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HIGH ENERGY STORAGE CERAMIC CAPACITOR

Third Quarterly Report

-for-

UNITED STATES ATOMIC ENERGY COMMISSION
Contract No. AT(30-1)-1963

Report Prepared by

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DISCLAIMER

DESCRIPTION OF AIMS

The chief purpose of this contract is the development of ceramic materials with high dielectric constant and high dielectric strength values, and suitable for use as dielectrics in capacitors capable of storing large amounts of energy in small volumes.

The following material and capacitor performance specifications are the goals of this work:

Dielectric constant: in the order of 1500

Working voltage gradient: 500 volts/mil or greater

Size of unit: to store 1000 joules per unit

Working voltage: 100,000 volts

Pulse operation: 10 microseconds to charge
.2 microseconds to discharge

Energy recovery: 75% per discharge pulse

Pulse repeat rate: one per minute

Life: 10,000 (minimum) charge-discharge cycles

Upon the successful development of materials and processing techniques, four prototype capacitors would be produced and delivered to the contractor.

INTRODUCTION

Work performed during the third quarter of this project has continued to follow the plan given in the First Quarterly Report. At this time, no major obstacles to final success can be seen. The principal advances made during the third quarter are concerned with the production of large ceramic pieces for the final test capacitor. A combined attack was made on this problem by the S. E. C. Department and the Research Laboratory. This has resulted in the development of a ceramic piece which has been successfully dried and fired, and is now undergoing contour machining.

Work in the Electronics Laboratory on material development has resulted in samples of 98.9% of theoretical density. Electrical tests have continued, and samples have been measured to 300 volts per mil stress. The dielectric constant continues to show an increase with increasing density.

A meeting was held at the Laboratory for Insulation Research, M. I. T., with Dr. A. von Hippel and his staff. Besides a considerable exchange of information, several very helpful suggestions were made at this meeting. For example, it was suggested that a gel binder be investigated, and this has been done. The information gained in discussing dielectric strength was put to use in revising test procedures and analyzing data.

In addition, samples and information have been exchanged with Dr. H. E. Weaver of Varian Associates. The possibility of using nuclear magnetic resonance or electron paramagnetic resonance spectroscopic analyses is being discussed.

The work continues under the direct technical responsibility of F. G. Recny, H. C. Craig, and D. A. Lupfer, with the following individuals making major contributions:

I. Material Development and Sample Preparation - Electronics Laboratory

V. A. Russell, assisted by R. J. Hill

II. Sample Testing - Electronics Laboratory

D. A. Lupfer and C. E. McCain

III. Capacitor Ceramic Fabrication - S. E. C. Department

S. E. C. Department: F. G. Recny and R. C. Knapp

Research Laboratory: P. D. S. St. Pierre and A. G. Pincus, assisted by J. A. Smith

Others who have contributed during the third quarter include the following:

E. C. Henry - Electronics Laboratory
A. A. Lachner - Electronics Laboratory
A. V. Illyn - Electronics Laboratory
R. C. DeVries - Research Laboratory
R. L. Fullman - Research Laboratory

I. MATERIAL DEVELOPMENT AND SAMPLE PREPARATION - V. A. Russell

Attempts to make barium-strontium titanates of high density have been continued with several lines of attack being pursued. Studies of the atmospheres used during firing have continued, along with experimentation with the procedures used in slip casting. In addition, a binder study has been initiated to find the binder which will give the best samples both in appearance and density.

Since oxygen has previously been determined to be the atmosphere giving the highest density samples, the study has been confined to this gas on the heating cycle. The temperature range through which the application of the oxygen is most effective has been the principal concern during this period. Experiments have shown that the temperature of initiation of the oxygen atmosphere is less important than the point at which it is suspended. The densities of samples where the oxygen was shut off at 1350°C on the temperature rise curve are better than those where oxygen was stopped at 1300°C, and also better than those whose oxygen atmosphere was continued to the soak at 1450°C.

As was mentioned previously, studies have also been made on the value of an argon atmosphere applied during the cooling cycle. After the oxygen firing, the samples had a brownish color which could be attributed to an oxygen excess. If this were true, an argon atmosphere applied during part of the cycle would help rid the sample of this excess oxygen. This has proved to be the case and excess argon appears to remove too much oxygen. Finding the correct amount of argon and the range of its application still have not been completely evaluated.

By the application of the best atmospheres, densities up to 5.585 g/cc have been attained. This is 98.9% of theoretical density as shown in Table I.

Several 2-1/4" diameter x 1/2" thick samples of calcined barium-strontium titanate were slip cast using the ingredients given by J. F. Murray in the NRL report No. 4483, "Slip Casting of Barium Titanate". The composition of the ingredients is given in Table III of the Second Quarterly Report and the make-up of the slip is shown in Table II of this current report. These samples were dried and then fired in a 4-1/2" diameter high-temperature porcelain combustion tube in an oxygen atmosphere. The densities attained on these large samples are shown in Table I. There is some evidence that a firing temperature of 1480°C gives poorer densities than 1450°C not only on these large samples but on some of the small ones. Due to other variables in these firings, an analysis cannot be made at this time.

Many of the samples which have been fabricated with Hyform binder have shown spots presumed to be the result of binder aggregation. These spots appear lighter than the sample in reflected light, and darker than the sample in transmitted light and are believed to be macroscopic voids. The source of these imperfections could be due to segregation of binder during the drying step. In attempts to correct this condition, two other binders and methods of adding them are being investigated. Methocel binder, as suggested by G. Economos of M. I. T., was used according to the procedure outlined by J. F. Murray and R. M. Callahan in the NRL Report #4176 published in 1953. Methocel binder has the advantage over Hyform in that the slurry from the ball mill gels quite rapidly upon heating to 85°—90°C, thus preventing separation during drying. The amounts of various ingredients used for the preparation of the ceramic pieces by this method are given in Table III. The dried powder is dampened with an aerosol and water solution just prior to pressing. Results on the samples made with methocel are encouraging and are also listed in Table I.

The other binder tried was PVA (polyvinyl alcohol) which is added to the dried powder in the amount of 4 ml. of a 10% solution per 100 grams of powder just before pressing. This method was suggested by Traub, Best, and Baldwin of Titanium Alloy Manufacturing Company. The results to date have not been very good with this method and it will probably be abandoned.

It was believed that the effects of temperature could well be further explored for this $(Ba,Sr)TiO_3$ composition. The temperature limitations of conventional electrically heated kilns using oxidizing atmospheres is presently 1500° - 1600°C. This range sets practical limits on the life of the heating elements by requiring excessive replacement. Consequently, a furnace was constructed and operated by H. C. Rothenberg for attaining temperatures beyond 1500°C in air.

This furnace was an inductively heated ceramic cylinder suitably insulated. The inner shell serving as the heat source was a ZrO_2 ceramic cylinder. It was separated from the Al_2O_3 outer cylinder by a loose packing of insulating ZrO_2 grain. The induction coil was wound outside the Al_2O_3 ceramic. The assembly was placed on ZrO_2 brick, covered with ZrO_2 brick, and surrounded with firebrick.

The inner ZrO_2 cylinder was heated inductively after first being heated to a temperature at which its electrical conductance reached some proper value. This was done by placing a graphite cylinder inside the ZrO_2 cylinder, and using this graphite for the susceptor. When the ZrO_2 was hot enough to serve as the susceptor, it electrically shielded the graphite. If necessary, the graphite could then be removed. Temperatures were measured with a Leeds and Northrup optical pyrometer sighted on the sample surface. In trial runs, temperatures greater than 2700°C were obtained. For better temperature control in the experimental range of 1500° - 1900°C, and to eliminate heat shock in the heating cycle, it was found better to leave the graphite cylinder in place.

