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Division 6 - Lincoln Laboratory
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SUBJECT: B-H LOOP SQUARENESS IN THE MAGNESIUM-MANGANESE FERRITES

To: David R. Brown

From: J. B. Goodenough

Date: October 22, 1953

Abstract: A mechanism is proposed whereby a tetragonal $2MOMnO_2$ phase is precipitated at $\{101\}$ surfaces in manganese-rich spinel systems. The critical Mn^{+4} content for precipitation is estimated from studies of lattice parameters in the Fe_3O_4-Mn_3O_4-$ZnMn_2O_4$-$ZnFe_2O_4$ system. A prediction is then made as to which compositions in the MgO-Fe_2O_3-Mn_3O_4 system will give favorable B-H loops. A large region is found to depend critically upon the firing atmosphere as a result of the variability of the degree of ionization of both the iron and manganese ions. It is suggested that both the original reacting and final firing should be done in oxygen-free atmospheres for optimum results in this region of the compositional diagram. A relatively high electric conductivity is also predicted for materials with these compositions. Finally the theoretical predictions are compared with some preliminary experiments which are being done in the chemistry section of Group 63 under the direction of F. E. Vinal.

I. Introduction

A magnetic memory core for a high-speed digital computer must, among other things, have a square B-H loop and a fast switching time. Eddy-current damping increases flux-reversal times. Ferrite systems with low electrical conductivity are therefore of interest as possible memory-core materials. Since production considerations require the use of polycrystalline samples which are prepared by sintering pressed metal oxides, the first problem is to obtain a polycrystalline ferrite which exhibits a square B-H loop.

A physical analysis of the factors which influence the shape of a B-H loop has already been made.[1] A criterion for a square B-H loop was found to be $H_n \geq H_c$ where H_n is the field strength at which new domains of reverse magnetization are formed. (Only the H_n of those reverse domains which are created within the hysteresis cycle directly effect the B-H loop.) The analysis of the lattice imperfections at which reverse domains may be nucleated showed that although granular inclusions strongly influence the coercivity of a material, they do not act as nucleating centers for mobile domain walls. Rather grain boundaries and lamellar precipitates, if present, were found to be the significant nucleation centers for mobile domains of reverse magnetization. The critical field strength for reverse-domain creation at these lattice imperfections is

$$H_n \propto (\sigma_w - \alpha\omega^{*2})$$

where α is a constant which depends upon the geometry of the created domain and the distance between internal planes of magnetic-pole surface density ω^*. The energy per unit area of 180° Bloch wall is σ_w. At a grain boundary $\omega^* = I_s(\cos\Theta_1 - \cos\Theta_2)$ where Θ_1 and Θ_2 are the angles the magnetization vector in two neighboring grains makes with the normal to their common boundary. At a lamellar precipitate $\omega^* = \omega_\ell^* = (I_s \cos\Theta_s - I_p \cos\Theta_p)$ where I_p is the spontaneous magnetization of the precipitate. If a material has a large magnetostriction coefficient, $(\cos\Theta_1 - \cos\Theta_2)$ will be stress sensitive, and the shape of the B-H loop will be stress sensitive. If lamellar precipitates are present, however, ω_ℓ^* will be relatively insensitive to stress.

Currently the most successful materials for memory cores come from the magnesium-manganese-ferrite system. These materials, if judged by the sensitivity of the B-H loop to externally applied stress, have relatively low magnetostrictive constants. The squareness of the B-H loop in these materials is not, therefore, attributed to internal stresses. It is rather believed due to a value of $\sigma_w/\omega^{*2} > \alpha$, or to a positive H_n. Although there is no grain orientation to reduce $(\cos\Theta_1 - \cos\Theta_2)$, the spontaneous magnetization in these materials is an order of magnitude smaller than in iron whereas $\sigma_w \propto \sqrt{KA}$ is as large as in iron. The anisotropy constant K will be sensitive to the chemical composition of the material. The exchange parameter A will

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[1] J. B. Goodenough and N. Menyuk, "A Theory of Domain Creation, Coercive Force, and Flux Reversal in Polycrystalline Ferromagnets", Lincoln Technical Report T-40 (1953).

probably be less sensitive to small variations in chemical composition.

It is of interest to predict, in the light of this analysis, in which compositional areas of the MgO-Fe_2O_3-MnO diagram the most satisfactory memory cores are to be found. In Section II an analysis is made of the mechanism of precipitation of lamellar $2MO \cdot MnO_2$. The symbol M standsfor a bivalent metal cation. In those compositional regions in which extensive lamellar precipitation occurs, $H_n < 0$ and the B-H loop is not square. In Section III an analysis is made of those compositional regions in which lamellar precipitation of $2MO \cdot MnO_2$ may be expected to occur. It is also shown how this region will change its area under different firing atmospheres. In Section IV there is a comparison of these predictions with the results of a preliminary experimental investigation of this system.

II. Precipitation of Mn_3O_4 on {101} Planes

Hausmannite (Mn_3O_4) has a tetragonal spinel structure with c/a = 1.16.[2] Its electrical conductivity is $10^8 - 10^9$ smaller than in Fe_3O_4.[3] Verwey and his co-workers have proposed that Mn_3O_4 consists of $2MnO \cdot MnO_2$ rather than $MnOMn_2O_3$ and that the spinel is inverse with the Mn^{+4} and Mn^{+2} ions so ordered in the octahedral sites as to occupy alternate planes parallel to the c axis.[4] Such ordering is similar to that found in magnetite (Fe_3O_4) below 120° K.[5] The suggestion was supported by a calculation of the Madelung-energy gain due to ordering. Further support for this hypothesis is given by Goodenough and Loeb[6] who also propose that γ-Mn_2O_3, which is tetragonal spinel with c/a = 1.16, is composed of $MnO \cdot MnO_2$ with a similar ordering of Mn^{+4} and Mn^{+2} ions on the octahedral sites.

