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ELEVENTH QUARTERLY PROGRESS REPORT

FERRITE DEVELOPMENT

CONTRACT NO.: DA-36-039-sc-5449

GENERAL CERAMICS & STEATITE CORP.

Keasbey, N.J.

11th QUARTERLY PROGRESS REPORT
SIGNAL CORPS CONTRACT NO. DA36-039 sc-5449
Period: 1 Oct. - 31 Dec., 1953

MAGNETIC FERRITES

GENERAL CERAMICS AND STEATITE CORPORATION
Keasbey, New Jersey

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11th QUARTERLY PROGRESS REPORT

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ELEVENTH QUARTERLY PROGRESS REPORT

CONTRACT FOR SERVICES, FACILITIES, AND MATERIALS REQUIRED TO INVESTIGATE AND STUDY MAGNETIC FERRITES

PERIOD COVERED: From October 1, 1953 to December 31, 1953

CONTRACT NO. DA 36-039-sc-5449

DEPT. OF ARMY PROJECT NO.: 3-93-00-503, 3-99-04-042

SIGNAL CORPS PROJECT NO.: 32-2005-D, 29-194B

PLACED BY: Laboratory Office, Signal Corps Procurement Agency
Contracting Division, Fort Monmouth, New Jersey

CONTRACTORS: General Ceramics and Steatite Corporation
Keasbey, New Jersey

OBJECT: To develop ferromagnetic ferrites of different permeabilities, and low magnetic losses, and to investigate conditions favorable to their production.

REPORTED BY: Dr. E. Albers-Schoenberg, Supervisor
Ephraim Gelbard, Project Engineer

LIST OF GRAPHS & TABLES

ABSTRACTS

PART I

A) The investigation of temperature-stable bodies of the lower permeability range has been continued. The work reported upon refers to the "fluxed" type similar to the old General Ceramics formula MF-90-"B". The effect of various fluxes and various firing temperatures has been studied. From the viewpoint of future production, the results are promising.

B) A preliminary property chart of Ferramic Q, a new low-loss, low-permeability body, is presented.

C) Three different methods of measuring μ_o and Q are compared. For a number of materials, the deviation of results is too large. Especially for the low-loss body, Ferramic Q, the figures are erratic.

PART II

A) The Reproducibility and Uniformity investigation has been extended on material MF-141-"I". Results for μ_o measurements on 400 cores (in the virgin state) are reported. One out of 6 batches shows 98% of all cores to fall within a tolerance of ± 2-1/2%. Some conditions for high uniformity are established.

PART I

Body Development and Measurements Made During The Eleventh Quarter of the Contract
DA Project No.: 3-93-00-503, S. C. Project No.: 32-2005-D

A) Temperature Stable Bodies of the Lower Permeability Range (Improvement of the Reproducibility of MF 90-"B" Body)

The majority of the program in temperature stability this quarter has been directed to the development of body MF 1835, a variation of the standard body, MF-90-"B". The body MF 1835 is an attempt to duplicate the "B"-body with the exception that purer raw ingredients are used.

The original body MF-90 as it was formulated in 1948 contained raw materials from natural sources. The natural impurities of these materials contributed to the peculiar temperature behaviour of the body, however, inconsistencies of composition were inevitable and reproducibility of μ_o, Q, and temperature coefficient of μ_o were frequently not attainable.

The aim of this part of our work was to duplicate the Ferramic "B" material by using pure raw materials only; i.e., to add the former impurities in the form of pure reagents. "Pure" in this case means the grade of purity, mostly better than 99%, as it is represented in technical raw materials.

Since silica and Ca-compounds are the most usual impurities in natural ores, a hydrated silica and calcium fluoride or calcium carbonate were added. After various attempts with additions of either silica or calcium compounds or both, and after discarding all those which failed to give a body approaching the B-body properties, finally the composition was established with an addition of 2.0% to 3.0% of silica (computed as water-free SiO_2) and CaF_2 in a fixed amount of 2.75% or calcium carbonate of 3.50%. The other (main) ingredients of the body remained essentially the same as those of the B-body:

5% MgO
6% MnO_2
10% ZnO
6% NiO

The remainder Fe_2O_3, which amounted to 68% in the case of high silica content and to 69% in the case of low silica content.

