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LOW PRESSURE CROSSED FIELD VACUUM SPUTTERING OF THIN FILMS FOR MULTIPACTOR SUPPRESSION USING A SIMPLE DIODE ARRAY*

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ABSTRACT

Using a modified crossed field diode configuration, thin films of titanium nitride, and oxides of titanium were reactively sputtered onto $A\ell_2O_3$ substrates. These $A\ell_2O_3$ substrates were high power microwave klystron windows. The thin films served to eliminate effects of multipactor heating due to the high secondary electron emission coefficient of aluminum oxide. Surfaces coated included large diameter cylindrical ceramics and flat disk shaped windows. Sputtering rates varied from less than one molecular layer per minute to several hundred angstroms per minute, and could be precisely controlled in a predictable fashion by varying parameters including magnetic field, sputtering potential, cell aspect ratio, diode array to substrate spacing, and pressure.

(Submitted to J. Vac. Sci. and Tech.)

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INTRODUCTION

Low pressure sputtering using "crossed" electrical and magnetic fields has been of interest for several decades. Penning filed patent on such a device in 1936.[1] The configurations reported at that time were extensions of his earlier patented single cell magnetron and hollow cell diodes used for pressure measurement.[2] Little was done with this as a film deposition scheme until recent work with inverted magnetron configurations by Gill and Kay,[3] and subsequent work by Hayakawa and Wasa[4-7] with conventional magnetron configurations. In all of these cases the substrate to be coated was contained within the discharge volume, and in some cases the substrate served as one of the electrodes of the sputtering potential. Other crossed field sputtering devices making use of non-uniform, fringing magnetic and electric fields are finding commercial application.[8,9]

Low pressure crossed field electrical discharges have been under limited investigation at SLAC for some time. This work was primarily directed toward applications in sputter-ion pumps and vacuum gauges.[10-13] Extensive experiments were conducted with configurations including versions of the typical single cell diode reported widely in the literature.[13] Figure 1 typifies this single cell configuration. Low pressure discharge characteristics of this "simple" configuration have been the subject of over 400 technical publications in the last thirty years. Yet, of the numerous discharge modes reported, only two to date may be discussed on a semi-quantitative or theoretical basis.[13-15]

While in the course of studies at SLAC, discharge characteristics of several "half diodes" were investigated. An example of such a configuration is given in Fig. 2. A reduction in discharge current of approximately a factor of two over a conventional comparible diode was observed. But, all of the other

characteristics of the discharge intensity were preserved. These included the linear relationship of discharge current to magnetic field, diode voltage potential, and pressure (i.e., controlled experiments were conducted in modes 1 and 2[13]). Possible applications in thin film sputtering were immediately apparent.

MULTIPACTOR IN MICROWAVE WINDOWS

Multipactor is an electron bombardment heating phenomenon unique to RF vacuum devices. The unfortunate combination of optimum surface orientation with respect to the electric field, high secondary electron emission coefficient of material surface(s), electron energy, and rf frequency will support multipactor.[16,17] The phenomenon has been understood for many years and is avoided in devices such as RF cavities and electron beam interaction gaps by material selection and dimensional considerations. Multipactor in such devices may be severe enough to melt cavity drift tube noses. Also, the steady state electron space charge cloud built up and sustained by the multipactor may result in RF matching problems due to the plasma dielectric equivalence, and/or significantly spoil the Q of an RF resonant circuit due to power lost in heating.

High power microwave windows are particularly subject to multipactor problems.[18,19] Physical properties including low loss tangent and high dielectric strength, and ease of fabrication make $A\ell_2O_3$ and BeO the best candidates for window material to date.[20] 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 in RF window design compound the problem of multipactor. A typical high power klystron window configuration is shown in Fig. 3. A transformation from the TE_{10} rectangular waveguide mode to a TE_{11} circular waveguide mode in this window structure creates very complicated fringing RF fields in the region of the window. It is not necessarily at very high RF power levels where multipactor problems occur. Due to variations in the RF field through a plane normal to the Poynting vector, energy bands supporting multipactor occur

at varying power levels. Regions of high multipactor shift in the window structure with variations in transmitted power. If loading and mismatch problems are not too severe it is sometimes possible to pass through these critical energy bands as demonstrated in Fig. 4. This figure shows calorimetric power dissipated in a window structure as a function of transmitted power in a high power microwave storage ring.

WINDOW COATING TO SUPPRESS MULTIPACTOR

In order to suppress multipactor very thin films of oxides of titanium have been used on window surfaces. These have been deposited by vacuum evaporation methods[21] and AC sputtering in a high partial pressure of argon.[20] Holland and Laurenson later demonstrated the secondary emission properties of films of titanium and its oxides.[22] Extensive work has subsequently been done with titanium and its oxides in the suppression of multipactor in microwave windows.[20,23]

A very large measure of success has been experienced at SLAC in use of reactively sputtered titanium films. However, procedures and equipment were largely the out-growth of empirical work, and did not readily translate to differing configurations. Also, rigorous control of film thickness is required or difficulty will be experienced with "hot" windows during test. Lastly, there is some suggestion that properties of the film change as a consequence of high temperature vacuum bakeout. This is demonstrated in Fig. 5 where calorimetric data are given for a sample of standard windows tested before and after bakeout. Work is continually be done with new coating materials in an effort to overcome the above short comings of oxides of titanium.[20,24,25]

SPUTTER COATING

It occurred to the writer that titanium nitride was thermally much more stable than oxides of titanium. Also, if it proved to have satisfactory secondary emission properties as well as thin film resistivity comparible to oxides of titanium, it might then prove to be a superior window coating material.

A double array of "half" 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. 6. 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 ~ 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 half diode arrays, new plates installed, 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 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 7 and 8 show RF ring test results for the titanium nitride and oxide of titanium coatings respectively. All 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,[26] standard coated window with copper plating on the cupronickel sleeves (see Fig. 3; 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_xO_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.

It should be stressed that the difference between a coated and uncoated window is significant. This difference is illustrated in Fig. 9, where power dissipation due to multipactor in an uncoated and partially coated window is given. It would be more than generous to say that our initial coating experiments lacked control. But, the initial results were very encouraging and prompted further experimentation.

SECOND GENERATION COATING APPARATUS

Our next objective was to develop an apparatus for coating the inside of a cylindrical $A\ell_2O_3$, L-band klystron output window (~15 cm dia. × 15 cm long). Test facilities did not exist for evaluating the window coating material and procedure prior to irrevocable commitment to the klystron assembly. A linear "half" diode array was therefore constructed, and used to coat the typical S-band windows (Fig. 3) which could be tested in the RF storage ring. This linear diode array is shown in Fig. 10. A rotary vacuum feedthrough was used to permit eventual coating of the inside of the L-band 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 insertion of the array in the cylindrical output window. Figure 11 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.

Numerous windows were coated with this apparatus. During these experiments both the above N_2-A and O_2-A gas mixtures were used as well as laboratory air, and pure argon. Evaporation coating experiments were also conducted with these gases. Results of these lengthy experiments are reported elsewhere.[27] However, TiN coating appeared superior to all other combinations both from the standpoint of power dissipation in windows and the virtual absence of the typical window glow patterns usually observed during microwave testing. Three windows were coated with TiN using the second generation apparatus. Results of tests on these windows before and after vacuum bakeout are shown in Figs. 12 and 13 respectively. Window No. 15e, shown in Fig. 13, was determined to have been contaminated with diffusion pump oil during vacuum bakeout.

The linear diode array was subsequently used to coat the cylindrical L-band 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. 14 with the L-band window removed from the system. This same apparatus is shown with the window installed in Fig. 15. The window served as the vacuum bottle during coating.

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 16 shows a window mounted on the top bell jar plate swivel assembly. This swivel assembly permited 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. 17. The diode sputtering array is shown in Fig. 18. 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. 19. This permitted ion beam scrubbing of the total window assembly and made possible more uniform film deposition during the coating operation. 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.

In previous coating experiments with different configurations, we made the following assumptions: (1) the rate of film deposition from the half diode array is directly proportional to the discharge current for a given potential; (2) the rate of film deposition onto the substrate is inversely proportional to the square of the distance of the array from the substrate, and lastly; (3) the rate of deposition from any one cell should be weighted in direct proportion to the

solid angle formed by the open end of the anode and a point source centered at the cathode.

