RADC-TDR-64-359

FAILURE MECHANISMS AT METAL-DIELECTRIC INTERFACES

TECHNICAL DOCUMENTARY REPORT NO. RADC-TDR-64-359

October 1964

Reliability Branch
Rome Air Development Center
Research and Technology Division
Air Force Systems Command
Griffiss Air Force Base, New York

Project No. 5519, Task No. 551902

(Prepared under Contract No. AF30(602)-3266 by Motorola, Inc., Semiconductor Products Division, 5005 East McDowell Road, Phoenix, Arizona. Prepared by: D. Peterson.)

ABSTRACT

The interface between a metal and a dielectric is representative of the type of interface found in microelectronic circuitry.

Capacitors using thin film dielectrics have been used as a means of studying the changes induced in a metal-dielectric interface by electrical and thermal stress.

Thin film capacitors which were exposed to 100 percent R.H. before top electrode evaporation showed larger changes under stress than similar units which were heated prior to electrode deposition.

In general, all capacitors decreased in capacitance, dissipation factor and T.C.C. after stressing.

Thin film capacitors stressed at 80 percent R.H. and 40 volts d.c. showed only minor changes in electrical properties.

High temperature stress studies of thin film capacitors have been initiated.

Thin film anodized Ta_2O_5 capacitors have shown little change in electrical properties under low temperature stress. Ta_2O_5 capacitors have been prepared for radioactive tracer studies, and will be stressed at higher levels than the first group.

Preliminary study indicates that radioactive tracer techniques may be used in the study of diffusion occurring at the metal-dielectric interfaces, where anodized Ta_2O_5, SiO_2 thermally grown on a silicon metal, and gas plated boro alumina silicate glass film are the dielectrics.

PUBLICATION REVIEW

This report has been reviewed and is approved. For further technical information on this project, contact Mr. Vincent C. Kapfer, EMERP, X 5168

Approved: Vincent C. Kapfer
VINCENT C. KAPFER
Reliability Physics Section
Reliability Branch

Approved: Milton Hans
D. F. BARBER
Chief, Reliability Branch
Engineering Division

AIR FORCE, GAFB, N.Y., 25 NOV 64-177

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1.0 INTRODUCTION

The interface between a metal and a dielectric material is a common feature of microelectronic circuits occurring in capacitors, and between deposited conductors and resistors and a passive substrate. The stability of such interfaces will determine to a large extent the overall reliability of the system in which such interfaces are contained.

By fabricating capacitors from very thin dielectric layers it is possible to maximize the effect of the metal dielectric interface and obtain a structure amenable to electrical characterization. Optical and electron microscopy and radioactive tracer studies will be utilized to supplement the information obtained from the capacitor measurements. Correlation of the information obtained from these studies will permit identification of the modes of failure at metal-dielectric interfaces and the electrical characteristics that are indicative of the probability of such failures occurring.

2.1.0 Technical Discussion

The interface between a metal and a dielectric is typical of the type of interface found in microelectronic circuitry. The characteristics of such interfaces will to a large extent determine the overall reliability of the circuitry.

The goal of this program is to study the properties and identify the modes of failure of the metal - dielectric interfaces.

Capacitors fabricated of thin dielectric films are a convenient tool for the study of the characteristics of metal-

dielectric interfaces, since the effect of the interfacial region may be maximized by using a very thin dielectric film.

The metal-dielectric systems to be investigated in this program are typical of those found in current microelectronic circuits.

2.1.1 Gas Plated Dielectric Capacitor Stress Test

A technique for the deposition of glass-like dielectrics has been developed and is finding application as a capacitor dielectric and a means of passivating the surface of active devices and thin film circuitry.

By means of this technique a series of capacitors were fabricated to evaluate the effect of various parameters of preparation. This series, (Test group #1 reported in first technical report), has now accumulated 1200 hours of stress test time.

The experimental variables of this test group are: electrode metal, stress condition and the conditions of capacitor preparation. For each processing parameter, i.e. exposure of the dielectric to 100% relative humidity for three days prior to electroding, four units of three capacitors each were prepared. Two of the units were provided with a gold top electrode and the remaining two were electroded with aluminum. Out of each pair of similar units one was placed on stress at 125°C and the other at 125°C 40 volts d. c.. The electrical stress was applied in such a direction as to make the top electrode positive. For the dielectric thickness used in these capacitors the applied electrical stress amounted to approximately 3×10^6 volts/cm.

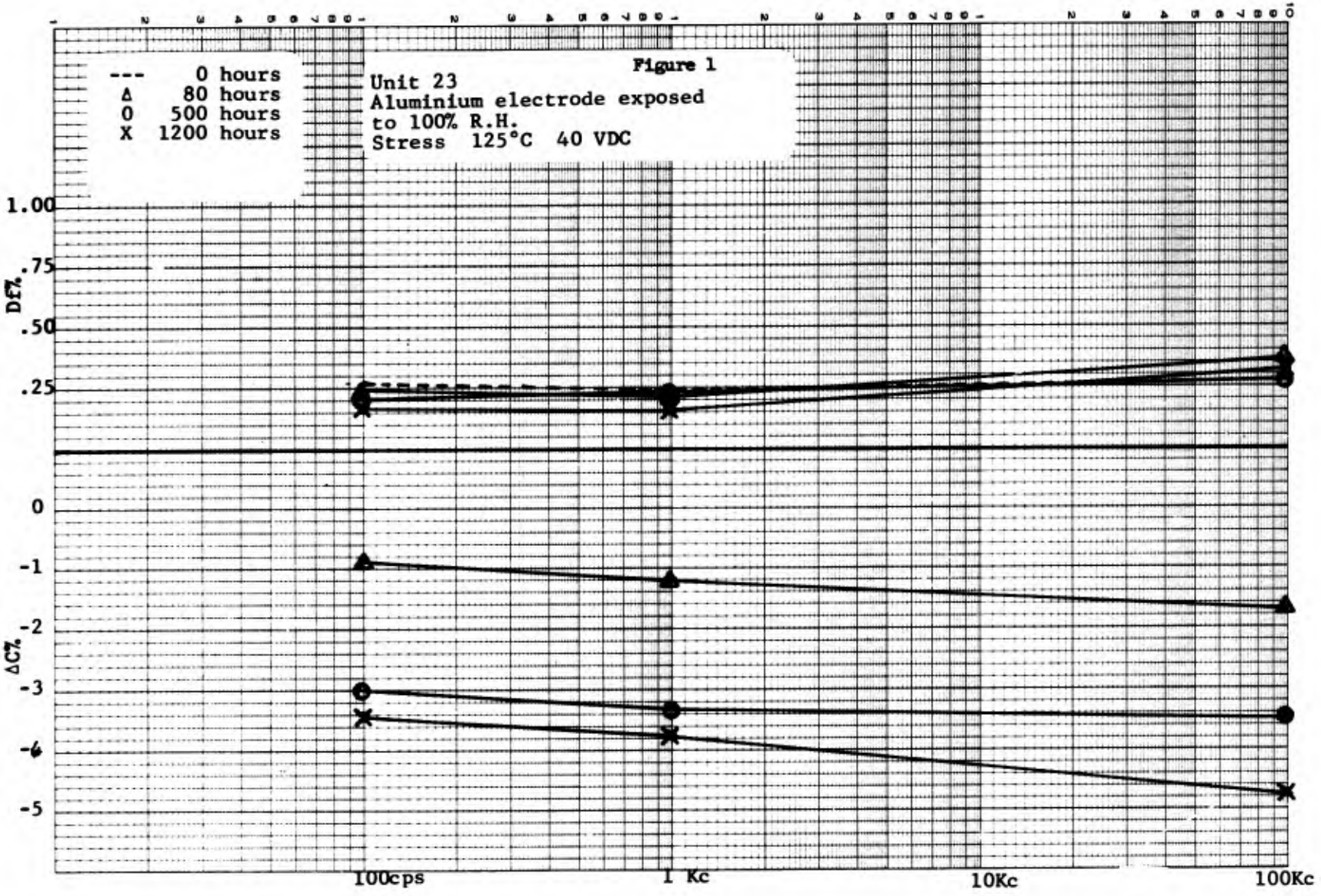

--- 0 hours
Δ 80 hours
0 500 hours
X 1200 hours
Unit 23
Aluminium electrode exposed
to 100% R.H.
Stress 125°C 40 VDC
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
D.F.%
ΔC%
100cps
1 Kc
10Kc
100Kc

Figure 1

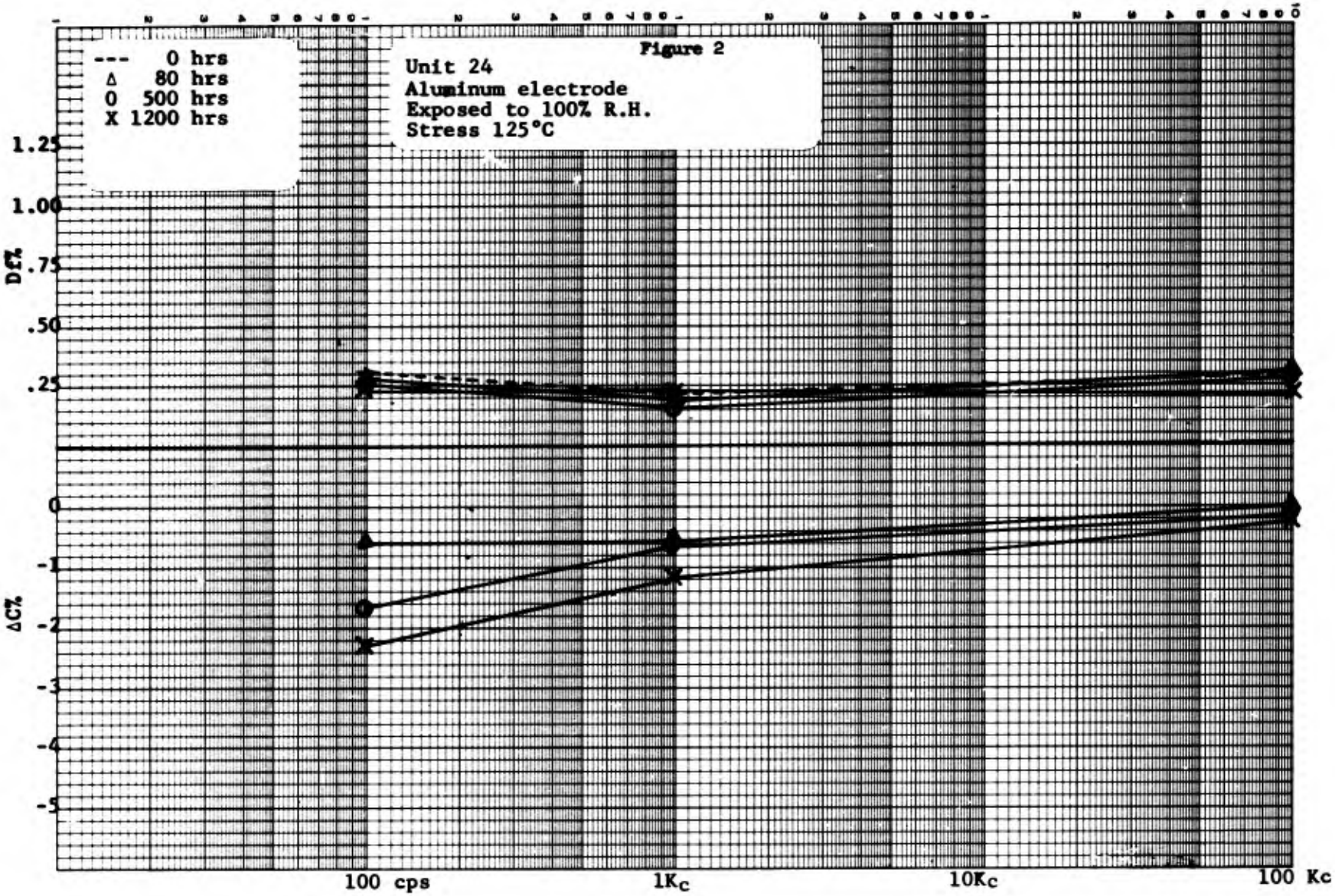
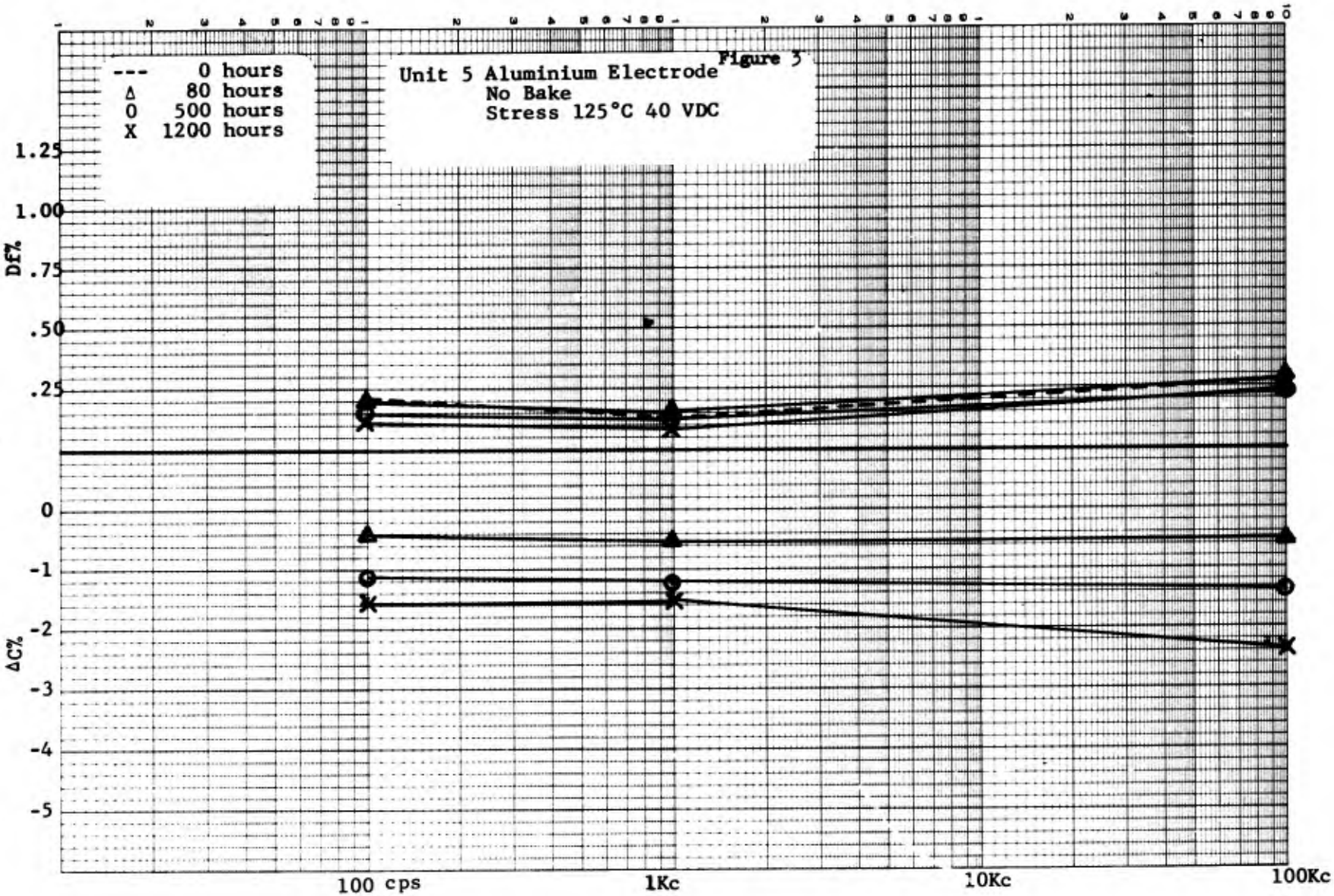


