ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN
ANN ARBOR

STUDY, DEVELOPMENT, AND PRODUCTION OF FERROSPINELS

APPLICABLE TO TUNING OF SEARCH RECEIVERS

QUARTERLY PROGRESS REPORT NO. 6, TASK ORDER NO. EDG-6
Period Covering January 1, 1954 to March 31, 1954

Electronic Defense Group
Department of Electrical Engineering

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April, 1954

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TASK ORDER

Title: STUDY, DEVELOPMENT, AND PRODUCTION OF FERROSPINELS APPLICABLE TO TUNING OF SEARCH RECEIVERS

Purpose of Task:

To further the development of ferrospinels of different incremental permeabilities and low losses, with reference to specific applications of interest to the Signal Corps such as RF tuning units.

Procedure:

The approach to the general objective will include:

a. The preparation, under controlled conditions, of specimens of different compositions;

b. The measurement of parameters such as the incremental and initial permeabilities, the saturation inductance, the coercive force and the Q (figure of merit) at various frequencies;

c. The interpretation of these magnetic parameters in terms of the composition, reaction temperature, pressure and other conditions in the preparation of the samples;

d. The relationship of the solid state properties of the crystallite with the various measured magnetic parameters;

e. Theoretical explanations, where possible, for the relationships found in d. above.

Reports and Conferences:

a. Quarterly Task Order Reports shall be submitted reporting technical detail and progress under this Task Order;

b. Task Order Technical Reports of a final summary type are in general desirable and shall be prepared at the conclusion of investigations of each major phase. Such reports shall be prepared as

decided in conference between the Electronic Defense Group and the Contracting Officer's Technical Representative in the Countermeasures Branch, Evans Signal Laboratory.

Personnel:

Electronic Defense Group:

Project Physicist: Mr. D. M. Grimes

Countermeasures Branch, Evans Signal Laboratory:

Project Engineer: Mr. Leon I. Mond

Components and Materials Branch, Squier Signal Laboratory

Project Scientist: Dr. E. Both

Comments:

The classification of this Task Order as Unclassified shall not preclude the classification of individual reports according to the information they contain, as determined in conference with the Contracting Officer's Technical Representative.

M. KEISER
Chief Scientist, Countermeasures Division
Contracting Officer's Technical
Representative

ABSTRACT

Problems in ferrite manufacture are considered. The structure sensitive and structure insensitive properties of the ferromagnetic material are discussed. Intimate mixing of the constituents has been achieved by coprecipitation. The introduction of excess Fe_2O_3 resulted in both desirable and undesirable properties. These properties are discussed and an effort to reduce the undesirable properties is proposed. Pits on the surface of our rings are analysed.

Additional data on Firing Series I and II are presented and analysed. Firing Series III, with pressing force, mixing, and firing temperature as parameters, and Firing Series IV, with calcining operations and composition as parameters, are outlined.

A graphical means of calculating μ_1 and μ_2 from data obtained with the coaxial inductor is derived.

STUDY, DEVELOPMENT, AND PRODUCTION OF FERROSPINELS APPLICABLE TO TUNING OF SEARCH RECEIVERS

QUARTERLY PROGRESS REPORT NO. 6, TASK ORDER NO. EDG-6
Period Covering January 1, 1954 to March 31, 1954

1. PURPOSE

The purpose of this report is to summarize the progress made by Task Group 6 of the Electronic Defense Group from January 1, 1954 to March 31, 1954 on the Signal Corps Contract No. DA-36-039 sc-15358.

The purpose of the task is to further the development of ferrospinels of different incremental permeabilities and low losses, with reference to specific applications of interest to the Signal Corps such as r-f tuning units.

The proposed program of Task Group EDG-6 was outlined in previous progress reports. Only those items will now be reported which have been worked on during the period.

2. PUBLICATIONS AND REPORTS

No publications were issued during the quarter. Dr. Welch, Messrs. Nace and Grimes attended a meeting at Squier Laboratories on 18 February; Mr. Jefferson attended a symposium on ceramic dielectrics and ferromagnetics at Rutgers University on 10 March; Mr. Nace attended the annual IRE convention in New York City from 22 to 25 March.

