

Division 6 - Lincoln Laboratory
Massachusetts Institute of Technology
Cambridge 39, Massachusetts

SUBJECT: FERRITE SYNTHESIS II

To: D. R. Brown

From: F. E. Vinal

Date: February 17, 1954

Abstract: The systematic investigation of the $\text{MgO-MnO-Fe}_2\text{O}_3$ system for which preliminary results were reported in Memorandum M-2442, has been extended and verified. The patterns of loop squareness vs. composition have remained essentially unchanged, but one new region has been added to the interpretation diagram for the loop-squareness patterns. The tentative phase interpretation which was offered in M-2442 has been confirmed by microstructure studies and has been extended by considering the chemistry of memory-core constituents during heat cycling. A hypothesis is made that any magnetic ferrite phase containing small amounts of Mn_3O_4 in cubic solid solution is potentially square hysteresis-loop material. Magnetic measurements taken on the various compositions include coercive force and magnetic induction at saturation and at maximum loop-squareness conditions as well as flux reversal times at maximum loop-squareness. The magnetic measurements are plotted, using the above compositional hypothesis.

I. HYSTERESIS LOOP SQUARENESS VS. COMPOSITION

A systematic investigation of the $\text{MgO-MnO-Fe}_2\text{O}_3$ system, which has thus far yielded the suitable ferrite memory cores, was undertaken to establish trends in the electrical and magnetic characteristics with changing composition. Progress in this investigation has been reported previously in Memorandum M-2442¹. The observed trends have now been

1. Division 6 - Lincoln Laboratory, M. I. T., Cambridge, Mass.; Memorandum M-2442, Subject: Ferrite Synthesis, To: D. R. Brown, From: F. E. Vinal, September 15, 1953.

extended and verified.

The basic composition diagram which has been used to express the relationships of the raw-material constituents is shown in Figure 1. The compositions are given in terms of the three constituent oxides MgO, MnO, and Fe₂O₃. Using a ternary diagram for these three materials, obvious points of significance are the midpoints of the MgO-Fe₂O₃ side and the MnO-Fe₂O₃ side. These points represent the compounds magnesium ferrite, MgO·Fe₂O₃ and manganese ferrite, MnO·Fe₂O₃ respectively. A line joining these two compositions is the locus of all the possible combinations of MgO, MnO and Fe₂O₃ in which the basic formula for a spinel, AB₂O₄, is satisfied. The compositions on this line are all stoichiometric in character; that is, the sum of the bivalent oxides is chemically equivalent to the trivalent oxide. All other compositions in Figure 1 represent combinations which are not stoichiometric, and hence contain excesses or deficiencies of one or more constituent oxides. The specific compositions thus far investigated are indicated in Figure 2. Each of the approximately 160 compositions shown has been subjected to 8 or 10 variations in processing. The data presented are based therefore on approximately 1600 samples whose properties have been observed.

The maximum loop-squareness obtained for each composition has been plotted on the basic composition diagram and a contour "weather map" prepared, showing in Figure 3 the loop-squareness pattern vs. composition. The extended data have not substantially modified the similar pattern shown in M-2442. The significance of the "stoichiometry line" as a boundary of the good squareness region is apparent, as is the line connecting the MnO apex with the midpoint of the base, MgO·Fe₂O₃. Within the area defined by these two lines, squareness deteriorates as compositions of higher manganese content are prepared. Although no clear-cut boundary is observed in the MnO direction, it is expected that this boundary will clear-up with further work. A simplified diagram for the interpretation of the loop squareness data is shown in Figure 4. The interpretation of the phases, first suggested in M-2442, has now been confirmed by microstructural examination of specimens. Briefly, region A + B + C is single phase, homogenous material while regions D, E and F are two-phase regions. Area A, containing all of the very-good-to-good squareness

values, is a cubic spinel phase while regions B and C are areas of limited solid solution of MgO and Fe_2O_3 , respectively, in the spinel phase. Fair-to-good loop squareness is possible in areas B and C but the usefulness of these compositions is limited by high firing temperatures and high values of coercive force. For good memory cores consideration may be limited to region A. The contours within region A are interesting because the best area appears, in general, to be in the region of high $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ content, diverging toward the $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ composition and the MnO apex. The deterioration of squareness in the upper central portion of region A is not fully understood, as verification of results in this area, now partially completed, indicate that with careful processing, values in this region may be improved. No improvement has been obtained for compositions close to the MnO apex.

Regions D, E and F are two-phase regions. The examination of suitable series of specimens by metallographic techniques shows clearly the increasing proportions of second phase as the boundaries of region A are crossed to the left and to the right. Region D lying to the left of region A shows a second phase of MgO appearing, at first as scattered grains in the spinel-phase matrix. Later, with increasing MgO content, the MgO may become the matrix and contain isolated grains of the spinel. These results might be expected because all compositions lying to the left of the center line in the composition diagram are so deficient in Fe_2O_3 that not even the MgO constituent alone may be satisfied. The granular appearance of the second-phase MgO is consistent with its cubic character. High electrical resistivity is known to be a characteristic in region D and creates some interest for applications where other qualities may be partially sacrificed to obtain high resistivity. Region E, in the lower right, is also a two-phase region but differs from D in that the second phase appears as lamellar sheets. Here the second phase is $\alpha\text{-Fe}_2\text{O}_3$ or haematite, whose growth as lamellar sheets is consistent with the hexagonal structure.

Region F, whose boundary with region A is not yet well defined, is of a more complex character. Microstructural examination of samples in region F has revealed twin-plane structures, containing what appears to be precipitates along the twin planes. The boundary between regions F

and A has been drawn somewhat arbitrarily on the basis of observed twin-plane formation and structural and energy considerations², although measurements of limits of good squareness with actual specimens do not at present conform to a straight line in this area. Microstructures illustrative of regions A, D, E, and F are shown in Figure 5.

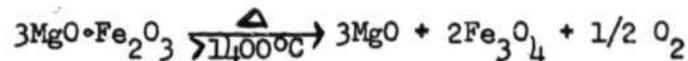
II. CHEMICAL CONSIDERATIONS ON SQUARE-LOOP FERRITES

In order to clarify the true nature of the system which yields the square-loop ferrites, the constituents and their chemical properties must be considered. As a start toward such clarification, consider again the pattern of loop-squareness vs. composition in Figure 3. As no high values of loop-squareness appear possible in the entire left half of the diagram, that portion may be eliminated from consideration of memory core ferrites. Similarly, the lower right one quarter is no longer of interest. The remaining quarter, in which all the good values of loop squareness have been observed, is still triangular in character, indicating a ternary system. This triangle, redrawn on an equilateral basis, would become a system-composition diagram specifically for the square-loop, memory-core ferrites. The constituents of this system as indicated from the apexes of the revised diagram are magnesium ferrite, $MgO \cdot Fe_2O_3$, manganese ferrite, $MnO \cdot Fe_2O_3$, and the manganese oxide constituent, MnO .