PART I (Cont'd.)

A) Cont'd.

The results of % $\frac{\Delta\mu_o}{\mu_o}$ vs Temperature are shown in Graphs 172 and 173. These represent the best obtainable up to the present. For further details see Table LII.

Out of the four compositions (MF-1835A to MF-1835D the A and D variations appear the most promising (see Graph 172). MF-1835A shows a low negative temperature coefficient of about -.014%/°C from 30°C to 100°C. The initial permeability of this body is 90 and the Q is 90 (toroidal measurements at 1 Mcs). MF-1835D has a positive temperature coefficient of about +.028%/°C from 30°C to 100°C. The initial permeability is 88 and the Q is 81 (toroidal measurements at 1 Mcs).

Graph 173 shows the effect of firing temperature on MF-1835B. It is clearly demonstrated here that the sign and magnitude of the temperature coefficient can be greatly changed by firing variations.

It has to be mentioned that MF-1835D is made without pre-calcining the materials. Therefore, it shows a relatively high shrinkage which is undesirable for a production body. It is, however, presumed, that body MF-1835D could be modified accordingly as to yield similar qualities after the raw mixture has undergone a prefiring treatment.

In any case, for all of these bodies, there remains a remarkable sensitivity as to the temperature coefficient of μ_o. A small coefficient easily changes its sign. At high temperatures from about 220°C on all curves as shown on Graph 172, turn positive and the coefficient increases rapidly toward the Curie point.

Summarizing, it may be stated that the goal has been reached and the temperature stable "B" composition, which has found widespread application during the past three or four years, has successfully been reshaped. The knowledge of proper handling a temperature stable body of the "fluxed" type has been greatly amplified.

Further development of the body MF-1832C (mentioned in the last quarterly report) has been aimed at determining the degree of firing required by that body in its present state of formulation. Numerous firings have disclosed that various shapes of the ferrite ware will affect the maturing temperature. F-108 (about 2" O.D., 1.5" I.D., 1/4" thick) mature at a slightly lower temperature than the F-256 tuning slugs (about 1/2" O.D., 1/4" I.D., 1-1/2" long). Good results have been obtained in both cases.

A few attempts to increase the effective fired density of MF-1829 have proved unsuccessful. In view of the better performance of the body MF-1832C, work on MF-1829 has been abandoned and MF-1832C can be adopted as a temperature stable ferrite of the permeability class 170-180 if necessary.

B) New Measurements of Ferramic Q

As indicated in the last quarterly progress report, more details of this ferrite are now shown. Table LIII gives preliminary measurements of this material in comparison with two other low-loss ferrites.

Ferramic "Q" is the lowest loss ferrite that has yet been developed. Some preliminary data shows it to have a useful frequency range at least up to 30 Mcs. The losses at 1 Mcs are so low that considerable difficulty has been experienced in evaluating the true tan δ m (see Section C).

In all aspects (regarding losses, temperature stability, inherent magnetic stability), this material is considered to be a most significant development.

C) Comparison of Measurement Techniques for Initial Permeability and Q

Since the last Quarterly Progress Report in which the field strength of the 1 Mcs μo measurement (R. F. Bridge) was evaluated, the attempt has been made to compare various measurement techniques with the objective of improving both accuracy and speed. On a set of F-268 toroids (O.D.= 1.25", I.D.= 0.75"), three different methods have been used and a fourth is being worked on. The three measurement methods already performed were:

1) R. F. Bridge (G. R. 916A)
2) Q-meter (Boonton 260-A)
3) R. F. Permeameter* (National Electronic Laboratories, Size B)

The first two methods utilized a toroidal winding (N=25 No. 20 AWG SF, evenly distributed), the third method needs no winding. All the measurements were performed at 1 Mcs to lend correlation to all previous work done on the R. B. Bridge at 1 Mcs.

Table LIV summarizes these results. The fourth method will consist of the evaluation of permeability by the measurement of H and B on a toroidal winding. When this is completed, an attempt to analyze the differences between the various methods will be made.

