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RECEIVING TUBE DIVISION
OWENSBORO, KENTUCKY

QUARTERLY REPORT NO. 4

INTERIM TECHNICAL REPORT ON

The Investigation of Synthetic and Substitute Materials in Domestic Supply for Use as Vacuum Tube Spacers.

For Period - 1 May 1952 to 31 July 1952

Date of Report - September, 1952

CONTRACT NO: NObsr 52535

Submitted by:

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Approved by:

A. P. Haase
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TECHNICAL PERSONNEL WORKING ON THIS CONTRACT
TECHNICAL PERSONNEL ADDED DURING FOURTH INTERVAL

<u>C. T. Durham, Jr.</u>

Date of Birth: February 19, 1927

Place of Birth: Chapel Hill, N.C.

Education and Experience:

U. S. Navy November, 1944 to July, 1946
B. S. Chemistry (1949) University of North Carolina
M. S. Ceramic Engr. (1951) North Carolina State
G. E. August 1951 to present - Chemist Program Trainee

<u>W. R. Schultz, Jr.</u>

Date of Birth: December 19, 1926

Place of Birth: Saginaw, Michigan

Education and Experience:

U.S. Navy February, 1945 to August, 1946
B. S. Chemical Engineering (1951) Purdue University
G. E. February, 1951 to present-Chemist Program Trainee

<u>C. H. Howard</u>

Date of Birth: October 12, 1912

Place of Birth: Hamilton, Missouri

Education and Experience:

A. B. Science (1930) Missouri Wesleyan
M. S. Bio. Chemistry (1932) Oklahoma A & M
Ph. D. Bio. Chemistry (1935) Rutgers University
E. R. Squibb & Sons, 1935-1947

C. B. Kendall Chemical Company, 1948-1949
Lincoln Income Insurance Company, 1949-1952
General Electric Company, January, 1952 to present

ABSTRACT

Physical test data are presented for samples of new or modified materials received and tested during this period. Data on a high-strength asbestos sheet material are discussed and evaluated.

Thermal conductivity measurements are presented with a description of the test conditions and the effect of this characteristic of the spacer on tube performance is discussed.

Tube data for tubes using many of the materials under investigation are presented and some of the apparently limiting factors discussed. Life test data are also presented and evaluated.

Technical manhours during this interval:

R. J. Coswell	309
Paul Dodgen	
A. P. Haase	13
J. C. Riddle	134
C. T. Durham, Jr.	31
W. R. Schultz, Jr.	70
C. H. Howard	74

The total expenditures on this contract through July 31, 1952 have been $20,800.

PURPOSE

The object of the work carried on under this contract is to find or develop a domestic material (or materials) to be used as vacuum tube spacers to replace or supplement the presently used mica.

The primary aim of the work will be to find a suitable material that will compete with mica in cost and ease of fabrication.

The major problems in connection with the fulfilment of this contract are

1. Find a material of suitable strength and flexibility to allow standard assembly techniques to be used
2. A material of such a nature that it may be fabricated to the standard tolerances used for mica at low cost and in large quantities.
3. Physical and chemical properties such that it may be degassed easily and not carry or contain elements or compounds detrimental to any of the other parts, particularly the oxide-coated cathodes.
4. A material or materials which will give equivalent electrical characteristics without major design changes in the structure of the tube, e. g., (emission, transconductance, leakage, capacitance, shock resistance, vibration, microphonics and noise).

General Factual Data

During the fourth interval of this project our efforts have been directed toward obtaining samples of new materials which would eliminate some of the shortcomings of previously tested materials. Also, we have continued to evaluate any new samples that we might receive.

Our efforts to obtain variations and changes in the various types of sheet materials under investigation necessitate prolonged discussions with the suppliers of these materials, and, even after agreements are reached on the type and amount of changes to be attempted, considerable time elapses before the new samples are available for test purposes. For these reasons, the progress, particularly on some types of these sheet materials, has been delayed during this interval.

In the previous report a statement was made to the effect that the Johns-Manville Corporation, supplier of the regular terratex raw material, had refused to make variations in the asbestos sheet stock. Since that time we have been successful in obtaining their cooperation on this work, and they have agreed to make new samples with clay contents from 0 to 8 percent. These samples should be available for evaluation during the next interval.

