DEVELOPMENT OF AN 1100°F CAPACITOR*

Robert E. Stapleton

Panelvision Corporation

265 Kappa Drive

Pittsburgh, PA 15238

SUMMARY

The feasibility of developing a high temperature capacitor for 1100°F operation which is as small and light as conventional capacitors for normal operating temperatures is discussed in this paper.

Pyrolyic boron nitride (PBN) was selected for the dielectric after evaluating three other candidate materials at temperatures up to 1100°F. PBN capacitors were made by slicing and lapping material from thick blocks and then sputtering thin film electrodes. These capacitors had breakdown strengths of 7,000 volts per mil and a dissipation factor of less than 0.001 at 1100°F.

Additional processing improvements were made after testing a multi-layer or stacked PBN capacitor for 1,000 hours at 1100°F. Sputter etching the wafers before depositing electrodes resulted in a 2-3 fold reduction in dissipation factor. A sputtered boron nitride film applied to the outer electrode surfaces produced a more stable capacitor. This data will be presented together with a design for a 0.1 µF capacitor and a summary of PBN wafer fabrication costs.

INTRODUCTION

Capacitors were one of the electrical components that limited the operating temperatures in the advanced electric power systems being developed for spacecraft in the 1960s. As electric power requirements in spacecraft increase, the amount of power lost as heat also increases. This heat must be removed to keep temperatures from building up beyond the operating limits of the electrical components. Specially designed mica capacitors were available for 750°F operation but these devices were larger and heavier than standard units. In order to build a higher temperature-lightweight capacitor, a better dielectric was needed.

MATERIAL SELECTION

At least ten different dielectric materials were considered initially as candidates for a high temperature capacitor. From published data, four likely materials were selected for test: single crystal Al_2O_3, polycrystalline Al_2O_3, hot pressed BeO and pyrolytic boron nitride (Pyrolytic boron nitride, formed by a chemical deposition process at 3600°F, is a denser and purer material than compressed and sintered boron nitride). Wafers were sliced from blocks or pieces of the candidate materials and then lapped and polished. Since capacitance varies inversely as the thickness of the dielectric material, the wafers were made as thin as practicable. The thinnest wafers were produced from pyrolytic boron nitride (PBN). This material is soft (Moh's scale-2) and less brittle. It was found that PBN could be lapped into flexible, pin-hole free wafers as thin as 0.0004 inches from thick blocks of starting material (1).

After careful cleaning, thin film electrodes of platinum - 20% rhodium were applied by DC triode sputtering. Glass masks were used for pattern definition. A small test furnace was built to fit inside an 18-inch glass bell jar that was pumped to the test pressure of 1-4 x 10-7 Torr with a liquid nitrogen trapped diffusion pump. Electrical tests of the single wafer capacitors in vacuum at temperatures up to 1100°F showed that pyrolyic boron nitride was by far the best material. The dissipation factor of PBN capacitors was less than 0.001, 10 to 100 times better than that of capacitors made from the other candidate materials as shown in Figure 1.

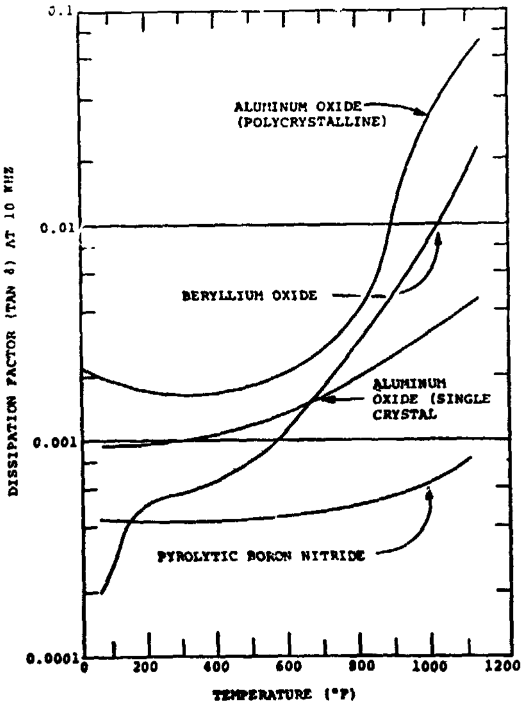

Figure 1. **Comparison of Dissipation Factor Versus Temperature for Candidate Purity Materials.**