Using a crystal monitor we measured film deposition rates for known discharge parameters in the third generation apparatus. 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 ≈ 75 Å. Results of subsequent RF tests of these windows are shown in Figs. 20 and 21. Calorimetric data were not taken on the first two windows (14 f(1) and 17 h) due to excessive multipactor and a desire not to "push" them to destruction. Test performance of the last two windows featuring "optimum parameter" coatings suggested good control and repeatability is achieved with this coating system.

DISCUSSION AND CONCLUSIONS

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 multipactor problems in large aluminum e^-e^+ storage ring RF cavities.[28] 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 suppression 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.[29]

ACKNOWLEDGMENTS

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FIGURE CAPTIONS

1. Typical single cell Penning diode.
2. Single cell "half" diode.
3. SLAC klystron microwave window.
4. Power dissipated in a typical Cr_2O_3 coated window as a function of RF storage ring power.
5. Power dissipated in windows with standard Ti_xO_y ac sputtered coating before and after 625°C vacuum bakeout.
6. Microwave window positioned in first generation sputtering apparatus.
7. 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.
8. 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.
9. Power dissipated in windows without coating.
10. Linear "half" diode sputtering array.
11. Linear sputtering array apparatus for L-band window coating feasibility studies.
12. Power dissipated in windows coated with titanium nitride in second generation apparatus compared with standard Ti_xO_y coated windows prior to vacuum bakeout.
13. 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.
14. L-band window coating apparatus with the window removed.
15. L-band window coating apparatus with window installed.
16. Window mounting bell jar plate assembly.

17. Third generation window coating apparatus.
18. Third generation coating apparatus showing diode sputtering array.
19. Sputtering apparatus without solenoid magnet.
20. Power dissipated in titanium nitride coated windows as a function of calculated film thickness.
21. Power dissipated in windows as a function of titanium nitride coating thickness and ring peak microwave power.

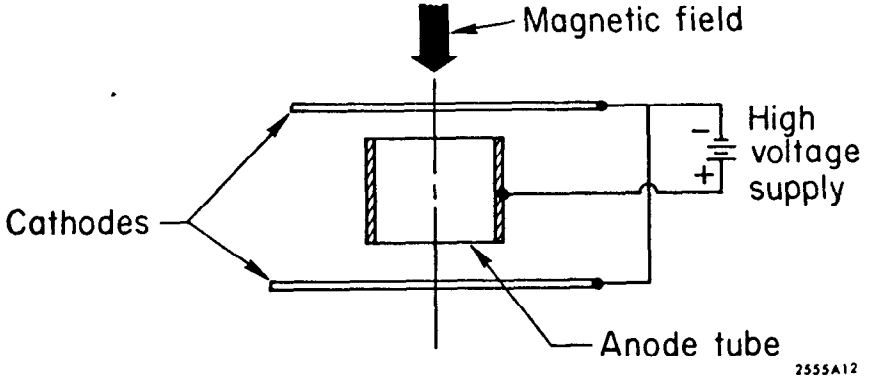

Fig. 1

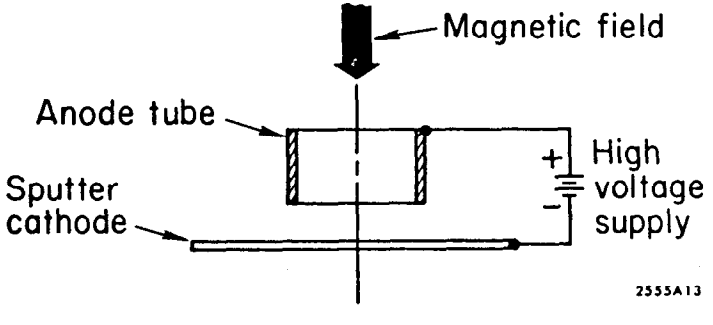

Fig. 2

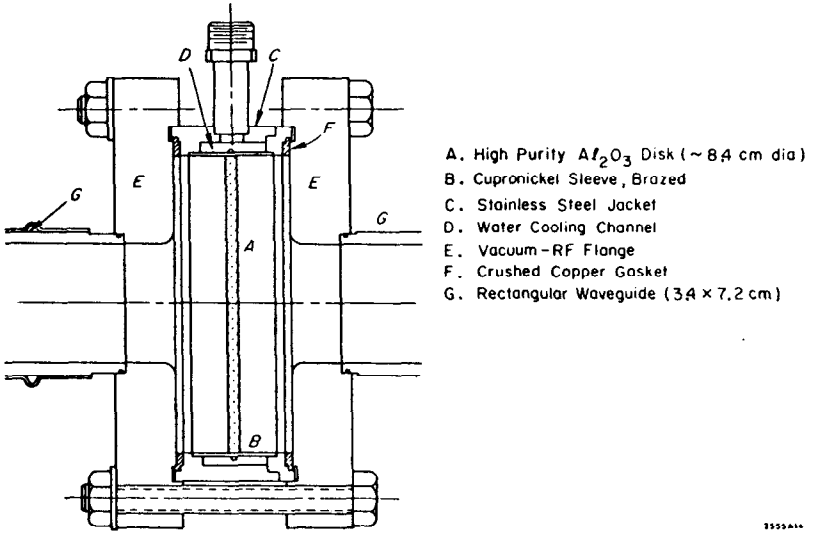

Fig. 3

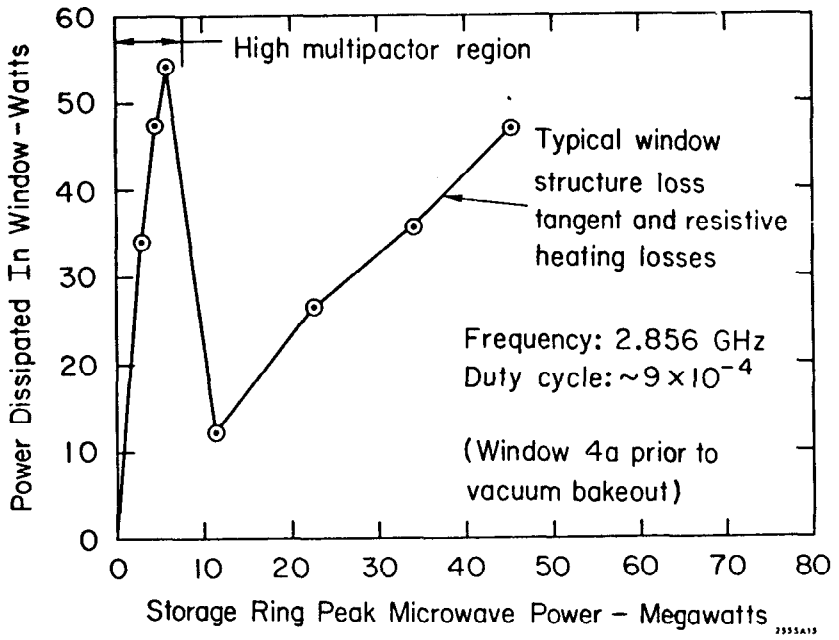
High multipactor region
Typical window structure loss tangent and resistive heating losses
Frequency: 2.856 GHz
Duty cycle: ~9 x 10^-4
(Window 4a prior to vacuum bakeout)
Power Dissipated In Window - Watts
Storage Ring Peak Microwave Power - Megawatts
0
10
20
30
40
50
60
70
80