As indicated in Section B, an attempt was made to measure the losses of Ferramic Q by these three methods. All the results are fairly doubtful and cannot be trusted as a true indication of the material for the following reasons:

On the R. F. Bridge, the loss resistance is so small that accurate reading is extremely difficult (R= 0.2 ohms). If the copper losses are then subtracted, the loss resistance approaches zero (or infinite Q). On the R. F. permeameter, this ferrite yields a negative tan δ m. On the Q-meter, a readable value can be measured but if the Q is over 500-600 the Q-meter accuracy is greatly reduced. It appears that other methods must be found to evaluate this material if further development is to be made.

*Developed by P. H. Haas, National Bureau of Standards

PART II

Body Development and Measurements Made During the Fourth Quarter of the Contract
DA Project No.: 3-99-04-402, S. C. Project No.: 29-194-B

A) Investigation of Reproducibility and Uniformity of Ferrite Materials

Since the initial phase of the work had been completed on Ferramic "G"-MF-254 in the last quarterly report, the uniformity and reproducibility investigation started over again with Ferramic "I"-MF-141. This ferrite material has been subjected to a very similar procedure of preparation, sample-pressing, firing and measurement. The μ_o figures have been taken from the cores in the virgin state and are reported and evaluated in the same manner as exercised for MF-254-"G" material. Another set of measurements, after magnetization and demagnetization, will follow; as well as the more important measurements of reversible permeability.

Preparation of the samples comprised the following steps of procedure:

A mix consisting of the following:

665 grams nickel oxide, grey
1645 grams zinc oxide, French process
1540 grams iron oxide, red, tech. grade, (C. K. Williams)
6700 ccm tap water

was milled for four hours with 7 kg of steel balls (3/8" diameter). Then, an additional 3150 grams of iron oxide (red), and 4800 ccm water were added and the milling continued for another four hours. After emptying and drying, the dry cakes were granulated through an 8-mesh screen, and the granulation filled into saggers for calcining.

At this point the preparation has been divided into two parts; each part being subjected to a separate calcining heat treatment. Sixty percent of the material was fired two hours at 1900°F, forty percent for the same time at 2250°F.

From these two calcines, two bodies have been prepared according to the following formulae:

PART II (Cont'd.)

MF-141-L (L standing for low-fired calcine)
80% by weight low-fired calcine
1.9% by weight of nickel oxide grey
4.7% by weight of zinc oxide, French process
13.4% by weight of iron oxide (red), techn. grade

MF-141-H (H standing for high-fired calcine)

50% by weight high-fired calcine
4.75% by weight of nickel oxide grey
11.75% by weight of zinc oxide, French process
33.50% by weight of iron oxide (red), tech. grade

The first batch (MF-141-L) weighed 5 kg, the second (MF-141-H weighed 4.6 kg;6500 ccm of water was added to the L-batch, 6000 ccm to the H-batch. Both batches were milled for 15 hours (over night).

After drying and breaking up the cakes, the material was worked in a Lancaster mixer with 1% (of the weight of dry material) of Polyvinyl alcohol dissolved in sufficient water to form a paste. One part of each body was screened through 20 mesh only, thus containing all grain sizes from 20 mesh down to fine dust; from another part of each body, the dust was taken out by an 80-mesh screen. Then 6% water was added to the granulated body.

Instead of pressing samples with three different pressures, this time only 5 tons and 10 tons have been applied. A pressure of 15 tons is too high with respect to the die-size (toroid F-109) and there were signs of overstressing the die. Since overpressing as well as underpressing has to be avoided in any case of practical production, there was no point in jeopardizing the die by applying excessive pressure. Thus, there were a total of eight variations to be fired.

With respect to the firing, there was a difference of procedure compared with the MF-254-"G" body work previously done. For the MF-141-"I"-body series, a medium size, gas-fired, car-type, tunnel oven with a firing cycle of 16 hours has been used. The slabs loaded with the toroidal cores were located on the middle level of the cars. There were 40 cores on each slab. Of all the kilns available, this kiln is the most reliable to handle medium size production lots. The peak temperature was 2360°F (almost cone 12 down).

