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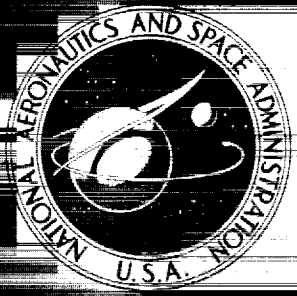

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HIGH TEMPERATURE CAPACITOR DEVELOPMENT

by R. E. Stapleton

Prepared by
WESTINGHOUSE ELECTRIC CORPORATION
Lima, Ohio
for Lewis Research Center

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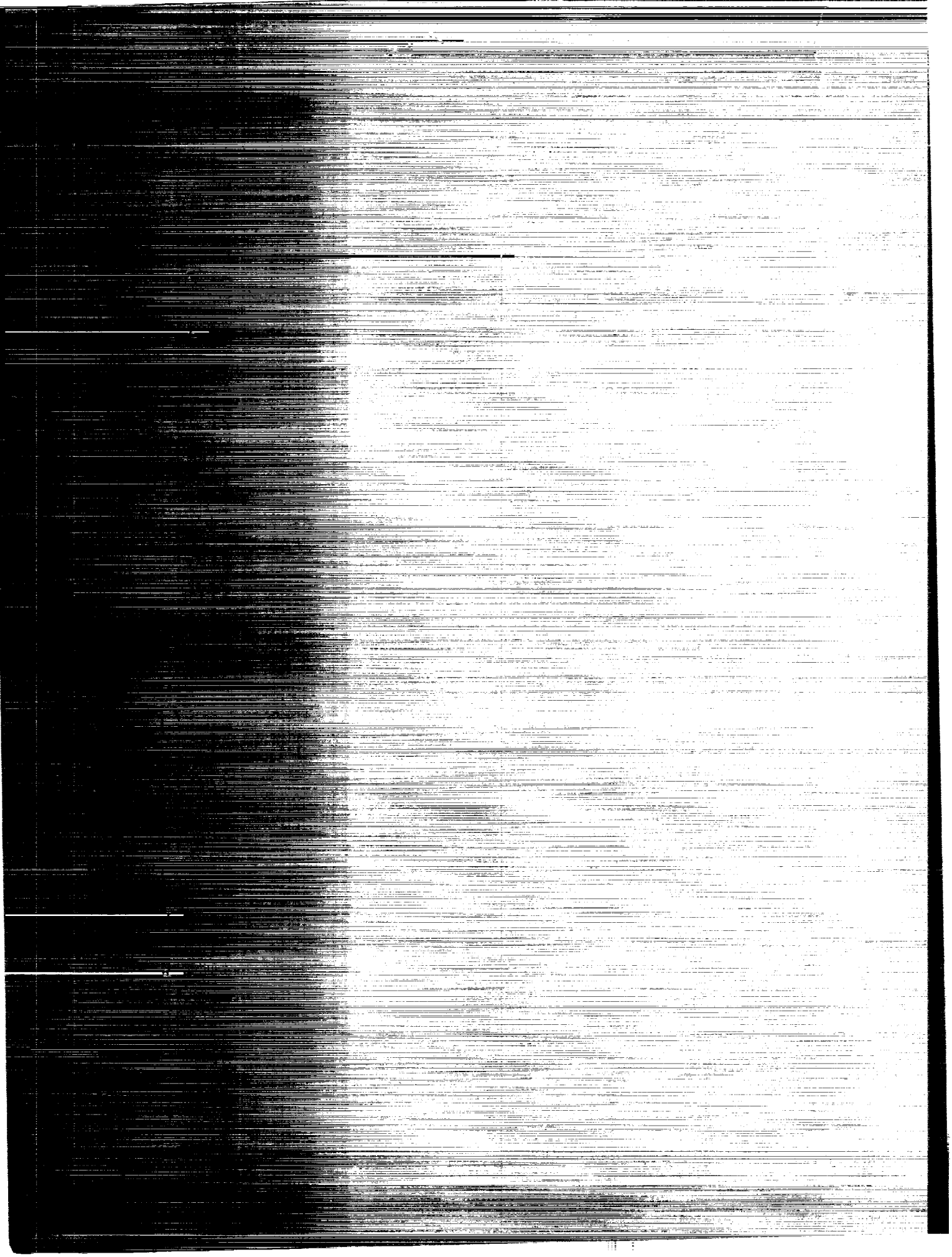

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16. Abstract

This report describes the progress made on improving the high-temperature stability and overall electrical properties of pyrolytic boron nitride capacitors which were developed under NASA Contract NAS 3-6465. One method investigated was surface texturizing of pyrolytic boron nitride wafer surfaces by radio frequency (rf) off-sputtering. Removal of 3000 angstroms of surface material before application of electrodes resulted in a two- to three-fold reduction in electrical losses. Surface texture (ratio of true to apparent or geometric area) was measured by a "double layer capacitance technique." These data were correlated with electrode adherence and capacitor losses and stability under thermal cycling and aging conditions at 1300^{o} and 1100^{o} F in vacuum ($<1\times10^{-6}$ torr). A second method that was studied was the deposition of a sputtered boron nitride barrier layer to a portion of the outer surfaces of the electrodes. Compression and aging tests conducted on multi-layered capacitors at 1100^{o} F in vacuum showed that the barrier layer prevented platinum electrodes, on alternate capacitor wafers in a stack, from diffusing (bonding) together. This resulted in greater capacitance stability and lower electrical losses.

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SECTION I

INTRODUCTION

Advanced space electric power systems will require magnetic materials, electrical conductors, and electrical insulations capable of long term stable operation at high temperatures in a vacuum or an alkali metal vapor environment. A series of programs has been directed toward determining the stability of materials and developing the materials and technology required to implement future space electric power systems.

This Topical Report is the result of work accomplished under contract NAS3-10941 for the Development of High-Temperature Electrical Materials. The program consists of three tasks as follows:

Task I - Continuation of a 1300° F Hot-Spot, 5000-Hour Test on Electrical Component Models to 10,000 Hours

Task II - High-Temperature Capacitor Development

Task III - Evaluation, Construction, and Endurance Testing of Compression Sealed Boron Nitride Slot Insulation

Task II is the subject of this report which presents the results of an investigation directed toward improving the high-temperature stability of pyrolytic boron nitride capacitors which were developed under NASA contract NAS3-6465.

High-temperature capacitor development was initiated on NAS3-6465 because capacitors available from commercial sources have limited maximum operating temperatures (up to 700° F for a few types). They are usually bulky and have rather high electrical losses at elevated temperatures. It will be necessary for advanced space electrical power systems to provide stable electric power and control for extended time periods. Operation of components such as capacitors at elevated temperatures and under high electric loads will be required.

The search for a better dielectric material for a light-weight, high-temperature capacitor has resulted in the selection of pyrolytic boron nitride (ref. 1). This material was available as thick blocks which were sliced and lapped into flexible and pinhole-free wafers as thin as 0.0004 inches. Capacitors were made by sputtering thin film electrodes on the wafer surfaces. Vacuum

tests showed that these capacators had a voltage breakdown of 7000 volts/mil at 1100° F, a capacitance change from room temperature to 1100° F of about 1.7 percent and a dissipation factor (tan δ) of less than 0.001 at 1100° F. A five-wafer stack of these capacitors which were continuously energized at voltages up to 1000 V dc/mil for over 1000 hours at 1100° F showed a capacitance change of less than three percent. This change in capacitance was attributed to a slight separation of electrodes from the pyrolytic boron nitride surfaces.

Section II of this report presents the results of two processing methods that substantially improved capacitor stability and overall electrical properties under aging and thermal cycling conditions. Section III contains the conclusions and recommendations derived from the experimental work, and Section IV lists the references specifically cited in this report. Also included is an appendix outlining specific pyrolytic boron nitride wafer slicing, lapping, and cleaning methods used to prepare test capacitors for this program. In addition, the test apparatus and methods used for electrical measurements are outlined in the appendix.

SECTION II

FABRICATION AND EVALUATION OF STABLE HIGH-TEMPERATURE PYROLYTIC BORON NITRIDE CAPACITORS

DISCUSSION

Background

The objective of this task was to make improvements in high-temperature pyrolytic boron nitride capacitor design, processing methods, and electrical performance. Results from a previous program (ref. 1) with pyrolytic boron nitride (PBN) capacitors showed a small decrease in capacitance and increase in losses (tan δ) after short (1000 hour) life tests at 1100° F in vacuum. These changes were attributed to a slight separation of electrodes from pyrolytic boron nitride surfaces in a stacked capacitor. This separation or loss of electrode adherence was minimized or eliminated by increasing the initial bonding of sputtered electrodes to boron nitride wafers and by preventing interelectrode diffusion bonding.

One method used to improve capacitor stability and electrical properties was surface texturizing. The texture or roughness of the wafers was varied over a range of values by mechanical (lapping) methods and by radio frequency (rf) off-sputtering up to 5000 angstroms of surface material. Removal of the mechanically disturbed surfaces was expected to result in lower capacitor losses as well as provide ultra clean surfaces for subsequent application of electrodes. Surface texture (ratio of true to apparent or geometric surface area) was measured by a "double layer capacitance" technique.

A second method studied was the application of a very thin barrier layer to a portion of the outer surfaces of the electrodes. The purpose of this layer was to prevent electrodes on alternate capacitor wafers in a stack from sticking together under the combined influence of compression, high temperature and vacuum. It was expected that greater capacitance, stability, and lower electrical losses after voltage excitation and thermal cycling would be achieved.

EXPERIMENTAL PROCEDURE AND TEST RESULTS

Electrical Characterization of Pyrolytic Boron Nitride

A group of tests were performed to determine if a new lot of pyrolytic boron nitride[1] obtained for this program was equivalent to that tested under an earlier program (ref. 1). Single-wafer capacitors approximately 1-mil-thick were prepared from a new lot of material and electrical properties (capacitance and dissipation factor) were measured at room temperature and 1100° F in vacuum. The direct current breakdown voltage was also measured at room temperature and 1100° F in vacuum. These data were compared to previous data obtained for single-wafer capacitors.

Fabrication of Single-Wafer Capacitors. - A new lot of pyrolytic boron nitride was received in the form of rectangular blocks 3 by 6 by 1/8 inches. Several one-inch square pieces were cut from these blocks and then sliced into wafers about 8 to 12 mils thick. These wafers were lapped to a nominal thickness of one mil. The slicing and lapping methods employed are described in Appendix A. The final surface finish on most of the wafers was achieved by final manual lapping with 3-micron alumina abrasive in a water slurry on a glass lapping plate. The wafer finish or surface texture produced in this manner is designated a "matte" finish. Wafers with this type of surface texture, when tested in a stacked configuration, exhibited the best overall electrical performance on the previous program. Several single-wafer capacitors were also produced with polished surfaces to compare dissipation factors (tan δ) of capacitors with matte surfaces. Polished surfaces were prepared using 0.3-micron alumina powder[2] on a silk lapping cloth. After final lapping or polishing and prior to the application of sputtered electrodes, the wafers were cleaned as described in Appendix B.

Sputtered platinum (99.95% purity) electrodes were applied using a radio frequency diode sputtering configuration described later in this report. Typical sputtering conditions were as follows:

(1)	Initial pump-down pressure	1×10^{-6} torr
(2)	Argon sputtering pressure	4 to 6 microns
(3)	Target to substrate distance	1.5 inches
(4)	Standing wave ratio	1.1

[1] "Boralloy" supplied by the Union Carbide Corporation, Carbon Products Division, New York, New York 10607

[2] Linde A supplied by the Linde Division, Crystal Products Dept., Union Carbide Corporation, East Chicago, Indiana

(5)	Radio frequency input power	380 watts
(6)	Magnet coil current	6 amperes
(7)	Sputtering time	20 minutes
(8)	Approximate electrode thickness	3500 angstroms

Wafers were about 0.8-inch square and electrodes were located in the center of the wafers. Electrodes were deposited first on one side of the wafers through a pyrolytic boron nitride mask. After venting the bell jar with argon to atmosphere, the wafers were turned over and the mask was repositioned coincident with the first electrode. The pump-down and sputtering sequence was then repeated as outlined above to apply an electrode on the opposite side of the wafer.

Room Temperature Electrical Properties. - Table I shows a comparison of room tempreature electrical properties of single-wafer capacitors with matte surfaces (group A) and with polished surfaces (group B). These data show that, in general, dissipation factor values for wafers with polished surfaces are lower than values of dissipation factor obtained on wafers with matte surfaces. It is also apparent from the data in table I, that, no significant differences in the high and low dissipation factor values were noted for capacitors made with lot 1 of pyrolytic boron nitride (ref. 1) and those made with the newer lot of material (lot 2) on this program. Different capacitance values are due to variations in wafer thicknesses that range from 0.6 to 1.1 mils and variations in electrode areas. The differences in dissipation factor values are believed to be caused by processing variables. These variables were small imperfections in the wafers introduced during the lapping and/or polishing operations and subsequent handling. Other factors contributing to these differences were moisture adsorption on the wafer surfaces during or just prior to measurement and the type of wafer surface finish.

As a further check of lot consistency, a group of capacitors were fabricated as a single batch (cleaning and sputtering of electrodes at the same time) to directly compare dissipation factor (tan δ) with different lots of material having the same surface finish. The results show that lower dissipation factor values were obtained on the new lot of pyrolytic boron nitride after the wafer surfaces were polished. These lower values are in the same range as those obtained for polished wafers previously (ref. 1).

Capacitor No. 1935456 originally was made with a matte surface finish and the capacitance and dissipation factor at 1 kHz were 282.407 picofarads and 0.001934 respectively. The electrodes were removed in aqua regia; the wafer was polished and cleaned and electrodes were reapplied. The measured capacitance value was 230.479 picofarads and the dissipation factor decreased to 0.000747. A smaller diameter electrode was applied which accounts for the

Table I. - Data Comparing Properties of Single-Wafer Capacitors Made with New and Old Lots of Pyrolytic Boron Nitride

Group	Pyrolytic Boron Nitride Lot[a]	Wafer Surface Finish	Platinum Electrode Area (in^2)	Calculated Wafer Thickness (inches)	Capacitance (pF)[b] and Dissipation Factor (tan δ) at Room Temperature				Comments
					1 kHz		10 kHz		
					C = pF	tan δ	C = pF	tan δ	
A	1	Matte	0.379	0.0008	353.91	0.00079	353.53	0.00060	Made on NAS3-10941
	1		0.379	0.0008	360.53	0.00102	360.02	0.00091	
	1		0.379	0.0007	395.88	0.00098	395.26	0.00065	
	1		0.379	0.0012	246.71	0.00119	246.33	0.00096	
	2		0.379	0.0010	287.61	0.00116	287.21	0.00097	Made on NAS3-10941
	2		0.364	0.0009	326.17	0.00094	325.75	0.00085	
	2		0.364	0.0010	277.86	0.00127	277.39	0.00108	Made on NAS3-6465
	2	Matte	0.364	0.0010	269.19	0.00071	268.93	0.00071	
B	1	Polished	0.217	0.0012	230.48	0.00075	230.25	0.00060	Made on NAS3-10941
	1		0.217	0.0009	182.12	0.00076	181.96	0.00058	
	2		0.364	0.0006	454.58	0.00061	454.23	0.00054	Made on Nas3-6465
	2		0.364	0.0004	657.28	0.00079	656.63	0.00072	
	2	Polished	0.364	0.0005	566.69	0.00108	565.90	0.00105	

(a) Lot identification refers to Boralloy, pyrolytic boron nitride plate material (Carbon Products Division, Union Carbide Corporation, 270 Park Ave., N.Y.C., N.Y.). Lot 1 was purchased on order 39-J-387991-CC and received August 24, 1965. Lot 2 was purchased on order 39-401673 and received January 11, 1967. Material specifications are in accordance with the manufacturer's quality control standards for Boralloy.

(b) Units of capacitance are in picofarads.

lower capacitance value. Another capacitor made from the same new lot of pyrolytic boron nitride material and with polished surfaces had a capacitance of 182.120 picofarads and a dissipation factor of 0.000761 measured at 1 kHz. All measurements were made with a digital capacitance measuring assembly (see Appendix C). The capacitors were lightly clamped (~50 grams pressure) in a fixture located in a shielded metal box. Connections were made to the bridge terminals with coaxial leads.

The overall results of the room temperature tests indicate that these variations in dissipation factor are primarily due to the type of surface finish (polished or matte) and to defects produced by lapping and/or polishing.

Electrical Measurements in Vacuum to 1100° F. - Table II shows the results of electrical characterization tests obtained for seven pyrolytic boron nitride capacitors made from the new lot of material. Each capacitor had 0.695-inch-diameter platinum electrodes (~3500 angstroms thick) sputtered on matte wafer surfaces. Wafer thicknesses were in the range of 1 mil (0.72 to 1.2 mils). Wafer thicknesses were calculated from the electrode area (0.379 square inches) using a dielectric constant of 3.4 for pyrolytic boron nitride and the measured capacitance.

Capacitance and dissipation factor (tan δ) were measured at room temperature and 1100° F in vacuum ($<1 \times 10^{-6}$ torr) for six of the capacitors shown in table II. (Refer to Appendix C for a description of test methods.) The direct current breakdown voltage was measured on four capacitors at room temperature and three capacitors at 1100° F in vacuum ($<1 \times 10^{-6}$ torr). The direct current breakdown voltage was obtained by manually increasing the voltage at a rate of approximately 1000 volts/second. A calibrated oscilloscope trace of voltage versus time was observed and photographed for each test run. The first indication of a voltage drop on the oscilloscope record was taken as the voltage breakdown level. The breakdown voltage level divided by the calculated wafer thickness (volts/mil) is shown in table II.

Oscilloscope photographs were made to show typical breakdown conditions at room temperature and 1100° F (capacitor Nos. 1935462 and 1935461 from table II). Figure 1 shows three successive breakdown runs at room temperature in vacuum for capacitor No. 1935462. The horizontal scale represents one second per centimeter (rate of increase in voltage is approximately 1000 volts per second) and the vertical scale is calibrated at 2000 volts per centimeter. During the first breakdown run, this capacitor punctured at about 11,000 volts and the power supply relay tripped dropping the voltage to zero. The power supply was then reactivated and a second breakdown run was made. A voltage level of 10,000 volts was reached before any indication of a voltage drop was recorded. A third run was made and it can be seen from the trace in figure 1 that momentary voltage drops occurred first at 9,000 volts but the relay did not trip until about 10,500 volts was reached.

Figure 2 shows a photomicrograph of the breakdown puncture hole in the capacitor wafer (capacitor No. 1935462). Evidence of removal by vaporization of the thin film platinum electrode from the immediate vicinity of the puncture may be seen in the photomicrograph. This self-healing effect explains the ability to reapply high voltage levels a number of times in vacuum after an initial dielectric breakdown or puncture occurs.

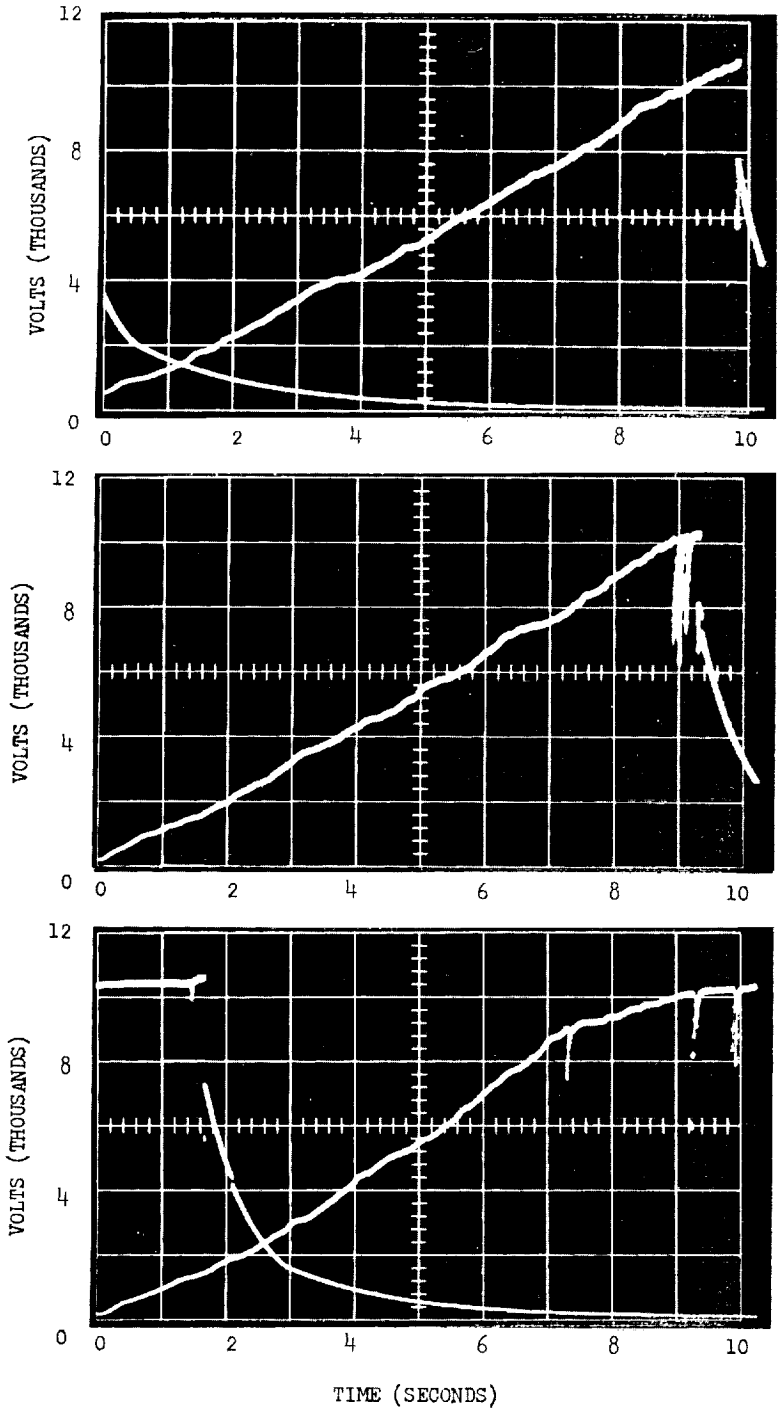
After the capacitor was subjected to this series of breakdowns, the measured capacitance and dissipation factor were 261.83

Table II. - Electrical Tests on Single-Wafer Capacitors Made From a New Lot (2) of Pyrolytic Boron Nitride (Sputtered Platinum Electrodes, Matte Surface Finish)

Capacitor Identification Number	Capacitance (pF) [b] and Dissipation Factor (tan δ) at Room Temperature (<1x10^{-6} torr)				Capacitance (pF) [b] and Dissipation Factor (tan δ) at 1100°F (<1x10^{-6} torr)				Calculated Wafer Thickness (Electrode Area = 0.379 in^2)	DC Voltage Breakdown in vacuum (<1x10^{-6} torr)	
	1 kHz		10 kHz		1 kHz		10 kHz			Room Temperature	1100°F
	C = pF	tan δ	C = pF	tan δ	C = pF	tan δ	C = pF	tan δ			
1935462	292.47	0.00138	291.98	0.00108	287.24	0.00590	285.68	0.00314	0.996 mils	11,000 v/mil	----
1935460	313.73	0.00139	313.18	0.00098	309.21	0.0088	307.40	0.00360	0.923 mils	10,800 v/mil	----
1935476	289.22	0.00246	285.93	0.00256	--	--	--	--	1.00 mils	11,200 v/mil	----
2466998	399.61	0.00137	398.95	0.00112	400.05	0.00771	398.76	0.00271	0.725 mils	6,900 v/mil [a]	----
1935475	242.74	0.00149	242.26	0.00140	241.72	0.00943	240.00	0.0045	1.200 mils	----	9400 v/mil
1935463	289.43	0.00116	289.18	0.00101	288.40	0.00491	287.41	0.00322	1.01 mils	----	7900 v/mil
1935461	319.36	0.00106	318.93	0.00085	320.65	0.00318	319.48	0.00252	0.912 mils	----	9300 v/mil

(a) The 6,900 volts/mil value was not used in obtaining an average room temperature breakdown value because the wafer was significantly thinner than 1 mil.

