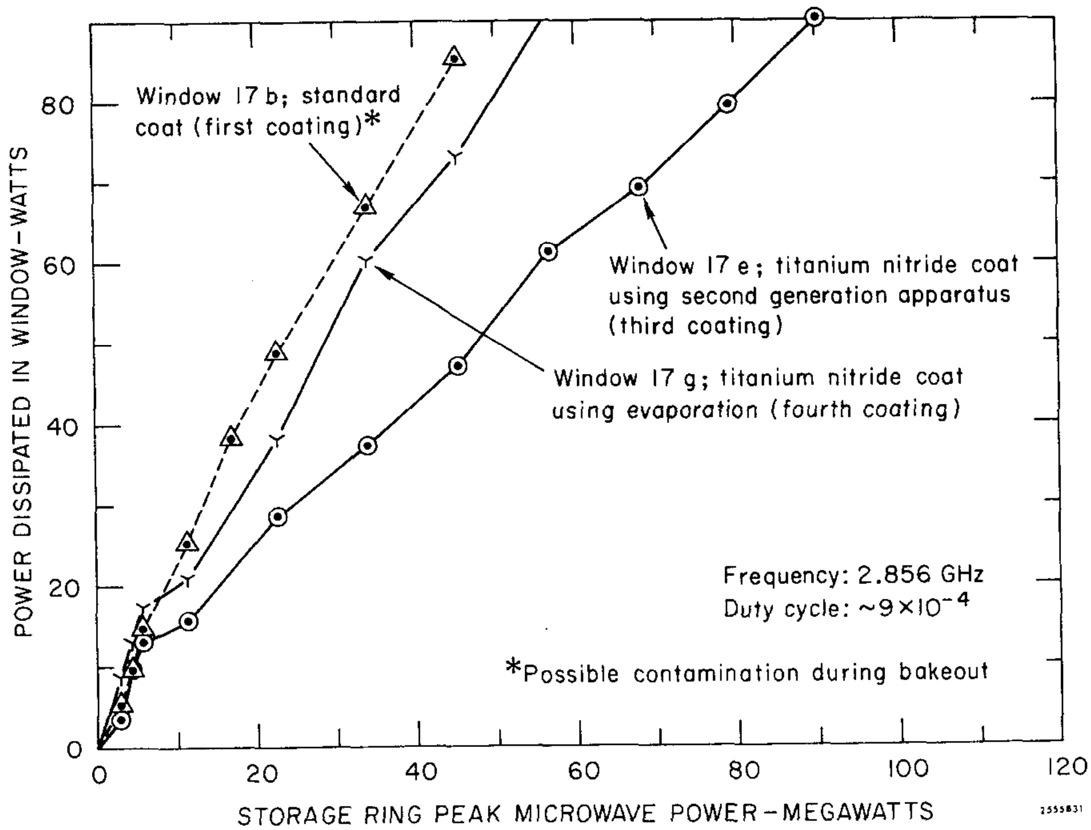


FIG. 23--Power dissipated in window number 17 on post bakeout test as a function of coating with different materials.