PART II (Cont'd.)

The total number of cores in this uniformity test was about 400. These were tested according to the standard procedure for μo at 1 Mcs and the measurements were corrected for temperature deviations from an ambient of 27°C. Since the test frequency was rather high for "I"-141 material due to the high losses, these rings will be retested at about 100 Kcs for μr and μo. This lower frequency is close to that of the field of application of this ferrite and any changes in the distributions as a function of frequency may be noticeable.

The test results are shown in Table LV and Graphs 174 and 175. Table LV indicates that the best results for uniformity of μo (1 Mcs) are obtained using a low temperature calcine in all comparable cases of particle size and pressure.* In the low-fired calcine preparation particularly, the 20 mesh granulation (i.e., the dust not removed) gives very good results at both the 10-ton and 5-ton pressures. Apparently when using the low-fired calcine any removal of the dust (through 20 mesh and on 80 mesh) reduces the uniformity. However, for the high-fired calcine preparation, there appears to be little change of the overall uniformity as a function of either the particle size or pressure.

It can be seen by comparing the distribution curves (Graphs 174 and 175) that the best conditions for uniformity (low fired calcine, through 20 mesh at pressure of either 5 tons or 10 tons) also results in the highest μo values by about 5% (using the peak of each distribution as an average indication of μo). Also, it is interesting to note that irrespective of calcine preparation, the 20 mesh granulation gives about equal μo values at each pressure while in the other granulation where the dust is removed the permeability shifts to lower values when the pressure is reduced.

Although at 1 Mcs the losses in this ferrite are very high and all the Q values are in the order of 2, the low-fired calcine preparation in each case yielded higher Q's. This small difference in losses at 1 Mcs may be magnified at 100 Kc.

*The pressures in psi reported in the two previous reports were in error. Actually, 3120 psi, 2080 psi and 1040 psi were respectively 15 tons, 10 tons and 5 tons hydraulic ram pressure on the F-109 die. (O.D.= 1.00" I.D.=.625")

PART II (Cont'd.)

The conclusions that can be drawn from this experiment and this particular firing temperature for MF-141 "I" body are:

a) the low-fired calcine preparation yields optimum results for uniformity of μ_o, and magnitudes of μ_o and Q.

b) removal of the dust yields poorer results in magnitude and uniformity of μ_o when using the optimum condition of low-fired calcine.

c) a marked increase in sensitivity to pressure variations occurs when the dust is removed using either calcine preparation.

Dimensional uniformity has still to be measured on these toroids. These results and many others of other magnetic properties will be reported in the future.

Another result of this uniformity test is that the conditions for test results (μ_o at 1 Mcs) are different from those of the MF-254 "G" body.

PART III

RESEARCH PLANNED FOR THE NEXT QUARTER

1) More information will be given on low-loss low-permeability body Ferramic "Q"

2) Investigation of Mn-Zn ferrites will be resumed. Special emphasis will be put on low-frequency measurements.

3) The Reproducibility and Uniformity test will be carried on. Information on the uniformity of MF-141-"I" will be evaluated for other magnetic properties.

MANHOURS SPENT ON CONTRACT FOR THE PERIOD OF

October 1, 1953 to December 31, 1953

NAME	TITLE	HOURS
Dr. E. Albers-Schoenberg	Supervisor	130
Ephraim Gelbard	Project Engineer	126
Dr. K. Wetzel	Ceramist	136
M. Eisenberg	Electrical Engineer	184
S. Ortepio	Ceramist	93
James Callahan	Ceramist	481
S. Golion	Technician	121
J. Kurtz	Technician	114
C. Purzas	Technician	104
W. Hegedus	Technician	91
A. Koseski	Technician	99
Mary Horton	Meas. Technician	319
Ruth Wainwright	Meas. Technician	204