Figure 2 shows that the change in capacitance from room temperature to 1100°F was minus 1.7 percent compared with plus 10 percent for single crystal Al_2O_3. The measured DC breakdown voltage was 7,000 volts per mil for a 0.001 inch PBN capacitor at 1100°F, compared to 1800 volts per mil for the closest competing material (single crystal Al_2O_3).

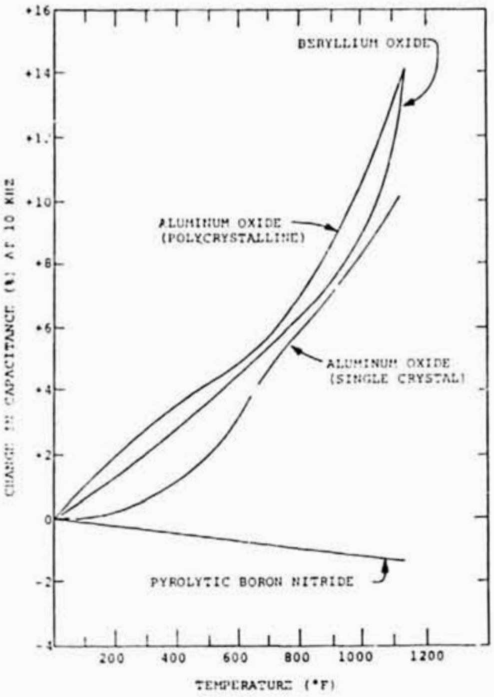

Figure 2. Change in Capacitance From Room Temperature to 1100°F for Candidate Materials.

PYROLYTIC BORON NITRIDE CAPACITORS

To obtain higher capacitance units, individual PBN capacitor wafers were shaped, electroded with sputtered platinum and stacked. Actual capacitor wafers (rectangular and round with tabs) are shown in Figure 3.

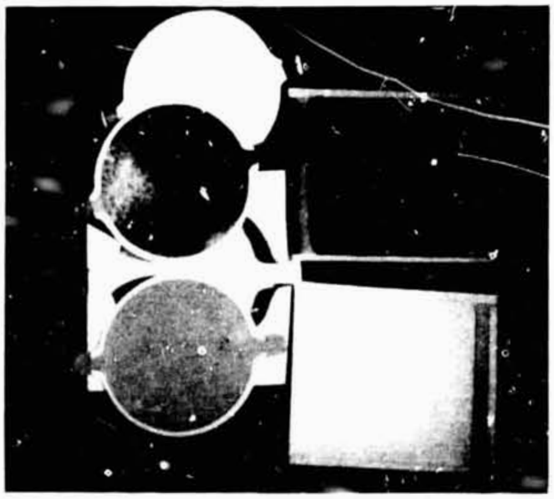

Figure 3. Photograph of Rectangular PBN Capacitor Wafers and tabbed 0.750-inch diameter wafers.