After encountering problems in reaction at elevated temperatures with the (Ba,Sr)TiO_3 ceramics and different support materials, $SrTiO_3$ ceramics were fabricated to serve as tables. A $SrTiO_3$ crucible was also cast and fired, and this was inverted and used as a protective cover.

The test temperatures selected were 1750° and 1850°C. A temperature of 1750°C is between the liquidus and solidus lines for $BaTiO_3$ - $SrTiO_3$ solutions shown in Figure 1 of the First Quarterly Report. A temperature of 1850°C is well into the liquid region.

In the first trial, the samples were heated to 1750°C, held for 10 minutes, and cooled. The x-ray pattern taken after this heating showed no difference from ceramics fired in the usual way. The samples were blue-gray and porous in appearance, indicating considerable reduction and poor sintering.

The second set of samples was heated to 1850°C, held for 15 minutes, and then cooled. This treatment had a pronounced effect. Shrinkage was pronounced; the diameter was decreased nearly one-half. X-ray examination showed two different solid solutions. One had a slightly smaller cell than for a conventional firing of the ceramic, but the other solid solution had a much smaller cell. In other words, $BaTiO_3$ was lost from the sample in both large and small amounts. It is believed that the $BaTiO_3$ was lost to the $SrTiO_3$ setter and crucible. The sample was highly discolored and reduced, and was highly conducting.

Additional work is planned using atmosphere control and longer reaction times in the temperature range 1600° - 1800°C.

Future Work

High density, void-free samples are still the immediate goal of the material development. Further work will be done in evaluating the range of application of the argon atmosphere. In addition, more work will be done on methocel binder and a comparison of it with Hyform. Additional larger samples will be made by slip casting, and then used for electrical tests. The effect of particle size on density will also be investigated further.

II. SAMPLE TESTING - D. A. Lupfer and C. E. McCain

The problems of ballistic testing were discussed with A. von Hippel and D. A. Powers of M. I. T., and it was concluded that the method should be carefully scrutinized before proceeding further with the tests. The principal objection to the ballistic method is that an attenuator of the Ayrton type is required at such high voltages. This can introduce capacitances and inductances giving a time constant problem in such a divided circuit. On the other hand, the ballistic method appears to be the only one giving information directly related to the application.

Consequently, the apparatus was modified and a new set-up made. Calibrations of the Leeds and Northrup No. 2285e galvanometer were performed with a General Radio 722-NQ variable capacitor for a standard (using the difference between two settings) and measuring the D.C. voltages by three standard methods. On the basis of averaging 12 consecutive readings for each test, the galvanometer deflection constant was determined and found to be reproducible to ±1.5%. Tests of calibrated polystyrene capacitors (General Radio Type ZCOP-12) verified the above calibration. All other layered dielectric capacitors gave higher capacitance values than their A.C. calibrations because of dielectric absorption. The performance to date indicates that ballistic readings are accurate within ±3%. Since the previous deflection constant differed by only 1.1% from the new value, the previous data do not require correction.

After calibration tests were concluded, the samples were measured as before. The results of the data are summarized in Figures 1 through 4. It will be noted that the AD samples broke down at much lower stresses than the AE samples. Breakdown for the AD sample shown occurred at 183 vpm and lower, while the breakdown value for the AE sample was 310 vpm. These tests show the only departure from the pattern observed to date that dielectric strength increases with increasing density. However, the AD samples chosen had extensive crystal growth, and this large crystal form is of lower electrical strength than a smaller crystallite ceramic of equal density. This is consistent with findings with other ceramics. With this exception, each increase in density has given an increase in dielectric strength. It may also be seen that the dielectric constant shows an increase with each increase in density, again with the exception of the AD series having large crystallites. This adds emphasis to the value of continuing work to increase the density of the ceramics.

The tests of one group of materials are omitted from this report. This group is the AB series which were earlier tested at 60 cycles and reported in the Second Quarterly Report. These samples showed a very high loss, and a wide range of capacitance values were obtained in the ballistic testing. The samples behave electrically as a two-layer dielectric, so they will be examined and analyzed carefully to determine any structural anomalies before reporting the data.

An additional test was performed on all samples reported here, and this was a corona test. The circuit is shown in Figure 5. This test, which has been developed for insulation testing, is well described in the literature(1). Its value is in differentiating the conduction current to a high degree. Any conduction processes, such as corona and prebreakdown currents, which occur in pulses containing more than 2 x 10^{-14} coulombs (practical) of charge can be detected in this measurement. Referring to Figure 5, any action which displaces greater than this amount of charge in the capacitor C_x is evidenced by a potential across the resonant circuit inductance. This may be amplified and displayed, and the use of a resonant circuit to give an underdamped transient simplifies the method of amplification.

To date, the circuit has been used in conjunction with the ballistic testing to give information on D.C. effects. It was found that nothing unusual happened with the application of D.C. potentials. Prebreakdown pulses of a large amplitude were noted on most samples just prior to breakdown. This corresponds to the behavior found with other solid dielectrics in uniform fields. It is concluded that there is nothing striking about the breakdown mechanism in these high dielectric constant materials.

The corona test will be discontinued on D.C. measurements, but will be used with 60 cycle A.C. voltages. This will serve as a quality check on voids and other discontinuities within the ceramics.

Further resistivity tests required that most attention be paid to the slopes of the ρ vs. $\frac{1}{T}$ curves at low temperatures. This is a difficult range of measurement because of the problems of measuring extremely low currents accurately, separating surface and volume effects, and preventing moisture and atmosphere attack. Consequently, it was felt that photoconductivity tests could be used to advantage in determining low energy levels, and apparatus is being readied to perform these tests.

Future Work

Future work will be concerned with continuing tests on ceramics of higher density and smaller crystallite size, and performing more short-time dielectric strength tests. Physical structure analyses of failed pieces will be performed, and the most probable causes of breakdown identified. Photoconductivity studies will be made to determine the pattern of energy levels for the ceramics under the various fabrication conditions.

III. FABRICATION OF CERAMIC CAPACITOR SHAPE - F. G. Recny, P. D. S. St. Pierre, J. A. Smith

Introduction

The Second Quarterly Report outlined the procedure whereby a test shape approximately 20" in diameter and 1-3/4" thick was successfully slip cast (Trial IV, page 25) and dried (Drying and Preparation for Burn Out, page 26). The subsequent burn off and firing treatment to which this piece was subjected, as well as additional slip casting tests, are reported here. Preliminary testing showed there were certain limitations on processing equipment available at the S. E. C. Department, particularly with the kiln used for firing the large shape. Consequently, during this quarter the Metallurgy and Ceramics Research Department of the General Electric Research Laboratory was enlisted in the attack on the problem of forming large shapes. This made available the specialized equipment as well as the skills and consultants available in the Ceramics Laboratory, Mechanical Equipment Development Shop, and other units of the Research Laboratory. Intensive study is being given to the problems of fabrication, drying, firing, and machining. Because of the particularly critical nature of the casting and drying problems, the trials and solution are given in detail below.

Experimental Results - S. E. C. Department

1. Burn-off cycle

The ceramic piece pictures in Figure 34 of the Second Quarterly Report was placed in a circulating hot air oven and subjected to the burn-off schedule given in Table IV. Although this schedule could eventually be shortened, it was felt necessary to remove the high temperature volatile materials in this first trial at a slow rate in order to prevent damage to the piece.