Consider first, the behavior of these three constituents individually during heat cycling, similar to that used in processing the ferrite memory cores. Magnesium ferrite is the most stable of the three. This ferrite, found in some quantity in chromite brick, is quite refractory and does not exhibit any tendency to oxidize at high temperatures. Confirmation of its stability has been obtained by thermal analysis. No thermochemical change is observed for $MgO \cdot Fe_2O_3$ between room temperature and $1400^\circ C$. At the upper limit of this temperature range there are indications of decreased stability with respect to oxygen loss³. The equilibrium partial pressure of oxygen has not been determined as a function of temperature but it is known to be significant in a region of $1400^\circ C$,

2. Division 6 - Lincoln Laboratory, M.I.T., Cambridge, Mass., M-2473, "B-H Loop Squareness in the Magnesium Manganese Ferrites", To: D. R. Brown, From: J. B. Goodenough, October 22, 1953.
3. Roberts, H. S. and Merwin, H. E., "The System $MgO \cdot FeO \cdot Fe_2O_3$ in Air at One Atmosphere", American Journal of Science 21, 145 (1931).

and to become larger as the temperature is further increased. Analogy to the loss of oxygen from Fe_2O_3 at high temperatures is suggested^{4,5}. Whether the oxygen loss from $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ occurs directly from the undissociated compound or occurs from the Fe_2O_3 portion after a dissociation into constituent oxides, is not known but the net result will be the same and may be summarized in the equation:

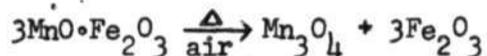


It is therefore seen how "over firing" may affect the electrical and magnetic characteristics of memory cores. The Fe_3O_4 formed will readily join in the ferrite phase solid solution and, in very small quantities, perhaps the MgO also; in larger quantities, MgO would be expected to precipitate as a second phase. Changes in magnetic and electrical properties with high firing of $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ may therefore be attributed to some combination of effects of the addition of Fe_3O_4 as a constituent and the presence of uncombined MgO . Considerable difficulty is experienced in the formation of suitable samples of $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ because the refractory character of the substance requires sintering temperatures which reach into the range of oxygen instability. In memory core compositions whose sintering temperatures are somewhat reduced over those required for pure $\text{MgO}\cdot\text{Fe}_2\text{O}_3$, the $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ constituent may be considerably stable and not easily susceptible to modification during a memory-core firing cycle in air.

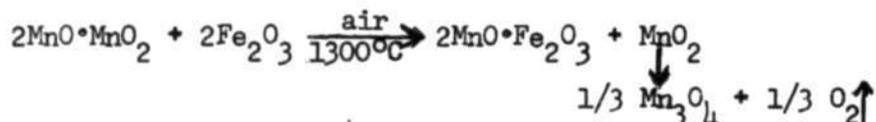
The manganese ferrite constituent on the other hand presents quite a different picture during heat cycling in air. A number of articles^{6,7,8} have described the results obtained when this material is heated

4. Ralston, O. C., U. S. Bur. Mines Paper 296 (1929).
5. Darken, L. S. and Gurry, R. W., "The System Iron-Oxygen II, Equilibrium and Thermodynamics of Liquid Oxide and Other Phases", J. Am. Chem. Soc. 68, 798 (1946).
6. Hilpert, S., Wille, A. and Linder, A.; Z. Physik. Chem. (B) 18, 291 (1932).
7. Harvey, R. L., Hegyi, I. J., and Leverenz, H. W., "Ferromagnetic Spinel for Radio Frequencies", RCA Review 11, 321 (1950).
8. VanArkel, A. E., Verwey, E. J. W. and VanBruggen, M. G., "Ferrites I", Rec. trav. chim. 55, 331 (1936).

in air. All agree that the manganese oxidizes and a mixture of oxides results.



As neither of these oxides is magnetic, the production of a magnetic phase resulting from the heating of $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ or the heating of an $\text{Mn}_3\text{O}_4 + \text{Fe}_2\text{O}_3$ oxide mixture, in air, must be explained by some other mechanism. A number of standard treatises⁹ state that Mn_3O_4 breaks down further to MnO , i.e., Ephraim, p. 382, 2nd ed., states "MnO....is only stable at atmospheric oxygen pressure at a white heat, and can only be obtained from Mn_3O_4 if this temperature is reached." From this, a mechanism of high temperature reaction between MnO and Fe_2O_3 to form $\text{MnO}\cdot\text{Fe}_2\text{O}_3$, might be devised, followed by sintering and grain growth into a dense mass. Another possible mechanism might be based on the fact that Mn_3O_4 is expressed more correctly as $2\text{MnO}\cdot\text{MnO}_2$, differing basically from the structure of Fe_3O_4 or $\text{FeO}\cdot\text{Fe}_2\text{O}_3$. At high temperatures Fe_2O_3 may react with the MnO portion of Mn_3O_4 to form $\text{MnO}\cdot\text{Fe}_2\text{O}_3$, leaving MnO_2 which, being above 960°C , would convert to more Mn_3O_4 as



Again, sintering and grain growth of the $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ to a dense mass is assumed. Which, if either, of these mechanisms is correct, is not known, but it is certain that a magnetic product is formed from high temperature treatment in air of $\text{Mn}_3\text{O}_4 + \text{Fe}_2\text{O}_3$, neither of which is alone magnetic. After the formation of this magnetic phase, which is assumed to be $\text{MnO}\cdot\text{Fe}_2\text{O}_3$, sintering and grain growth would restrict the access of air and tend to off-set reoxidation of the mass during cooling. Solid solution of a small amount of $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ in a bulk of air-stable $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ would greatly assist in desensitizing the manganese to reoxidation. In the study of memory core compositions it has been observed that compositions close to the $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ point are much more sensitive to atmospheric oxidation than those whose composition is mostly $\text{MgO}\cdot\text{Fe}_2\text{O}_3$.

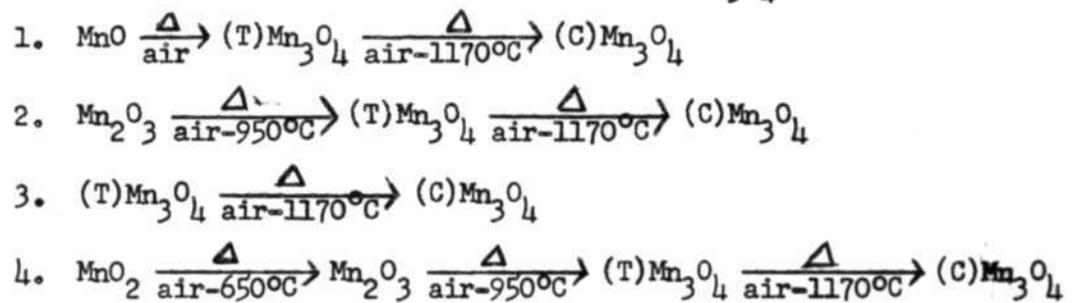
9. See standard treatises on Inorganic Chemistry, i.e., Mellor, Treatise on Inorganic Chemistry; Ephraim, Inorganic Chemistry; etc.