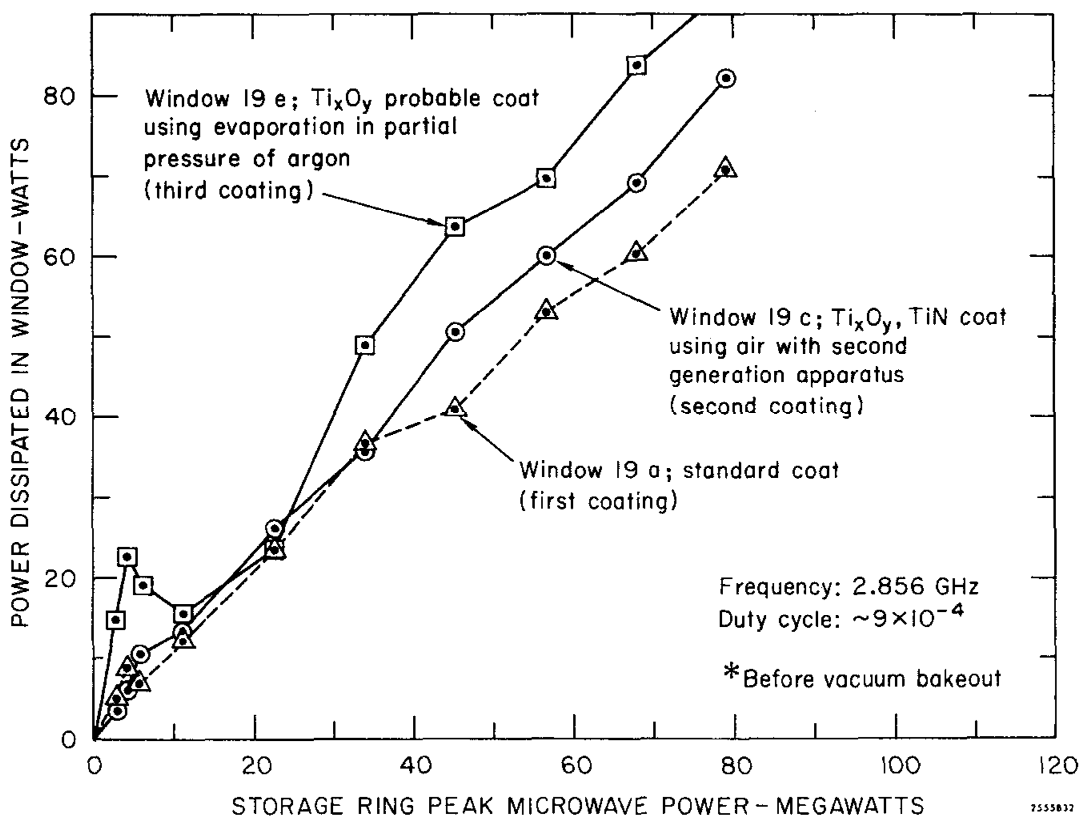


FIG. 24--Power dissipated in window number 19 as a function of sequential coating with different materials.

