# STUDY
# HIGH DIELECTRIC CONSTANT THIN FILM CAPACITOR MATERIALS

FINAL REPORT



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# I. INTRODUCTION

This report covers the results of a study program conducted to investigate the high vacuum approach for thin film capacitors with high specific capacitances in order to permit reductions in the size of micro-circuits. The study was performed in accordance with the requirements of NASA Manned Spacecraft Center, General Research Procurement Branch, under Contract No. NAS 9-5592. In general, materials of the $ABO_3$ type and having dielectric constant "k" were investigated.

The study program was separated into three phases as follows:

Phase 1. This phase consisted of a literature survey of materials for investigation. The survey included research of material properties in both their bulk and thin film form. The results of the survey, including a bibliography, were previously reported in an interim report dated March 25, 1966. The interim report included five materials recommended for technical exploration in Phase 2.

Phase 2. This phase was the actual study portion of the contract and was started upon receipt of NASA's approval of the materials recommended for further study. Material investigation was limited to the following materials:

1) Sodium niobate
2) Solid solution of barium zirconate
3) Solid solution of lead zirconate

Numberous depositional approaches, such as laser, electron bombardment, flash evaporation, and dual evaporation, were explored. The physics, crystallography, and chemistry of the resultant thin films were established by utilization of optical and electron microscopy, X-ray diffraction, and spectrographic analysis. The electrical characteristics of the thin films when utilized as capacitor dielectrics were established with regard to temperature coefficient of capacitance, specific capacitance, dielectric constant, frequency response, voltage breakdown characteristics and stability.

Phase 3. This phase is to consist of fabrication and delivery of working capacitors to the NASA Manned Spacecraft Center in Houston along with appropriate drawings and process procedures utilized in the fabrication of the capacitors. This phase is to cover a two-month time spectrum and will be begun upon approval of Phase 2 by NASA.

Mr. L. A. Darling, Manager of the TRW Systems Microelectronics Department, served as program manager; and Mr. R. P. Radke was in charge of the investigation in the capacity of project engineer. Mr. Radke was assisted by thin film development engineers of the Microelectronics Department, as well as specialists in electron microscope and X-ray diffraction evaluation of thin films, as shown in Figure 1.

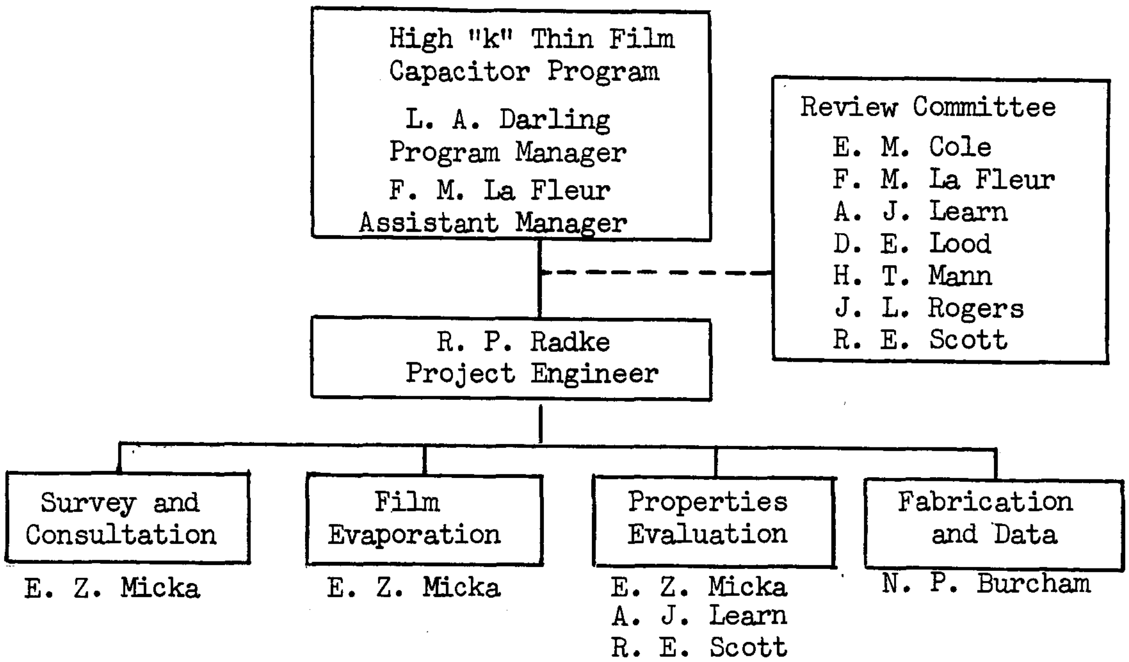


Figure 1. Program Organization

Sources used as references throughout this study are included in the bibliography at the end of this report.

## II. BACKGROUND

NASA requested in "Exhibit A" of the contract that a specified list of twenty compounds be surveyed during the Phase 1 effort. All of these plus an additional nine materials were surveyed. A list of these materials appears in Table I, along with references to their room temperature crystal structure, electrical properties, Curie point, and dielectric constant.

The previously submitted interim report included a discussion of ferroelectricity and antiferroelectricity and the bulk properties of numerous compounds and the thin film properties of the same compounds. The materials were grouped and discussed according to type (i.e., titanates, zirconates, hafnates, stannates, niobates, tantalates, and other materials).

The materials recommended in the interim report for further study in Phase 2 were:

$BaTiO_3$
$PbTiO_3$
$(Ba\text{-}Pb)ZrO_3$
$KNbO_3$
$NaNbO_3$

In a communication received from Marion C. Owens, Contracting Officer for NASA Houston (reference BG731-39, C. D. Stamps), dated April 12, 1966, and received by TRW Systems on April 19, 1966, NASA authorized TRW, Inc. to proceed with Phase 2, limited to future investigation of the following materials only:

Sodium niobate
Solid solution of barium zirconate
Solid solution of lead zirconate

The remaining materials recommended by TRW Systems for further investigation were to be explored only in the event that investigation of the above noted compounds proved fruitless.

TABLE I. COMPOUNDS SURVEYED DURING PHASE 1

| MATERIAL | ROOM TEMPERATURE CRYSTAL STRUCTURE | ELECTRICAL PROPERTIES | CURIE POINT | DIELECTRIC CONSTANT | TEST CONDITIONS | | COMMENTS |
|---|---|---|---|---|---|---|---|
| | | | | | TEMP. | FREQ. | |
| $BaTiO_3$ | Tetragonal | Ferroelectric | 120°C | 1400 | 25°C | 1KC | |
| $PbTiO_3$ | Tetragonal | Ferroelectric | 490°C | 450 | 25°C | 1KC | Thin film form |
| $SrTiO_3$ | Cubic | Ferroelectric | ~40°K | 250 | 25°C | 50 cps | |
| $CaTiO_3$ | Orthorhombic | Paraelectric | N/A | 143 | 25°C | 50KC | |
| $CdTiO_3$ | Orthorhombic | Ferroelectric | -218°C | 250 | 25°C | — | |
| $PbZrO_3$ | Orthorhombic | Antiferroelectric | 233°C | ~100 | 25°C | — | |
| $BaZrO_3$ | Cubic | Paraelectric | N/A | low | — | — | |
| $(Ba_{0.4}Pb_{0.6})ZrO_3$ | — | Paraelectric | N/A | 2600 | 25°C | 1KC | |
| $SrZrO_3$ | Cubic | Paraelectric | N/A | low | — | — | |
| $CaZrO_3$ | Orthorhombic | Paraelectric | N/A | low | — | — | |
| $PbHfO_3$ | Tetragonal | Antiferroelectric | 215°C | 90 | 25°C | 10KC | |
| $SrHfO_3$ | — | Paraelectric | N/A | low | — | — | |
| $BaHfO_3$ | — | Paraelectric | N/A | low | — | — | |
| $BaSnO_3$ | Cubic | Paraelectric | N/A | 17 | 25°C | 1KC | |
| $SrSnO_3$ | Cubic | Paraelectric | N/A | 17 | 25°C | 1KC | |
| $CaSnO_3$ | Orthorhomic | Paraelectric | N/A | 15 | 25°C | 1KC | |
| $MgSnO_3$ | — | — | — | 62 | 25°C | 1KC | |
| $Bi_2(SnO_3)_2$ | — | — | — | 34 | 25°C | 1KC | |
| $PbSnO_3$ | — | — | — | 16 | 25°C | 1KC | |
| $CaSnO_3$ | — | — | — | 16 | 25°C | 1KC | |
| $NiSnO_3$ | — | — | — | 235 | 25°C | 1KC | |
| $KNbO_3$ | Orthorhombic | Ferroelectric | 434°C | ~500 | 25°C | 1KC | |
| $NaNbO_3$ | Orthorhombic | Antiferroelectric | 355°C | 575 | 25°C | 1KC | |
| $Cd_2Nb_2O_7$ | — | — | 170°K | 320/500 | 25°C | — | |
| $Pb(NbO_3)_2$ | — | — | 575°C | 300 | 25°C | — | |
| $KTaO_3$ | Cubic | Ferroelectric | ~ 13°K | Below 500 | 25°C | — | |
| $NaTaO_3$ | Orthorhombic | Paraelectric | 475°C | — | — | — | |
| $WO_3$ | Monoclinic | Antiferroelectric | -50°C | — | — | — | |
| SbSI | Monoclinic | Ferroelectric | 22°C | 50,000 | 22°C | — | Parallel to needle axis |

— No information available

N/A Not applicable

## III. TEST CAPACITOR GEOMETRIES

The test capacitor configuration utilized for the depositions in the earlier part of the program allowed for four capacitors per substrate, all using a common base electrode (Figure 2).

During the latter stages of Phase 2 when successful capacitor depositions were being performed, an improved test geometry was utilized (Figure 3). This particular configuration allowed for sixteen capacitors per substrate and obviously produced a greater number of units for test for any given deposition cycle. This second test pattern suffered from the shortcoming of producing "cross talk" due to the presence of common base and counter electrodes when a short in a given capacitor was obtained.

A modification of the second test pattern was devised and is the one currently in use (Figure 4). This configuration still allows for sixteen capacitors per substrate, but each now has an isolated counter electrode. In addition, the pattern has been so designed that a substantial proportion of reflective material overlaps the dielectric step, thereby facilitating the taking of interferrometric thickness measurement directly on the substrate.

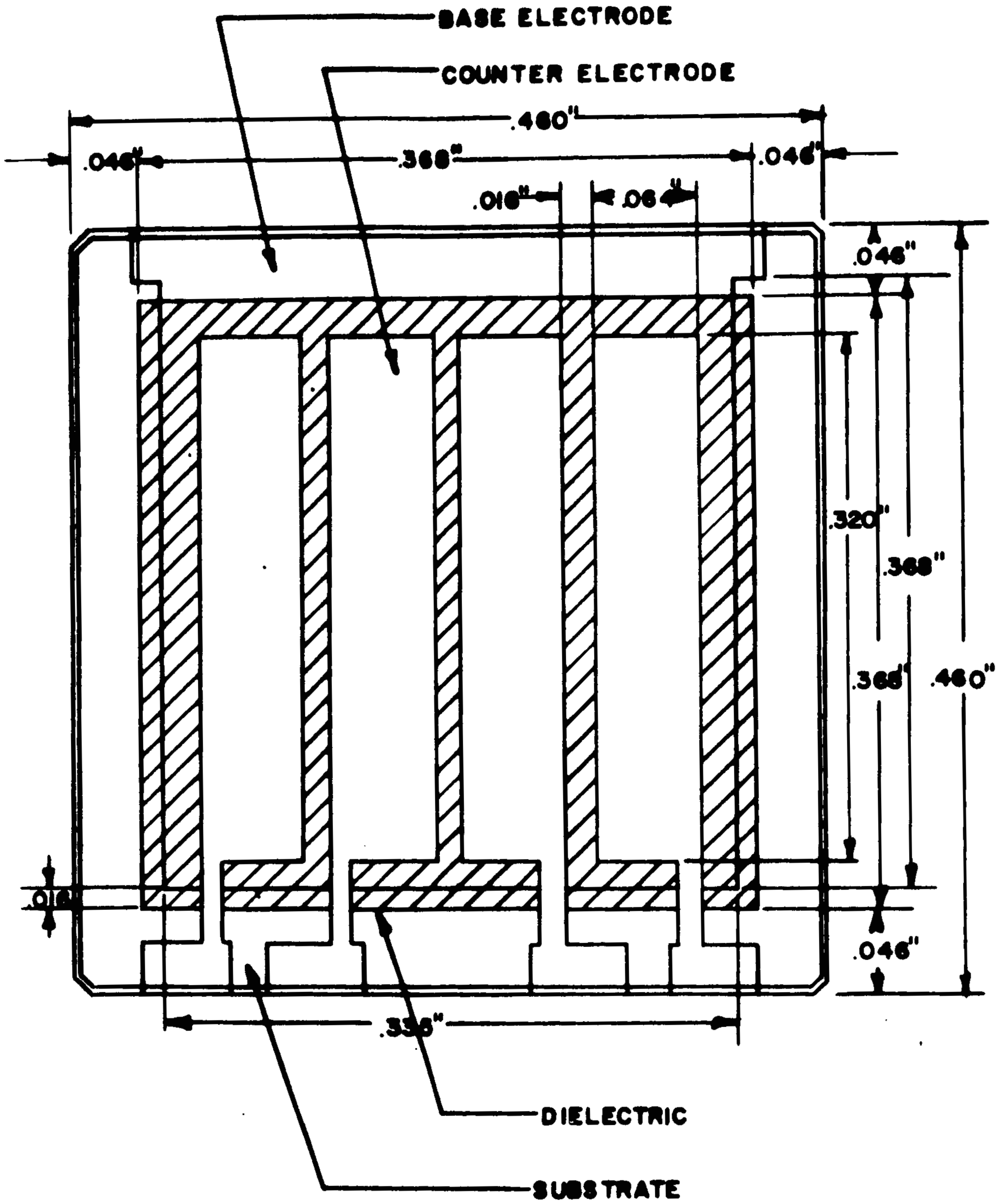


Figure 2. Capacitor Test Pattern (First)