Fig. 4

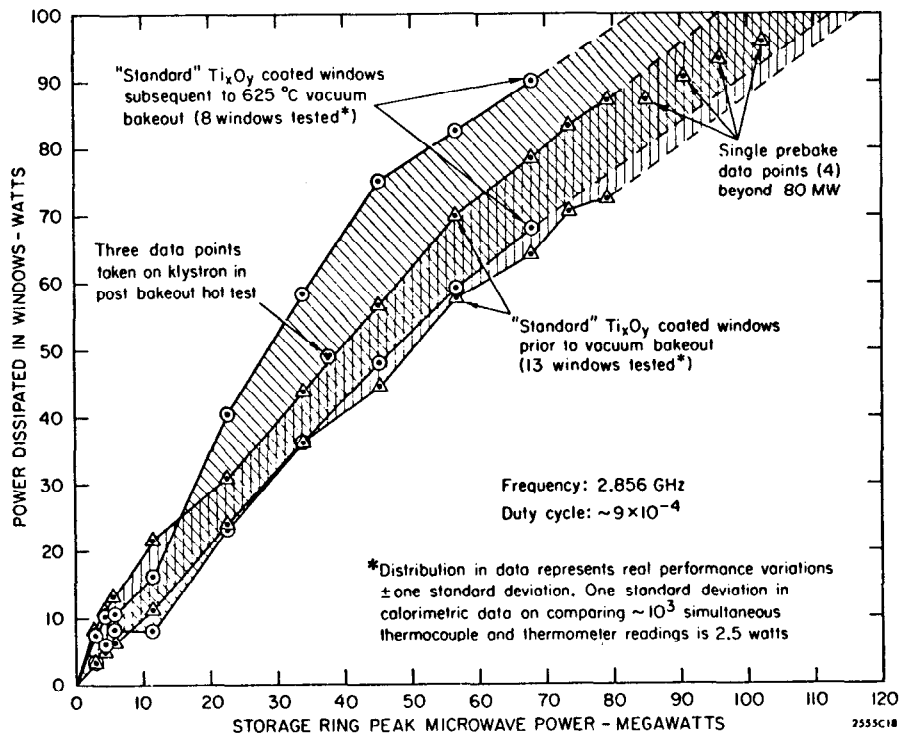
"Standard" Ti_xO_y coated windows subsequent to 625 °C vacuum bakeout (8 windows tested*)
Single prebake data points (4) beyond 80 MW
Three data points taken on klystron in post bakeout hot test
"Standard" Ti_xO_y coated windows prior to vacuum bakeout (13 windows tested*)
Frequency: 2.856 GHz
Duty cycle: ~9×10^-4
*Distribution in data represents real performance variations ±one standard deviation. One standard deviation in calorimetric data on comparing ~10^3 simultaneous thermocouple and thermometer readings is 2.5 watts
POWER DISSIPATED IN WINDOWS - WATTS
STORAGE RING PEAK MICROWAVE POWER - MEGAWATTS

Fig. 5

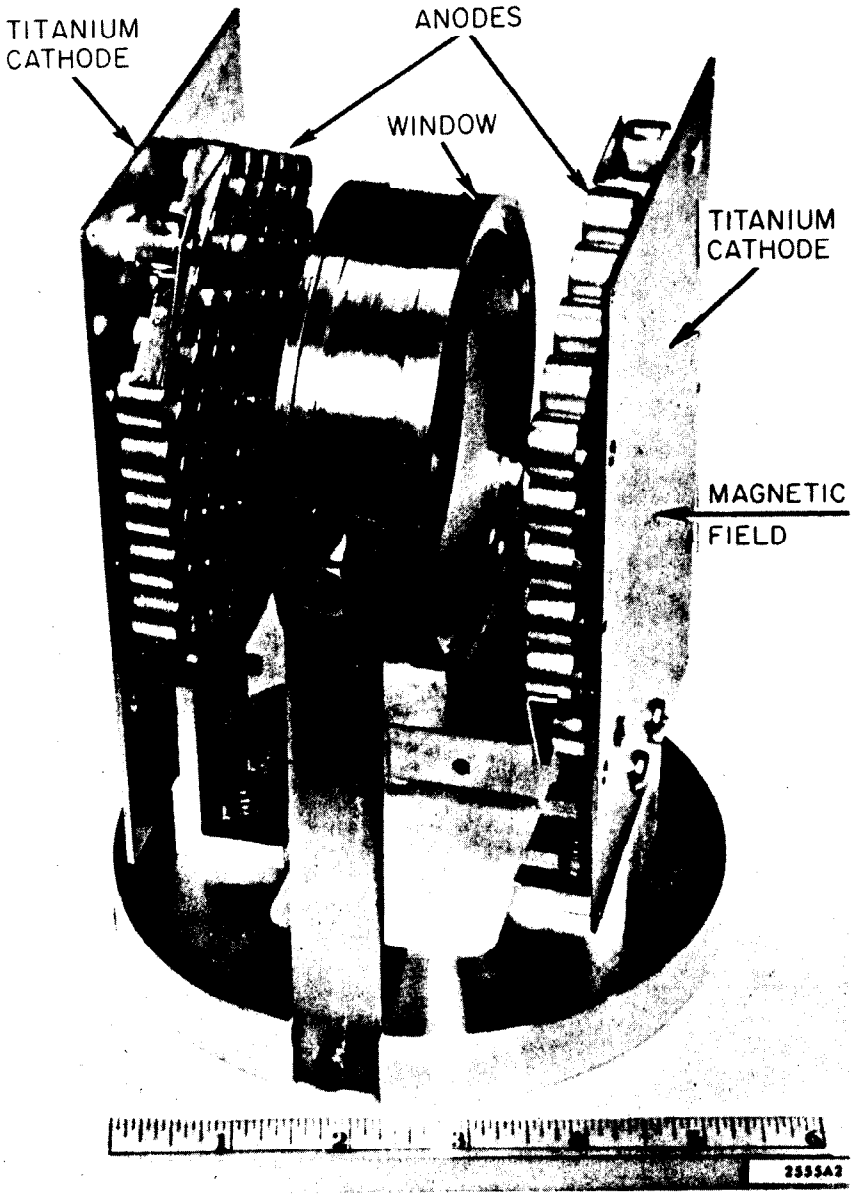
TITANIUM CATHODE
ANODES
WINDOW
TITANIUM CATHODE
MAGNETIC FIELD
2555A2

Fig. 6

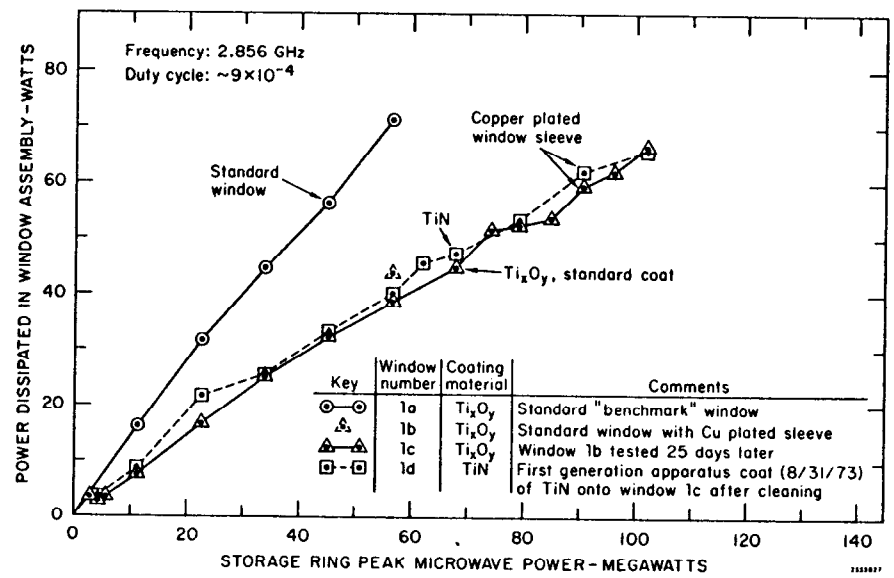
Frequency: 2.856 GHz
Duty cycle: ~9×10⁻⁴
Standard window
Copper plated window sleeve
TiN
Ti_xO_y, standard coat
POWER DISSIPATED IN WINDOW ASSEMBLY - WATTS
STORAGE RING PEAK MICROWAVE POWER - MEGAWATTS
0
20
40
60
80
100
120
140
Key | Window number | Coating material | Comments
1a | Ti_xO_y | Standard "benchmark" window
1b | Ti_xO_y | Standard window with Cu plated sleeve
1c | Ti_xO_y | Window 1b tested 25 days later
1d | TiN | First generation apparatus coat (8/31/73) of TiN onto window 1c after cleaning