3. FACTUAL DATA

3.1 Theory of the Magnetization Process (D. W. Martin; D. M. Grimes)

The theoretical studies of the magnetization process have been combined with the measurement of the reversible permeabilities. The report is currently being prepared that was expected to have been issued during the past quarter. It is now expected to be issued during the coming quarter.

3.2 The Manufacturing Program (D. M. Grimes; B. Hershenov; C. F. Jefferson; B. T. Kimura; P. E. Nace; L. Thomassen)

3.2.1 A Description of the Problem. The problem of ferrite manufacture is two fold. First, there is the choosing of the proper gross chemistry for the desired system and second there is the proper choice of manufacturing parameters. Each choice must be dependent upon the desired final properties. For the application of interest to this program there are three specific magnetic quantities which it is desirable to be able to control. These are (a) the effective permeability as a function of frequency, (b) the magnetic losses associated with this permeability, (c) the temperature dependence of the permeability and its associated losses.

These properties depend upon both the structure insensitive and the structure sensitive properties of the ferromagnetic material. The structure insensitive properties are magnetostriction, anisotropy, and saturation moment. The structure sensitive properties are the internal localized demagnetizing fields and stresses. In Section 3.1.1 of QPR No. 5, the quasi-reversible curve would be assumed to arise from the internal localized demagnetizing fields, the superimposed variations would be assumed to arise from the internal localized stresses. Thus the magnetic properties of particular interest to this program would in great measure be determined by the localized internal demagnetizing fields and the

localized internal stresses. Therefore, we chose a specific gross composition ferrite and are now attempting to optimize those characteristics which result from the structure sensitive properties. This part of the program could then be carried over to apply to specimens with different compositions.

The internal demagnetizing factors will depend upon the grain size and the orientation difference between neighboring grains. The stresses will depend upon the annealing and upon the completeness of the solid state reaction. If regions existed where the cation diffusion was incomplete, the result would be localized differences in the degree of inversion of the spinel with resulting differences in lattice constant and thus large internal stresses. The latter effect would be expected to be important at the lower firing temperatures.

At the higher firing temperatures cation diffusion will be more nearly complete.[1] Grain size will be large enough so that walls can move rather freely over wide regions with resulting high permeability and high losses.

Cubical particles, one micron on a side, contain on the order of 10^{10} atoms. Therefore, the spinel reaction must be accomplished by cation diffusion over a large number of unit cells. If the oxides were intimately mixed before firing, the reaction would be completed at a much lower temperature. It is known that the coprecipitation of ferrous and ferric hydroxide produces a precipitate that changes color and becomes ferromagnetic shortly after its formation.

We have coprecipitated iron, nickel, and zinc with Na_2CO_3 and have found that the precipitate was slightly magnetic after being dried at about 110° C and was quite magnetic after heating to 350° C. This introduces the possibility of grain orientation by application of a magnetic field during the firing. The Curie temperature for the material we are now making, 20 mole % NiO, 30 mole % ZnO, and 50 mole % Fe_2O_3, is around 250° C.

3.2.2 The j' Material. During the preparation of Firing Series II an error was made in the composition of the material described as type j. This material, hereafter called j', was composed of a nickel ferrite and a zinc ferrite. The zinc ferrite contained 60 mole % Fe_2O_3 and 40 mole % ZnO instead of the usual 50-50 ratio. Fig. 1 shows the variation of μ_1 and μ_2 with firing temperature for this material at a frequency of 2 mc/sec. The result is a μQ product of around 5×10^4 at this frequency. The material looked very promising since (a) it possesses a large μQ product and (b) the firing temperature is sufficiently high to eliminate any differences that might arise from differences in green density, in minor temperature deviations in the oven, etc. However, it was found that the material contained about 4 mole % ferrous iron. Associated with the ferrous iron content is a high Richter Type[3] after-effect which makes the material unusable.

We are currently making an effort to produce similar material without the high ferrous iron content by replacing some of the nickel with lithium to prevent ferric ion reduction. We will also use replacement by sodium ions and the addition of pentavalent vanadium as an internal oxygen source. However, it is known that magnetite has a negative magnetostriction and most other ferrites a positive magnetostriction. The composition of j' is very near one which gives zero magnetostriction according to Harvey, Hegyi and Leverenz.[2] The presence of a low value of magnetostriction results in a small value of the coefficient β in the differential equation of motion for the domain wall (Eq. 32, p. 15, of QPR No. 5) and thus in a high Q. Therefore, the high Q may arise from the ferrous ions we are trying to eliminate.