New samples of terratex type material prepared as handsheets by the TAP Laboratory were received and tested. Sample 146B proved to be the best terratex material tested to date. The test results are tabulated in the Detailed Data Section of this report. Attempts were again made to decrease the amount of gas given off by the material when under vacuum. These tests consisted of treatments with various chemical reagents in an effort to change the surface characteristics, and thus lower the adsorption tendencies of the sheet material. No definite improvements in gas content were observed after these tests, which are described later in the report.

Evaluation of the mica-based materials continued in an effort to determine what factors were limiting the application of these materials as spacers and what techniques could be employed to reduce or eliminate the undesirable properties.

Correspondence with one of the suppliers of a mica-based sheet material disclosed that they are convinced that the binder they were using was causing some trouble and they have agreed to change to a new type, which it is hoped will be an improvement. Samples of this new material will be available in the next interval.

We have also received samples of a new silicone impregnated mica sheet from another supplier and thus have two possible sources of this type of material.

Thermal conductivity data have been received for some of the more promising of the materials tested in the previous intervals, and the data are presented in the Detailed Data Section of this report.

No results on the gas analysis tests have been received to date, but this work is being actively pursued and some results will be available in the very near future.

Detailed Factual Data

The following descriptions* identify the new materials and variations in some of the previously tested materials investigated during the fourth interval of this project.

Asbestos-Based Material

Lot #146-A - Terratex type material - (4% clay) - 0.018-0.020" thick

Lot #146-B - Terratex type material - (6% clay) - 0.018-0.020" thick

Lot 147 - Type I.N. Quinterra (no clay) - 0.012" thick

Lot 178 - Type I.N. Quinterra (no clay) - 0.015" thick

Lot 2A-1 - Lot 2A Terratex - distilled water treatment for control purposes

Lot 2A-2 - Lot 2A Terratex - treated with dilute nitric acid

Lot 2A-3 - Lot 2A Terratex - treated with 10% sodium hydroxide

Lot 2A-4 - Lot 2A Terratex - treated with 10% potassium hydroxide

Lot 2A-5 - Lot 2A Terratex - treated with approx. 20% calcium hydroxide

Lot 2A-6 - Lot 2A Terratex - treated with approx. 20% barium hydroxide

Lot 2A-7 - Lot 2A Terratex - treated with approx 20% cerium nitrate

All of the above samples were immersed in the designated solutions and placed in an evacuated chamber for 1 hour, except #2 & #3 which were in solution 10 minutes and under vacuum 5 minutes.

This reduced time was necessary to prevent disintegration of the paper during the treatment.

(All of the above materials were treated with ethyl silicate by the solvent method prior to the tests described.)

* Handsheets prepared by the T.A.P. Lab., G.E. Pittsfield Works

Mica-Based Sheet Material

M2SH2B and PC2SH2B	- Silicone resin treated integrated mica samples supplied by the Integrated Mica Corporation
S-1032	- Silicone resin treated Micamat supplied by General Electric Company
Lot 155A	- Silicone resin bonded Micamat laminate (2 layers) 0.012" - 0.014"
Lot 155B	- Silicone resin bonded Micamat laminate (3 layers) 0.017" - 0.020"

The physical test results for these materials are tabulated and discussed in the following pages.

Material	(°C) Firing Temp	(Min) Firing Time	Bend Angle	Load at Break	Punching Characteristics & General Remarks	Ease of Degassing & Gas Content
Lot 146-A Terratex	As Rec'd		31°	0.41	Sample punched similarily to previous samples of Terratex. Punchings are cleaner and have better definition at the higher temperatures.	Similar to previous Terratex samples
	400°	5	21	0.42		
	500	5	19	0.41		
	600	5	15	0.31		
Lot 146-B Terratex	As Rec'd		26°	0.45	Similar punching characteristics to lot 146-A above. This is the best Terratex type material tested to date with respect to physical strength.	Same as above
	400°	5	19	0.46		
	500	5	16	0.38		
	600	5	12	0.37		
Lot 147	As Rec'd		32°	0.12	Material punchings are fuzzy as received and when fired at 400C, but are fairly clean at 500C and quite clean at 600C.	"
	400°	5	39	0.15		
	500	5	36	0.16		
	600	5	23	0.11		
Lot 148	As Rec'd		33°	0.19	This sample did not punch as well as Lot 147 above, although it did improve with firing temperature.	"
	400°	5	31	0.22		
	500	5	21	0.22		
	600	5	18	0.18		
S-1032 G-E Micamat	As Rec'd		6.6	0.43#	Punches cleanly with very little tendency to flake or delaminate	Similar to regular mica in quantity. No information on composition.