LII

PROPERTIES OF NEW "B"-BODY TYPE COMPOSITIONS

	Fluxes	Firing Facility	Firing Temp.	Magnetic Properties				Temp. Coeff. of μ_o % $\frac{\Delta\mu_o}{\mu_o}$
				μ_o (1 Mcs)	μ_{max}	Bs	Q (1 Mcs)	
1835-A	CaF_2 SiO_2	Large production tunnel kiln	2285°F	90	190	2100	90	-1.0 (30-100°C)
1835-B	CaF_2 SiO_2	Small production tunnel kiln	2330°F	87	176	2100	66	+3.5 (30-100°C)
1835-D	$CaCO_3$ SiO_2	Large production tunnel kiln	2285°F	88	---	---	81	+2.0 (30-100°C)

TABLE LIII

COMPARISON OF MAGNETIC PROPERTIES OF FERRAMIC Q (PRELIMINARY DATA) WITH TWO OTHER LOW LOSS FERRITES

	Ferramic Q	Ferramic N-874	Ferramic J-472
μ_o (1 Mcs)	125	200	330
Q (1 Mcs)	$\geq$ 400	120	61
$\frac{1}{\mu_o Q}$ (1 Mcs)	$\leqq$ 0.00002	0.00004	0.00005
Curie Temp. (°C)	> 250	290	180
$\% \frac{\Delta\mu_o}{\mu_o}$ (%/°C-1 Mcs) (25°C-100°C)	+0.10	+0.14	+0.22
Bs (Gauss-HDC=25) oersteds	2900	3000	2900
BR (Gauss)	1300	2300	1600
Hc (oersted)	1.80	0.50	0.80
μ_{max}	400	500	750

TABLE LIV

COMPARISON OF μ_o AND Q BY VARIOUS MEASUREMENT METHODS (1 Mcs) (F-268 Toroid)

BODY	R.F.Bridge (916A)**** μ'	R.F.Bridge (916A)**** Q*	Boonton Q Meter (290A) μ'	Boonton Q Meter (290A) Q*	R. F. Permeameter (Size B)*** μ'	R. F. Permeameter (Size B)*** Q*
A-106	20.1	139.3	20.4	115**	19.3	192.0
B-90	74.5	72.9	75.6	60	65.8	75.0
C-159	240.7	52.8	243	50	216	63.0
D-216	350.9	39.9	353	37.5	321	39.0
E-174	632.3	10.6	652	11	625	11.9
[illegible]-254	354.2	21.6	364	21	312	23.5
[illegible]-19	715.2	4.6	762	<10	622	5.85
[illegible] 1-1102	599.4	3.6	744	<10	622	4.44
I-141	825.9	2.1	948	<10	1276	1.57
[illegible]-72	289.6	59.8	294	56	278	63.7
[illegible]-974	188.2	90.1	187	80	180	106
[illegible]-1676	906.2	4.3	948	<10	831	4.25

* $Q_m = \frac{1}{\tan \delta_m}$

** Low Q mica parallel condenser

*** C_o, Q_o established with parallel air condenser

**** H_m less than 0.45 milli oersteds

TABLE LV

DISTRIBUTION OF μ_o (1 Mcs) MF 141-"I" AT 27°C FOR TWO CALCINES TWO DIFFERENT PARTICLE SIZES, AND TWO DIFFERENT PRESSURES DIE F 109 - (PERCENTAGES OF CORES WITHIN ± 2-1/2% and ± 5% FOR μ_o USING THE PEAK OF THE DISTRIBUTION CURVES AS THE CENTER VALUE)

CALCINING PREPARATION	PRESSING PRESSURE	THROUGH 20 MESH		THROUGH 20 MESH ON 80 MESH	
		WITHIN ± 2-1/2%	WITHIN ± 5%	WITHIN ± 2-1/2%	WITHIN ± 5%
L Preparation (Low Fired Calcine)	10 Tons	98.0%	100.0%	84.0%	100.0%
	5 Tons	94.1%	98.0%	72.0%	92.0%
H Preparation (High Fired Calcine)	10 Tons	68.6%	98.0%	77.1%	93.8%
	5 Tons	70.6%	98.0%	77.5%	96.2%