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[2] G. Aminoff, "Uber die Kristallstruktur von Hausmannite ($MnMn_2O_4$)" Z. Krist. 64, 475-90 (1926).

[3] E. J. Verwey and J. H. de Boer, "Cation Arrangement in a Few Oxides with Crystal Structures of the Spinel Type," Rec. Trav. Pays-Bas 55, 531-54 (1936).

[4] E. J. Verwey et al., "Die Verteilung der Metallionen im Spinellgitter und deren Einfluss auf die Physikalischen Eigenschaften," Z. Physik. Chem. 198, 6-22 (1951).

[5] E. J. Verwey, P. W. Haayman, and F. C. Romeijn, "Physical Properties and Cation Arrangements of Oxides with Spinel Structures: II. Electronic Conductivity," J. Chem. Phys. 15, 181-187 (1947).

[6] J. B. Goodenough and A. Loeb, "Ordering in Manganese Oxides with Spinel Structure", Digital Computer Laboratory Memorandum M-2474, October 22, 1953.

Mason[7] has made a study of the variation of lattice parameters with composition in the Fe_3O_4-Mn_3O_4-$ZnMn_2O_4$-$ZnFe_2O_4$ system. His measurements of c/a and his phase diagram for this system are reproduced in Figures 1 and 2. The iron-rich portion of the two-phase region (Vredenburgite) is reported to consist of a lamellar precipitation of Mn_3O_4 in a cubic matrix. A mechanism for the formation of this lamellar precipitate is now proposed.

It is assumed that Manganese is present in the spinel systems as Mn^{+2} and Mn^{+4}. It is further proposed that distortions of the lattice from cubic to tetragonal symmetry are the result of an ordering of the Mn^{+4} ions into every other plane parallel to a c axis. Since all three cubic axes have an equal probability of becoming a c axis, twinning results along $\{101\}$ planes of the lattice in order to relieve the internal stresses which result. Such a twinning surface is shown diagramatically in Figure 3(a). After twinning has occurred, the twinning surface becomes a region of low stress. Therefore there is a stress gradient on the odd-sized Mn^{+4} ions to cause them to diffuse toward the region of low stress. This gradient is proportional to eE/a where E is Young's modulus and the strain e is given by $c/a = (1+e)$. Diffusion of the Mn^{+4} ions toward the twinning surface will stop when the bulk matrix becomes cubic and $e = 0$. If the spinel lattice is maintained, M^{+2} ions must diffuse with the Mn^{+4} ions in order to maintain local electrical neutrality. It is assumed that cubic and tetragonal spinels are the only lattice types present. In Figure 3(b) is shown a diagram of the B-site lattice after precipitation is complete. Tetragonal $2MO \cdot MnO_2$ has precipitated at the twinning surface with its c axis in a $[\bar{1}01]$ direction parallel to the $\{101\}$ twinning surface. The bulk matrix becomes cubic when precipitation is complete. If precipitation is not complete, the bulk matrix is tetragonal, but its c/a ratio is smaller than that of the precipitate.

In support of this mechanism of lamellar precipitate formation, lattice-dimension and phase measurements for three compositions, marked R, S, BB in Figure 2, are taken from Mason's work and recorded in Table I. At compositions R and BB precipitation is not completed until lower temperatures are reached. Each of these cases is on the Manganese-rich side of the two-phase

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[7] B. Mason, "Minerological Aspects of the System Fe_3O_4-Mn_3O_4-$ZnMn_2O_4$-$ZnFe_2O_4$," American Minerologist 32, 426-40 (1947).

region where considerable precipitation must occur before the parent matrix can become cubic. This view is quite consistent with the fact that the temperature range over which precipitation occurs increases with Manganese content. It is significant that the tetragonal phase of smaller c/a ratio becomes cubic. Composition BB shows a continuous decrease in the c/a ratio of the parent lattice until it becomes cubic whereas there is little change in the c/a ratio of the precipitate. This is just the behavior predicted by the proposed precipitation mechanism.

Table I

Prep.	Composition				Phases	Cubic	Tetragonal			Remarks
	Fe	Mn	Zn	T		a	a	c	c/a	
R	16.7	50.0	33.3	890	T		8.162	8.991	1.102	
				800	T+(T_1)		8.120	9.107	1.121	Brackets indicate small amount
							(8.204)	(8.872)	(1.081)	
				710	T+(C)	(8.423)	8.114	9.115	1.123	
				590	T+(C)	(8.422)	8.111	9.139	1.127	
S	26.7	40.0	33.3	920	T		8.217	8.869	1.080	
				810	T		8.214	8.861	1.079	
				700	C+T	8.424	8.125	9.099	1.120	
				600	C+T	8.422	8.117	9.121	1.124	
BB	18.8	56.2	25	1000	T		8.181	9.065	1.108	
				920	T+T_1		8.138	9.143	1.124	Brackets indicate small amount
							(8.183)	(9.025)	(1.103)	
				810	T+T_1		8.137	9.136	1.123	
							(8.212)	(8.953)	(1.090)	
				710	T+T_1		8.133	9.136	1.123	
							(8.250)	(8.934)	(1.083)	
				600	C+T	8.433	8.129	9.169	1.128	

III. Prediction of Compositions with Square B-H Loops

If lamellar $2MO \cdot MnO_2$ is readily formed in a spinel system which is rich in Mn^{+4} and if the presence of extensive lamellar precipitation prevents the occurance of a square B-H loop, as has been proposed, it should be possible to predict in which compositional regions of the $MgO-Fe_2O_3-MnO$ system square B-H loops are attainable. If, further, there were information available on the variations of K, A, and I_s with composition, it should be possible to predict an optimum compositional value. This latter accomplishment must await measurements of the physical constants. A rough prediction of the general regions in which optimum core characteristics are to be found can , however, be made on the basis of our present knowledge.