FIG 1

COMPLEX PERMEABILITY VS FIRING TEMPERATURE

j' MATERIAL f = 2 Mc

3.2.3 Results of Firing Series I and II. The density and permeability of some of the cores of Firing Series I and II were given in QPR No. 5. Sufficient data are not yet available to determine how much variation is present between the different core types manufactured in each series or for a comparison between series.

Figs. 2, 3, and 4 show μ_1 versus material types a, c, e, f, g, and h, as defined on p. 18 of QPR No. 5, at different frequencies. These should be interpreted in conjunction with Figs. 11, 12, and 13 of that report. When interpreting the data of Figs. 2, 3, and 4 it is necessary to keep in mind that each plotted point on a curve represents measurements obtained from a different core. Since there are fluctuations in the properties of core subjected to the same manufacturing parameters, the results will deviate from a smooth curve. Present values of μ_2 are not considered sufficiently accurate. Therefore, a better coaxial inductor has been made together with modifications of measurement analysis to obtain μ_2.

Comparison of the frequency spectrum with the equations of Section 4.3 of QPR No. 5 is being undertaken. The permeability curves versus the firing temperature (Figs. 2, 3, and 4) indicate that the reaction which allows wall movement and produces a high value of permeability below resonance is nearly completed at a firing temperature of 1200° C. That the frequency spectrum actually undergoes a resonance can be seen from the negative value of susceptibility at 500 mc/sec for cores fired at temperatures higher than 1150° C. As was expected, the permeability fall-off with frequency of the cores fired below 1000° is much smaller than those fired above. Although accurate data is not available at the moment, the Q of the core is satisfactory only for those cores fired at a low temperature. Once again, as expected, the cores whose characteristics are most