2. The test shape to be bisque fired was placed in a side heated globar kiln and the top of the piece covered with a layer of -35 mesh Norton Zirconia grain I. The sides of the test piece were protected with alundum saggers which acted as shields to prevent direct radiation from the globars.

The firing schedule was as follows:

a. Room temperature to 550°F at 80°F per hour
b. 550°F to 850°F at 70°F per hour
c. 850°F to 2100°F at 50°F per hour
d. 2 hour soak at 2100°F
e. Cool from 2100°F to 1300°F at 100°F per hour
f. 1300°F to room temperature (natural cooling of the kiln) in three days.

When the piece was removed from the kiln, it was cracked into several individual pieces with approximately 1/2" to 5/8" separation between each piece indicating the cracking had occurred during the early part of the heating cycle. Figure 6 illustrates the condition of the piece after the bisque fire.

The time-temperature firing cycle for this shape was reasonable, but it appeared that too large a temperature gradient existed in the kiln during the heating period. It was felt the kilns available at the Research Laboratory would be more suitable for firing these large shapes, particularly when controlled atmosphere firings are tested.

3. Additional Slip Casting Tests

To determine the reproducibility of the method previously used, and to study further the slip preparation and casting procedure, the following tests were made:

A) Trial V

The procedure as outlined in Trial IV of the Second Quarterly Report was duplicated. The filled plaster mold cracked while it was being placed in the vertical position. Since the mold started to leak the test was discontinued.

B) Trial VI

The procedure from Trial IV was again used with one exception. The pressure maintained during fill and setting up was reduced from 10 psi to 7-1/2 psi. The cast piece cracked as previously described in Trial I and II and Figures 31 and 32 in the Second Quarterly Report.

C) Trial VII

The material from previous test castings that had cracked was dried at 110°C in a circulating air oven and passed through a pulverizer to break up the lumps. It was then remilled with the original amounts of Solutions I and II and cast. The result was extremely poor since the slip would not set up and remained very thixotropic in the plaster mold.

D) Trial VIII

Another duplicate run as in Trial VI was tried except the wet milling procedure was changed since it appeared to yield a more fluid slip with the same specific gravity. A milling time of three hours, overnight aging of approximately 16 hours, and 5 minutes milling previous to dumping the slip from the ball mill was followed. The cast piece again was cracked as in Trial VI.

It was observed that the cast piece exhibited a thixotropic nature even though it had been in the plaster mold for approximately 18 hours. This condition was also noticed on previous trials, and it was felt this undesirable feature could be associated with the subsequent cracking of the piece during the initial drying stages. It was then decided to eliminate the glycerine in Solution 1 (Table III, page 32, Second Quarterly Report) and substitute water. This new composition is shown as Solution 1 in Table VI of this report.

E) Trial IX

A batch in which water was substituted for the glycerine was milled and cast at 7-1/2 psi. The mold was opened after approximately 18 hours. The cast piece had no cracks, no signs of being thixotropic, and appeared to be set up quite hard. The piece was left in the mold and allowed to set inclined at about 30° from the vertical for 16 days (during vacation shutdown). The drying cycle for this piece is reported in Table V.

This dry piece was then submitted to the Research Laboratory for firing.

Experimental Results - Research Laboratory

1. Preliminary Tests

To answer the doubts about the feasibility of producing pieces larger than 8" in diameter, a 12" disc was slipcast by the open mold method. It was fired to maturity at 1400°C and is apparently free of cracks, as shown in Figure 7. One such final fired 9-1/2" disc has been turned over to the Capacitor Department for their experiments on application of electrodes. Another 9-1/2" disc has been retained at the Research Laboratory for similar purposes.

2. Firing and Grinding Tests

The full size 20" diameter disc which had been cast and dried by the S. E. C. Department was fired in the Research Laboratory kiln. The results are shown in Figures 8 and 9. Slight surface cracks on the underside ran from the periphery for 1/4"; however these were removed during grinding of the piece. There was some scaling of the top face, but this was also removed during grinding. The disc shrank to 15" diameter, as shown in Figure 9.

The production of this body has proved that a large disc can be fired in existing equipment without serious cracking. Experiments have been undertaken on the turning and grinding of prefired and finish-fired pieces. Preliminary experiments have shown that material prefired to 1150°C machines well, and fully matured ware can be ground with either diamond or silicon carbide.

3. Sample Preparation

Details on the preparation of samples are given in the following summary:

A) Plaster Molds

In addition to the pressurized feed molds described on Page 23 of the Second Quarterly Report, two other casting methods are being tried: gravity type feed, and open mold. The respective molds are shown in Figures 10 and 11. Equipment is also being assembled for vibratory casting of slurries with lower water contents.

B) Materials

Simplification of the original slip formula is being pursued by omitting some of the ingredients of Solution 1 (Table III, Page 32 of Second Quarterly Report). The formula used as currently developed by the S. E. C. Department is given in Table VI.

C) Slip Preparation

All batches have been prepared by wet milling for three hours, aging overnight, and then milling for five minutes before pouring off. Remaining procedures are the same as previously reported.

D) Casting Operations

a) Pressure Type Molds

Initial work was done with molds provided by the S. E. C. Department.

b) Gravity Type Molds

The mold is held together with three C-clamps and set in a vertical position. Slip pressurized to 5 psi is pumped out of the tank through a hose line which is inserted through the opening at the top of the mold. The hose is slowly raised and withdrawn as the mold is filled. Additional slip is trickled into the open riser as needed during the casting period. Feeding requires 1 to 1-1/2 hours; then the piece is left in this position for four hours. One half of the mold is then removed and the remainder set at 60° from the horizontal for drying.

c) Open Type Molds

The plaster mold is placed under the slip-tank and the slip is then run directly into the mold. The mold was kept full by controlling the opening of the valve in the line from the tank. After four hours of setting horizontally, the casting is left on edge at 60° angle for drying.

E) Drying

In the first trials, only room drying techniques were used although effort is currently being directed toward speeding up the drying rate with the aid of an air-circulating oven. It is felt this stage is of the greatest importance. A scaling of the surface has been noted on drying the castings. Although this is a serious matter, there is reason to believe that it will be easily overcome.

F) Firing

Firings have been on a slow heating and cooling cycle, by using a 12" x 18" x 36" General Electric program controlled globar-heated kiln, part of which is shown in Figure 8. For prefiring, the cycle has been 25°/hr heating, holding at 1150°C for one hour, and cooling at 25°/hr. Final firing, heating, and cooling rates were the same, but the bodies were held for one hour at 1400°C.

G) Measurements of Fired Density as a Control Technique

Test samples were made from each batch of slip by casting 1" x 1" x 1-1/2" pieces. Densities were measured using the buoyancy principle. Polished sections were also made. Minor experimental difficulties necessitate further work to confirm the data obtained.

Future Work

The first objective of the Research Laboratory activity is to supply at least one finished disc as soon as possible. The longer range objective is to understand and describe the parameters significant in the process.

The approach will be to investigate concurrently the factors outlined:

1. Slip Composition

   Relation to stability, casting behavior, and uniformity of the as-cast structure.

2. Methods of casting

   Under pressure

   Gravity feed

   Open mold

   Mold design

   Can initial contours be put into casting?

3. Drying

   Setting

   Rate of heating

   Humidity Controls

4. Firing

   Setting and saggering

   Heating rate

Maturing temperature

Cooling rate

Shrinkage

One or two firings

5. Machining

Place in manufacturing sequence -- Compare turning a prefired piece with grinding a fully matured one.