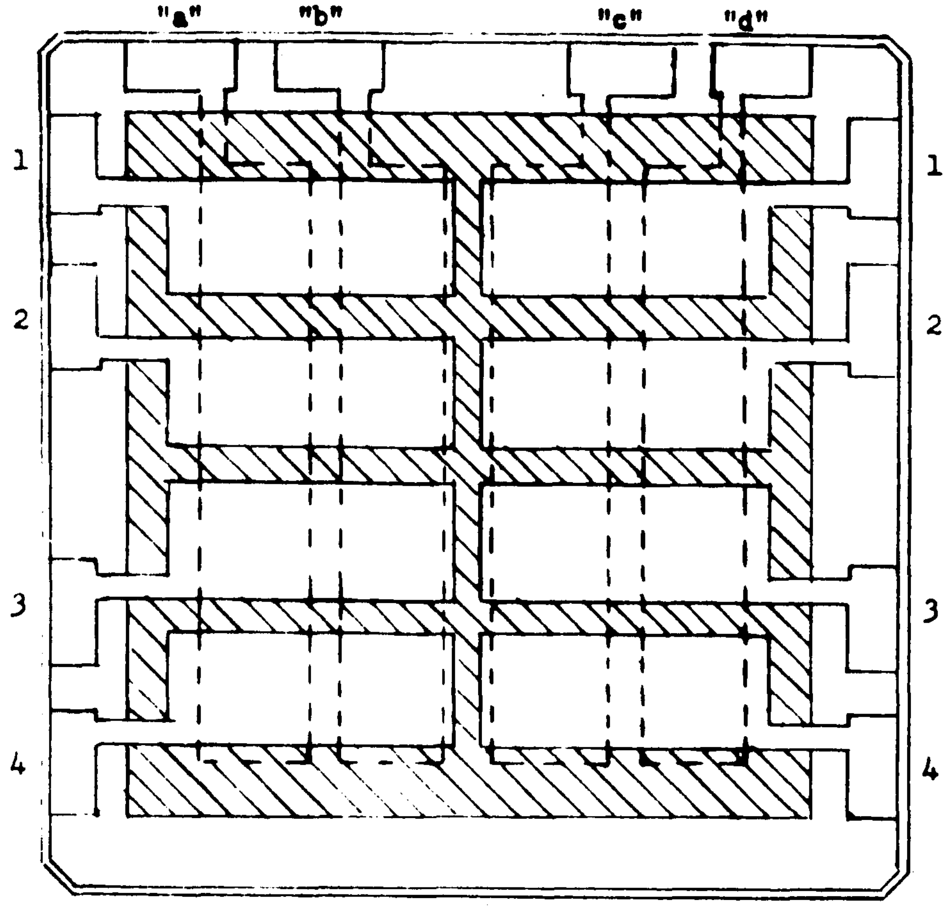


Figure 3. Capacitor Test Pattern (Second)

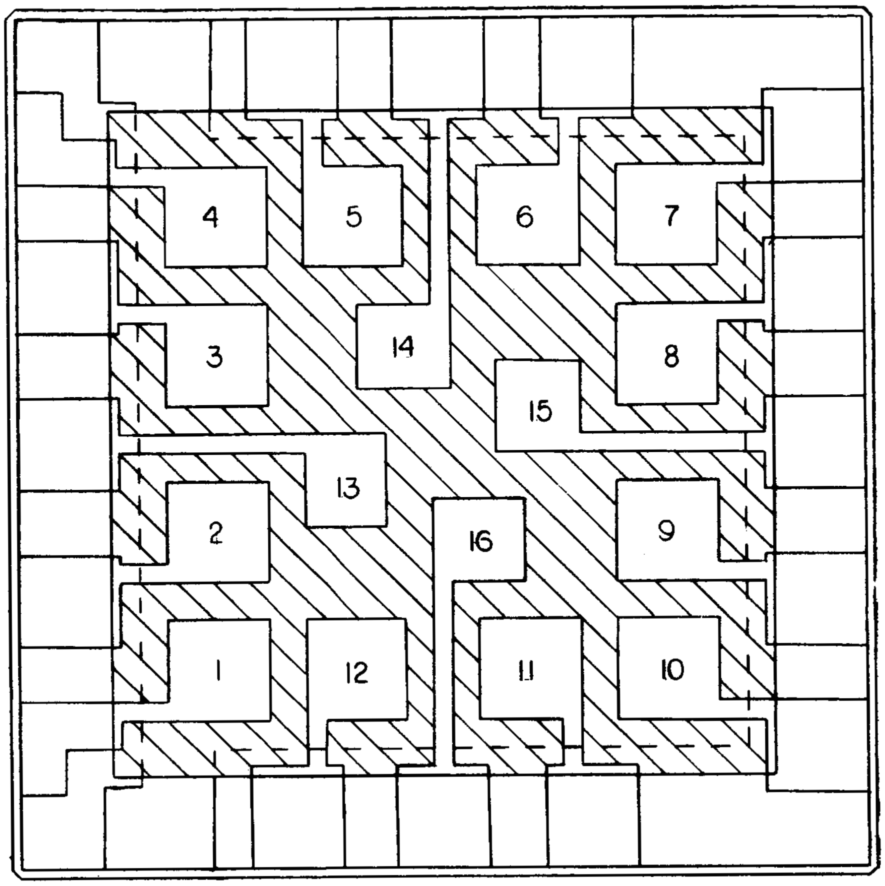


Figure 4. Capacitor Test Pattern (Final)

## IV. DEPOSITION MODES

### FLASH EVAPORATION

A dual flash evaporation unit was designed and constructed in the beginning months of the program (Figures 5, 6, and 7). Evaporations of barium-lead zirconate, barium titanate, and $(Ba_{0.6},Pb_{0.4})ZrO_3$ were performed utilizing this mechanism. Spectrographic analysis of samples prepared via this technique (i.e., flash evaporation) has shown high contamination levels from the refractory metal source material. Table II below shows a reasonably typical analysis of sodium niobate films deposited by flash evaporation.

TABLE II. ANALYSIS OF SODIUM NIOBATE FILMS DEPOSITED BY FLASH EVAPORATION

| Element | % Present | |
|---|---|---|
| Niobium | 51. % | 49. % |
| Sodium | 5.6 | 5.0 |
| Tungsten | 15. | 18. |
| Iron | 0.15 | 0.40 |
| Silicon | 0.28 | 0.51 |
| Boron | nil | nil |
| Manganese | nil | nil |
| Magnesium | 0.019 | 0.078 |
| Chromium | trace | nil |
| Aluminum | 0.046 | 0.19 |
| Copper | 0.056 | 0.25 |
| Silver | nil | nil |
| Titanium | trace | nil |
| Nickel | nil | nil |
| Calcium | 0.36 | 0.31 |
| Molybdenum | nil | trace 0.04 |
| Lead | nil | 0.12 |
| Other Elements | nil | nil |