Fig. 7

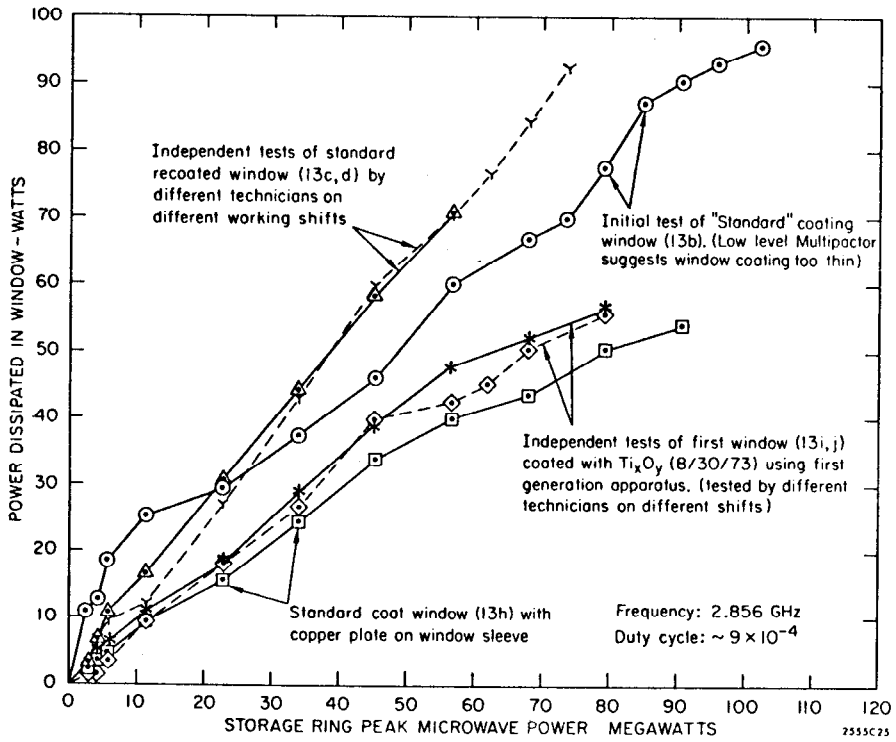
POWER DISSIPATED IN WINDOW - WATTS
STORAGE RING PEAK MICROWAVE POWER MEGAWATTS
Independent tests of standard recoated window (13c,d) by different technicians on different working shifts
Initial test of "Standard" coating window (13b). (Low level Multipactor suggests window coating too thin)
Independent tests of first window (13i,j) coated with Ti_xO_y (8/30/73) using first generation apparatus. (tested by different technicians on different shifts)
Standard coat window (13h) with copper plate on window sleeve
Frequency: 2.856 GHz
Duty cycle: ~ 9×10^{-4}
2555C25

Fig. 8

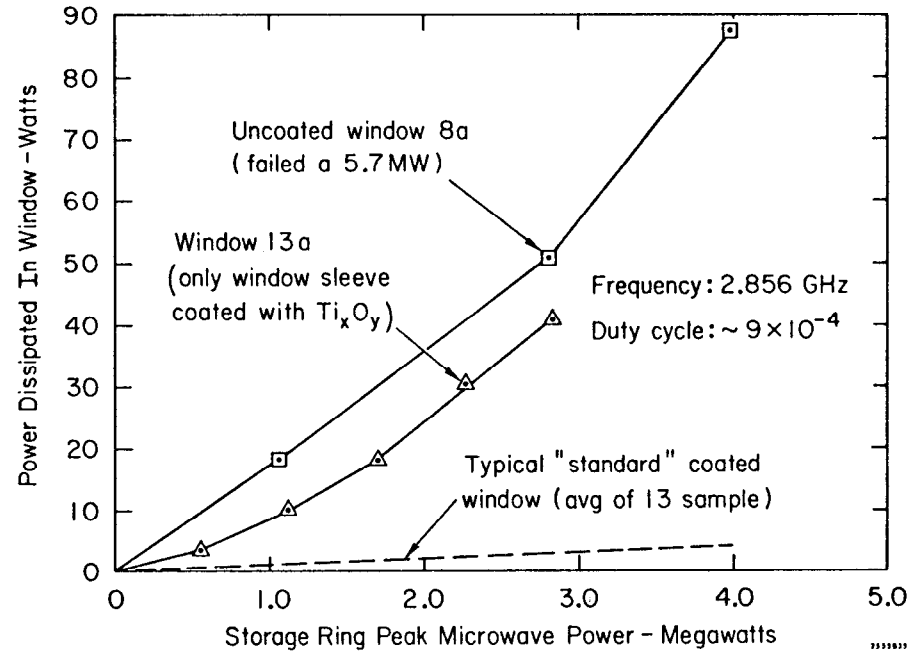
Power Dissipated In Window - Watts
Storage Ring Peak Microwave Power - Megawatts
Uncoated window 8a
(failed a 5.7 MW)
Window 13a
(only window sleeve
coated with Ti_xO_y)
Frequency: 2.856 GHz
Duty cycle: ~ 9×10^{-4}
Typical "standard" coated
window (avg of 13 sample)