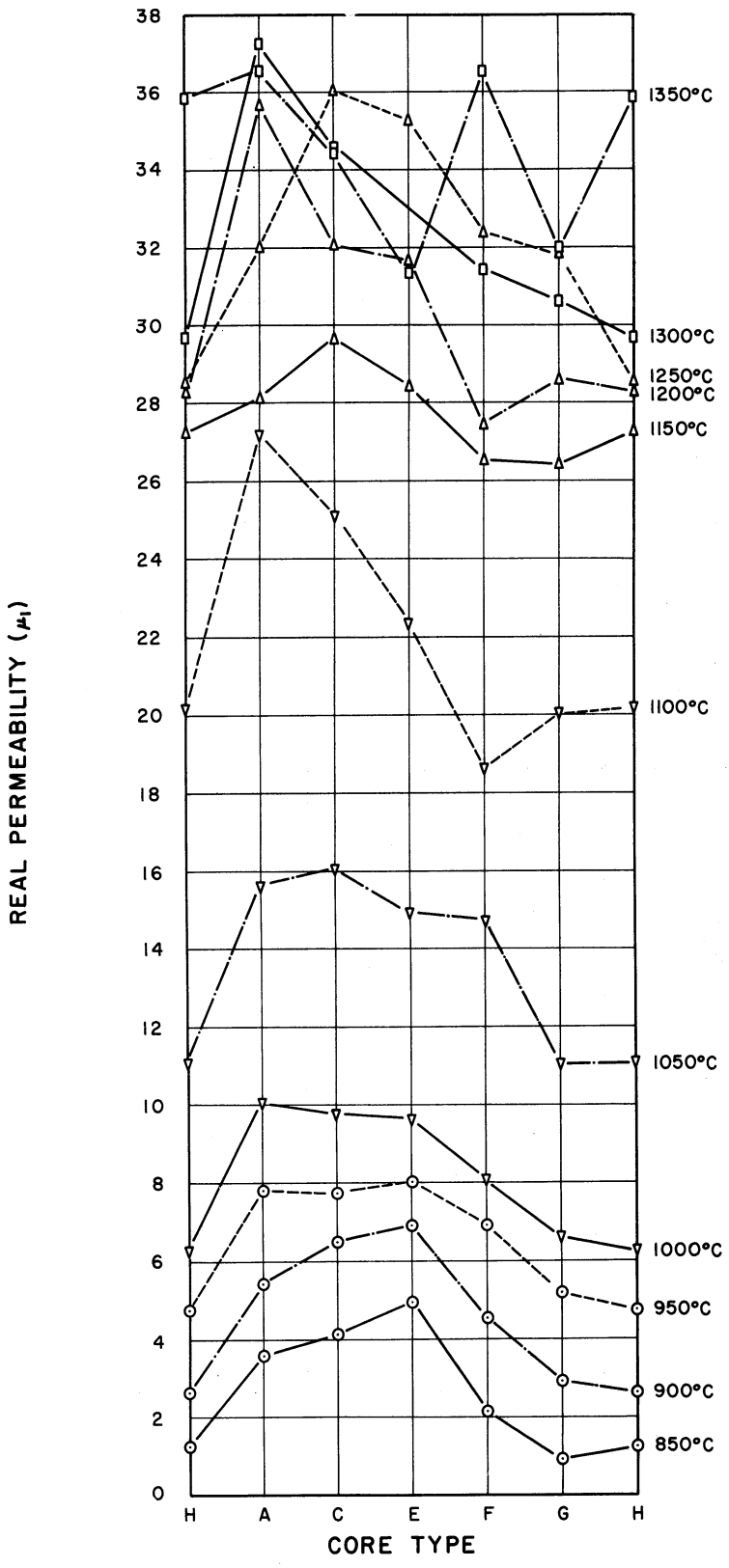

FIG 2

INITIAL PERMEABILITY VS CORE TYPE
f = 100 Mc

FIG 3

INITIAL PERMEABILITY VS CORE TYPE

f = 200 Mc

FIG 4

INITIAL PERMEABILITY VS CORE TYPE

f = 500 Mc

desirable at low frequencies are those cores which were fired at a high temperature producing a large amount of wall movement.

3.2.4 Firing Series III. The object of Firing Series III was to determine the effect of the pressure on the original "pill" and the effect of different mixing procedures. The code designations for the variations will be:*

TABLE I

FIRING TEMPERATURE, °C

Pressing Force, lbs.	1000	1050	1100	1150	1200	1250
250	162	170	178	186	194	202
500	163	171	179	187	195	203
1000	164	172	180	188	196	204
1500	165	173	181	189	197	205
2000	166	174	182	190	198	206
3000	56	64	72	80	88	96
4000	167	175	183	191	199	207
5000	168	176	184	192	200	208
6000	169	177	185	193	201	209
Mixing:						
a) Ultrasonic						
300 kc/sec			210	214	218	222
500 kc/sec			211	215	219	223
750 kc/sec			212	216	220	224
1000 kc/sec			213	217	221	225
b) Ball Milling						
10 min.			226	228	230	232
24 hrs.			227	229	231	233

* For an explanation of the code system see p. 18 of QPR No. 5.

The numbers represent the code designation for each material. To indicate a nickel-zinc ferrite, all numbers carry the prefix A.

Because the dies stick enough to make the total force applied to the die quite different from that felt by the material the green density of each core was measured and taken as the real criteria of the pressure. The variation of green density at a constant applied force can be seen from the example of 3000 lbs. applied force. Here the green density was measured as: 2.14; 2.28; 2.33; 2.37; 2.26; 2.28; 2.36; 2.29.

The ultrasonic mixing was done for us by the General Electric Company. They were insonated for one minute at 300 kc/sec, then at the frequency listed in Table I for another minute and returned to us. A period of perhaps a week elapsed between the insonating and the removal of the oxides from the slurry. It is not known how much the material settled.

The following observations were made on the surface of the material mixed by the different methods. The material ball milled for 24 hours and fired at 1150° C showed indented regions up to a millimeter in diameter with a metallic luster as compared with the brownish black of the rest of the core. These indentations ranged in size down to as small as could be seen on the 500 power of the microscope. The material which had been ball milled for shorter periods of time and that had been insonated showed fewer large holes and more small holes. The 24 hour ball milled material showed about 10 spots per cm^2 with a diameter of over 0.5 mm, and about 200 spots of the order of .01 mm. The 10 minute ball milled material showed about 1 large spot per cm^2 and around 400 of order .01 mm diameter. The insonated material looked very much like the 10 minute material.