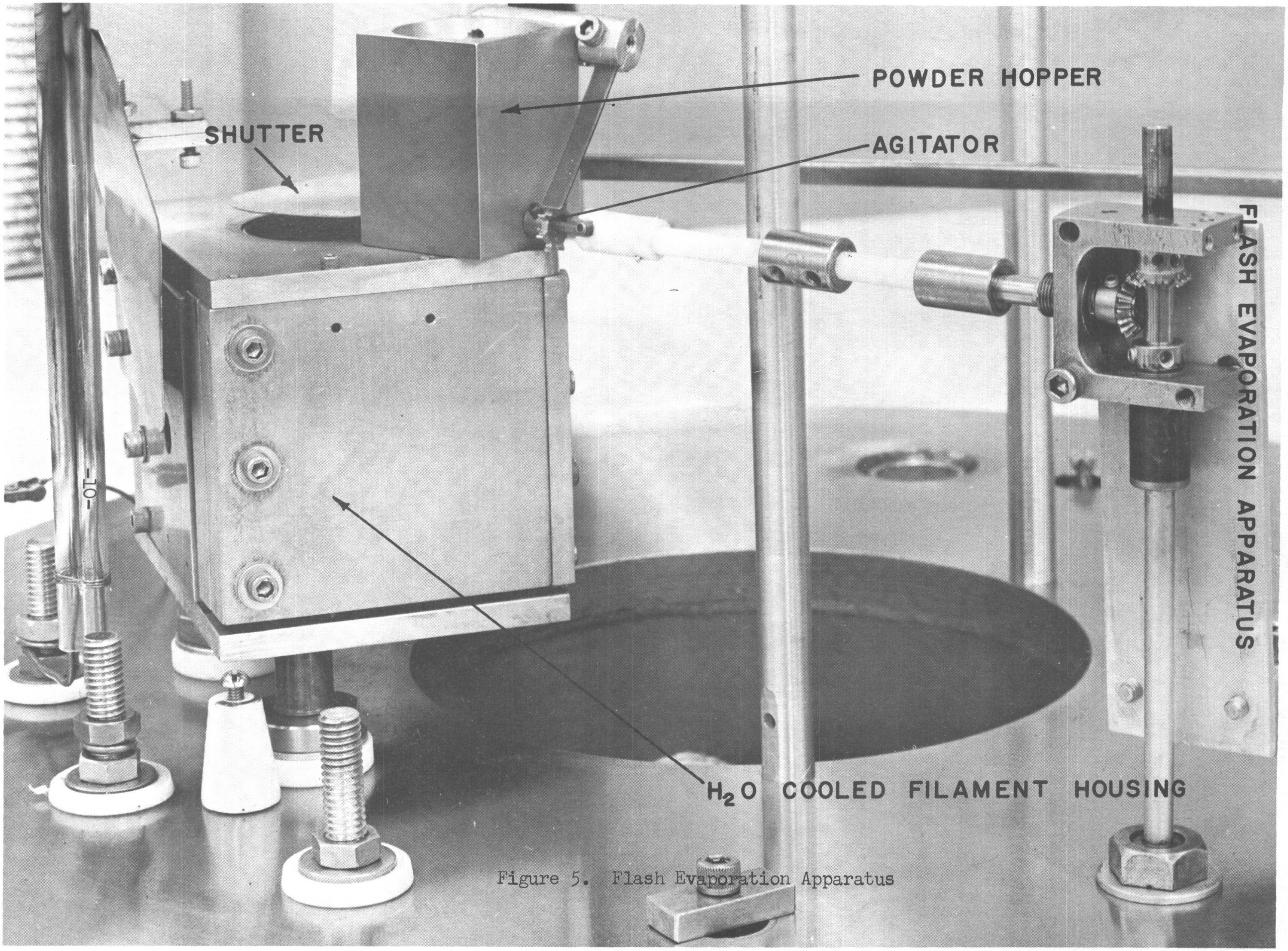


Figure 5. Flash Evaporation Apparatus

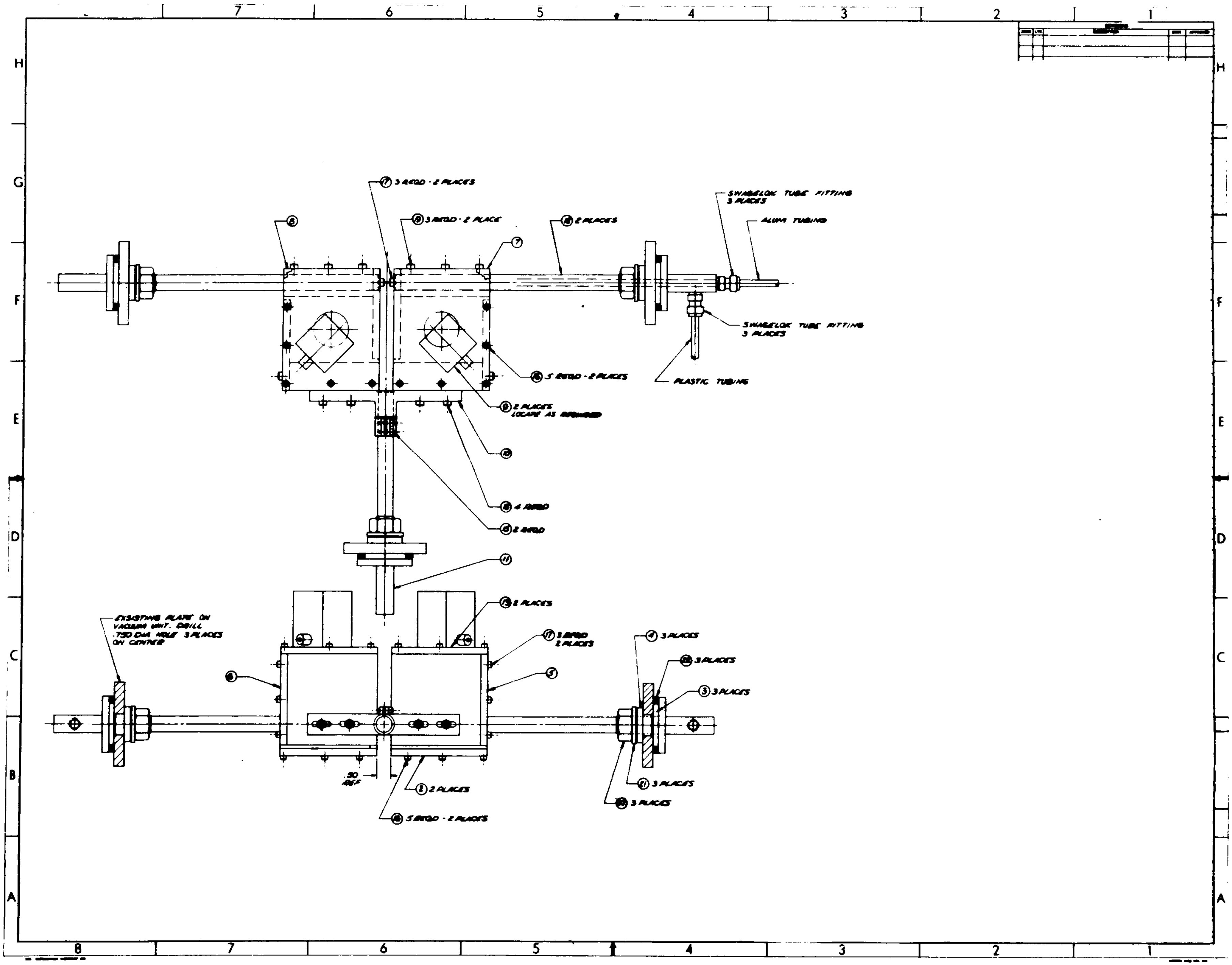


Figure 6. Dual Flash Evaporation
Fixture Assembly

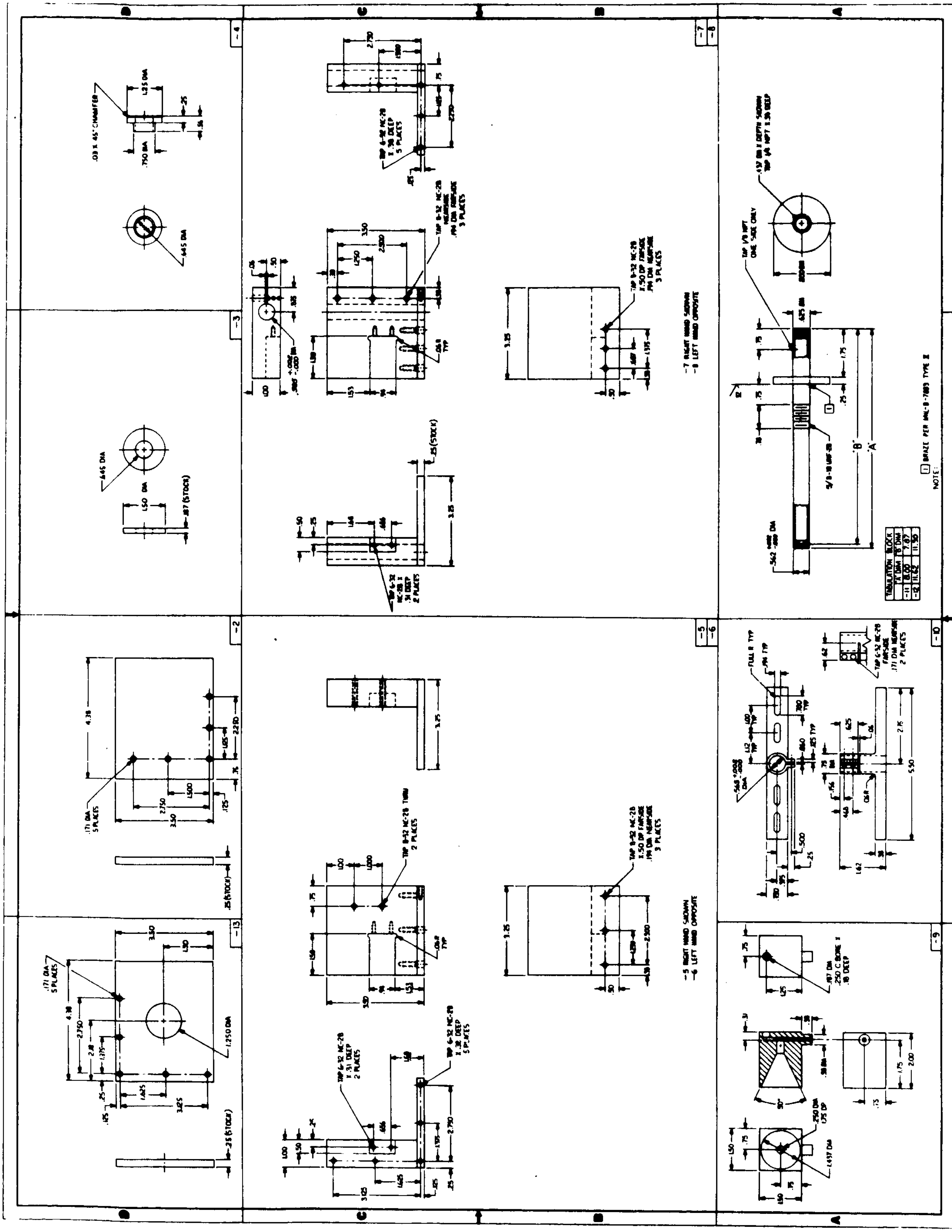

Figure 7. Dual Flash Evaporation Fixture Detail

Numerous attempts at reducing the contamination level of the refractory source included utilization of $Al_2O_3$-coated molybdenum, zirconium diboride ($ZrB_2$)-coated molybdenum, irridium, electron beam heating of carbon, and electron beam heating of vitreous carbon (a new material which is essentially a vitrified carbon and which exhibits extreme inertness and low thermal coefficient of expansion). The oxide-coated refractory metals were incapable of being heated to sufficiently high temperatures to allow for flash evaporation without decomposing. The irridium melted at the required temperatures. The electron beam heating of the carbon and vitreous carbon eroded the source and caused a deposit of source material to appear on the substrate.

The problem of source contamination was eventually solved by devising a unique and practical method designated as the "Molten Sphere Technique". The specifics of this technique are described later in this report.

## ELECTRON BOMBARDMENT

Electron bombardment evaporation of $(Ba_{0.6},Pb_{0.4})ZrO_3$ and of sodium niobate ($NaNbO_3$) was performed with a Filmtech Associates multiple source electron gun, Model TG-1 (Figure 8). A powder press was utilized to fabricate 1/4" pellets of appropriate evaporants. In order to provide for a high residual $O_2$ in the vicinity of the substrate, a scheme was devised, constructed, and put into operation which would allow for adequate recombination of the decomposed and fractionated constituents, while still maintaining a sufficiently low pressure in the vicinity of the hot tungsten electron emitter to preclude decomposition and oxidation of the emitter (Figure 9). An example of typical results is shown in Figure 10. The pictured "slugs" are shown in various stages of evaporation. The arrows point to a tungsten wire which was inserted through the core of the slug to provide an electron drain. These electron drains prove to be a serious source of contamination

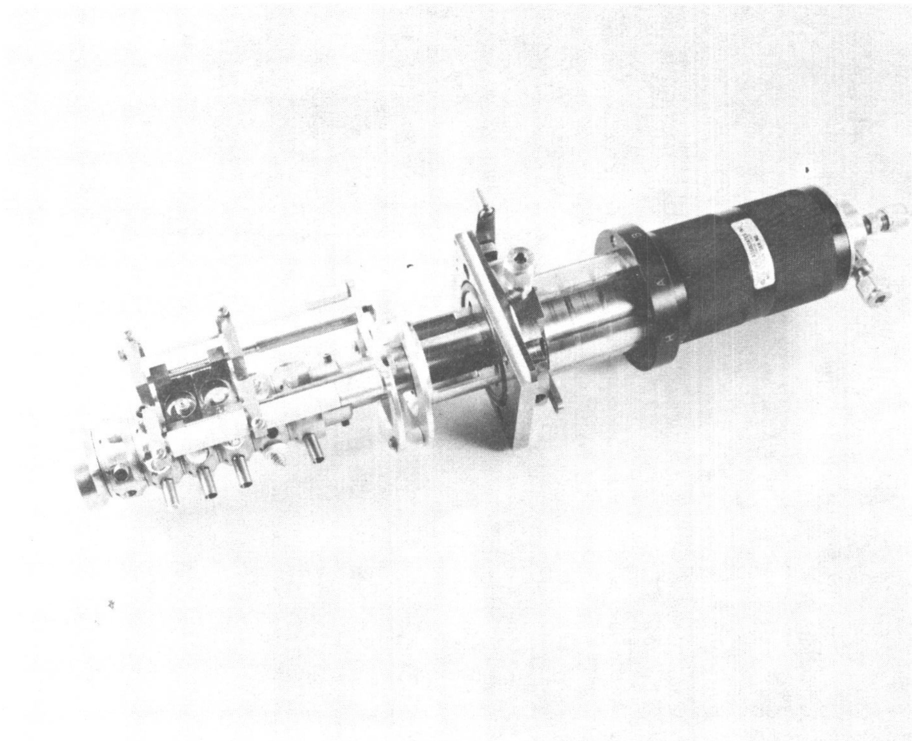

Figure 8. Multiple Source Electron Gun

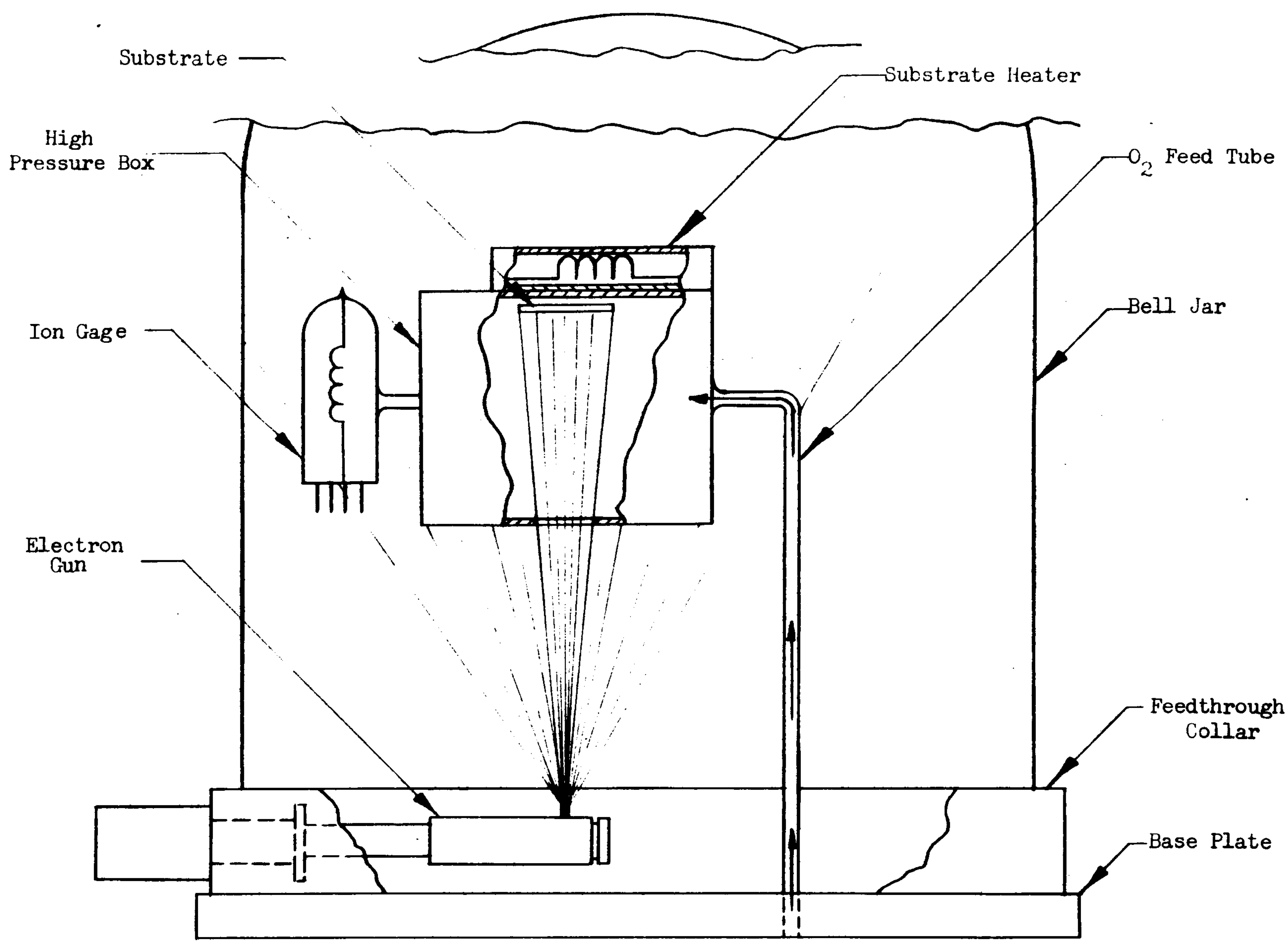


Figure 9. Differential Pressure Apparatus

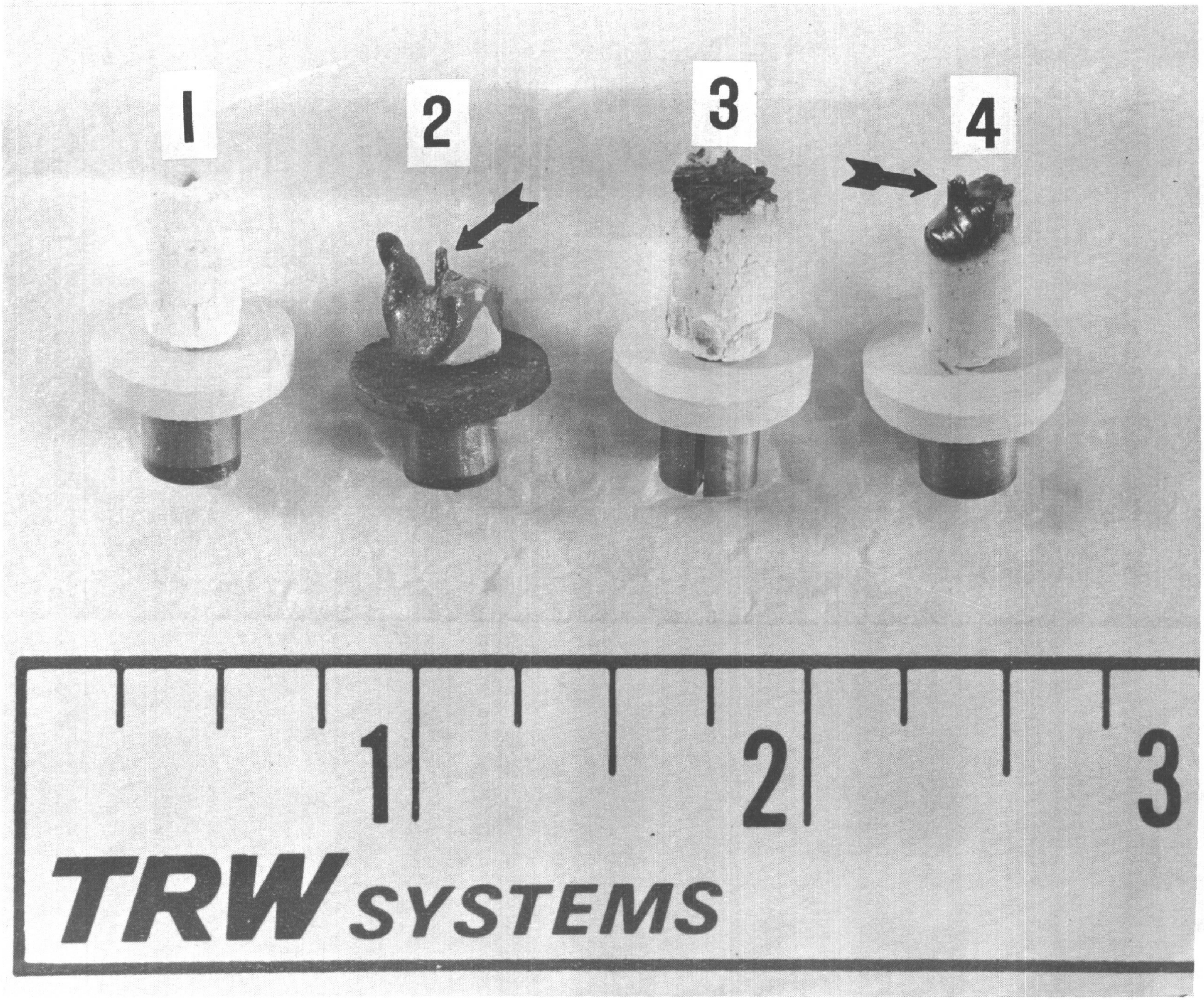


Figure 10. "Slugs" Shown in Various Stages of Evaporation

as shown in Table III. Substitution of niobium wire in place of tungsten precluded contamination by an extraneous constituent, but the technique still proved to be impractical, primarily as a result of the relatively low rate of evaporation which was maintained. Successive evaporation of as many as six slugs yielded films only in the order of 500-800 Å. This was partially due, of course, to the low mean-free path resulting from the necessity of maintaining a high residual $O_2$ pressure.