(b) Units of capacitance are in picofarads.



(a)

First Breakdown

(b)

Second Breakdown

(c)

Third Breakdown

Figure 1. - Oscilloscope Traces of DC Voltage Versus Time of Three Breakdown Tests on Pyrolytic Boron Nitride Capacitor No. 1935462 (~1.0-Mil Thick) at Room Temperature in Vacuum ($<1 \times 10^{-6}$ torr)

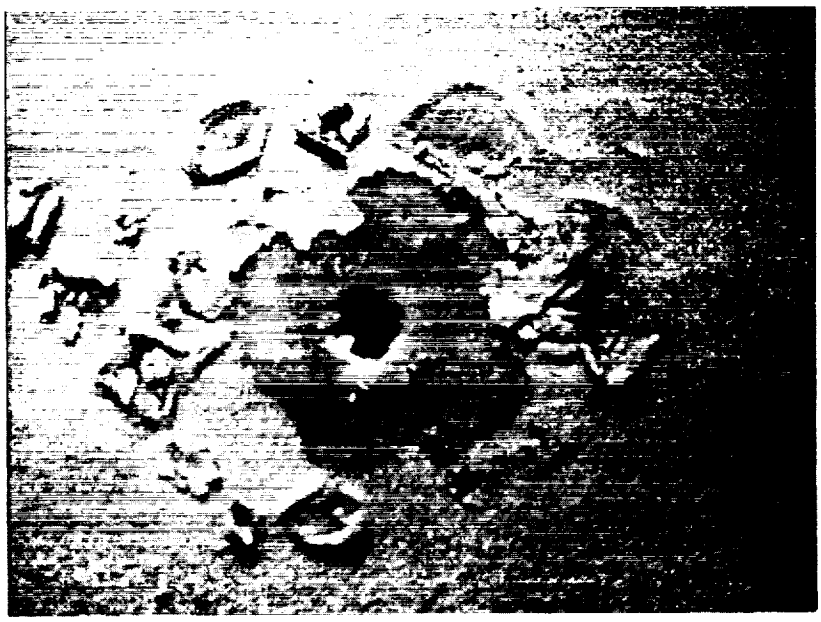
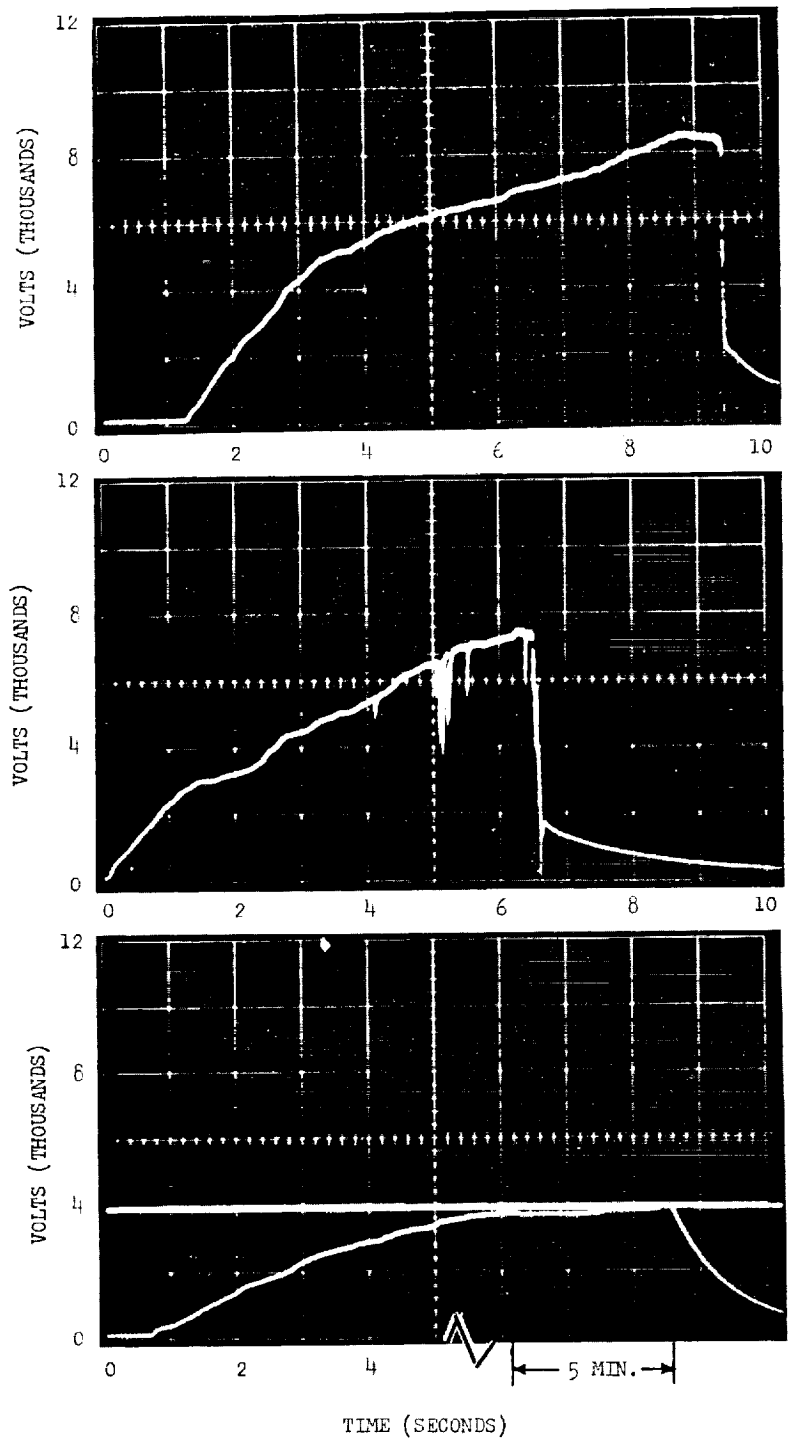


Figure 2. - Photomicrograph of the Breakdown Area on Pyrolytic Boron Nitride Capacitor No. 1935462 Tested at Room Temperature in Vacuum (<1 x 10^{-6} torr) 80 X

picofarads and 0.000903 respectively at room temperature, indicating no apparent degradation in electrical properties other than a reduction in capacitance.

Figure 3 shows oscilloscope traces of two successive breakdown tests at 1100° F in vacuum on capacitor No. 1935461. The first breakdown for this capacitor occurred at about 8500 volts. This lower voltage level compared to 11,000 volts for capacitor No. 1935462 is due to the higher test temperature (1100° F versus 72° F). After two breakdowns at 1100° F, a third test was made at a lower voltage level (about half the breakdown voltage or 4000 volts). The voltage was held constant at this level for five minutes without any indication of arcing or failure.

Figure 4 shows a photomicrograph of the breakdown area in this capacitor. It should be noted that a number of small puncture holes were developed compared to only one large hole for the capacitor tested at room temperature (figure 2). Both photomicrographs were at the same magnification (80 X).



(a)

First Breakdown

(b)

Second Breakdown

(c)

Third Run - Voltage increased to 4000 V dc, held for 5 minutes

Figure 3. - Oscilloscope Traces of DC Voltage Versus Time of Two Breakdown Tests Followed by a Five-Minute Constant Voltage Test on Pyrolytic Boron Nitride Capacitor No. 1935461 (~0.91 Mils Thick) at 1100° F in Vacuum ($<1 \times 10^{-6}$ torr)

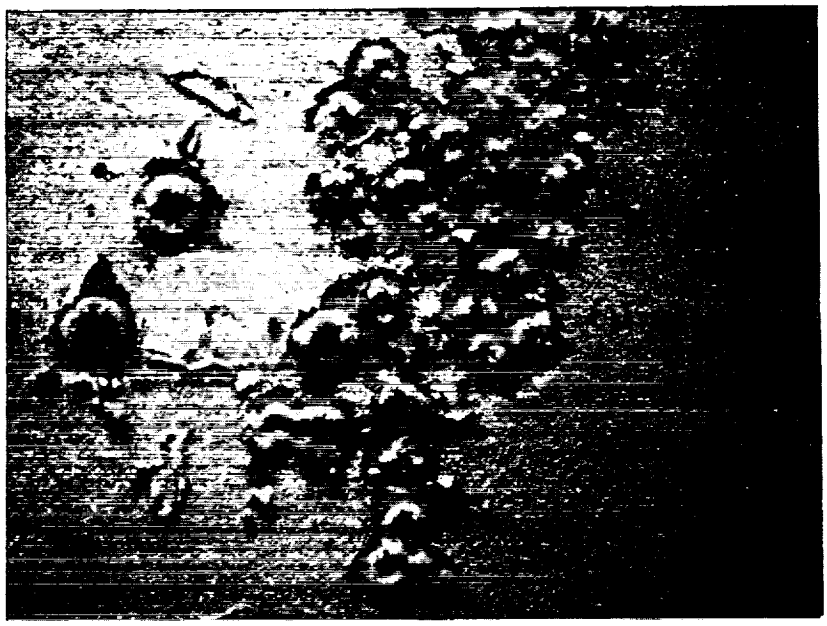


Figure 4. - Photomicrograph of the Breakdown Area on Pyrolytic Boron Nitride Capacitor No. 1935461 Tested at 1100° F in Vacuum ($<1 \times 10^{-6}$ torr) 80 X

To summarize, these data show that the average direct current voltage breakdown strength at room temperature in vacuum was 11,000 volts per mil (three capacitors, 0.92 to 1.01 mils thick, 0.695-inch diameter electrodes) and 8770 volts per mil at 1100° F (three capacitors, 0.91 to 1.2 mils thick, 0.695-inch diameter electrodes). Therefore, breakdown voltages are better than those obtained for pyrolytic boron nitride capacitors made previously (ref. 2). Dissipation factor values are comparable at 1100° F to those obtained previously for matte wafers tested in a stacked configuration.

Pyrolytic Boron Nitride Wafer Texturizing (Surface Texture Measurements By The Double Layer Capacitance Method)

The capacitance of the electric double layer of a hydrogen film on the surface of platinum replica films immersed in an aqueous electrolyte was selected as the approach to measure the true surface areas of pyrolytic boron nitride capacitor wafers. The purpose of these measurements was to:

(1) Establish a feasible range of surface texture ratios that can be produced on pyrolytic boron nitride surfaces by controlled mechanical methods (lapping and polishing) and by radio frequency off-sputtering.

(2) Determine an optimum platinum film thickness needed to accurately measure the expected range of surface textures produced on reference surfaces of pyrolytic boron nitride. The optimum thickness is considered to be the thickness where a complete, conformal conducting layer is developed over the entire surface topography of the substrate. A film that is thicker than the optimal value may tend to form a secondary texture that is not representative of the actual pyrolytic boron nitride surface.

If it is assumed that the thickness and dielectric constant of the double layer are constant, the capacitance of the double layer will be directly proportional to the surface area of the electrode on which it forms. An ideally smooth surface will have a true area equal to its geometric area. But a surface with some degree of roughness will have a true area that is greater than the geometric area. Therefore, the capacitance of the double layer will increase in direct proportion to the increase in surface area due to surface roughness. It is also assumed that a sputtered thin platinum film deposited on smooth and roughened surfaces forms a good replica of the underlaying substrate surface texture. A highly polished glass surface represents a solid substrate which has a true to geometric surface area ratio equal to one.

Double Layer Capacitance Test Assembly. - The surface area of various specimens has been tested in a one-normal Na_2SO_4 solution using a circuit similar to that of McMullen and Hackerman (ref. 3) Figure 5 is a schematic diagram of the electrical circuit used. A variable-frequency, variable-amplitude, square-wave oscillator operating into a 50-watt audio amplifier provided the square-wave generation. The resistor R_S should be non-inductive to avoid preferential attenuation of the high-frequency components of the square wave. The chemical cell was a standard 2-liter chemical reaction vessel of Pyrex brand glass; the electrometer measured the direct current bias of the test electrode with respect to the standard calomel cell through a sodium sulfate salt bridge. The direct current bias was applied by a variable direct current voltage source through a 12-henry choke to keep the square-wave voltage from affecting the direct current instruments. The platinum reference electrode N was a platinum wire screen cylinder of 2-inch diameter. The sample electrode was supported by a platinum wire holder; the capacitance of the holder without the sample was determined prior to insertion of the sample. Platinum lead wires were used to make electrical connections to the test sample immersed in the electrolyte.

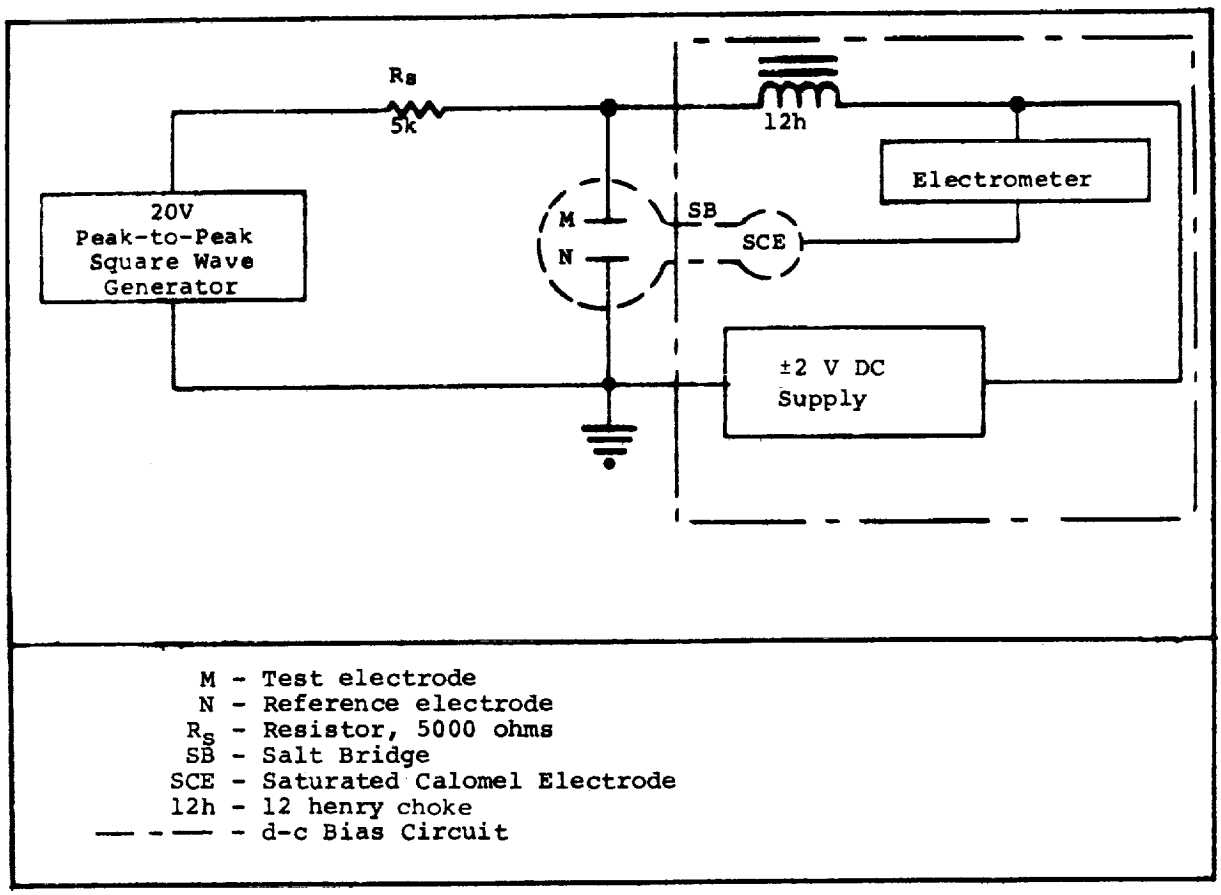


Figure 5. - Schematic Diagram of the Double Layer Capacitance Test Circuit

The values of double layer capacitance developed during the measurement of the surface area of polarized electrodes was expected to be in the range from about 1 to 600 microfarads. An electrical circuit for the electrochemical test apparatus which was used in measuring double layer capacitance was assembled. Initial tests were conducted to verify the range and accuracy capability of the capacitance measuring circuit.

This set-up was used to measure the values of non-polarized capacitors from approximately 1 to 600 microfarads. This simulation showed the anticipated accuracy of capacitance measurement to be ±5 percent up to 500 microfarads. Above 500 microfarads, the accuracy was expected to degrade because the very small alternating current signal which must be detected approaches the level of the background noise.

Table III shows the capacitance measurement obtained on ten

Table III. - Comparison of Capacitance Values Obtained by the Charging Curve Method of Measurement With Those Obtained by the Capacitance Bridge Method

Frequency (hertz)	Incremental Time (seconds)	Series Resistance (ohms)	Applied Voltage (volts)	Voltage Increment Across Capacitor (volts)	Capacitance-Charging Curve Method (microfarads)	Capacitance-Capacitance Bridge Method (microfarads)
1000	5×10^{-4}	15×10^{3}	5	0.092	0.91	0.943
1000	5×10^{-4}	15×10^{3}	10	0.18	0.926	0.943
1000	5×10^{-4}	15×10^{3}	5	0.008	10.4	10.1
1000	5×10^{-4}	15×10^{3}	15	0.024	10.4	10.1
1000	5×10^{-4}	15×10^{3}	20	0.014	23.8	23.6
1000	5×10^{-4}	15×10^{3}	20	0.001	33.3	33.5
500	10^{-3}	15×10^{3}	20	0.0135	49.4	49.0
500	10^{-3}	15×10^{3}	20	0.009	74	71.6
500	10^{-3}	15×10^{3}	20	0.008	83	81.5
500	10^{-3}	1×10^{4}	20	0.003	222	241
500	10^{-3}	2×10^{3}	20	0.021	238	241
500	10^{-3}	1×10^{3}	20	0.040	250	241
500	10^{-3}	1×10^{3}	20	0.027	370	377
500	10^{-3}	1×10^{3}	20	0.019	526	500

test capacitors using the electrical test assembly which was utilized for measuring double layer capacitance. It may be noted that the accuracy of measurements obtained by the charging curve method is ±5 percent.

Measurements of the Surface Area of Polarized Electrodes. - Double layer capacitance measurements were made using a standard size pyrolytic boron nitride disk (0.99 cm^2 apparent or geometric surface area). The first test series to determine an optimum platinum film thickness was made with four different initial reference textures. These were polished and lapped or matte finishes produced with 0.05, 3, 9, and 15 micron alumina abrasives. Successively thicker platinum electrodes were sputtered onto the texturized surfaces. The double layer capacitance was measured after each incremental increase in film thickness over a range from about 200 to 5000 angstroms. These measurements were made on the exposed (top) platinum electrode surface.

A definite functional trend was observed. A very rapid increase in the double layer capacitance occurred for all surface textures as the platinum film thickness was increased from 200 to

approximately 1500 angstroms. A gradual leveling off in capacitance values took place for thicker platinum films (>1500 angstroms) and generally constant values were observed for films thicker than approximately 3000 angstroms. Based on these data, a 3000-angstrom-thick platinum film was selected for subsequent tests.

The various data points in figure 6 were obtained by first polishing standard pyrolytic boron nitride disks and then off-sputtering different sample groups for 5, 20, 35, and 50 minutes and 1, 2, 3, and 4 hours. Platinum films (3000-angstroms-thick) were then sputtered onto the off-sputtered surfaces and the double layer capacitance was measured. The surface texture ratio was calculated by dividing each of these values by 18.2 $\mu F/cm^2$.

A value of 18.2 $\mu F/cm^2$ was taken for the apparent geometric area of a platinum film sputtered onto a highly polished glass surface. This value is an average of five test samples. It is evident from figure 6 that a very non-linear and rapid increase in texture ratio occurs between zero and one hour of off-sputtering. A smooth curve has been fitted between these data points to approximate the relationship between surface texture and off-sputtering time for polished substrate starting surfaces. Figure 6 also shows that a polished pyrolytic boron nitride surface (no off-sputtering) has a texture ratio of 1.3 or the surface is about 30 percent rougher than a highly polished glass surface. Examination of polished pyrolytic boron nitride surfaces under high magnification reveal a number of fine scratches. These scratches are very difficult to avoid during polishing because of the intrinsic softness of pyrolytic boron nitride (two on the Mohs scale). Some improvement in the polishing technique may be possible, but it does not appear practical to achieve further significant increase in smoothness.

Figure 7 shows that no important change in the surface texture ratio is produced by off-sputtering (up to 8 hours) when the starting surfaces are of the as-lapped or matte type (3-micron alumina abrasive). Data have also been obtained that show this relationship holds for matte surfaces produced with coarser abrasives (9 and 15 micron alumina).

Data were consistent as long as the electrolyte was not contaminated. Extreme cleanliness is required to obtain repeatable electric double layer capacitance measurements. The presence of even traces of organic contaminants in the electrolyte has a large scattering effect on the data. All samples were vapor degreased in iso-propyl alcohol before testing. Individual readings varied by as much as ten percent because of the limits of resolution and low signal-to-noise ratio of the measured signal.