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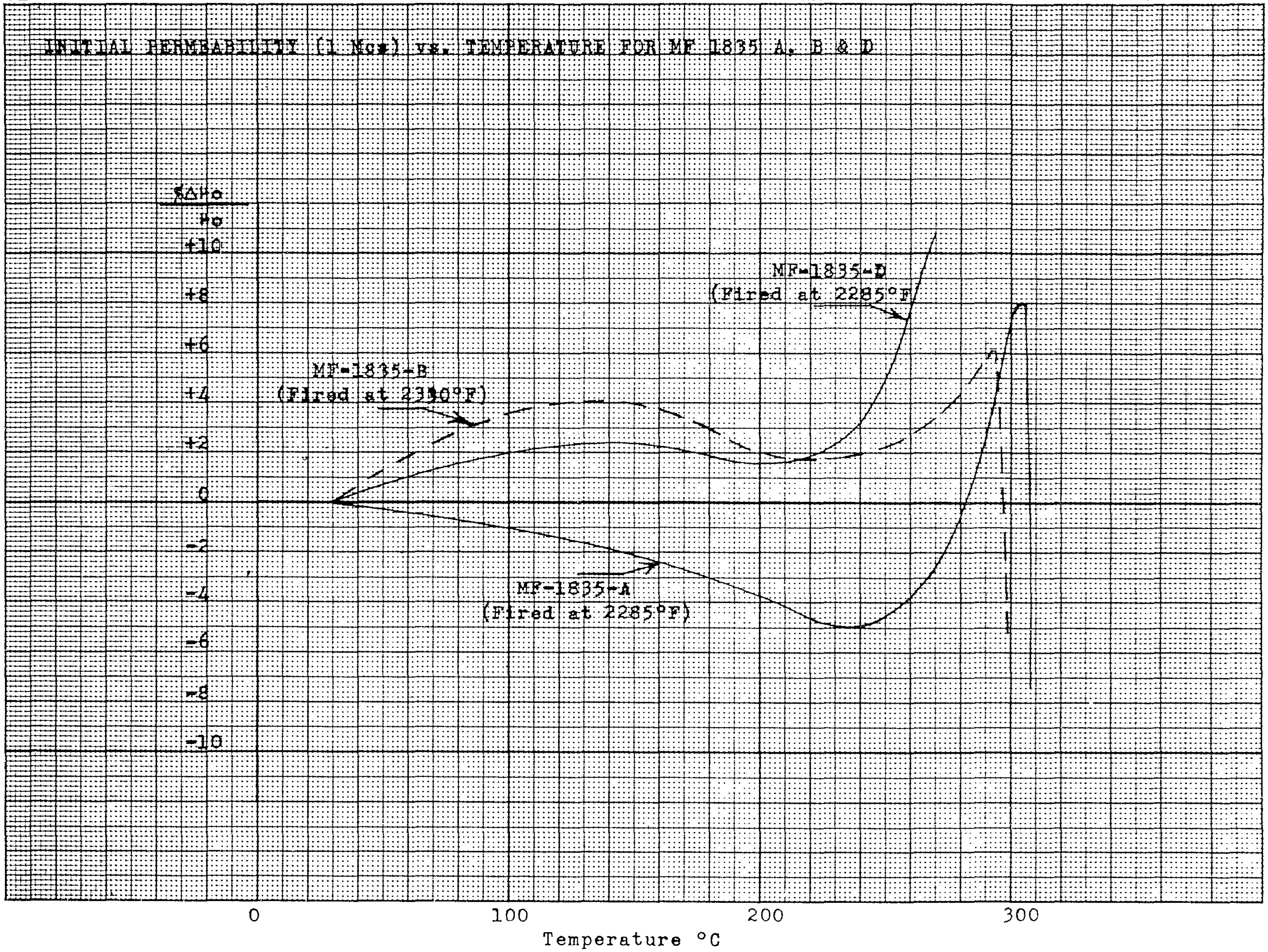
GRAPH 172