TABLE III. SPECTROGRAPHIC ANALYSIS OF ELECTRON BOMBARDMENT DEPOSITED THIN FILM OF $NaNbO_3$ UTILIZING TUNGSTEN ELECTRON DRAIN.

| Element | % Present |
|---|---|
| Niobium | 24. % |
| Sodium | 29. |
| Tungsten | 13. |
| Iron | 0.42 |
| Silicon | 2.8 |
| Boron | 0.027 |
| Manganese | trace |
| Magnesium | 0.095 |
| Chromium | 0.34 |
| Aluminum | 0.58 |
| Copper | 0.35 |
| Silver | 0.018 |
| Titanium | 0.15 |
| Nickel | 0.089 |
| Calcium | 2.0 |
| Other Elements | nil |

LASER

A neodymium-doped glass laser capable of a peak pulse output of approximately 14 joules per pulse was designed and constructed within the TRW facilities. The laser utilizes a water-cooled 1/2" x 4" neodymium glass rod and is capable of pulse repetition rates of approximately three to four per minute. However, encouraging results obtained with sodium niobate capacitors deposited via the "Molten Sphere" technique precluded a concommitant investigation of laser evaporation.

"MOLTEN SPHERE"

As an extension of the technique of electron beam heating of a refractory source (such as carbon or vitreous carbon)in order to reduce contamination, a novel and effective technique was evolved which in essence consisted of powder feeding sodium-containing compounds onto a molten and evaporating sphere of electron beam heated niobium. The technique (Figure 11) has been designated the "Molten Sphere" technique.

The sodium containing compounds investigated were selected on the basis of their likelihood of decomposing upon hitting the hot niobium sphere to a form of sodium or sodium oxide and a gas, which would be pumped from the system. $NaNO_2$, NaCl, and $Na_2CO_3$ were all attempted as the feed powder. The first two proved to be impractical, but the $Na_2CO_3$ produced encouraging results. It is hypothesized that the sodium carbonate, upon hitting the hot niobium, decomposes in any one of the following ways:

$$2\ Na_2CO_3 \longrightarrow 2\ Na_2O_2 + 2\ CO$$

$$2\ Na_2CO_3 + O_2 \longrightarrow 2\ Na_2O_2 + 2\ CO_2$$

$$Na_2CO_3 \longrightarrow Na_2O + CO_2$$

$$3\ Na_2CO_3 + O_2 \longrightarrow Na_2O + 2\ Na_2O_2 + 3\ CO_2$$

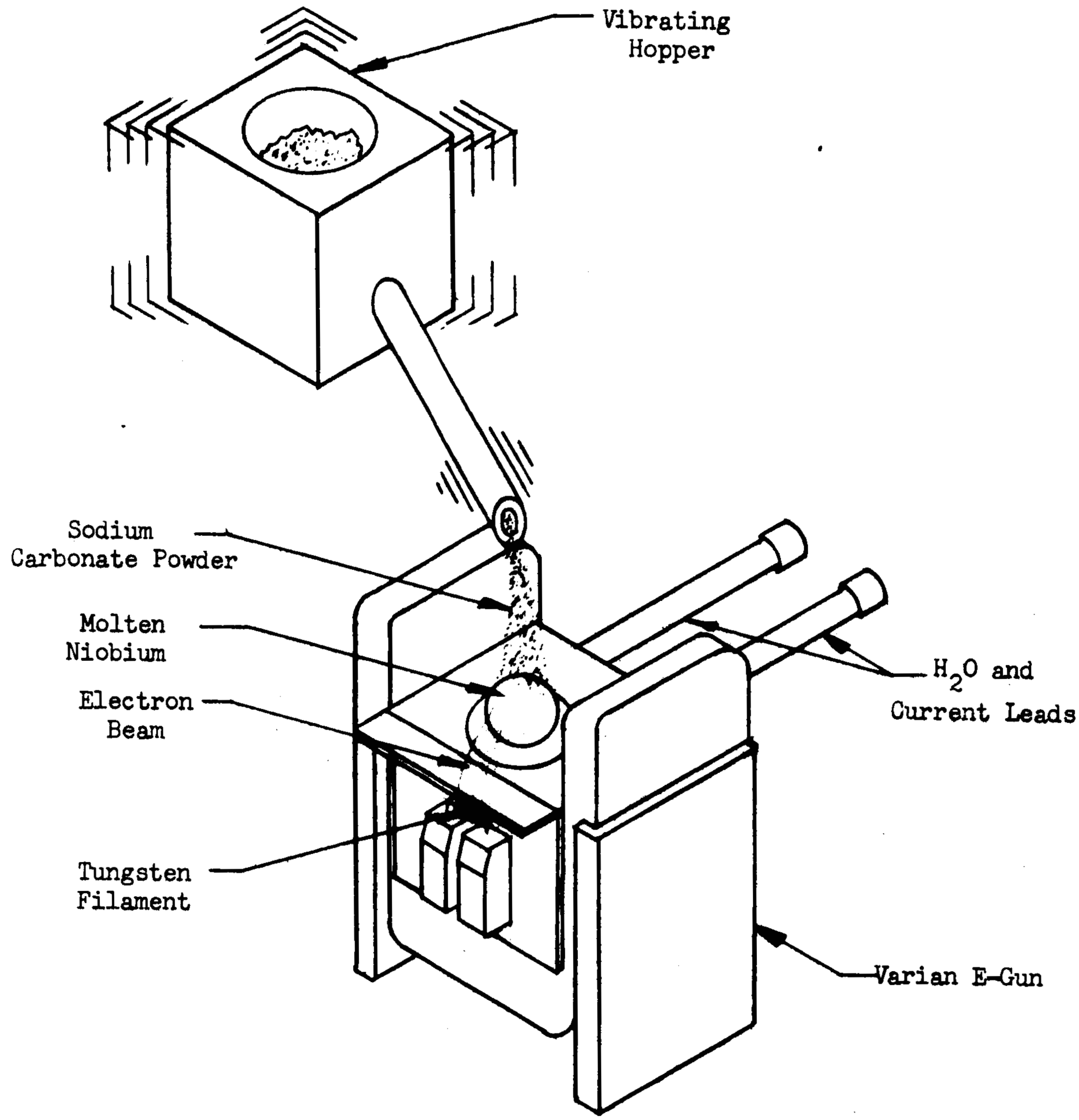


Figure 11. "Molten Sphere Technique"

Spectrographic analysis was performed on films deposited via this technique and did indeed corroborate the hypothesis that refractory metal contamination would be eliminated. The initial analyses (Table IV) indicated a disproportionately high silicon contamination. This was assumed to be resulting from the failure of the liquid nitrogen feed mechanism which caused backstreaming of cracked silicon diffusion pump oil components into the system. Spectrographic analyses of the raw materials (Table V) indicated that the source of the silicon contamination was not inherent in the parent material. A subsequent spectrographic analysis of a "Molten Sphere" evaporated sodium niobate film (Table VI) indicated a silicon contamination of only 0.1% and a reasonably good ratio of niobium to sodium (55:12, as compared with the idealized stoichiometric ratio of 56.7:14.0).

Good yields of sodium niobate capacitors were obtained with the "Molten Sphere" technique. The dissipation factors were encouragingly low (in the vicinity of 5-10% for a majority of the capacitors). The data sheets, Tables VII, VIII, and IX, show the initial readings of these capacitors.

Both optical and electron microscopy were utilized to establish insights into the crystallographic character of the evaporated thin films. Particularly good examples of the orthorhombic crystallinity are shown in Figures 12 and 13.

In order to establish what degree of repeatability could be obtained with the "Molten Sphere" technique, three additional runs (T-39, 40, and 41) were deposited in a manner exactly analagous to runs T-28, 29, and 30. The only test difference is inherent in the fact that two quartz and two sapphire substrates were utilized in each run rather than four quartz substrates. After the initial readings were taken, the substrates from all three runs

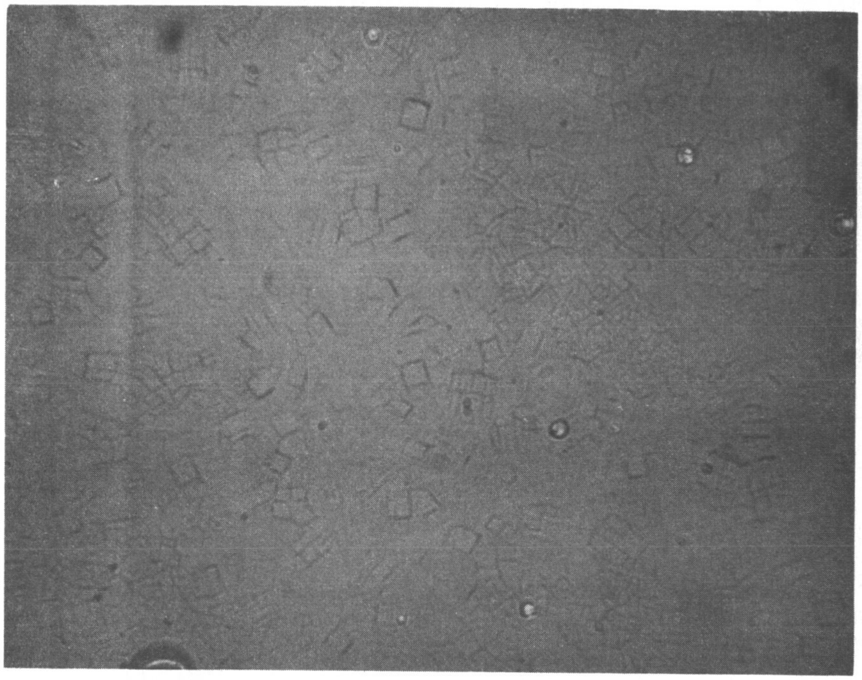

Figure 12. Orthorhombic Crystallinity (500X)

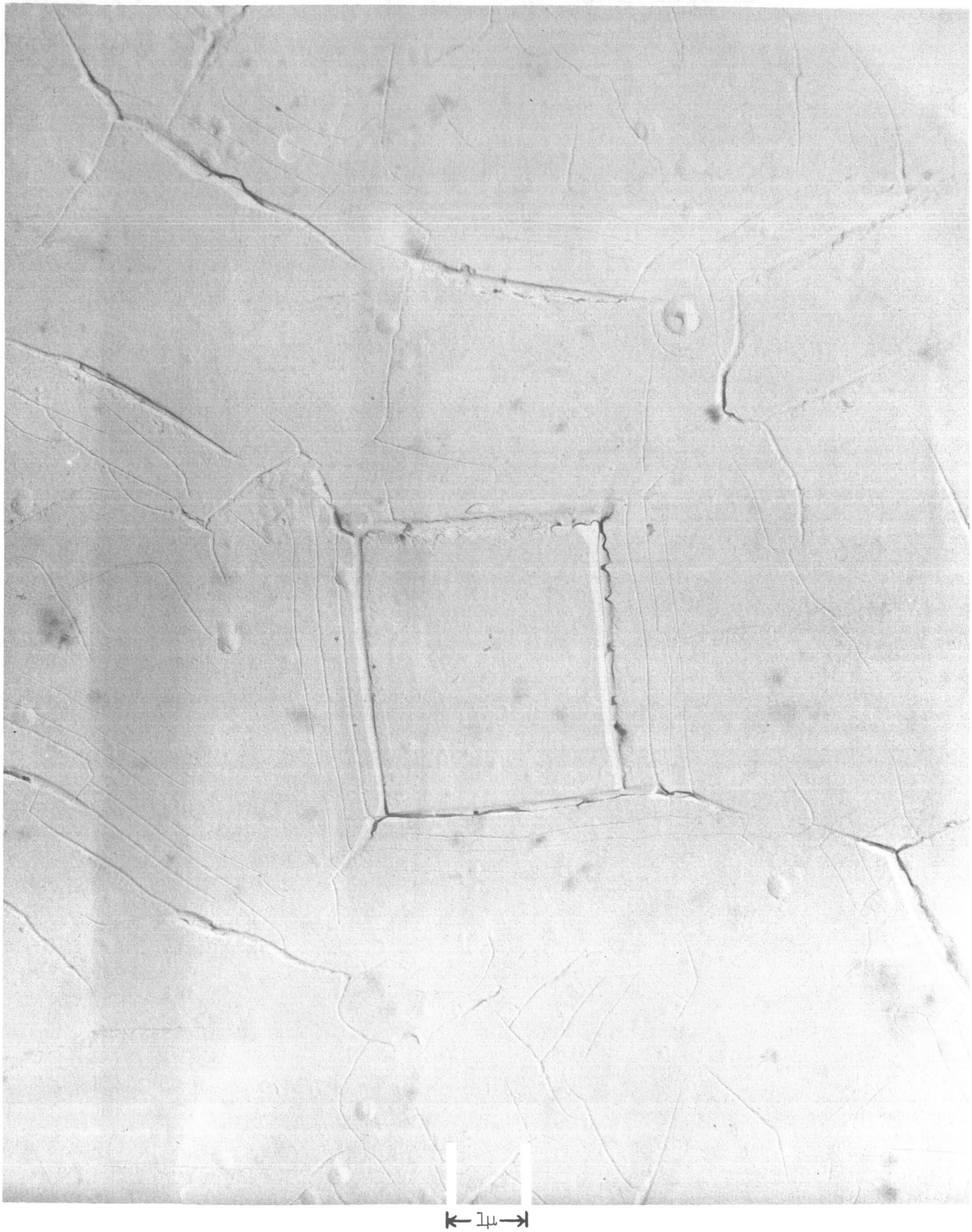


Figure 13. Orthorhombic Crystallinity
(24,000X)

TABLE IV. $NaNbO_3$ SAMPLES (VACUUM DEPOSITED THIN FILM ON CARBON)

| Element | No. 1 | No. 2 |
|---|---|---|
| Niobium | 50. % | 53. % |
| Silicon | 4.1 | 3.7 |
| Gold | 0.085 | nil |
| Iron | 0.21 | 0.28 |
| Magnesium | 0.073 | 0.058 |
| Lead | 0.18 | nil |
| Aluminum | 0.43 | 0.19 |
| Molybdenum | 0.054 | 0.043 |
| Copper | 0.27 | 0.25 |
| Silver | 0.011 | nil |
| Sodium | 13. | 10. |
| Nickel | trace | nil |
| Calcium | 0.28 | 0.15 |
| Other Elements | nil | nil |