The pits are seemingly a product of shrinkage due to grain coagulation. Thus the large pits presumably occur for a reaction nearing completion. It also appears from preliminary permeability measurements that the better the material is mixed, the larger the fraction of material reacted at a given temperature. The difference between ball milling for 6 and 24 hours is inconclusive as could be expected.

3.2.5 Firing Series IV. Firing Series IV will investigate (a) the effect of completing the reaction of the individual spinels prior to mixing the spinels as compared with mixing of the original oxides. This will be, in essence, a variation of the type i material with a longer and higher calcining temperature. It will also (b) both investigate the effect of additions of univalent material to keep the iron in the ferric valence state when more than 50 mole % iron is introduced and the effect of adding pentavalent vanadium.

3.3 Low Frequency Measurements (B. Hershenov)

The manufacturer of the permeameter is as yet unable to furnish a primary core which will allow measurements in the frequency range 100 to 800 kc/sec for the type B permeameter. Therefore, we are attempting to carry out these measurements at given fixed frequencies in this range by winding a General Ceramics type Q core as the primary. This core gives agreement with the core supplied by the manufacturer within 6% at 1 mc/sec. It has not yet been evaluated at lower frequencies.

3.4 P_1 and P_2 Contours (P. E. Nace)

The calculations required to determine permeability and losses of a ferrite toroid from impedance measurements on the coaxial inductor are laborious. If Zg and Θg are the impedance and phase angle of the coaxial inductor as measured on the Hewlett-Packard VHF bridge at a frequency f, one must make the following calculations:

$Zg' = Zg\ (1 + a)$ where a is a small percentage correction obtained (1)
from calibration curves supplied by the manufacturer.

$\Theta g' = \Theta g \frac{f}{f_o} + \Theta_c$ where Θ_c is a small correction likewise obtained (2)
from the manufacturer's calibration curves. f_o = 100 megacycles/sec.

$$Z_L^2 = \frac{(Zg' \cos \Theta g')^2 + (Zg' \sin \Theta g' - Zg'')^2}{\left[1 + \frac{Zg''Zg'}{Z_o^2} \sin \Theta g'\right]^2 + \left[\frac{Zg''Zg'}{Z_o^2} \cos \Theta g'\right]^2} \tag{3}$$

$$\Theta_L = \arctan \frac{Zg' \sin \Theta g' - Zg''}{Zg' \cos \Theta g'} + \arctan \frac{\frac{Zg''Zg'}{Z_o^2} \cos \Theta g'}{1 + \frac{Zg'Zg''}{Z_o^2} \sin \Theta g'} \tag{4}$$

where Z_o is the characteristic impedance of the short transmission line joining the toroidal section of the coaxial inductor to the point of measurement in the bridge. $Zg'' = Z_o \tan \frac{2\pi\ell}{\lambda}$ where ℓ is the length of the transmission line.

Finally: $$\mu_1 = 1 + \frac{f_o}{4\pi f\ t\ \ell n \frac{r_2}{r_1}} (Z_L \sin \Theta_L - Z_A) \qquad \text{(MKS units)} \tag{5}$$

$$\mu_2 = \frac{f_o Z_L \cos \Theta_L}{4\pi f\ t\ \ell n \frac{r_2}{r_1}} \tag{6}$$

Here $Z_A = \mu_o df\ \ell n \frac{r_o}{r_i}$; r_o and r_i are the outer and inner radii of the coaxial inductor. d is the length of the toroidal section of the coaxial inductor. t = the thickness of the core. r_2 and r_1 are the outer and inner radii of the core. For all of our cores $\frac{r_2}{r_1}$ is a constant.