12-15-53



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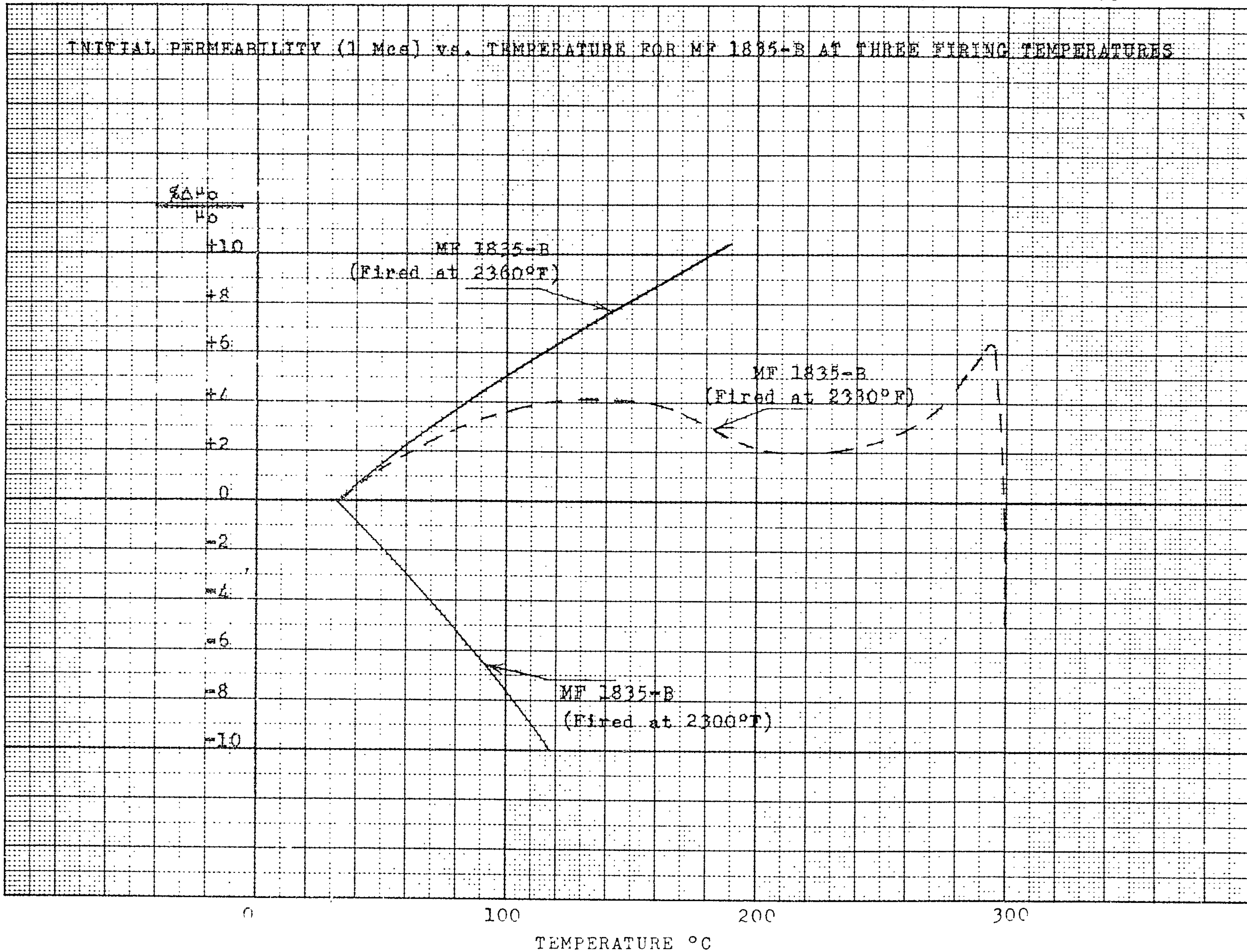