From the preceding physical test results and Fig. 1, which is a plot of bend test information on typical samples of the various materials tested to date, it can be seen that the Lot 146-B Terratex material has the highest mechanical strength of any of the materials under test and approximately 60 percent of that of the presently used sheet mica. While the Terratex material lacks stiffness at small loads; as compared to the regular mica, the B-2 Integrated Mica, and the S-1032 Micamat, it does have good strength. Fig. II is also a plot of bend test information which shows the effect on strength and stiffness of air firing the Lot 146-B material. From this plot it can be seen that the properties of the material can be varied, if necessary, to fulfil specific applications where flexibility or stiffness at small bonds would be desirable.

Bend test information for the variations in the Lot 2A material was not obtained, since the purpose of these treatments was to observe their effect, if any, on the gas and vapor affinity of this material. This information is discussed under Tube Data later in this report.

Data on the two samples of laminated Micamat (Lots 155A and 155B) were not taken when it was found that these samples when heated tended to delaminate andwould not be satisfactory as tube spacers.

Because the effect of cathode cooling by heat conduction through the spacer material is of great importance in tube design, the following tests were performed by the General Engineering Laboratory of the General Electric Company to determine the thermal conductivity of several of the materials under investigation. This work was necessary because thermal conductivity data for these types of sheet materials with heat flow parallel to the surface of the sheet were not available in the literature.

These tests were conducted on sample blocks of the various materials approximately 5" x 5" x 1/2" built up from strips 5" x 1/2" and supported in transite clamps for the test. A measure of the thermal resistivity was obtained when these blocks were heated to a known temperature on one side and the resulting steady state temperature measured on the opposite side.

The results on regular sheet mica, Lot 2A Terratex, M2SH2B and PC2SH2B Integrated Mica are as follows:

Material	Sample Size In Inches	Hot Side Temp °C	Cold Side Temp °C	Thermal Resistivity °C/in/watt/in^2
Sheet Mica	5" x 4 1/2" x 1/2"	570	543	21
Terratex (Lot 2A)	5" x 5" x 1/2"	618	512	92
M2SH2B Int Mica	5" x 5" x 1/2"	572	530	33
PC2SH2B	5" x 4" x 1/2"	590	549	34

The estimated accuracy of these results is ± 10 percent. The least accurate of the values measured and necessary to calculate the resistivity of the sample, is the sample thickness. Due to the laminated character of these samples, the accuracy of the thickness measurements was quite low.

From this information it appears that from the heat conductivity standpoint all three of the materials under test are superior to the regular sheet mica. Thus, from this consideration, it would be possible to use greater thicknesses of these materials than those currently used with sheet mica.

Tube Data

The JAN test limits for the two tube types for which data are presented in this report are as follows:

Type SD-6C (12AT7)

- $-I_g$ = 2.25 ua (max)
- I_p = 7 - 14 ma
- Sm = 4500 - 6500 umhos
- 11Sm = 4000 umhos
- I_s = 50 ma (min)

Type SD-6L (1U4)

- $-I_g$ = -1.0 ua (max)
- I_p = 1 - 2.1 ma
- I_{g2} = 0.28 - 0.62 ma
- Sm = 720 - 1080 umhos
- 1.1 Sm = 610 - 1080 umhos
- [illegible] = 32 mw (max)

To determine the effect of the various chemical treatments of the Lot 2A Terratex described earlier in this report Type SD-6C-F (mod 12AT7) tubes were made and processed on the bench exhaust equipment where the relative quantities of gas evolved could be observed.

Due to the small number of tubes in each lot and random variations, the data on relative quantities of gas evolved were not conclusive. However, there did appear to be certain trends which might be of interest. All of the Terratex-spaced evolved at least twice as much gas as the average mica-spaced ones. Also, the distilled water treated and nitric acid treated samples seemed to give off the gas more slowly so that the higher pressures came later in the exhaust schedule than the other Terratex treatments. Of the other five treatments, the calcium and barium hydroxide treated samples gave off the largest quantities of gas, with the potassium hydroxide treated sample third, the sodium hydroxide treated sample fourth, and the cerium nitrate sample appeared to give off the least gas of any of these Terratex samples. None of the treatments appeared to improve the gas content of the Terratex enough to be called a major improvement, so this continues to be a big problem in the application of this type of material.