It has been found that the direct current bias and reference cell can be omitted from the circuit without affecting the consis-

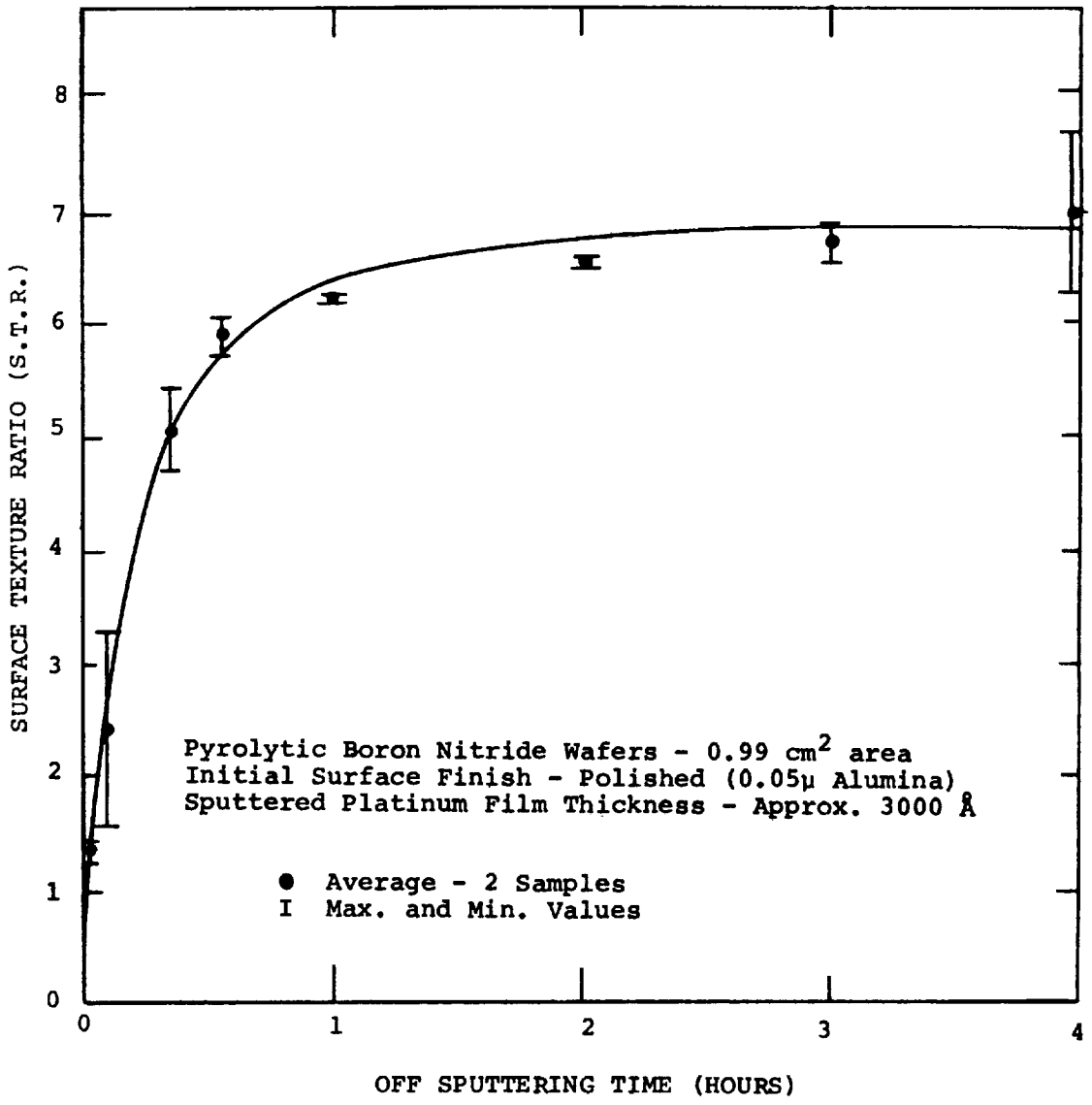


Figure 6. - Surface Texture Ratio Versus Off-Sputtering Time (Hours) for Pyrolytic Boron Nitride Wafers Initially Prepared with Polished Surfaces

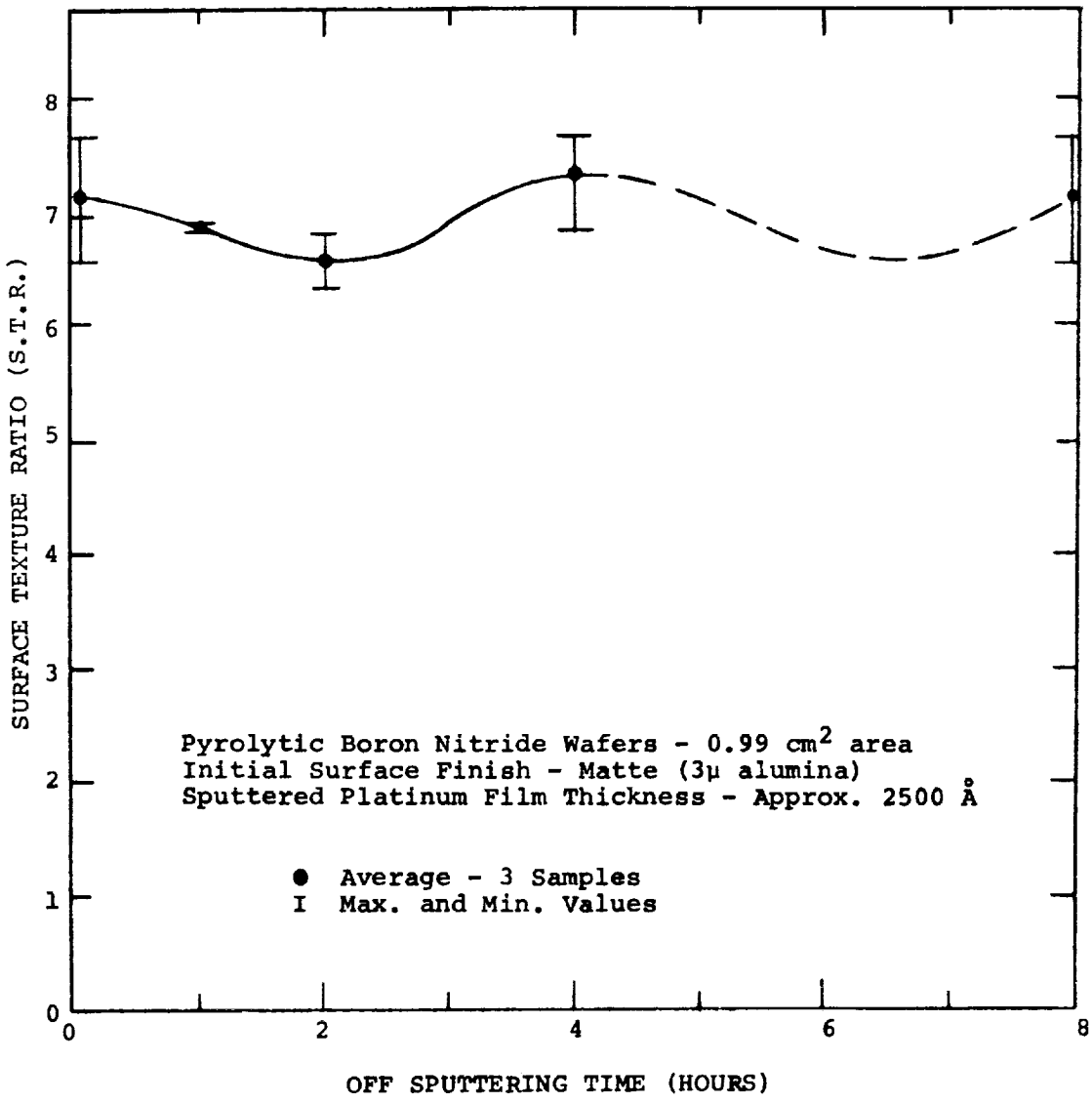


Figure 7. - Surface Texture Ratio Versus Off-Sputtering Time (Hours) for Pyrolytic Boron Nitride Wafers Initially Prepared with Matte (3μ Alumina) Surfaces

tency of the data. The numerical results given above were obtained with the bias and reference circuit disconnected.

In summary, these data show that surface texture ratios between 1.3 and 7.1 can be produced on pyrolytic boron nitride surfaces. To obtain ratios less than six it was necessary to start with polished surfaces and off-sputter for periods of time less than about 35 minutes.

Surface Area Measurements of the Pyrolytic Boron Nitride - Platinum Interface. - A platinum film transfer technique was investigated in order to compare the capacitance of the electric double layer of a hydrogen film on both surfaces (outer surface and the interface surface) of sputtered platinum films on texturized pyrolytic boron nitride. Data reported in the preceeding paragraphs are for the outer platinum surface.

A transfer technique was used to bond the platinum to a glass surface at 1300° F while the platinum film was lightly and uniformly pressed against the glass surface. This method was found to be satisfactory for platinum films sputtered onto polished boron nitride. However, after a number of trials were made with platinum on texturized boron nitride, it was evident that the method was not reliable. In all instances, it was observed that small sections of boron nitride were pulled from the wafer and remained bonded to the platinum film as shown in figure 8. This reduced the geometric surface area of the platinum and resulted in misleading double layer capacitance measurements on the platinum interface surface.

Thermal Cycling From 1300° and 1100° F

The previous discussion has outlined the methods and results of surface texturizing treatments on pyrolytic boron nitride. It has been shown that a variety of surface textures (surface texture ratios) can be produced ranging from a minimum value of 1.3 to a maximum value of 7.1. The following paragraphs will present the results of a series of thermal cycling tests performed on actual pyrolytic boron nitride capacitor wafers with six different surface texture ratios ranging from 1.3 to 7.1.

Thermal Cycling Tests (Pyrolytic Boron Nitride Capacitors - Platinum Electrodes). - Tabbed capacitor wafers were fabricated for this test series as follows:

1) Surface Texture Ratio of 1.3. Standard slicing and lapping procedures were used to produce wafers about 1.5 mils thick (see Appendix A). Final lapping was completed with 3-micron alumina abrasive. The wafers were then carefully polished on both sides with 0.05-micron alumina. A glossy surface appearance was produced. The polishing operations had removed 0.5 mils of surface material

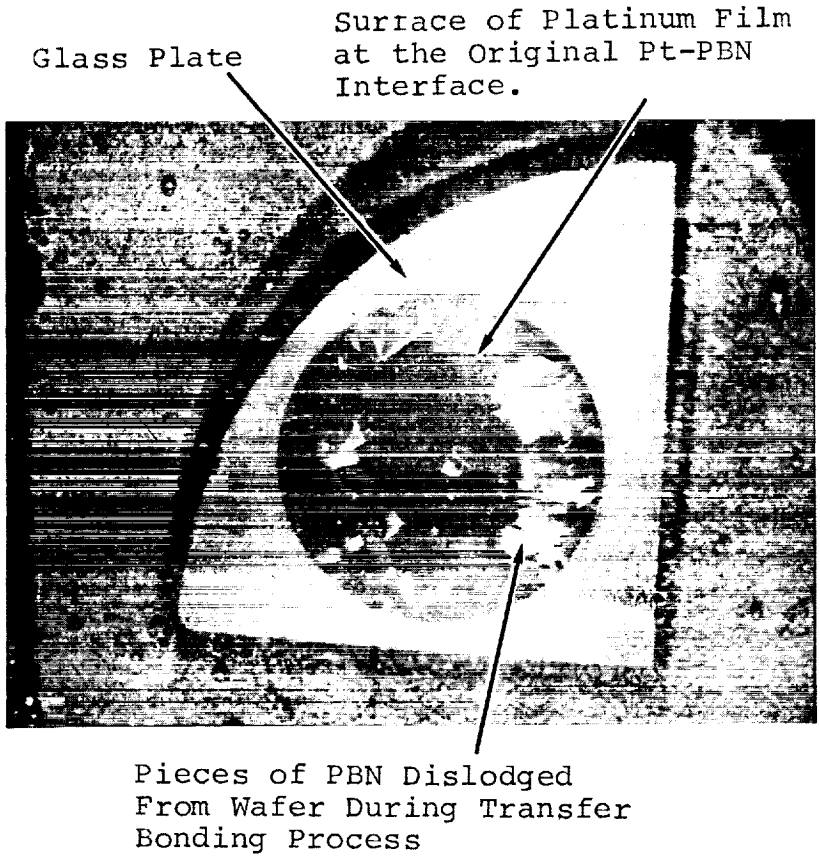


Figure 8. - Photomicrograph of the Surface at the Platinum-Pyrolytic Boron Nitride (Pt-PBN) Interface of a Sputtered Platinum Film Initially Deposited on a Texturized (STR = 7.2) Pyrolytic Boron Nitride Wafer. The Platinum Film was Reverse Transfer Bonded to a Glass Plate.

yielding a final wafer thickness of about 1.0 mils. After thorough cleaning (see Appendix B) sputtered platinum electrodes were applied.

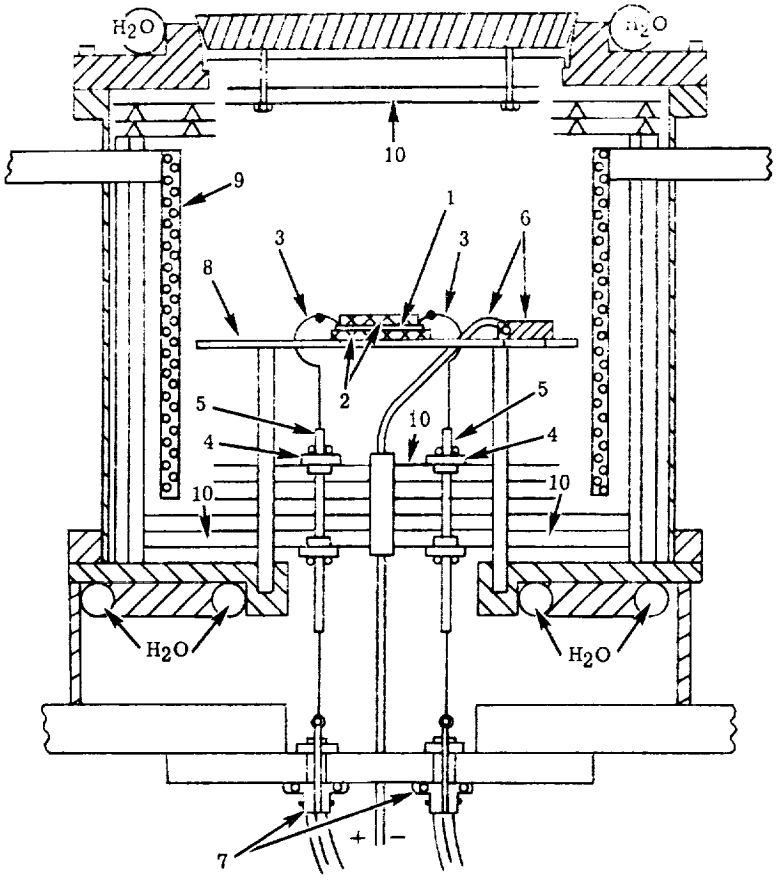
2) Surface Textures Ratios of 3.3 to 5.8. The same procedure was followed as outlined above. After polishing and cleaning however, the wafers were off-sputtered using the methods described previously. One wafer was off-sputtered (both sides) for seven minutes. Figure 6 showed that the surface texture ratio was 3.3. Another wafer was off-sputtered for 14 minutes producing a surface texture ratio of 4.3. The third wafer was off-sputtered for 35 minutes resulting in a surface texture ratio of 5.8. After off-sputtering, platinum electrodes were applied.

3) Surface Texture Ratio of 7.0. This surface texture ratio was produced by off-sputtering a wafer with a lapped or matte surface rather than a polished surface. Figure 7 showed that 35 minutes of off-sputtering will produce a surface texture ratio of about 7 when the starting surface has a lapped finish (3-micron alumina). In addition, figure 7 shows that off-sputtering a pyrolytic boron nitride wafer with a matte starting surface produces no appreciable increase in the surface texture ratio. Therefore, a sixth capacitor was included in this test series that was final lapped with 3-micron alumina but not off-sputtered.

In summary, a total of six single wafer, tabbed pyrolytic boron nitride capacitors were prepared. Five capacitors had surface texture ratios of 1.3, 3.3, 4.3, 5.8, and 7.0. The sixth capacitor had a texture ratio of about 7.1 but was not off-sputtered. Figures 6 and 7 provided the basis for determining surface texture ratios.

A cold-wall, liquid-nitrogen-trapped vacuum furnace was modified and adapted to test a maximum of three individual test capacitors during a single pump-down cycle. The furnace can be automatically cycled between room temperature and 1300° F. Six coaxial electrical feedthroughs were installed so that shielded cable can be connected between the feedthroughs and the capacitance bridge thus minimizing pick-up and stray capacitance effects. Figure 9 shows a sectional representation of the test furnace. A tungsten mesh heating element, which is capable of heating the furnace to 5000° F, was used.

Initially, five capacitors were setup in the furnace and electrically interconnected to the feedthroughs. One side of each capacitor was connected to a common lead wire and the other side was connected to individual lead wires and feedthroughs. Capacitance and dissipation factor measurements were made at room temperature and 1100° F. However, large differences in electrical properties were observed when one capacitor was being tested, and the others were either grounded or ungrounded. It was decided to



1 - Tabbed Pyrolytic Boron Nitride Capacitor Wafer

2 - Pyrolytic Boron Nitride Holding Fixture

3 - Platinum Lead Wires Welded to Platinum Foil in Contact With Platinum Capacitor Electrodes that Extend over Tab Areas

4 - Pyrolytic Boron Nitride Insulators

5 - Alumina Guide Tubes for Platinum Lead Wires

6 - Chromel-Alumel Thermocouple Embedded in a Pyrolytic Boron Nitride Block for Temperature Measurement and Control

7 - Coaxial Feedthroughs and Cable to Capacitance Bridge

8 - Molybdenum Support Table

9 - Tungsten Mesh Heating Elements

10 - Tantalum and Molybdenum Heat Shields

Figure 9. - Sectional View of the Cold-Wall Vacuum Test Furnace Showing Thermocouple Placement and the Electrical Interconnection Between a Tabbed Pyrolytic Boron Nitride Capacitor and Holding Fixture and a Set of Coaxial Feedthroughs

remove two capacitors so that separate lead wires could be connected to each capacitor. This approach eliminated the interference effects caused by the "common lead wire" connection method.

Figure 9 shows a single capacitor connection to schematically illustrate the overall furnace and final test configuration. A tabbed capacitor was clamped between two pyrolytic boron nitride retaining plates and electrical contact made at the tab areas. Pyrolytic boron nitride was also used to electrically isolate each lead wire as it passed through the heat shields. The pyrolytic boron nitride insulators were firmly fixed to retain their position during the tests. A chromel-alumel thermocouple was embedded in a block of pyrolytic boron nitride to monitor and control the capacitor wafer temperature. The thermocouple was located in close proximity to the array of test capacitors during a test run.

Three complete thermal cycling test runs were made. In the first run one capacitor was tested for 50 cycles between 300° and 1300° F to determine equipment capability. During the second 50 cycle run, three capacitors were tested and during the third 50 cycle run, two capacitors were tested. A total of six capacitors were evaluated. The heating rate was controlled at 1100° F per hour (300° to 1300° F) and the cooling rate was determined by the thermal inertia of the furnace and fixturing. (Approximately 1000° F per hour from 1300° to 300° F.) The furnace was maintained at 1×10^{-6} torr or less during the thermal cycling tests.

Figures 10 and 11 show the change in dissipation factor as a function of the number of thermal cycles for the six capacitors. Figure 10 shows these data measured, after the furnace had been allowed to cool to room temperature (~72° F). Figure 11 shows the 1300° F data. It is evident from both of these figures that no important degradation effects occurred. The most significant observation is the comparison of absolute values of dissipation factor for capacitors with different surface texturizing treatments. Both 72° and 1300° F data show the following general trend:

(1) The highest dissipation factors were obtained for capacitors that were not off-sputtered. This includes both the capacitor with a lapped or matte surface finish (surface texture ratio = 7.1) and the capacitor with a polished surface finish (surface texture ratio = 1.3).

(2) The lowest dissipation factors were obtained for capacitors with off-sputtered surfaces. Progressively lower dissipation factors are evident as the off-sputtering time was increased from 7 to 35 minutes. The data show that a surface texture ratio of 7 obtained by off-sputtering a matte wafer for 35 minutes produces a capacitor with the lowest dissipation factor.

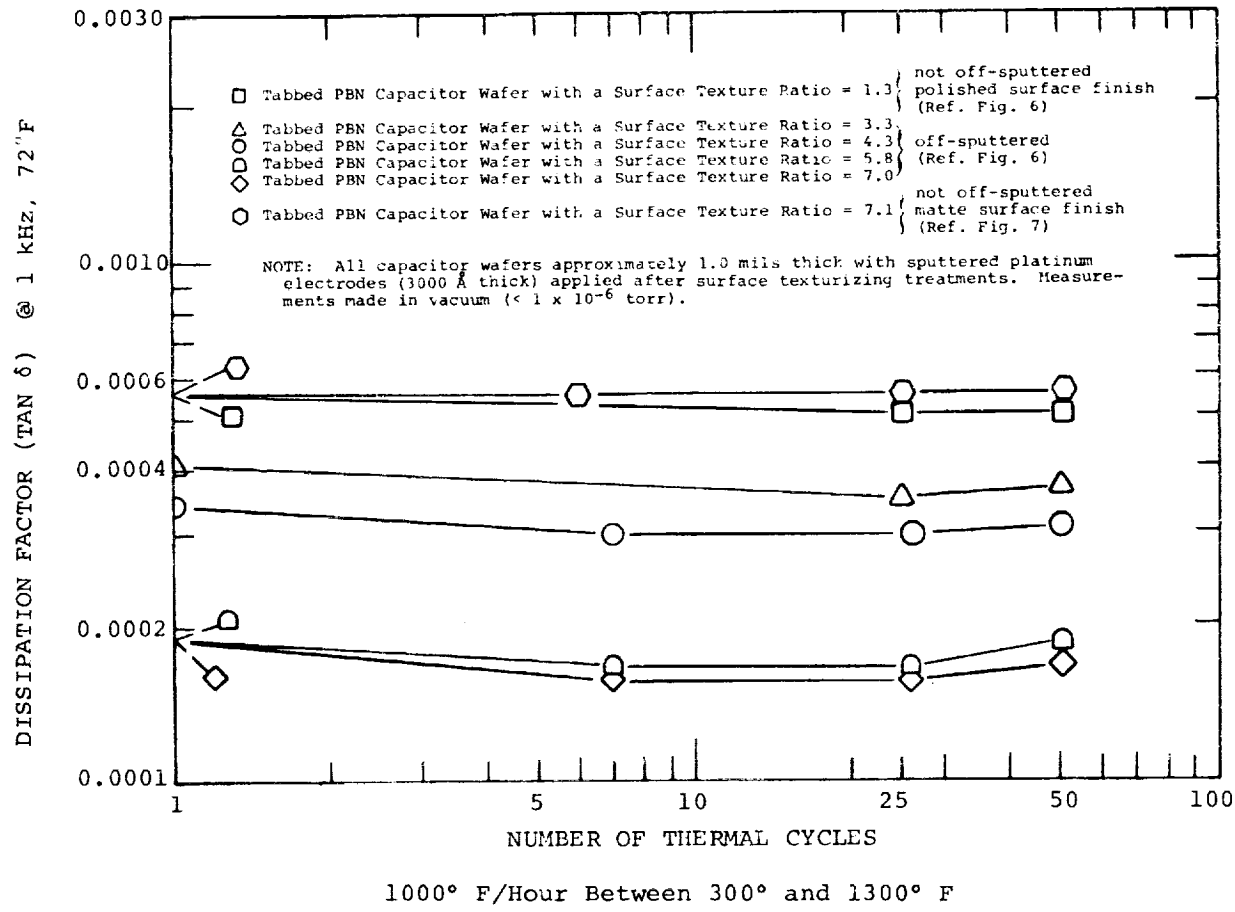


Figure 10. - Comparison of Dissipation Factors (Tan δ) Measured at 72° F for Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes) Versus the Number of Thermal Cycles in Vacuum Between 300° and 1300° F. Tabbed PBN Capacitors Made From Wafers with Different Surface Texture Ratios (1.3 to 7.1). Rate of Temperature Change was 1000° F/Hour Between 300° and 1300° F.