Fig. 9

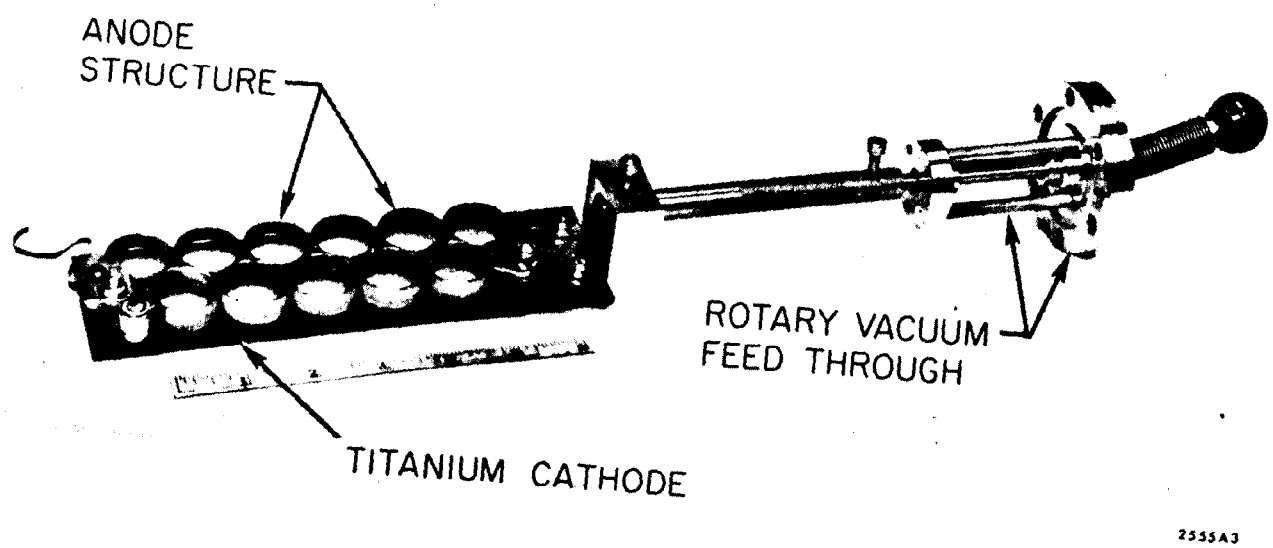
ANODE
STRUCTURE
ROTARY VACUUM
FEED THROUGH
TITANIUM CATHODE
2555A3

Fig. 10

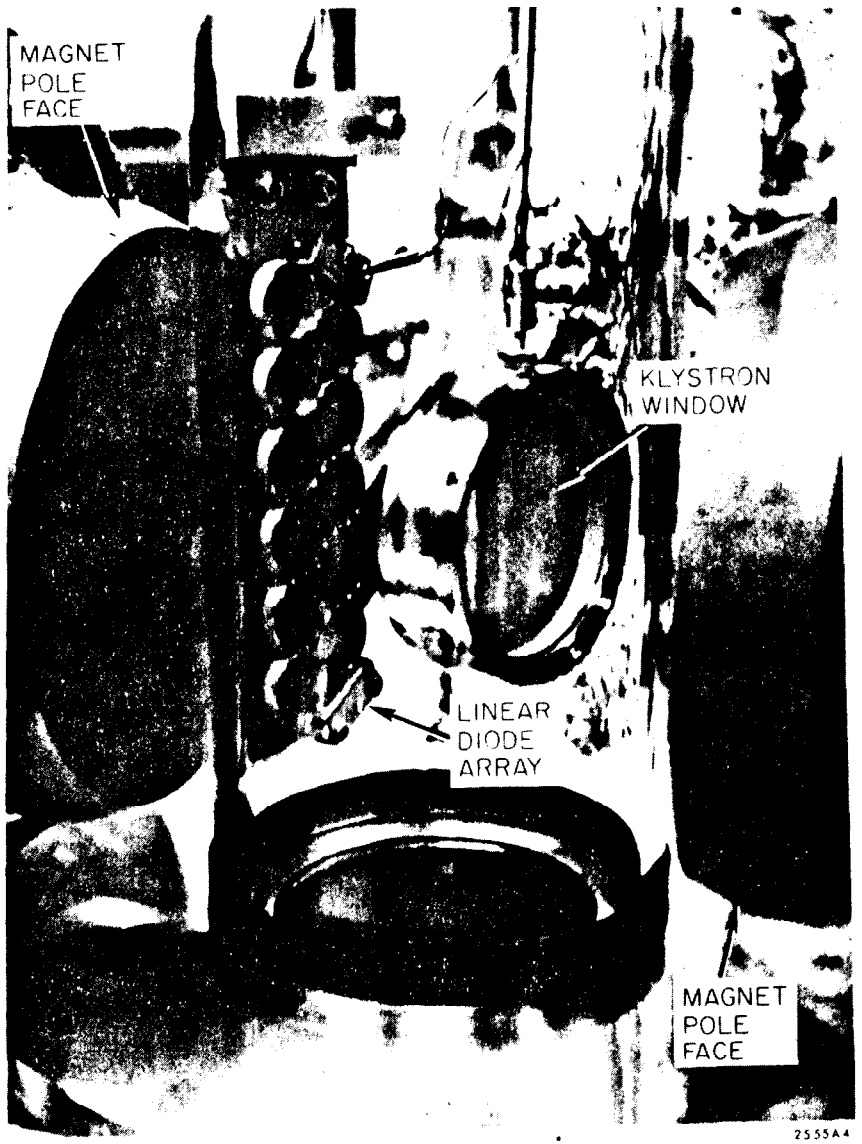
MAGNET
POLE
FACE
KLYSTRON
WINDOW
LINEAR
DIODE
ARRAY
MAGNET
POLE
FACE
2555A4

Fig. 11

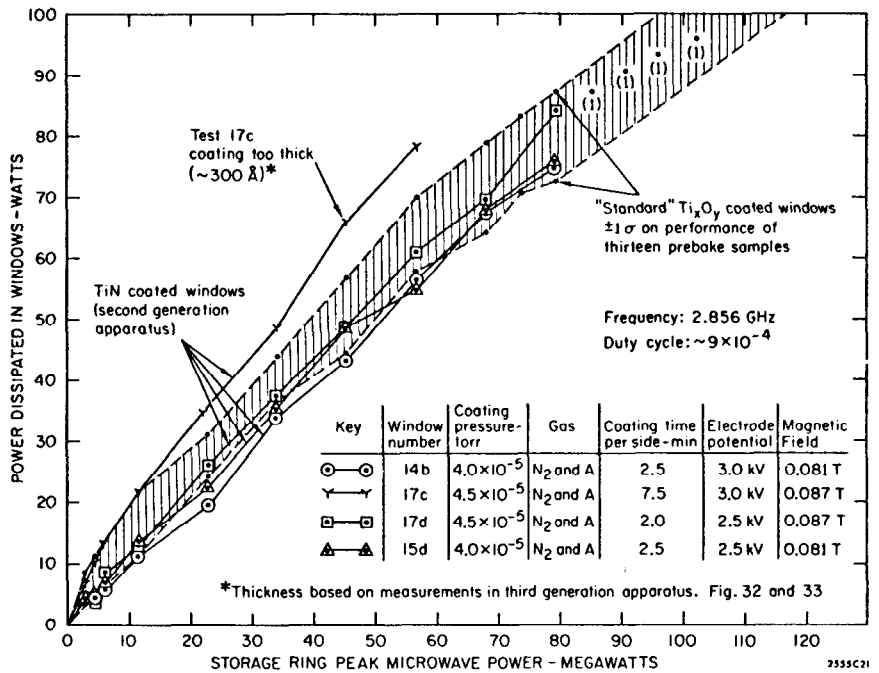
POWER DISSIPATED IN WINDOWS - WATTS
STORAGE RING PEAK MICROWAVE POWER - MEGAWATTS
Test 17c
coating too thick
(~300 Å)*
TiN coated windows
(second generation
apparatus)
"Standard" Ti_xO_y coated windows
±1 σ on performance of
thirteen prebake samples
Frequency: 2.856 GHz
Duty cycle: ~9×10⁻⁴
Key | Window number | Coating pressure-torr | Gas | Coating time per side-min | Electrode potential | Magnetic Field
14b | 4.0×10⁻⁵ | N₂ and A | 2.5 | 3.0 kV | 0.081 T
17c | 4.5×10⁻⁵ | N₂ and A | 7.5 | 3.0 kV | 0.087 T
17d | 4.5×10⁻⁵ | N₂ and A | 2.0 | 2.5 kV | 0.087 T
15d | 4.0×10⁻⁵ | N₂ and A | 2.5 | 2.5 kV | 0.081 T
*Thickness based on measurements in third generation apparatus. Fig. 32 and 33
2555C21

Fig. 12

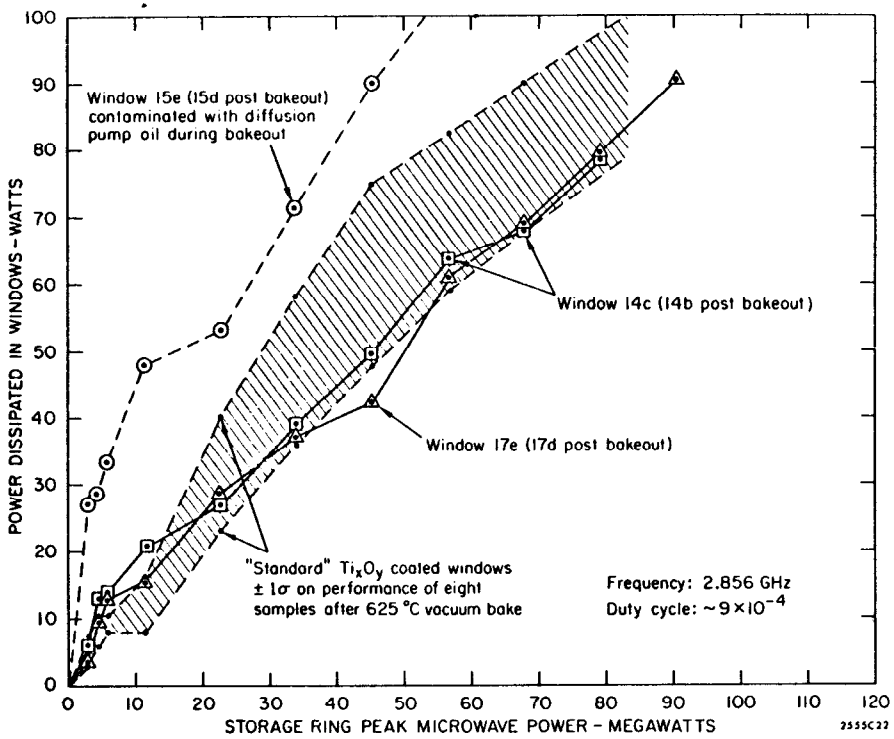
POWER DISSIPATED IN WINDOWS - WATTS
STORAGE RING PEAK MICROWAVE POWER - MEGAWATTS
Window 15e (15d post bakeout) contaminated with diffusion pump oil during bakeout
Window 14c (14b post bakeout)
Window 17e (17d post bakeout)
"Standard" Ti_xO_y coated windows ± 1σ on performance of eight samples after 625 °C vacuum bake
Frequency: 2.856 GHz
Duty cycle: ~9×10^{-4}
2555C22