Figure 2
--- 0 hrs
Δ 80 hrs
0 500 hrs
X 1200 hrs
Unit 24
Aluminum electrode
Exposed to 100% R.H.
Stress 125°C
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
D%
ΔC%
100 cps
1Kc
10Kc
100 Kc


--- 0 hours
Δ 80 hours
0 500 hours
X 1200 hours
Figure 3
Unit 5 Aluminium Electrode
No Bake
Stress 125°C 40 VDC
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
Df%
ΔC%
100 cps
1Kc
10Kc
100Kc

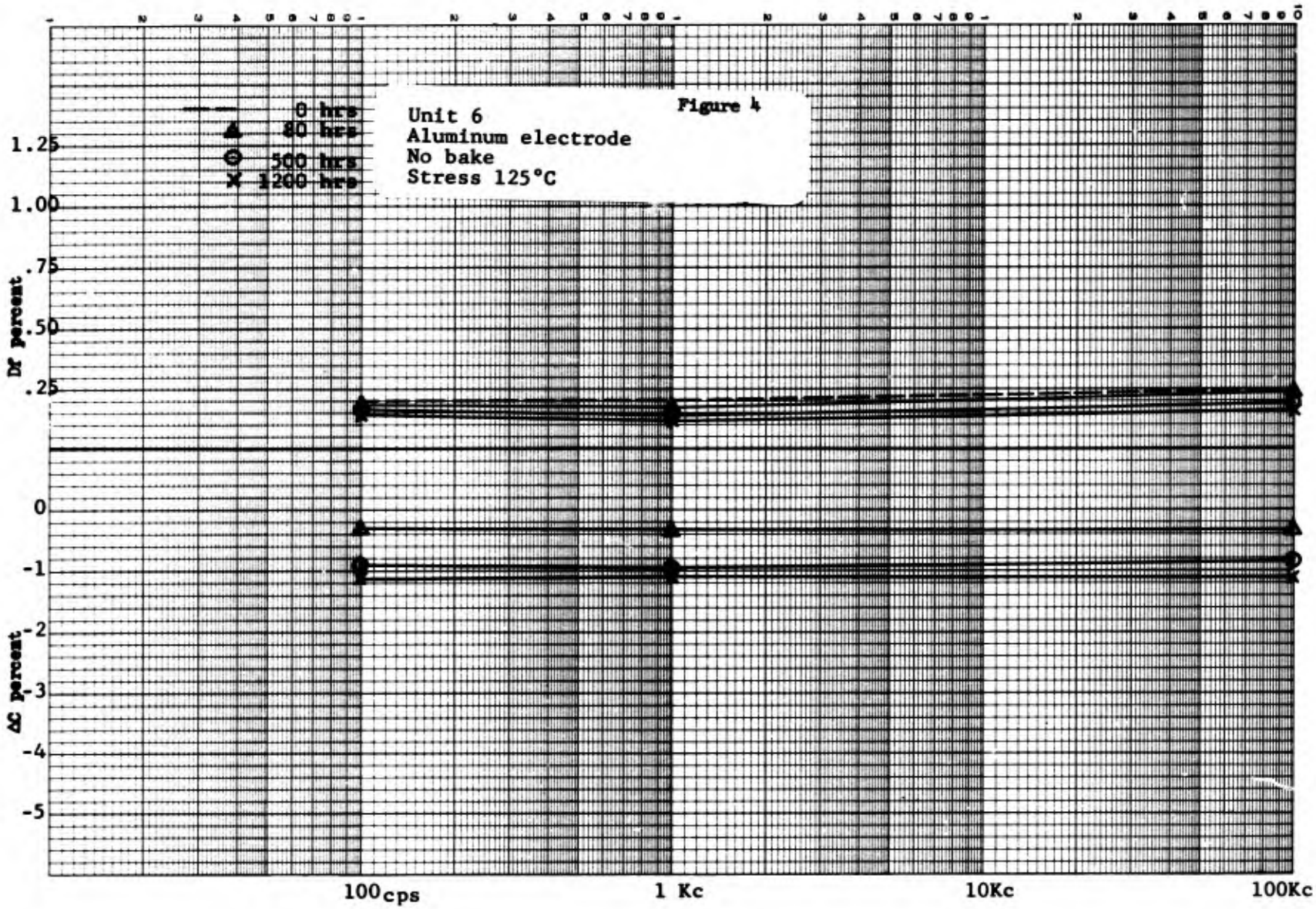


Figure 4
Unit 6
Aluminum electrode
No bake
Stress 125°C
0 hrs
80 hrs
500 hrs
1200 hrs
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
Df percent
ΔC percent
100cps
1 Kc
10Kc
100Kc

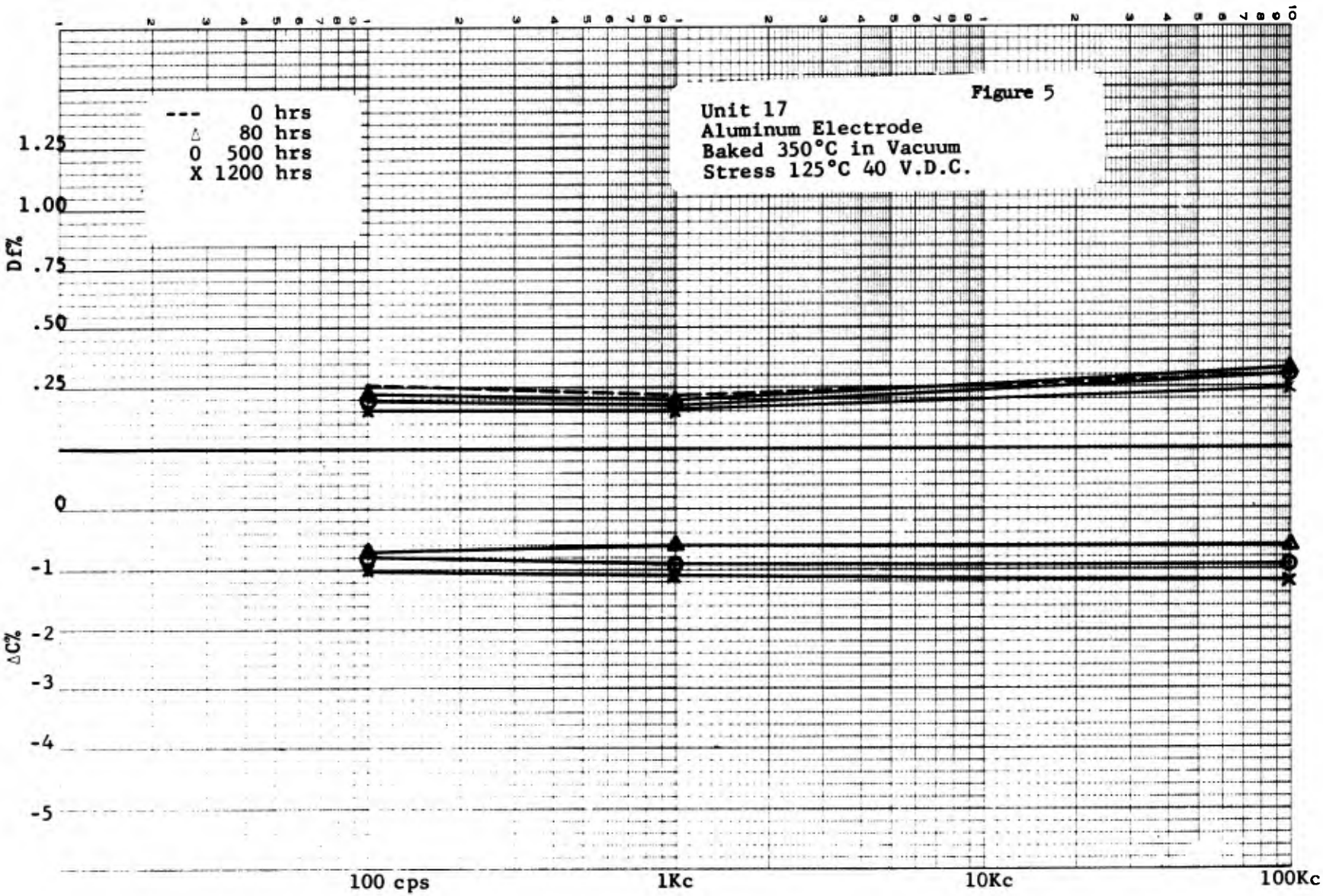


Figure 5
Unit 17
Aluminum Electrode
Baked 350°C in Vacuum
Stress 125°C 40 V.D.C.
--- 0 hrs
Δ 80 hrs
0 500 hrs
X 1200 hrs
Df%
1.25
1.00
.75
.50
.25
0
-1
ΔC%
-2
-3
-4
-5
100 cps
1Kc
10Kc
100Kc

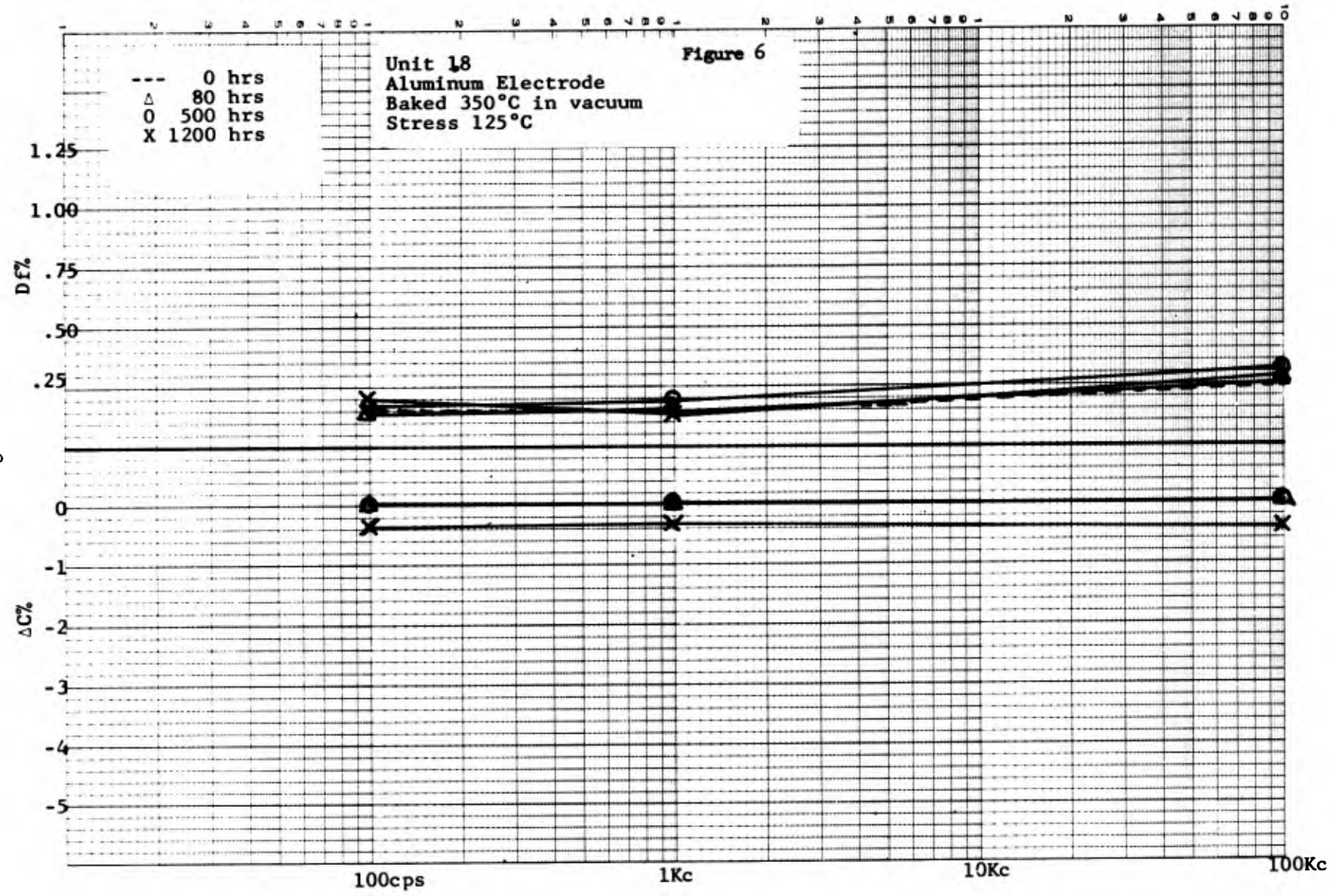


Figure 6
Unit 18
Aluminum Electrode
Baked 350°C in vacuum
Stress 125°C
--- 0 hrs
Δ 80 hrs
0 500 hrs
X 1200 hrs
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
Df%
ΔC%
100cps
1Kc
10Kc
100Kc