Examination of the microstructure of a large core, basically a manganese-zinc ferrite, as is used in TV sweep voltage generation, shows the interior to be large, well-sintered, homogenous grains but the surface is two phase and poorly sintered. The two phase portion will follow cracks and flaws into the interior of the piece. In memory core applications the extent to which this oxidation process occurs is a function of the relative proportions of manganese ferrite and the other constituents, the compaction, the sintering and the temperature. An oxidized film on the exterior of a TV transformer core may well be insignificant, but the same film may constitute a high percentage of a memory core whose wall thickness is .015". Again, with manganese ferrite we must not ignore the loss of O_2 at very high temperatures from either the ferrite itself or the Fe_2O_3 oxidation product, with the consequent addition of Fe_3O_4 to the spinel phase.

It has been determined experimentally, that cooling from peak temperature in an inert (nitrogen) atmosphere yields a better memory core product than is obtained with air cooling. Closer examination of the effects of the inert atmosphere indicates that protection of a magnetic manganese ferrite against reoxidation is not the only possible effect. The oxygen instability and loss from Fe_2O_3 and ferrites at high temperatures will be affected by an inert atmosphere. In the case of uncombined Fe_2O_3 a nitrogen atmosphere would aid the conversion to Fe_3O_4 with respect to both rate and temperature of conversion. The Fe_3O_4 , being a cubic spinel, would favor the formation of a single phase product and thus enhance the possibilities for loop squareness from that point of view. On the other hand, the effects of Fe_3O_4 as a constituent have not been ascertained in regard to the other electrical and magnetic properties of memory cores. It therefore is not clear, at this time, whether nitrogen cooling is entirely beneficial with respect to assisting in the conversion of Fe_2O_3 to Fe_3O_4 . For the case of a ferrite such as $MgO \cdot Fe_2O_3$ showing oxygen loss at high temperature, the increased conversion of Fe_2O_3 to Fe_3O_4 , by a nitrogen atmosphere, would inevitably increase the uncombined MgO content of the cooled ferrite. This would not be favorable to the production of good memory cores. Optimum processing conditions would therefore seem to require a compromise between high temperatures, favorable to the formation of a magnetic manganese ferrite and the sintering of

$MgO \cdot Fe_2O_3$, and somewhat lower temperatures, favoring oxygen stability in the ferrites. Using a nitrogen atmosphere for cooling accentuates the need for lower sintering temperatures.

The manganese oxide constituent also requires attention in heat cycling, primarily because it has been demonstrated that approximately the same result is obtained no matter which oxide of manganese is employed as a starting material, provided the variation in molar relationships among the oxides is placed on an equivalent basis for combination with Fe_2O_3 . The oxides of manganese are the subject of considerable literature⁹. As they are heated in air they all are changed to cubic Mn_3O_4 above $1170^\circ C$.



In the sintering range for ferrites, in air, uncombined manganese oxide will be present as $(C)Mn_3O_4$ no matter what oxide of manganese was used as a starting material. Upon cooling through $1170^\circ C$, Mn_3O_4 reverts to the tetragonal form and so remains down to room temperature.

It appears, therefore, that starting with a mixture of $MgO \cdot Fe_2O_3$, $MnO \cdot Fe_2O_3$, and MnO , then raising the temperature in air to the sintering range for ferrites, the following are relatively stable forms which can co-exist: $MgO \cdot Fe_2O_3$, $(C)Mn_3O_4$, $\alpha-Fe_2O_3$, and $MnO \cdot Fe_2O_3$. The particular combination which will exist for a given starting mixture will depend principally on the relative proportions of the starting mixture. The forms stable at high temperature are all stable during cooling to room temperature except for the polymorphic change of $(C)Mn_3O_4$ to $(T)Mn_3O_4$ at $1170^\circ C$ and reoxidation of $MnO \cdot Fe_2O_3$ at temperatures a little below white heat. If any sort of protection is provided against reoxidation of the $MnO \cdot Fe_2O_3$, it too will reach room temperature without change. Such protection may be provided by grain growth and sintering to a dense mass, nitrogen cooling atmosphere, solid solution of a small amount of $MnO \cdot Fe_2O_3$ in a bulk of an air-stable ferrite, quenching or some combination of these four methods. Mn_3O_4 in small amounts has been found to remain in cubic

solid solution with a spinel ferrite structure below 1170°C ^{10,11}. In predominant amounts, the tetragonality of the Mn_3O_4 shows its effect in the twin-plane structures of region F, Figure 4, where the twinning may be considered as a stress-relief mechanism in a tetragonal-cubic mixture.

The good values of loop-squareness have all been obtained with compositions of the general formula

Mols	Constituent oxide
x	MgO
y	MnO
z	Fe_2O_3

where $z \geq x$ but $z < x + y$.

This is interpreted to mean that the MgO must be satisfied by Fe_2O_3 and that the formation of $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ occurs preferentially. After the formation of x mols of $\text{MgO}\cdot\text{Fe}_2\text{O}_3$, any Fe_2O_3 remaining may form $(z - x)$ mols of $\text{MnO}\cdot\text{Fe}_2\text{O}_3$. Any uncombined MnO then remaining will be equal to $y - (z - x)$ mols which, on heating, would convert to $\frac{y - (z - x)}{3}$ mols of Mn_3O_4 . Assuming protection against oxidation of the $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ by solid solution with $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ or by other means, the final ferrite memory core should consist of a single phase cubic spinel solid solution of $\text{MgO}\cdot\text{Fe}_2\text{O}_3 + \text{MnO}\cdot\text{Fe}_2\text{O}_3 + \text{Mn}_3\text{O}_4$. In this composition it is assumed also that the Mn_3O_4 content is small compared to the total ferrite phase, so that the Mn_3O_4 will be sure to join the ferrite phase in cubic solid solution. Good loop-squareness has been observed for compositions which would be satisfied by a solid solution of $\text{MgO}\cdot\text{Fe}_2\text{O}_3$ and Mn_3O_4 . It therefore appears that $\text{MnO}\cdot\text{Fe}_2\text{O}_3$ is not basic to loop-squareness but the Mn_3O_4 is a basic constituent.

From the above analysis, a hypothesis is made. The hypothesis is stated as follows:

"Any magnetic ferrite plus Mn_3O_4 , existing together as a single phase cubic spinel, is potentially a square hysteresis-loop material."

This hypothesis implies that the magnetic ferrite phase may be composed

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10. McMurdie, Sullivan and Mauer, Journal of Research of the National Bureau of Standards, 45, 35, (1950).
 11. Romeijn, F. C., Phillips Technical Reports, 8, 304, (1953).

of a single ferrite or more than one ferrite, not all of which need be magnetic provided the overall blend is a magnetic ferrite phase.

III. MAGNETIC MEASUREMENTS OF MEMORY CORES

For most of the memory core compositions detailed in Section I, measurements of the following magnetic properties have also been taken:

1. Coercive force at maximum loop-squareness conditions
2. Coercive force at saturation (30 Oersteds)
3. Magnetic Induction at maximum loop-squareness conditions
4. Magnetic Induction at saturation (30 Oersteds)
5. Flux reversal time at maximum loop-squareness conditions.

The handling of these data had proven troublesome until they were treated as a function of the composition, where the composition was expressed as a ratio of the total possible mols of the ferrite phase to the mols of the excess manganese oxide constituent, Mn_3O_4 . Essentially the same curves would be obtained using MnO instead of Mn_3O_4 as the form of the excess manganese but this form would not exist after air firing of the powder compact.