It is known experimentally that in any spinel certain ions prefer the four coordination of a tetrahedral site. The order of preference for ions which are significant to this discussion is Zn^{2+}, Fe^{3+}, Mg^{2+}. This order of preference has little to do with the ion size. Rather it is due to the strength of the covalent bonding between the oxygen ions and the cation in a tetrahedral site. Hybridization of (d^3s) orbits[8] gives four electron orbitals which point to the corners of a tetrahedron. The Zn^{2+} ion characteristically forms covalent bonds with the tetrahedral coordination. The Fe^{3+} ion can also readily hybridize (d^3s) orbitals to enter into covalent bonds of tetrahedral symmetry. However, that $ZnOFe_2O_3$ forms a normal spinel shows Zn^{2+} to have the stronger preference for the tetrahedral site. In the Mg^{2+} ion (p^3s) orbits can be hybridized. These have the tetrahedral symmetry which is necessary for covalent bonding in a tetrahedral site. The energy of the s state is sufficiently above the p-state energies, however, that a smaller amount of energy is gained through this admixture of states than through the admixture of (d^3s) states in Fe^{3+}. This is evidenced by the fact that $MgO \cdot Fe_2O_3$ is mostly inverse spinel.[9] It is concluded, therefore, that in a spinel lattice in which Zn^{2+}, Fe^{3+}, and Mg^{2+} ions are present, the tetrahedral, or A, sites will be occupied by the Zn^{2+} ions.

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[8] G. E. Kimball, "Directed Valence," J. Chem. Phys. 8, 188-98 (1940).

[9] L. M. Corliss, J. M. Hastings, and F. G. Brockman, "Neutron Diffraction Study of Magnesium Ferrite," Phys. Rev. 90, 1013-18 (1953).

If there are still some vacant A sites, these will be predominatly occupied by Fe^{3+} ions. The Mg^{2+} ions will be assumed, in the arguments to follow, to occupy the octahedral, or B, sites.

If manganese is present in the system, the Mn^{2+} ion, which has the same outer electron configuration as Fe^{3+}, will also have a preference for covalent bonding in the tetrahedral site. Since $MnO \cdot Fe_2O_3$ appears to be an inverse spinel,[10] it is concluded that the Fe^{3+} ions have the greater preference for the A sites. In the considerations which follow, all Mn^{2+} ions will be considered to occupy B sites unless there are not sufficient Zn^{2+} or Fe^{3+} ions to fill the A sites. The Fe^{2+} ion, with six outer electrons, can form a six-fold covalent coordination. It will be considered to always occupy B sites as it does in magnetite (Fe_3O_4). The Mn^{4+} ion will also be considered to always occupy B sites as it does in Hausmannite (Mn_3O_4).[6]

Just as the tetragonality of Mn_3O_4 and γ-Mn_2O_3 is attributed to an ordering of the Mn^{4+} ions into every other plane of B sites parallel to a c axis, so it is proposed that tetragonality in any spinel system containing manganese is due to a similar ordering of Mn^{4+} ions. A fraction f will be defined as the fraction of Mn^{4+} ions per B site which are necessary before ordering occurs. Since distortions to tetragonality introduce twinning planes at which the precipitation of $2MO \cdot MnO_2$ can occur, this fraction f will also be the critical fraction for lamellar precipitate formation. If the fraction of Mn^{4+} ions per B site exceeds f, the B-H loop will not be square. The lamellar precipitates will be acting as nucleating centers for domains of reverse magnetization, and $H_n < H_c$.

Although the valencies of both the manganese and the iron ions may be variable, a minimum value of f can be obtained from Mason's[7] work. If the iron ions are all Fe^{3+} in the $ZnFe_2O_4$-$ZnMn_2O_4$ composition at which tetragonality sets in, f can be calculated from Figure 1. The unit cell contains 8 Zn^{2+} on the A sites and 3.1 Mn^{4+} 3.1 Mn^{2+} 9.8 Fe^{3+} on the B sites. This gives $f = 3.1/16$. In the Fe_3O_4-Mn_3O_4 system, the highest manganese content at which tetragonality first occurs is 60 mol percent Mn_3O_4. If all of the iron ions are Fe^{3+} at this composition, the unit cell contains 8 Fe^{3+} on the A sites and 1.6 Fe^{3+} 3.2 Mn^{4+} 11.2 Mn^{2+} on the B sites. This gives an $f = 1/5$. The value $f = 1/5$ is therefore taken as the critical fraction of Mn^{4+} ions per B site. If a

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[10] E. J. W. Verwey and E. L. Heilmann, "Physical Properties and Cation Arrangement of Oxides with Spinel Structures; I. Cation Arrangement in Spinels," J. Chem. Phys. 15, 174-180 (1947).

greater concentration of Mn^{4+} ions is present, lamellar precipitate will presumably be present. The B-H loop is predicted to be nonsquare for these compositions.