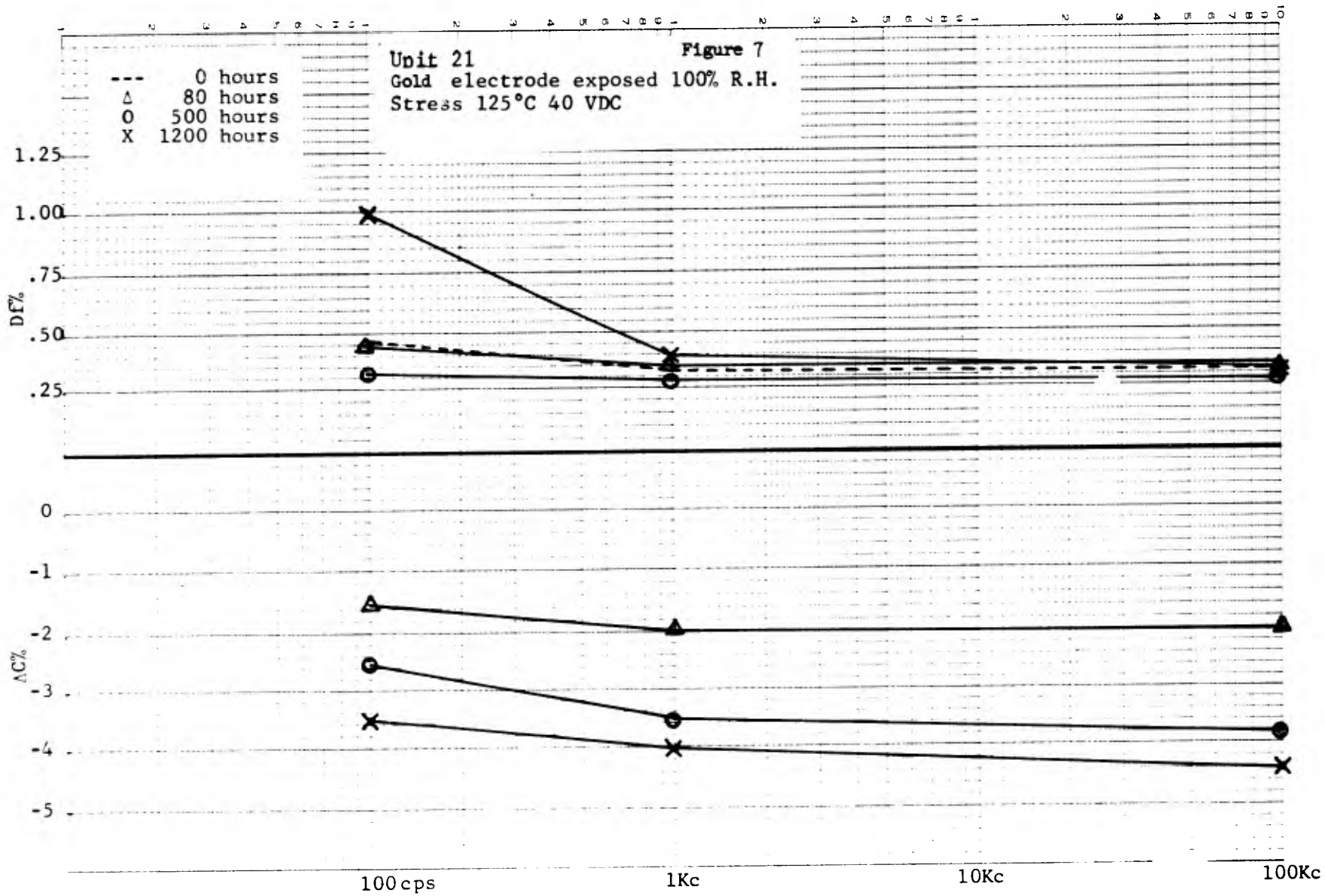

Unit 21
Gold electrode exposed 100% R.H.
Stress 125°C 40 VDC
--- 0 hours
Δ 80 hours
O 500 hours
X 1200 hours
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
DF%
ΔC%
100 cps
1Kc
10Kc
100Kc

Figure 7

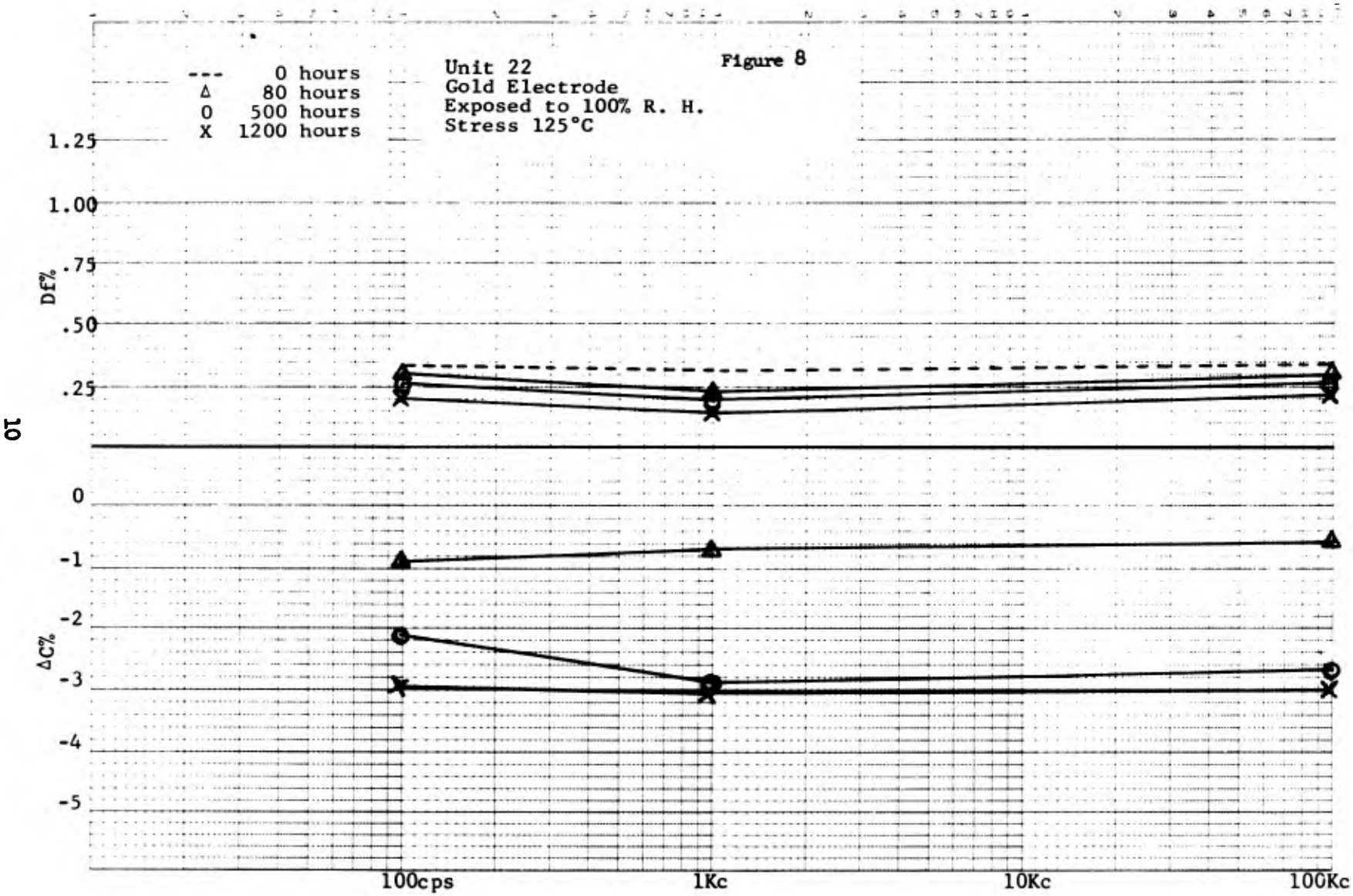

--- 0 hours
Δ 80 hours
0 500 hours
X 1200 hours
Unit 22
Gold Electrode
Exposed to 100% R. H.
Stress 125°C
Df%
ΔC%
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
100cps
1Kc
10Kc
100Kc

Figure 8

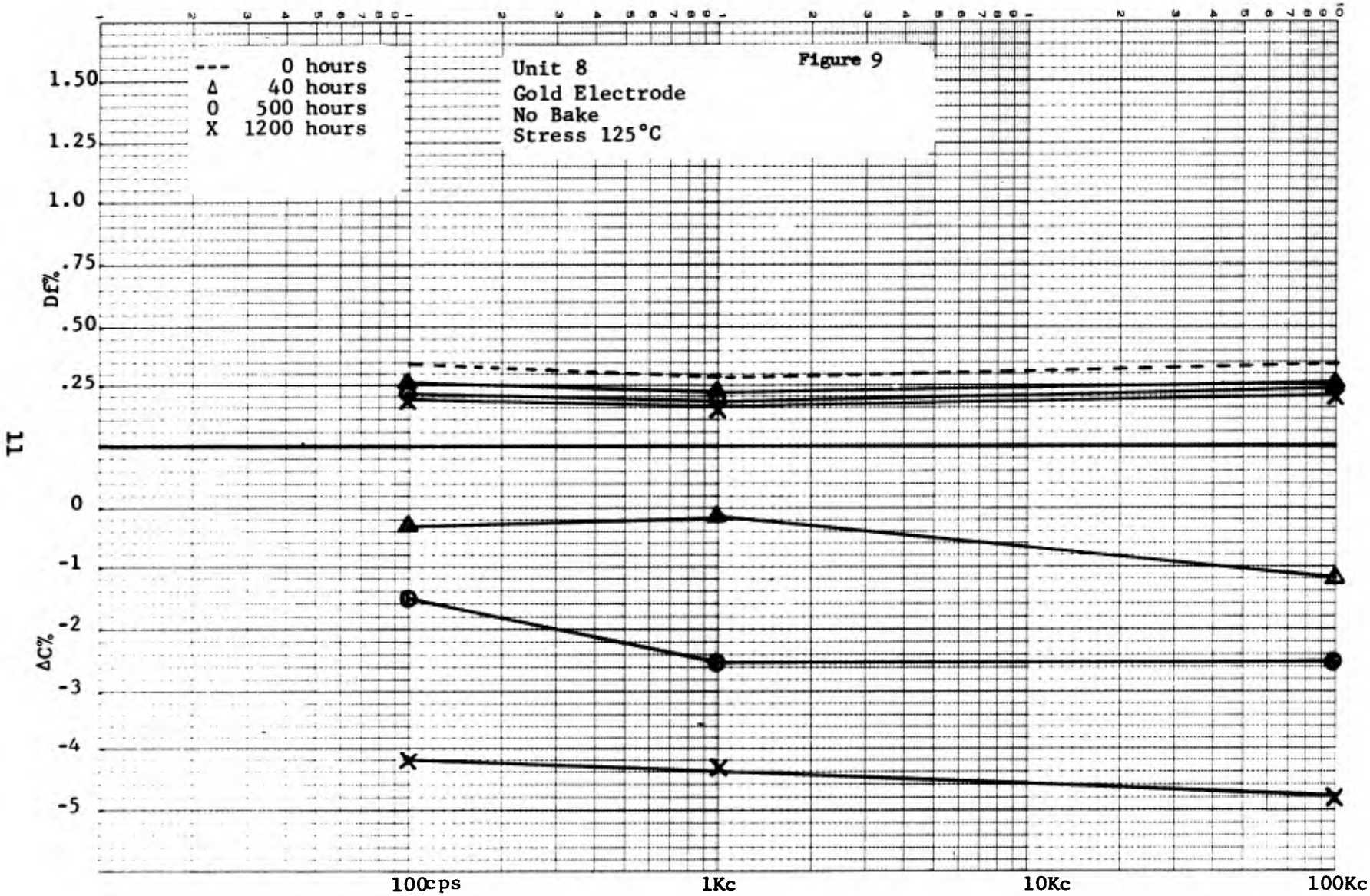


Figure 9
--- 0 hours
Δ 40 hours
0 500 hours
X 1200 hours
Unit 8
Gold Electrode
No Bake
Stress 125°C
1.50
1.25
1.0
.75
.50
.25
0
-1
-2
-3
-4
-5
DF%
ΔC%
100cps
1Kc
10Kc
100Kc

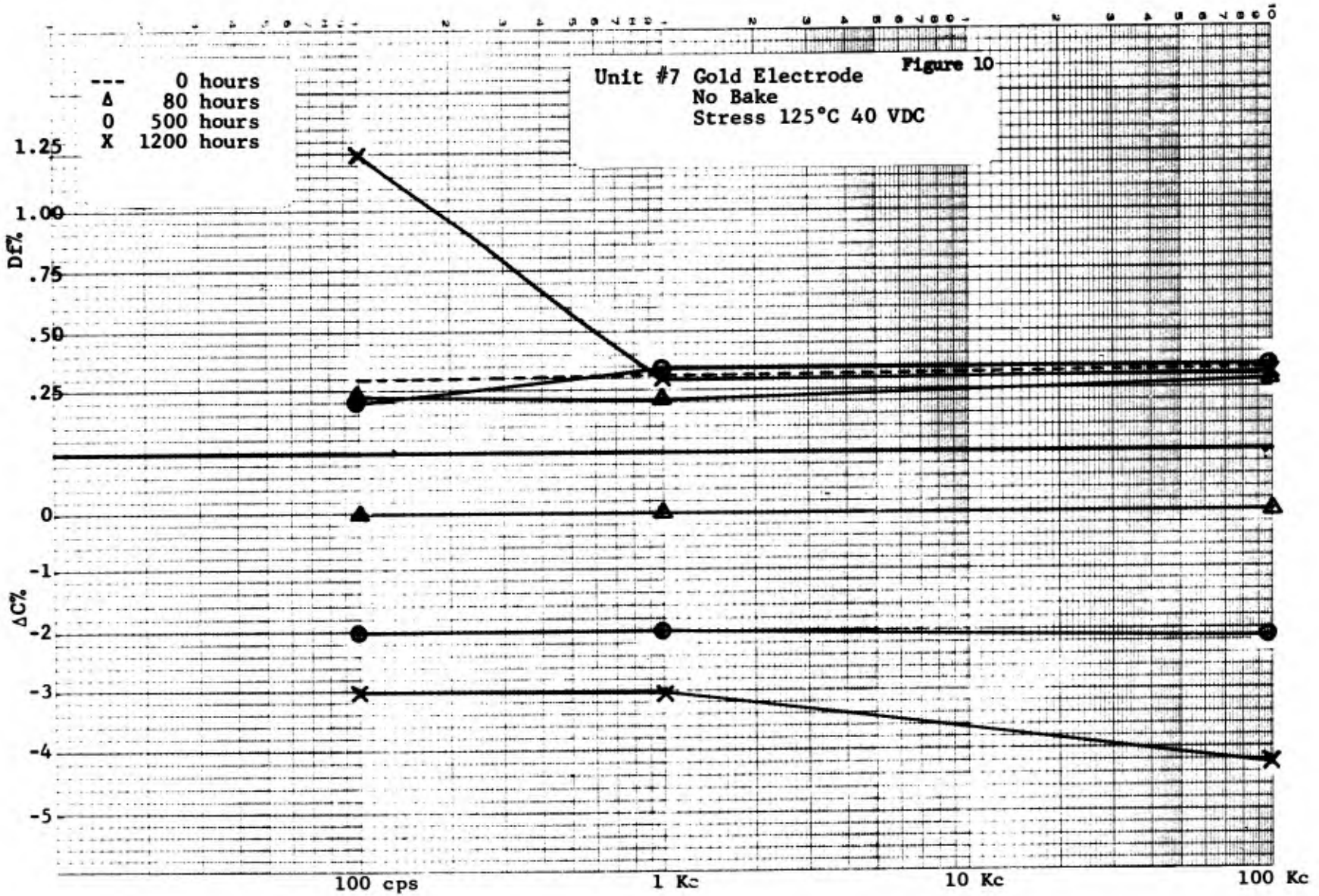


Figure 10
Unit #7 Gold Electrode
No Bake
Stress 125°C 40 VDC
--- 0 hours
Δ 80 hours
0 500 hours
X 1200 hours
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
Df%
ΔC%
100 cps
1 Kc
10 Kc
100 Kc