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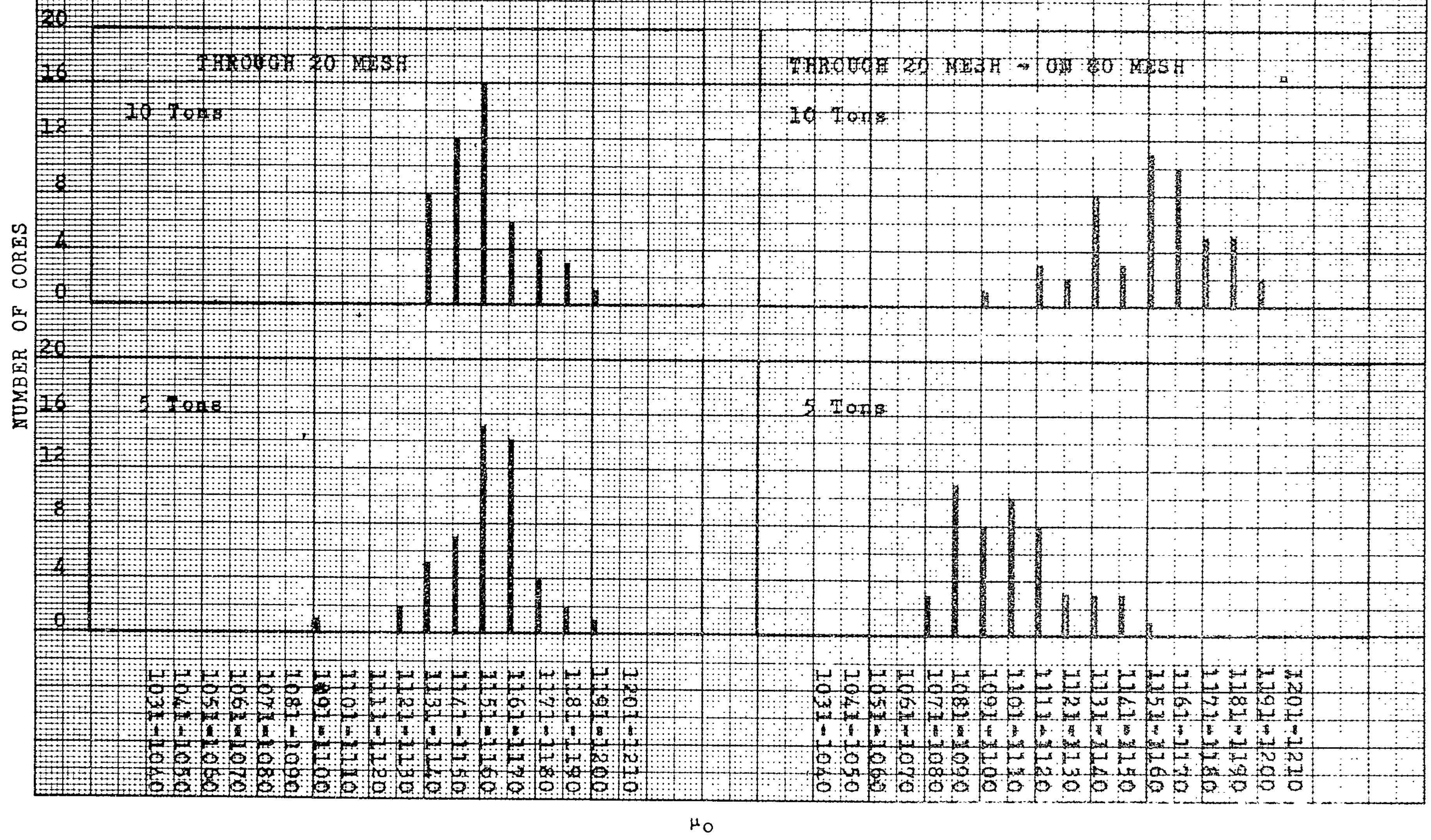
DISTRIBUTION OF μo (1 Mcs) OF MF 141 "I" BODY AT 27°C
TWO DIFFERENT PARTICLE SIZES, TWO DIFFERENT PRESSURES
L PREPARATION (LOW FIRED CALCINE) DIE F 109



GENERAL CERAMICS & STEATITE CORP. GRAPH 175 12-15-53

DISTRIBUTION OF μ_o (1 Mcs) OF MF 141 "I" BODY AT 27°C
TWO DIFFERENT PARTICLE SIZES; TWO DIFFERENT PRESSURES
H PREPARATION (HIGH FIRED CALCINE) DIE F 109