Fig. 13

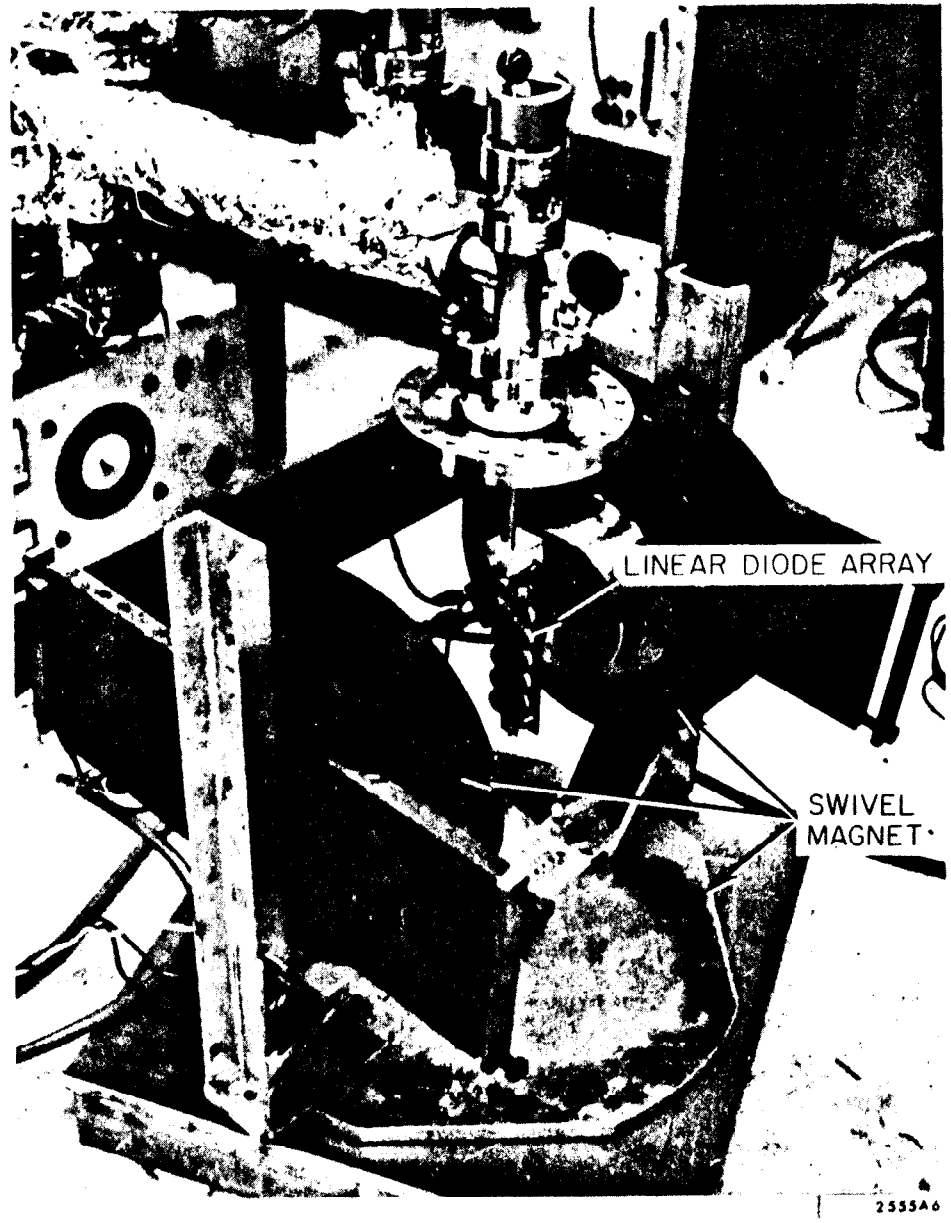
LINEAR DIODE ARRAY
SWIVEL MAGNET
2555A6

Fig. 14

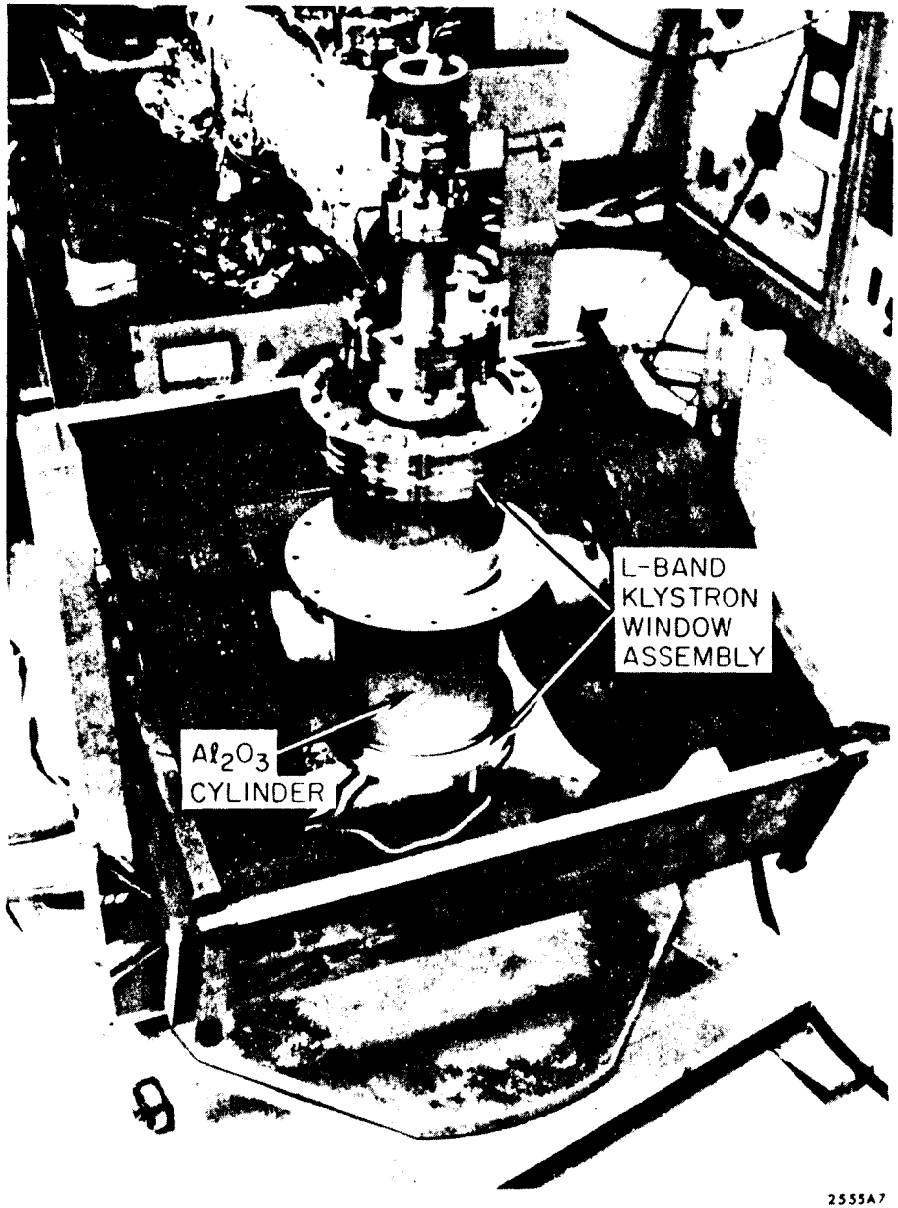
L-BAND
KLYSTRON
WINDOW
ASSEMBLY
Al_2O_3
CYLINDER
2555A7

Fig. 15

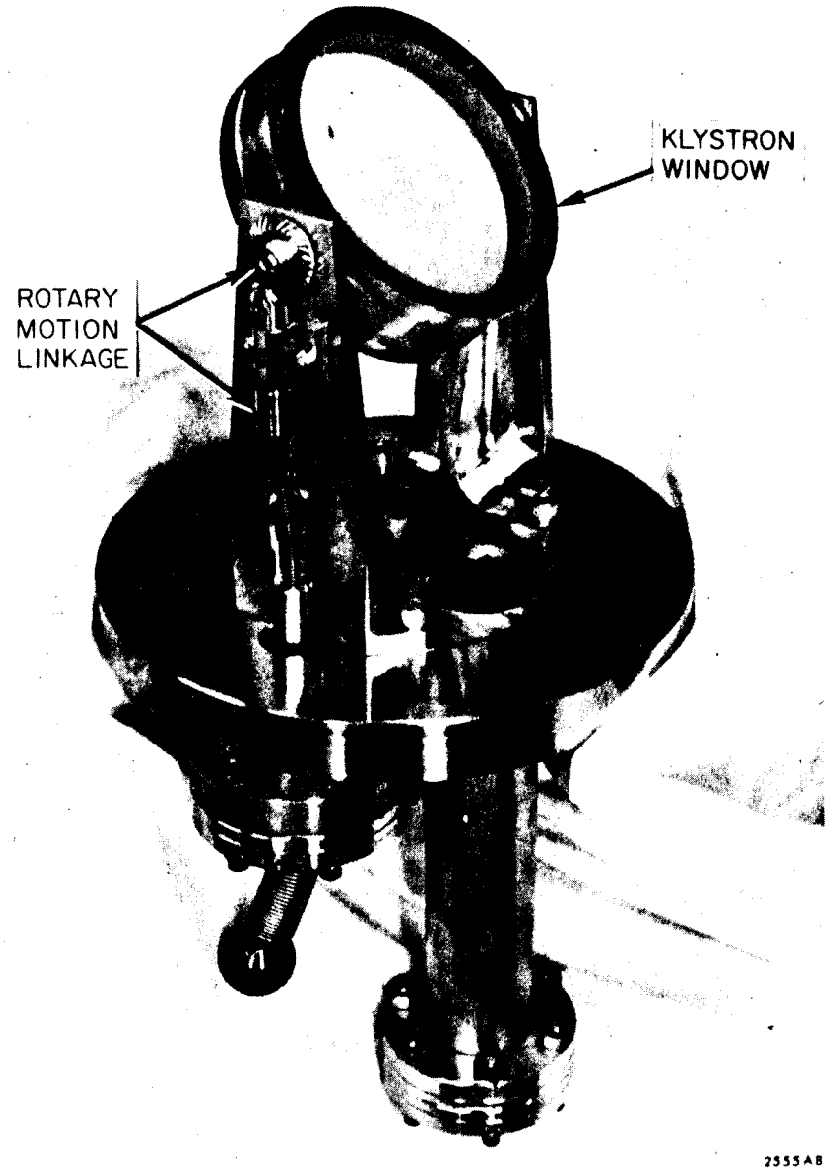
KLYSTRON
WINDOW
ROTARY
MOTION
LINKAGE
2555A8