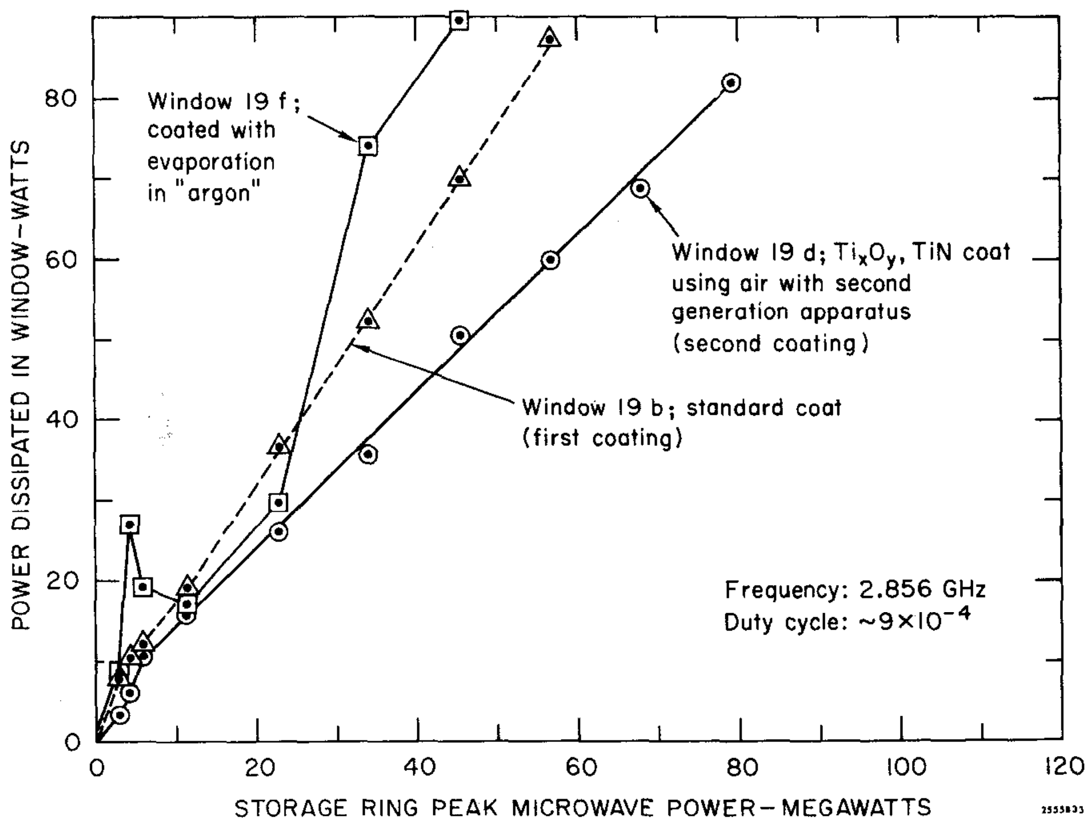
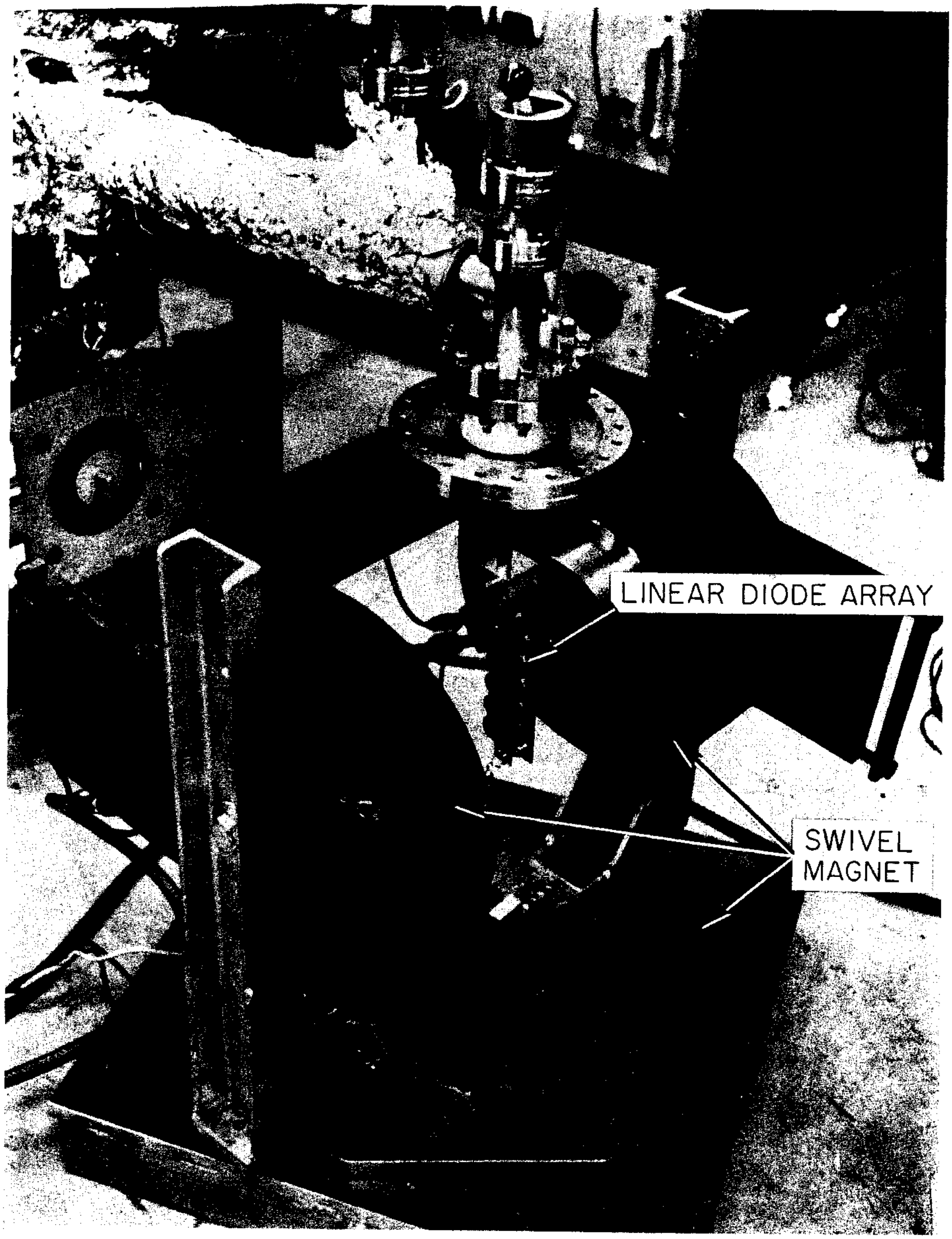
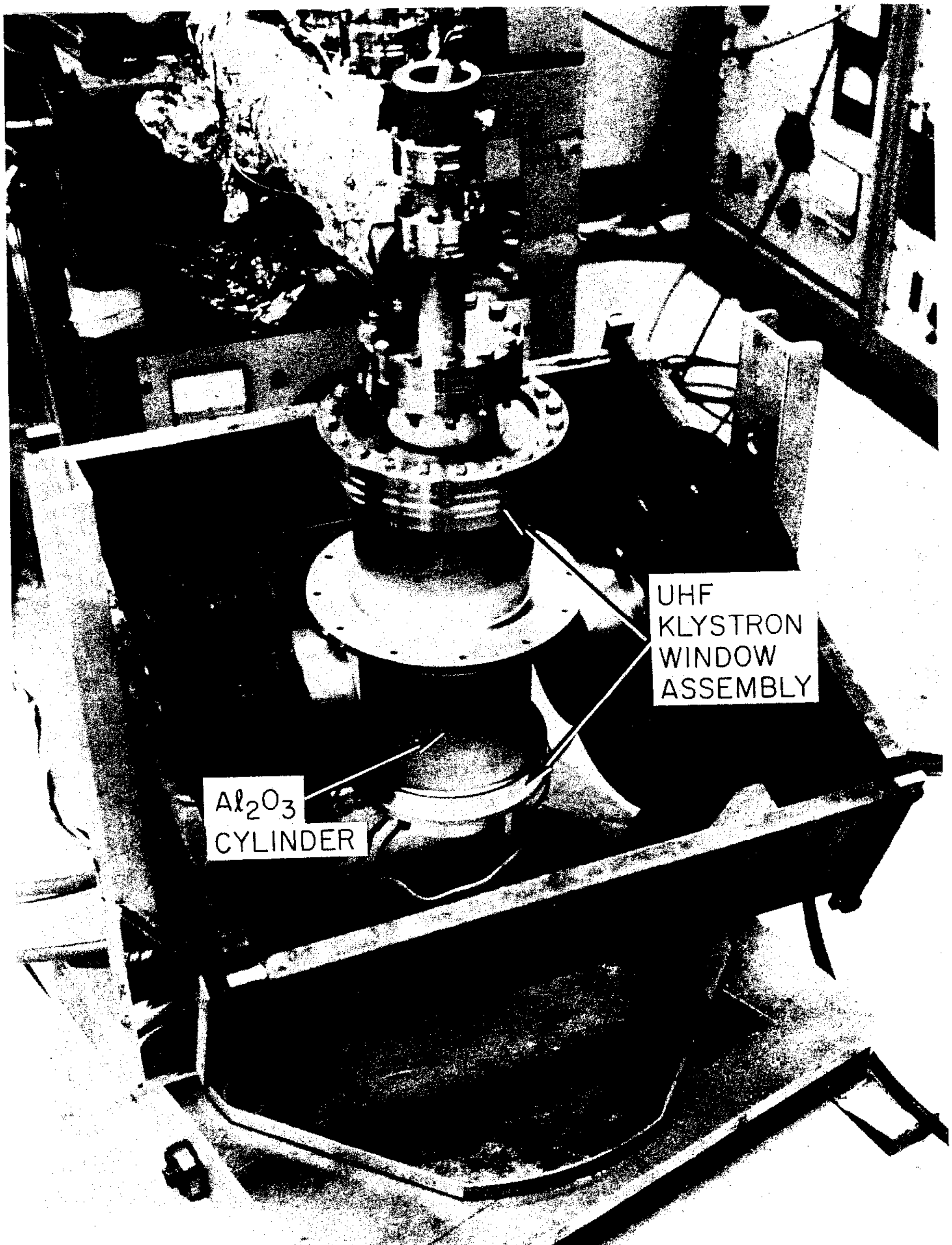