The ternary diagram of Figure 4 is marked off into five compositional regions. In Region I MgO will precipitate. It is probable that Fe_2O_3 precipitates in Region II. The nature of these precipitates is not yet known. Suffice it to say that the presence of precipitation in these regions may be expected to destroy the squareness of the B-H loop. In Region III $f > 1/5$ regardless of the degree of ionization of the iron atoms. This will therefore be a two-phase region in which tetragonal $2MOMnO_2$ forms a lamellar precipitate along $\{101\}$ surfaces of a cubic matrix. Materials of these compositions will form magnetic cores with nonsquare B-H loops unless they are rapidly quenched from a temperature above the ordering temperature. A rapid quench may succeed in "freezing in" the disordered arrangement of Mn^{4+} ions. The disordered lattice will be cubic, and the precipitation mechanism will not exist. In Region IV $f < 1/5$ regardless of the degree of ionization of the iron atoms. This represents the region of complete miscibility. The single phase will be cubic, and the B-H loops of cores made from these compositions should have good squareness. The boundary between Regions IV and V represent $f = 1/5$ if all the manganese present is Mn^{4+}. Region V is the compositional region in which the B-H loop shape will be sensitive to firing and annealing conditions.

The magnitude of f can be critically controlled in Region V by the firing and annealing atomspheres used. In an oxygen-rich atmosphere there will be a tendency to form Mn^{4+} ions. If the spinel lattice is maintained, this may be accomplished by a transfer of electrons from an Mn^{2+} to two Fe^{3+} ions to give an Mn^{4+} and two Fe^{2+} ions. More probably, however, the lattice becomes cation deficient as oxygen is added to the lattice. For every oxygen added, an Mn^{2+} becomes Mn^{4+} and a normally-occupied B site becomes vacant.

It has been found experimentally that if a material of composition in Region V is fired in air, the atmosphere is sufficiently rich in oxygen for lamellar precipitation to form. These lamellar precipitates are removed either by quenching rapidly from above the ordering temperature or by annealing in an oxygen-free atmosphere above 1000°C. In the latter process oxygen ions are removed from the lattice and enough Mn^{4+} ions become Mn^{2+} to make $f < 1/5$. Temperatures in excess of 1000°C are sufficient, according to

Mason's data, for the Mn^{4+} ions to diffuse away from a twinning surface at which precipitation has occurred. Since there are no longer sufficient Mn^{4+} ions present to give ordering, the lattice is cubic, and there is no precipitate at room temperature.

It should be noted that some reducing atmospheres may reduce the Fe^{3+} ions to Fe^{2+} and thus force some Mn^{2+} ions to become Mn^{4+}. Controlled oxygen atmospheres may be necessary for optimum conditions.

Finally one can predict an electrical conductivity approaching that of magnetite in those samples which contain disordered Fe^{2+}, Fe^{3+} ions on the B sites. This conductivity will be due to the exchange of electrons between like atoms of different degrees of ionization. The exchange between Mn^{2+} and Mn^{4+} ions will give a lower contribution to the conductivity.[6]

IV. Comparison with Preliminary Experiments

A. Best Squareness Ratios vs. Composition

The chemistry section of Group 63, under the direction of F. E. Vinal, has prepared ferrite cores of a large number of different compositions within the MgO-Fe_2O_3-Mn_3O_4 system. Each composition was fired in air at 1300°C, 1350°C, 1400°C, 1450°C and refired in N_2 at 1100°C. In Figure 5 is a contour map for the best squareness ratios at each composition. In Figure 6 is shown a contour map of the average change in squareness with refiring. The following conclusions are at once apparent.

1. The compositions of favorable squareness lie within Regions IV and V as predicted.
2. The compositions which are sensitive to anneal in an oxygen-free atmosphere lie in Region V as predicted.
3. The lower squareness compositions within Region V are manganese-rich samples which failed to respond appreciably to an anneal in an oxygen-free atmosphere. It is believed that improved squareness can be obtained in these regions.

B. Microstructure Studies

Some microstructures have been prepared by the chemistry section of Group 63 and by A. Kulin of Group 35. These studies have shown lamellar precipitation in air-fired samples of Region V. This precipitate is most commonly found near the surface of the material or at flaws. A typical micrograph for such a region is shown in Figure 7. It corresponds to the composition marked A in Figures 5 and 6. For four materials of this composition originally fired in air at 1300°C, 1350°C, 1400°C, 1450°C, the average increase in squareness ratio with refire in Nitrogen was 0.27.

The General Ceramics Corp. MF-1371 body is located in Region V. The micrograph and B-H loop for an air-fired and steam-fired specimen of this material are shown in Figure 8. Extensive lamellar precipitation is apparent. The precipitation pattern is strongly suggestive of a heavy twinning pattern. Such a precipitate pattern is predicted by Section II and Section III.

C. Miscellaneous

The Group 63 chemistry staff has found that some cores of the 7.5 percent iron-deficient series in the $MgO-Fe_2O_3-MnO$ system are difficult to press and to fire. These compositions are all located in Region V. The raw oxides are first reacted in air at elevated temperatures to allow shrinkage to occur. The product is reground and then pressed into cores for the final firing. Mason[7] found that a material in which lamellar precipitation had just begun crumbled to pieces on handling because of the presence of large internal strains. It is suggested, therefore, that sufficient oxydation of the manganese has occurred during the initial reacting process for precipitation to have begun. If this is true, the difficulty may be removed by reacting the materials initially in an inert atmosphere.

I should like to express my appreciation to F. E. Vinal for access to his data which is yet in incomplete compilation.