The electrical test data for these tubes are as follows:

Lot 2A-1 TX (Dist Water)

$-I_g$	I_p	Sm	11Sm	I_s
.30	10.6	5790	5530	85
.06	10.1	5300	5020	90
.20	8.8	5290	5030	80
.30	9.5	5420	5130	85
.08	11.6	5400	5010	100

Lot 2A-2 TX (HNO_3)

$-I_g$	I_p	Sm	11Sm	I_s
.08	9.2	5300	5030	80
.08	10.1	5120	3700	80
.10	10.8	5780	5390	85
.08	9.4	5460	5090	85
.10	10.3	5450	4980	90

Lot 2A-3 Tx (NaOH)

-Ig	Ip	Sm	11Sm	Is
.15	10.6	5850	5580	85
.08	8.8	4850	2690	70
.15	9.8	5350	4320	65
.05	9.9	5360	4070	75
.08	9.8	5040	3160	75

Lot 2A-4 Tx (KOH)

-Ig	Ip	Sm	11Sm	Is
.08	9.4	4990	4450	75
.08	9.8	5360	4920	85
.10	9.8	5180	4250	80
.06	9.4	5280	4320	80
.10	10.6	5400	5060	95

Lot 2A-5 Tx Ca(OH)2

-Ig	Ip	Sm	11Sm	Is
.10	9.6	5380	5090	90
.10	8.2	4780	4620	75
.20	9.0	5140	5000	85
.08	10.6	5620	5450	95
.08	8.5	5300	4900	85

Lot 2A-6 Tx Ba(OH)2

-Ig	Ip	Sm	11Sm	Is
.10	10.7	5400	5240	110
.08	10.3	5400	5200	100
.10	10.6	5600	5350	100
.10	9.4	5200	5060	90
.08	9.8	5260	5180	95

Lot 2A-7 Tx Ce (NO3)4

-Ig	Ip	Sm	11Sm	Is
.08	9.5	5160	4990	80
.03	9.0	5180	4850	85
.05	8.9	5060	4900	80
.10	9.2	5030	4850	85
.05	9.2	5160	4800	85

Mica Control

-Ig	Ip	Sm	11Sm	Is
.02	8.4	4750	4330	60
.05	9.7	5040	4620	75
.03	8.8	5000	4630	80
.05	10.7	5140	4740	80
.07	9.1	5110	4620	75

These data do not indicate any marked differences between the various lots of tubes using the chemically treated Terratex. However, it is of interest to note that none of the treatment appeared to be detrimental to the emission of the cathodes. Our hopes of reducing the gas content of this material by these treatments were not realized, and we must continue to explore means to reach this end.

To further evaluate the samples of Terratex and Quinterra designated as Lots 146A, 146B, and 148, type SD-60-G (Mod. 12AT7) tubes were made. Lot 147 Quinterra was not used in this test because of its low mechanical strength and the fact that it was the same basic material as Lot 148 and differed only in thickness. Lot 2A Terratex-spaced tubes were made along with regular mica-spaced tubes for control and comparison purposes.

All tubes were evacuated on the bench exhaust system where a measure of the gas given off by the various spacer materials could be made. The tubes with Lots 146A, 146B and 148 spacers gave off quantities of gas of the same order of magnitude as the Lot 2A Terratex control tubes. No differences could be observed. All four of these materials gave off several times the quantity of gas obtained in the mica-spaced control tubes.

The electrical test data for these five lots of tubes showed 70 percent or more of the tubes using Lots 146A and 146B Terratex spacers to have very high grid current readings. This could be attributed to the fact that these two samples, being hand-made sheets, were of greater density than the machine-made Lot 2A Terratex and the Lot 148 Quinterra. Therefore, they were more difficult to degas during the normal exhaust procedure. This had not been observed during evacuation of the tubes, but it probably would take a longer exhaust schedule or a different heat treatment prior to tube making to insure a more complete degassing of the spacers. As only limited quantities of these two samples were available, these factors could not be checked at this time.

Tube data are given below on the Lot 2A Terratex, Lot 148 Quinterra, and the control tubes using regular mica spacers.