FIG. 25--Power dissipated in window number 19 in post bakeout test as a function of sequential coating with different materials.



2555A6

FIG. 26--~~L-band~~ UHF window coating apparatus with the window removed.



2555A7

FIG. 27--~~L-band~~ UHF window coating apparatus with window installed.

THIRD GENERATION COATING APPARATUS

Our next objective was to construct a window coating system which could serve as an alternative to the standard coating system in the event of difficulties in the future, and which could be used in future studies with TiN thin films and perhaps other new materials. Features of this new apparatus are shown in the following figures. Figure 28 shows a window mounted on the top bell jar plate swivel assembly. This swivel assembly permitted rotation of the window for sequential coating of each side, and if desired, edge heating of the window with the discharge array ion beams. A solenoid coil was used to provide the magnetic field at the discharge array. The complete apparatus with magnet coil is shown in Fig. 29. The diode sputtering array is shown in Fig. 30. The window is tilted in this figure to provide a better view of the array. The sputter array may be moved in a direction transverse to the system axis by manipulation of a bellows at the bottom of the bell jar base plate assembly as shown in Fig. 31. This permitted ion beam "scrubbing" of the total window assembly and made possible more uniform film deposition during the coating operations. With the given configuration, the klystron window assembly could be electrically biased with respect to the anode and cathode of the array. As a rule windows were edge heated with the array ion beam prior to coating, but at times subsequently coated while biased at anode potential.

A series of eight windows were coated in the third generation apparatus with progressively thicker films until the scaled second generation apparatus optimum parameters were achieved. This corresponded to a total optimum film thickness of $\sim$75 Å as subsequently measured with a crystal film thickness monitor. Results of subsequent RF tests of these windows are shown in Figs. 32 and 33. Calorimetric data were not taken on the first two windows (14f (1) and 17h) due to excessive multipactor and a desire not to push them to destruction. Test performances of the last two windows featuring "optimum parameter" coatings suggested that good control and repeatibility is achieved with this coating system, and also that the technology can be easily translated from one apparatus to another.

CONCLUSION

A new material has been reported for use in the suppression of multipactor in microwave windows. This material, titanium nitride, was subsequently used at the writers suggestion, with considerable success in eliminating serious

