for the resistivity of polycrystalline yttrium using the following average rule.[3]

$$\rho_{\text{poly}}=(2\rho_{\perp}+\rho_{\parallel})/3. \quad (1)$$

The result is shown in Fig. 1 as a continuous line. The dashed line is a fit of Grüneisen's relation[4] [Eq. (2)] to the prediction

$$\frac{\rho}{\rho_\theta}=4\left(\frac{T}{\theta}\right)^5\int_0^{\theta/T}\frac{x^5dx}{(e^x-1)(1-e^{-x})}, \quad (2)$$

as calculated by Eq. (1). The values that gave the best fit were $\rho_\theta=37.47$ μ ohm-cm and $\theta=187.5$°K. The high-temperature specific heat Debye θ for yttrium is about 213°K.[5] The agreement between these two temperatures is better than for most metals,[2] although there seems to be no good reason why they should have to be the same.[6]

[3] Compare J. L. Nichols, J. Appl. Phys. **26**, 470 (1955).
[4] E. Grüneisen, Ann. Physik **16**, 530 (1933).
[5] R. E. Miller, Ames Laboratory (private communication, 1959).
[6] M. Blackman, Proc. Phys. Soc. (London) **A64**, 681 (1951).

The polycrystalline curve is represented by the dots on Fig. 1. This curve agrees with the prediction within 5% at room temperature, and follows it fairly well all the way down to 1.3°K.

DISCUSSION

The purity of the single crystals is estimated to be 99%, the major contaminants being 0.5% Zr, and 0.3% O_2. The purity of the polycrystalline sample is estimated as 99.2%, the major contaminants being 0.5% Ti and 0.1% F. The Zr and Ti originated from the crucibles in which the metal was melted.

The data presented here are interpreted as substantiating the applicability of Eqs. (1) and (2) to yttrium metal, but no interpretation of the large anisotropy is proposed at this time.

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Magnetic Properties of Hematite Single Crystals. I. Magnetization Isotherms, Antiferromagnetic Susceptibility, and Weak Ferromagnetism of a Natural Crystal*†

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Two sets of magnetization isotherms of pure natural hematite single crystals from Elba have been obtained in the temperature range from 488°K down to liquid helium temperatures. The first set of curves, along a certain direction in the basal plane, support Néel's magnetic model of a superposition of a weak ferromagnetism on a normal antiferromagnetism. The second set of curves, along the ternary axis, display very unusual form. The analysis of the isotherms shows that the antiferromagnetic susceptibility-temperature curves, $\chi-T$, are in good agreement with those obtained by Néel and Pauthenet but the weak ferromagnetic properties are apparently contradictory to their interpretations. The spontaneous magnetization-temperature curves, σ_0-T, indicate that there is no isotropic ferromagnetism, and that the weak anisotropic ferromagnetism in the basal plane above transition and along the ternary axis below transition seems to have the same nature and origin. The $\chi-T$ and σ_0-T curves show that the wide transition takes place gradually and continuously. A general magnetic model of canted antiferromagnetism with unequal sublattice moments has been proposed which explains all the experimental data satisfactorily. From the present model Haigh's data of remanent magnetization of hematite powder seems to be explained naturally.

I. INTRODUCTION

THE magnetic properties of hematite (αFe_2O_3) have been a controversial problem for several decades. In spite of considerable study some fundamental properties of this material are still questionable. Although in the early days, a great deal of important work[1] was carried out, the most important contributions were made in the last decade when Néel[2] discovered that hematite is primarily an antiferromagnetic material (with Néel temperature equal to 950°K) superimposed

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† Part of this work was published in the *Proceedings of the Conference on Magnetism and Magnetic Materials* [Suppl. J. Appl. Phys. **30**, 306S (1959)].

[1] T. T. Smith, Phys. Rev. **8**, 721 (1916); R. Chevallier and S. Mathieu, Ann. Physik **18**, 258 (1943); and many other works.
[2] L. Néel, Ann. Physik **3**, 137 (1948); **4**, 249 (1949).

on a weak ferromagnetism and Morin[3] found a transition at about 250°K at which the antiferromagnetic axis changes orientation from the basal plane to the ternary axis or vice versa, depending upon whether the temperature is lowered or raised through this transition. These two antiferromagnetic properties, confirmed by neutron diffraction,[4] have been generally accepted as the characteristics of hematite. The most controversial problems are (1) the origin and the nature of the weak ferromagnetism above transition and (2) the existence and the nature of the weaker ferromagnetism below transition.