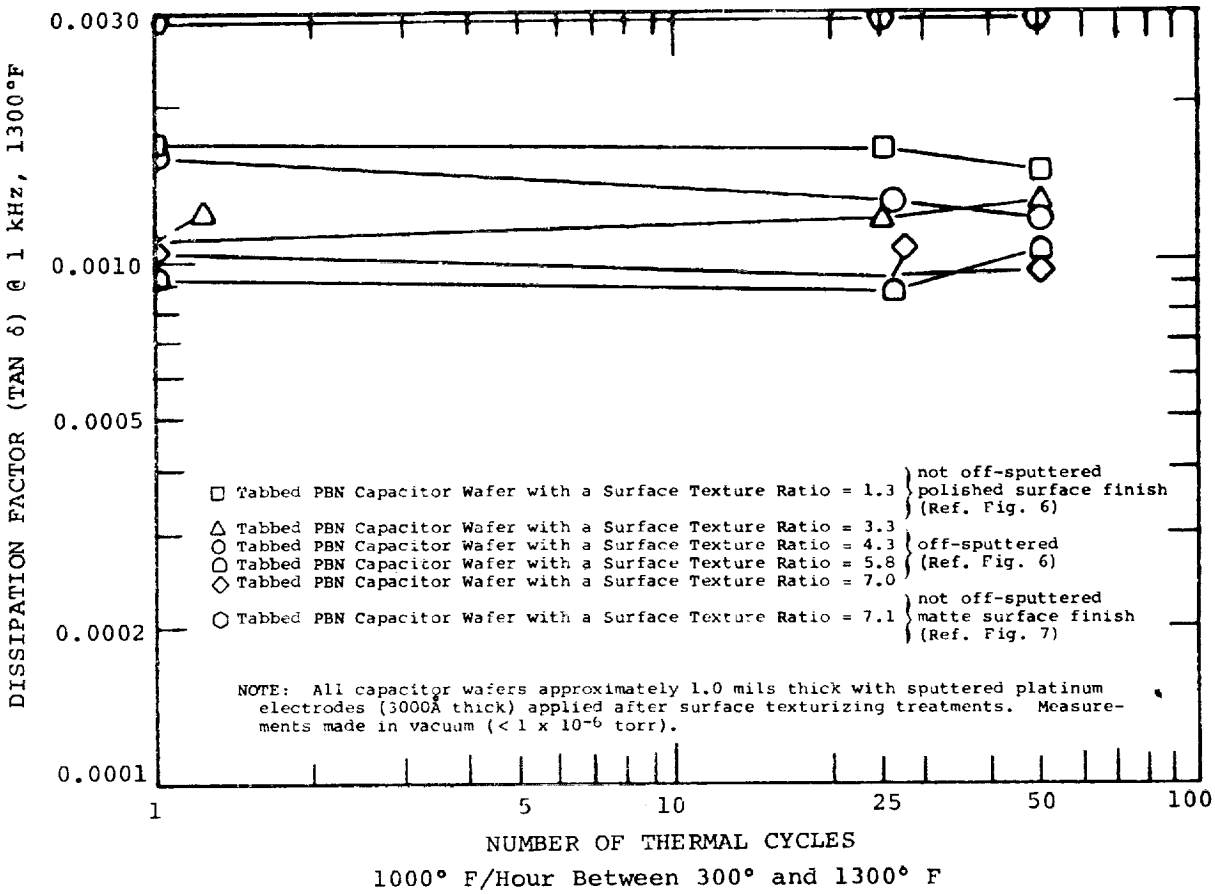


Figure 11. - Comparison of Dissipation Factors (Tan δ) Measured at 1300° F for Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes) Versus the Number of Thermal Cycles in Vacuum Between 300° and 1300° F. Tabbed PBN Capacitors Made From Wafers with Different Surface Texture Ratios (1.3 to 7.1). Rate of Temperature Change was 1000° F/Hour Between 300° and 1300° F.

Figure 12 clearly shows the decrease in dissipation factor measured at 72° F as the off-sputtering time was increased to produce surface texture ratios from 1.3 to 7.0. In addition, figure 13 shows that the ordering of dissipation factors with different surface texturizing treatments generally holds over the entire temperature range from 72° to 1100° F.

These results show that a two- to three-fold improvement (decrease) in dissipation factor is obtained when a wafer surface is off-sputtered.

Figure 14 compares the change in the ratio $\frac{\Delta C}{C_o} \times 100$ as a function of the number of thermal cycles for each of the six test capacitors. These data show that changes in capacitance are typically less than ±0.2 percent over the 50 cycle test. This variation is quite small and is attributed to experimental conditions.

Thermal Cycling Tests (Pyrolytic Boron Nitride Capacitors - Gold Electrodes). - Three tabbed pyrolytic boron nitride capacitors with gold electrodes were prepared for thermal cycling tests. Each capacitor wafer had a surface texture ratio of 7 produced by radio frequency off-sputtering matte wafer surfaces for 35 minutes. Pure gold (>99.9% Au) electrodes were sputtered onto the wafers at a rate of approximately 350 angstroms per minute to obtain electrodes in the 3000- to 4000-angstrom thickness range.

Room temperature electrical measurements showed that these capacitors had dissipation factors as low as those prepared with platinum electrodes on wafers with a surface texture ratio of 7. However, at 1100° F in vacuum the dissipation factor values of capacitors with gold electrodes were from 3 to 8 times higher than those with platinum electrodes.

The same cold-wall vacuum furnace used for thermal cycling and aging tests of capacitors with platinum electrodes was used to test the capacitors with gold electrodes. Figure 15 shows the test results after 64 thermal cycles between 300° and 1100° F in vacuum. Two of the capacitors with gold electrodes exhibited a significantly greater rate of increase in dissipation factor at 1100° F than observed for the capacitor with platinum electrodes. The third capacitor exhibited no significant changes in electrical properties.

Figure 16 shows the change in capacitance for the three capacitors with gold electrodes over 64 thermal cycles between 300° and 1100° F. It is evident that the same two capacitors that exhibited the greatest increase in dissipation factor (figure 15) also show the greatest change (decrease) in capacitance.

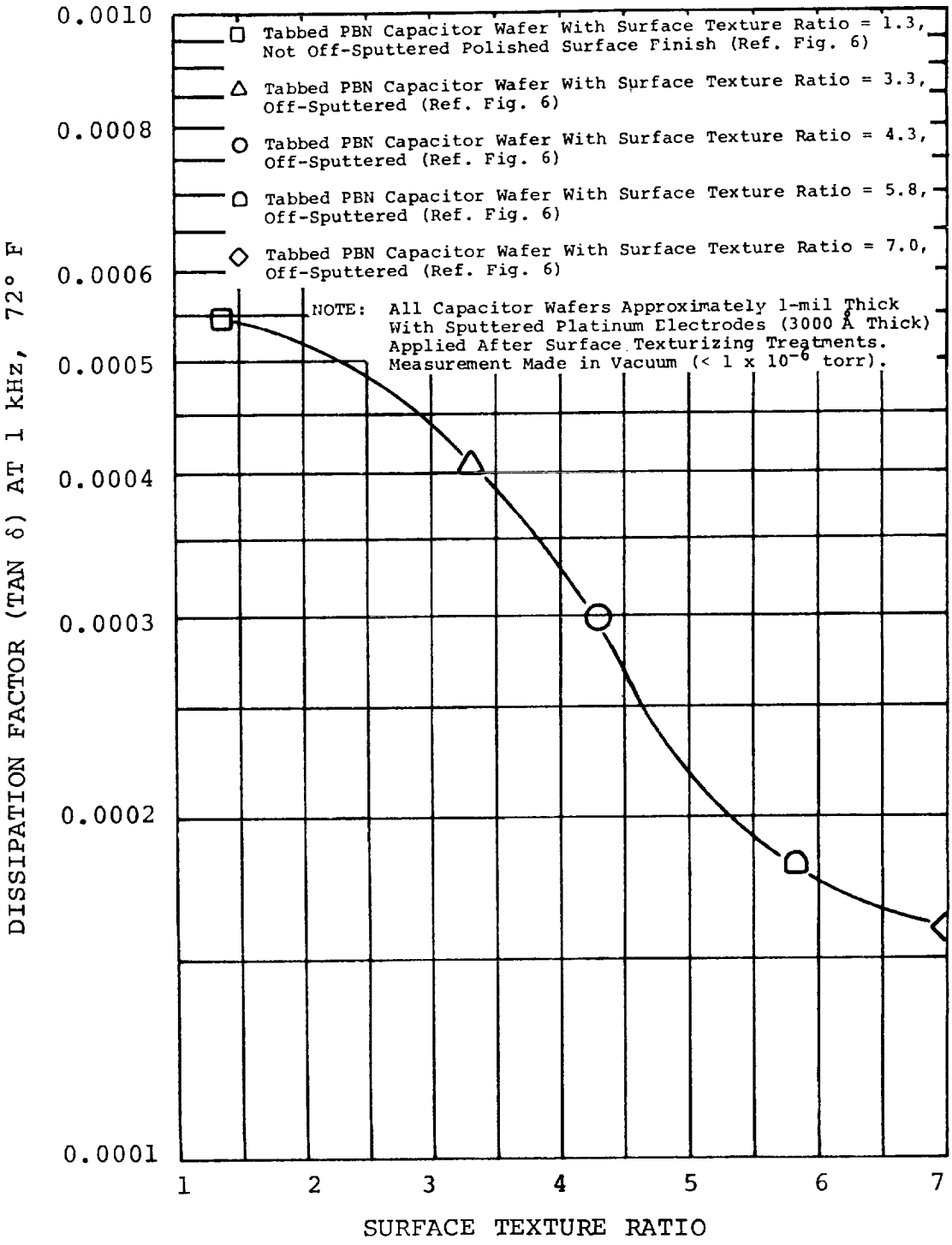


Figure 12. - Dissipation Factor (Tan δ) Measured at 72° F in Vacuum Versus Surface Texture Ratio for Tabbed Pyrolytic Boron Nitride Capacitors with Platinum Electrodes

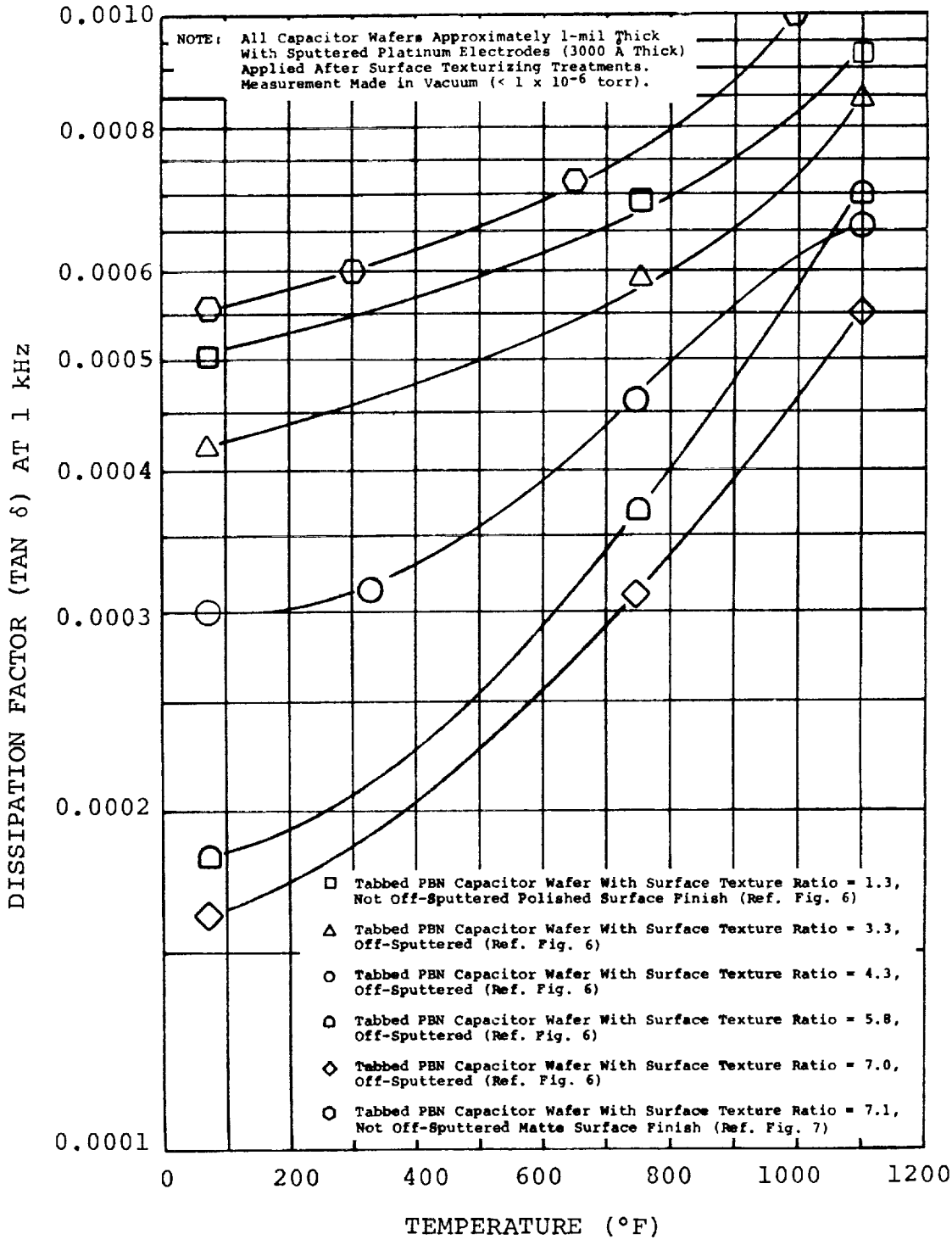


Figure 13. - Comparison of Dissipation Factor (Tan δ) Versus Temperature for Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes). Made from Wafers with Different Surface Texture Ratios Ranging from 1.3 to 7.1.

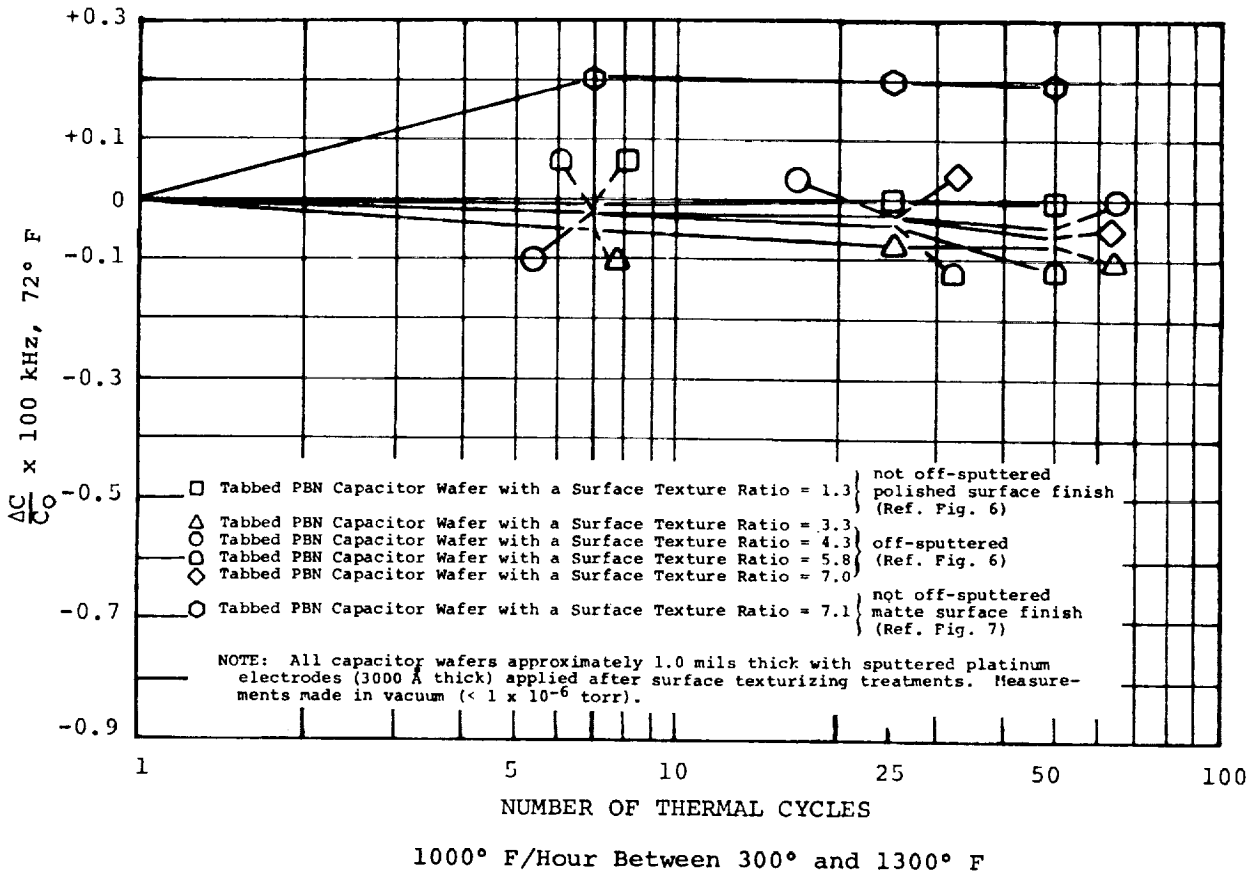


Figure 14. - Comparison of Ratio $\frac{\Delta C}{C_o}$ Measured at 72° F For Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes) Versus The Number of Thermal Cycles in Vacuum Between 300° and 1300° F. Tabbed PBN Capacitors Made From Wafers With Different Surface Texture Ratios (1.3 to 7.1). Rate of Temperature Change was 1000° F/Hour Between 300° and 1300° F.

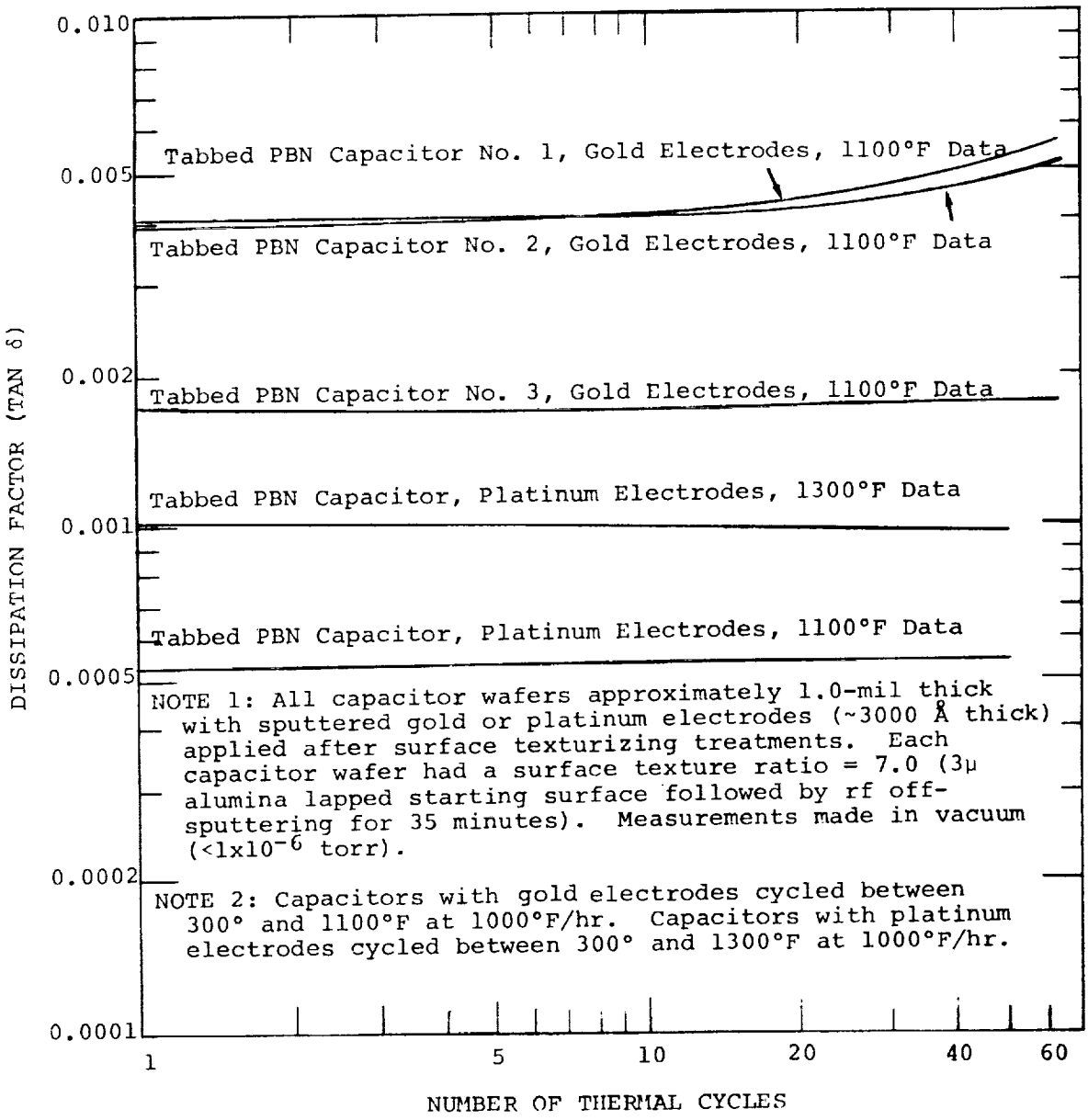


Figure 15. - Comparison of Dissipation Factors (Tan δ) for Three Tabbed Pyrolytic Boron Nitride Capacitors With Gold Electrodes and One Capacitor With Platinum Electrodes Versus the Number of Thermal Cycles in Vacuum Between 300° and 1100° F or 1300° F. All Capacitor Wafers Have a Constant Surface Texture Ratio of 7.0.

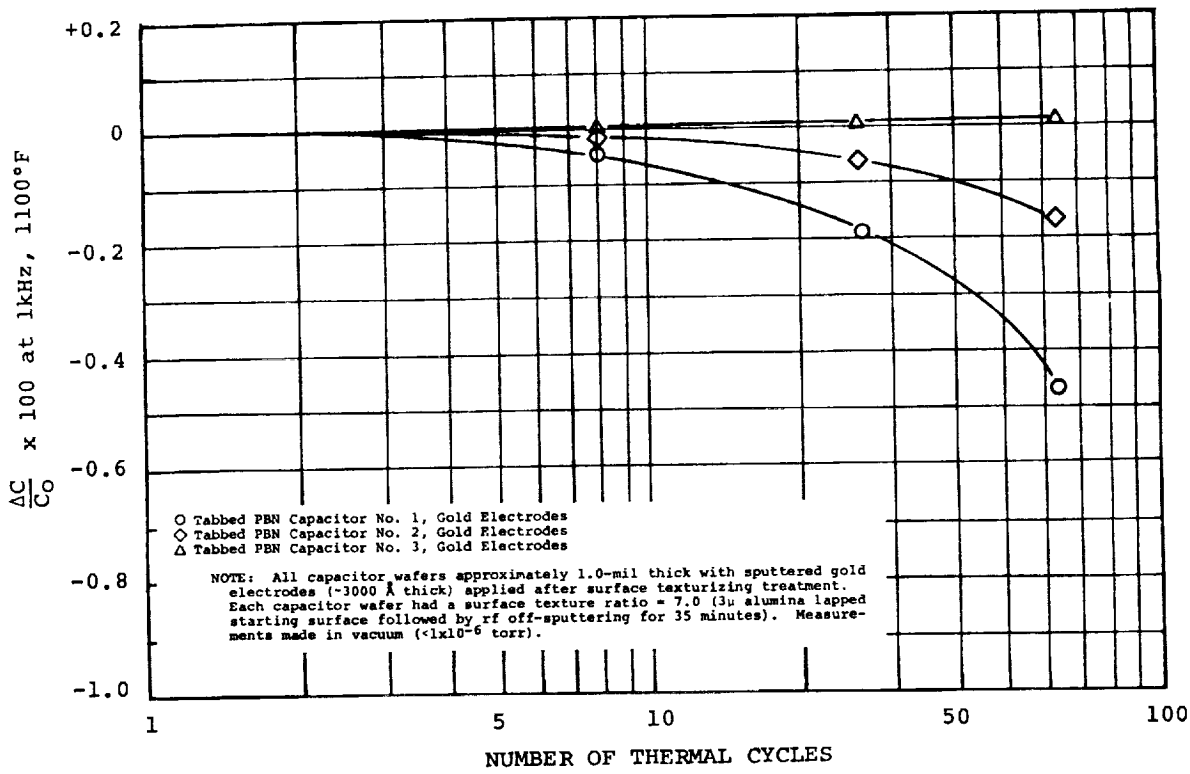


Figure 16. - Comparison of Ratio $\frac{\Delta C}{C_o}$ Measured at 1100° F for Three Tabbed Pyrolytic Boron Nitride Capacitors with Gold Electrodes Versus the Number of Thermal Cycles in Vacuum Between 300° and 1100° F. All Capacitor Wafers Have a Constant Surface Texture Ratio of 7.0.