These calculations are too long if one is faced with hundreds of measurements. Fortunately, at a given frequency the factor t is the only quantity that changes with measurements on different cores. Therefore charts can be made in terms of contours involving P_1 and P_2 which are defined by:

$$P_1 = t(\mu_1 - 1) \quad \text{and} \quad P_2 = t\,\mu_2$$

The contours are computed from the following implicit expressions which can be derived from the formulas given above:

$$Zg' \sin \theta_1 = \frac{P_1K + Z_A + Zg''}{1 - \frac{Zg''}{Z_o^2}(P_1K + Z_A - P_2K \cot \theta g')} \tag{7}$$

$$Zg' \cos \theta_1 = \frac{P_2K}{1 - \frac{Zg''}{Z_o^2}(P_1K + Z_A + P_2K \tan \theta g')} \tag{8}$$

where $K = \frac{4\pi f}{f_o} \ell n \frac{r_2}{r_1}$

The contours are calculated as follows:

1. Select a value for P_1 and a value for P_2.
2. Guess a value of $\theta g'$
3. Calculate $\theta_1 = \arctan\left(\frac{Zg' \sin \theta_1}{Zg' \cos \theta_1}\right)$ and compare θ_1 with $\theta g'$. This procedure is repeated until θ_1 is obtained equal to $\theta g'$. Then one calculates Zg' from either Eq. 7 or 8.
4. Next, one calculates θg and Zg from Eqs. 1 and 2.
5. Finally, one plots the calculated data on the Zg-θg plane, joining points of constant P_1 to form P_1 contours and similarly for P_2 contours.

One need only take the measured impedance Zg and phase angle θg, use the P contours to obtain P_1 and P_2, and divide by t to obtain μ_1-1 and μ_2. P-contours have been calculated for two frequencies, 50 and 200 mc/s. They will be calculated for some additional frequencies. They will result in a material savings of time and labor.

3.5 Experimental Determination of the Heat Capacity of 20-30 Nickel Zinc Ferrite
(E. F. Westrum Jr.)

The heat capacity of this material was determined over the range 5.5 to 300° K in the adiabatic calorimeter described in Section 4.57 of QPR No. 1, Task Order EDG-6. The cryostat and manner of operation are also described in the same section. The actual sample contained (laboratory designation W-5) is described more fully in Section 3.32 of QPR No. 2, Task Order EDG-6. The pressure of helium gas in the sample container was 3.8 cm. similar to that used during heat capacity measurements on 10-40 Nickel-Zinc Ferrite.

The sample (code 48-70) was prepared by this task group. It was fired at 1200° C for four hours, then cooled at 60° C/hour in an oxygen atmosphere to 400° C. Although the same cooling rate was employed to the ambient room temperature the oxygen flow was discontinued below 400° C. Analysis indicated 0.2% by weight ferrous iron present. To correct this situation the material was reground, refired to 900° C for 85 minutes and cooled in oxygen as before.

The sample was prepared from C.P. grade oxides mixed by weight to the following composition:

Fe_2O_3	66.984%
NiO	12.533%
ZnO	20.483%

corresponding to a theoretical 46.84% iron. Determination of the total iron present by titration with potassium dichromate indicated 46.8 ± 0.1% total iron.* The determination of ferrous iron in the final material was less than 0.1% by weight. The empirical chemical formula therefore may be represented as

$$\left(NiO_{0.4}ZnO_{0.6}\right) Fe_2O_3$$

with a gram formula weight of 238.404 grams. The sample employed for the heat capacity measurements weighed 203.4344 grams.

The heat capacity data are presented in Table II, per gram formula weight, in terms of a defined thermochemical calorie equal to 4.1840 absolute joules. The values of the heat capacity are believed to be accurate to within ± 0.2% above 35° K with an uncertainty which increases to about ± 0.5% at 20° K and to several percent at the lowest temperatures. These data have been tabulated as C_s (equilibrium values at the 3.8 cm pressure of helium within the calorimeter) and hence are virtually identical with C_p (heat capacity at constant pressure) values and have been corrected for curvature to true heat capacities.

The graphical presentation of the heat capacities (Figure 17 of QPR No. 5) reveals that the heat capacity is a very smooth function of the temperature and that the data are very precise. Comparison of these data with those made earlier on Ferramic-E indicates a generally parallel behavior in the two materials of somewhat similar composition. A slight hump in the heat capacity appears in the region of 10° K. This is however, small in comparison with that found in the 10-40 nickel-zinc ferrite.