The platinum sputtering targets were positioned on opposite sides of a wafer. The wafer was clamped between two glass masks as shown in Figure 4 so that both surfaces of

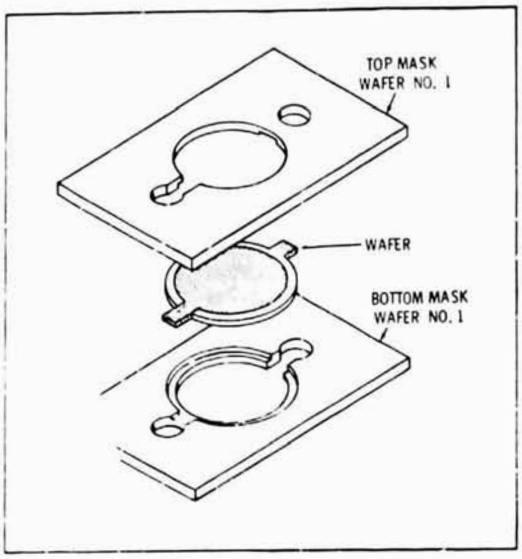

Figure 4. Glass masks used for sputtering electrodes on tabbed wafers.

the wafer were coated at the same time including the conducting path around each tab. By properly orienting the tabbed wafers, alternate electrodes are connected together as shown in Figure 5. The total measured capacitance of each stack is then the sum of the capacitances of all wafers.

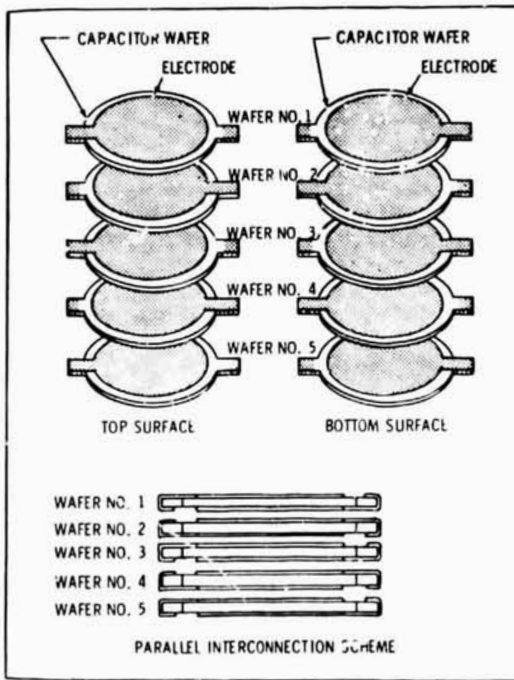

Figure 5. A Five-Layer Stacked Capacitor Showing Tabbed Wafers and Electrode Geometries and Electrode Orientation Necessary for Parallel Electrical Interconnection.

TABLE 1

Pyrolytic Boron Nitride Capacitor Compared with Lower-Temperature Capacitors*

Capacitor Type	DC Working Voltage	Maximum Operating Temp. (°F)	Capacitance per Unit Volume (µF/in.^{3})	Volumetric Efficiency (µF-V/in.^{3})	Capacitance Change from Room Temp. to	Dissipation Factor At 1 kHz
Metallized Polycarbonate	600	200	0.54	320	200°F,+1%	0.002 at 200°F
Teflon, Foil Electrodes	200	400	0.64	127	400°F,-4%	0.001 at 400°F
Mica (commercial)	150	750	0.13	19	750°F,-4%	0.02 at 750°F
Mica (experimental)	250	900	0.03	8	900°F,-25%	0.10 at 900°F
Pyrolytic Boron Nitride (5-wafer stack)	500 to 1000	1100	0.8 to 1.74 (uncased)	400 to 1740 (uncased)	400°F,-0.5% 1100°F,-1.[illegible]%	0.001 at 400°F 0.003 at 1100°F

*Values are typical for the general types of dielectric systems indicated.

Electrode thickness is negligible (about 0.00001 inch) making the total stack height essentially the sum of the thicknesses of the PBN wafers. This construction produces higher capacitance per unit volume than that of other capacitor types, and also considerably higher volumetric efficiency. These and other qualities are compared in Table 1 for a PBN capacitor and several commercial capacitors.