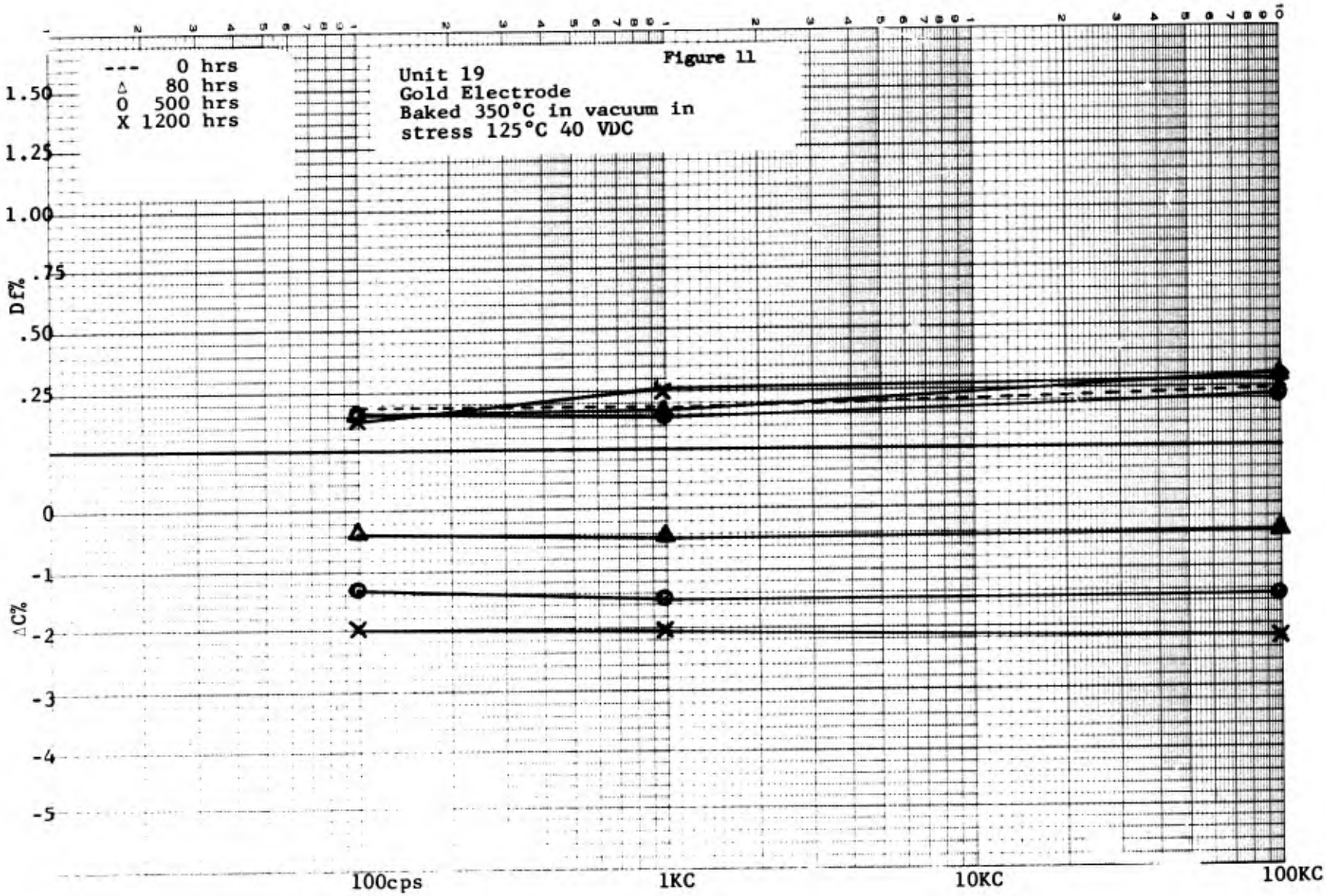
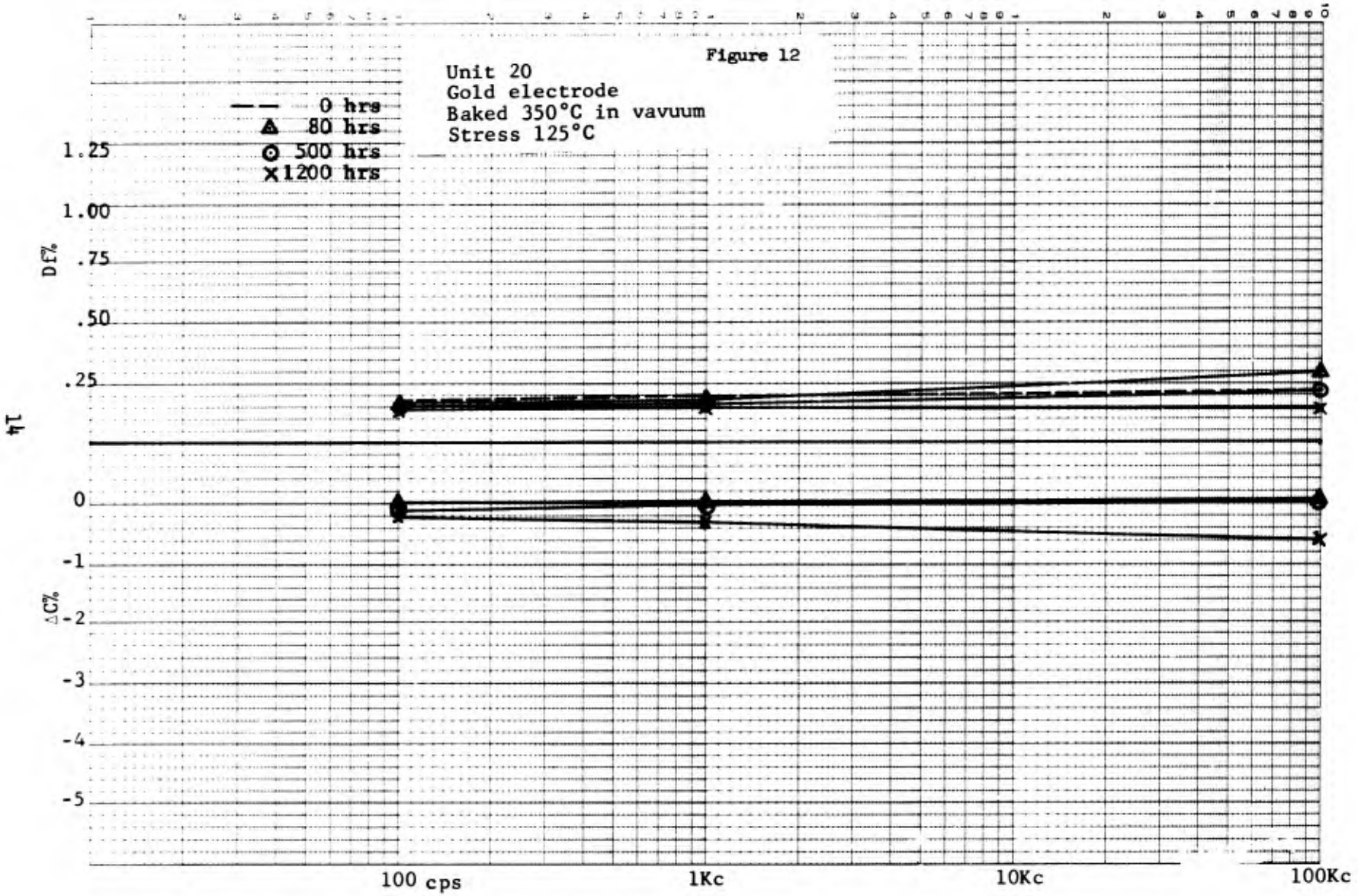


Figure 11
Unit 19
Gold Electrode
Baked 350°C in vacuum in
stress 125°C 40 VDC
--- 0 hrs
Δ 80 hrs
0 500 hrs
X 1200 hrs
1.50
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
D£%
ΔC%
100cps
1KC
10KC
100KC

Figure 12


Unit 20
Gold electrode
Baked 350°C in vavuum
Stress 125°C
0 hrs
80 hrs
500 hrs
1200 hrs
1.25
1.00
.75
.50
.25
0
-1
-2
-3
-4
-5
DF%
ΔC%
100 cps
1Kc
10Kc
100Kc

2.1.2 Capacitance and Dissipation Factor Changes

Figures 1 through 12 plot the percent of change in capacitance and the dissipation factor after 80, 500 and 1200 hours of stress testing.

It is seen that in all cases the capacitance has decreased with stress. The units exposed to 100% R. H. showed the greatest overall change in capacitance with aging. Within each pair of units having the same processing parameter and electrode type, the unit receiving the 40 volt 125°C stress changed somewhat more than the unit receiving 125°C stress only.

It can also be seen that for capacitors prepared under the same processing conditions, those units with a gold top electrode showed the largest changes. With respect to dissipation factor none of the capacitors showed significant changes after stressing, except for units #7 and #21. These units had gold electrodes and received a stress of 40 volts d. c. at a temperature of 125°C. Unit #7 was prepared by evaporation of the top electrode immediately after dielectric deposition, while in unit #21 the dielectric was exposed to 100% relative humidity for three days prior to electroding. It is expected that these units would have larger amounts of absorbed water than the other gold electrode units, all of which received some kind of heat treatment prior to electrode deposition.

Any water present at the dielectric interface could react with the gold electrode to produce a gold ion. Any ions produced would then migrate into the dielectric due to the influence of the strong field applied. The presence of large, easily polarizable ions such as Au^{+} within the dielectric matrix should result in an increase in the dissipation factor such as that observed for these units.

Units prepared under the same conditions but with electrodes of aluminum did not show an increase in dissipation factor. This is expected, since aluminum would react with any water present to form an aluminum oxide. Ionic migration of the aluminum would require breaking of the extremely stable aluminum-oxygen bond. For this reason, field-induced migration of aluminum could not be expected under the conditions of this test.

Confirmation of this interpretation of the test data must await further test results, particularly the results of the radioactive diffusion experiments.

2.1.3 T. C. C. Measurements

The temperature coefficient of capacitance appears to be particularly sensitive to the influence of interfacial effects.

Table 1 gives the T. C. C. for the units exposed to 100% R. H. and those receiving a vacuum bake.

It is seen that the units which were baked in vacuum prior to electroding have changed very little during this stress period. The units exposed to 100% R. H. have all shown a decrease in T. C. C., the improvement in the gold electrode units being particularly marked. The aluminum electroded unit exposed to 100% R. H. and stressed at 125°C 40 volts d. c. did not show the same improvement at low frequency (100 cps) as the unit stressed at 125°C only, although at 100 kc their T. C. C. is identical.

Further measurements are being made to confirm this result.

TABLE I

Electrode	Surface Treatment	Stress	T. C. C. ppm/c° Initial 100cps	1kc	100kc	After 350 hrs. stress 100cps	1kc	100kc
Au	Exposed 100% R. H.	125°C	1200	750	470	320	258	196
		125°C 40v D.C.	"	"	"	228	175	120
Al	Exposed 100% R. H.	125°C	500	425	380	275	215	150
		125°C 40v D.C.	"	"	"	470	350	150
Au	Baked in vacuum	125°C	210	200	125	149	135	107
		125°C 40v D.C.	"	"	"	149	134	119
Al	Baked in vacuum	125°C	150	125	90	140	125	115
		125°C 40v D.C.	"	"	"	154	134	130

2.1.4 Failure Rate

The total number of failures for the two electrode types and the two stress levels is given below:

ELECTRODE	STRESS	UNITS ON TEST	NUMBER OF FAILURES	TIME TO FAILURE
Al	125°C	9	1	1200 hrs.
Al	125°C 40v D.C.	14	1	80 "
Au	125°C	14	1	1200 "
Au	125°C 40v D.C.	10	4	80 "
			1	500 "

If those failures which occurred during the first "burn in" period are ignored, there is statistically no difference in the failure rate between electrode types or stress level.

The high initial failure rate of the gold electroded units is likely due to a field-induced migration of the electrode material along localized micro defects within the dielectric, as the period of time would seem too short to permit diffusion through the bulk.

Additional evidence for this interpretation is that many of the units which were shorted later cleared themselves and thereafter showed no significant deviation in electrical properties from capacitors which had not shorted.

There is no observable correlation between failures and surface treatment of the dielectric at this point.

Stress testing of these units is continuing.

2.1.5 High Humidity Stress Test

A group of 20 aluminum electroded capacitors using a dielectric film of gas plated boro alumina silicate was prepared for high humidity stress test.

The units were held at 80% relative humidity at room temperature with 40 volts d. c. electrical stress. At the end of 200 hours test time none had failed and the average capacitance change was + .1% with the dissipation factor unchanged.

It was decided to attempt to accelerate the test by increasing the stress temperature to 150°F with the relative humidity remaining at 80% and the electrical stress at 40 volts d. c.. At the end of 150 hours test under these conditions average capacitance change was + .5% and the dissipation factor had increased from an average of .17% at 100 cps to .19% at 100 cps.

The increase in D. F. and capacitance at the lower frequency, although minor, does indicate a migration of water or possibly of the electrode material into the dielectric film or it may be due to a surface leakage current.

The units were replaced in the humidity chamber for further test, but an equipment failure permitted condensation of water droplets onto the capacitor plates causing them to short out. For this reason testing of this group was discontinued at this point.

2.1.6 High Temperature Stress Testing

Ten capacitor test units were prepared, using a boro alumina silicate dielectric film and nickel electrodes.

Five of these units were stress tested at 500°C for a period of 16 hours. After this period the units were removed. The average capacitance decrease was 1.3% and the dissipation factor had changed from .18% to .16% at 1 kc. No units had failed at this time.

The units were replaced in the 500°C oven for another period of 20 hours. At the end of the period the top nickel electrode had oxidized so completely that further capacitance measurements could not be made.