Lot 2A Terratex

-Ig	Ip	Sm	11Sm	Is
.12	9.9	5250	4950	100
.05	8.8	4750	4400	92
.13	8.8	4800	4490	98
.10	9.3	4920	4640	100
.08	10.5	5090	4760	100

Lot 148 Quinterra

-Ig	Ip	Sm	11Sm	Is
.10	10.4	5000	4400	97
.10	9.2	4560	4200	94
.05	8.7	4750	4120	70
.05	8.1	4530	4200	85
.10	9.7	4830	4200	100

Mica Control

-Ig	Ip	Sm	11Sm	Is
.15	9.2	4840	4200	75
.15	9.5	5030	3800	95
.10	8.9	5000	4600	95
.10	9.4	4820	4400	105
.10	9.4	4850	4500	100

The results on the Terratex-and Quinterra-spaced tubes compare favorably with the mica control tubes and indicate that these materials could have some application, if a technique could be found for reducing their gas content.

Life test data for SD-6G (Mod. 12AT7) tubes, for which initial test data were presented in the second interval report, have been obtained. These lots of tubes were designated as follows:

Life Lot #490 - Regular Mica (Control)
" " #491 - Lot 129 Quinorgo
" " #492 - Lot 134 Terratex
" " #493 - Lot 135 Terratex

Figure III is a plot of transconductance versus hours on life for the median values of these four lots. From these data it can be seen that the Lot 129 Quinorgo-spaced tubes compare very favorably with the mica control tubes, and that the two lots of Terratex-spaced tubes are not greatly different from the control tubes. The apparent rise in transconductance from 315 hours to 504 hours for the Lot 134 Terratex tubes is difficult to explain, although it amounts to less than 5 percent. Additional time in life test would be necessary to determine whether this result is really a trend or only a random variation because of the small number of tubes tested. These data are encouraging and seem to confirm the belief that if initial gasing of the material could be overcome, some variation of an asbestos-based sheet could find application for improved tube spacers.

To obtain additional information on the Terratex type of material, another tube type was tried to see what new facts might come to light. This tube type was the SD-6L (1U4), which is a low power filament type. Tubes were made to compare

Lot 2A Terratex to regular mica and were processed on automatic production type equipment. The electrical test data for these tubes are tabulated below.

Mica (Control)

-Ig	Ip	Sm	1.1 Sm	Is	RF Noise
0	1.60	943	875	36	OK
0	1.32	897	833	30	"
0	1.50	899	826	34	"
0	1.55	917	842	36	"
0	1.50	922	860	35	"
0	1.50	906	854	34	"
0	1.55	924	863	35	"
.15	1.61	954	900	34	"
0	1.73	942	882	35	"
0	1.52	888	837	34	"

Lot 2A Terratex

-Ig	Ip	Sm	1.1 Sm	Is	RF Noise
0	1.52	903	827	34	OK
0	1.55	930	816	28	"
.05	1.54	918	828	36	"
0	1.56	952	875	36	"
.02	1.54	888	800	34	"
0	1.55	936	829	35	"
0	1.50	900	828	34	"
.05	1.62	900	825	34	"
.02	1.49	913	832	34	"
0	1.69	973	878	37	"

These data indicate that the Terratex spacers gave tubes comparable to regular mica spacers. This is encouraging because the tubes were processed on the automatic equipment at normal production speeds.

One particular drawback of the Terratex material was demonstrated during the mounting operation on these tubes. This drawback concerned the mounting of the filament after the cage had been assembled and where it is desirable to be able to see the grids so that the filament may be inserted without damage. With the clear, unsprayed micas this was possible, but the Terratex material, being opaque, required greater skill and effort on the part of the operators to realize good tubes. This factor may limit the application of this type of material where it is desirable or necessary to observe grid alignment or other features after the mount cage is completed.

CONCLUSIONS

The physical strength of the new Terratex samples, designated Lots 146A and and 146B, appears to be quite good and shows promise of a material that could replace mica, at least from the mechanical considerations. The high grid current readings on tubes using spacers of these two materials may be due to the greater density of the sheet making it more difficult to degas or to foreign material being incorporated in the sheet while it is being made. Since these two materials had a higher clay content than the Lot 2A Terratex, it indicates the need for a full evaluation of the effect of clay in this material.

The test results on the Lot 148 Quinterra samples indicate that it apparently does not impair the emissive characteristics of the cathode materials and is similar to Terratex from the gas consideration. It does have fairly low strength, but this might be offset by using a greater thickness.

There have not been any major improvements in reducing the gasing characteristics of the asbestos-based materials, and this is still the big drawback to this material. The gas analysis data should help in solving this problem.