Signed John B. Goodenough
John B. Goodenough

Approved DRB
David R. Brown

JBG/jk

Drawings attached:
Figures 1, 2 A-56625
Figure 3 A-56626
Figure 4 A-56627
Figure 5 A-56628
Figure 6 A-56629
Figure 7 A-56630
Figure 8 A-56064

cc: Group 63 staff
S. Foner - Group 37

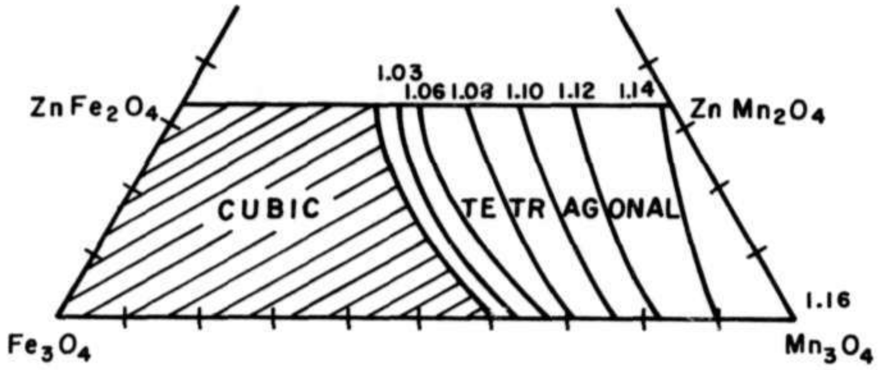


FIG. 1

VARIATION OF THE AXIAL RATIO c/a WITH COMPOSITION.

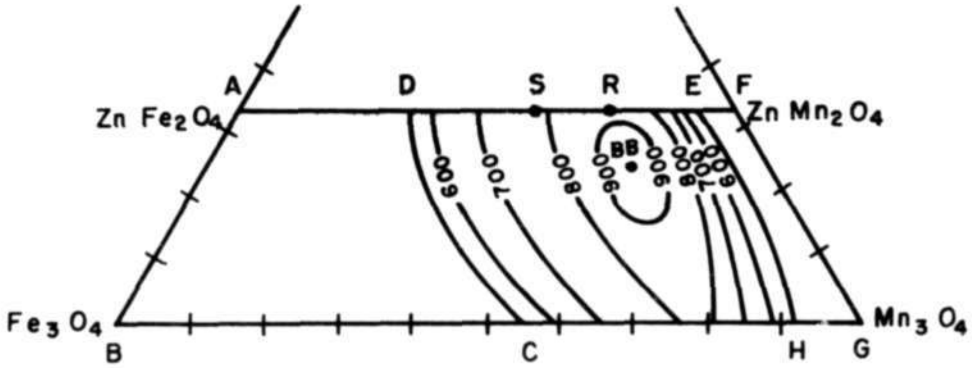
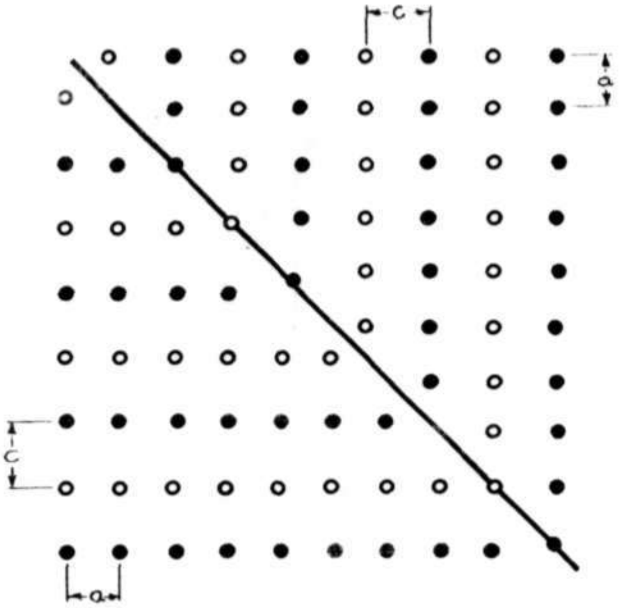


FIG. 2

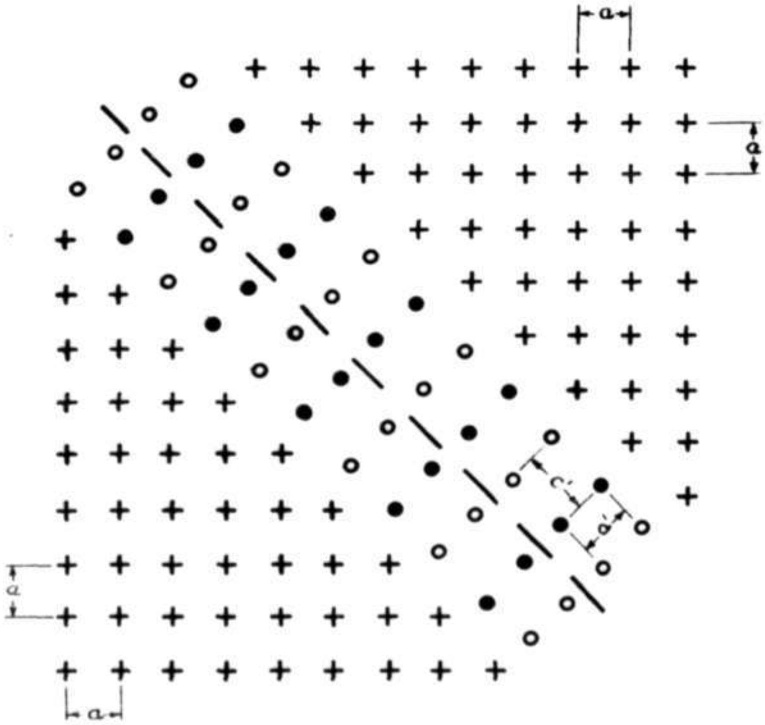
ABCD IS THE FIELD OF CUBIC MIX – CRYSTALS. EFGH IS THE FIELD OF TETRAGONAL MIX-CRYSTALS. CDEH IS THE FIELD OF INCOMPLETE MISCIBILITY; ISOTHERNS SHOW THE BOUNDARIES OF THE FIELD OF INCOMPLETE MISCIBILITY AT DIFFERENT TEMPERATURES.