To overcome this difficulty the second group of five units was encapsulated with 1000 Å of boro alumina silicate glass. These units showed a decrease of capacitance of only .1% after 40 hours stress at 500°C. The dissipation factor changed from .13% before stressing, to .09% after stress. The encapsulation was successful in preventing oxidation of the top electrode and allowed further stress testing of these units. A further stress period of 20 hours at 500°C produced no further changes in these capacitors.

The temperature was increased to 600°C and the capacitors stressed for another twenty hour period. At the end of the stress period the average capacitance had decreased .2% and the dissipation factor was unchanged. No units failed at this point.

A further stress period of twenty hours caused failure of the bonded aluminum leads used to make contact to the capacitor electrodes and the test was discontinued.

Although high temperature testing is necessary to establish failure rates within a reasonable time and sample size, there is the possibility of introducing failure modes which are inoperable at lower stress levels.

The rather high temperature, 500°C, used in this test was selected since it is slightly greater than the sealing temperature used in the ceramic flat pack for integrated circuits and so modes of failure introduced by this stress level are of primary importance in a practical application.

2.1.7 Diffusion Study

The diffusion of ambient gases through a dielectric film to an underlying metal may have a deleterious effect at the metal-dielectric interface that would lead to device failure. In particular, thin film metal resistors are susceptible to attack by oxygen ambients and must be protected by encapsulation with a dielectric film.

To obtain a semi-quantitative estimate of the diffusion coefficient of O_2 through the dielectric film, the following procedure was used.

A film of copper was vacuum evaporated onto a glass substrate and a 1000 Å thick film of boro alumina silicate gas plated over the surface.

The optical density of the sample under white light was then determined with the Welch Densicron.

The test specimen was then heated in air at 500°C for a two hour period and the optical density remeasured. Diffusion of oxygen through the dielectric film would be indicated by increased light absorption at the dielectric metal interface due to formation of the black copper oxide. Under the conditions of this test an unprotected copper film would be completely oxidized in less than 30 seconds.

Experimentally, an increase of 10% in the optical density was taken as being indicative of the arrival of oxygen at the metal dielectric interface.

From Fick's Law one may derive the expression:

$$T_d = \frac{l^2}{6D}$$

where l is the film thickness, in cm, D the diffusivity constant and T_d the time required for a molecule to diffuse through the film.

Experimentally, it required from two to four hours exposure to a 500°C air ambient to obtain a 10% increase in optical density indicating from the above equation a value for $D < 10^{-14}$ cm^2/sec for these gas plated films. This value for the diffusion constant of gas plated glass films compares favorably with values reported in the literature for 3.5 to 3.8 x 10^{-12} cm^2/sec [1] for O_2 diffusion through a glass at 450°C.

2.2 Future Work

Extended stress testing of the first test group of capacitors will continue. The T. C. C. of all capacitors on test will be obtained after the 1500 hours of stress.

Additional capacitors will be prepared for high humidity stress testing and high temperature accelerated stress test.

Both A. C. and D. C. electrical stress will be used in this series of tests.

Electron micrographs of units which fail during test will be obtained and compared to micrographs of good units.

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[1] V. Carino-Canina, Compton's Revised Edition, p. 248 (1959). 1319

3.0 RADIOTRACER STUDIES OF METAL DIFFUSION AS A MODE OF FAILURE

3.1 Discussion

At the interfaces of the metal film electrodes and the thin-film dielectric of a thin film capacitor, thermal or electrical stress may produce diffusion of the electrode metal into the dielectric material and alter the resistivity and other electrical and physical properties of the dielectric to a depth equivalent to the depth of the diffusion. This may change the electrical characteristics of the capacitor, or, if the diffusion has progressed to a considerable degree, may produce high "leakage" or catastrophic failure. To determine quantitatively the concentration profile of the diffused metal through the dielectric, the metal is activated by neutron irradiation and radiotracer analysis employed. Utilizing incremental etches of the dielectric layer and a radioactivity count of each etch solution, the profile is established. The following conditions must apply if this analytical method is to be practical:

1. One metal electrode must be removable without destruction of the dielectric and its supporting structure to enable incremental etching of the dielectric.

2. The dielectric must etch uniformly at a controllable rate and without destruction of the bottom capacitor electrode.

3. A non-destructive means must be utilized to measure dielectric thickness before and after each etch.

4. The diffused metal must have a sufficiently high radioactivity level to enable a significant count to be made and the half-life must be sufficiently long to permit a reasonable experiment time.

5. The dielectric material must not contain enough beta or gamma radiation emitting elements (other than the element being analyzed) to mask the radio activity count of the metal being analyzed.

3.2 Tantalum-Tantalum Oxide Capacitors

3.2.1 Discussion

The tantalum-tantalum oxide interface was initially chosen for study because of its use as an electronic component and its applicability to radio tracer techniques. This method, initiated under Contract AF30(602)-2593, was used to reveal irreversible migration of tantalum atoms across a thermally and electrically stressed tantalum-tantalum oxide interface found in a thin-film tantalum oxide capacitor formed by anodizing a radioactive tantalum film. After stressing, the tantalum oxide was etched in small incremental thicknesses, using a hydrogen fluoride solution. The radioactivity of the sample was measured after each etch and the thickness of the remaining tantalum oxide film measured by non-destructive optical methods. Under the present contract the radioactivity of the etch solution, rather than the radioactivity of the entire unit, will be measured. Previous experience indicated that the radioactivity level of the entire unit (substrate, tantalum anode and unetched dielectric) was sufficiently high to saturate or partially insensitize the sodium iodide detector of the radioisotope disintegration counting equipment

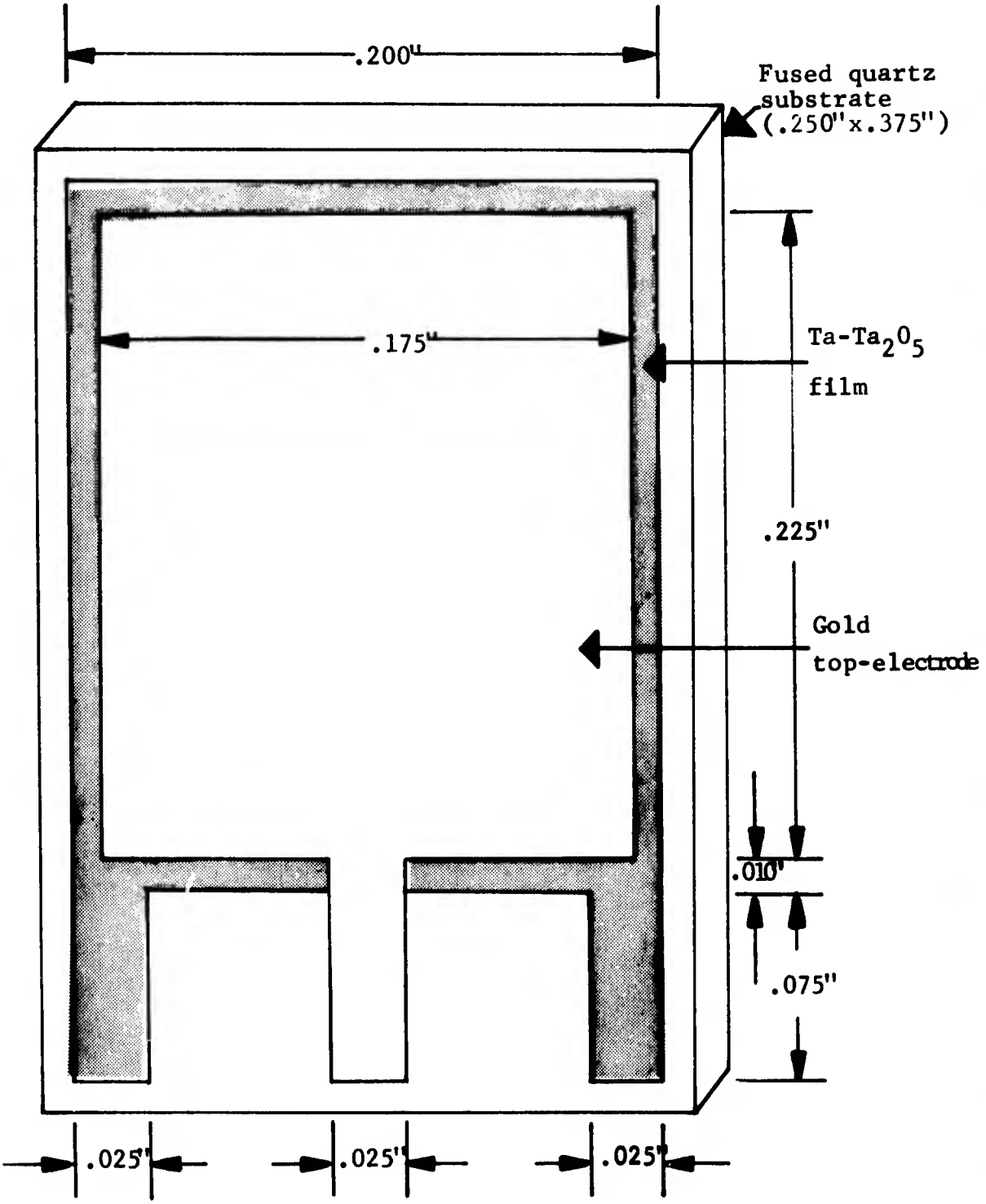


Figure 13

Ta-Ta_2O_5-Au Stress-Test Unit

and consistent readings could not be made. The radioactivity level of the etch solution is expected to be well within the practical range of this equipment. An activity profile depicting the distribution of radioactive tantalum isotopes through the oxide may be obtained. By comparing the profile of a stressed unit with that of an unstressed unit, differences resulting from the migration of tantalum atoms through the oxide or across the tantalum-tantalum oxide interfaces may be correlated with stress conditions. A correlation of change of the tantalum atom profile through the dielectric with change of electrical characteristics of the capacitor unit incorporating this dielectric is to be established.

3.2.2 Preparation and Description of Samples

Forty tantalum films ranging from 2340Å to 2730Å in thickness were deposited on optically polished 0.375 x 0.250 inch fused-quartz substrates by vacuum evaporation. The configuration of the tantalum deposition and a subsequent gold deposition are shown in Figure 13. A second series of twenty-nine samples ranging in thickness from 2800Å to 3440Å were prepared in like manner. The failure of the first series to withstand incremental etching was ascribed to insufficient thickness of the deposited metal. The second series was accordingly made thicker as a potential solution to the etch problem. The configuration of the tantalum deposition was such that the tantalum film would function as the capacitor bottom electrode (anode) and would also, upon anodization, form the dielectric. Prior to anodization the units were sent to the A. E. C. Oak Ridge National Laboratories for neutron irradiation. (See Appendix for activity level and irradiation time calculations.)

The first series of films was anodized at a forming voltage of 82 volts in a 5% solution of $(NH_3)_2\ HPO_4$ by a process

described by R. W. Berry (Bell Telephone Lab.), U. S. Patent No. 2993266. To complete the capacitor, a gold top electrode (cathode of suitable configuration as indicated by Figure 1) was applied by vacuum deposition and leads were attached.

The second series has been sent to Oak Ridge for irradiation, and has not been returned at the time of this writing.

3.2.3 Thermal, Electrical and Environmental Stressing

In preparation for stressing of the first series of 40 units, gold top electrodes were evaporated onto 20 of the units. Capacitance and dissipation at 1000 cps were then measured directly by means of a Model 650-A General Radio Capacitance bridge, a Model 200 C Hewlett Packard audio oscillator as a signal source and a Model 400D Hewlett Packard voltmeter as a null indicator. The capacitance of 17 units ranged from 33.0 mμf to 34.8 mμf and dissipation ranged from 0.9% to 1.9%. One unit was extremely erratic electrically and 2 units displayed direct shorts. The 17 good units were momentarily stressed at 22½ volts direct current with the tantalum positive. Seven units failed immediately. Since 10 of the original 20 units failed, the total yield at this point was 50%, a typical yield for capacitors of this size and type. Those that failed electrically were still suitable for thermal, but not electrical stressing and therefore were included in the stressing program. For the following reasons, units with and without gold top-electrodes were included in the stressing schedule:

A.) Oxidation of the dielectric by the surrounding air might alter the radioactivity profile of the dielectric. A gold top-electrode might inhibit this oxidation if such oxidation exists. Comparison of similarly stressed units with and without top

electrodes might indicate whether or not oxygen does diffuse into the Ta_2O_5 layer and, if so, what influence it has.

B.) During gold electrode application the units are heated to approximately 200°C for several minutes. This thermal stressing may contribute to tantalum diffusion.

Later observations indicated that any diffusion anticipated in B.) is negligible.

The stress program anticipated at the start of the stress period is presented by the following schedule:

GOLD ELECTRODE	STRESS		ENVIRONMENT
No	Room temp.	4 weeks	Air
No	Room temp.	8 weeks	Air
No	70°C	4 weeks	Air
No	70°C	4 weeks	Argon
No	125°C	4 weeks	Air
No	125°C	4 weeks	Argon
Yes	Room temp.	4 weeks	Air
Yes	Room temp.	22½V dc 2 weeks	Air
Yes	Room temp.	22½V dc 4 weeks	Air
Yes	Room temp.	22½V dc 8 weeks	Air
Yes	70°C	4 weeks	Air
Yes	70°C	4 weeks	Argon
Yes	70°C	22½V dc 4 weeks	Air
Yes	125°C	4 weeks	Air
Yes	125°C	4 weeks	Argon
Yes	125°C	22½V dc 4 weeks	Air

Stress periods indicated above were tentative and were dependent on observed changes in electrical characteristics as monitored by capacitance, dissipation and current leakage measurements. As discussed later, electrical measurements indicate that 8 weeks stressing time is insufficient for significant diffusion changes.

At the time of this writing the units on electrical stress are in the tenth week of stressing. Capacitance, dissipation and leakage have been monitored at two week intervals and a plot of the capacitance and dissipation results presented in Figures 14, 15 and 16. Immediately after capacitance and dissipation measurements, current leakage at 10 volts d. c. with the tantalum positive was determined by use of a General Radio Type 1230-A D. C. Amplifier and Electrometer, and the readings presented in Table II.

