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MENLO PARK, CALIFORNIA

June 1966

Quarterly Report No. III | *Covering the Period 1 March to 31 May 1966*

STUDY OF THIN DIELECTRIC FILMS FOR THERMO-DIELECTRIC ENERGY CONVERSION

Prepared for:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER
MOFFETT FIELD, CALIFORNIA

CONTRACT NAS2-3197

By: C. PELTZER

SRI Project FMU-5671

Approved: A. E. GORUM, ASSOCIATE EXECUTIVE DIRECTOR
PHYSICAL AND INDUSTRIAL SCIENCES

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CONTENTS

LIST OF ILLUSTRATIONS

ABSTRACT

This report covers the third quarter's effort on the study of thin dielectric films for thermo-dielectric energy conversion. The objective of this program is to obtain a better understanding of the physical mechanisms underlying the response of certain thin film dielectrics during temperature cycling and under high electric field conditions.

The temperature dependence of the dielectric constant of thin Mylar film has been investigated in vacuum as a function of frequency. Thermal cycling runs in vacuum between $-50^{\circ}C$ and $30^{\circ}C$ gave values of β of the order of -0.0001; these values are in apparent contradiction to the positive value of the order of +0.001 to be expected from the capacitance bridge measurements.

I INTRODUCTION

This report covers the third quarter of a research program on the use of thin dielectric films for thermo-dielectric energy conversion.[1]

The principle underlying this energy conversion scheme may be described as follows: radiant energy incident on a capacitor is stored in the dielectric in the form of polarization energy and is converted into usable electric energy upon discharge of the capacitor in a load circuit. The purpose of this study is to investigate the behavior of certain thin film capacitors under the conditions required by this energy conversion scheme, i.e., thermal cycling and high electric fields. Our first objective is a careful examination of the temperature dependence of the dielectric constant ϵ of the dielectric material, in particular of any divergence between the values of $\beta = \frac{1}{C}\frac{dC(T)}{dt}$* obtained from static measurements and those deduced from thermal cycling runs.[2] Parameters to be considered here, aside from the temperature, include the applied electric field, cycling period, environment, and nature of the electrodes.

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* C(T) = capacitance of the capacitor at the temperature T.

II WORK ACCOMPLISHED - ANALYSIS

The test setup used for the thermal cycling experiments carried out during this quarter is basically the same as that described in the first and second quarterly reports. The major difference is that the internally mounted furnace in the equipment previously used has now been replaced by a 650 watt movie flood iodine lamp.

A comprehensive series of capacitance measurements in vacuum was completed. The sample used for these experiments was a 1/2 mil Mylar film capacitor having evaporated copper electrodes. The capacitance versus temperature [C(T)] for this material was measured over the range of -50°C to 90°C at 1 kcps, 50 cps, and 5 cps, with a 1.5- or 3-volt source (Figs. 1 through 4).

A similar series of runs was made on the same material, using a 300-volt d.c. bias. The results of the measurements made at 1 kcps are in agreement with those previously made and reported in which the bridge technique was used for measurement. The only apparent effect of the d.c. bias was a shift of the C(T) curves in the high direction. The change was on the order of 10% higher, in this case, with no noticeable change in the slope of the curves. On the other hand, the 50 cps and 5 cps measurements showed a large decrease in the slope of C(T) at lower temperatures--a capacitance drop of about 3% at 50 cps (resp. $< 1\%$ at 5 cps) from room temperature to -50°C, as compared with a drop of $> 6\%$ at 1 kcps. Static measurements using the standard discharge method were also performed over the same temperature range with charging voltages of 6 and 300 volts. At temperatures above 70°C, considerable leakage occurred. No appreciable decrease in the capacitance was observed from room temperature down to -50°C. We should also note that the static capacitance was higher than the ones obtained at the different frequencies.

The open circuit test[2] in vacuum gave the following results: when the capacitor was charged (6, 18, and 300 volts) for the first time at the low temperature (-50 to -80°C), upon rapid heating we observed a very small drop in the voltage across the capacitor, corresponding to a positive β. On subsequent thermal cycling, however, negative β's were observed. As long as the temperature of the capacitor stayed below ~ 60°C, the magnitude of the effect was small (~ 1%); going to higher temperature resulted in a large, mainly irreversible drop of the voltage, indicative of leakage.