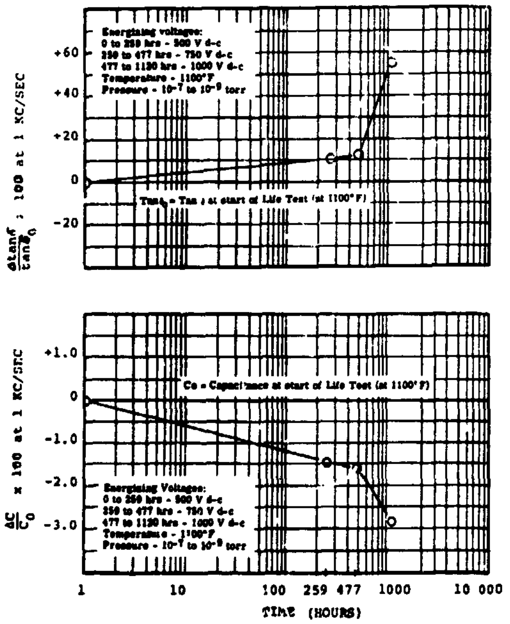

Figure 6. Change in the Ratios of $\frac{\Delta\tan\delta}{\tan\delta_0}$ x 100 and $\frac{\Delta C}{C_0}$ x 100 as a Function of Time and Increased DC Energizing Voltages for a Five-Wafer Multi-Layer Pyrolytic Boron Nitride Capacitor with Sputtered Platinum Electrodes in Vacuum at 1100° F

A 5 wafer PBN capacitor was life tested at 1100°F in vacuum for a total of 1120 hours at a DC voltage stress up to 1,000 volts per mil. Figure 6 shows the change in dissipation factor and capacitance as functions of time and increasing voltage. Note, however, that a more rapid change in capacitance occurred at 477 hours which corresponds to an increase in energizing voltage from 750 to 1,000 volts per mil. Subsequent analysis showed that these changes were probably due to a slight separation of electrodes from the wafer surfaces in the stacked capacitor.

FABRICATION IMPROVEMENTS

Two methods were developed to improve the electrode adherence on PBN wafers in a stacked capacitor. The first method was to RF sputter etch (texturize) both surfaces of a PBN wafer just prior to depositing electrodes. This treatment produced an ultra clean surface and electrode adhearance values greater than 1,000 psi. A 2-3 fold reduction in dissipation factor was an unexpected bonus compared to capacitors made without etching. The reduction in dissipation factor is attributed to the removal of mechanically disturbed surface layers produced during final lapping. About 3,000 angstroms was removed from each surface of a PBN wafer by sputter etching. Removal of additional material had a negligible affect on dissipation factor.

The second improvement was to deposit a diffusion barrier layer over the outer surfaces of each electrode to prevent interelectrode bonding in a stacked capacitor. Boron nitride was RF sputtered from a PBN target using a glass mask to protect the contact tabs. About 500 angstroms of boron nitride was deposited on each electrode at 70 angstroms per minute.

A three wafer capacitor was tested that incorporated these improvements (sputter etching and BN barrier layers). Figure 7 compares the rate of change in capacitance

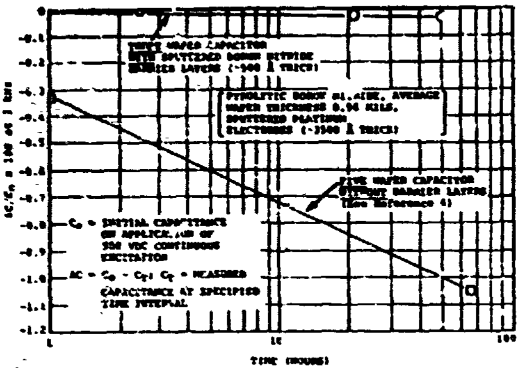

Figure 7. Comparison of the Change in Capacitance Versus Time at 500 V dc/Mil in Vacuum at 1100° F for Pyrolytic Boron Nitride Multi-Layer Capacitors With and Without Sputtered Boron Nitride Barrier Layers.

versus time for the improved 3 wafer capacitor and the original 5 wafer capacitor. The 3 wafer capacitor shows a negligible change in capacitance for the duration of the test (75 hours at 1100°F).