Typical data are shown in Figures 6 to 9 for materials in which the amount of Fe_2O_3 was always chemically equivalent to the amount of MgO, hence the manganese content should all be present uncombined, as Mn_3O_4 in solid solution with the spinel phase. These samples are obtained by progressing up the centerline of Figure 4 toward the MnO apex from pure $MgO \cdot Fe_2O_3$ at the base. Figure 6 shows the loop-squareness values expressed as the squareness ratio¹². In general terms, values of 0.80 to 0.85 are most suitable for memory core use. Figures 7 through 9 show the coercive force, magnetic induction and flux reversal time values respectively. While these data do not in any way prove the compositional hypothesis, the appearance of the curves lends support to the idea.

IV. SUMMARY

The present state of the art regarding memory-cores and other square-loop ferrites may be summarized as follows:

12. Division 6 - Lincoln Laboratory, M.I.T., Cambridge, Mass., "A Squareness Ratio for Coincident-Current Memory Cores", Engineering Note E-464, To: Group 63 Staff, From: D. R. Brown, July 17, 1952.

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1. The most satisfactory compositions in the $\text{MgO-MnO-Fe}_2\text{O}_3$ system are known.
2. Explanations have been found for the failure of some compositions and the success of others.
3. A considerable understanding of reasons for the critical character of the required processing has been achieved.
4. It is now possible to recognize many of the significant chemical problems which must be solved to put loop-squareness on a sound scientific basis.
5. The present knowledge of square-loop ferrites, even though limited, would permit a logical start in the development of new square-loop materials, as needed, with pre-specified magnetic and electrical characteristics.

Signed



F. E. Vinal

Approved



D. R. Brown

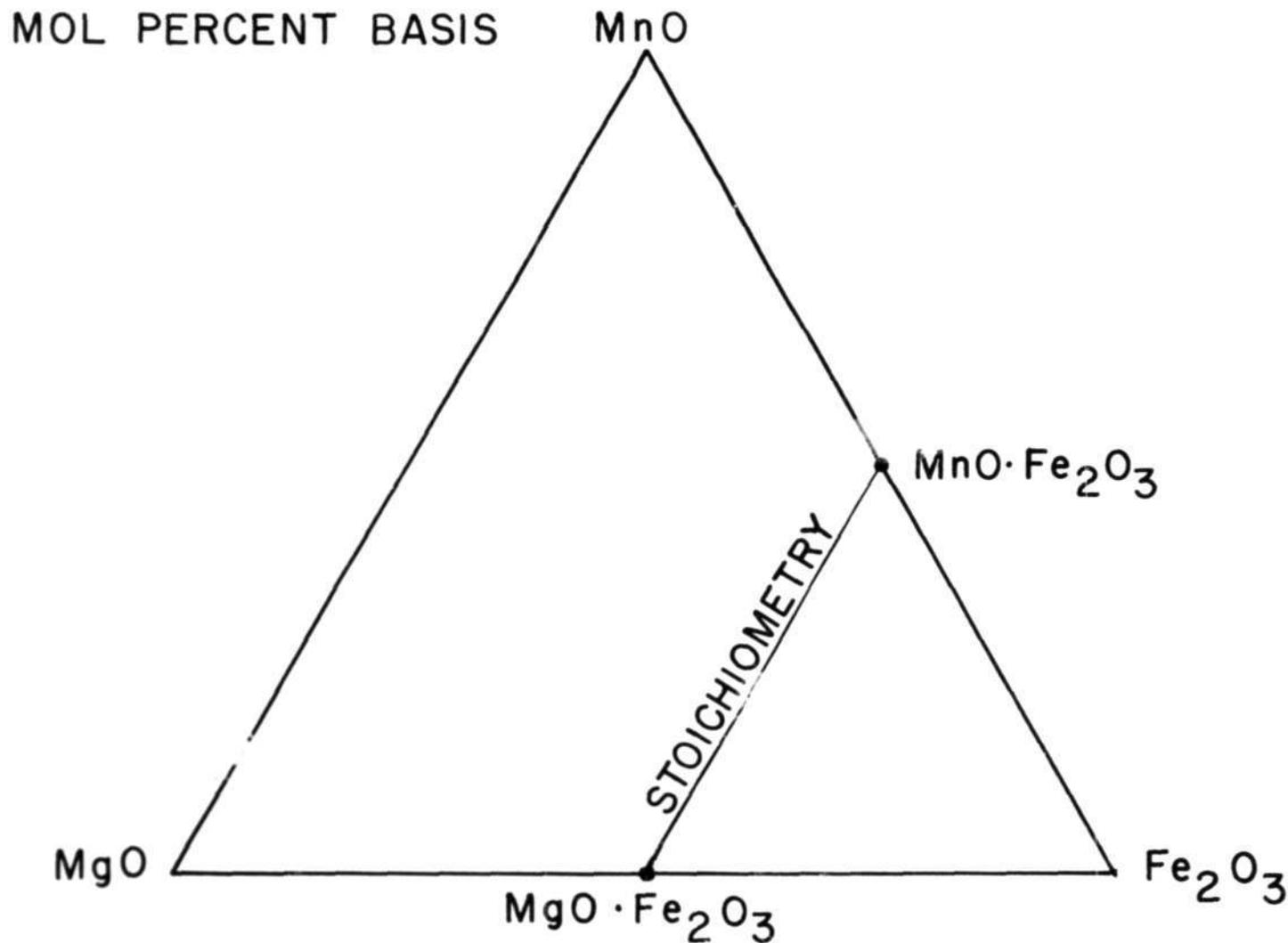
FEV/djd

Drawings Attached: Figure 1 B-57066
Figure 2 B-56130-1
Figure 3 C-56411
Figure 4 B-56368-1
Figure 5 A-57953
Figure 6 B-57070
Figure 7 B-57062
Figure 8 B-57071
Figure 9 B-57061

cc: Group 63 Staff
Group 62 - N. H. Taylor and Section Leaders
Group 37 Staff
Group 35 - H. Priest, W. Z. Leavitt
IBM (Kromer)