A – 56625



(a)

B SITES IN A DISTORTED SPINEL LATTICE IN THE REGION OF A {101} TWINNING SURFACE. DARK SITES ARE THOSE WHICH MAY BE OCCUPIED BY Mn^{4+} IONS



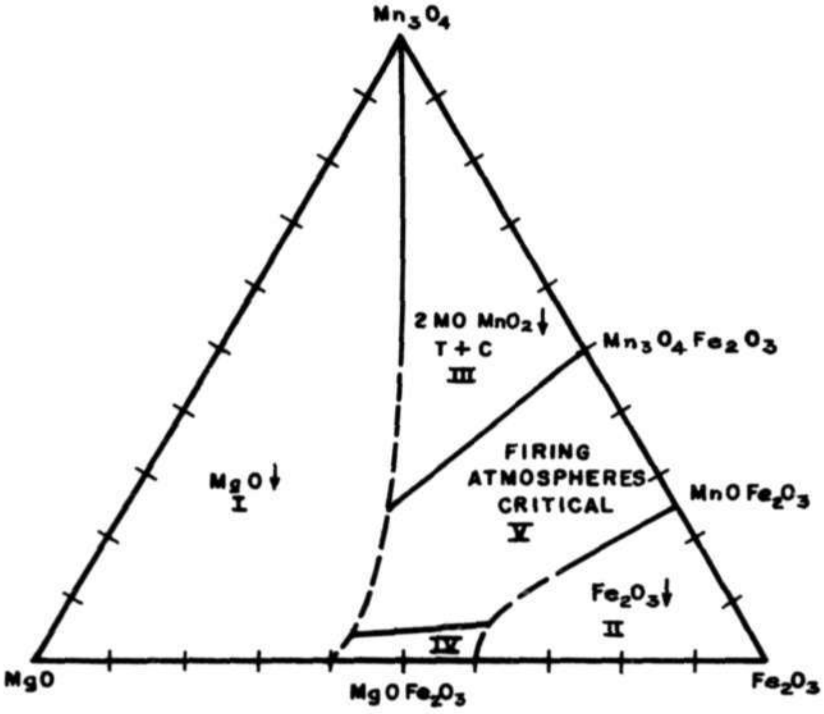
(b)

B SITES AFTER PRECIPITATION IS COMPLETE. DARK SITES ARE OCCUPIED BY Mn^{4+} IONS

FIG. 3

DIAGRAMATIC REPRESENTATION OF PRECIPITATE FORMATION AT A TWINNING SURFACE

A-56626



REGIONS I, II, III GIVE POOR SQUARENESS.
REGION IV FAVORABLE TO GOOD SQUARENESS.
REGION V FAVORABLE TO GOOD SQUARENESS PROVIDED ALL FIRING PROCESSES ARE DONE IN A REDUCING ATMOSPHERE.