Aging at 1100° F (Pyrolytic Boron Nitride Capacitors - Platinum Electrodes)

A series of thermal aging tests have been made to compare the electrical performance of pyrolytic boron nitride capacitors with different surface texture ratios (1.3 to 7.0). The same capacitor group that was tested and evaluated under thermal cycling conditions were aged in the cold-wall vacuum furnace for 50 hours at 1100° F. Periodic measurements of capacitance and dissipation factor at 1 and 10 kHz were made.

Figure 17 shows the effects of constant temperature aging on the dissipation factor measured at 1100° F. The capacitor with a surface texture ratio of 5.8 was the only capacitor that showed a significant increase in dissipation factor (0.0007 to 0.001 after

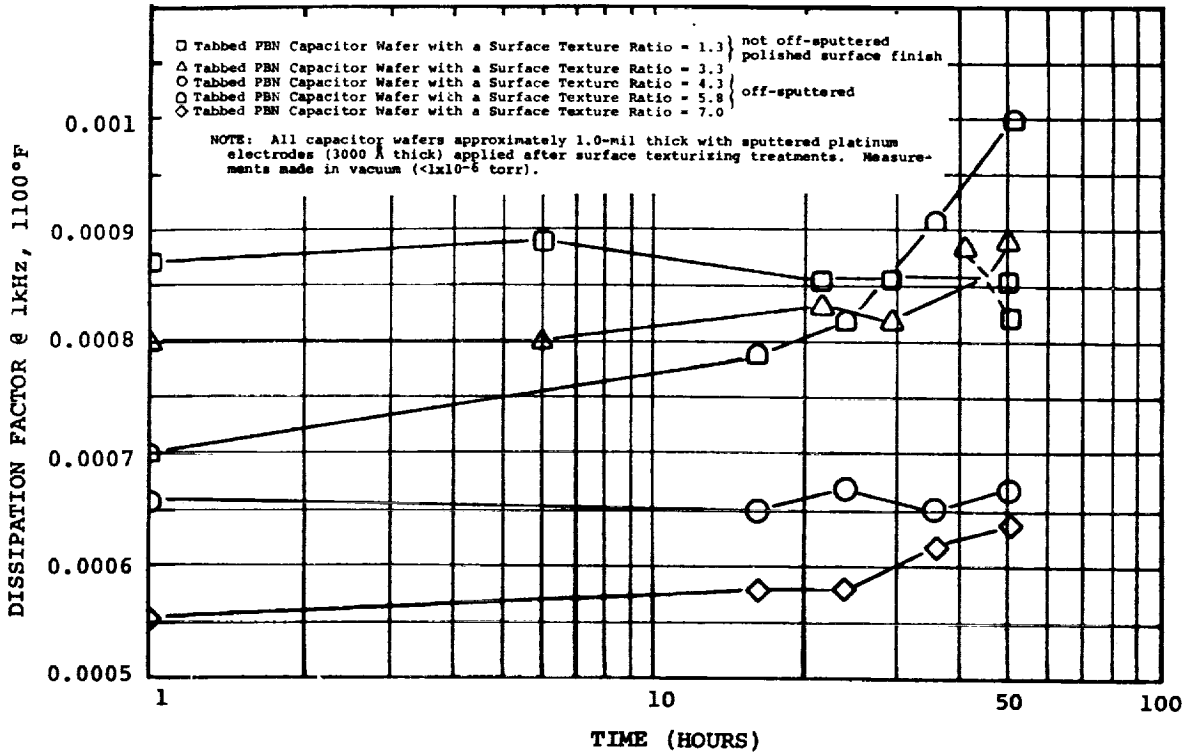


Figure 17. - Comparison of Dissipation Factors (Tan δ) Measured at 1100° F for Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes) Versus Aging Time at 1100° F in Vacuum (<1 x 10^{-6} torr). Tabbed PBN Capacitors Made From Wafers With Different Surface Texture Ratios (1.3 to 7.0)

50 hours at 1100° F). However, a dissipation factor of 0.001 at 1100° F was still substantially lower than those measured previously (ref. 1) for the pyrolytic boron nitride capacitors that had received no off-sputtering treatment. Figure 18 shows that the capacitance change after 50 hours of thermal aging was less than -0.15 percent for all five capacitors.

An important trend that is evident from figure 17 is the effect of off-sputtering on the absolute values of dissipation factor. The highest dissipation factors were obtained for capacitors that were not off-sputtered as observed previously during thermal cycling. Progressively lower dissipation factors were evident as the off-sputtering time was increased from 7 to 35 minutes. These data show that a surface texture ratio of 7 obtained by off-sputtering a matte wafer for 35 minutes produced a capacitor with the lowest dissipation factor before and after being subjected to thermal cycling and aging tests. A pyrolytic

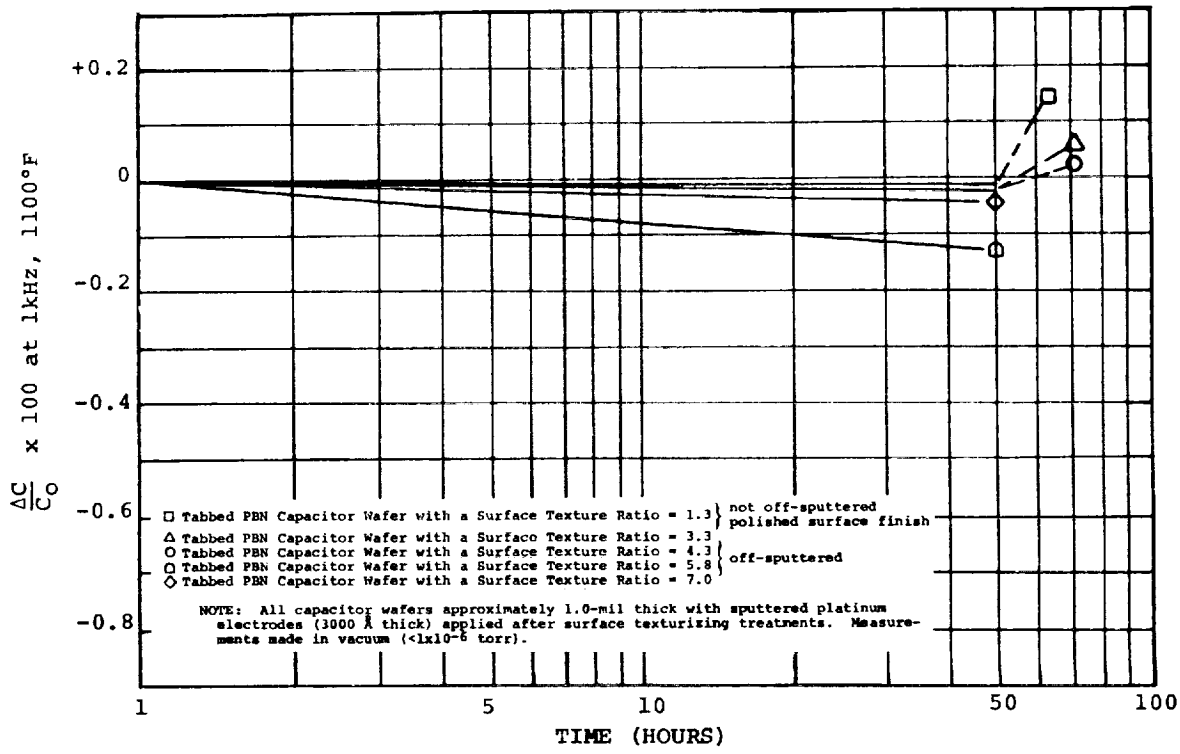


Figure 18. - Comparison of the Ratio $\frac{\Delta C}{C_o}$ Measured at 1100° F for Tabbed Pyrolytic Boron Nitride Capacitors (Platinum Electrodes) Versus Aging Time at 1100° F in Vacuum (<1 x 10^{-6} torr). Tabbed PBN Capacitors Made From Wafers with Different Surface Texture Ratios (1.3 to 7.1).

boron nitride capacitor with this surface texture is also easily fabricated because a polished starting surface is not required prior to off-sputtering. In general, a two- to three-fold improvement (decrease) in dissipation factor was obtained when a pyrolytic boron nitride wafer surface was off-sputtered. This effect is attributed to the removal of the mechanically disturbed surface layers during off-sputtering.

Stability Testing of Pyrolytic Boron Nitride Capacitors Fabricated With Texturized Dielectric Surfaces and Boron Nitride Diffusion Barrier

The objective of this test was to 1) fabricate a multi-layer capacitor with off-sputtered wafer surfaces and radio frequency sputtered boron nitride barrier layers on the surfaces of each individual capacitor electrode and, 2) perform a short term

(50-hour) aging test at 1100° F in vacuum to determine whether the barrier layers are effective in reducing the rate of change of capacitance with time and preventing interelectrode diffusion bonding. The results of this test are compared with the data from a 1120-hour aging test performed on a multi-layer capacitor without electrode barrier layer. (See ref. 1.)

Off-Sputtering (texturizing) of Capacitor Wafers. - A group of tabbed, one-mil-thick, pyrolytic boron nitride wafers were prepared for off-sputtering. The wafer surfaces were final-lapped with 3-micron alumina abrasive.[3] A particle size analysis[4] of this abrasive gave an average particle diameter of 2.17 microns. After lapping, the wafers were cleaned as detailed in Appendix B.

Figure 19 shows a photograph of the off-sputtering assembly. The radio frequency off-sputtering target consists of a 5-inch diameter by 1/16-inch-thick disk of pyrolytic boron nitride with a sputtered copper back-electrode. The wafers to be off-sputtered are placed on the surface of the pyrolytic boron nitride disk. Typical sputtering conditions are as follows:

(1)	Initial pump-down pressure	$<1 \times 10^{-6}$ torr
(2)	Sputtering pressure	13 to 18 microns
(3)	Sputtering gas	Research grade, ultrapure nitrogen (total impurities <1 ppm)
(4)	Magnet coil current	6 amperes
(5)	Radio frequency power	400 watts
(6)	Standing wave ratio	1.2 to 1.5
(7)	Sputtering time (each side of wafer)	80 minutes per side

Five tabbed wafers were off-sputtered at one time as outlined above. These wafers were weighed before and after off-sputtering. Based on the loss in weight, approximately 12,000 angstroms of surface material were removed from each wafer (6000 angstroms from each surface). This represents an average off-sputtering rate of 75 angstroms per minute.

[3]Type AC-003 Al_2O_3, Geoscience Co., New York, New York 10017
[4]Sub-Sieve Analyzer, Fisher Scientific Co., New York, New York 10017

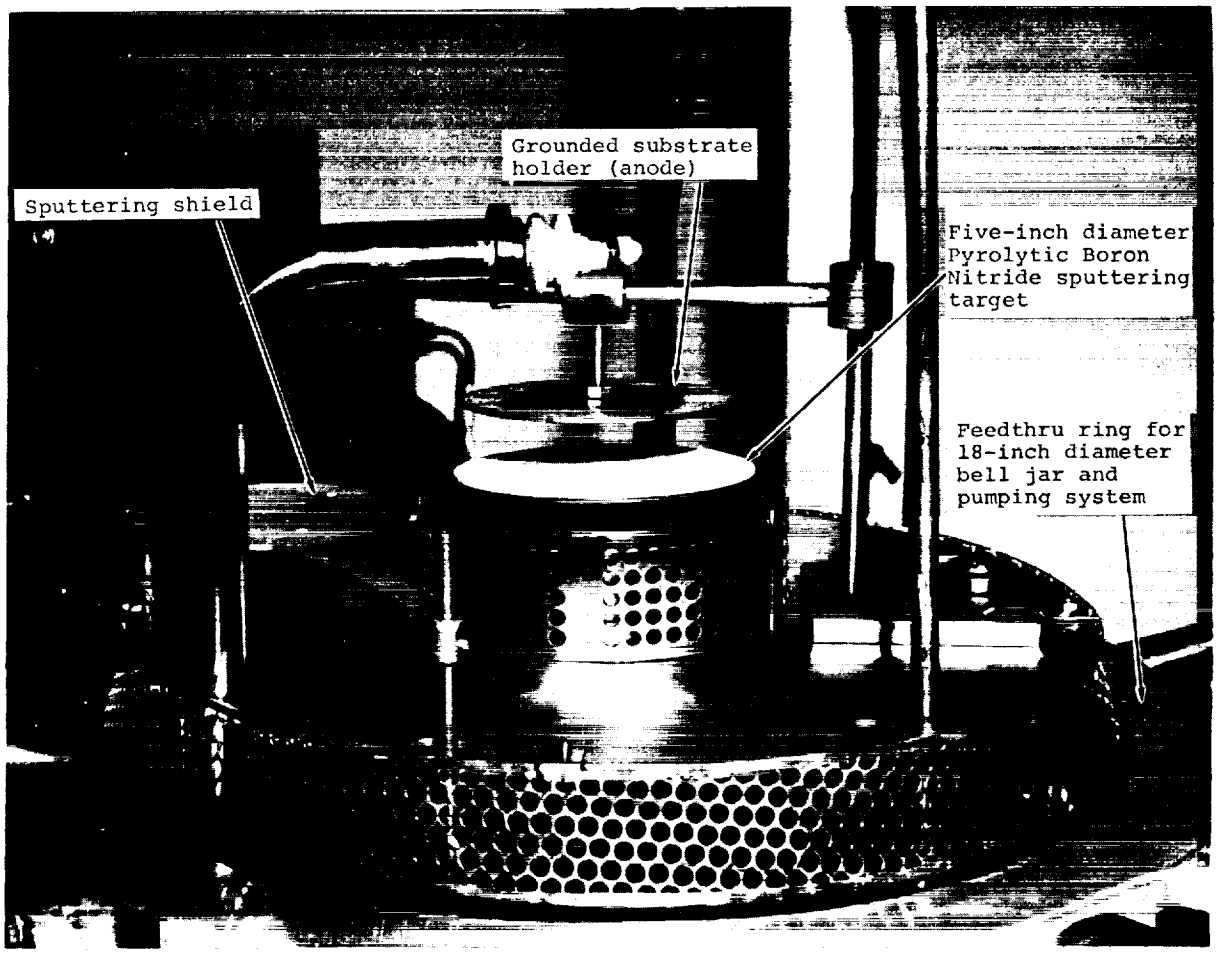


Figure 19. - Photograph of the Sputter-Up Radio Frequency Diode Configuration Used for Off-Sputtering of Pyrolytic Boron Nitride Wafers and Deposition of Boron Nitride Barrier Layers

Radio Frequency Diode Sputtering of Electrodes. - Three of the off-sputtered wafers were clamped between glass masks.[5] The masks were ultrasonically machined from 3 by 3 by 0.040-inch plates and are similar in design to those shown in figure 20. Figure 21 shows a schematic representation of the radio frequency diode sputtering assembly for sputter-down deposition of platinum electrodes. Platinum was deposited onto the exposed wafer surfaces under the conditions previously outlined in this report to obtain 3500-angstrom-thick electrodes.

Radio Frequency Sputtering of Boron Nitride Barrier Layers. - After depositing the electrodes, the capacitance and dissipation factor of each wafer were measured. These data are shown in table IV. The wafers were then clamped in another glass mask with different openings so that only the major circular portion of the platinum electrodes would be coated with a sputtered boron nitride film. Figure 22 shows the mask configuration. The wafer and mask assembly were fastened to the grounded top plate using the same sputter-up configuration shown in figure 19. Boron nitride was sputtered from the pyrolytic boron nitride target onto the exposed electrode surfaces. The sputtering conditions were as follows:

(1)	Initial pump-down pressure	$<1 \times 10^{-6}$ torr
(2)	Sputtering gas	Research grade ultrapure nitrogen (total impurities <1 ppm)
(3)	Sputtering pressure	11 to 12 microns
(4)	Preliminary off-sputtering onto shutter	30 minutes
(5)	Standing wave ratio	1.4 to 1.6
(6)	Radio frequency power	470 to 490 watts
(7)	Magnet coil current	6 amperes
(8)	Sputtering time	7 minutes per side

It is estimated that the film thickness was about 500 angstroms.

Results of Aging Test at 1100° F in Vacuum (50 Hours). - The completed wafers were stacked as illustrated in figure 23. A pyrolytic boron nitride clamping and test fixture similar to that

[5] 7059 glass, Corning Glass Works, Corning, New York

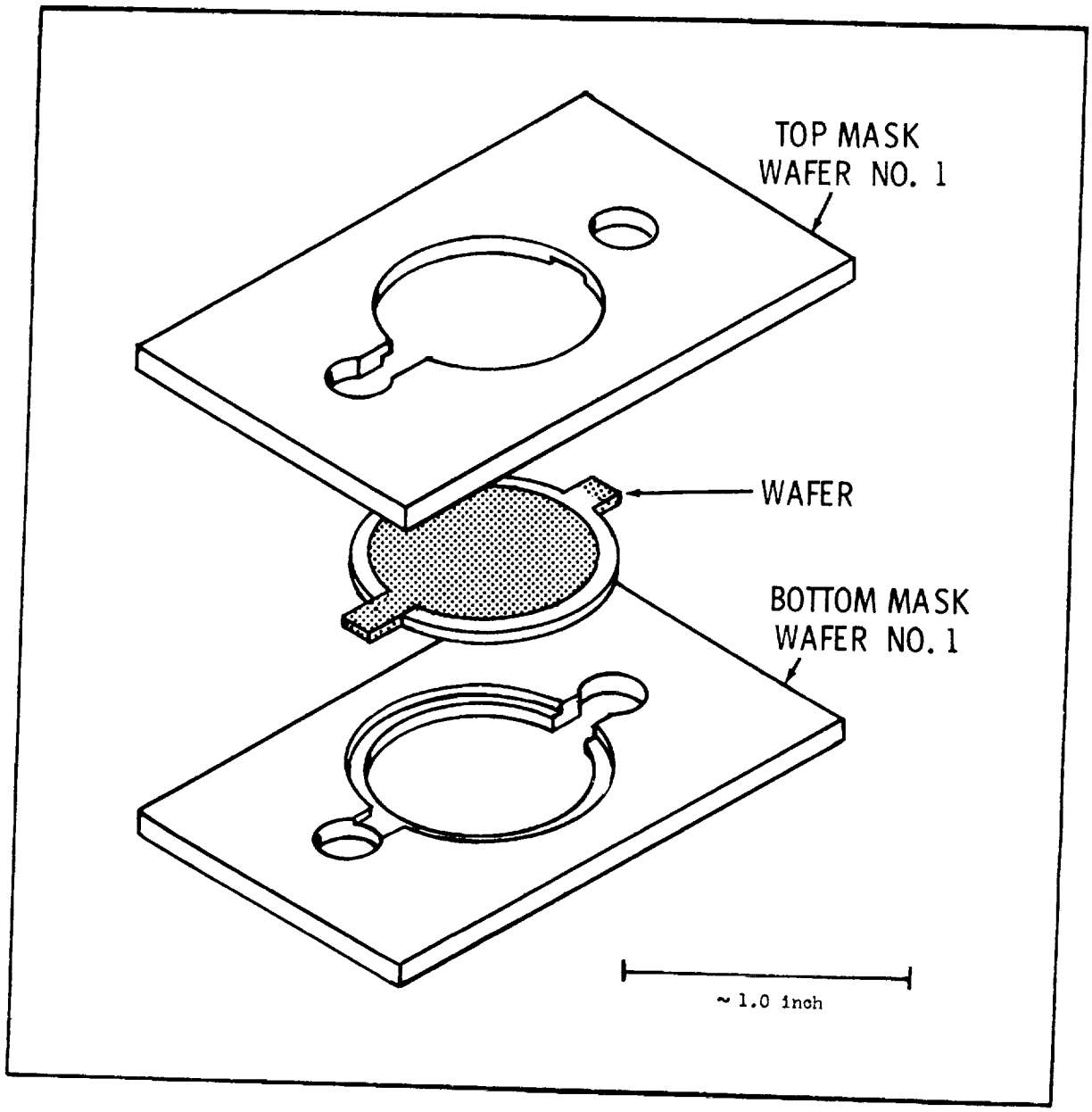


Figure 20. - A Typical Set of Masks Used for Sputtering Electrodes on Tabbed Wafers

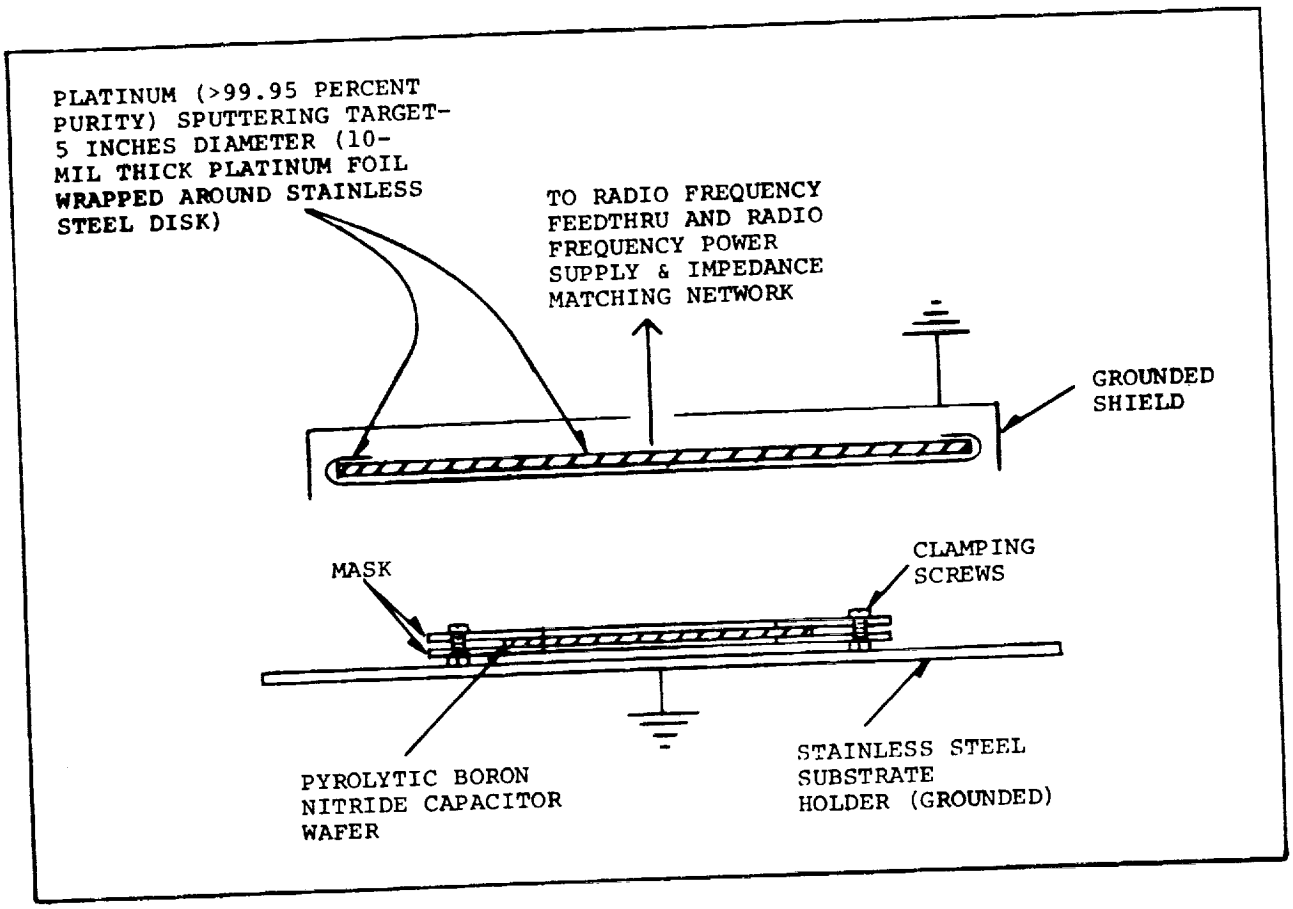


Figure 21. - Diagram of Sputter-Down Radio Frequency Diode Configuration Used for Deposition of Platinum Electrodes

shown in figure 24 was used to provide external lead contacts and align the wafers. A 200-gram aluminum oxide[6] cylinder was positioned on top of the boron nitride pressure plate during the aging test to provide a constant force of about one pound per square inch on the wafer surfaces. The test was conducted in a sputter-ion pumped, cold-wall vacuum furnace.