Using the same sample we have made thermal cycling runs in vacuum (Figs. 5 and 6) from -50°C to 30°C. The temperature was measured with a thin wire chromel-alumel thermocouple. Voltages of 100 to 500 volts were applied across the capacitor, and the current flowing in and out of the capacitor during the thermal cycling was obtained from the voltage drop across a 100 kΩ resistor placed in series with the capacitor. This voltage was picked up through a high impedance (1MΩ) meter, amplified, and registered with the thermocouple emf on a Visicorder. In a series of runs, we obtained records indicating a negative β of the order of -0.0001 to -0.0002, in contrast to an expected β from the bridge measurements of +0.0012. We should note that on the basis of the 5 cps capacitance curve, or the static one, we would expect a β of less than 0.0002 in absolute magnitude.

For this reason, we cannot conclude on the basis of these data alone that there exists a conflict between static and thermal cycling β's. Before reaching such a conclusion it will be necessary to obtain further comparative data on other materials besides Mylar. We are presently engaged in obtaining such data. "Alcar," another plastic film, is being investigated first. Inorganic films will be studied next. We have set up a deposition apparatus for forming titanium dioxide films by reactive vapor deposition and we also plan to study silicon monoxide and aluminum oxide films in the coming quarter. The deposition apparatus for titanium dioxide is the same as the one reported by Feuersanger.[3] Titanium dioxide is produced by low temperature hydrolysis of titanium tetrachloride: $TiCl_4 + 2H_2O \rightarrow TiO_2 + 4HCl\uparrow$. Nitrogen is being used as carrier gas and platinum as substratum.