The life test data on the SD-6C (Mod. 12AT7) tubes with Terratex and Quinorgo spacers is encouraging and indicates that these materials have real possibilities in vacuum tubes. Additional life testing of these and other tubes will be helpful in determining to what extent the materials may give up gas during tube life.

Up to the present time no really satisfactory samples of any built-up mica or integrated mica sheet materials have been tested. Most of these have been mechanically weak and manifested a tendency to reduce the emission characteristics of the cathode materials. However, as more interest is developed by people familiar with mica and the processing and manufacturing of these materials, it should be possible to obtain a material which could be used in certain applications.

WORK TO BE PERFORMED NEXT INTERVAL

Secure the gas analysis results on some of the materials under test and interpret these results with an objective of reducing the gas content of these materials.

Obtain and evaluate new samples of Terratex and integrated mica in an effort to find materials of higher strength and with less detrimental affect on the cathode materials.

Continue to attempt new processing techniques and treatments that will improve some of the characteristics of materials currently on hand or available.

Obtain experience on various tube types to determine what types can or cannot be made utilizing some of the materials already tested. This work is to be directed at representative tube types of the various tube classifications.

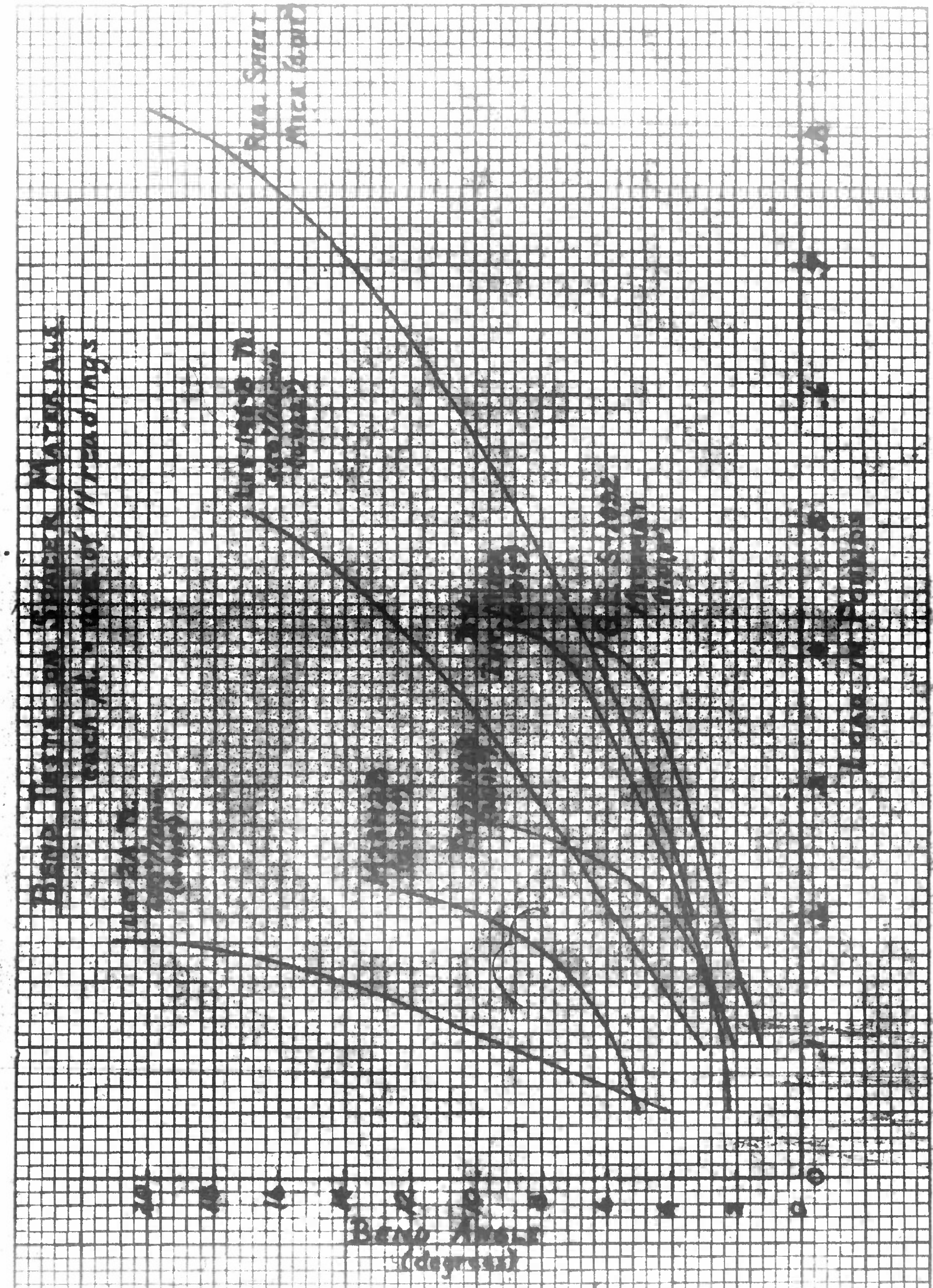
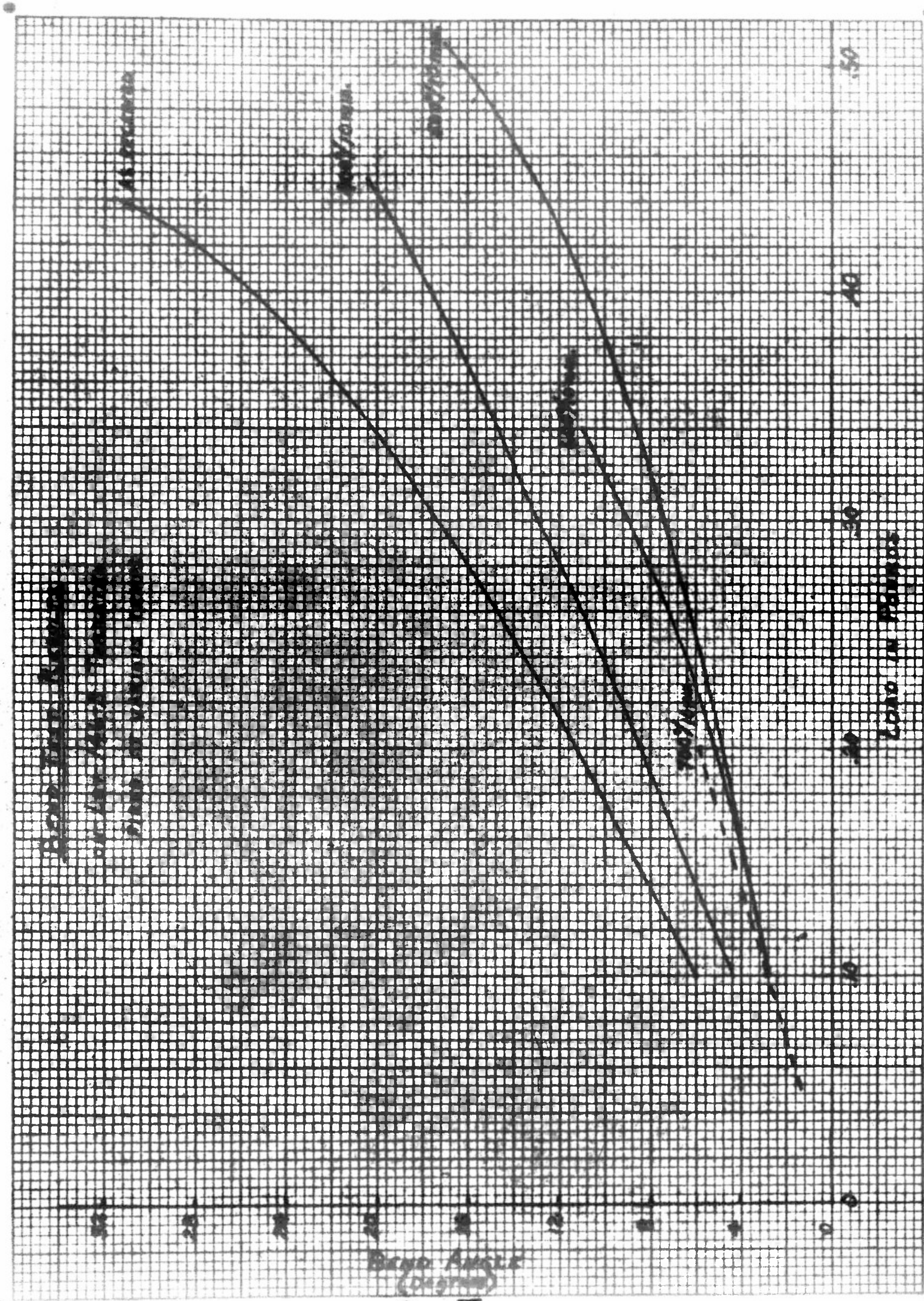