B-57066

F-2153
SN-664

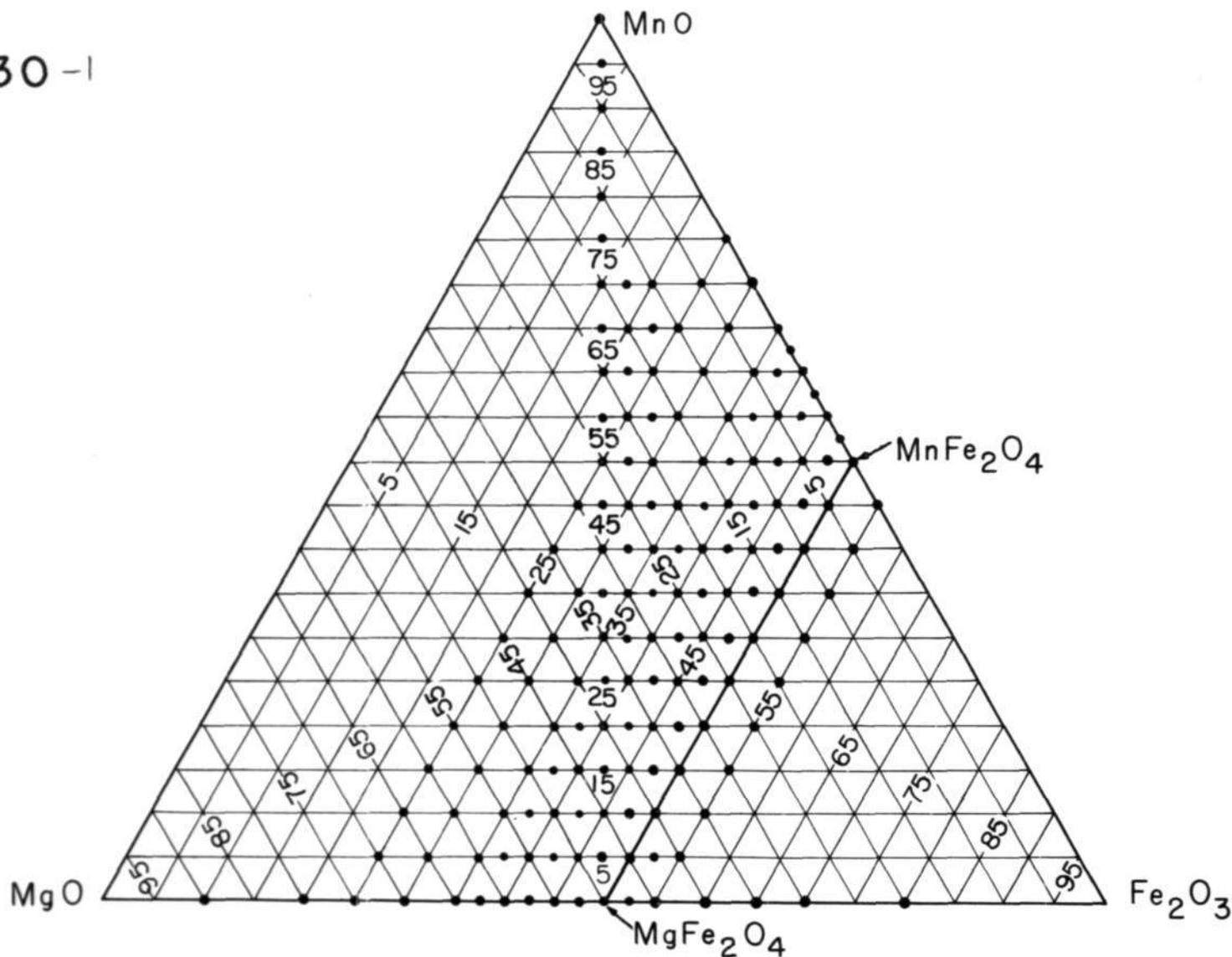


FERRITE MEMORY CORE

TERNARY COMPOSITION DIAGRAM

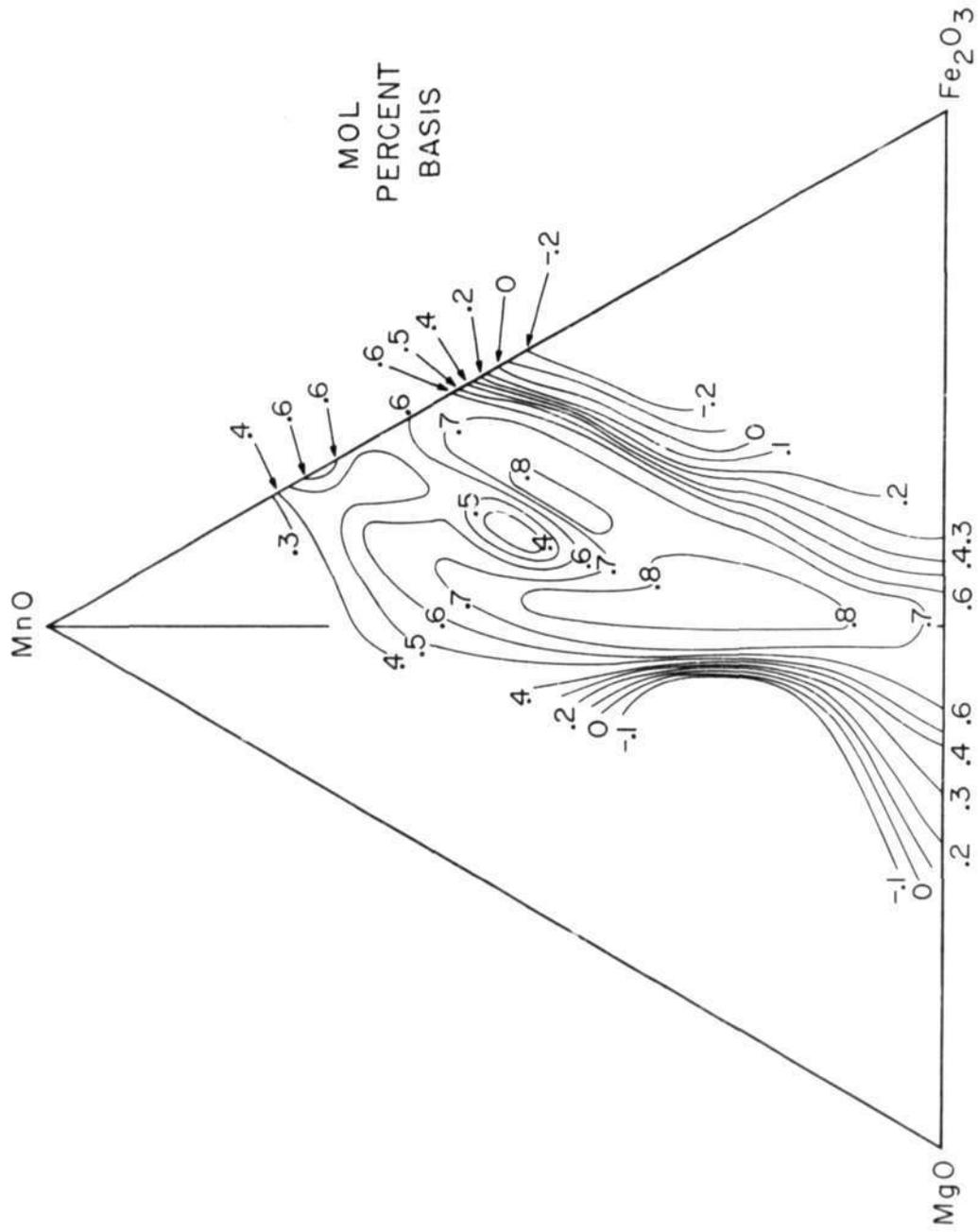
B-56130-1

F-2146
SN-657
MN



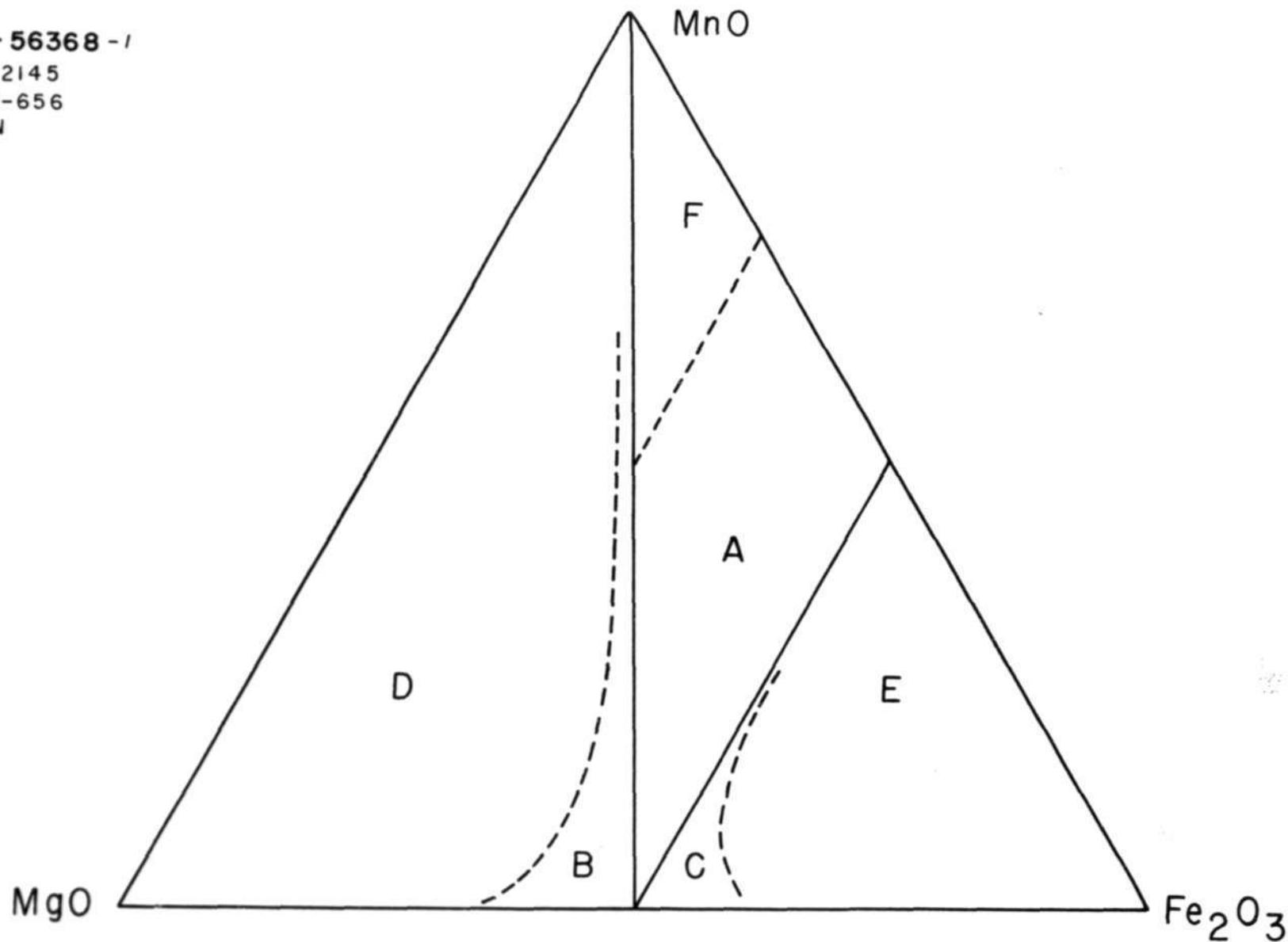
COMPOSITIONS INVESTIGATED IN THE
 $MgO \cdot MnO \cdot Fe_2O_3$ OXIDE SYSTEM MOL PERCENT BASIS

C-56411 - 1
F-2154
SN-665
MN



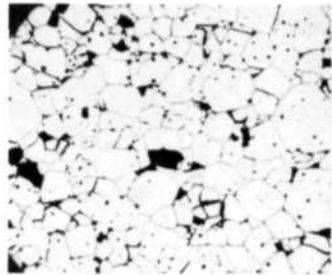
LOOP SQUARENESS VS. COMPOSITION FOR THE MgO · MnO · Fe₂O₃ SYSTEM