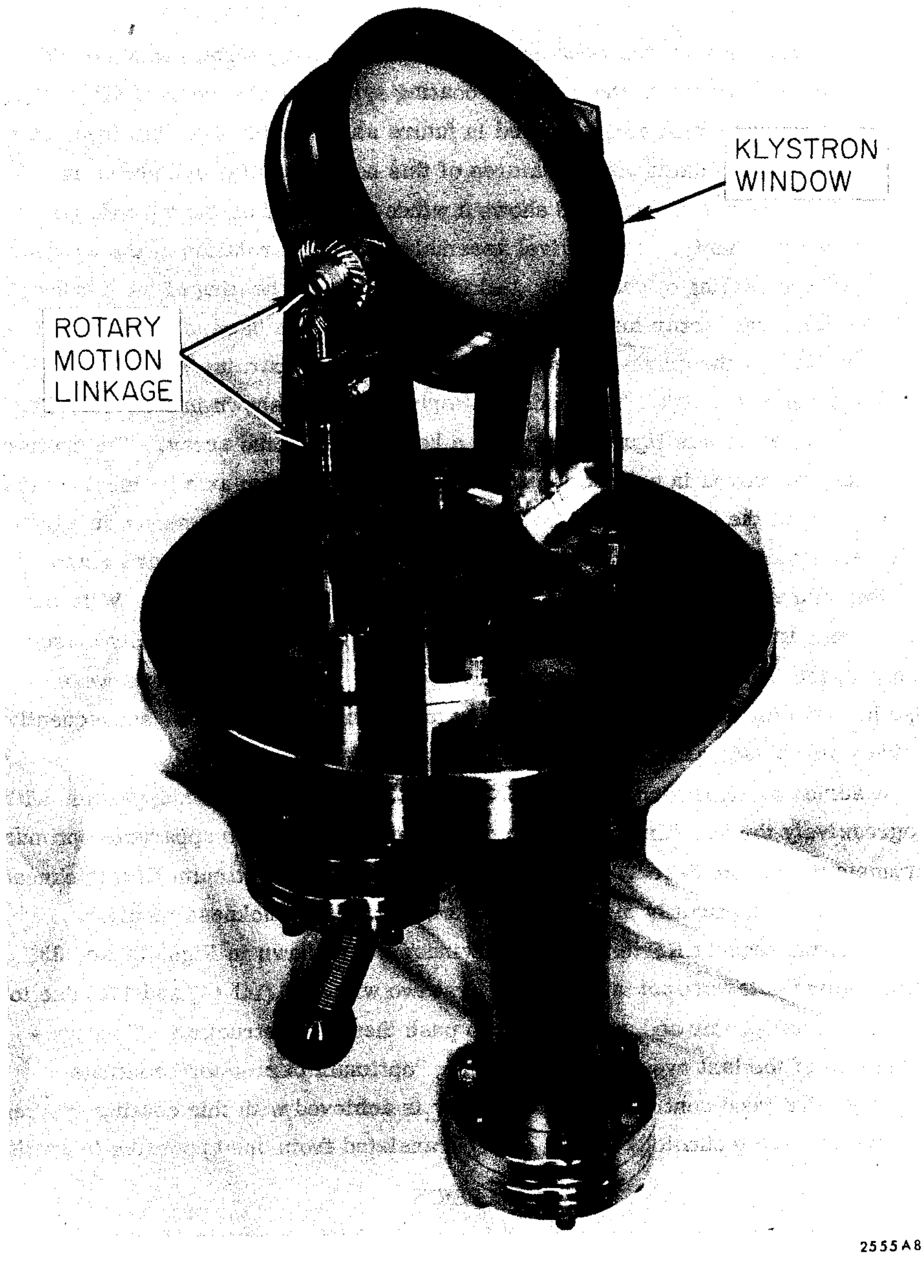


FIG. 28--Window mounting bell jar plate assembly.

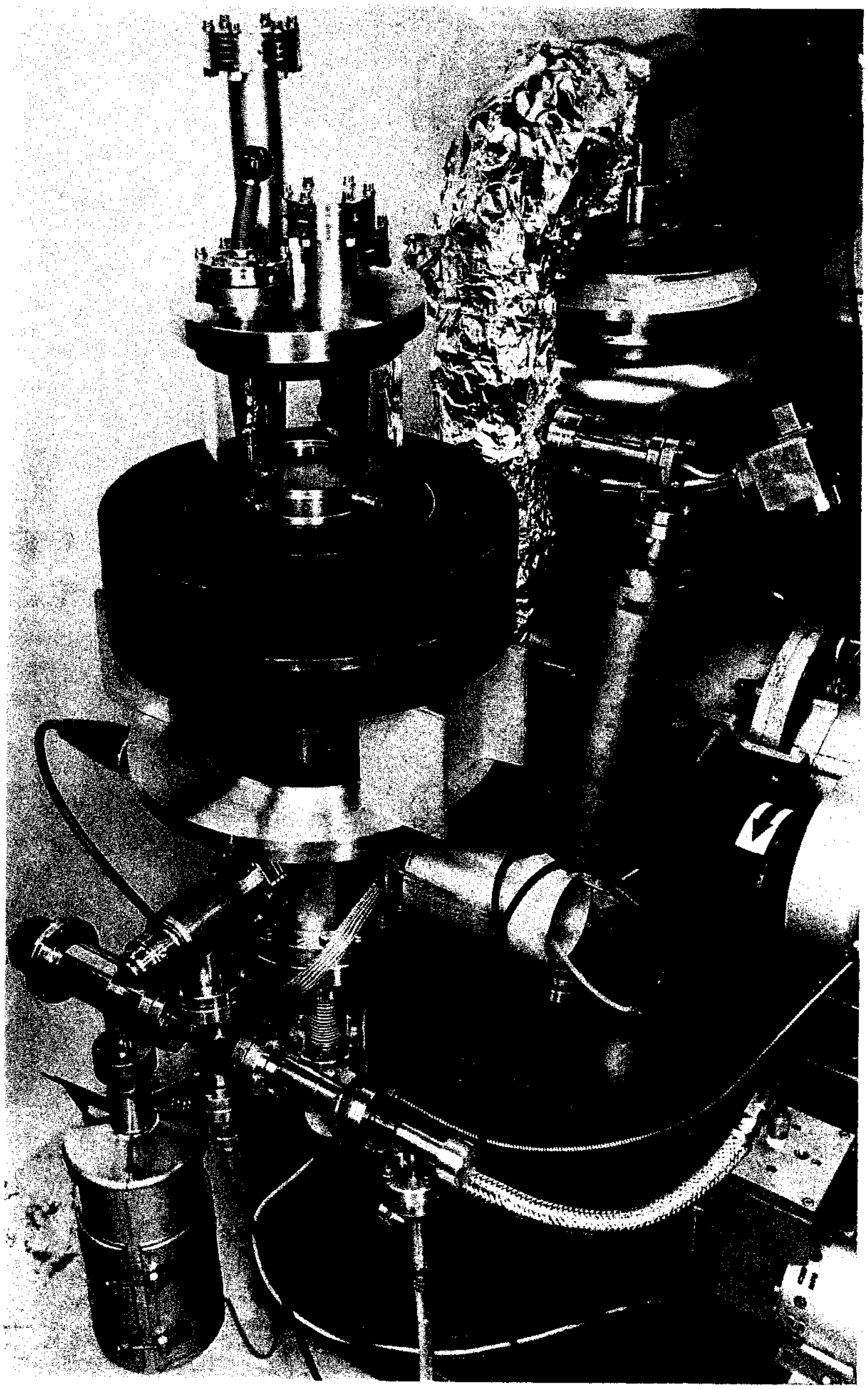


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FIG. 29--Third generation window coating apparatus.