TABLE V. RAW MATERIALS ANALYSIS

| Niobium Wire | | Sodium Carbonate Powder | |
|---|---|---|---|
| Niobium | Remainder | Sodium | 43. % |
| Titanium | 0.056% | Silicon | 0.0065 |
| Iron | nil | Iron | 0.070 |
| Aluminum | " | Aluminum | 0.0028 |
| Copper | " | Copper | 0.0019 |
| Titanium | " | Titanium | 0.0028 |
| Calcium | " | Calcium | 0.0051 |
| Chromium | " | Chromium | 0.0071 |
| Magnesium | " | Magnesium | 0.0015 |
| Other Elements | " | Other Elements | nil |

TABLE VI. SPECTROGRAPHIC ANALYSIS OF $NaNbO_3$ FILM, "MOLTEN SPHERE" EVAPORATED

| Element | % Present |
|---|---|
| Niobium | 55. % |
| Sodium | 12. |
| Iron | 0.044 |
| Silicon | 0.100 |
| Manganese | 0.054 |
| Magnesium | 0.033 |
| Copper | 0.091 |
| Calcium | 0.31 |
| Other Elements | nil |

TABLE VII. CAPACITOR DATA, INITIAL READINGS
(T28)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T28 | | - - - | | - - - | | - - - | |
| Substrate Material | | Quartz | | - - - | | - - - | | - - - | |
| Substrate Temperature | | 480°C | | - - - | | - - - | | - - - | |
| Postevaporative Bake | | 17 hr. at 450°C | | - - - | | - - - | | - - - | |
| Thickness | | 1600 Å | | - - - | | - - - | | - - - | |
| "k" (Cap. of Calc.) | | 140 (18 nf) | | - - - | | - - - | | - - - | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | 18.6 nf | 0.068 | 17.8 nf | 0.075 | 20.3 nf | 0.075 | 20.6 nf | 0.063 |
| | 2 | 18.0 | 0.095 | 16.3 | 0.093 | 17.6 | 0.12 | 18.3 | 0.11 |
| | 3 | 17.4 | 0.13 | 16.0 | 0.12 | 17.2 | 0.13 | 17.6 | 0.12 |
| | 4 | 18.3 | 0.13 | 17.2 | 0.12 | 18.3 | 0.12 | 19.3 | 0.14 |
| B | 1 | — | — | 29.4 | 0.32 | — | — | — | — |
| | 2 | 16.2 | 0.14 | 14.8 | 0.13 | — | — | — | — |
| | 3 | — | — | 31.0 | 0.16 | — | — | — | — |
| | 4 | — | — | 30.6 | 0.14 | — | — | — | — |
| C | 1 | 14.3 | 0.077 | 14.5 | 0.073 | 14.9 | 0.082 | 14.6 | 0.084 |
| | 2 | 14.5 | 0.080 | 14.5 | 0.077 | 15.4 | 0.091 | 15.4 | 0.094 |
| | 3 | 14.4 | 0.084 | 14.4 | 0.087 | 15.2 | 0.085 | 15.6 | 0.090 |
| | 4 | 22.4 | 0.074 | — | — | — | — | 23.0 | 0.100 |
| D | 1 | — | — | — | — | 29.3 | 3.0 | 44.5 | 2.8 |
| | 2 | — | — | — | — | 27.0 | 3.2 | 27.5 | 8.2 |
| | 3 | — | — | — | — | 27.0 | 3.0 | 29.0 | 3.7 |
| | 4 | — | — | — | — | 31.0 | 3.4 | 32.0 | 2.6 |

TABLE VIII. CAPACITOR DATA, INITIAL READINGS (T29)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T29 | | - - - | | - - - | | - - - | |
| Substrate Material | | Quartz | | - - - | | - - - | | - - - | |
| Substrate Temperature | | 375°C | | - - - | | - - - | | - - - | |
| Postevaporative Bake | | 15 hr at 450°C | | - - - | | - - - | | - - - | |
| Thickness | | 2400 Å | | - - - | | - - - | | - - - | |
| "k" (Cap. of Calc.) | | 100 (8.5 nf) | | - - - | | - - - | | - - - | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | — | — | — | — | — | — | 18.8 nf | 0.04 |
| | 2 | 13.0 | 0.15 | 9.8 | 0.04 | 18.8 nf | 0.05 | — | — |
| | 3 | 10.0 | 0.033 | — | — | — | — | 18.4 | 0.85 |
| | 4 | — | — | — | — | 10.0 | 0.03 | 10.0 | 0.036 |
| B | 1 | — | — | 12.7 | 0.22 | — | — | 20.1 | 0.32 |
| | 2 | 9.1 | 0.10 | 8.8 | 0.18 | 9.4 | 0.039 | 10.8 | 0.28 |
| | 3 | 9.1 | 0.059 | 8.4 | 0.060 | 20.2 | 0.25 | — | — |
| | 4 | 9.2 | 0.062 | 8.6 | 0.060 | — | — | 20.0 | 0.62 |
| C | 1 | 8.6 | 0.12 | 8.0 | 0.048 | 8.1 | 0.049 | 8.6 | 0.05 |
| | 2 | 8.0 | 0.014 | 7.8 | 0.045 | 7.5 | 0.056 | 7.7 | 0.06 |
| | 3 | 8.2 | 0.054 | 7.7 | 0.064 | 7.0 | 0.056 | 7.4 | 0.055 |
| | 4 | 8.7 | 0.049 | 8.1 | 0.044 | 7.0 | 0.053 | 7.5 | 0.049 |
| D | 1 | 9.0 | 0.041 | 8.1 | 0.042 | 11.9 | 0.47 | 17.0 | 1.0 |
| | 2 | 8.7 | 0.042 | 8.7 | 0.046 | 13.7 | 1.8 | — | — |
| | 3 | 8.6 | 0.043 | 8.6 | 0.040 | 8.8 | 0.057 | 9.1 | 0.070 |
| | 4 | 8.6 | 0.046 | 9.1 | 0.040 | 8.5 | 0.055 | 8.7 | 0.045 |

TABLE . CAPACITOR DATA, INITIAL READINGS
(T30)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T30 | | - - - - | | - - - - | | - - - - | |
| Substrate Material | | Quartz | | - - - - | | - - - - | | - - - - | |
| Substrate Temperature | | 380°C | | - - - - | | - - - - | | - - - - | |
| Postevaporative Bake | | 16 hr. at 450°C | | - - - - | | - - - - | | - - - - | |
| Thickness | | 2400 Å | | - - - - | | - - - - | | - - - - | |
| "k" (Cap. of Calc.) | | 155 (13) | | - - - - | | - - - - | | - - - - | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | 16.8 nf | 0.25 | 16.7 nf | 0.046 | 21.0 nf | 0.32 | 20.0 nf | 0.32 |
| | 2 | 16.8 | 0.45 | 17.0 | 0.29 | 18.3 | 0.11 | 18.0 | 0.151 |
| | 3 | — | — | — | — | 18.5 | 0.092 | 18.0 | 0.151 |
| | 4 | — | — | — | — | — | — | — | — |
| B | 1 | — | — | — | — | 16.8 | 0.1 | 15.0 | 0.03 |
| | 2 | 15.0 | 0.04 | 14.5 | 0.07 | — | — | — | — |
| | 3 | 15.2 | 0.04 | 15.0 | 0.07 | — | — | — | — |
| | 4 | — | — | — | — | 17.0 | 0.11 | 18.0 | 0.03 |
| C | 1 | 16.0 | 0.46 | 16.0 | 0.44 | 12.4 | 0.157 | 12.7 | 0.22 |
| | 2 | 13.0 | 0.015 | 12.8 | 0.015 | 12.0 | 0.025 | 11.9 | 0.02 |
| | 3 | 12.7 | 0.016 | 12.8 | 0.016 | 12.0 | 0.025 | 12.0 | 0.02 |
| | 4 | 13.0 | 0.065 | 13.0 | 0.05 | 12.0 | 0.08 | 12.0 | 0.09 |
| D | 1 | 13.0 | 0.019 | 13.0 | 0.015 | 13.0 | 0.028 | 13.0 | 0.03 |
| | 2 | 13.0 | 0.019 | 12.6 | 0.015 | 12.0 | 0.015 | 12.0 | 0.04 |
| | 3 | 12.4 | 0.009 | 12.6 | 0.015 | — | — | — | — |
| | 4 | 14.0 | 0.37 | 14.0 | 0.29 | 12.0 | 0.014 | 12.0 | 0.014 |

were reread at specified time intervals up to 36 days in order to get some indication as to the stability. The data is presented in Tables X through XII.

Two things that are immediately apparent are the excellent yield and dissipation factors obtained in all three of these runs. Concurrent with this, however, is noted a lower dielectric constant than was obtained in runs T-28, 29, and 30. The reasons for this reduction in dielectric constant are unknown at this time, but point up the fact that obtaining crystallinity and stoichiometry is not a routine accomplishment.

The aging data indicates degradation of a reasonably significant nature with respect to numbers of capacitors lost with time. It appears with microscopic observation that the aluminum-sodium niobate-chromium system is subject to a certain amount of instability with regard to chemical interaction between the dielectric and the electrodes. Free sodium may be the offending item, but this has not been systematically determined.

Runs T-50, 51, 52, and 53 (all utilizing the final capacitor geometry, Figure 4) were made with the intent of utilizing a technique which might provide the sodium a greater opportunity to oxidize and which might preclude the splattering of undissociated sodium carbonate onto the thin film. Essentially, the technique consisted of performing the "seeding" operations by feeding sodium oxide onto an electron beam heated carbon slab rather than onto the hot, but not molten, niobium sphere. The carbon was heated from beneath to prevent the possibility of carbonaceous contamination. High dielectric constants were obtained with this technique, as will be noted on the data sheets (Tables XIII through XVI); but, unfortunately, poor dissipation factors were universally obtained.

TABLE X(a). CAPACITOR DATA, INITIAL READINGS
(T-39)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-39 | | | | | | | |
| Substrate Material | | Quartz, Sapphire | | | | | | | |
| Substrate Temperature | | 380°C | | | | | | | |
| Postevaporative Bake | | 17 hr @ 450°C | | | | | | | |
| Thickness | | 5500 | | | | | | | |
| "k" (Cap. of Calculation) | | 73 (3.0 nf) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1 | 3.26 nf | 0.019 | 3.14 nf | 0.024 | 3.41 nf | 0.026 | 3.45 | 0.029 |
| | 2 | 3.22 | 0.029 | 3.06 | 0.028 | 3.35 | 0.033 | 3.40 | 0.034 |
| | 3 | 3.23 | 0.036 | 3.05 | 0.030 | 3.37 | 0.036 | 3.42 | 0.038 |
| | 4 | 3.27 | 0.032 | 3.11 | 0.029 | 3.43 | 0.030 | 3.50 | 0.033 |
| B (Quartz) | 1 | 3.26 | 0.039 | 2.96 | 0.036 | 3.51 | 0.048 | 3.50 | 0.050 |
| | 2 | 3.15 | 0.040 | 2.85 | 0.037 | 3.37 | 0.053 | 3.36 | 0.053 |
| | 3 | 3.15 | 0.075 | 2.82 | 0.047 | 3.40 | 0.050 | 3.40 | 0.050 |
| | 4 | 3.03 | 0.044 | 2.73 | 0.041 | 3.35 | 0.052 | 3.23 | 0.053 |
| C (Quartz) | 1 | 2.81 | 0.071 | 2,87 | 0.073 | 2.62 | 0.105 | 2.76 | 0.091 |
| | 2 | 3.20 | 0.072 | 3.25 | 0.068 | 2.90 | 0.110 | 3.18 | 0.084 |
| | 3 | 3.23 | 0.065 | 3.25 | 0.060 | 2.91 | 0.105 | 3.21 | 0.086 |
| | 4 | 3.31 | 0.061 | 3.37 | 0.050 | 3.00 | 0.105 | 3.21 | 0.086 |
| D (Sapphire) | 1 | 2.81 | 0.046 | 2.75 | 0.044 | 2.55 | 0.038 | 2.64 | 0.038 |
| | 2 | 3.21 | 0.037 | 3.13 | 0.036 | 2.84 | 0.033 | 3.00 | 0.038 |
| | 3 | 3.20 | 0.039 | 3.13 | 0.036 | 2.84 | 0.033 | 3.03 | 0.035 |
| | 4 | 3.24 | 0.040 | 3.20 | 0.037 | 2.94 | 0.035 | 3.12 | 0.038 |

TABLE X(b). CAPACITOR DATA, AFTER 8 DAYS STORAGE (T-39)

| Run Number | | "a" T-39 | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | 3.33 nf | 0.028 | 3.20 nf | 0.026 | ** | | | |
| | 2 | 3.28 | 0.034 | 3.14 | 0.034 | 4.25 | 0.035 | 4.26 | 0.037 |
| | 3 | 3.29 | 0.034 | 3.13 | 0.034 | | | | |
| | 4 | 3.30 | 0.032 | 3.17 | 0.030 | | | | |
| B* | 1 | | | | | | | | |
| | 2 | | | | | | | | |
| | 3 | | | | | | | | |
| | 4 | | | | | | | | |
| C | 1 | 2.94 | 0.900 | 2.97 | 0.073 | 2.57 | 0.064 | 2.75 | 0.082 |
| | 2 | 3.35 | 0.046 | 3.40 | 0.052 | 2.00 | 0.068 | 3.18 | 0.057 |
| | 3 | --- | --- | 5.10 | 0.130 | 2.93 | 0.065 | 3.20 | 0.057 |
| | 4 | 3.54 | 0.041 | 3.56 | 0.041 | 3.00 | 0.061 | 3.23 | 0.055 |
| D | 1 | 2.94 | 0.035 | 2.91 | 0.034 | 3.17 | 0.042 | 3.17 | 0.038 |
| | 2 | 3.40 | 0.034 | 3.34 | 0.033 | 3.67 | 0.039 | 3.69 | 0.034 |
| | 3 | 4.93 | 0.054 | --- | --- | --- | --- | --- | --- |
| | 4 | 3.44 | 0.035 | 3.40 | 0.034 | --- | --- | --- | --- |