B-56368-1
F-2145
SN-656
MN

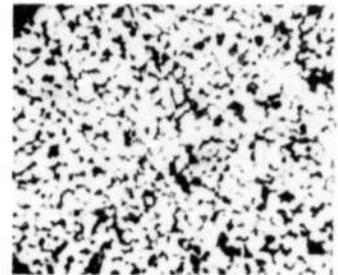


INTERPRETATION OF HYSTERESIS-LOOP-SQUARENESS

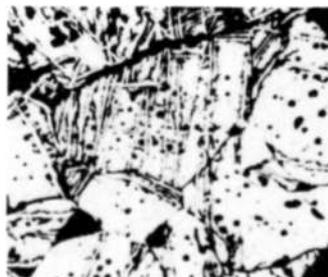
FIG 4



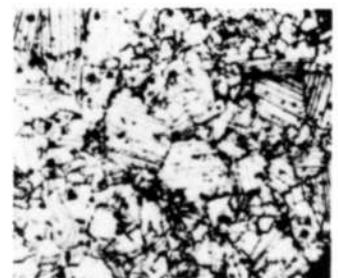
REGION A



REGION D



REGION E



REGION F

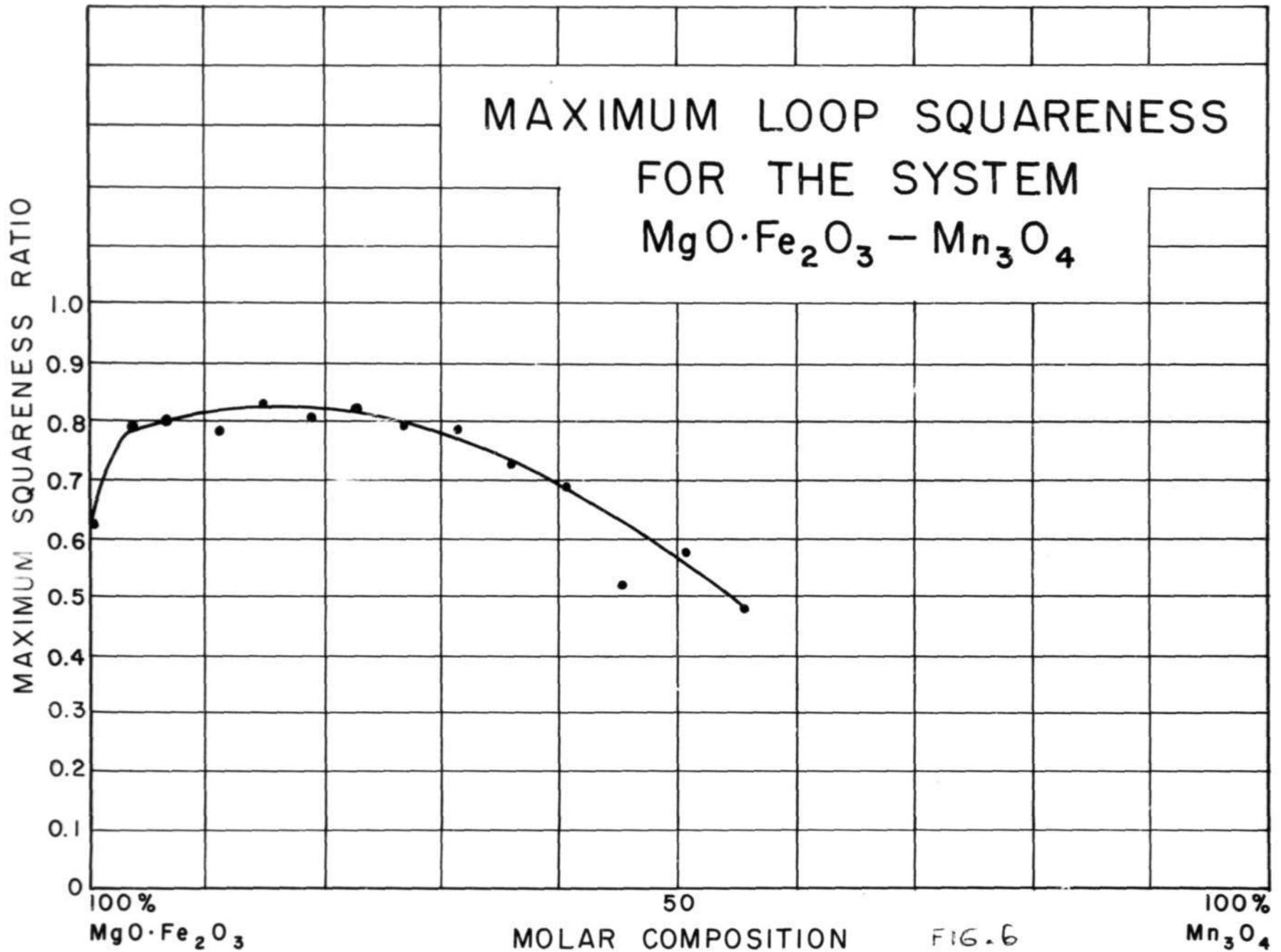
FIG. 5

MICROSTRUCTURES OF FERRITES
IN THE $\text{MgO} \cdot \text{MnO} \cdot \text{Fe}_2\text{O}_3$ SYSTEM

A-57953

B-5770
F-2152
SN-663
MN

APPROVED FOR PUBLIC RELEASE. CASE 06-1104.



B-5 062
F-2151
SN-662
MN

APPROVED FOR PUBLIC RELEASE. CASE 06-1104.