TABLE II

Current Leakage of Anodized Tantalum Capacitors Before and After Stressing

(Leakage in picoamps at 10 V d.c. (Ta+)

Unit Number	Initial	2 wks.	4 wks.	6 wks.	8 wks.	Stress
0	4	--	3	10	7	Room Temp. 22.5 V
75	7	--	20	500	14	"
135	130	5	2	3	3	"
17	7	--	20	5	15	70°C 22.5 V
195	7	--	10	7	7	"
20	2	--	5	7	10	"
8	22	3	(shorted in 24 days)			125°C 22.5 V
11	16	7	10	8	6	"
12	20	(shorted in 1 hour)				"
13	20	4	35	420	2	"

The net change in capacitance in eight weeks for those capacitors that did not short (75 not included because of top-electrode damage) is shown below:

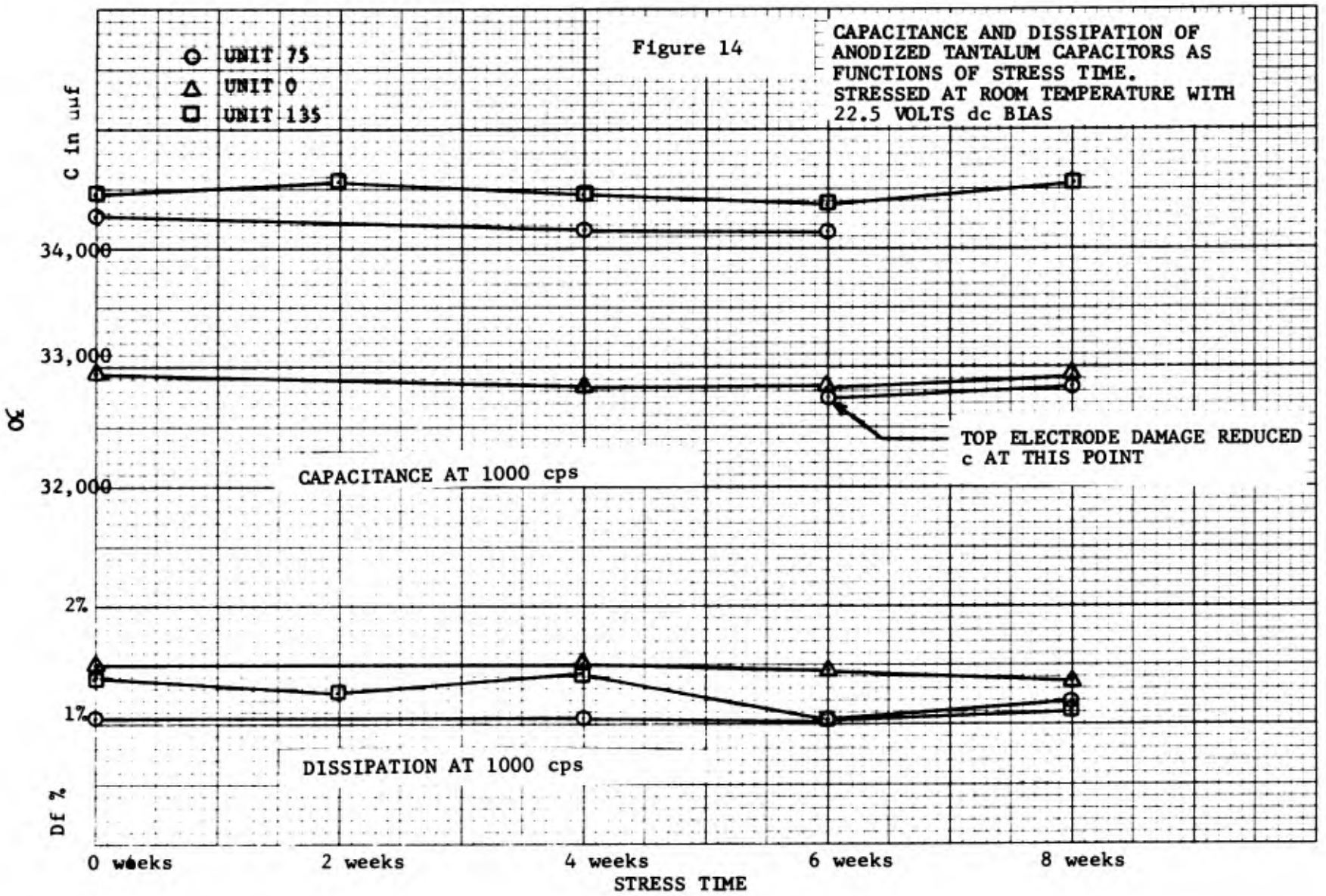


Figure 14
CAPACITANCE AND DISSIPATION OF ANODIZED TANTALUM CAPACITORS AS FUNCTIONS OF STRESS TIME. STRESSED AT ROOM TEMPERATURE WITH 22.5 VOLTS dc BIAS
UNIT 75
UNIT 0
UNIT 135
C in μμf
34,000
33,000
32,000
%
2%
1%
Df %
CAPACITANCE AT 1000 cps
DISSIPATION AT 1000 cps
TOP ELECTRODE DAMAGE REDUCED c AT THIS POINT
0 weeks
2 weeks
4 weeks
6 weeks
8 weeks
STRESS TIME

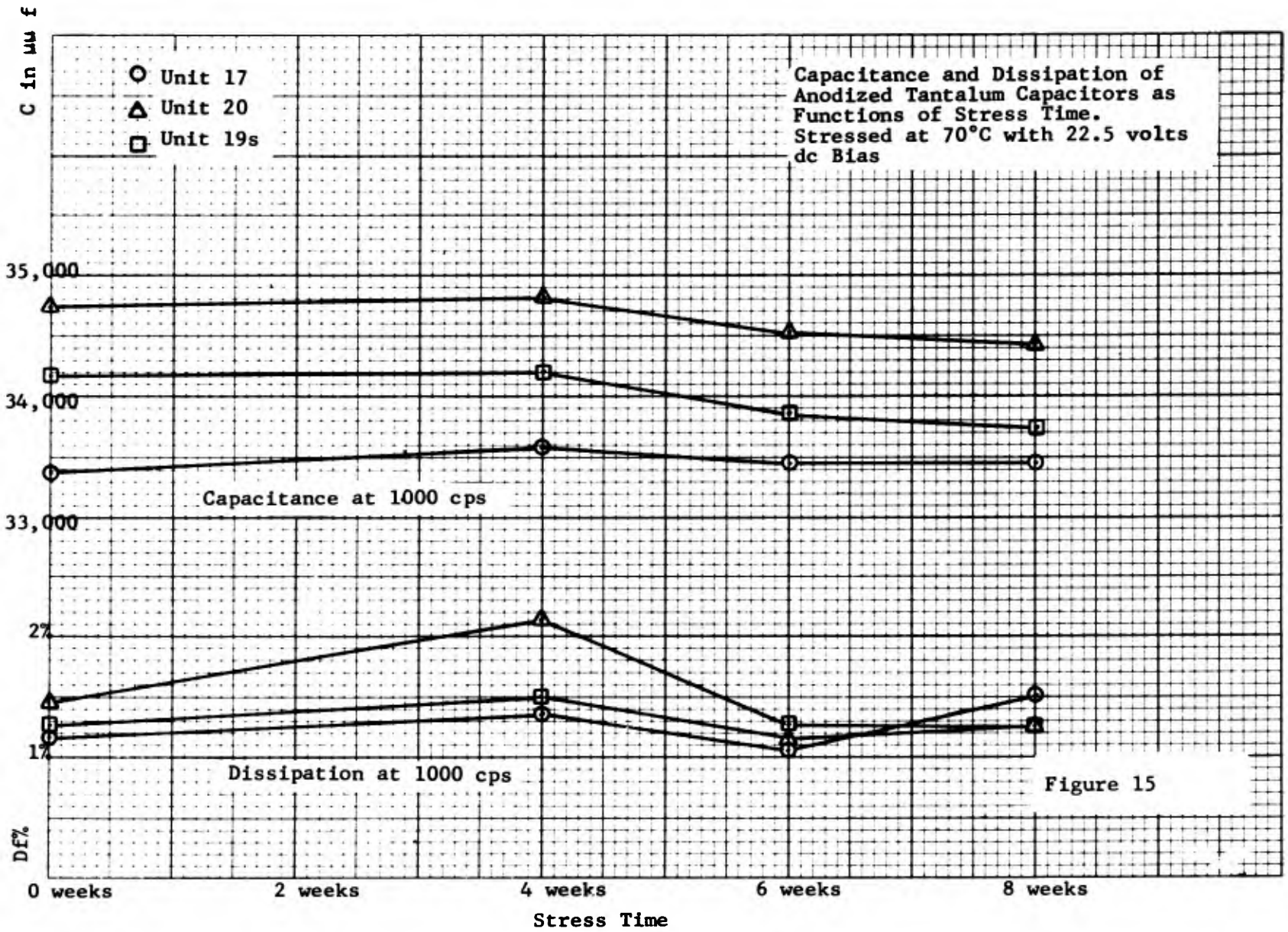

C in μμf
Unit 17
Unit 20
Unit 19s
Capacitance and Dissipation of Anodized Tantalum Capacitors as Functions of Stress Time. Stressed at 70°C with 22.5 volts dc Bias
35,000
34,000
33,000
Capacitance at 1000 cps
2%
1%
Dissipation at 1000 cps
Df%
0 weeks
2 weeks
4 weeks
6 weeks
8 weeks
Stress Time

Figure 15

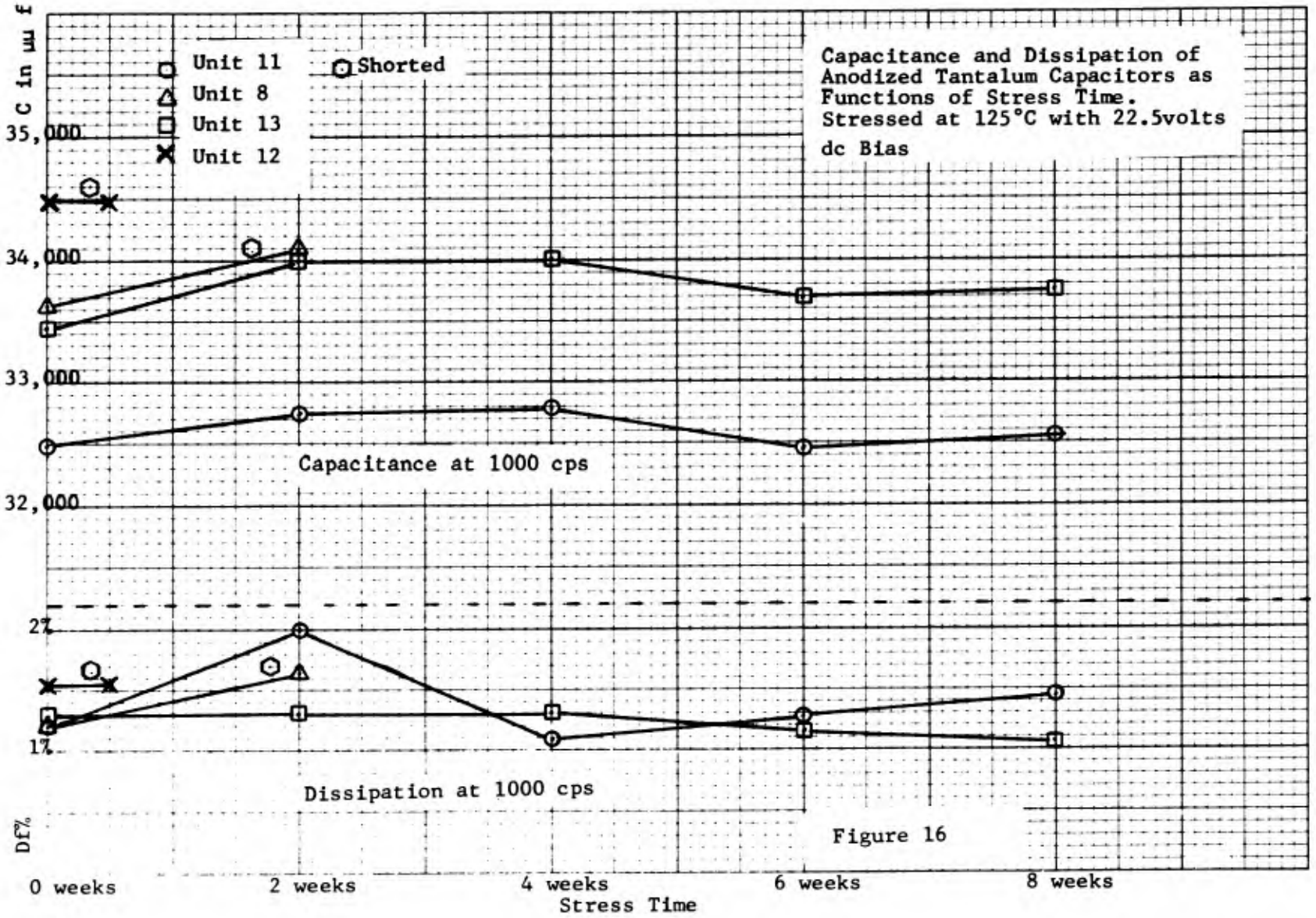

Capacitance and Dissipation of Anodized Tantalum Capacitors as Functions of Stress Time. Stressed at 125°C with 22.5volts dc Bias
Unit 11
Shorted
Unit 8
Unit 13
Unit 12
C in μf
35,000
34,000
33,000
32,000
Capacitance at 1000 cps
2%
1%
Dissipation at 1000 cps
Df%
0 weeks
2 weeks
4 weeks
6 weeks
8 weeks
Stress Time

Figure 16

Number	Stress Temp.	Change of C in %	
0	Room	-.09	
135	Room	+.17	Average +.04
17	70°C	+.15	
195	70°C	-1.26	Average -.68
20	70°C	-.92	
11	125°C	+.03	
13	125°C	+.78	Average +.40

Theoretically, diffusion of tantalum into the tantalum oxide dielectric upon stress produces an increase in thickness of a semiconducting layer between the almost stoichiometric tantalum bottom-electrode and tantalum oxide dielectric. Increase in thickness of this semiconducting layer is accompanied by a decrease in thickness in the dielectric layer and capacitance rises. In this investigation the average capacitance of all units stressed for eight weeks dropped 0.15%, a statistically insignificant change that could be credited to experimental error, drift in instrumentation or slight changes in ambient conditions.

It appears that eight weeks stressing at the thermal and electrical levels indicated is insufficient to diffuse a significant quantity of tantalum into the oxide dielectric.

Current leakage results as presented in Table II may further confirm this contention. If, upon stress, tantalum diffuses into the dielectric, the resistance of the dielectric should be reduced and leakage current rise. As calculated from Table 1, the average leakage (not including shorted units 8 and 12) dropped from 23.5 picoamps at 10V d.c. to 6.4 picoamps after 8 weeks of

stressing. Experience indicates that capacitors of this type have "self healing" ability. It appears that minute local areas of high current density may temporarily produce relatively high total current leakage and then heal. The total leakage may then return to a low value. This mechanism is indicated by units 75, 135 and 13. As shown by Table II, units that shorted (8 and 12) did not have significantly high initial leakage; so, at least in this series, high initial leakage cannot be considered as a precursor to failure.