As to problem (1) there are several different opinions. Many authors, especially in the early days, believe that the weak ferromagnetism is due to ferromagnetic impurities such as magnetite. Recently, Néel[5] has suggested that it might be due to the lattice defect, such as errors in the regular alternation of layers of iron atoms magnetized antiparallel. This defect might be related to dislocations of the lattice, or it could be due to an imperfection in the antiferromagnetism which causes a slight asymmetry between the two sublattices. Li[6] and Jacobs and Bean[7] interpret that the weak ferromagnetism comes from the unbalanced antiferromagnetic domain walls. Dzyaloshinsky[8] and Vonsovsky[9] suggest that the weak ferromagnetism is due to the result of canted antiferromagnetism. The general opinion on problem (2) is as follows: The natural crystal from Elba[10] has weak ferromagnetism not only in the basal plane above transition but also along the ternary axis below transition, although the latter is usually weaker than the former. However, pure synthetic hematite[11] has no ferromagnetism along the [111] direction. According to Dzyaloshinsky and Vonsovsky, there should be no ferromagnetism along the ternary axis. As to the nature of the ferromagnetism, Néel[12] has thought that the parasitic ferromagnetism consists of two parts, one isotropic, which is independent of direction in the crystal, and the other anisotropic, which is tightly coupled with the direction of antiferromagnetism, and can be observed only above 250°K, in a direction perpendicular to the ternary axis. Both the isotropic and anisotropic components of ferromagnetism disappear at about 950°K.

It seems to the author that the confusion probably comes from the inadequacy of the experimental data. Single crystal data, which is most important for the decision of the above mentioned problems is very rare. For example, Smith's data[1] is limited to room temperature only, Bizette and his co-workers' data[13] is limited to room temperature and liquid nitrogen temperature, and Néel and Pauthenet's data,[10] although covering a wide range of temperature, is limited to a narrow range of fields. In order to understand the whole picture of the magnetic properties, it is necessary to obtain enough isotherms for a wide enough range of temperatures and fields along different directions of the single crystals. From these isotherms many important magnetic properties can be derived. The present study has been planned to achieve this purpose. We are also planning the study of the magnetic behavior in the transition region by measuring the remanent magnetization along different directions of spherical samples of different single crystals. This study is expected to reveal some valuable information which would appear in the next paper of this series.

II. PREPARATION OF SAMPLE AND TECHNIQUE OF MEASUREMENT

The natural single crystal of hematite from Elba was kindly supplied by Dr. W. H. Dennen from the Department of Geology, M. I. T. The chemical analysis showed that the iron composition in the sample was almost exactly stoichiometric. The spectroscopic analysis showed that the material was very pure and the result is shown in Table I. Two samples in the form of rectangular prisms of about $\frac{1}{8}$ in.$\times\frac{1}{8}$ in.$\times\frac{1}{4}$ in. were prepared from a large single crystal. The $\frac{1}{4}$-in. dimension of one sample was along the [111] direction of the rhombohedral axis and the other was along a certain direction in the basal plane which was perpendicular to the [111] direction. The crystal axis was determined by x-rays.

TABLE I. Spectroscopic analysis of hematite single crystal from Elba (by Nuclear Metals, Inc., Concord, Massachusetts).[a]

Sample	Al	Ca	Cu	Mg	Ni	Si	Sn	Fe	Zn	Ti
Hematite	*tr*	*tr*	*tr*	*tr*	*tr*	*tr*	*tr*	*M*	*nd*	*nd*

[a] This hematite is fairly pure, much purer than Baker analyzed Fe_2O_3 which is 99.75% pure. The purity is comparable to that of the oxide prepared from Johnson, Matthey spectroscopically pure Fe which is 99.98% Fe.