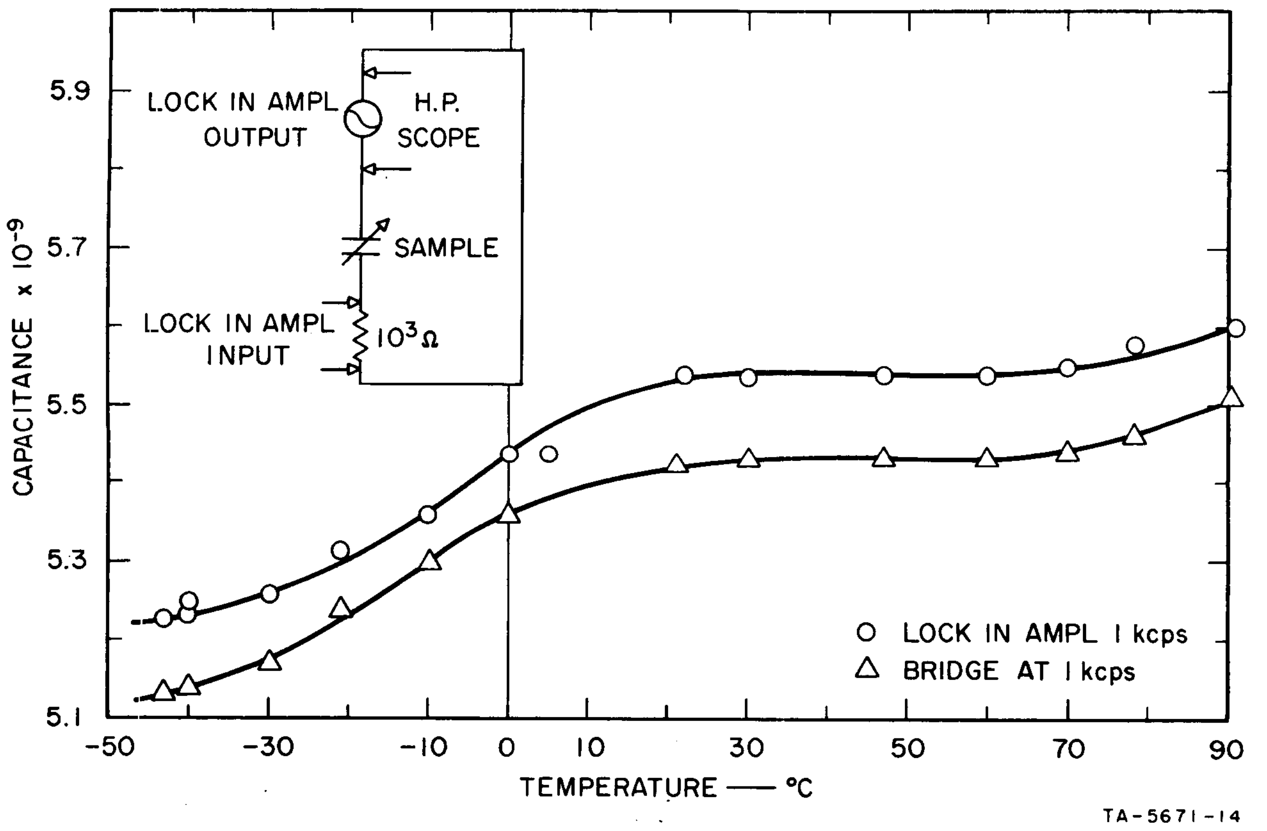

FIG. 1 CAPACITANCE VERSUS TEMPERATURE AT 1 kcps

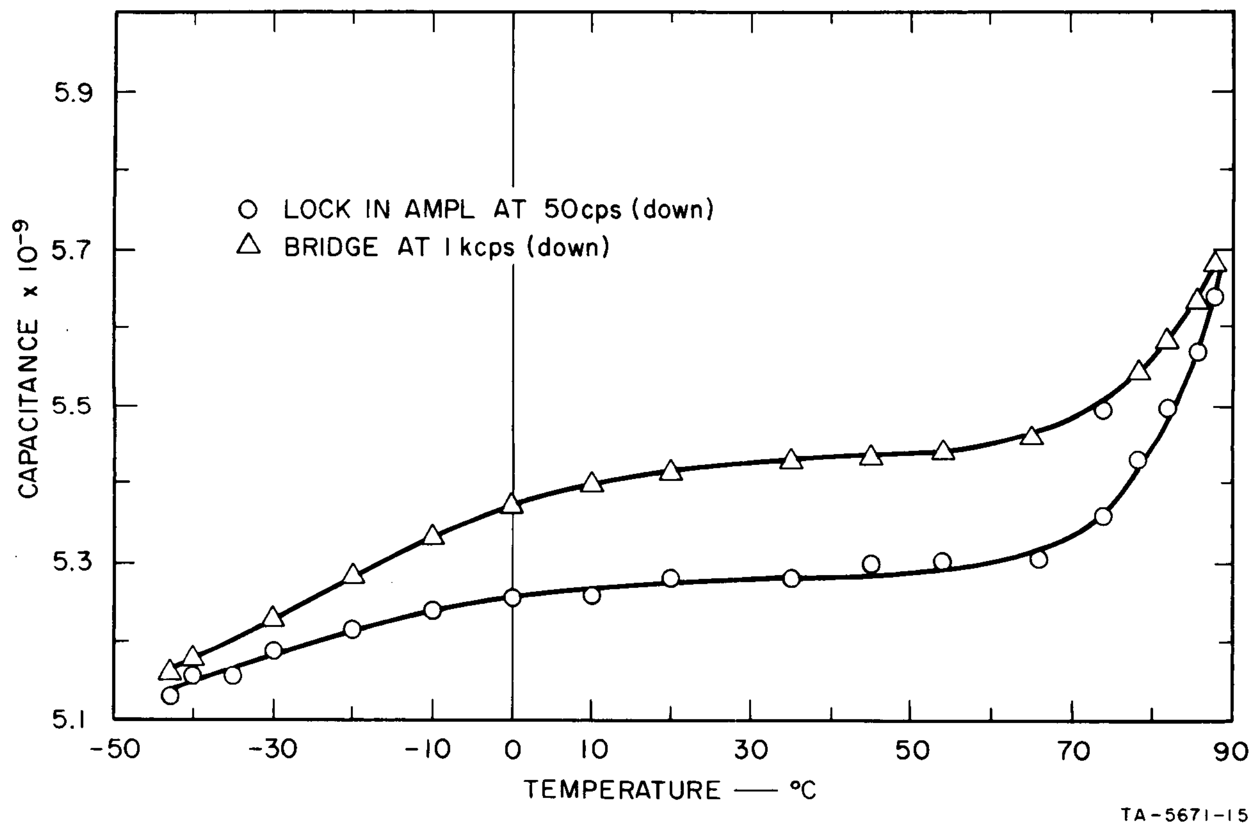

FIG. 2 CAPACITANCE VERSUS TEMPERATURE AT 50 cps

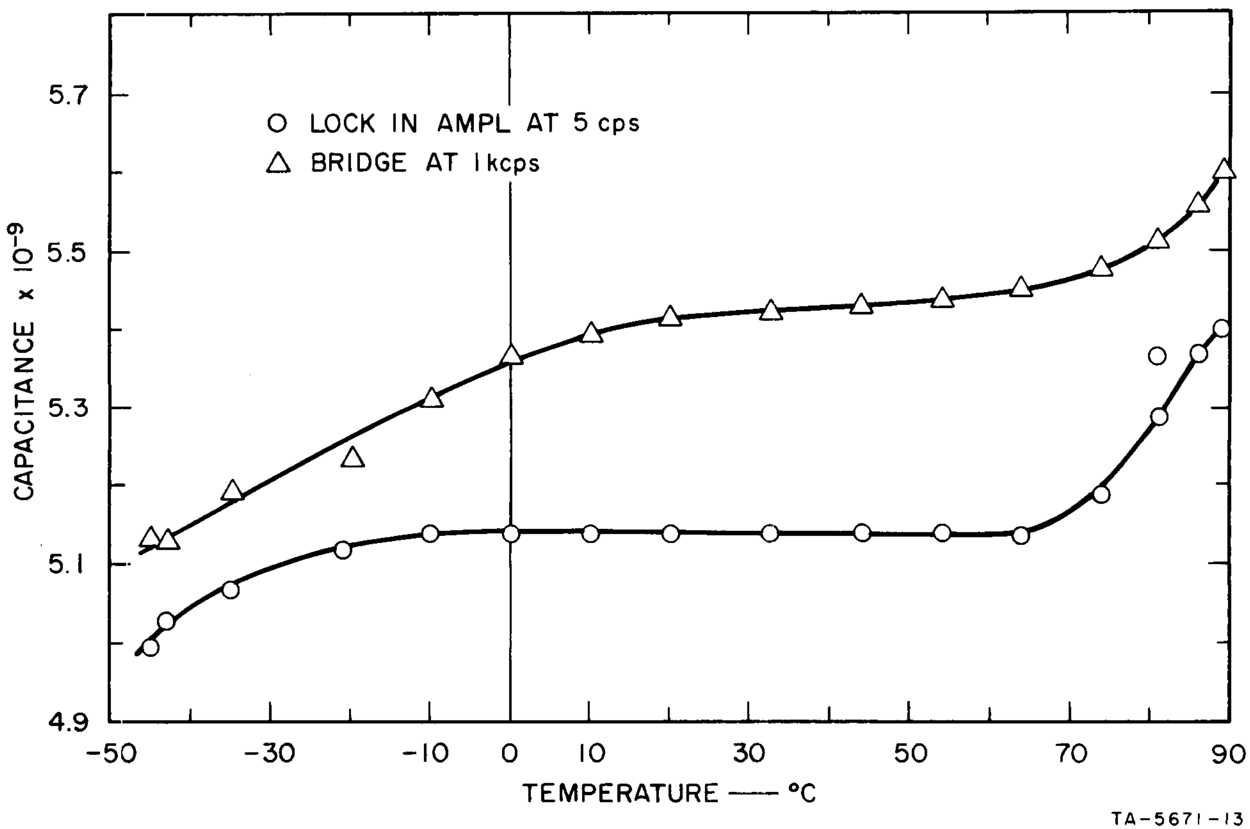