FIG. 4

PREDICTED COMPOSITIONAL REGIONS OF HIGH AND LOW B-H LOOP SQUARENESS

A-56627

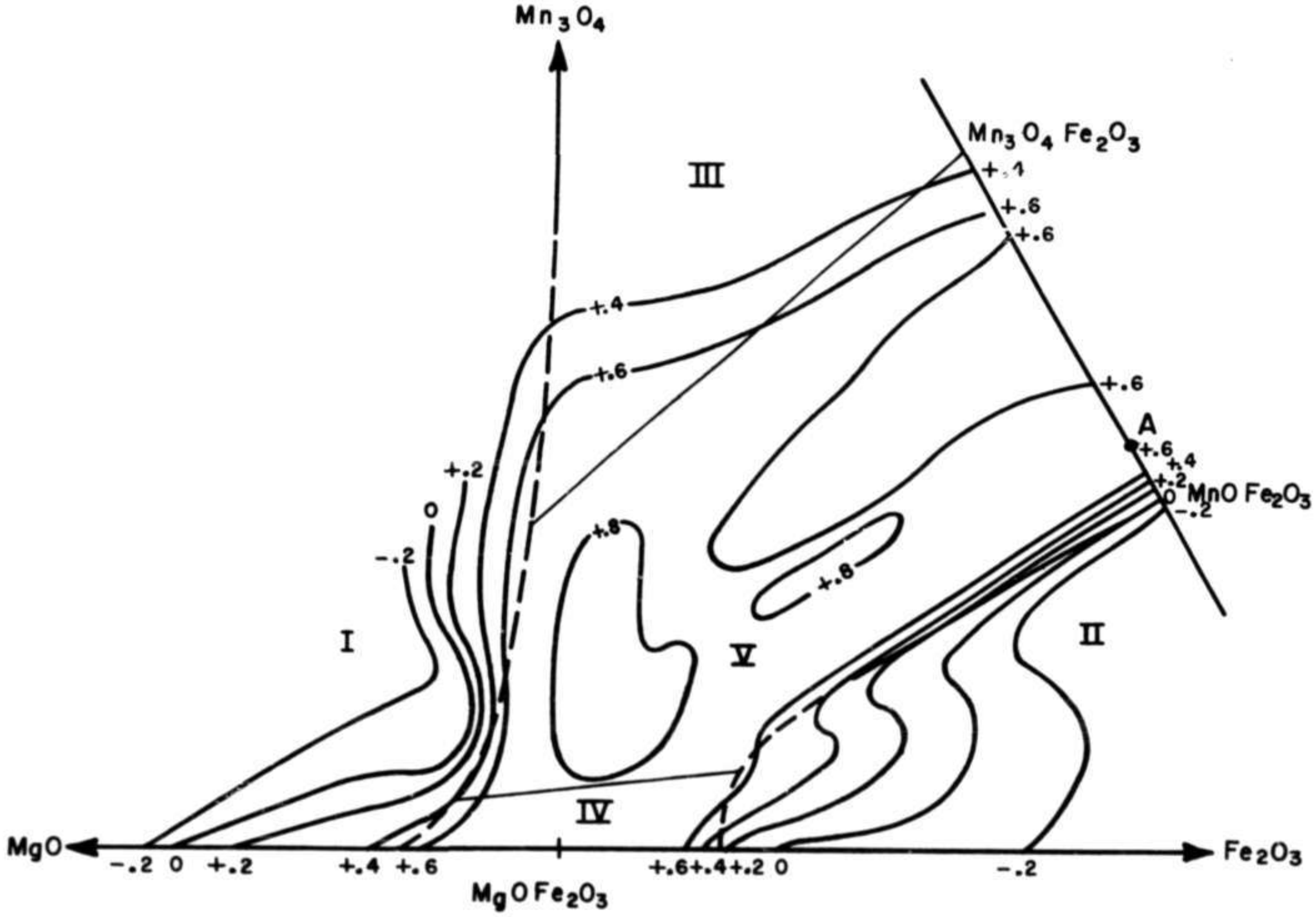


FIG. 5

CONTOURS OF HIGHEST SQUARENESS RATIO $R_s = B_m/B_d$ WHICH HAVE BEEN OBTAINED TO DATE UNDER VARIOUS FIRING AND PROCESSING CONDITIONS.