* TC Test, Unavailable for readings
** Voltage Test, Destroyed

TABLE X(c). **CAPACITOR DATA AFTER 13 DAYS STORAGE**
(T-39)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-39 | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | 3.40 nf | 0.032 | 3.26 nf | 0.039 | --- | --- | --- | --- |
| | 2 | 3.35 | 0.041 | 3.21 | 0.037 | 4.34 | 0.038 | 4.35 | 0.037 |
| | 3 | 3.37 | 0.040 | 3.20 | 0.038 | --- | --- | --- | --- |
| | 4 | 3.37 | 0.035 | 3.22 | 0.034 | --- | --- | --- | --- |
| B | 1 | 20.5 | 0.65 | --- | --- | 12.7 | 0.7 | 12.7 | 0.72 |
| | 2 | 14.4 | 0.65 | 12.8 | 0.66 | 22.3 | 0.67 | --- | --- |
| | 3 | 15.7 | 0.63 | 14.2 | 0.62 | 17.6 | 0.65 | 18.4 | 0.77 |
| | 4 | 16.3 | 0.72 | 14.0 | 0.63 | 17.0 | 0.64 | 16.7 | 0.67 |
| C | 1 | 3.5 | 0.043 | 6.0 | 0.18 | 2.64 | 0.06 | 2.80 | 0.054 |
| | 2 | --- | --- | 3.52 | 0.048 | 2.94 | 0.06 | 3.21 | 0.054 |
| | 3 | --- | --- | 6.0 | 0.145 | 2.98 | 0.062 | 3.25 | 0.055 |
| | 4 | 3.7 | 0.037 | 3.74 | 0.040 | 3.06 | 0.056 | 3.28 | 0.050 |
| D | 1 | --- | --- | 6.80 | 0.17 | * --- | --- | * --- | --- |
| | 2 | 3.6 | 0.037 | 3.54 | 0.035 | * | --- | * --- | --- |
| | 3 | 6.81 | 0.081 | --- | --- | --- | --- | --- | --- |
| | 4 | 6.85 | 0.096 | --- | --- | --- | --- | --- | --- |

* Scratched

TABLE X(d). CAPACITOR DATA FTER 36 DAYS STORAGE
(T-39)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-39 | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | 3.50 nf | 0.042 | 3.37 nf | 0.041 | --- | --- | --- | --- |
| | 2 | 3.45 | 0.055 | 3.27 | 0.052 | 4.70 nf | 0.060 | --- | --- |
| | 3 | 3.47 | 0.055 | 3.25 | 0.054 | --- | --- | --- | --- |
| | 4 | 3.45 | 0.042 | 3.30 | 0.039 | --- | --- | --- | --- |
| B | 1 | | | --- | --- | --- | --- | --- | --- |
| | 2 | | | 1.25 | 0.064 | 2.40 | 0.700 | --- | --- |
| | 3 | | | 1.40 | 0.059 | 1.76 | 0.680 | 1.86 nf | 0.72 |
| | 4 | | | 1.40 | 0.062 | 1.72 | 0.640 | 1.63 | 0.64 |
| C | 1 | | | 6.00 | 0.180 | 2.80 | 0.040 | 2.90 | 0.05 |
| | 2 | | | 3.70 | 0.045 | 3.00 | 0.052 | 3.30 | 0.05 |
| | 3 | --- | --- | 6.00 | 0.130 | 3.20 | 0.053 | 3.40 | 0.046 |
| | 4 | 3.95 | 0.045 | 3.95 | 0.044 | 3.20 | 0.046 | 3.40 | 0.048 |
| D | 1 | --- | --- | 7.00 | 0.175 | --- | --- | * --- | --- |
| | 2 | 3.80 | 0.037 | 3.75 | 0.038 | * --- | --- | --- | --- |
| | 3 | 7.00 | 0.100 | --- | --- | --- | --- | --- | --- |
| | 4 | 7.00 | 0.100 | --- | --- | --- | --- | --- | --- |

*Scratched

TABLE XI(a). CAPACITOR DATA, INITIAL READINGS (T-40)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-40 | | | | | | | |
| Substrate Material | | Quartz, Sapphire | | | | | | | |
| Substrate Temperature | | 380°C | | | | | | | |
| Postevaporative Bake | | 17 hrs. at 450°C | | | | | | | |
| Thickness | | 7560 Å | | | | | | | |
| "k" (Cap. of Calculation) | | 93 (2.8) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1 | 2.45 nf | 0.038 | --- | --- | 2.33 nf | 0.033 | 2.39 nf | 0.030 |
| | 2 | 2.86 | 0.038 | --- | --- | 2.60 | 0.023 | 2.72 | 0.024 |
| | 3 | 2.86 | 0.038 | --- | --- | 2.58 | 0.023 | 2.68 | 0.024 |
| | 4 | 2.89 | 0.038 | 3.60 nf | 0.048 | 2.72 | 0.028 | 2.77 | 0.028 |
| B (Quartz) | 1 | 3.33 | 0.073 | 3.85 | 0.080 | 3.50 | 0.040 | 3.56 | 0.042 |
| | 2 | 3.49 | 0.070 | --- | --- | 3.29 | 0.048 | 3.36 | 0.038 |
| | 3 | 3.49 | 0.066 | 3.51 | 0.082 | 3.19 | 0.037 | 3.26 | 0.036 |
| | 4 | 3.38 | 0.063 | 3.37 | 0.078 | 3.00 | 0.032 | 3.05 | 0.036 |
| C (Quartz) | 1 | 2.57 | 0.027 | 2.54 | 0.022 | 2.29 | 0.022 | 2.48 | 0.024 |
| | 2 | 2.74 | 0.023 | 2.73 | 0.019 | 2.48 | 0.020 | 2.68 | 0.018 |
| | 3 | 2.74 | 0.023 | 2.73 | 0.019 | 2.51 | 0.024 | 2.72 | 0.026 |
| | 4 | 2.80 | 0.027 | 2.80 | 0.020 | 2.54 | 0.025 | 2.73 | 0.024 |
| D (Sapphire) | 1 | --- | --- | --- | 0.095 | 3.20 | 0.037 | 3.27 | 0.021 |
| | 2 | 3.45 | 0.028 | --- | 0.024 | 3.20 | 0.037 | 3.30 | 0.024 |
| | 3 | 3.45 | 0.028 | --- | 0.024 | 3.26 | 0.026 | 3.30 | 0.024 |
| | 4 | 3.49 | 0.028 | --- | 0.024 | 3.36 | 0.028 | 3.38 | 0.025 |

TABLE XI(b). **CAPACITOR DATA AFTER 7 DAY STORAGE**
(T-40)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-40 | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | --- | --- | --- | --- | --- | --- | 5.10 nf | .040 |
| | 2 | --- | --- | --- | --- | 2.96 nf | .031 | 3.05 | .032 |
| | 3 | --- | --- | --- | --- | 2.94 | .030 | 3.0 | .033 |
| | 4 | --- | --- | --- | --- | --- | --- | 5.1 | .039 |
| B | 1 | 3.94 nf | .075 | 3.95 | .081 | --- | --- | 5.40 | .048 |
| | 2 | --- | --- | --- | --- | 3.51 | .045 | 3.58 | .043 |
| | 3 | 3.57 | .073 | 3.55 | .082 | 3.40 | .044 | 3.48 | .040 |
| | 4 | 3.46 | .070 | 3.45 | .083 | 3.18 | .046 | 3.23 | .044 |
| C | 1 | 3.00 | .050 | 3.01 | .050 | --- | --- | --- | --- |
| | 2 | 5.72 | .24 | --- | --- | 5.02 | .075 | 5.05 | .075 |
| | 3 | 5.80 | .22 | --- | --- | 2.96 | .034 | 2.96 | .034 |
| | 4 | 3.26 | .049 | 3.27 | .052 | 2.96 | .034 | 2.96 | .034 |
| D | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | 4.10 | .067 | 4.10 | .030 |
| | 3 | 4.13 | .038 | 4.13 | .035 | 4.17 | .070 | 4.19 | .026 |
| | 4 | --- | --- | --- | --- | 4.28 | .072 | 4.30 | .030 |

TABLE XI(c). CAPACITOR DATA AFTER 35 DAYS STORAGE
(T-40)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-40 | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | --- | --- | --- | --- | --- | --- | 3.4 nf | 0.045 |
| | 2 | --- | --- | --- | --- | 3.2 nf | 0.036 | 3.2 | 0.034 |
| | 3 | --- | --- | --- | --- | 3.2 | 0.038 | 3.2 | 0.038 |
| | 4 | --- | --- | --- | --- | --- | --- | 5.4 | 0.042 |
| B | 1 | 4.2 nf | 0.082 | 4.1 nf | 0.087 | --- | --- | 5.6 nf | 0.054 |
| | 2 | --- | --- | --- | --- | 3.7 | 0.053 | 3.7 | 0.046 |
| | 3 | 3.7 | 0.075 | 3.7 | 0.092 | 3.5 | 0.048 | 3.6 | 0.049 |
| | 4 | 3.6 | 0.072 | 3.5 | 0.085 | 3.3 | 0.046 | 3.3 | 0.046 |
| C | 1 | 3.2 nf | 0.044 | 3.2 | 0.038 | --- | --- | --- | --- |
| | 2 | 6.8 | 0.08 | --- | --- | 5.4 | 0.085 | --- | --- |
| | 3 | 6.8 | 0.057 | --- | --- | 3.2 | 0.039 | 3.3 | 0.04 |
| | 4 | 6.7 | 0.054 | | | 3.2 | 0.039 | 3.3 | 0.04 |
| D | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | 4.2 nf | 0.032 |
| | 3 | 4.2 | 0.038 | 4.3 nf | 0.04 | --- | --- | 4.3 | 0.031 |
| | 4 | --- | --- | --- | --- | --- | --- | 4.4 | 0.030 |

TABLE XII(a). **CAPACITOR DATA, INITIAL READINGS**
(T-41)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-41 | | | | | | | |
| Substrate Material | | Quartz, Sapphire | | | | | | | |
| Substrate Temperature | | 380° | | | | | | | |
| Postevaporative Bake | | 17 hr. @ 450°C | | | | | | | |
| Thickness | | 1600 A° | | | | | | | |
| "k" (Cap. of Calculation) | | 34 (4.8 nf) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1 | 4.75 nf | .045 | 4.64 | .036 | --- | --- | 5.62 | .041 |
| | 2 | 4.55 | .042 | 4.39 | .037 | 4.48 | .056 | 4.62 | .056 |
| | 3 | 4.50 | .042 | 4.39 | .037 | 6.62 | .150 | --- | --- |
| | 4 | --- | --- | 6.50 | .061 | 4.13 | .057 | 4.23 | .060 |
| B (Quartz) | 1 | 4.48 | .032 | 4.37 | .028 | 5.5 | .025 | 5.80 | .027 |
| | 2 | 4.22 | .024 | 4.07 | .026 | 5.2 | .028 | 5.19 | .027 |
| | 3 | --- | --- | 6.15 | .033 | 5.2 | .028 | 5.19 | .027 |
| | 4 | 4.30 | .036 | 4.14 | .034 | --- | --- | --- | --- |
| C (Quartz) | 1 | 3.63 | .043 | 3.52 | .043 | 3.70 | .075 | 3.76 | .074 |
| | 2 | 4.12 | .039 | 3.92 | .035 | 4.18 | .066 | 4.33 | .066 |
| | 3 | 4.12 | .039 | 3.96 | .039 | 4.30 | .063 | 4.40 | .059 |
| | 4 | 4.27 | .043 | 4.06 | .041 | 4.38 | .063 | 4.50 | .065 |
| D (Sapphire) | 1 | 3.86 | .105 | 6.31 | .96 | --- | --- | --- | --- |
| | 2 | 4.55 | .097 | 6.31 | .96 | 5.39 | .028 | 5.37 | .024 |
| | 3 | 4.55 | .097 | 6.31 | .96 | 5.29 | .031 | 5.28 | .031 |
| | 4 | 5.95 | 1.05 | --- | --- | --- | --- | --- | --- |

TABLE XII(b). CAPACITOR DATA AFTER 7 DAYS STORAGE
(T-41)

| Run Number | | "a" T-41 | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 3 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 4 | --- | --- | --- | --- | --- | --- | --- | --- |
| B | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 3 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 4 | --- | --- | --- | --- | 4.91 | .046 | 4.93 | .043 |
| C | 1 | 3.66 | .036 | 3.58 | .034 | --- | --- | --- | --- |
| | 2 | 4.16 | .030 | 4.01 | .031 | --- | --- | --- | --- |
| | 3 | 4.17 | .031 | 4.01 | .031 | 5.60 | .035 | 5.60 | .036 |
| | 4 | 4.25 | .032 | 4.10 | .030 | 5.69 | .034 | 5.69 | .034 |
| D | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 3 | 5.10 | .030 | 5.10 | .032 | --- | --- | --- | --- |
| | 4 | --- | --- | --- | --- | --- | --- | --- | --- |

TABLE XII(c). **CAPACITOR DATA AFTER 35 DAYS STORAGE**
(T-41)

| | | "a" | | "b" | | "c" | | "d" | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-41 | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| **Substrate** | **Capacitor** | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 3 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 4 | --- | --- | --- | --- | --- | --- | --- | --- |
| B | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 3 | --- | --- | --- | --- | 7.6 nf | 4.2 | --- | --- |
| | 4 | --- | --- | --- | --- | 5 | 0.038 | 5 nf | 0.038 |
| C | 1 | 3.6 nf | 0.031 | 3.6 nf | 0.032 | --- | --- | --- | --- |
| | 2 | 4.2 | 0.028 | 4.0 | 0.027 | --- | --- | --- | --- |
| | 3 | 4.2 | 0.029 | 4.0 | 0.027 | 5.5 nf | 0.034 | 5.5 nf | 0.03 |
| | 4 | 4.3 | 0.03 | 4.1 | 0.026 | 5.6 | 0.032 | 5.6 | 0.032 |
| D | 1 | --- | --- | --- | --- | --- | --- | --- | --- |
| | 2 | --- | --- | --- | --- | 5.8 nf | 0.2 | --- | --- |
| | 3 | 5.2 nf | 0.032 | 5.2 | 0.032 | 5.6 | 0.3 | --- | --- |
| | 4 | --- | --- | --- | --- | --- | --- | --- | --- |

TABLE XIII. CAPACITOR DATA, INITIAL READINGS
(T-50)