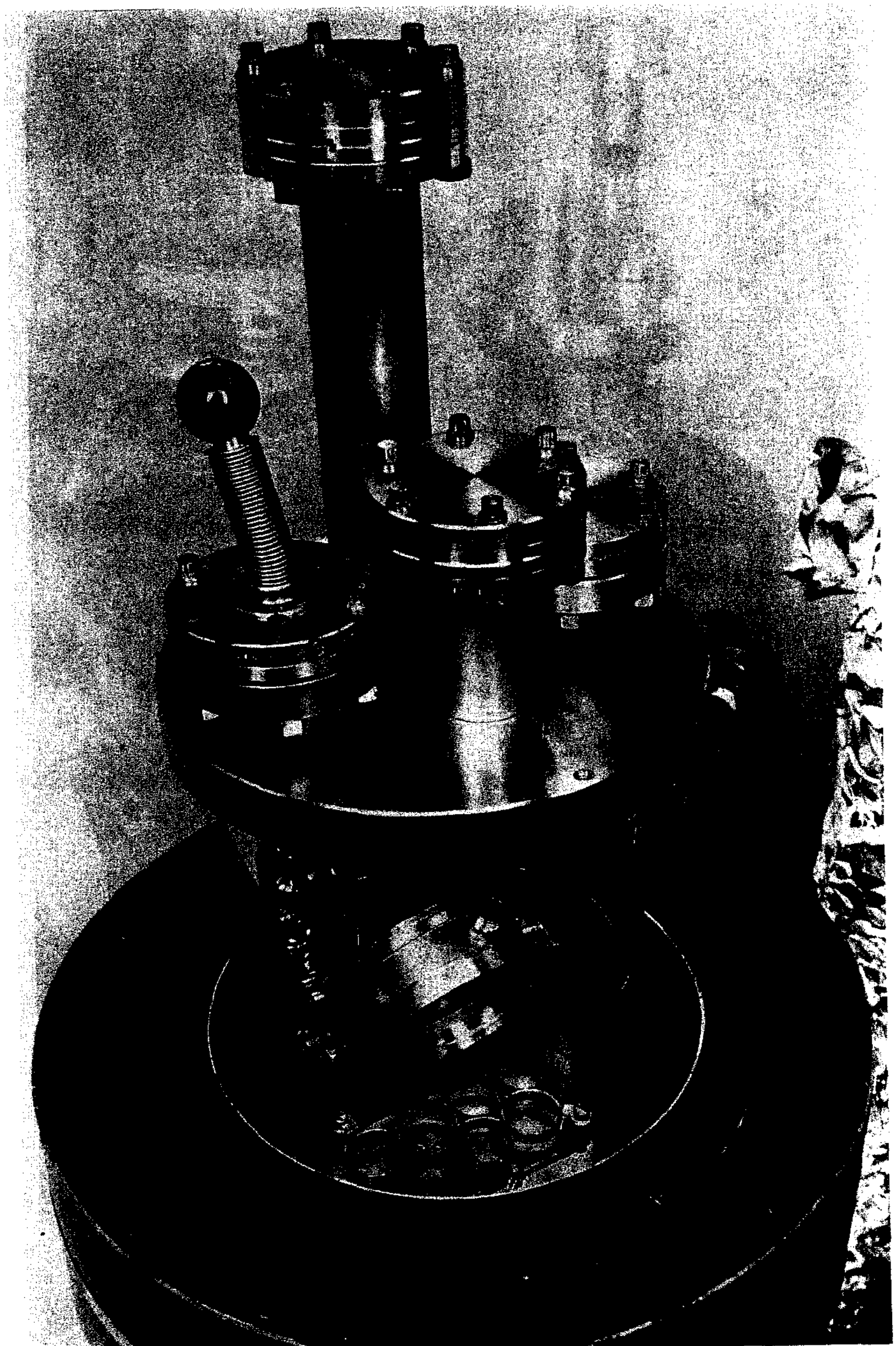

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FIG. 30--Third generation coating apparatus showing diode sputtering array.