A-56628

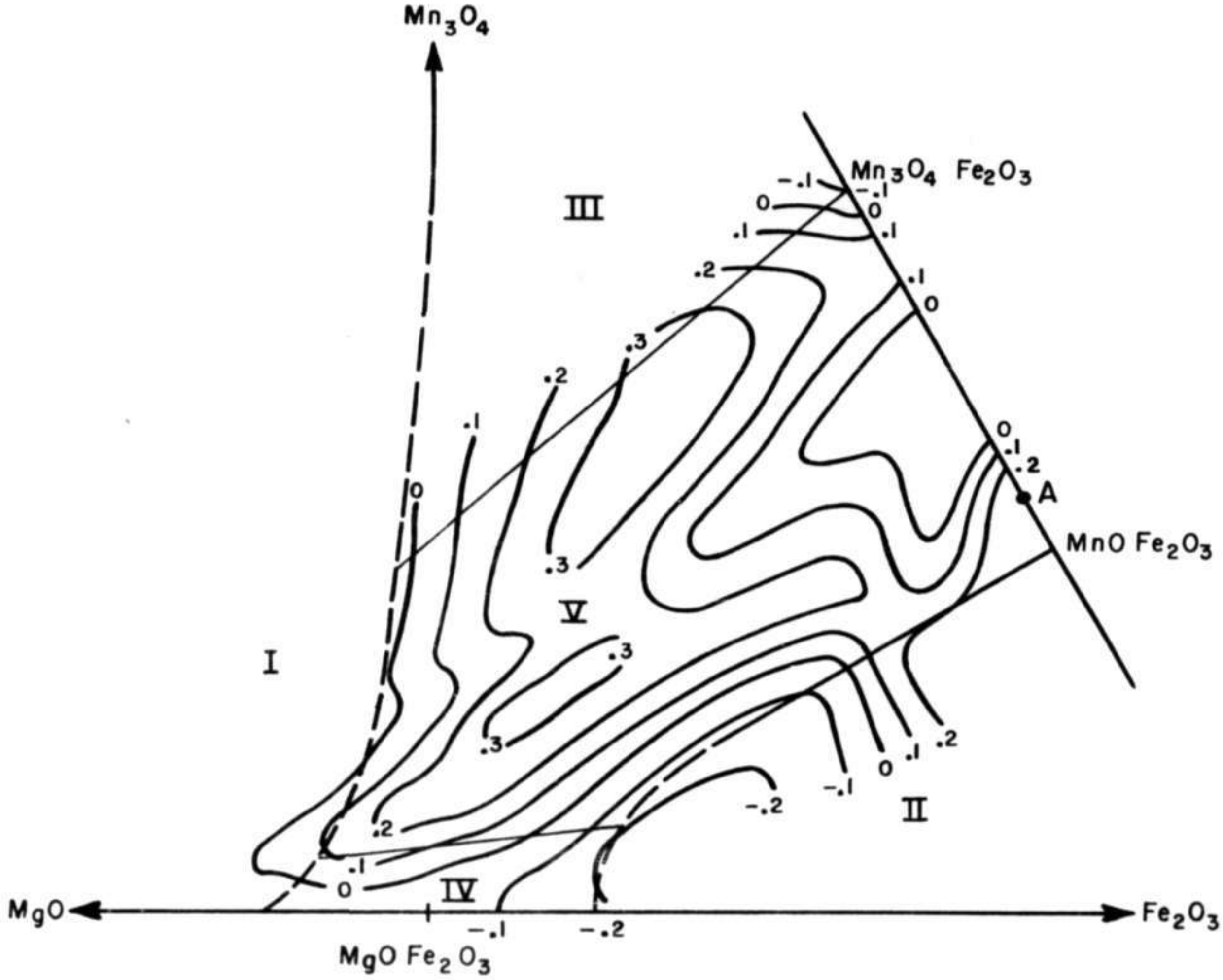


FIG. 6

CONTOURS OF AVERAGE CHANGE IN R_S AFTER REFIRING CORES FOR ONE HOUR AT 1100°C IN AN N_2 ATMOSPHERE. THE CORES WERE ORIGINALLY FIRED IN AIR FOR ONE HOUR AT 1300°C, 1350°C, 1400°C, AND 1450°C.

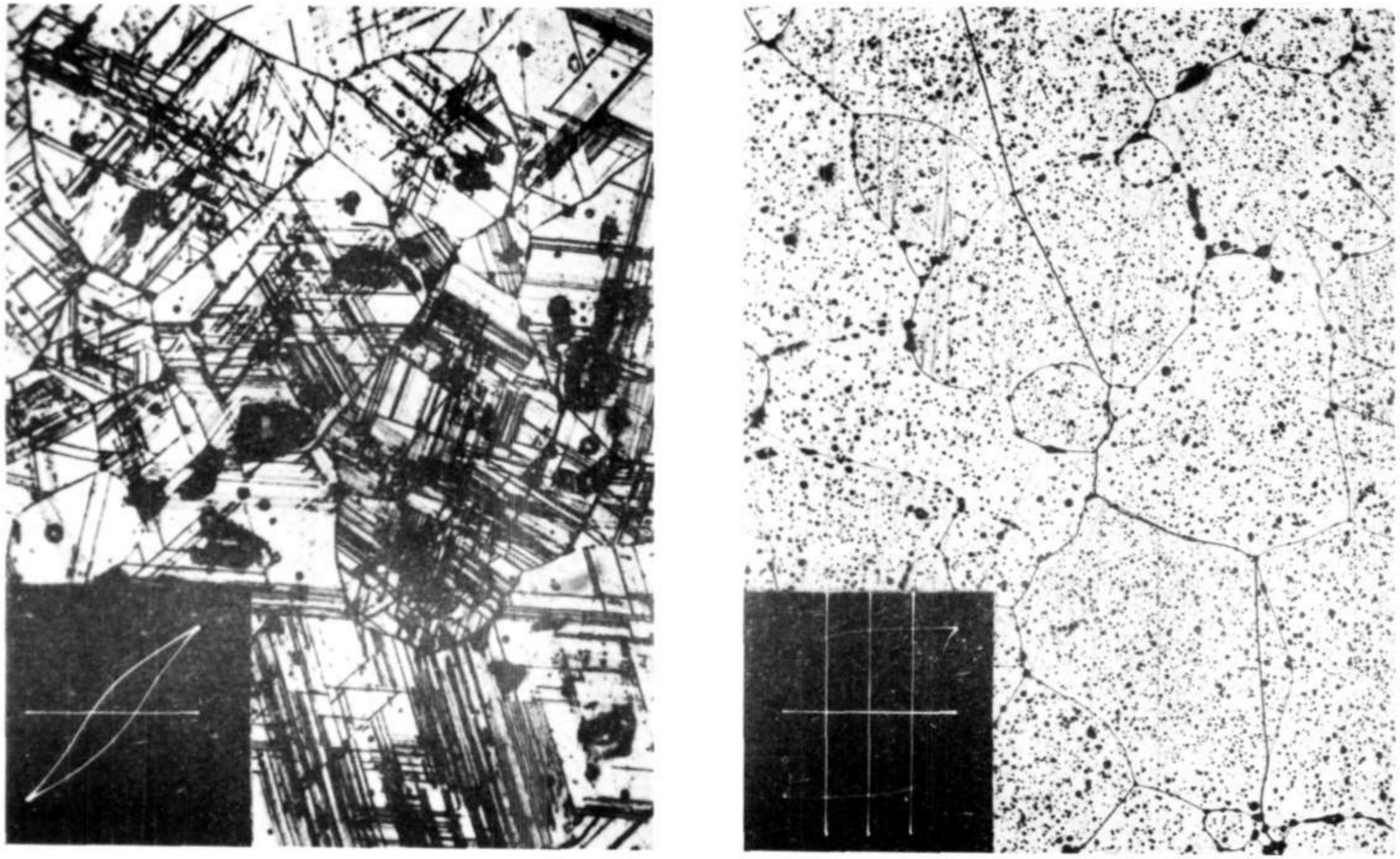
A-56629

FIG. 7

TYPICAL LAMELLAR PRECIPITATE FORMATION IN 55 mol % MnO 45 mol % Fe_2O_3. OXIDES WERE SINTERED IN AIR FOR ONE HOUR AT 1400° C. (WHITE LIGHT X167)

A-56630

A-56064



(a) B_m = 150 GAUSS
H_m = 2.25 Oe

(b) B_m = 1128 GAUSS
H_m = 0.46 Oe

FIG. 8

MICROSTRUCTURE AND MAXIMUM SQUARENESS RATIO B-H LOOP FOR A GENERAL CERAMIC CO. MF 1371B FERRITE BODY (a) BEFORE ANNEAL (WHITE LIGHT X 498) AND (b) AFTER ANNEAL (WHITE LIGHT X 100)