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-50 | | | | | | | |
| Substrate Material | | Quartz | | | | | | | |
| Substrate Temperature | | 350°C | | | | | | | |
| Post Evaporative Bake | | 16 hrs in air @450°C | | | | | | | |
| Thickness | | 1500 A° | | | | | | | |
| "k" (Cap. of Calculation) | | 220 (25 nf) | | | | | | | |
| **Substrate** | **Capacitor** | **Cap.** | **D.F.** | **Cap.** | **D.F.** | **Cap.** | **D.F.** | **Cap.** | **D.F.** |
| A (Sapphire) | 1-5-9-13 | 7.7nf | 1.3 | --- | --- | --- | --- | 27.7 | .73 |
| | 2-6-10-14 | 8.9 | 1.3 | --- | --- | --- | --- | 25.6 | .35 |
| | 3-7-11-15 | 8.9 | 1.5 | --- | --- | 24.2 | .39 | | |
| | 4-8-12-16 | 6.6 | 2.0 | --- | --- | 22.8 | .46 | 23.2 | .36 |
| B (Quartz) | 1-5-9-13 | 4.8nf | .32 | 9.5 | .71 | --- | --- | 5.1 | 1.3 |
| | 2-6-10-14 | 4.67 | .33 | 21.4 | .50 | --- | --- | 16.7 | .50 |
| | 3-7-11-15 | 5.0 | .36 | --- | --- | 4.28 | 1.4 | --- | --- |
| | 4-8-12-16 | --- | --- | --- | --- | 4.98 | .56 | --- | --- |
| C (Quartz) | 1-5-9-13 | 28.6 | .32 | 28.1 | .37 | --- | --- | 31.7 | .36 |
| | 2-6-10-14 | 30.1 | .32 | 29.3 | .36 | --- | --- | 29.1 | .35 |
| | 3-7-11-15 | 29.0 | .35 | --- | --- | 29.3 | .33 | --- | --- |
| | 4-8-12-16 | 24.7 | .40 | --- | --- | 29.5 | .33 | 30.7 | .35 |
| D (Sapphire) | 1-5-9-13 | 21.7 | .45 | 8.8 | 1.7 | --- | --- | --- | --- |
| | 2-6-10-14 | 25.2 | .55 | 8.8 | 1.6 | --- | --- | 6.0 | 2.3 |
| | 3-7-11-15 | 26.6 | .94 | --- | --- | 31.5 | .80 | --- | --- |
| | 4-8-12-16 | 10.3 | 1.2 | --- | --- | 28.0 | .41 | 12.4 | 1.5 |

TABLE XIV. CAPACITOR DATA, INITIAL READINGS (T-51)

| Run Number | | T-51 | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Substrate Material | | Quartz | | | | | | | |
| Substrate Temperature | | 350° | | | | | | | |
| Postevaporative Bake | | 16 hr. @ 450° C | | | | | | | |
| Thickness | | 1750 A° | | | | | | | |
| "k" (Cap. of Calculation) | | 305 (30) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1-5-9-13 | 40.5 nf | .41 | 36.7 | .40 | 59.8 | .56 | 36.0 | .40 |
| | 2-6-10-14 | 21.2 | .36 | 39.6 | .44 | 68.0 | .53 | 47.0 | .49 |
| | 3-7-11-15 | 28.0 | .38 | 49.5 | .48 | 59.0 | .54 | 56.0 | .57 |
| | 4-8-12-16 | 41.5 | .49 | 55.5 | .57 | 52.5 | .49 | 63.5 | .79 |
| B (Quartz) | 1-5-9-13 | 33.2 nf | .24 | 35.5 | .26 | 36.3 | .27 | 32.7 | .29 |
| | 2-6-10-14 | 30.3 | .29 | 31.7 | .32 | 36.2 | .31 | 35.8 | .29 |
| | 3-7-11-15 | 26.6 | .28 | 31.2 | .31 | 36.8 | .28 | 40.2 | .28 |
| | 4-8-12-16 | 39.2 | .19 | 31.1 | .29 | 34.8 | .29 | 42.8 | .28 |
| C (Quartz) | 1-5-9-13 | 28.0 | .20 | 14.2 | .30 | 14.9 | .30 | 14.9 | .24 |
| | 2-6-10-14 | 26.4 | .19 | 8.9 | .28 | 15.5 | .28 | 13.2 | .31 |
| | 3-7-11-15 | 9.7 | .23 | 9.2 | .23 | 21.2 | .27 | 14.2 | .28 |
| | 4-8-12-16 | 23.8 | .20 | 9.4 | .27 | 25.3 | .24 | 22.1 | .26 |
| D (Sapphire) | 1-5-9-13 | 38.2 | .41 | 89.0 | .62 | 30.7 | .48 | 97.0 | .70 |
| | 2-6-10-14 | 80.5 | .57 | --- | --- | 20. | .34 | 53.2 | .73 |
| | 3-7-11-15 | 89.0 | .50 | 34.8 | .48 | 25.3 | .40 | 67. | .78 |
| | 4-8-12-16 | 81.0 | .50 | 39.0 | .53 | 28.5 | .44 | 75.0 | .63 |

TABLE XV. CAPACITOR DATA, INITIAL READINGS
(T-52)

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Run Number | | T-52 | | | | | | | |
| Substrate Material | | Quartz | | | | | | | |
| Substrate Temperature | | $350^{o}$ | | | | | | | |
| Postevaporative Bake | | 16 hr. @ $450^{o}C$ | | | | | | | |
| Thickness | | 1800 $A^{o}$ | | | | | | | |
| "k" (Cap. of Calculation) | | 136 (13) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1-5-9-13 | 13.8 | .23 | 13.6 | .18 | 12.3 | .17 | 13.9 | .19 |
| | 2-6-10-14 | 14.4 | .20 | 15.0 | .19 | 11.6 | .16 | 16.3 | .18 |
| | 3-7-11-15 | 13.6 | .20 | 11.7 | .16 | 10.7 | .15 | 12.7 | .17 |
| | 4-8-12-16 | 14.1 | .18 | 13.6 | .18 | 14.1 | .20 | 14.1 | .17 |
| B (Quartz) | 1-5-9-13 | 27.2 | .34 | 27.2 | .33 | 23.2 | .36 | 27.0 | .34 |
| | 2-6-10-14 | 24.3 | .32 | 22.1 | .32 | 22.0 | .35 | 33.0 | .38 |
| | 3-7-11-15 | 25.7 | .31 | 25.7 | .31 | 20.3 | .34 | 21.6 | .36 |
| | 4-8-12-16 | 20.8 | .31 | 22.2 | .32 | 23.3 | .36 | 25.7 | .38 |
| C (Quartz) | 1-5-9-13 | 16.0 | .33 | --- | --- | 21.1 | .36 | 13.4 | .30 |
| | 2-6-10-14 | 15.3 | .33 | 18.4 | .33 | 15.8 | .31 | 14.5 | .30 |
| | 3-7-11-15 | 16.6 | .33 | 10.6 | .31 | 17.5 | .32 | 15.6 | .34 |
| | 4-8-12-16 | --- | --- | 12.8 | .31 | 19.3 | .33 | 11.8 | .29 |
| D (Sapphire) | 1-5-9-13 | 12.8 nf | .15 | 11.1 | .16 | 13.0 | .17 | 18.2 | .32 |
| | 2-6-10-14 | 15.2 | .18 | --- | --- | 14.9 | .16 | 14.4 | .17 |
| | 3-7-11-15 | 18.1 | .19 | 11.0 | .17 | 15.7 | .17 | 15.2 | .18 |
| | 4-8-12-16 | 11.6 | .17 | 13.5 | .18 | 22.7 | .70 | 19.2 | .19 |

TABLE XVI. CAPACITOR DATA, INITIAL READINGS
(T-53)

| Run Number | | T-53 | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Substrate Material | | Quartz | | | | | | | |
| Substrate Temperature | | 350° | | | | | | | |
| Postevaporative Bake | | 16 hrs. at 450°C | | | | | | | |
| Thickness | | 2430 A° | | | | | | | |
| "k" (Cap. of Calculation) | | 490 (35) | | | | | | | |
| Substrate | Capacitor | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. | Cap. | D.F. |
| A (Sapphire) | 1-5-9-13 | --- | --- | 33.9 | .97 | 37.5 | 1.4 | 28.0 | .68 |
| | 2-6-10-14 | 40.5nf | 1.5 | 19.8 | 1.9 | 39.8 | 1.1 | 22.0 | .88 |
| | 3-7-11-15 | 23.2 | 1.3 | 31.2 | 1.5 | 35.1 | 1.9 | 30.0 | 1.1 |
| | 4-8-12-16 | 23.3 | 1.3 | 25.3 | 1.1 | 41.5 | 1.9 | 32.5 | 1.8 |
| B (Quartz) | 1-5-9-13 | 38.6 | 1.1 | 38.0 | 1.1 | 39.0 | 1.5 | 50.5 | 1.8 |
| | 2-6-10-14 | 42.2 | 1.9 | 34.4 | 1.1 | 37.0 | 1.1 | 54.5 | .80 |
| | 3-7-11-15 | 46.0 | 1.1 | 32.8 | 1.3 | 42.3 | 1.3 | 50.0 | .86 |
| | 4-8-12-16 | 38.2 | 1.0 | 40.7 | 1.1 | 40.1 | 1.7 | 50.5 | 1.5 |
| C (Quartz) | 1-5-9-13 | 32.4 | .69 | 24.8 | .62 | 19.7 | .35 | --- | --- |
| | 2-6-10-14 | 30.6 | .63 | 20.2 | .44 | 19.9 | .40 | 28.7 | .60 |
| | 3-7-11-15 | 21.5 | .52 | 28.5 | .68 | 21.7 | .41 | --- | --- |
| | 4-8-12-16 | 20.0 | .38 | 23.7 | .45 | 24.6 | .50 | 24.5 | .47 |
| D (Sapphire) | 1-5-9-13 | 22.1 | .95 ad | 12.8 | 1.1 | 29.6 | 1.1 | 31.5 | 1.0 ad |
| | 2-6-10-14 | 51.5 | .98 | 17.8 | .85 ad | 21.9 | 1.2 | 15.2 | 1.1 |
| | 3-7-11-15 | 68.0 | 1.15 ad | 25.0 | .90 ad | 30.5 | 1.1 | 54.0 | .80 |
| | 4-8-12-16 | 19.5 | 1.2 | 25.2 | .95 ad | 26.7 | 1.2 | 65.5 | .88 |

## V. CAPACITOR CHARACTERIZATION

### ELECTRICAL

The deposited capacitors were all read initially for capacity and dissipation factor and had the thicknesses of the dielectric films determined by utilization of interferrometric techniques. This thickness was then used to calculate the dielectric constants.

Temperature coefficients of capacitance were run on selected samples throughout the course of Phase 2. Typical temperature coefficient curves obtained in the latter stages of Phase 2 are as shown in Figures 14.

Voltage breakdown tests were also performed on selected samples, and in general showed voltage capability only to about 3 or 4 volts (Figure 15).

### PHYSICAL

Spectrographic analysis, optical and electron microscopy, and X-ray diffraction techniques were all utilized to characterize the physical, chemical, and crystallographic makeup of the sodium-niobium-oxygen complex.

Utilizing X-ray diffraction, employing sodium niobate Standard No. 14-603 (Table XVII) and a conversion chart of d-spacings to degrees (Table XVIII), the presence of orthorhombic crystalline sodium niobate in the "Molten Sphere" evaporated films was established. Tape traces of the diffraction pattern (Figure 16) are representative of numerous films discovered. The ratio between relative intensities of the (004) reflection and the (200) reflection in the diffraction pattern provides a measure of the preferred orientation within the thin film.

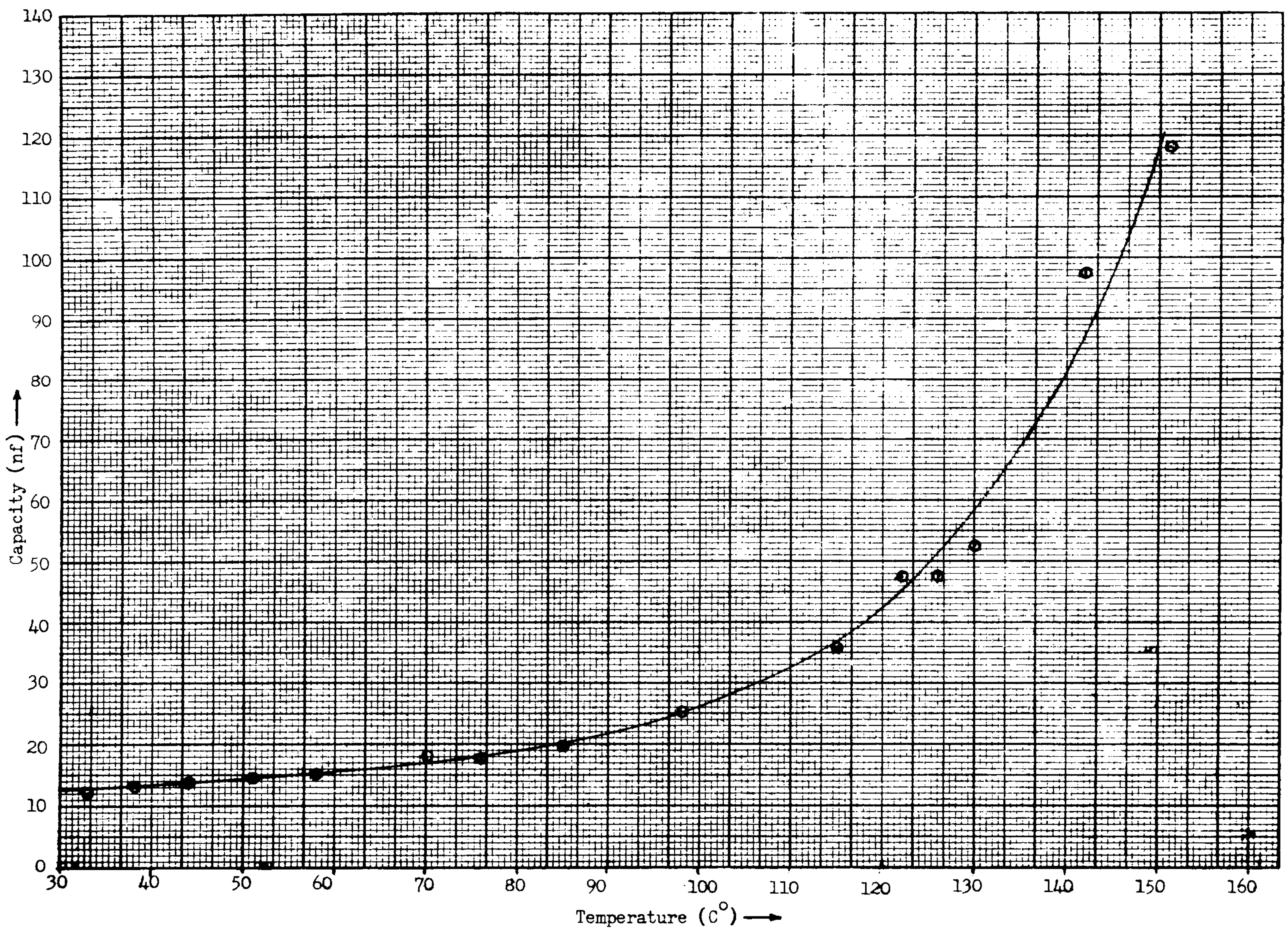


Figure 14. Capacity vs. Temperature
Run T29A, Sample (D-3)