As indicated by Figures 14, 15 and 16, dissipation fell within a range of approximately 1% to 2% and was erratic for some units. Average dissipation for all units that did not short dropped from 1.28% to 1.27% after 8 weeks stress, an insignificant change. The erratic changes in some units may be attributed to questionable integrity of the bond between the electrodes and their connection wires. Ohmic connection was made by means of conductive silver paint. The mechanical strength of this bond was poor and occasional connection breaks made repair necessary. It is possible that variations in series resistance due to bond damage caused the dissipation variation.

No definite correlation of changes in capacitance, dissipation and leakage was evident. Those that were erratic in dissipation were no more erratic than the others in capacitance or leakage and those erratic in leakage displayed no trend of being erratic in capacitance or dissipation.

3.2.4 Radiotracer Profile of Stressed Ta_2O_5 Dielectric

As indicated in the "First Technical Documentary Report," 6 December 1963 - 5 March 1964, of this investigation, stressing of Ta, Ta_2O_5 units will be continued until significant changes in

the electrical properties are noted. When such changes occur, the radioactive profile will be determined. As has been stated in section 3.2.3, eight weeks stress at 22.5 volts bias and at room temperature, 70°C and 125°C, has not produced significant changes in the electrical characteristics and apparently is not sufficient, for tantalum diffusion, to begin radioactivity profile studies. Because the establishment of profiles of unstressed units is a necessary part of the program, several anodized but unstressed units from the first series of 40 units (see section 3.2.2) irradiated at Oak Ridge were incrementally etched in HF according to the procedure developed under AF 30(602)-2593 contract. Failure of these units to etch uniformly as the Ta-Ta_2O_5 interface was approached precluded continuation of radiotracer studies of this series. Because the most significant dielectric alteration is anticipated near the metal-metal oxide interface, it is essential that the etch in this region be very uniform. A second series of 29 units (see section 3.2.2) was then sent to Oak Ridge for activation. Greater diligence was exercised with the second series to improve the substrate-tantalum bond and thus assure good etch performance. Although the first series of 40 capacitors was not suitable for radiotracer studies, the capacitors were suited for electrical characterization as a function of stressing and therefore were put on stress as reported in section 3.2.3.

Initial etch attempts with the first series of capacitors were 40% on HF basis - 8 parts of 49% reagent grade HF were used with 2 parts of water. This concentration, used successfully under Contract AF 30(602)-2593, etched away about 5 Å of Ta_2O_5 per second. After removal of about 700 Å (about 43%) of the Ta_2O_5, film pitting and loosening started and upon etching of about 200 Å more Ta_2O_5, destruction was so serious that radiotracer analysis of the etch increments would be meaningless. Other concentrations of HF ranging from 10% to 100% were tried, but

not found to be better than the 40% concentration. Addition of H_2SO_4 to HF solution accelerated the etch rate but did not improve etch performance or inhibit film destruction.

As in Contract AF 30(602)-2593, the oxide thickness was monitored optically before and after each etch by wavelength minimum reflectivity measured by means of a specially constructed reflectance attachment made to fit a Beckman Model B spectrophotometer (see Figure 17). For a nonabsorbing oxide the following relationship applies:

$$(D + X) = \left(\frac{2r-1}{2}\right)\left(\frac{\lambda}{2n \cos \theta_1}\right)$$

where

D = thickness
X = constant which allows for reflection phase changes
r = 1, 2, 3, etc. denoting orders of interference
λ = wavelength of minimum reflection
n = refractive index of oxide at wavelength λ
θ_1 = angle of refraction

The refractive index is calculated using the formula derived by Young:[2]

$$n = 2.14 + 0.292/(\lambda/10^3 A - 2.305)^{1.2}$$

θ_o = 11° as established by the mirror placement of the reflection attachment (Figure 5).

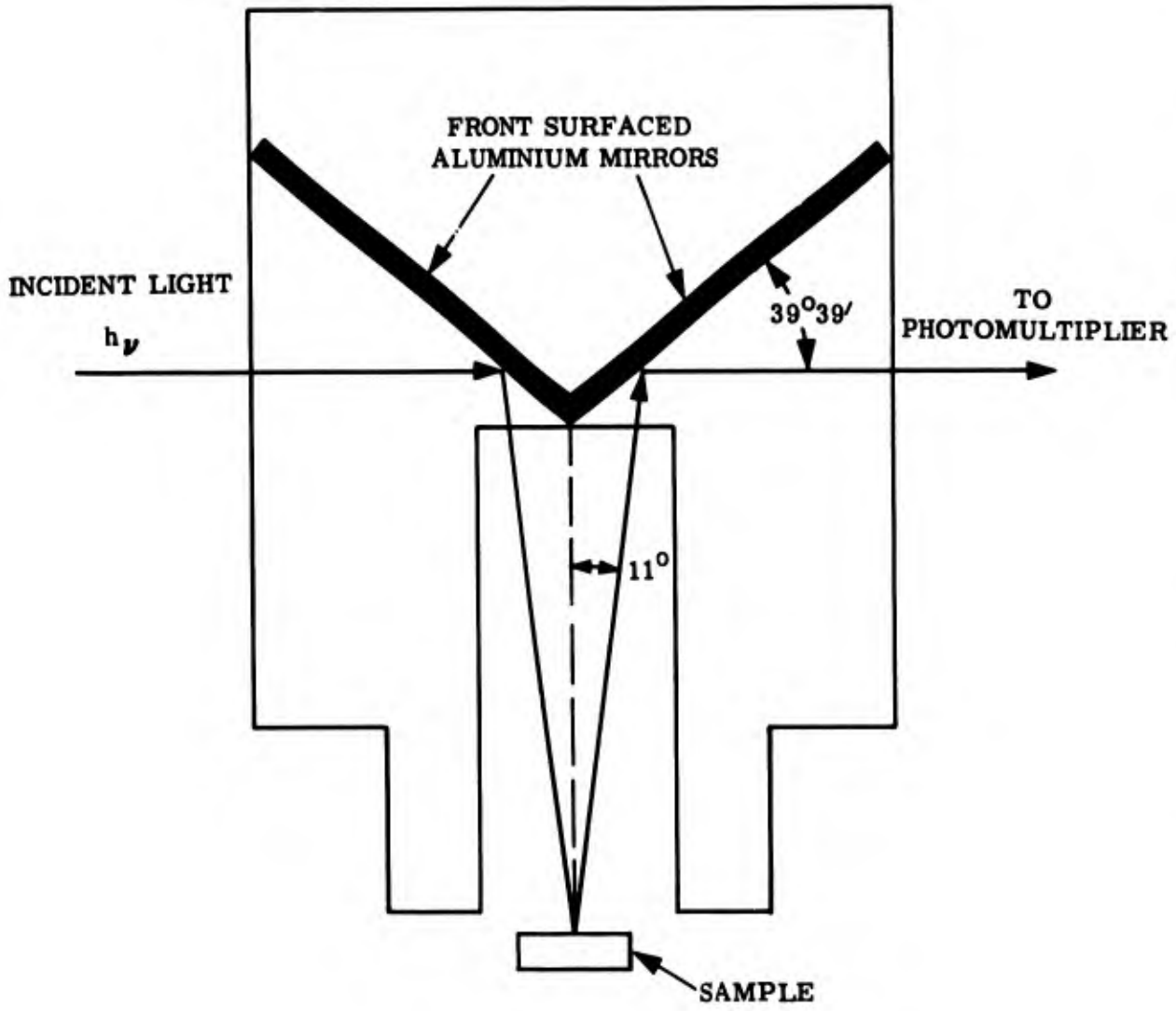
Since θ_1 is dependent on refractive index according to the following relationship,

$$\sin \theta_1 = \frac{\sin \theta_o}{n}$$

$\sin \theta_1$ must be calculated for each wavelength.

Interference occurs between the light reflected from the nonabsorbing oxide with a phase change of 180 degrees and

[2] Young, L., "Anodic Oxide Films," p. 82, Academic Press, N.Y. 1961.



Spectrophotometer
Reflectance Arrangement

Figure 17

light which is reflected from the metal with an unknown phase change. This gives rise to the constant X. The difference between the two phase changes results in a calculated thickness greater than the actual oxide thickness. This thickness difference was established as 156 Å by work done under Contract AF 30(602)-2593.

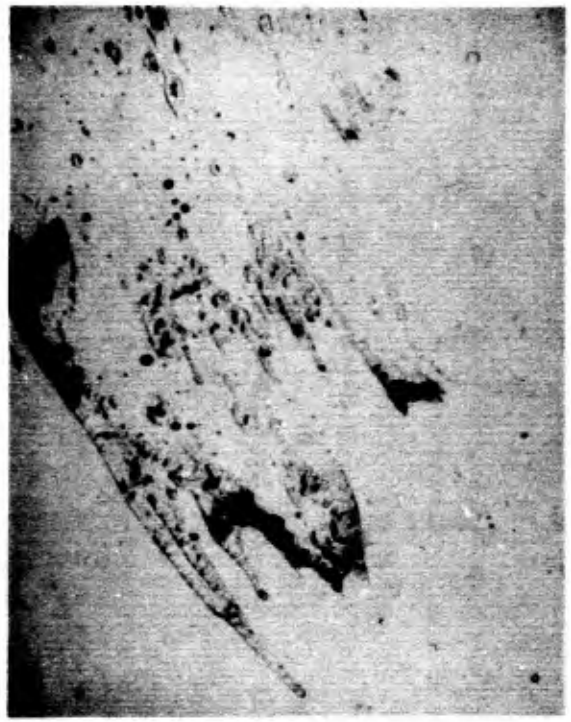
All radioactivity measurements are to be made with a Nuclear Chicago Corp. Model DS 5-4 scintillation detector, a Model 1810 radiation analyzer, and a Tracerlab Model SC-32 scaler.

3.2.5 Radiographic Indication of Potential Failure Sites in Tantalum Film

Area modulated irregularities in radioactivity of of an activated film may be detected and recorded by direct contact of the radioactive film with a suitable silver halide photographic emulsion after sufficient exposure time.

Upon neutron irradiation, contamination sites that might be potential failure sites will be activated to a different degree than the surrounding uncontaminated tantalum film. A radiographic examination may locate these sites.

In this investigation an activated and anodized tantalum film was placed in contact with Kodak Type M single-coated X-ray film. Purposely chosen was a tantalum film having many irregularities so that an easy correlation could be made between the blemish areas and the X-ray film image-modulation (see Figure 18) It appears that with this arrangement and film, areas having less activity than the surrounding Ta and Ta_2O_5 must be about .03mm in diameter, or greater, to be visible on the radiograph. It is likely



Original Film 50X

Radiographic Image 50X

Figure 18

Radiograph of Anodized Tantalum Film

that contamination sites having relatively high activity could be much smaller and yet be visible as dark spots on a negative radiograph.

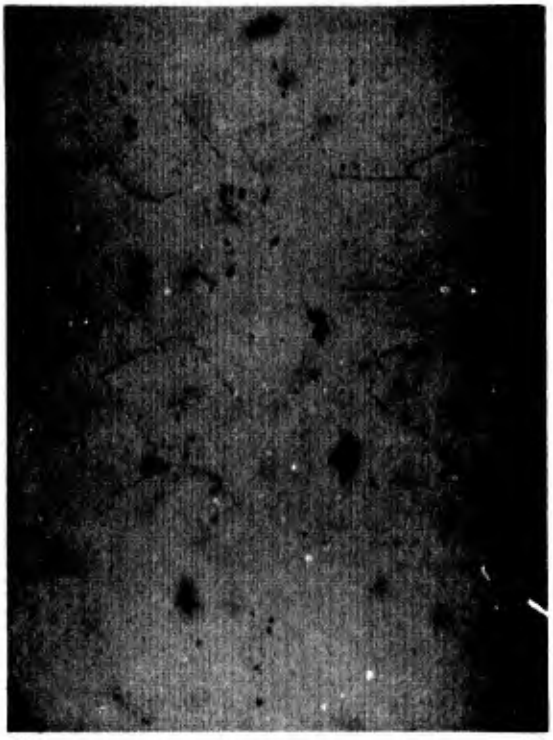
This has only been cursory examination of radiography as a technique for finding potential failure sites and the feasibility of this method has not been established. Probably the resolution would have to be improved by at least two orders of magnitude to produce meaningful results.

3.2.6 Recrystallization of Anodized Tantalum

Young[3] reports dielectric recrystallization during the oxide formation process in the manufacture of tantalum capacitors to be an important mode of dielectric failure and states that crystalline oxide may be caused to appear in some anodic films by holding them under voltage at temperature well below 100°C. The crystalline areas may be seen under an optical microscope at a magnification of a few hundred times as irregularities having a characteristic 5- or 6- sided polygonal shape.

It has been assumed as a task in this investigation to scrutinize the oxide surface of the stressed tantalum units for similar crystallization or evidence of crystallization of any sort. Although no crystalline areas similar to those reported by Young have been observed, a geometric crystalline structure (Figure 19) was found on the oxide surface of a tantalum film that had been anodized at 82 volts in 5% $(NH_3)_2HPO_4$ but which had no additional thermal or electrical stress applied. The smaller crystals were about 30 microns in length. A 5 minute soak in 80°C water did not remove this structure, but a 5 second etch in 80% HF

[3] Young, L., "Anodic Oxide Films," Chapter 9, Academic Press, N.Y. 1961



500X

Crystalline Structure Observed on Ta_2O_5 Surface of Anodized Tantalum Film

Figure 19

completely obliterated it to optical microscope visibility. This etch removed about 25Å of oxide. No firm explanation of this phenomenon has been established, but will be studied further if this crystalline structure appears on other units.

3.3 Radiotracer Studies of Diffusion in Other Thin Film Capacitor Systems

3.3.1 Discussion

Requisites that must be met to make radiotracer techniques applicable to diffusion studies of thin film capacitance systems are presented in section 3.1. Systems other than the Ta-Ta_2O_5 system that are or have been under investigation are reported in the next four sub-sections.

3.3.2 Boro Alumina Silicate Glass on Aluminum

Thin-film boro alumina silicate glass deposited from metal organic vapors has proved to be a reliable dielectric and passivating material.

In a preliminary evaluation of the suitability of this glass for diffusion studies by radiotracer techniques, films of this glass were deposited on vacuum evaporated aluminum, a dielectric and bottom electrode combination that has found practical usage for capacitors of thin film circuitry. Because of aluminum surface oxidation, strong interference colors were not produced. An attempt was made to establish a thickness versus wavelength of minimum reflectivity curve as discussed in section 3.2.4, but the weak interference colors produced such broad minimum reflectivity nulls that the accuracy was unacceptable. Because of lack of a

suitable non-destructive method for dielectric thickness measurement, this fabrication is inapplicable to radiotracer diffusion studies.