Can finish-fired piece be turned by diamond or cemented oxide tools?

6. Alternative forming methods

Vibratory compacting

Cold press and sinter

Hot press

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   b) Development of Corona Measurements and Their Relation to the Dielectric Strength of Capacitors; R. J. Hopkins, T. R. Walters, and M. E. Scoville

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TABLE I

DATA ON SMALL SAMPLES

Series No.	Sample No.	Pre-treatment and binder	Atmosphere	Temp. of Soak	Time of Soak	Density	% Theor. Dens.
BH	DL-72-RN-SW	calcined at 1300°C, Hyform	O_2(1200-1450)	1450	4 hrs.	5.55	98.2
BC	DL-72-RN-TA	calcined at 1300°C, Hyform	O_2(1200-1450)	1450	4-1/2	5.574	98.7
BD	DL-72-RN-TA	calcined at 1300°C, Hyform	O_2(1200-1350)	1450	4-1/2	5.585	98.9
BI	DL-72-TM-S-TO	calcined at 1300°C 2-1/4" discs slip cast	O_2(1200-1350)	1450	2	5.45	96.5
BJ	DL-72-TM-S-TT	calcined at 1300°C 2-1/4" discs slip cast	O_2(1200-1350)	1480	1-1/4	5.36	94.8
BK	DL-72-TH-TT	calcined at 1300°C, Methocel binder	O_2(1200-1350)	1480	1-1/4	5.54	98.2
BL	DL-72-TH-TV	calcined at 1300°C, Methocel binder	O_2(1150-1300)	1450	4	5.38	95.3
BM	DL-72-TH-TV	calcined at 1300°C, Methocel binder	O_2(1150-1350)	1450	4	5.55	98.2
BN	DL-72-TH-TV	calcined at 1300°C, Methocel binder	O_2(1200-1300)	1450	4	5.39	95.5
BO	DL-72-TH-TV	calcined at 1300°C, Methocel binder	O_2(1200-1350)	1450	4	5.55	98.2
BP	DL-72-TX-UD	calcined at 1300°C, Hyform binder	O_2(1200-1350)	1450	3	5.46	96.7
	DL-72-TX-UD	calcined at 1300°C, PVA binder	O_2(1200-1350)	1450	3	5.20	92.0
	DL-72-UF-UI	calcined at 1300°C, Methocel binder	O_2(1200-1350)	1480	3	5.12	91.0

TABLE II

COMPOSITION OF PURE MATERIAL SLIP

450 gm	Calcined (Ba,Sr)TiO_3
57.5 cc	H_2O
7.6 cc	Solution No. 1
6.1 cc	Solution No. 2
4.2 cc	No. 551 Ross and Rowe Interfacial Modifier

TABLE III

COMPOSITION USING GEL BINDER

910 gm	Calcined (Ba,Sr)TiO_3
547 cc	H_2O
6.8 gm	Stearic acid
13.6 gm	Methyl cellulose
9.1 cc	Octyl alcohol-aerosol OT solution

TABLE IV

BURN OFF SCHEDULE

TOTAL TIME (Hrs.)	TEMPERATURE (°F)
0	Start at 80°F
1/2	125
1-1/2	150
3	200
4-1/2	250
21	275
21-1/2	300
28-1/2	350*
77	400
92-1/2	450
98	500
116	off

* Temperature at which visible signs of volatile materials being driven off were first noticed.

TABLE V

DRYING CYCLE ON TEST SHAPE FROM TRIAL IX*

Air dry one week

24 hours at 85°F

9 hours at 95°F

15 hours at 105°F

57 hours at 125°F

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* Test shape was left in the bottom half of the plaster mold and placed in horizontal position.

TABLE VI

SLIP CASTING SOLUTION

Solution No. 1

905 cc	Distilled water
32 gm	Tannic Acid
20 cc	Triethenolamine
5 cc	Octyl Alcohol

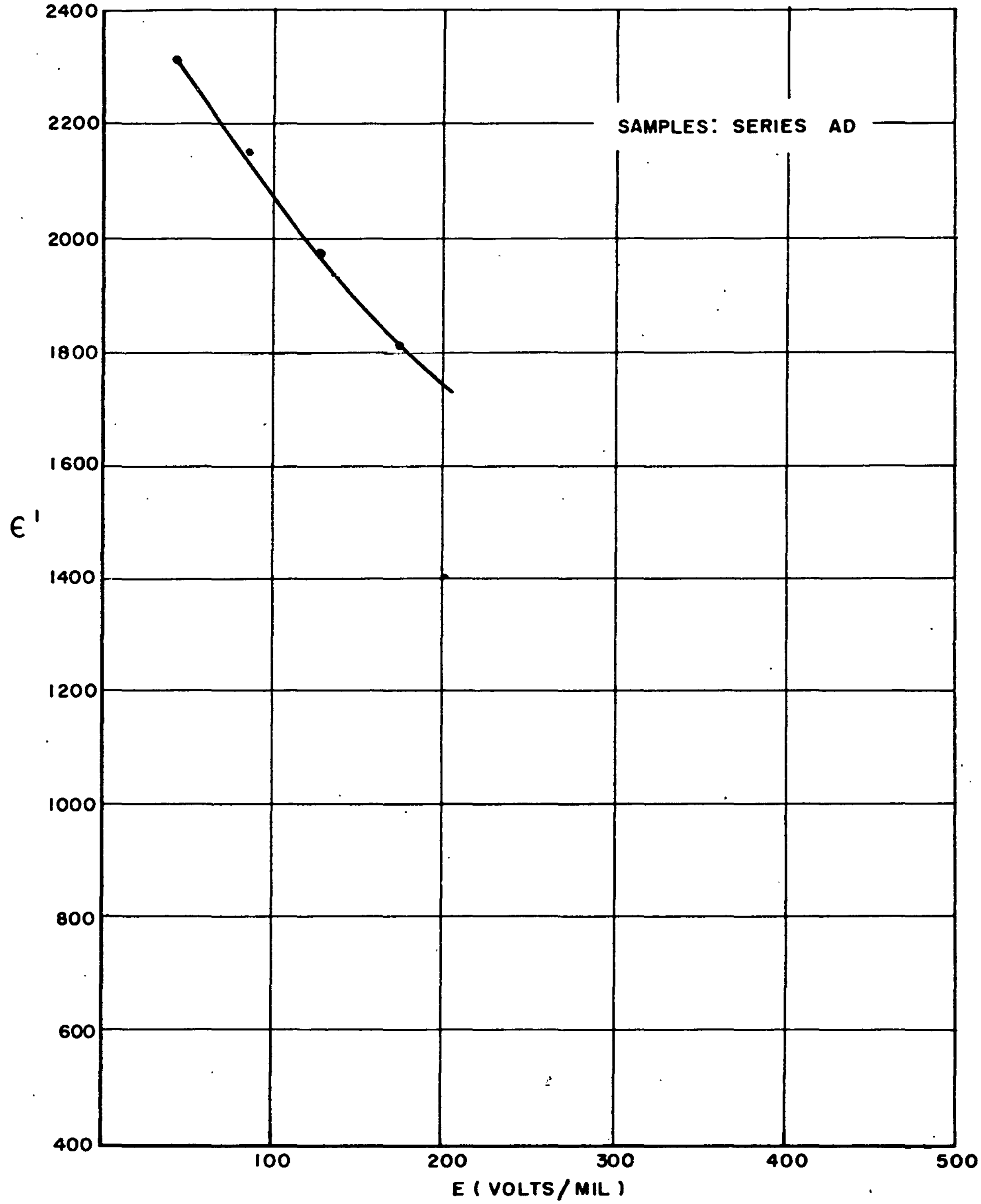
Solution No. 2

10% Polyvinyl Alcohol Solution in Water

(Elvanol 51-05)