All the measurements were carried out by the Curie method. The magnet[14] used consists of two parts, a very uniform main field and a very uniform field gradient. The direction of both the field and the gradient is along the vertical direction. This device is very important in this kind of study. Because we were interested in the magnetic properties of a particular direction of the sample, we limited both the field and the gradient in that particular direction. If they were not in the same direction as in the case of the conventional electromagnet, the problem would have been complicated and the data confused. Desiring the sample to be in a uniform field we used a small sample and a small gradient of about a few oersteds per centimeter. The polarity of

[3] F. J. Morin, Phys. Rev. **78**, 819 (1950).
[4] Shull, Strauser, and Wollan, Phys. Rev. **83**, 333 (1951).
[5] L. Néel, *Advances in Physics* edited by N. F. Mott (Taylor and Francis, Ltd., London, 1955), Vol. 4, p. 191.
[6] Y. Y. Li, Phys. Rev. **101**, 1450 (1956).
[7] I. S. Jacobs and C. P. Bean, J. Appl. Phys. **29**, 537 (1958).
[8] I. E. Dzyaloshinsky, J. Phys. Chem. Solids **4**, 241 (1958).
[9] S. V. Vonsovsky and E. A. Turov, J. Appl. Phys. **30**, 9S (1959).
[10] Reference 1; L. Néel and R. Pauthenet, Compt. rend. **234**, 2172 (1952).
[11] C. Guilaud, J. phys. radium **12**, 489 (1951).
[12] L. Néel, Revs. Modern Phys. **25**, 58 (1953).
[13] Bizette, Chevallier and Tsai, Compt. rend. **236**, 2043 (1953).
[14] S. T. Lin and A. R. Kaufmann, Revs. Modern Phys. **25**, 182 (1953).

the small gradient could be changed in order to eliminate the uncertainty of the measurement introduced by the condensation etc. as described[15] elsewhere. The cryostat and the furnace used for obtaining the appropriate temperatures were the same as described in the previous article.[16]

III. EXPERIMENTAL PROCEDURE

a. Magnetization Isotherms

1. The magnetization isotherms along a certain direction in the basal plane perpendicular to the rhombohedral axis of the hematite single crystal, for the temperature range from 488°K down to liquid helium temperature, are shown in Fig. 1. The general features of the curves are as follows: (1) At low field, the curves bend toward the field axis. (2) At high field, the curves are almost linear with the field. (3) The linear portion of all the curves are almost parallel except in the transition region. These are the characteristics of a material with weak ferromagnetism superimposed on an antiferromagnetism observed in the region of small anisotropy energy. Therefore, this set of curves confirms the magnetic sturcture proposed by Néel.[2] It is to be noted that below 250°K the intercepts on the ordinate axis of the straight lines extrapolated from the linear parts of the isotherms are small and that the isotherms below 250°K and above 360°K are all crowded together at their respective limiting positions. These phenomena are very significant in analyzing the isotherms.

2. The isotherms along the ternary axis which is perpendicular to the basal plane for the same temperature range as in the previous case are shown in Fig. 2. These curves display a very unusual form. At temperatures well below transition, the isotherms are almost independent of magnetic field up to 15 000 oersteds except at very low field at which the curves are concave downward. Above 250°K the curves in general bend toward the field axis for low fields and then go linearly with the field. After the field reaches a certain value (we may call it critical field) it bends away from the field axis, and for still higher fields, it bends toward the field axis again. The critical field increases with decreasing temperature. The curves are also crowded together at their respective limiting positions corresponding to the temperature below 250°K and above 360°K. The interpretation of this peculiar form of isotherms will be described in the last section.

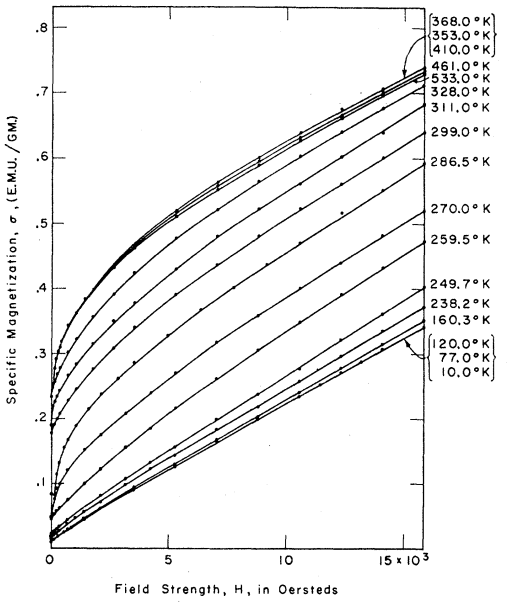

FIG. 1. Magnetization isotherms of hematite single crystal from Elba along a certain direction in the basal plane perpendicular to the ternary axis.