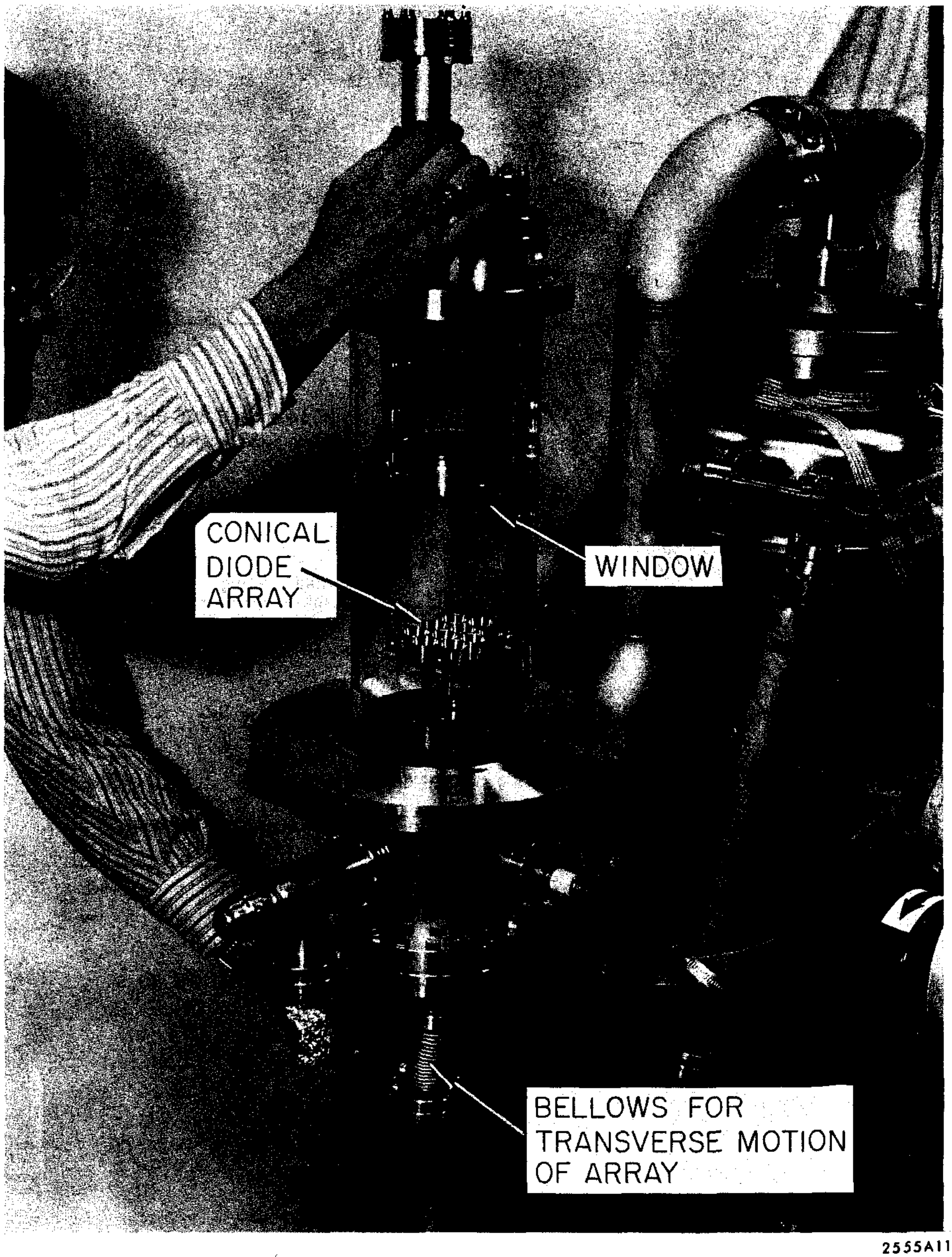

FIG. 31--Third generation sputtering apparatus with solenoid magnet removed.

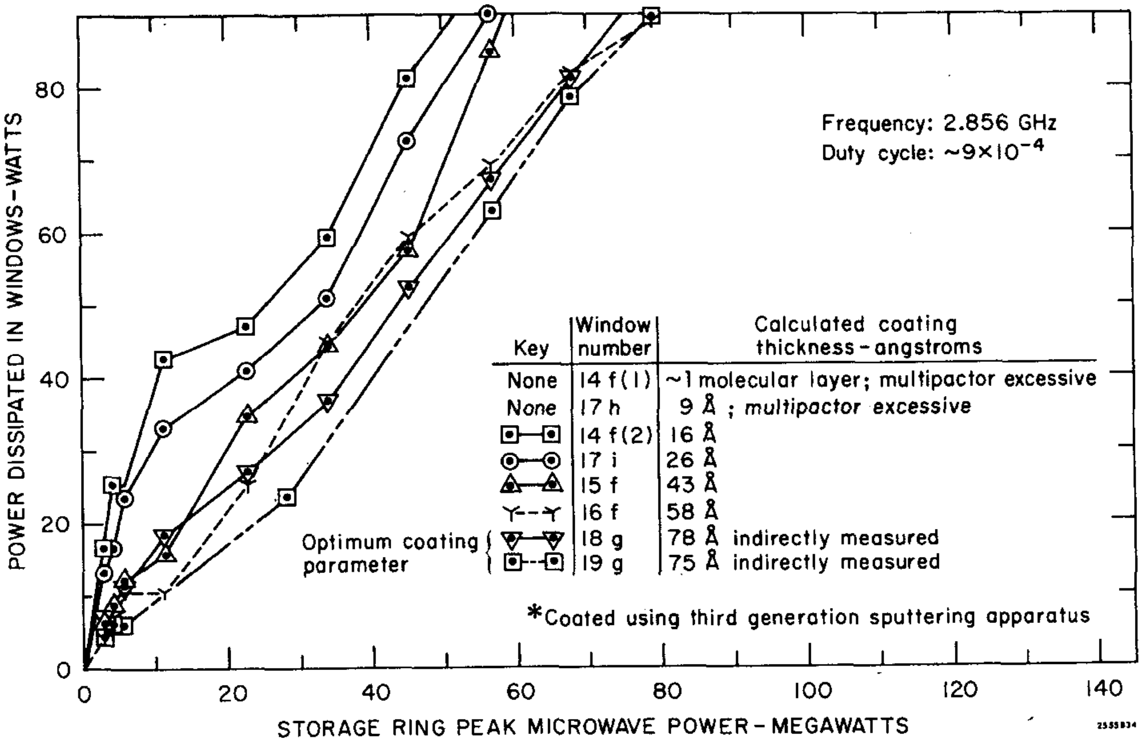

FIG. 32--Power dissipated in titanium nitride coated windows as a function of calculated film thickness.