Fig. 16

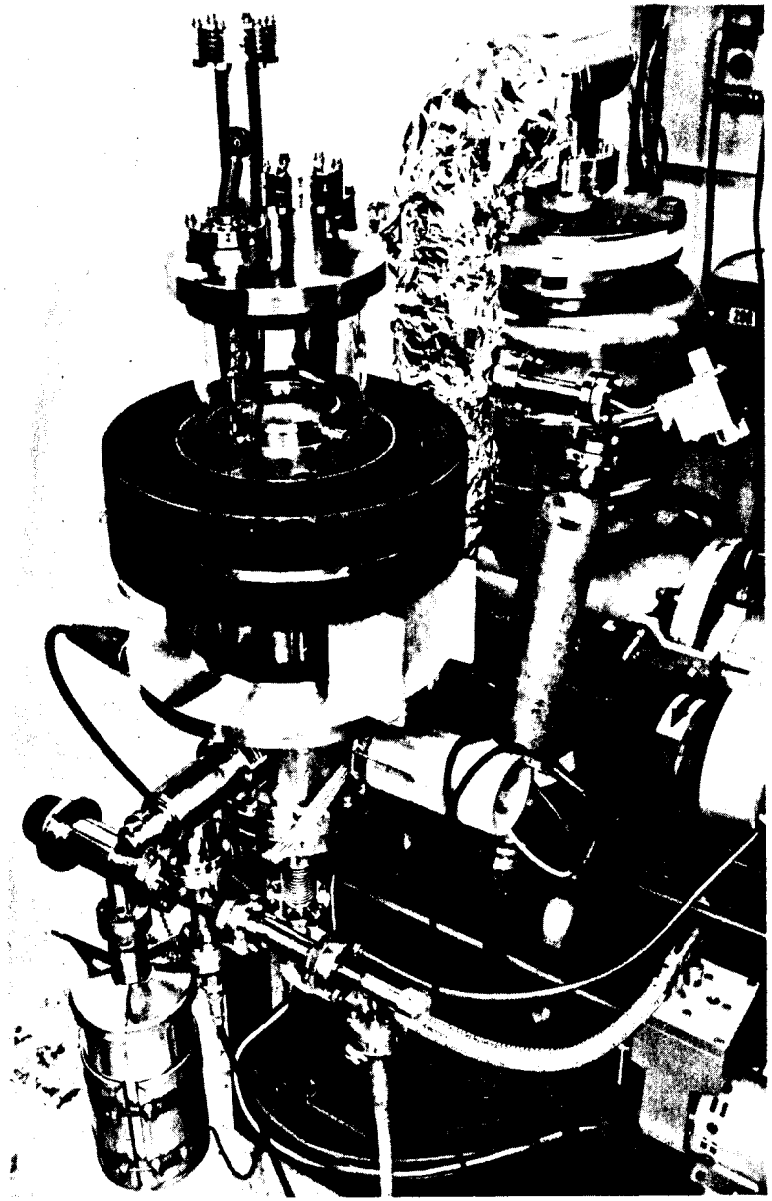

Fig. 17

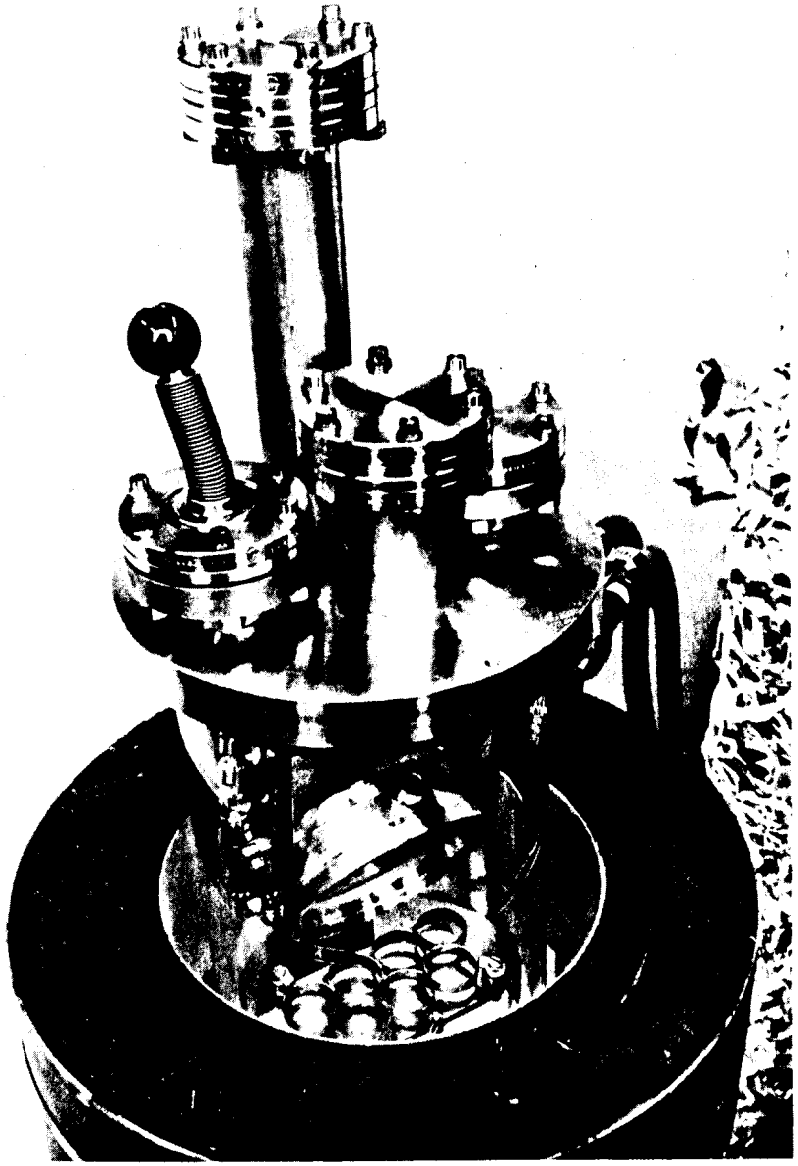

Fig. 18

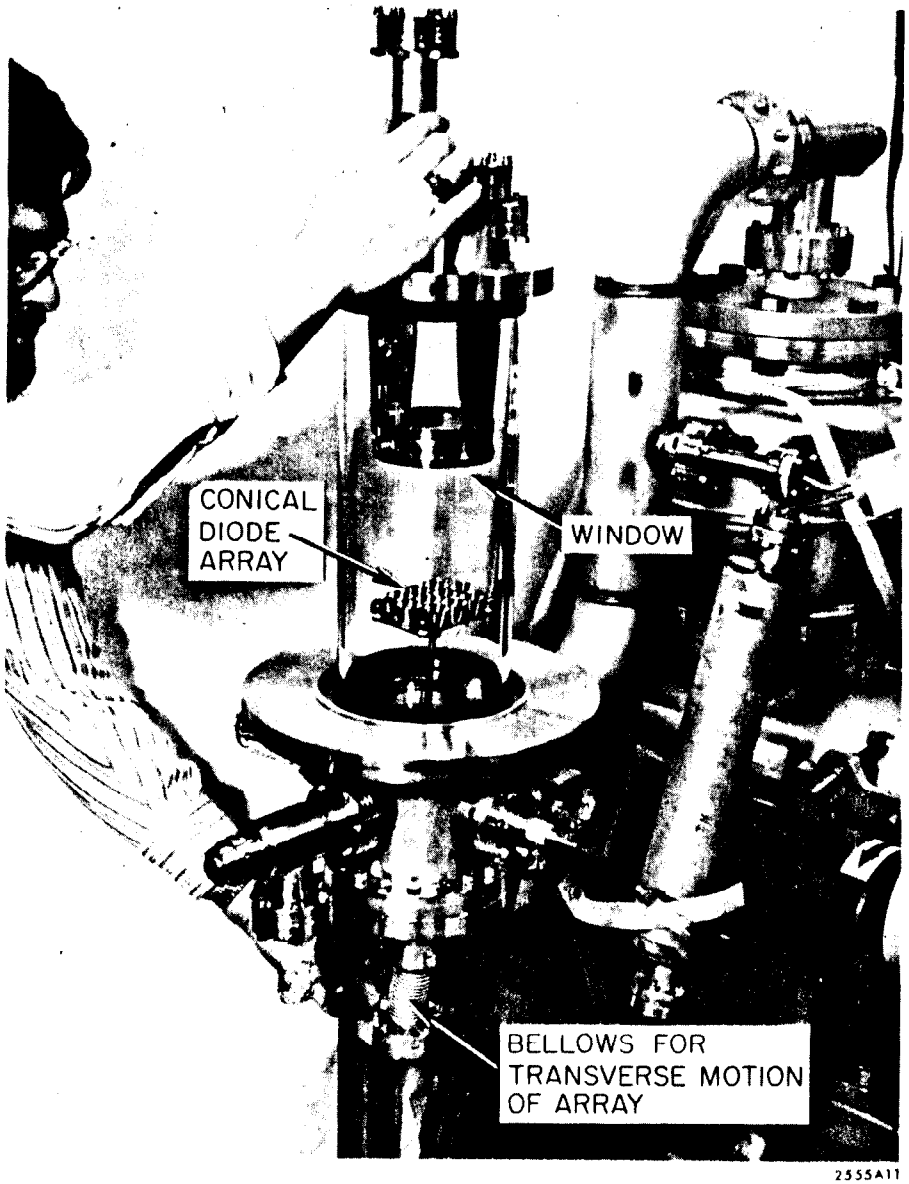
CONICAL
DIODE
ARRAY
WINDOW
BELLOWS FOR
TRANSVERSE MOTION
OF ARRAY
2555A11

Fig. 19

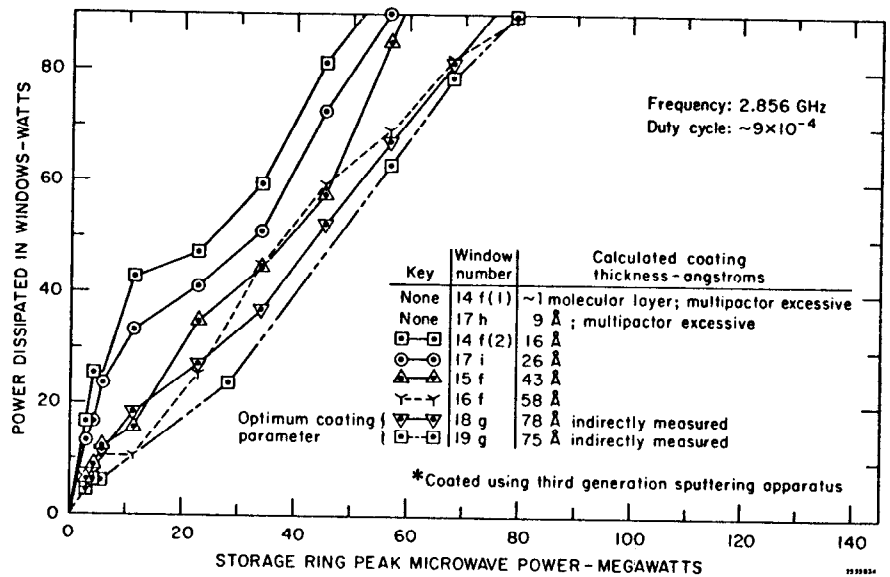
POWER DISSIPATED IN WINDOWS-WATTS
STORAGE RING PEAK MICROWAVE POWER-MEGAWATTS
0
20
40
60
80
100
120
140
Frequency: 2.856 GHz
Duty cycle: ~9×10⁻⁴
Key | Window number | Calculated coating thickness-angstroms
None | 14 f(1) | ~1 molecular layer; multipactor excessive