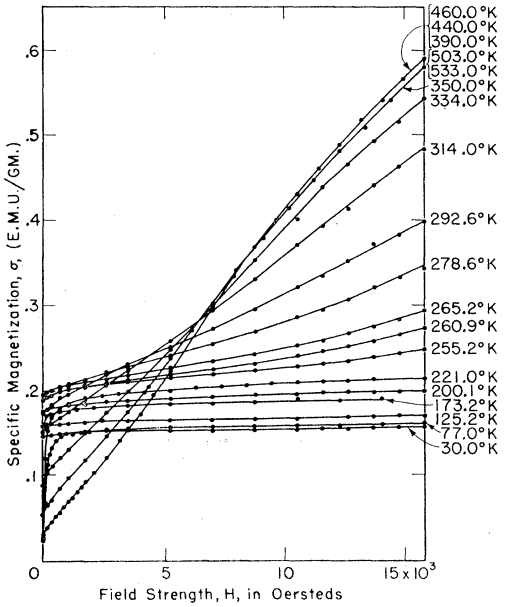

FIG. 2. Magnetization isotherms of hematite single crystal from Elba along the ternary axis.

It should be pointed out that due to the effect of the weak ferromagnetism the sample had a little remanence which was very difficult to demagnetize in the magnet system. For the purpose of high relative accuracy at high fields, we did not want to take the sample out of the magnet system for demagnetization at each temperature. We therefore ignored the little remanence. This would not affect the analysis of the curve at high field. The only drawback was the inaccuracy of the isotherms at very low field. As we were interested in high field data, the demagnetizing field was not corrected for all the magnetization curves.

b. Analysis of the Isotherms

From the magnetization isotherms and the information derived from them, it is believed that the weak

[15] S. T. Lin and A. R. Kaufmann, Phys. Rev. **102**, 640 (1956).
[16] S. T. Lin and A. R. Kaufmann, Phys. Rev. **108**, 1171 (1957).

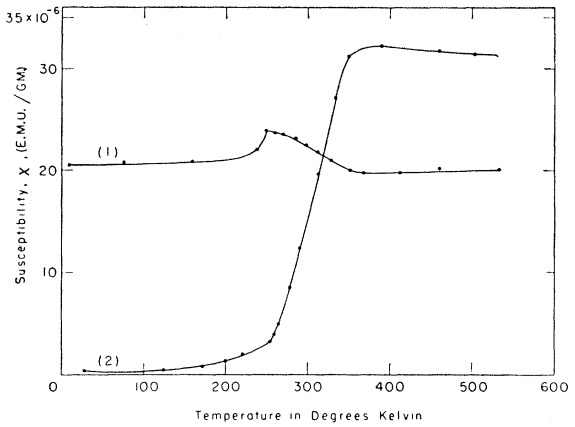

FIG. 3. Variation with temperature of the antiferromagnetic susceptibility of hematite single crystal from Elba: (1) along a certain direction in the basal plane perpendicular to the ternary axis, (2) along the ternary axis.

ferromagnetism (at least for the natural single crystal from Elba) is observable along the ternary axis below and in the transition region. The analysis of the experimental data can be divided naturally into three different temperature ranges: (1) above the transition, where the weak ferromagnetism lies in the basal plane; (2) below the transition, where it orients along the ternary axis; and (3) in the transition region, (some samples cover a wide range of temperature from 360°K to 250°K) where the ferromagnetic moment lies between the ternary axis and the basal plane.