FIG. 3 CAPACITANCE VERSUS TEMPERATURE AT 5 cps

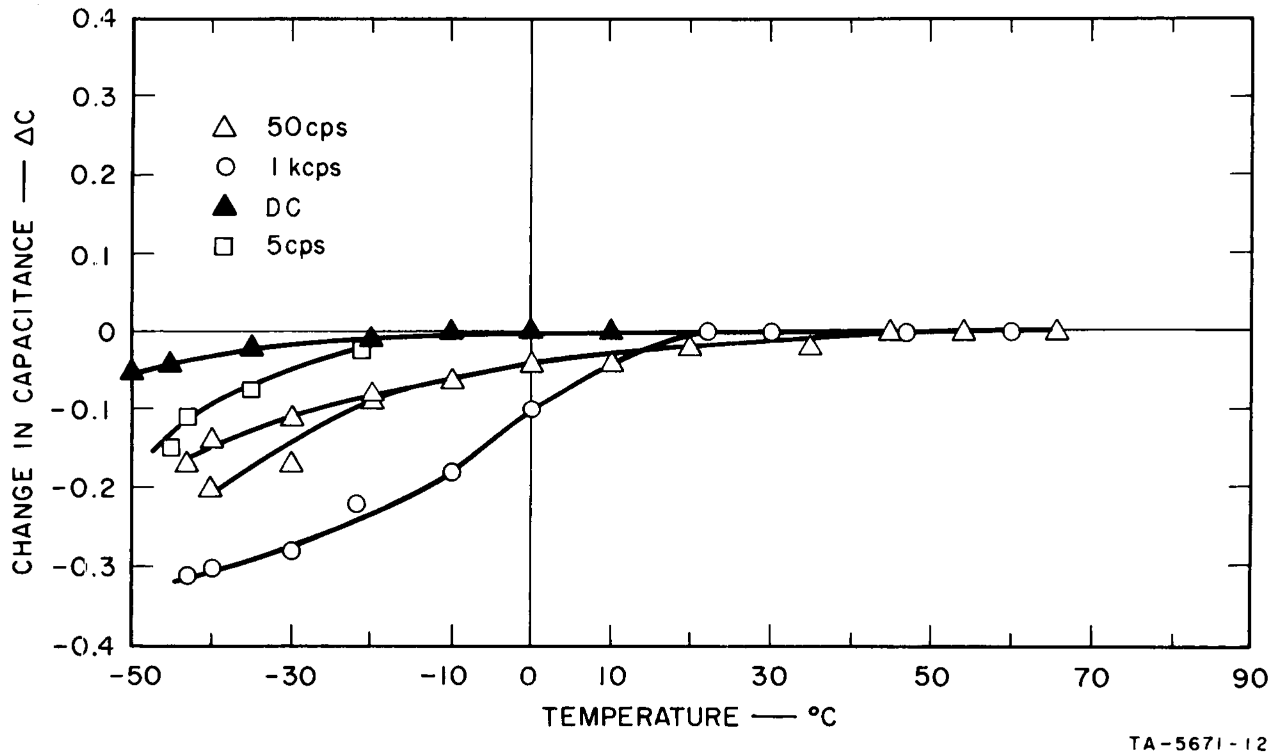

FIG. 4 CHANGE IN CAPACITANCE VERSUS TEMPERATURE

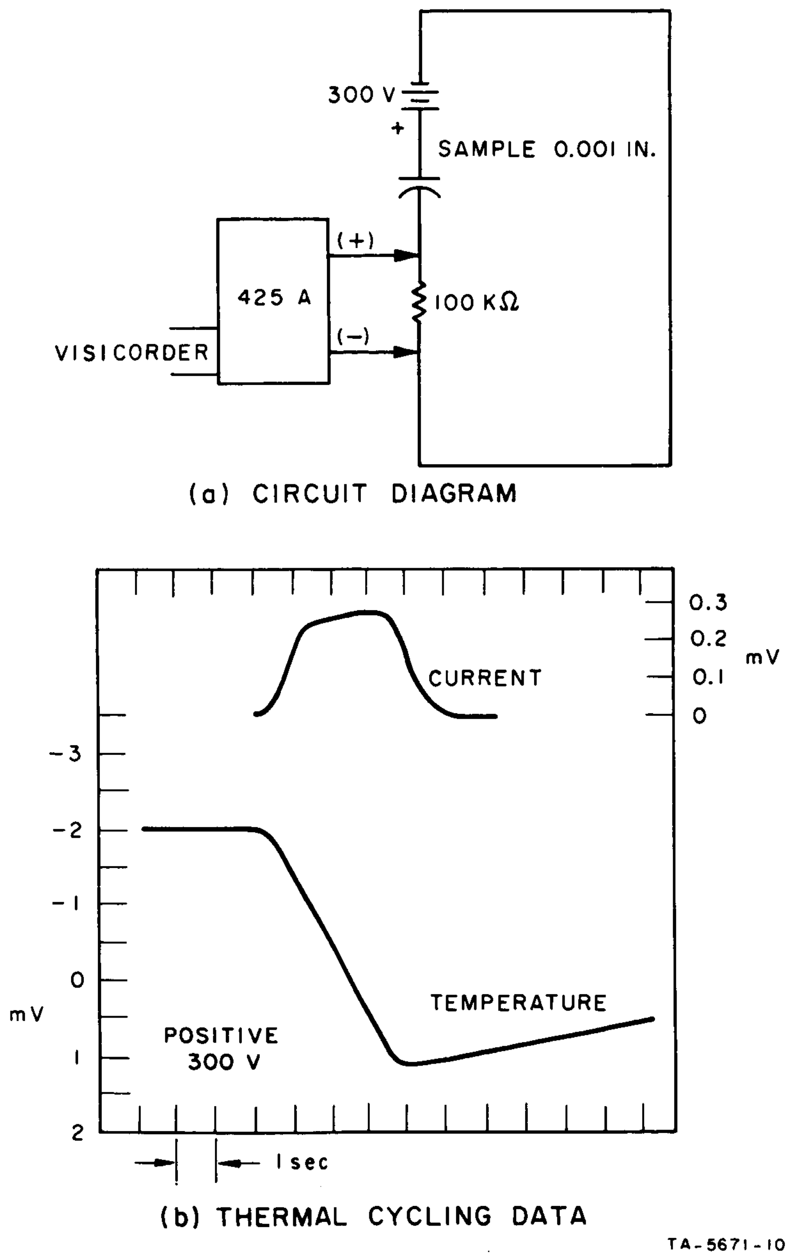