Plans for the immediate future include preparation of samples and determination of the heat capacity of 5-45 and 15-35 nickel-zinc ferrite materials

* The determinations were made by Mr. C. F. Jefferson.

TABLE II

HEAT CAPACITY OF 20-30 NICKEL ZINC FERRITE

$$\left(NiO_{0.4}ZnO_{0.6}\right) Fe_2O_3$$

Gram Formula Weight = 238.404 0° C = 273.16° K

SERIES I

T, °K	ΔT, °K	C_8 cal(gm form. wt)$^{-1}$deg^{-1}
63.23	5.592	6.324
69.11	6.167	7.354
75.83	7.285	8.556
83.16	7.374	9.900
91.49	9.270	11.42
90.54	7.468	11.25
98.30	8.063	12.61
106.78	8.890	14.10
115.28	8.120	15.59
123.12	7.553	16.93
130.70	7.608	18.19
138.60	8.193	19.45
147.06	8.718	20.77
155.74	8.652	22.06
164.52	8.892	23.30
173.47	9.012	24.51
182.58	9.207	25.65
191.87	9.372	26.77
201.18	9.239	27.83
210.40	9.203	28.83
219.59	9.177	29.73
228.65	8.940	30.61
237.58	8.921	31.41
246.56	9.062	32.19
255.62	9.050	32.91
264.65	9.008	33.62
273.70	9.094	34.30
282.71	8.946	34.92
291.70	9.042	35.52
300.83	9.217	36.09

TABLE II (continued)

SERIES II

T, °K	ΔT, °K	C_8 cal(gm form. wt)$^{-1}$deg^{-1}
4.50	0.267	0.0682
4.87	0.623	0.0659
5.65	1.096	0.0845
4.75	0.668	0.0698
5.58	1.087	0.0818
5.49	1.188	0.0792
6.66	1.287	0.1052
7.87	1.183	0.1420
8.96	1.060	0.1836
10.00	1.047	0.2317
11.11	1.202	0.2569
12.38	1.367	0.2950
13.77	1.449	0.3415
15.27	1.555	0.4018
16.86	1.629	0.4653
18.52	1.704	0.5393
20.28	1.813	0.6274
22.17	1.982	0.7363
24.37	2.422	0.8862
26.80	2.447	1.073
29.37	2.697	1.304
32.14	2.831	1.591
35.29	3.458	1.956
38.67	3.313	2.394
42.16	3.661	2.879
46.10	4.215	3.468
50.74	5.044	4.204
55.57	4.632	5.003
60.34	4.885	5.818
65.57	5.576	6.376

to survey the nature of the low temperature thermal anomaly.

4. CONCLUSIONS

There are two basically different types of processes which determine magnetic behavior, i.e. structure sensitive and structure insensitive. For control of the finished product more knowledge of how to control the structure sensitive properties is essential. Therefore, we have chosen to pick a specific composition and to see what could be done with the structure sensitive effects. These we believe to depend upon the internal demagnetizing factors and the internal stresses.

From Firing Series I we conclude that the variation in properties with temperature is so rapid in the region of 1050 to 1200° C that it is necessary to use rather elaborate methods of temperature control. At higher temperatures high values of permeabilities exist, but the Q falls rapidly with frequency above a few megacycles. The exact value of the frequency where the fall off occurs is controllable to a degree. This value, for a given composition, usually decreases with increasing firing temperature. In this temperature region, the green density and thus the pressure is unimportant.

Cores to be used above the frequency of rapid drop in Q must be fired at a low temperature. This does not preclude the possibility of firing an initial powder at a high temperature, mixing with oxides and refiring at a low temperature. In this temperature region the permeability spectrum indicates magnetization by rotation.

Oxides mixed by chemical coprecipitation as hydroxides and carbonates become slightly ferromagnetic at 110° and quite magnetic at 350° C.

5. PROGRAM FOR THE NEXT INTERVAL

Analysis of the firing series will continue. The high frequency measurements will be considerably speeded by the P contours. The effect of green densities upon low fired material is being examined from the cores of Series III. Series IV will continue, mixing by chemical coprecipitation will be carried to the point where several cores will be tested. The future program will depend upon these results.

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