The furnace was evacuated to the 10^{-6} range and capacitance, dissipation factor, and direct current resistance (at 500 V dc) were measured at room temperature. Furnace power was applied, the temperature stabilized at approximately 1100° F and a series of electrical measurements were made at various time intervals after reaching 1100° F. An energizing voltage of 500 volts dc was

[6]Lucalox, General Electric Co., Lamp Glass Dept., Nela Park, Cleveland, Ohio

Table IV. - Electrical Measurements on Individual Pyrolytic Boron Nitride Wafers Used in the Diffusion Barrier Feasibility Test Capacitor (Before Deposition of Barrier Layers)

Tabbed Wafer Number	Platinum Electrode Thickness[a] (Å)	Wafer Surface Finish[b]	Calculated Wafer Thickness (inches)	Capacitance (pF)[c] and Dissipation Factor (tan δ) at Room Temperature			
				1 kHz		10 kHz	
				C = pF	tan δ	C = pF	tan δ
1	3500	Matte Off-Sputtered	0.001 (1.0 mils)	278.080	0.00056	277.879	0.00042
2	3500	Matte Off-Sputtered	0.00086 (0.86 mils)	332.021	0.00063	331.760	0.00044
3	3500	Matte Off-Sputtered	0.001 (1.0 mils)	272.020	0.00057	271.832	0.00039

(a) Estimated from sputtering time (20 minutes ≈ 3500 Å for radio frequency diode configuration, figure 21).

(b) Final lapping with 3-micron alumina abrasive - then off sputtered for 80 minutes on each side to remove approximately 12,000 Å of surface material radio frequency diode configuration, figure 21).

(c) Units of capacitance are in picofarads.

continuously applied to the capacitor for 50 hours. Table V shows these data as well as capacitance and dissipation factor measurements after 50 hours when the furnace had been cooled to room temperature. Also shown in table V, for comparison, is the data taken during the first 65-hour period for the five-wafer capacitor tested for 1120 hours previously (ref. 4).

Two important differences are apparent in comparing these data:

1) The rate of change of capacitance over similar time intervals at the same direct current energizing voltage is significantly less for the three-wafer capacitor with the diffusion barrier layers.

2) The dissipation factor values for the three-wafer capacitor are substantially lower at room temperature in vacuum before and after aging.

Somewhat higher dissipation factor values were recorded for the three wafer capacitor at 1100° F during the test. This is believed to be caused by deposition of a thin, slightly conductive surface coating from evaporation and condensation of metallic mat-

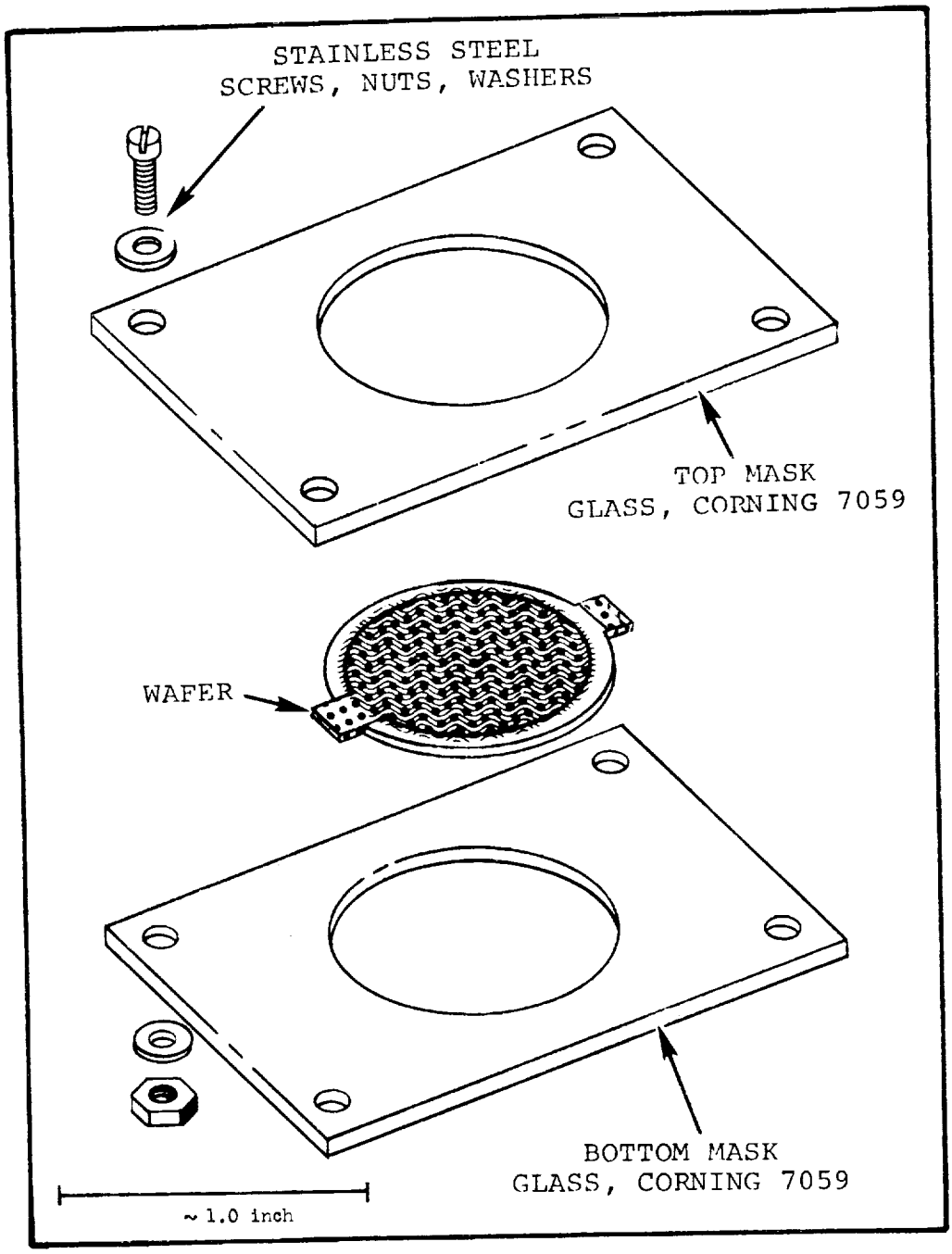


Figure 22. - A Typical Set of Masks Used for Radio Frequency Sputtering Boron Nitride Barrier Layers Onto Electrode Surfaces

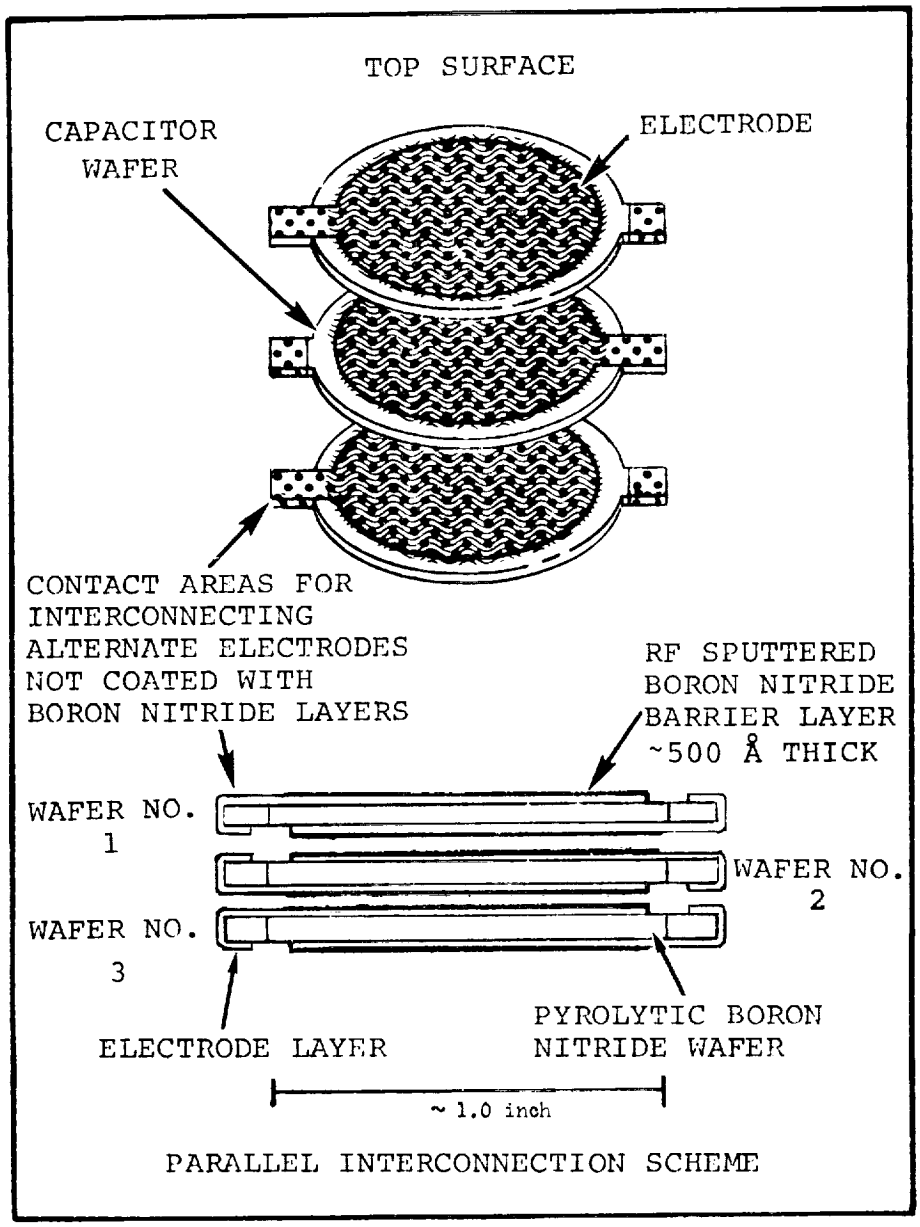


Figure 23. - A Representation of the Three-Wafer Tabbed Capacitor Showing Location of Radio Frequency Sputtered Boron Nitride Barrier Layers on Top and Bottom Electrode Surfaces of Each Capacitor Wafer

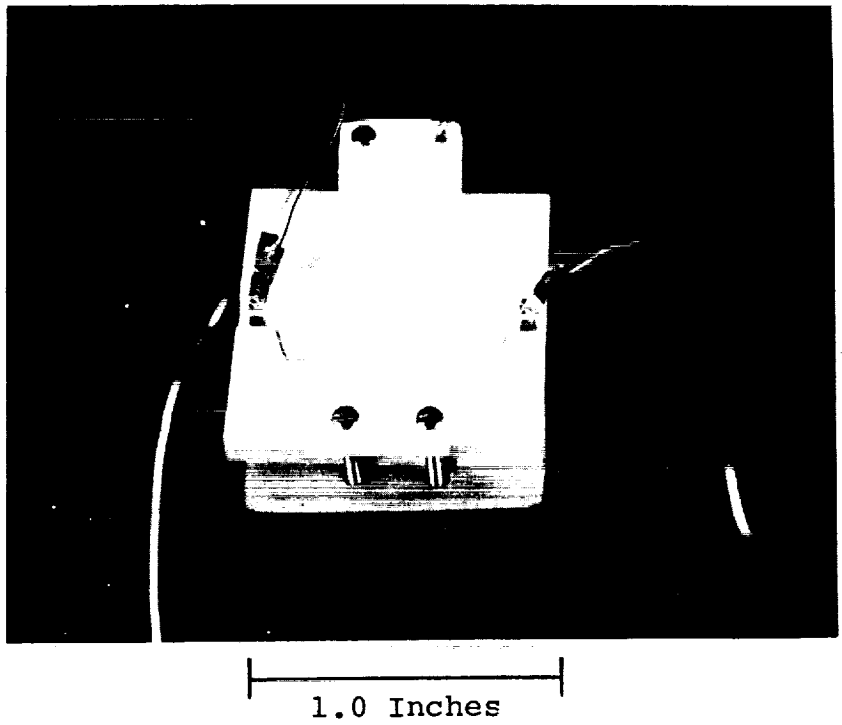


Figure 24. - Fixture for Making Electrical Measurements on Stacked Tabbed Capacitor Wafers

erials in the furnace onto the feedthrough insulators used to isolate the capacitor leads from ground. Inspection of these insulators after the test showed signs of surface darkening from deposited metallics.

Figure 25 compares the rate of change in capacitance $\left(\frac{\Delta C}{C_o} \times 100 \text{ @ } 1 \text{ kHz}\right)$ versus time for the three- and five-wafer capacitors. It is evident from these curves that the decrease in capacitance with time is reduced considerably by incorporating electrode barrier layers. It was theorized previously (ref. 5) that the decrease in capacitance with time was caused by a separation of the electrodes from the wafer surfaces. This separation was the result of interelectrode diffusion bonding between electrodes on adjacent wafers. A thin gap is produced by this process at a wafer/electrode interface. This introduces a high capacitance in series with the original capacitance and the net effect is a small reduction in the measured capacitance. The preliminary results reported above appear to confirm this hypothesis.

Further evidence of the effectiveness of the sputtered boron nitride barrier layers in increasing capacitance stability is shown

Table V. - Aging Test Data for Pyrolytic Boron Nitride Capacitors With or Without Sputtered Diffusion Barrier Layers

Elapsed Time With 500 VDC Applied	Furnace Temperature (°F)	Pressure (torr)	Capacitance (pF)[c] and Dissipation Factor (tan δ)				$\Delta C/C_o$ x 100 at 1 kHz	RC Product DC Resistance (MΩ) x Capacitance (μF)
			1 kHz		10 kHz			
			C = pF	tan δ	C = pF	tan δ		
DATA FOR THREE WAFER CAPACITOR WITH DIFFUSION BARRIER LAYERS								
Room Temp.	~80	$1x10^{-6}$	883.27	0.000192	883.08	0.00025	--	$8.8x10^{3}$
Start	1100	$9x10^{-8}$	861.73	0.0024	858.78	0.0018	--	--
21 hrs.	1090	$9x10^{-8}$	861.56	0.0037	858.02	0.0021	--	11.0
50 hrs.	1100	$8x10^{-8}$	861.46	0.0044	857.38	0.0024	-0.031%	9.0
--	~100	$7.9x10^{-8}$	875.01	0.000087	874.95	0.00015	--	$4.9x10^{4}$
DATA FOR FIVE WAFER CAPACITOR - NO DIFFUSION BARRIER LAYERS (NAS3-6465)								
Room Temp.	~80	$2.6x10^{-7}$	1422.85	0.00058	1421.75	0.00064	--	$2.4x10^{5}$
Start	1111	$4x10^{-7}$	1381.39	0.00231	1378.77	0.00119	--	14.7
65 hrs.	1113	$2x10^{-8}$	1366.84	0.00267	1363.30	0.00145	-1.05%	17.1
--	~80	$5.3x10^{-9}$	1363.69	0.00117	1361.67	0.00131	--	$1.06x10^{3}$

(a) Chromel/Alumel thermocouple located approximately 2 inches from test specimen.

(b) C_o = capacitance value at 1 kHz when 500 VDC is initially applied.
$\Delta C = C_o - C_T$; where C_T = capacitance value at 1 kHz after specified elapsed time with 500VDC applied continuously.

(c) Units of capacitance are in picofarads.

in figure 26. The capacitor wafer in the top photograph is one of the wafers from the five-wafer capacitor previously tested for 1120 hours. During the process of separating this wafer from an adjacent wafer, the electrode peeled from the wafer surface because it had bonded to the electrode on the adjacent wafer. By comparison, the bottom photograph shows one of the wafers in the three-wafer capacitor with a diffusion barrier layer. No disruption of the electrode can be seen. None of the wafers in the stack showed any indication of interelectrode diffusion except at the tab contact areas which had not been coated with a barrier layer.

Adherence of Platinum Electrodes to Texturized Pyrolytic Boron Nitride Wafers

Two methods were investigated for measuring the adherence of sputtered platinum electrodes applied to texturized pyrolytic boron

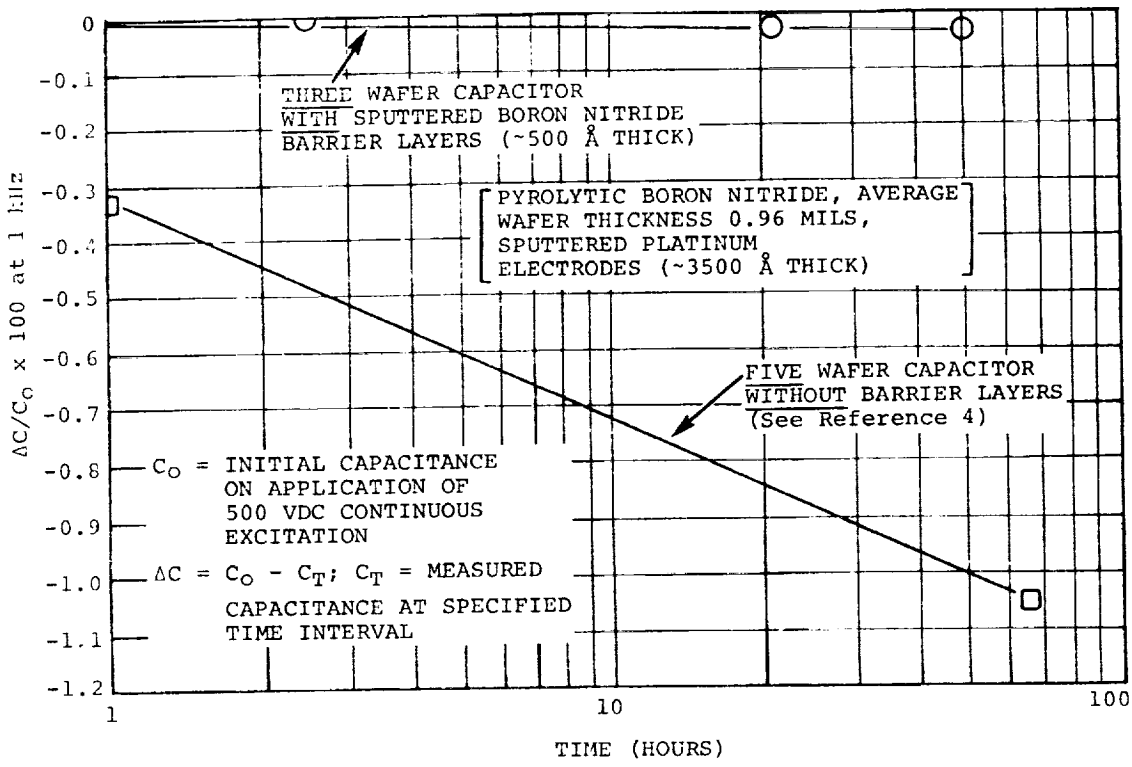


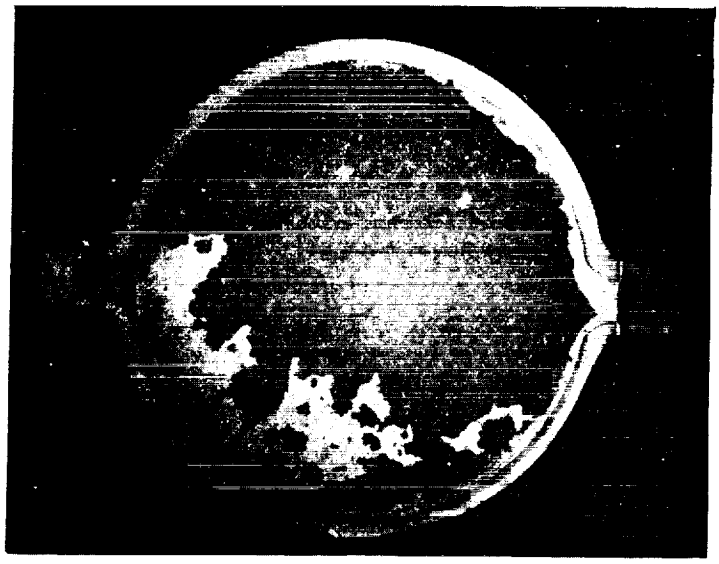
Figure 25. - Comparison of the Change in Capacitance Versus Time at 500 V dc/Mil in Vacuum at 1100° F for Pyrolytic Boron Nitride Multi-Layer Capacitors With and Without Sputtered Boron Nitride Barrier Layers

nitride wafers. The first method was based on a 180-degree peel test and the second method was a solder-pull test.