LARGER PBN CAPACITORS AND COST ANALYSIS

The specially designed ceramic package shown in Figure 8 was fabricated to provide the necessary compressive forces, orient and hold PBN capacitor wafers and provide a hermetic enclosure. More than 40 defect free PBN capacitors were made with sputter etched surfaces and boron nitride barrier layers for this package. The package has sufficient internal volume to hold more than 300 capacitor wafers which would be equivalent to a 0.1 uF capacitor. In 1976 a cost analysis was made based on yield data from previous laboratory experience with this process. A 16 percent overall yield assumption was made (from raw material to final test). The cost to fabricate 572 finished wafers (equivalent to 0.16 uF) was about $51,000. Half of this cost was for purchased raw materials (PBN) in the form of 1 x 1 x 1/8 inch blocks.

CONCLUSIONS

Pyrolyic boron nitride capacitors offer the promise of high stability and reliability over long periods in a wide range of environments and operating conditions. These new capacitors should find use in many demanding applications. The cost to make these capacitors by slicing and lapping thick blocks of material is a deterrent to commercialization. A study of methods of producing low defect thin films of PBN would provide the basis for a more cost effective high temperature capacitor technology.

ACKNOWLEDGMENTS*

This development program was funded by NASA Lewis Research Center and conducted at Westinghouse Aerospace Electrical Division under contracts NAS 3-6465 and NAS 3-10941.

The author would like to thank A. C. Beiler and R. A. Lindberg for their support and guidance and S. D. Burkholder, H. Banks and R. Schumate for capacitor fabrication and testing.

REFERENCES

(1) R. E. Stapleton, NASA-CR-1213 (1968) and NASA-CR-1799 (1971). These reports contain a full discussion of PBN wafer slicing, lapping, cleaning, electrode deposition and testing.

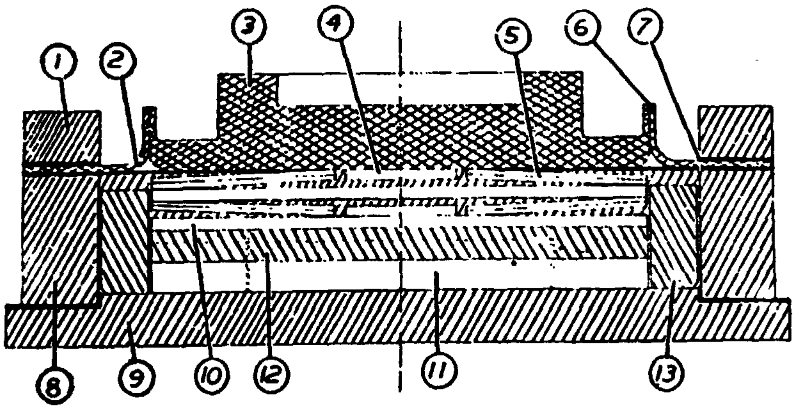

Figure 8. PBN Capacitor Hermetic Package - Volume 0.87 In^3 (to 0.1uF)

LEGEND (Figure 8)

1. Al_2O_3 Cylindrical Ring (1.5 inch O.D.)
2. Top Ring, Cb-1%Zr
3. Top Plug, Cb-1%Zr
4. Spring Alignment disk, Mo
5. Bellville Spring, Ta (T-111)
6. Electron Beam Weld
7. Braze Alloy, 60Zr-25V-15Cb
8. Al_2O_3 Cylinder
9. Metallized BeO Disk, Sputtered Mo
10. Spring Support Plate, Mo
11. PBN Capacitor Stack, 0.78 inch dia.
12. Pressure Plate, PBN
13. Alignment Bushing, PBN