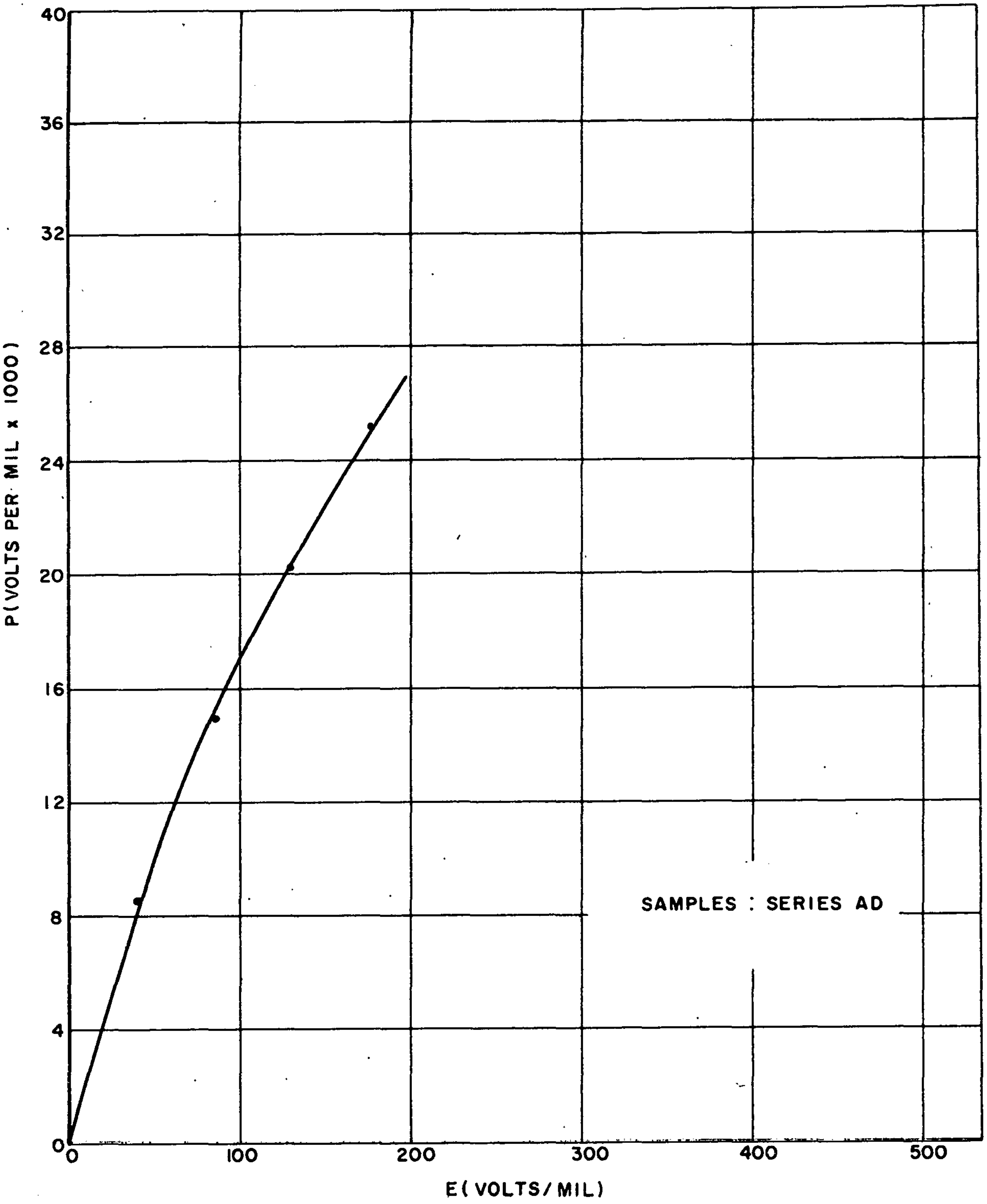
Ball Mill Charge

Barium Titanate TAM	9600 gm
Strontium Titanate TAM	6400 gm
Distilled Water	3105 cc
Solution No. 1	615 cc
Solution No. 2	410 cc
Conc. Ammonium Hydroxide	25 cc
Flint Pebbles	65 lbs. (approx.)