FIG. 5 THERMAL CYCLING (Positive Voltage)

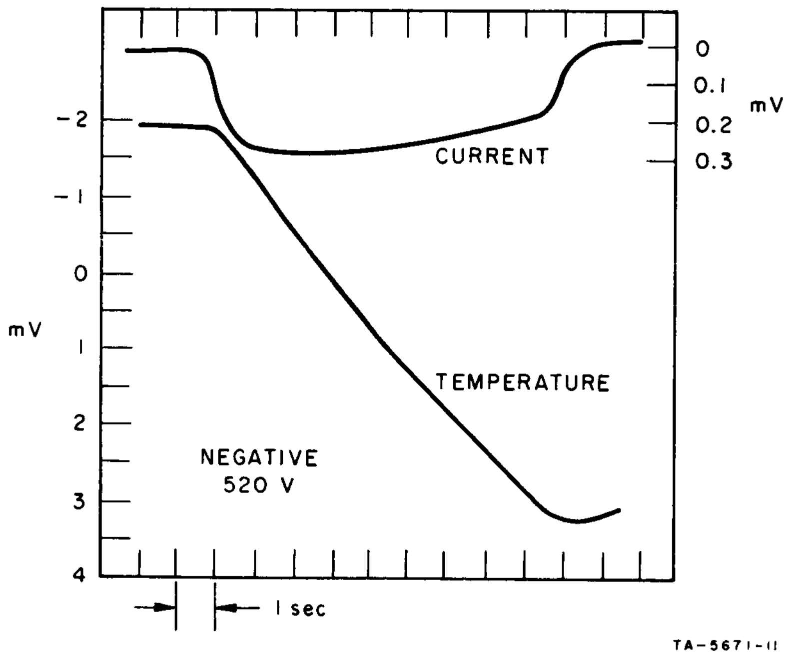

FIG. 6 THERMAL CYCLING (Negative Voltage)

REFERENCES

1. B. H. Beam, NASA TMX-50, 136;
   J. D. Childress, J. Appl. Phys. 33, 1793 (1962).

2. B. H. Beam, et al., "Experiments on radiant energy conversion using thin dielectric films," (preprint).

3. A. E. Feuersanger, "Titanium dioxide films prepared by vapor reaction," Proc. IEEE, Dec. 1964, p. 1463.