None | 17 h | 9 Å ; multipactor excessive
14 f(2) | 16 Å
17 i | 26 Å
15 f | 43 Å
16 f | 58 Å
Optimum coating parameter
18 g | 78 Å indirectly measured
19 g | 75 Å indirectly measured
*Coated using third generation sputtering apparatus

Fig. 20

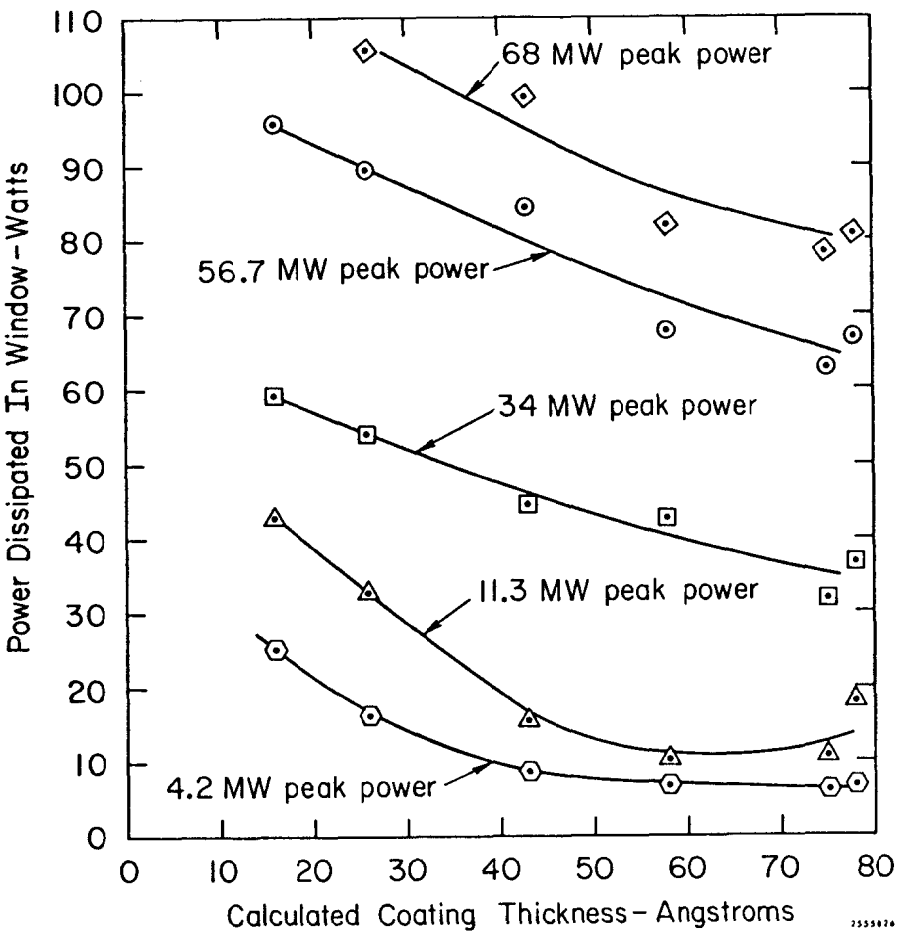
68 MW peak power
56.7 MW peak power
34 MW peak power
11.3 MW peak power
4.2 MW peak power
Power Dissipated In Window - Watts
Calculated Coating Thickness - Angstroms
0
10
20
30
40
50
60
70
80
90
100
110

Fig. 21