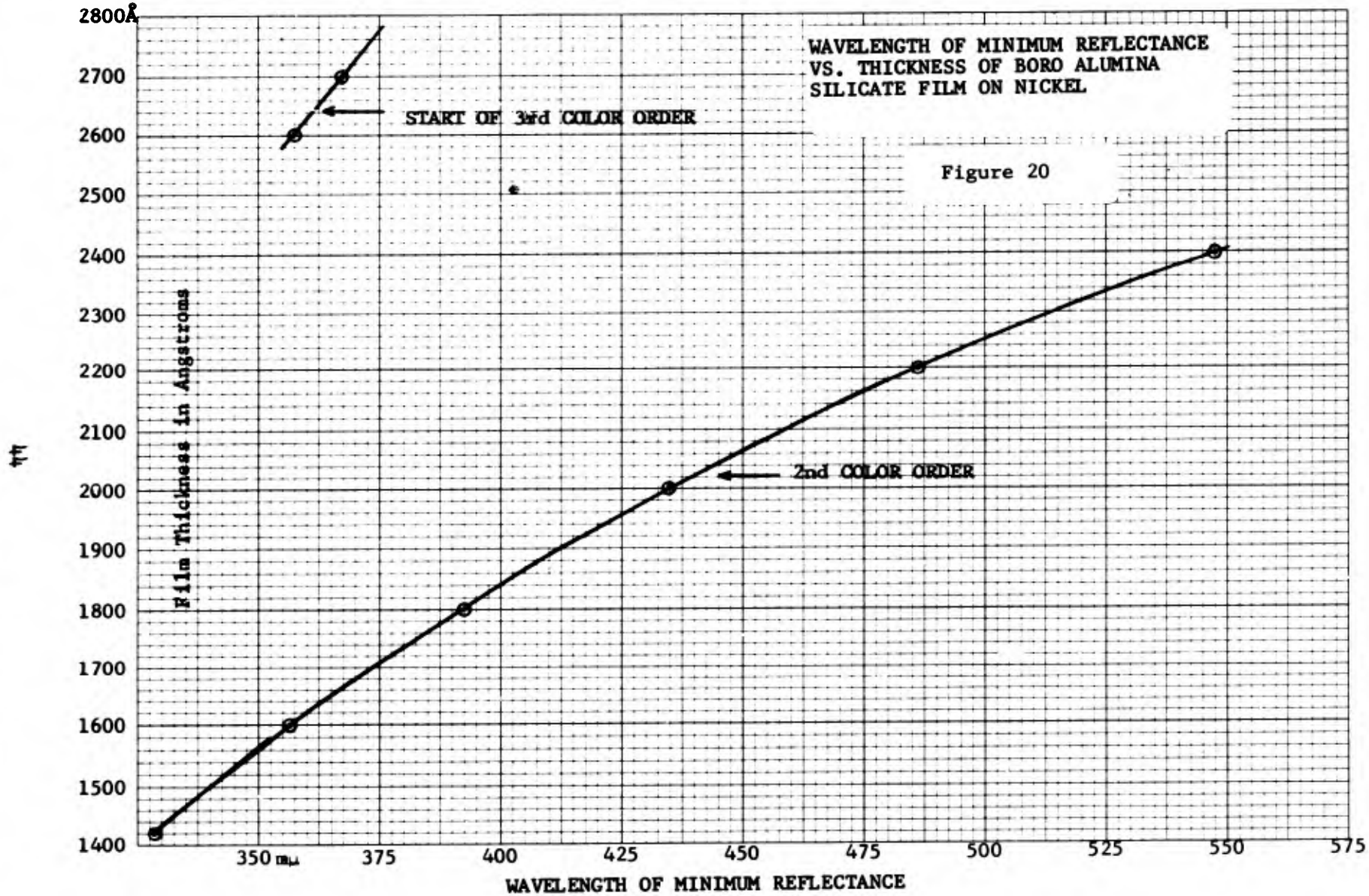
3.3.3 Boro Alumina Silicate Glass on Nickel

Thin film nickel has been used successfully as electrodes for practical capacitor fabrication utilizing thin-film boro alumina silicate glass as a dielectric.

For radiotracer studies, a series of units was prepared with 500Å to 4300Å boro alumina silicate films applied to 1000Å nickel films. The nickel was previously vacuum evaporated onto glazed ceramic substrates. Interference colors produced by this arrangement were sufficiently strong that accurate minimum reflectivity nulls and thickness measurements could be obtained using the modified Beckman Model B Spectrophotometer discussed in Section 3.2.4. It was found that second order colors were produced by glass thicknesses ranging from about 1300Å at the blue end to about 2300Å at the red end of the spectrum. Because this range of thickness is suitable for capacitor dielectric use and because optical thickness measurements proved to be more consistent in the second order than in other orders, many points were established in the 2nd order and a plot of the dielectric thickness versus wavelength of minimum reflectivity was established (Figure 20).

It was anticipated that boron, with its high thermal neutron cross section of 3990 barns, would contribute to radiation destruction of the boro alumina silicate film and this anticipation was further confirmed by a statement in "Handbook of Glass Manufacture,"[4] that intense neutron irradiation of boron glasses destroys the glass network and physical destruction ensues. As a cursory

[4]Tooley, F., "Handbook of Glass Manufacture," Vol. II Section XV, p. 53, Ogden Publishing Co., N. Y. 1960


WAVELENGTH OF MINIMUM REFLECTANCE
VS. THICKNESS OF BORO ALUMINA
SILICATE FILM ON NICKEL
Figure 20
START OF 3rd COLOR ORDER
2nd COLOR ORDER
Film Thickness in Angstroms
2800Å
2700
2600
2500
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
350 mμ
375
400
425
450
475
500
525
550
575
WAVELENGTH OF MINIMUM REFLECTANCE

investigation of this potential destruction, two units having films of boro alumina silicate were irradiated at Oak Ridge for one hour at a flux of 7×10^{12} neutrons/cm^2/sec. These had 1000Å thick nickel film vacuum deposited on one polished side of a fused quartz substrate. Boro alumina silicate glass was then applied to the nickel in thicknesses of 2420Å for unit ABS 1 and 1715Å for unit ABS 2. Wavelength of minimum reflectance readings were made before and after irradiation and the results reported below.

UNIT	λ Before Irradiation	λ After Irradiation
ABS 1	377 mμ	378 mμ
ABS 2	555 mμ	571 mμ

Because unit ABS 2 has its interference color in an extremely sensitive region (red), it is not unusual that the optical change is more pronounced than the change of unit ABS 1. Theoretically, alteration of the glass network will change its refractive index, which will produce a change in wavelength of minimum reflectivity. Although the radiation alteration of the boro alumina silicate units investigated appears to be minor, it is not established whether or not this alteration is sufficient to negate the feasibility of radiotracer study of this material.

Boro alumina silicate glass films on a nickel base may be etched uniformly and at a conveniently controllable rate by use of dilute HF. It has been established that an 0.5% HF aqueous solution etches at a rate of 50Å a second, 0.1% solution etches at a rate of 15Å a second and a 0.03% solution etches at a rate of 4Å a second.

3.3.4 SiO on Nickel

Vacuum evaporated SiO has been considered as a dielectric material for metal diffusion studies employing radiotracer techniques.

A series of units was prepared, having SiO films ranging in thickness from about 500Å to about 3000Å. The SiO was applied over 1000Å vacuum deposited nickel films deposited on Corning 7059 glass substrates. First order interference colors were fairly vivid but colors of second and third order films (about 1000Å to 3000Å) degraded to a golden tan color having very little color variation with variation of film thickness.

In an attempt to etch incrementally the SiO films, many combinations of HF, HNO_3 and H_2SO_4 were studied as were various strong caustic solutions. All attempts were completely unsuccessful. In no case was the etch uniform, a powdery condition usually developed on the oxide surface and in many cases the nickel underlay was seriously attacked or loosened from the substrate.

As has been stated, first order SiO colors are vivid and are therefore suitable for wavelength of minimum reflectivity readings by means of the spectrophotometer arrangement discussed in section 3.2.3, but thicker films are unreadable. However, manufacturing experience indicates that first order thickness SiO used as a dielectric material for thin-film capacitors produces a very high incidence of "leaky" capacitors. Because in this investigation electrical stress is applied to the dielectric material, this is a serious impediment.

The detriments associated with SiO were so overwhelming that this material was dropped from consideration for radiotracer studies.

3.3.5 SiO_2 Thermally Grown on Si Substrates

A dielectric film of SiO_2 grown bulk silicon by thermal oxidation has many applications in the electronic component industry for passivation, masking and capacitor fabrication. SiO_2 films produced in this way are under consideration for radiotracer metal diffusion study and preliminary investigation has been started.

Interference colors of this film are very vivid in thicknesses up to 1 micron or more and are suitable for thickness measurement by the minimum reflectivity method.

The SiO_2 film may be conveniently and uniformly etched in an aqueous solution of 1 ml. of 49% HF in 50 ml. of 20% ammonium biflouride at a rate of approximately 20Å per second.

To determine the susceptibility of SiO_2 film to thermal neutron alteration, two arsenic doped Si-SiO_2 units were irradiated at Oak Ridge for one hour at a flux of 7×10^{12} neutrons/cm^2/sec. The SiO_2 films were approximately 2500Å and 3000Å in thickness and had minimum reflectance measurements made before and after irradiation. No change in wavelength of minimum reflectance was detected indicating that the refractive index changed little or not at all. This is an indication that little or no structural alteration occurred.

3.3.6 Conclusions and Plans for Future Work

Eight weeks stress at 22½ volts d. c. and 125°C is insufficient to change significantly capacitance or dissipation at 1000 kc or leakage at 10 volts d. c. of thin-film Ta-Ta_2O_5

capacitors. Initial tantalum film thickness of test units was 2340Å to 2730Å, anodizing was at 82 volts, electrical stress was with the tantalum positive and d. c. leakage measurement was with the tantalum positive.

Since 50%, or 10 out of 20 Ta-Ta_2O_5 units, failed immediately upon application of electrical stress, it can justifiably be concluded that these units failed for reasons other than metal diffusion into the dielectric, because diffusion did not have time to occur. Since two more units failed within two weeks of thermal and electrical stressing and similar units that did not fail displayed no significant change in electrical stressing, it seems justifiable to conclude that these failures also were not the result of metal diffusion across the metal-oxide interface.

The two units that failed within the two weeks of stressing displayed no electrical irregularities precursory to failure and the 8 units that have not failed in 8 weeks of stressing have shown no significant change in electrical characteristics.

The second series of Ta-Ta_2O_5 units will be anodized at 60 volts rather than at 82 volts. The resulting decrease in dielectric thickness should produce a greater change in electrical characteristics for a given degree of diffusion of tantalum into the oxide. Also stress conditions (electrical and thermal) may have to be made more severe to produce measurable diffusion in an experimentally practical stress period.

Ta-Ta_2O_5 units presently on stress will be continued at present stress levels unless it is decided advisable to accelerate diffusion by raising the stress level.

The investigations of metal electrode diffusion into boro alumina silicate glass and into thermally grown SiO_2 as a mode of failure will be continued.

APPENDIX

Activity Production Calculation

a. Weight of Ta film: 1.56×10^{-4} gm

b. Activity desired: 2×10^{4} disintegrations/sec./unit

c. Calculated Irradiation Time Requested:

Solve for S

$$S = \frac{A}{N.F.C.}$$

A = activity production (dis/sec/sample)

N = number of atoms of target

F = neutron flux (neutrons/cm^2/sec)

C = activation cross section (cm^2/atom)

S = saturation factor

$A = 2 \times 10^{4}$ dis/sec/unit

$$N = \frac{1.56 \times 10^{-4} \text{ gm}}{181 \text{ gm atom wt}} \times 6.02 \times 10^{23} \text{ atoms/mole} = 5.3 \times 10^{17}$$

$F = 7 \times 10^{12}$ neutrons/cm/sec (Oak Ridge)

$C = 19 \times 10^{-24}$ cm^2/atom

$$S = \frac{2 \times 10^{4}}{(5.3 \times 10^{17})\ (7 \times 10^{12})\ (19 \times 10^{-24})} = 2.8 \times 10^{-4}$$

Solve for t t = irradiation time in days

$$S = 1 - \epsilon^{-\lambda t}$$

$$\lambda = \frac{0.693}{T_{\frac{1}{2}}}$$ T = half-life in days = 112

t = .047 days or 1.13 hours

Because irradiation periods at the Oak Ridge facility are in increments of one hour, one hour was requested.

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*
Motorola Inc.
Semiconductor Product Division
Phoenix, Arizona

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP

3. REPORT TITLE
Failure Mechanisms at Metal Dielectric Interfaces

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Technical Documentary Report, Progress 6 March 1964 - 5 June 1964

5. AUTHOR(S) *(Last name, first name, initial)*
Peterson, David

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
October 1964	50	4

8a. CONTRACT OR GRANT NO.
AF 30(602)-3266
b. PROJECT NO. 5519
c. Task No. 551902
d.

9a. ORIGINATOR'S REPORT NUMBER(S)
RADC-TDR-64-359 - Second Quarterly

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

10. AVAILABILITY/LIMITATION NOTICES
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11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY
Rome Air Development Center

13. ABSTRACT

The interface between a metal and a dielectric is representative of the type of interface found in microelectronic circuitry.

Capacitors using thin film dielectrics have been used as a means of studying the changes induced in a metal-dielectric interface by electrical and thermal stress.

Thin film capacitors which were exposed to 100 percent R.H. before top electrode evaporation showed larger changes under stress than similar units which were heated prior to electrode deposition.

In general, all capacitors decreased in capacitance, dissipation factor and T.C.C. after stressing.

Thin film capacitors stressed at 80 percent R.H. and 40 volts d.c. showed only minor changes in electrical properties.

High temperature stress studies of thin film capacitors have been initiated.

Thin film anodized Ta_2O_5 capacitors have shown little change in electrical properties under low temperature stress. Ta_2O_5 capacitors have been prepared for radioactive tracer studies, and will be stressed at higher levels than the first group.

Preliminary study indicates that radioactive tracer techniques may be used in the study of diffusion occurring at the metal-dielectric interfaces, where anodized Ta_2O_5, SiO_2 thermally grown on a silicon metal, and gas plated boro alumina silicate glass film are the dielectrics.

DD FORM 1 JAN 64 1473

Unclassified
Security Classification

Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Reliability (Electronic and Electrical Equipment) Failure Mechanisms Metal Films Dielectric Films Surface Properties (Interface Properties)						

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