Fig. I



Load in Pounds
0
10
20
30
40
50
Bend Angle
(Degrees)
0
4
8
12
16
20
24
28
32

Fig. II

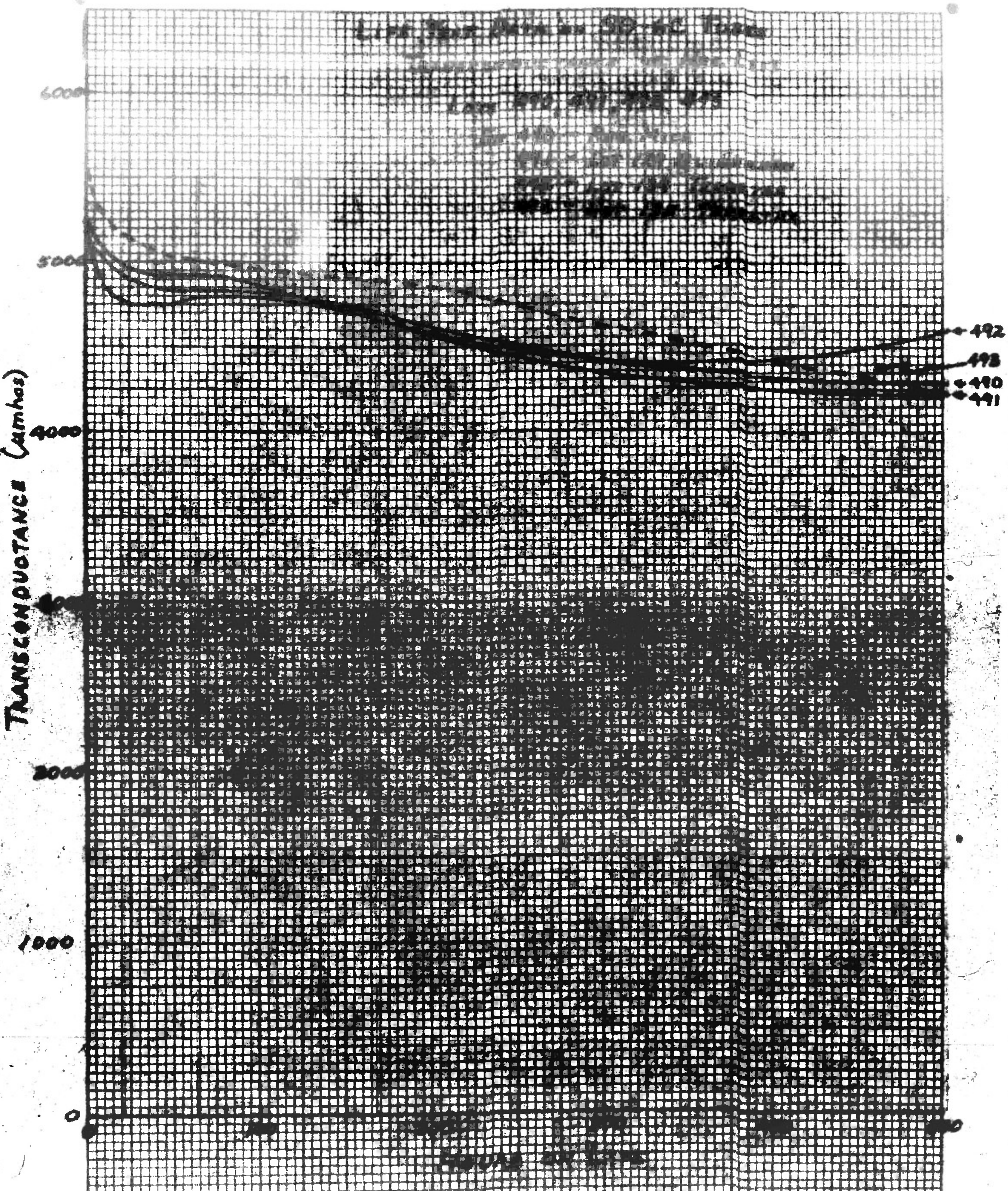

6000
5000
4000
2000
1000
0
TRANSCONDUCTANCE (μmhos)
492
493
490
491

Fig. III