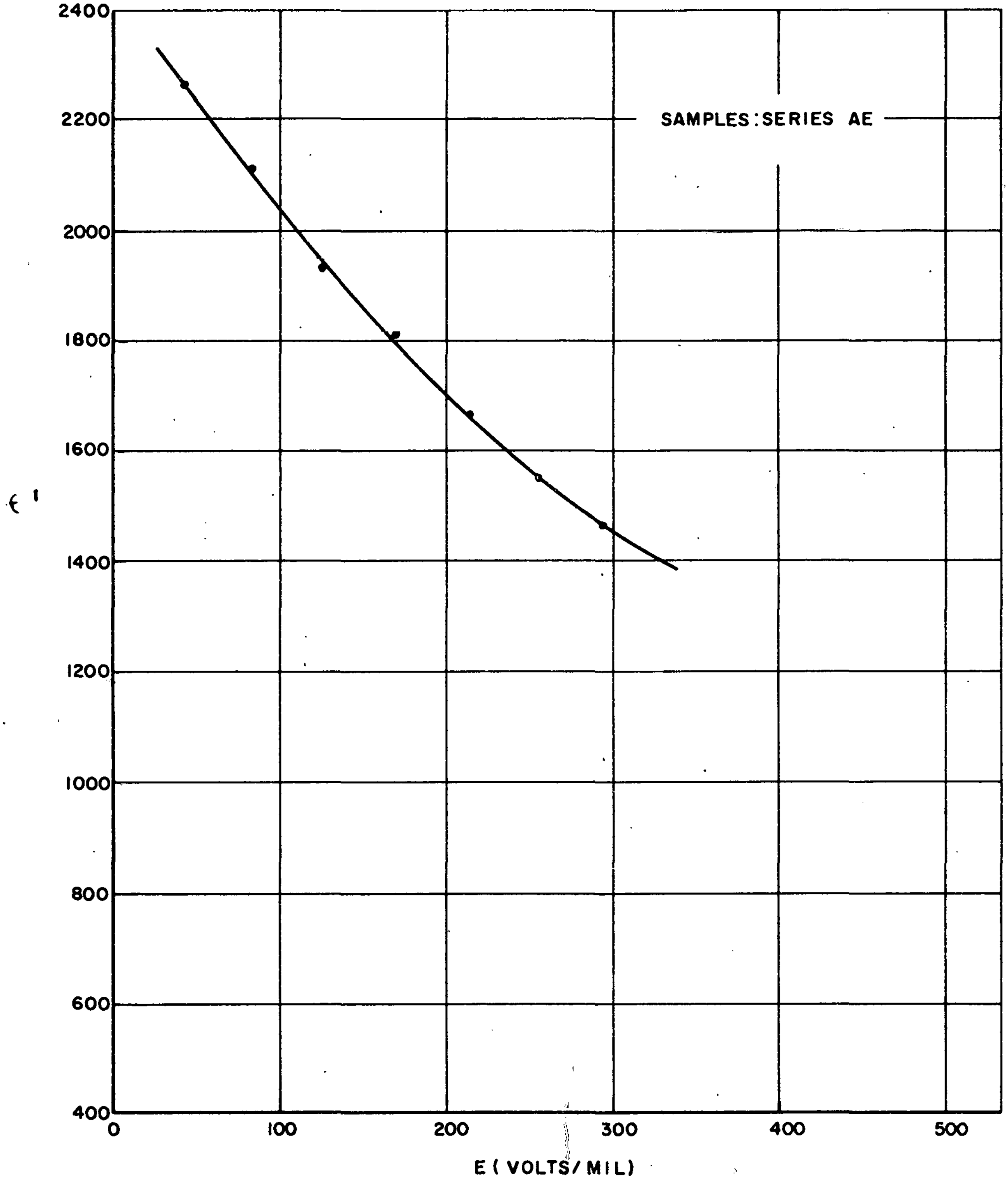
DIELECTRIC CONSTANT VS. FIELD STRENGTH
MEASURED BALLISTICALLY

FIGURE I



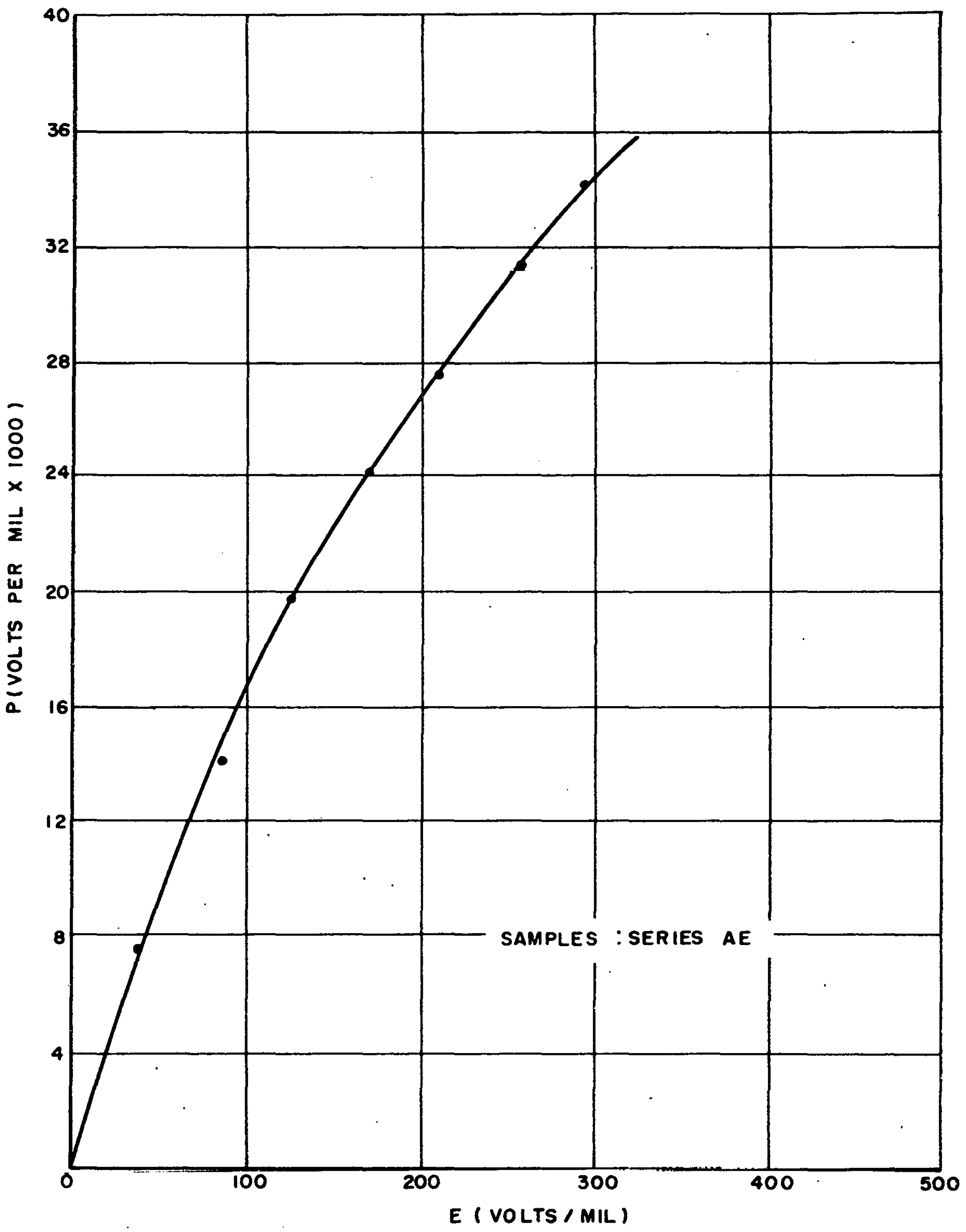
POLARIZATION VS. FIELD STRENGTH
MEASURED BALLISTICALLY