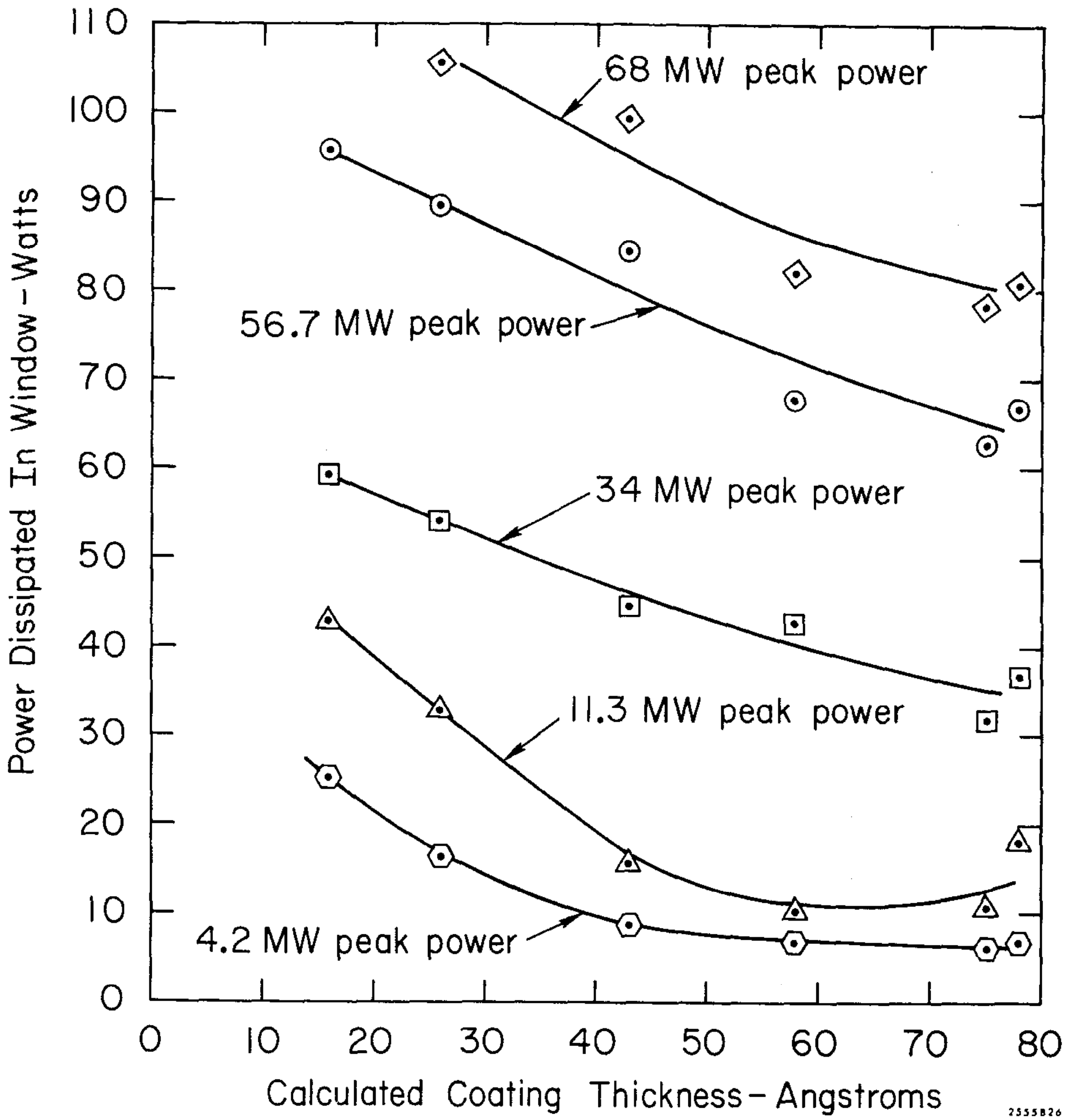

FIG. 33--Power dissipated in windows as a function of titanium nitride coating thickness and ring peak microwave power.

multipactor problems in large aluminum e^-e^+ storage ring RF cavities.[20] Initial findings in window tests indicate that TiN may be superior to the less stable oxides of titanium.

A new method has been reported for sputtering thin films. It was successfully used to reactively sputter films of titanium onto windows for the suppressions of multipactor. Sputtering rates are predictable and films of uniform thickness were obtained by manipulation of the substrate and discharge array. This coating scheme shows considerable promise in future research and industrial applications.[21]

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