Two-mil-thick polyester terephthalate electrical tape[7] with a pressure-sensitive adhesive backing was selected for the peel test. Pyrolytic boron nitride substrates (matte surfaces) with sputtered platinum films were tested to determine the force required to peel the platinum from the substrate surface. It was found that, although forces[8] in excess of 400 grams per inch of tape width were required to peel the tape (pressure bond) from the surface of the platinum, the sputtered film remained attached to the pyrolytic boron nitride wafer. Tests were also made with the addition of a

[7]Mylar tape, Minnesota Mining and Manufacturing Co., St. Paul Minnesota

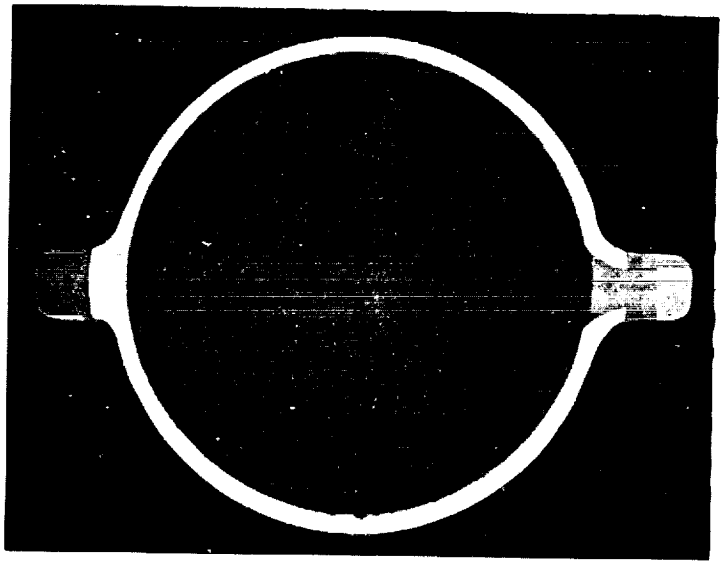
[8]Instron Machine equipped with a type A (10- to 500-gram range) load cell, Instron Engineering Corporation, Canton, Mass.



NO BARRIER LAYER ON PLATINUM ELECTRODE

Wafer separated from a five wafer multi-layer capacitor life tested in vacuum at 1100°F.

Note electrode removal from Pyrolytic Boron Nitride. After aging at 1100°F for 1120 hours (approx. 3.5 X actual size).



RADIO FREQUENCY SPUTTERED BORON NITRIDE DIFFUSION BARRIER (~ 500 Å thick) ON PLATINUM ELECTRODE

Wafer separated from a three wafer multi-layer capacitor life tested in vacuum at 1100°F.

Note effectiveness of barrier layer in maintaining electrode integrity after aging for 50 hours at 1100°F (approx. 3.5 X actual size).

Figure 26. - Photographs Showing Comparison of Tabbed Capacitor Wafers With and Without Electrode Barrier Layers After Aging as a Multi-Layer Capacitor Assembly at 1100° F in Vacuum

small amount of cyanoacrylate adhesive[9] applied to the tape--platinum interface. The forces required to peel the tape were higher, but failure occurred at the tape-pressure sensitive adhesive interface.

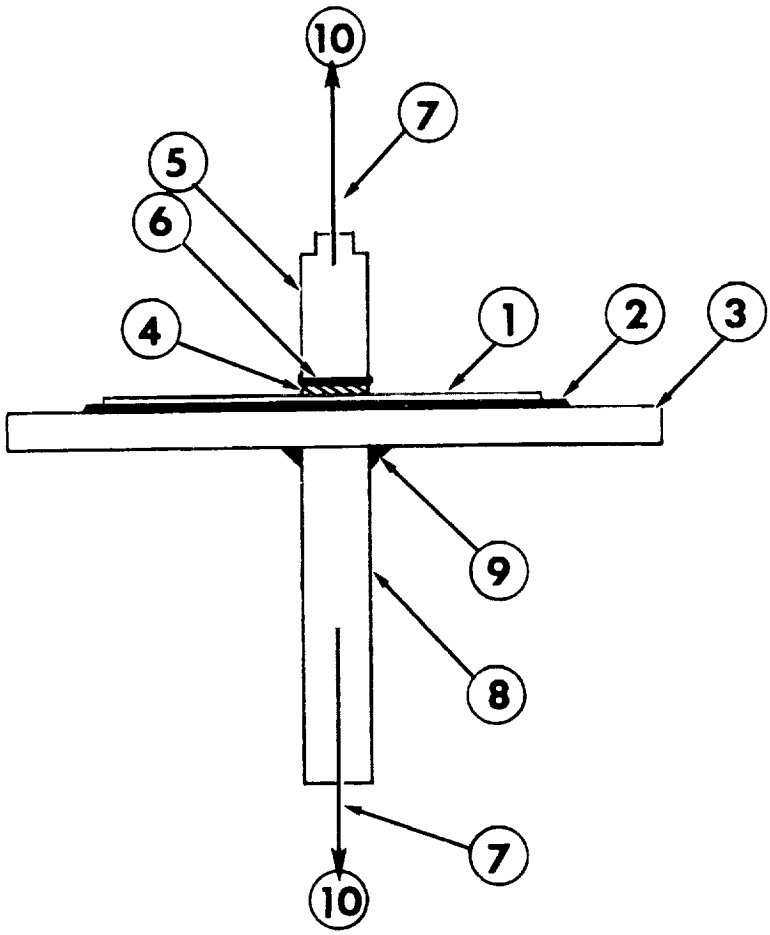
The solder-pull test method showed considerable promise over the tape-peel test.

The adherence of sputtered platinum films on texturized pyrolytic boron nitride wafers (approximately 1-mil thick) was measured using a butt-type solder bond. Figure 27 shows the test specimen configuration. A 0.150-inch-diameter brass rod with a Chromel wire (0.040-inch-diameter) clamped on one end was soft-soldered to a 0.150-inch diameter platinum film. The pyrolytic boron nitride wafer on which the platinum film had been deposited was epoxy-bonded to a small glass support plate. A 1/4-inch-diameter copper tube was then epoxy-bonded to the opposite side of the glass. The copper tube and Chromel wire were then carefully placed in the jaws of a tensile test machine.[10] The rate of travel was set at 0.01 inches per minute. Stress versus strain was recorded until failure, or until excessive yielding occurred.

Table VI shows the test results for adherence of platinum to texturized pyrolytic boron nitride wafers with four different surface treatments (surface texture ratios ranging from 5.8 to 7.2). The average values for three or four specimens from each group are approximately the same and range from 589 to 667 psi. Table 6 also indicates that many of the test specimens failed below the platinum-boron nitride interface, i.e. pieces or sections of boron nitride were dislodged from the bulk of the wafer and remained attached to the surface of the platinum electrode. This effect is shown in figure 28. The photograph on the left is the surface of the brass rod showing the nodular shaped sections of boron nitride that had been pulled from the boron nitride wafer and remain bonded to the platinum film. The photograph on the right in figure 28 is the surface of the boron nitride wafer showing the pulled out areas. The specimen shown in figure 28 had been off-sputtered for 60 minutes after a final lapping with 3-micron alumina abrasive. The surface texture ratio was 6.8 and the specimen failed at 777 psi (see table 6). In other instances a complete separation at the platinum-boron nitride interface was observed. Figure 29 is a photograph of a specimen that showed a complete separation at the platinum-boron nitride interface. This specimen failed at 902 psi and has a surface texture ratio of 7.2 (final lapping with a 3-micron alumina abrasive, not off-sputtered). These results show

[9]Eastman 910, Tennessee Eastman Co., Division of Eastman Kodak, Kingsport, Tennessee

[10]Instron Machine equipped with a type A (10- to 500-gram range) load cell, Instron Engineering Corporation, Canton, Mass.



(1) Pyrolytic Boron Nitride Wafer (~1-mil thick) Epoxy Bonded (2) to Glass Plate (3)

(4) Platinum Film 0.150-inch Diameter Sputtered onto Surface of Pyrolytic Boron Nitride Wafer

(5) Brass Cylinder Soldered (6) to Platinum Film

(7) Chromel Wire Clamped to Brass Cylinder

(8) Copper Tube Epoxy Bonded (9) to Glass Plate

(10) Direction of Applied Force by Means of an Instron Universal Tester (0.01-inch/minute)

Figure 27. - Test Specimen Configuration for Measuring Sputtered Platinum Film Adherence on Texturized Pyrolytic Boron Nitride Surfaces

Table VI. - Results of Platinum Film Adherence Tests on Texturized Pyrolytic Boron Nitride Wafers

PBN Surface Treatment and Nominal Surface Texture Ratio (STR)	Specimen Number	Stress at Failure[a] (psi)		Comments (Visual Inspection of Failed Specimens)
		Specimen Value	Average Value	
Final Lap With 3μ Alumina; Not Off-Sputtered STR 7.2	#1	442	667	Glass Plate Cracked
	#2	424		Complete Separation at PBN/Pt Interface
	#3	902		Complete Separation at PBN/Pt Interface[b]
Final Lap With 3μ Alumina; Off-Sputter 60 Minutes STR 6.8	#1	777	589	~ 70% of PBN Pulled From Surface[c]
	#2	753		Complete Separation at PBN/Pt Interface
	#3	323		Complete Separation at PBN/Pt Interface
	#4	815		~ 50% of PBN Pulled From Surface
Final Lap With 3μ Alumina; Off-Sputter 180 Minutes STR 7.2	#1	>1130	664	No Specimen Failure Due to Excessive Yielding
	#2	454		~ 30% of PBN Pulled From Surface
	#3	408		~ 40% of PBN Pulled From Surface
Polished; Off-Sputter 30 Minutes STR 5.8	#1	>1190	605	No Specimen Failure Due to Excessive Yielding
	#2	543		~ 50% of PBN Pulled From Surface
	#3	119		Complete Separation at PBN/Pt Interface
	#4	340		~ 30% of PBN Pulled From Surface

(a) Based on 0.150-Inch Diameter Platinum Electrode With Same Diameter Brass Cylinder Soldered to Platinum Surface (see Figure 27); Solder Used was 60/40 Lead/Tin; Force Applied Normal to Platinum Film Surface.

(b) See Figure 29.

(c) See Figure 28.

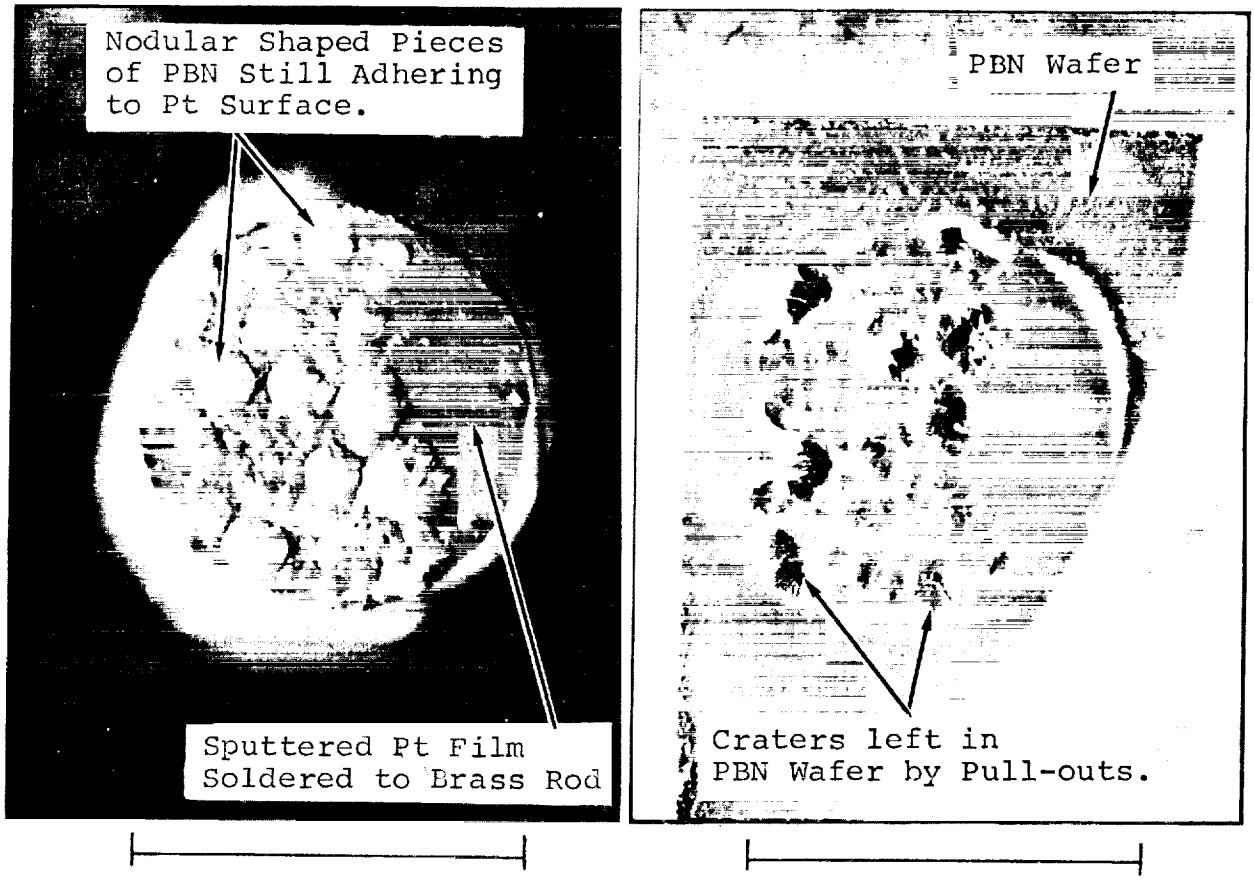


Figure 28. - Photomicrograph of a Platinum-Pyrolytic Boron Nitride (Pt-PBN) Interface (Surface Texture Ratio of 6.8) After Adherence Pull-Test Showing Nodular Shaped Pieces of Boron Nitride Pulled from the Test Wafer

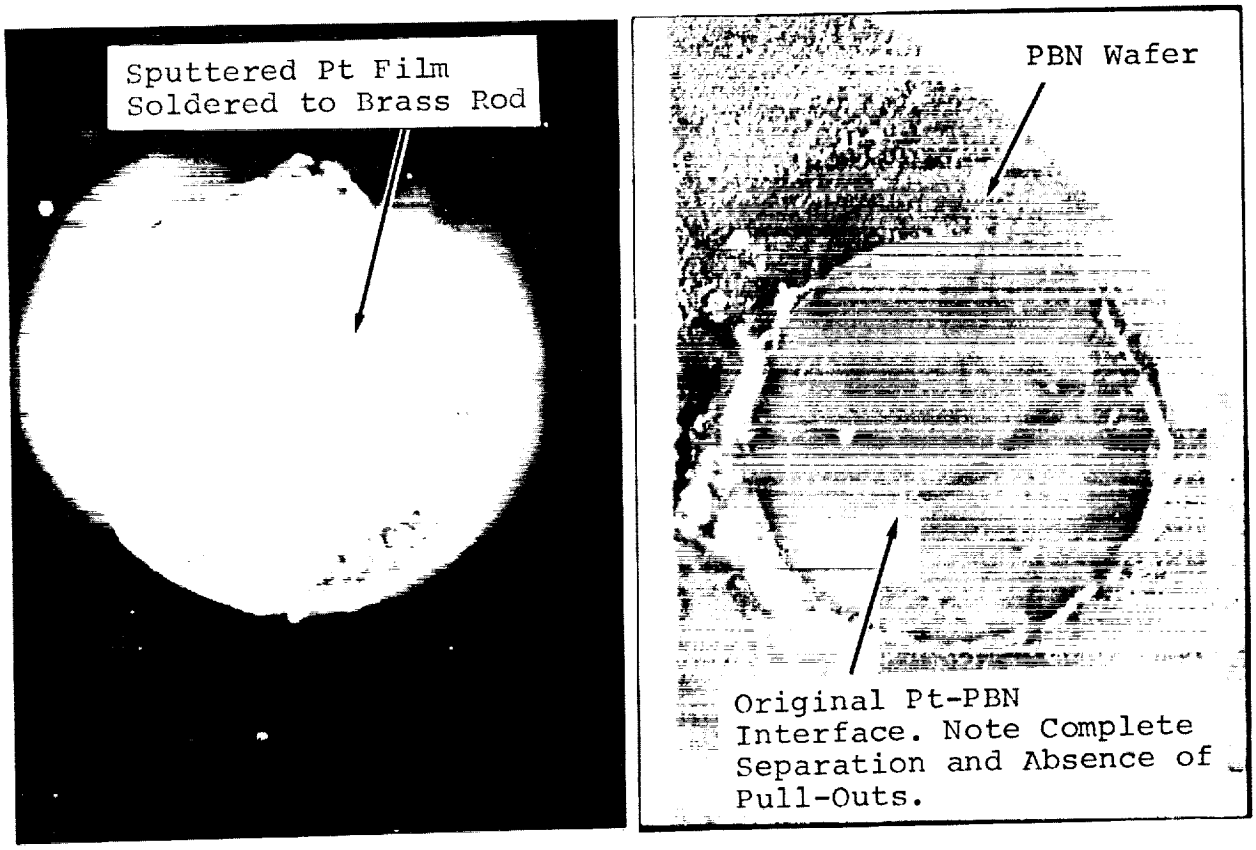


Figure 29. - Photomicrograph of a Platinum-Pyrolytic Boron Nitride (Pt-PBN) Interface (Surface Texture Ratio of 7.2) After Adherence Pull-Test Showing Complete Separation at the Interface

that the cohesive strength of the laminar-type pyrolytic boron nitride structure (ref. 6) is in some instances less than the strength of the platinum-boron nitride interface. In addition, these data are in general agreement with surface texture ratios which are in a fairly narrow range from 5.8 to 7.2. The methods used to obtain these surface texture ratios, however, were significantly different (mechanical lapping and/or radio frequency off-sputtering). Thus, the platinum film adherence is primarily dependent on surface roughness (true surface area). However, it is anticipated that due to cleanliness the RF sputtering will always be superior.

Previous data (figure 6) showed that the surface texture ratio or surface roughness of pyrolytic boron nitride increases rapidly with off-sputtering time and then levels off to a constant value. These data imply that the adherence of sputtered platinum on boron nitride will also reach a maximum and constant value. This appears to be the case based on the data shown in table 6.

Similar butt-type soldered bonds were made to platinum films sputtered onto polished pyrolytic boron nitride surfaces. However, the adherence of these films was so low that the specimens failed before they could be mounted in the tensile test machine. An attempt was made to obtain adherence data using larger area platinum films, but consistent data could not be obtained.

Effect of Compression on the Elevated Temperature Electrical Properties of Pyrolytic Boron Nitride Capacitors at 1100° F in Vacuum

The short time influence of pressure on dissipation factor (tan δ) and capacitance was measured in vacuum on a two-wafer capacitor assembly. Each capacitor wafer had sputtered boron nitride barrier layers (500 angstroms thick) deposited over sputtered platinum electrodes (3500 angstroms thick). The surfaces of the pyrolytic boron nitride wafers had been off-sputtered for 80 minutes (surface texture ratio = 7.0) before depositing platinum electrodes. These particular capacitor wafers were previously used in a three-wafer capacitor assembly that was thermally aged for 50 hours at 1100° F in vacuum (see table 4).

Pressure was applied to the two-wafer capacitor assembly at levels at 2, 5, and 15 psi. Arc cast tungsten rods were used as weights. The specified force per unit area was based on the total area of a standard tabbed wafer (0.475 square inches). The test was conducted in the same cold-wall vacuum furnace previously used for thermal cycling and aging tests (see figure 9).

After the capacitor assembly was loaded in the furnace, the appropriate number of tungsten rods were placed on top of the pyrolytic boron nitride tab contact and pressure plate to obtain a

lytic boron nitride tab contact and pressure plate to obtain a 2-psi load. The furnace was pumped-down to 1×10^{-6} torr and room temperature (70° F) electrical measurements were made. The furnace temperature was then increased to 750° and 1100° F.

Electrical measurements were made at these temperatures five minutes after a stable temperature level was achieved. After cooling to room temperature, electrical measurements were made before opening the chamber. Additional tungsten weights were added to obtain a 5-psi load and the temperature test cycle was repeated as outlined above. A similar procedure was followed for the 15-psi pressure loading.

Figures 30 and 31 show the effects of increasing compressive force on dissipation factor (tan δ) and capacitance at room temperature, 750° and 1100° F. The 1100° F data show a general decrease in dissipation factor and a small decrease in capacitance with increasing pressure. However, the room temperature and 750° F data show an initial increase in dissipation factor at 5 psi and no significant changes at 15 psi. The increase in dissipation factor at the lower temperature (70° and 750° F) at 5-psi pressure was influenced by the previous thermal history of the capacitor. The pyrolytic boron nitride holding fixture and/or insulation protecting the lead wires could have become coated with a semiconducting surface film from metallic furnace parts. This would result in increased leakage currents and the measured dissipation factor and capacitance would increase accordingly.

Following the short time pressure test series, the capacitor assembly with a 15-psi load was aged for 24 hours at 1100° F in a vacuum (10^{-6} torr). The capacitance and dissipation factor increased to 537.23 pF and 0.00427 at 1 kHz from their initial values of 536.90 pF and 0.00370 respectively. Examination of each capacitor wafer after these tests showed no visible evidence of interelectrode sticking or bonding except at the tab contacts which were not coated with boron nitride barrier films.

In general these data show that a stacked capacitor assembly can be operated at compressive loads up to 15 psi in vacuum. This test demonstrates that the aging and pressure effects are diminished by the boron nitride diffusion barrier layer.

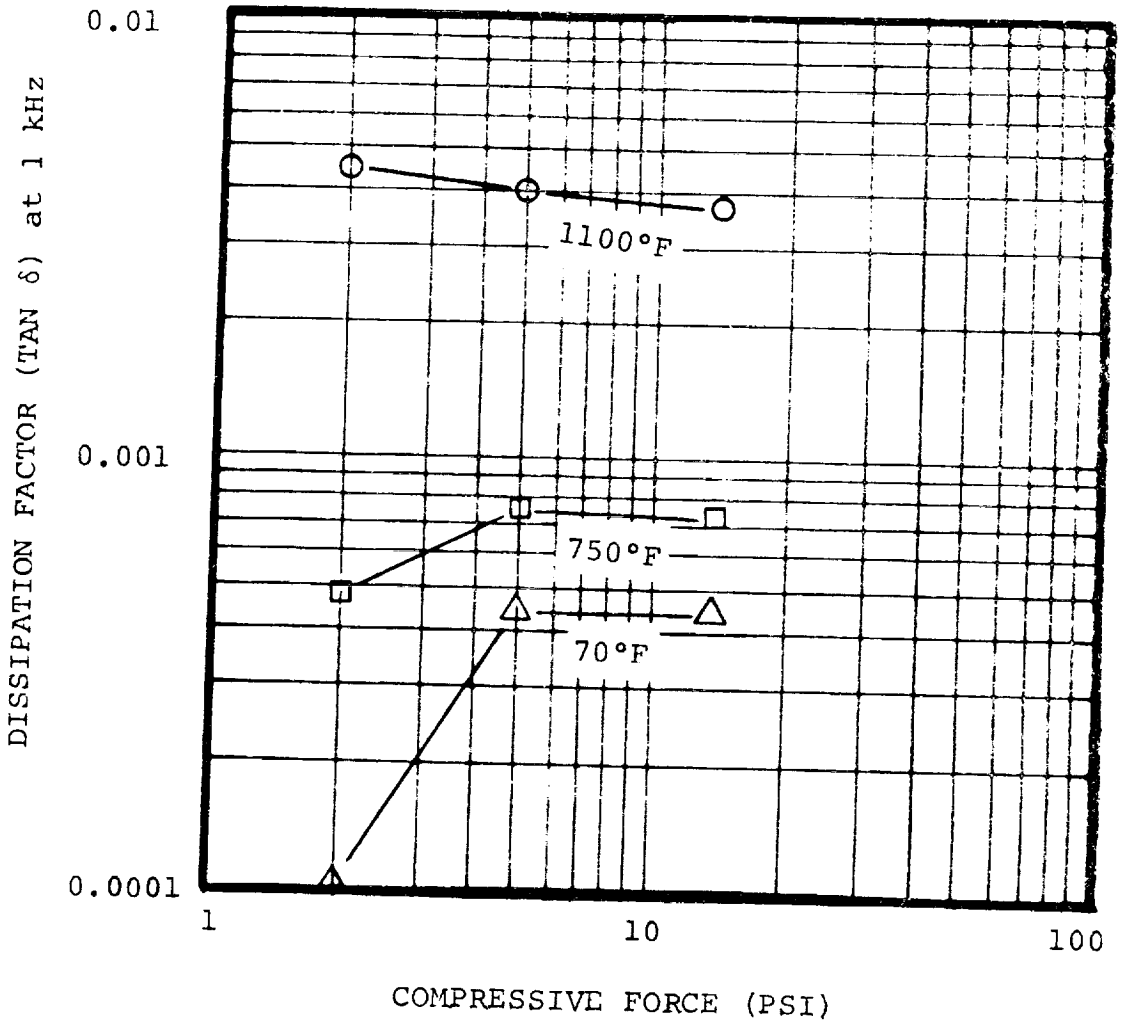


Figure 30. - Effect of Compression on Dissipation Factor at Room Temperature, 750°, and 1100° F in Vacuum (1 x 10^{-6} torr range). (See Text for History of Samples.)