FIGURE 2



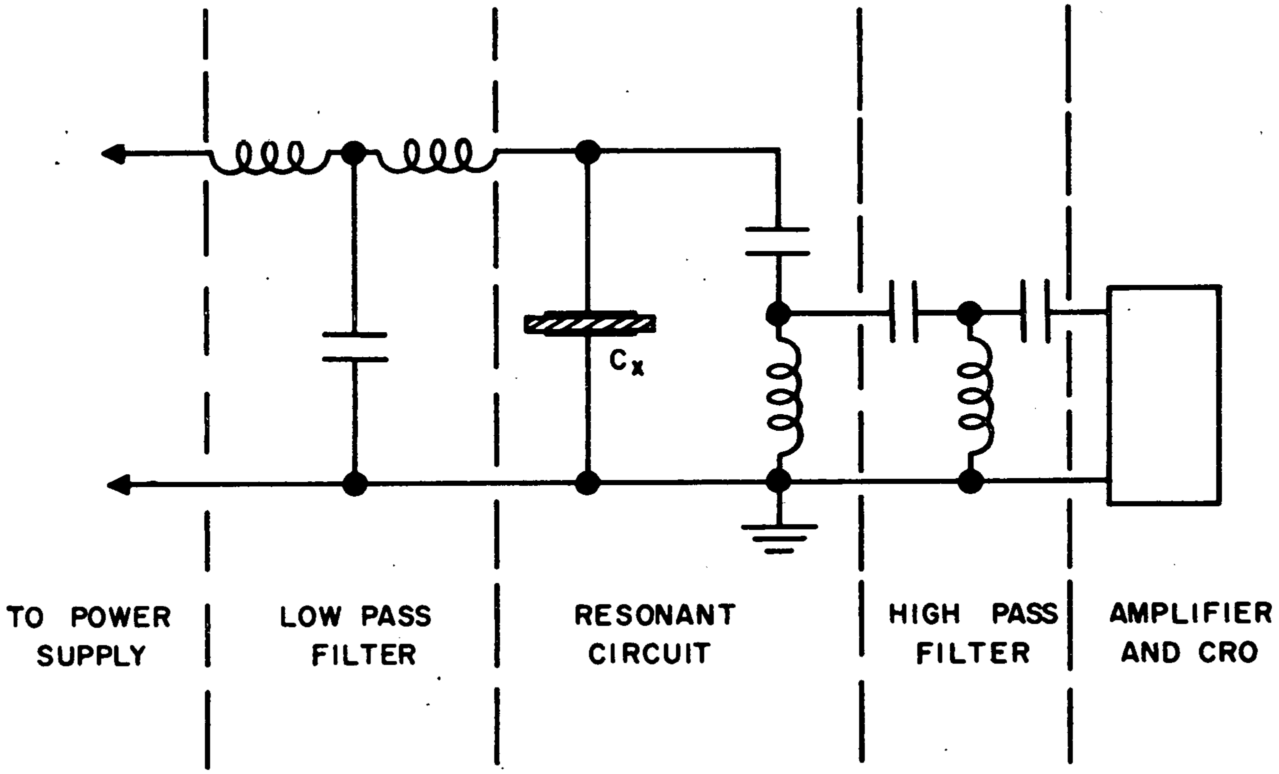
DIELECTRIC CONSTANT VS. FIELD STRENGTH
MEASURED BALLISTICALLY

FIGURE 3



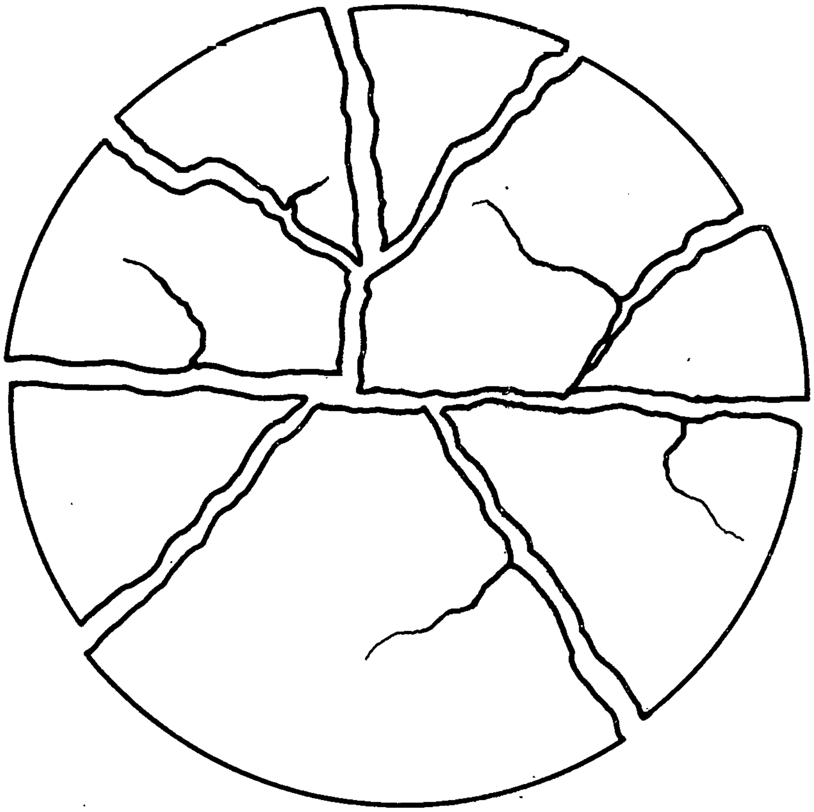
POLARIZATION VS. FIELD STRENGTH
MEASURED BALLISTICALY