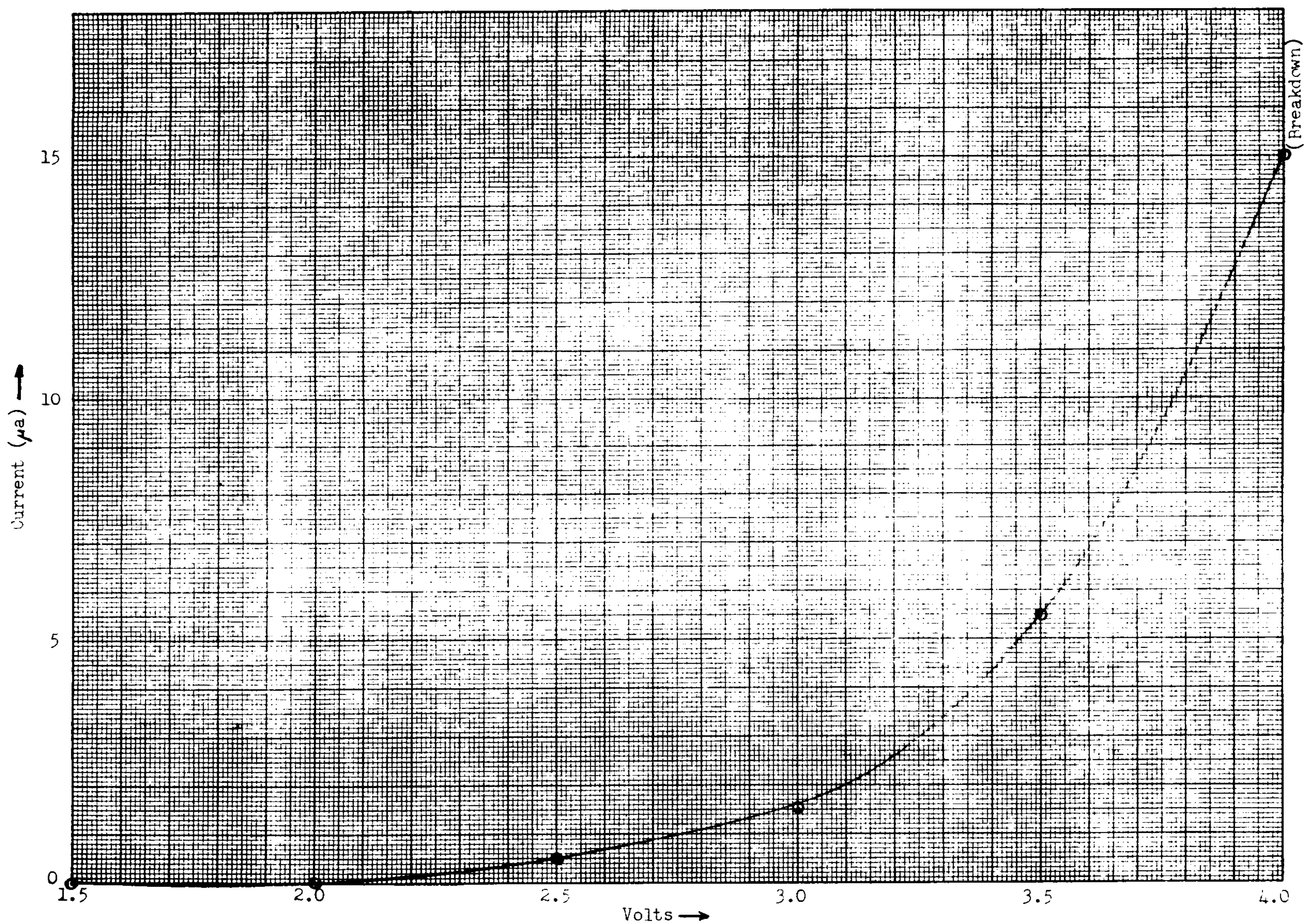


**Figure 15.** Current vs. Voltage
Run HD31, Sample B-3

**14-603**

| d | 2.74 | 1.58 | 3.93 | 3.93 | $NaNbO_3$ (AT 25$^{0}$C) |
|---|---|---|---|---|---|
| $I/I_1$ | 100 | 90+ | 90 | 90 | SODIUM NIOBATE (LUESHITE) |

Rad. $CuK\alpha_1$ $\lambda$ 1.54050 Filter Ni Dia. 114.6MM
Cut off $I/I_1$ VISUAL INSPECTION
Ref. E. WOOD, BELL TELEPHONE LABORATORIES, MURRAY HILL, NEW JERSEY

Sys. *ORTHORHOMBIC S.G. $P222_1$ (17)**
$a_0$ 5.512 $b_0$ 5.557 $c_0$ 4x3.885 A C
$\alpha$ $\beta$ $\gamma$ Z 8 Dx
Ref. WOOD, ACTA CRYST. 4 353-362 (1951)

$\epsilon\alpha$ $n\omega\beta$ 2.30** $\epsilon\gamma$ Sign
2V D 4.44** mp Color CLEAR, COLORLESS BUT TURNS TAN ON EXPOSURE TO LIGHT
Ref.

*PSEUDOTETRAGONAL: Z=16, $A_0$=2x3.921, $C_0$=4x3.885
*MONOCLINIC: $A_0$ AND $C_0$=2x3.921, $B_0$=4x3.885, $\beta$=90°40'; CHANGES TO TETRAGONAL FORM AT ABOUT 370°; TO CUBIC AT ABOUT 640°C.
**SAFIANNIKOFF, BULL. ACAD. ROY. SOC. COL. BELGES 5 1251 (1959)

| d Å | $I/I_1$ | hkl | d Å | $I/I_1$ | hkl |
|---|---|---|---|---|---|
| 3.93 | 90 | 110 | 1.523 | 15 | INDEXED BY LGB |
| 3.85 | 90 | 004 | 1.389 | 40 | |
| 2.77 | 90 | 114 | 1.377 | 80 | |
| 2.74 | 100 | 021(200?) | 1.302 | 50 | |
| 2.59 | 5 | 202,006 | 1.295 | 60 | |
| 2.44 | 20 | 211,115+ | 1.265 | 10 | |
| 2.37 | 10 | 122 | 1.242 | 20 | |
| 2.34 | 10 | 016,106 | 1.236 | 70 | |
| 2.25 | 20 | 204 | | | |
| 2.24 | 20 | 123,213 | | | |
| 1.96 | 80 | 220 | | | |
| 1.94 | 60 | 008 | | | |
| 1.78 | 5 | - | | | |
| 1.75 | 50 | 224 | | | |
| 1.74 | 90 | 310 | | | |
| 1.66 | 20 | 304 | | | |
| 1.653 | 5 | - | | | |
| 1.598 | 50 | 134,028 | | | |
| 1.583 | 90+ | 208 | | | |
| 1.537 | 5 | 230,320+ | | | |

TABLE XVII. SODIUM NIOBATE STANDARD

TABLE XVIII. d(Å) vs. 2θ for $NaNbO_3$ at 25°C

| d(Å) | 2θ (degrees) |
|---|---|
| 3.93 | 22.62 |
| 3.85 | 23.08 |
| 2.77 | 32.3 |
| 2.74 | 32.66 |
| 2.59 | 34.62 |
| 2.44 | 36.80 |
| 2.37 | 37.94 |
| 2.34 | 38.44 |
| 2.25 | 40.04 |
| 2.24 | 40.22 |
| 1.96 | 46.28 |
| 1.94 | 46.78 |
| 1.78 | 51.28 |
| 1.75 | 52.24 |
| 1.74 | 52.56 |
| 1.66 | 55.30 |
| 1.653 | 55.56 |
| 1.598 | 57.64 |
| 1.583 | 58.24 |
| 1.537 | 60.40 |
| 1.523 | 60.76 |
| 1.389 | 67.36 |
| 1.377 | 68.02 |
| 1.302 | 72.54 |
| 1.295 | 73.00 |
| 1.265 | 75.02 |
| 1.242 | 76.66 |
| 1.236 | 77.10 |

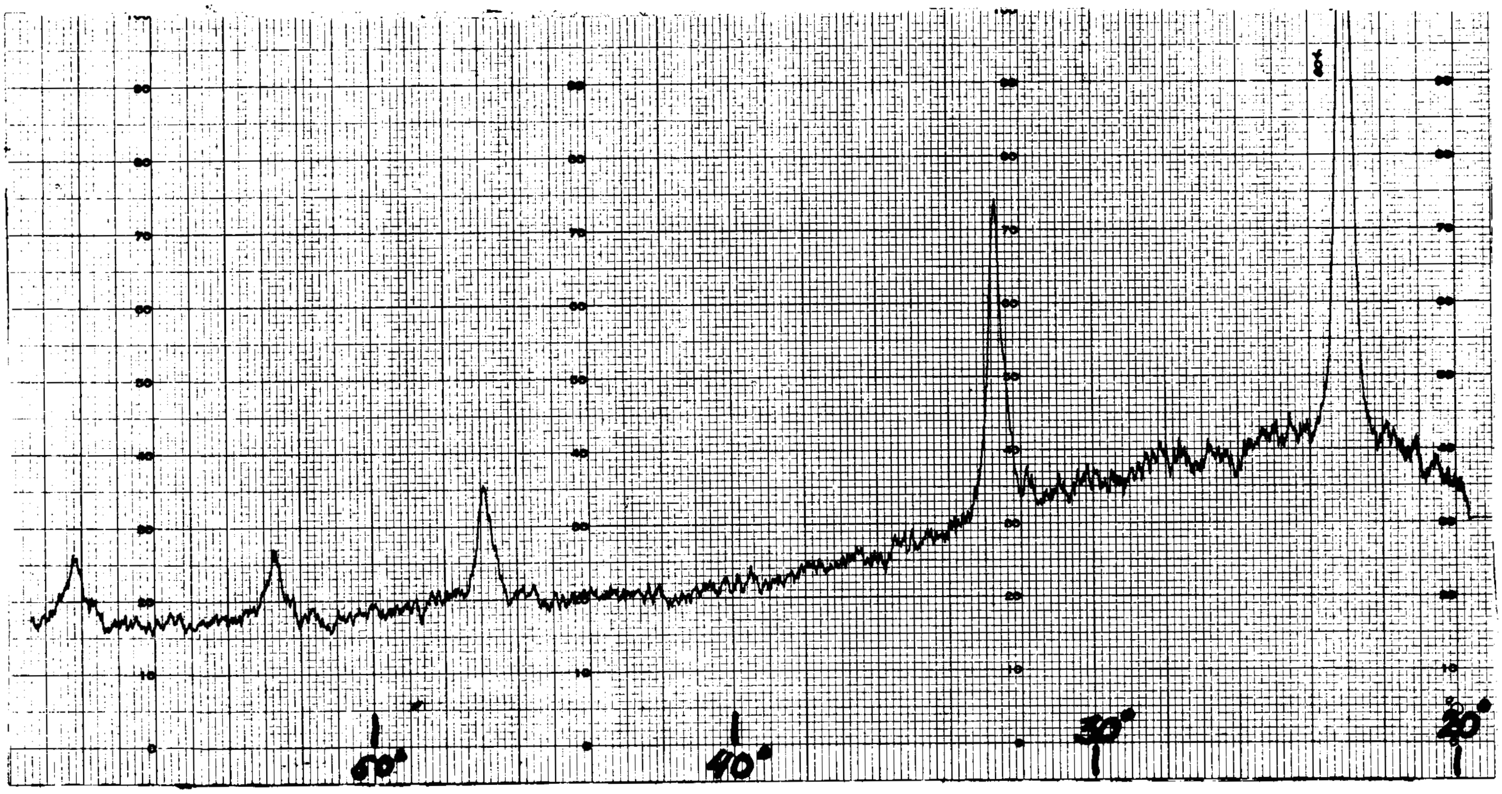


Figure 16. Diffraction Pattern
Tape Traces

## VI. CONCLUSIONS

The "Molten Sphere" deposition approach has been shown to be one which holds high potential for successful fabrication of high dielectric constant thin film capacitors of sodium niobate. The technique is especially advantageous from the standpoint of eliminating contamination from a refractory metal source, a problem which has persistently plagued investigators attempting to evaporate compounds of the $ABO_3$ type.

Good yield factors of $NaNbO_3$ capacitors deposited via this approach have been obtained, but their long-range stability is open to question. Dissipation factors have been variable, ranging from good (1-10%) to questionable (10-25%). Dielectric constants have ranged from 50-150. While this does not approach the figure of 500-600 obtained with pure, single-crystalline and preferentially oriented bulk material, it nevertheless represents a significant improvement in the specific capacitance obtainable with vacuum evaporated capacitors. The data obtained to date regarding temperature coefficient of capacitance is not conclusive, but does indicate that reasonable flatness of temperature response is obtained up to about 100°C. Voltage breakdown characteristics remain as the most notable deficiency at the present time. Breakdowns in excess of 5 volts DC have been obtained only infrequently.

One primary area of concern at the present time involves the selection of appropriate base and counter electrode material. Nichrome, gold, titanium, aluminum, and chromium have all been attempted for one or the other or both electrodes during the Phase 2 investigation, but the necessity for frequent modification of the dielectric deposition parameters precluded the possibility of obtaining well integrated and conclusive data regarding electrode material. The behavior of the base electrode during the postevaporative bake cycle and the chemical interaction between both electrodes and the dielectric are extremely complex and important considerations, and undoubtedly have a significant effect on the dissipation factors and voltage breakdown characteristics.

An important deficiency during the Phase 2 investigation had been the lack of control of the powder being fed onto the molten sphere of niobium. Frequent "dumpings" of large amounts of the powder caused relatively violent eruptions. As a result of this, particulate matter was injected into the thin film on the substrate, causing undesirable roughness. The implications of this with regard to voltage breakdown characteristics is obvious. To correct this deficiency, Bendix Balzer's vibratory feeder unit, Model No. C-20460, has been obtained and is presently being installed in the vacuum system. It is felt that this unit will alleviate the problem to a very large extent, if not entirely.

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