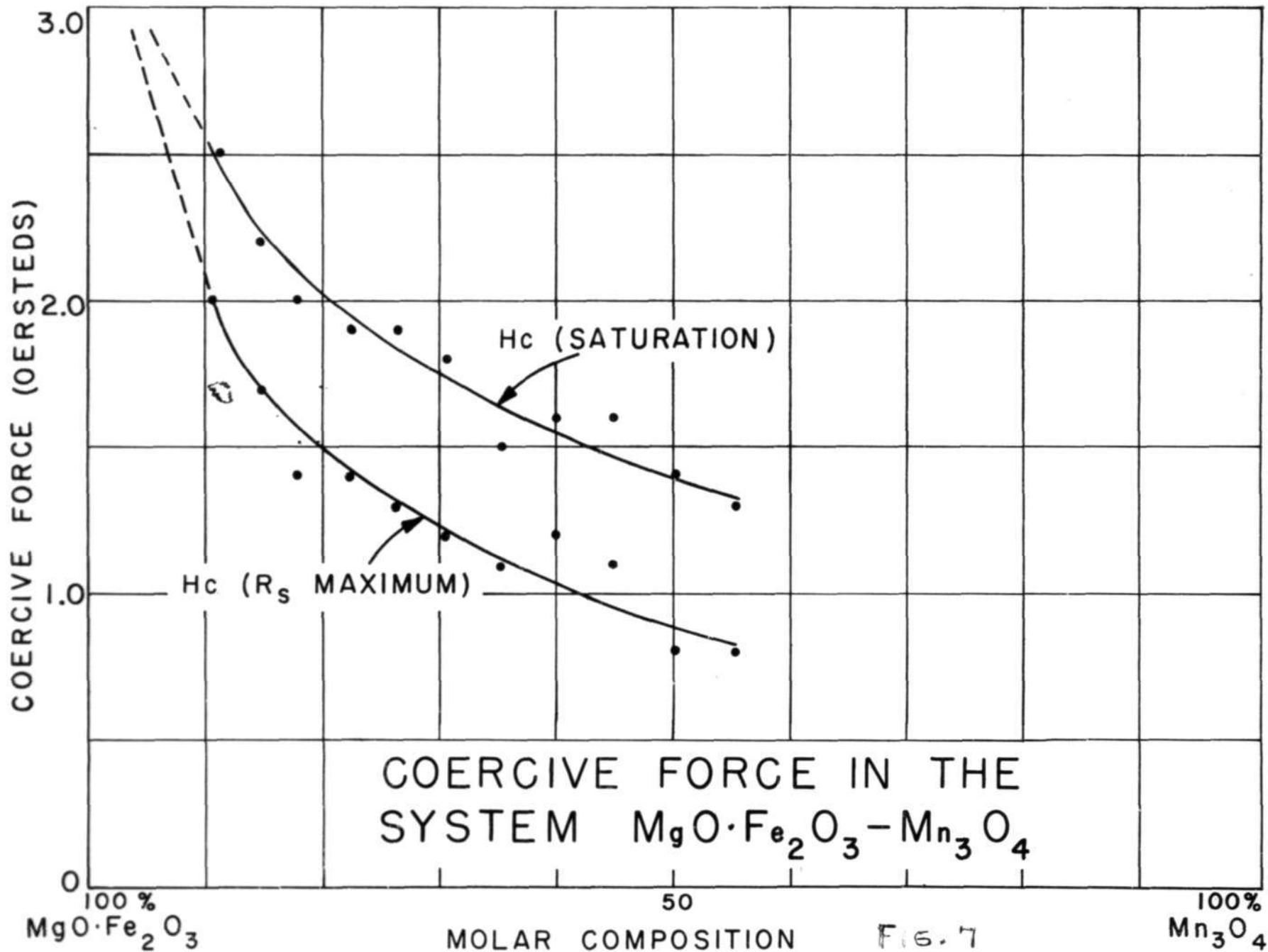


FIG. 7

B-57071
F-2149
SN-660
MN

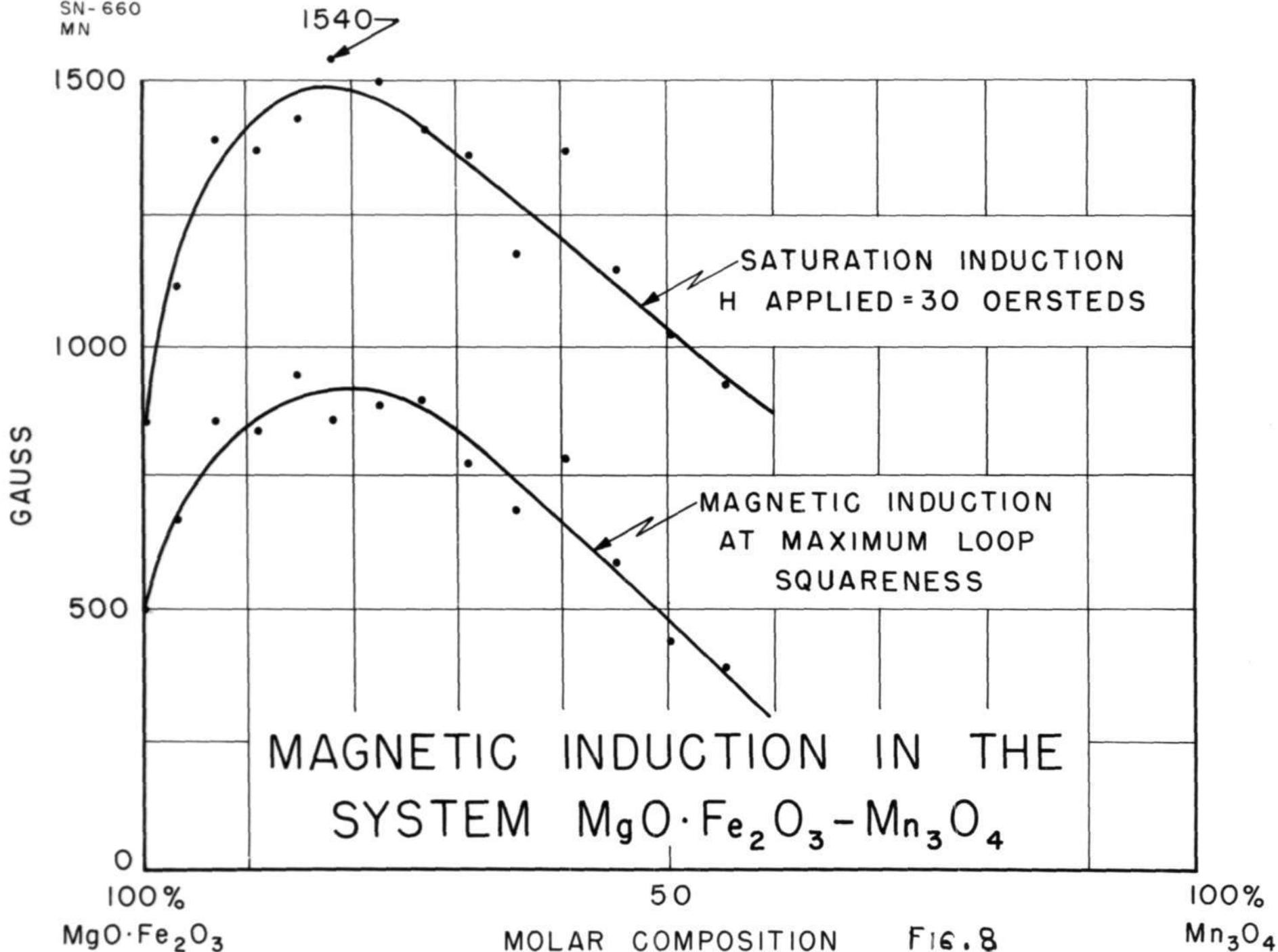
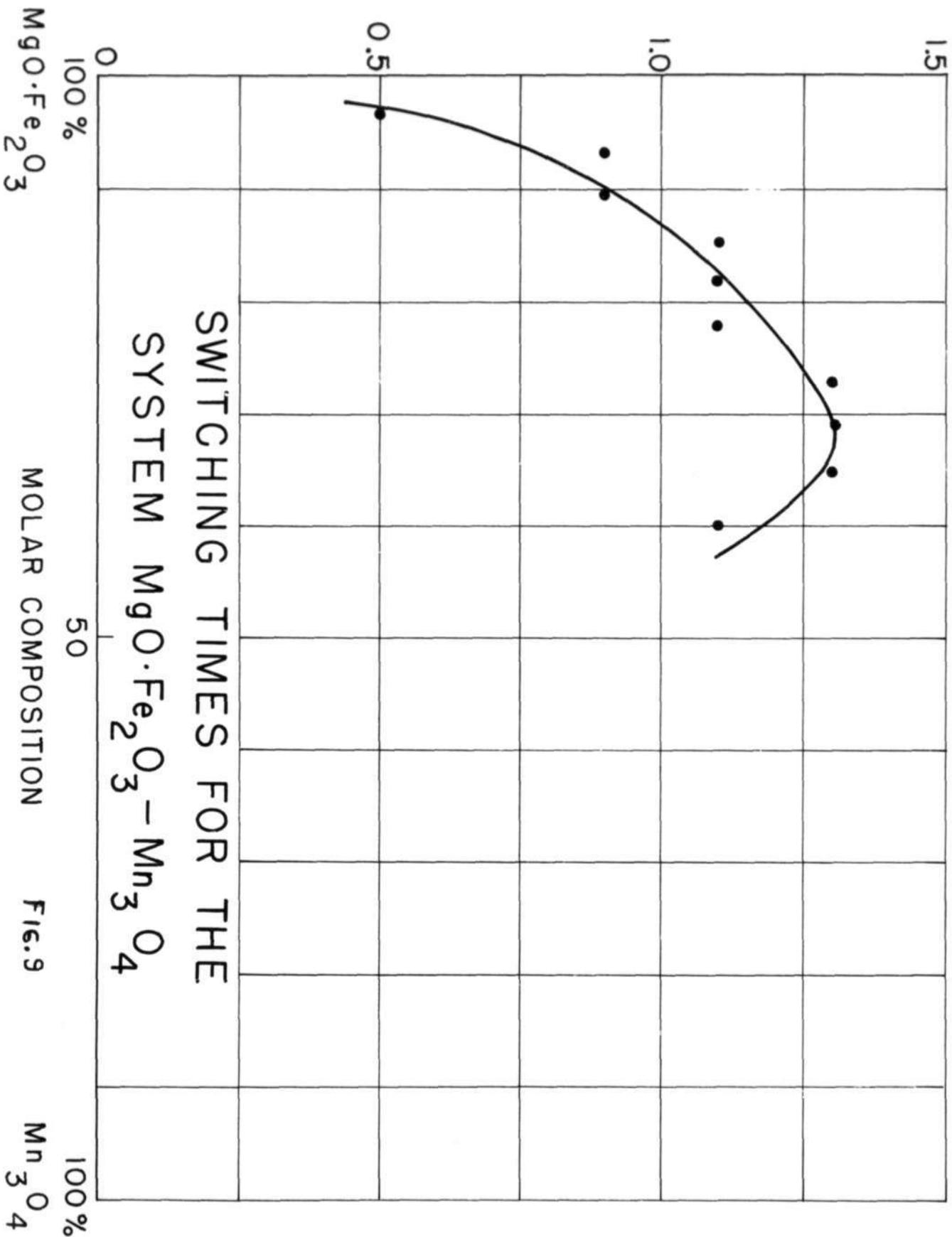


FIG. 8

B-57
F-2147
SN-658
MN



MOLAR COMPOSITION

FIG. 9

B-57
F-2147
SN-658
MN