FIGURE 4



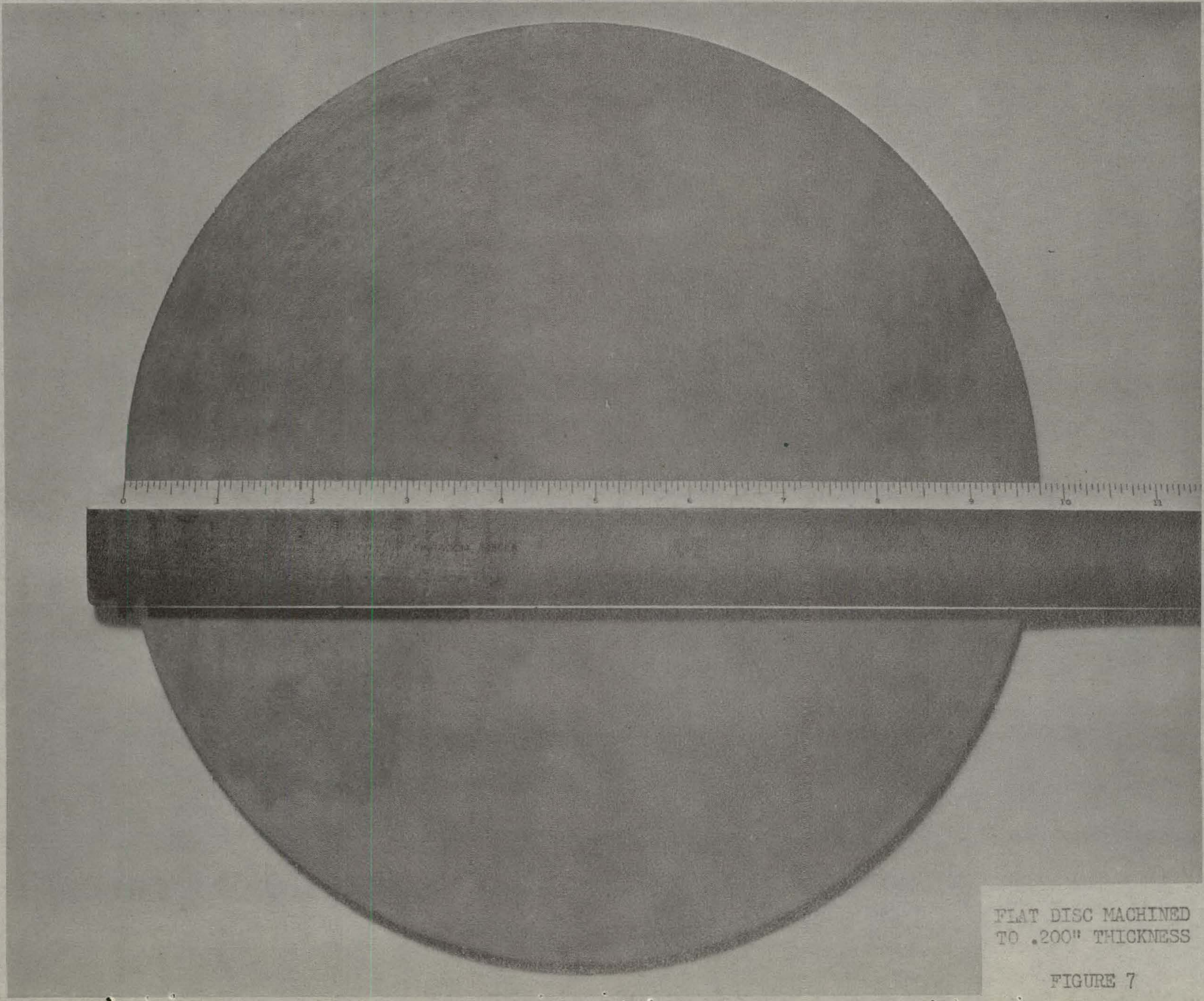
CORONA TEST CIRCUIT

FIGURE 5



TEST DISC AFTER BISQUE FIRE

FIGURE 6

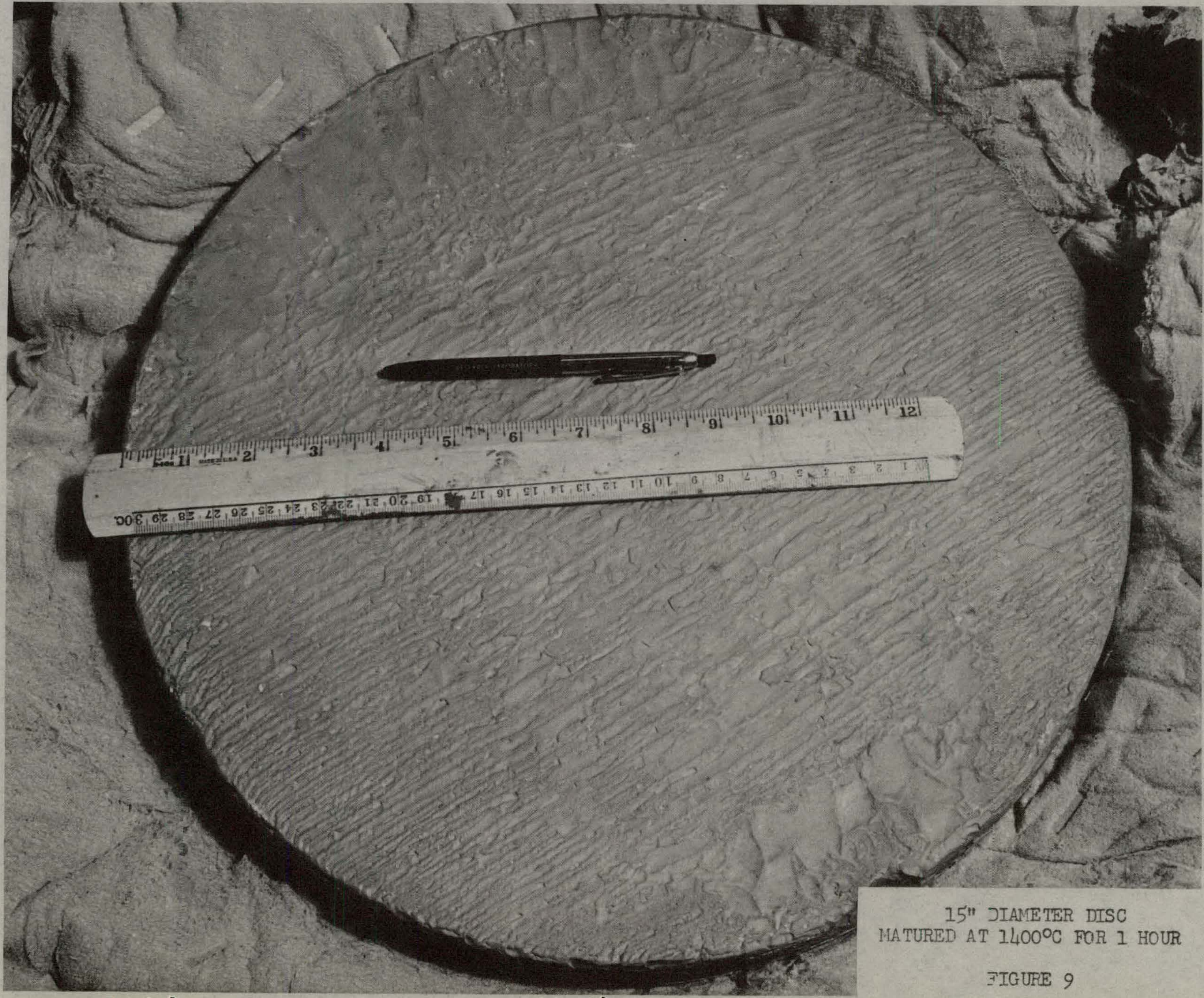


FLAT DISC MACHINED
TO .200" THICKNESS

FIGURE 7

15" DIAMETER DISC SET IN
ZIRCONIA GRAIN IN A GLOBAR KILN

FIGURE 8

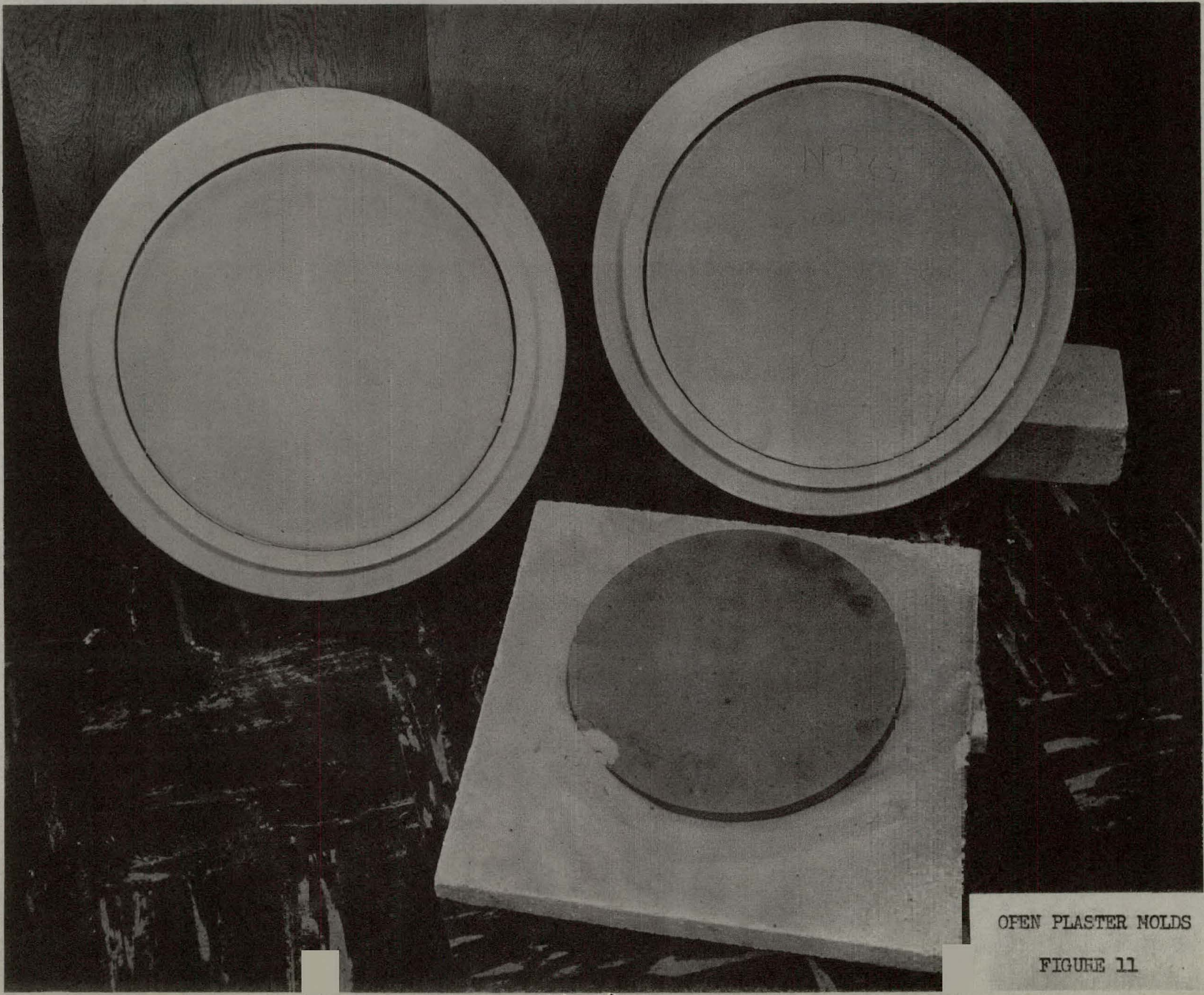


15" DIAMETER DISC
MATURED AT 1400°C FOR 1 HOUR

FIGURE 9

GRAVITY FED PLASTER MOLDS

FIGURE 10



OPEN PLASTER MOLDS

FIGURE 11