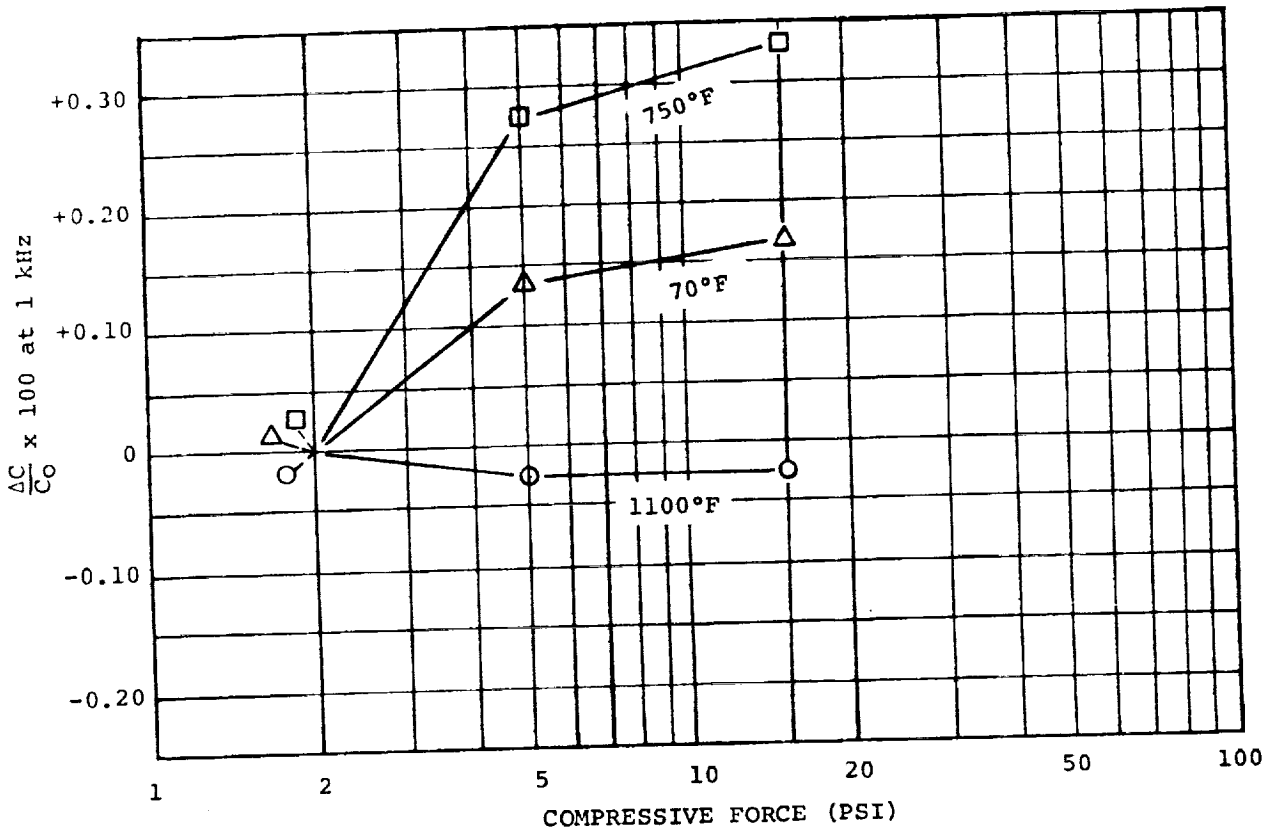


Figure 31. - Effect of Compression on Capacitance at Room Temperature, 750°, and 1100° F in Vacuum (1×10^{-6} torr range). (See Text for History of Samples)

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1. The effectiveness of a radio frequency-sputtered boron nitride barrier layer on the platinum electrodes of a pyrolytic boron nitride capacitor was demonstrated. Capacitors constructed in this manner were subjected to a 50-hour test at 1100° F and have been subjected to compressive loads up to 15 psi at temperatures to 1100° F. The capacitors performed with a slight change in capacitance (<0.05%). Examination of the capacitors subjected to these tests showed that the barrier layers prevented interelectrode diffusion bonding between electrodes on adjacent wafers.

2. Pyrolytic boron nitride capacitor losses (dissipation factor) at 1100° F can be reduced 3-fold by radio frequency off-sputtering or surface texturizing pyrolytic boron nitride wafer surfaces prior to the deposition of sputtered platinum electrodes. This effect is attributed to the removal of the mechanically disturbed surface layers produced during final lapping and subsequently removed during off-sputtering.

3. Surface texturizing of pyrolytic boron nitride capacitor wafers by mechanical and off-sputtering methods have shown a surface area ratio of 6 to 1 (actual to apparent). These results insure a wider range of surface textures for improving electrode adherence. Double layer capacitance measurements were used to obtain this information. It appears that the surface area of off-sputtered pyrolytic boron nitride wafers reach a constant value (with off-sputtering time) irrespective of the initial surface texture (smooth or mechanically roughened) of the wafer.

4. Adherence tests conducted on platinum electrodes which had been sputtered on to texturized pyrolytic boron nitride capacitor wafers showed bond strengths to 1000 psi. On the other hand, the bond strength of platinum to polished pyrolytic boron nitride wafers (earlier method of pyrolytic boron nitride capacitor preparation) was too low to determine.

5. Electrical characterization tests were performed on a number of single wafer capacitors fabricated from a new lot of pyrolytic boron nitride. The overall results indicate there are no intrinsic differences in the electrical properties of the new lot of pyrolytic boron nitride when compared to the

material used on an earlier program (NAS3-6465). The average direct current (dc) breakdown strength was 11,000 volts/mil at room temperature and 8770 volts/mil at 1100° F in vacuum.

6. Pyrolytic boron nitride capacitors with platinum electrodes subjected to 50-thermal cycles between 300° and 1300° F and then aged for 50 hours at 1100° F in vacuum showed no significant changes in electrical properties (capacitance and dissipation factor). Six capacitors were tested. Each capacitor had a different surface texture ratio ranging from 1.3 to 7.1. The capacitor with a surface texture ratio of 7 obtained by off-sputtering a matte wafer for 35 minutes had the lowest dissipation factor throughout the test series.

7. Three pyrolytic boron nitride capacitors with sputtered gold electrodes were subjected to 64 thermal cycles between 300° and 1100° F. Although each capacitor had a surface texture ratio of 7 obtained by off-sputtering a matte wafer for 35 minutes, the dissipation factor of two capacitors increased 40 and 26 percent. The third capacitor showed no appreciable change in dissipation factor. These results indicate that gold electrodes on pyrolytic boron nitride capacitors are less stable than platinum electrodes.

Recommendations

1. Pyrolytic boron nitride capacitor wafers that are mechanically lapped to final thickness should be pre-conditioned by radio frequency off-sputtering wafer surfaces before deposition of electrodes. At least 2800 angstroms of surface material should be removed.

2. To prevent interelectrode diffusion bonding between platinum electrodes on adjacent wafers in a stacked and compressed pyrolytic boron nitride capacitor assembly approximately 500 angstroms of radio frequency sputtered boron nitride should be deposited on the electrodes. Sputtered platinum electrodes approximately 3500 angstroms thick should be used rather than gold electrodes to obtain the best overall electrical performance at elevated temperatures in vacuum.

3. A hermetically packaged, stacked pyrolytic boron nitride capacitor assembly consisting of 10 or more wafers under compressive load (2 to 15 psi) should be tested under thermal aging and cycling conditions. Performance should be compared with that obtained for one and three wafer capacitors.

4. The feasibility of fabricating larger area (2 to 5 inches square) pyrolytic boron nitride capacitors should be deter-

mined. Mechanically lapped pyrolytic boron nitride wafers as well as "as deposited" boron nitride film should be evaluated.

5. Pyrolytic boron nitride capacitors should be tested under actual or simulated load conditions such as operation in a 3-phase full wave rectifier assembly.

SECTION IV

REFERENCES

1. Stapleton, R. E.: High-Temperature Capacitor Feasibility, NASA-CR-1213, December 1968.

2. Ibid., p. 36, and pp. 62-64.

3. McMullen, J. J., and Hackerman, N. : Capacitors of Solid Metal-Solution Interfaces, Journal of Electrochemical Society, 106, pp. 341-346, April 1959.

4. Stapleton, R. E. : , High-Temperature Capacitor Feasibility, NASA-CR-1213, December 1968, p. 92.

5. Ibid., p. 110.

6. Ibid., p. 12.

APPENDIX A

SLICING AND LAPPING PROCEDURE FOR PYROLYTIC BORON NITRIDE WAFERS

Slicing

The following is a description of the slicing process used to prepare pyrolytic boron nitride wafers about 0.011 inch thick.

1. One inch square blocks of pyrolytic boron nitride are wax bonded to a 1 x 1 x 2 inch steel gauge block with LOC Wax-10 (Geoscience Instruments Corporation).

2. Pyrolytic boron nitride wafers approximately 0.011 inch thick are sliced on a Micro-matic Precision Wafering Machine, Model WMSA-2221 (Micromesh Manufacturing Corporation).

    a. Cut-off wheel speed -- 5500 rpm
    b. Feed rate -- 0.6 inch/minute
    c. Cut-off wheel type 37C240-V8R-30, 0.015-inch thick x 5-inch O.D., Crystolon rubber bonded (Norton Co.)

Lapping

The following is a description of the lapping process used to reduce pyrolytic boron nitride wafers from as-sliced thickness to the 0.001 inch thickness range.

1. As-sliced wafers are wax bonded (LOC Wax-10) to a steel holder. A typical holder with two mounted wafers is shown in figure A-1. Metal shim strips are spot welded to the surface of the holder and act as stops to control the wafer thickness during lapping.

2. Figure A-2 shows the Mazur Lapping/Polishing Machine with a wafer-holding fixture about to be placed on the glass lapping plate. The holder and wafers are placed face down onto the lapping plate containing a slurry of abrasive (15 micron alumina) and water. The machine is run at its slowest speed setting and stock is removed from the wafers until they reach the height of the shim stop (0.006-inch).

3. The above process is repeated on the opposite side of the wafers after they are remounted on another steel holder with 0.004-inch shim stops.

Figure A-1. Steel Holder with Wax Bonded Pyrolytic Boron Nitride Wafer

4. The 0.004-inch thick wafers are removed from the holder. Final thickness reduction to about 0.0016-inch is done by lapping the wafer between one fixed and one floating glass plate. Figure A-3 shows a wafer positioned between the glass lapping plate on the Mazur Machine and a hand-held glass plate. The top plate is moved manually in a figure eight motion. The wafer is removed occassionally for thickness measurements until the desired thickness is attained.

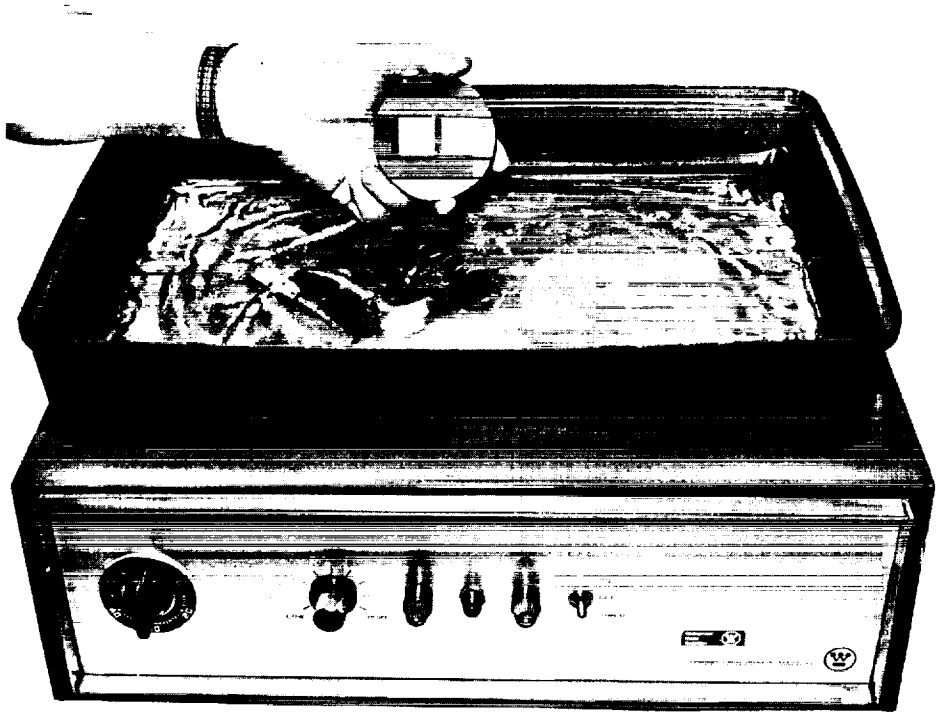


Figure A-2. Mazur Lapping/Polishing Machine Showing a Wafer Holding Fixture About to be Placed on Glass Lapping Plate

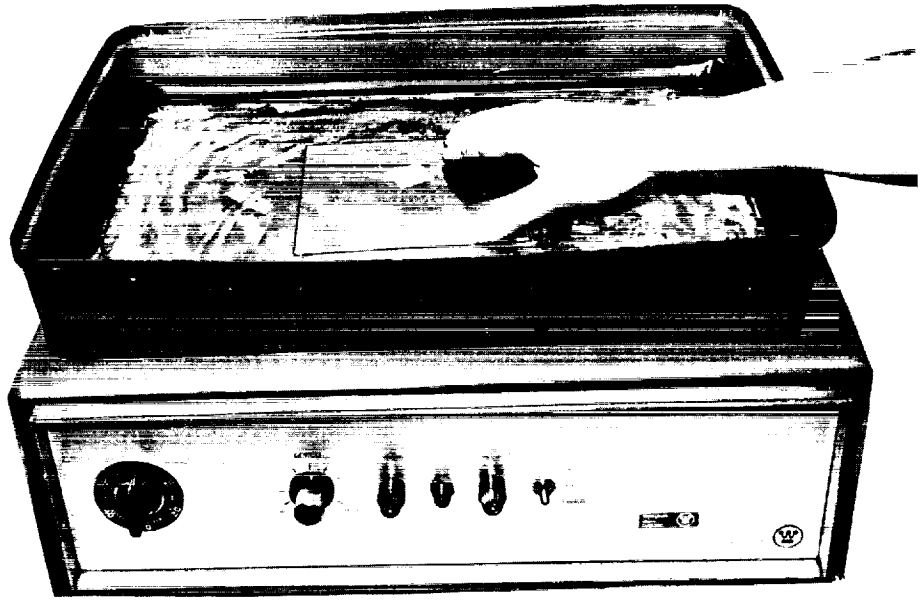


Figure A-3. Pyrolytic Boron Nitride Wafer Being Lapped to 1-Mil Thickness by Alternate Method (between two glass plates)

APPENDIX B

CLEANING PROCEDURE FOR PYROLYTIC BORON NITRIDE WAFERS

The following is a description of the techniques used to clean pyrolytic boron nitride wafers after final lapping.

1. Let wafers soak in de-ionized water until ready for further cleaning.
2. Pour off water and gently refill with de-ionized water; pour off water; repeat three times.
3. Very gently heat wafers in detergent (Alconox) solution (0.1 gram per 50 ml de-ionized water) for 10 minutes at about 195° F.
4. Pour off hot detergent solution; gently refill with de-ionized water and then let de-ionized water flow into beaker for 5 minutes so that wafers are gently agitated.
5. Pour off cold water and refill with hot (195° F) de-ionized water; soak for 3 minutes; pour off hot water; refill with hot water; repeat two times.
6. Pour off hot water and gently rinse in flowing de-ionized water for 5 minutes.
7. Pour off cold water and refill with 2-Propanol.
8. Pour off 2-Propanol and air dry wafers.
9. Boil wafers gently in aqua regia for 10 minutes.
10. Pour off hot aqua regia and gently rinse in flowing de-ionized water for 15 minutes.
11. Rinse four times in 2-Propanol.
12. Rinse three times in hot 2-Propanol.
13. Vapor degrease in 2-Propanol.
14. Oven dry at 300° F; cool in dessicator.

APPENDIX C

TEST APPARATUS AND METHODS FOR CAPACITANCE, DISSIPATION FACTOR AND D-C RESISTANCE MEASUREMENTS

The following is a description of the apparatus and methods used to make electrical measurements on pyrolytic boron nitride capacitors at room temperature and 1100° F.

1. All capacitance and dissipation factor (tan δ) measurements are made using a three terminal coaxial connection technique as described in section 3.7.3 and 4.2.2 of the General Radio Operation Instruction Manual for the type 1620-A Capacitance Measuring Assembly (Form 1615-A-0100-C, 1D887, April 1966). The assembly consists of a type 1615-A capacitance bridge with six digit capacitance readout and four digit dissipation factor readout, a type 1311-A Audio Oscillator, and a type 1232-A Tuned Amplifier and Null Detector.

2. Elevated temperature measurements in vacuum are made in a small resistance heated furnace insulated with tantalum radiation shields. Figure C-1 shows a cutaway view of the test furnace which is designed to be used within the same glass bell jar system used for sputtering electrodes. The capacitor is placed on top of a columbium disk as shown in figure C-1. A d-c power supply is used to energize the furnace winding.

3. D-C resistance measurements are made with a Keithly Model 610B Electrometer and a Keithly Model 240 Regulated D-C Power Supply. The d-c resistance of the test furnace insulators (less test specimen) was measured at 1000 Vd-c in vacuum (3×10^{-7} torr) up to 1100° F and found to be in the range 10^{17} to 10^{14} ohms from 72° to 1100° F.

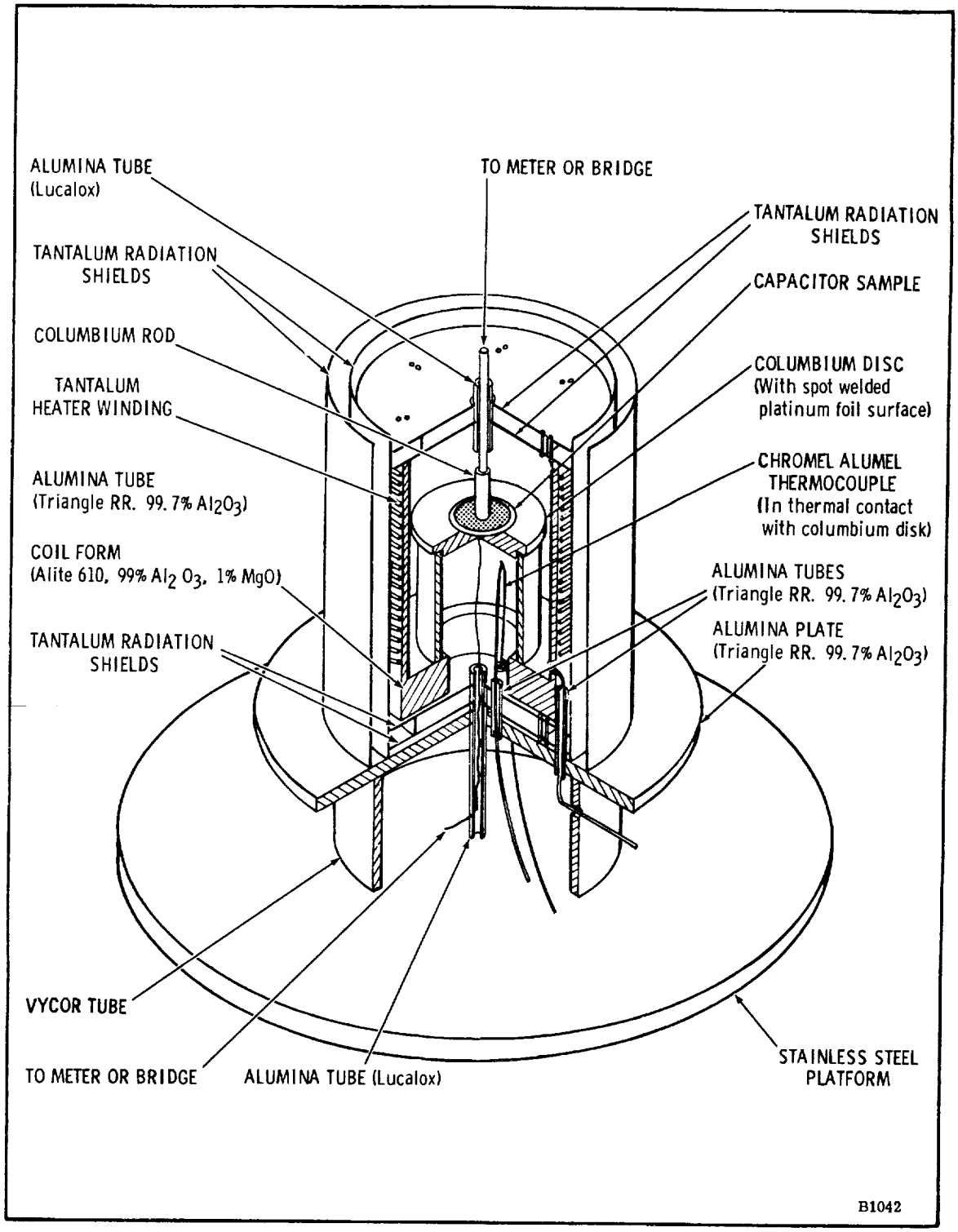


Figure C-1. Cut Away View of 1100° F Vacuum Furnace Constructed For Electrical Testing of Single Layer Capacitors