From Anderson's resonance experiment[17] and Smith's magnetic measurement,[1] the anisotropy field was found to be small (about 60 oersteds) in the basal plane above transition. If a magnetic field higher than the anisotropy field is applied along any direction in this plane, the ferromagnetic moment would set itself along the field. When an external field is applied either in the basal plane or along the ternary axis, the total magnetic moment of the sample along the field direction may be expressed by the simple formula:

$$\begin{aligned}\sigma_T&=\sigma_F+\sigma_A, \\ &=\sigma_0+\chi H,\end{aligned} \tag{1}$$

where σ_T is the total moment, σ_F is the moment of the weak ferromagnetism, and σ_A is the moment of the antiferromagnetism. If the applied field is high enough to saturate the ferromagnetic part but not enough to disturb the equilibrium position of the magnetization vector at the corresponding temperature, σ_F becomes a constant, σ_0, and σ_A may be expressed as χH, where χ is the antiferromagnetic susceptibility, which is generally independent of the field. Hence, at constant temperature the total magnetic moment is a linear function of the applied field if it is high enough. It should be noted that the linear portion of the isotherms along the ternary axis is not always at the high field side. The interpretation of σ_0 in Eq. (1) is a little different when applied to the different temperature regions. In the temperature regions (1) and (2) described above σ_0 may represent the spontaneous magnetization of the ferromagnetism. In case (3), as the magnetization vector lies between the ternary axis and the basal plane, it would not coincide with the external field. Then σ_0 is the component of the spontaneous magnetization along the external field at the corresponding temperature.

The values of σ_0 and χ may be separated by the usual technique of extrapolating the linear portion of the isotherm to zero field; the intercept on the moment axis representing σ_0 and the slope of the linear portion of the isotherm representing χ.

Figures 3 and 4 show the results derived from Figs. 1 2 and by using the above analysis. Curve 1, Fig. 3 is almost independent of temperature. The temperature dependence of χ helps us to infer the spin transition process discussed in the last section. The two $\chi-T$ curves, Fig. 3, are about the same as have been obtained by Néel and Pauthenet except for low temperatures of Curve 2. Below 250°K, the susceptibility of Curve 2 is practically equal to zero. The spontaneous magnetization against temperature curves, σ_0-T, Fig. 4, are very different from the corresponding ones of Néel and Pauthenet. The values of σ_0 in Curve 1 above transition and Curve 2 below the transition are almost equal to zero. These phenomena indicate that there is no evidence of isotropic ferromagnetism. This will be discussed in more detail later on.

IV. DISCUSSION

From the four curves of Figs. 3 and 4, the following conclusions can be drawn:

1. Since σ_0 is practically equal to zero above 360°K in Curve 2, Fig. 4, and below 250°K in Curve 1, Fig. 4, it indicates that there is no evidence of isotropic ferromagnetism.

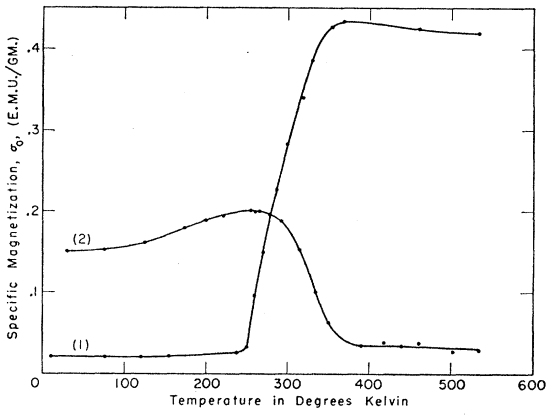

FIG. 4. Variation with temperature of the weak spontaneous magnetization of hematite single crystal from Elba: (1) along a certain direction in the basal plane perpendicular to the ternary axis; (2) along the ternary axis.

[17] P. W. Anderson *et al.*, Phys. Rev. **93**, 717 (1954).

2. From Curve 2, Fig. 4, the anisotropic weak ferromagnetism is observable below transition (250°K) and the magnitude is about 0.2 emu/g.

3. From the four curves of $\chi-T$, and σ_0-T, it is evident that in a wide region of over a hundred degrees, the transition takes place gradually and continuously insetad of rapidly and discontinuously.

4. The two curves of Fig. 4 are complementary to each other. Above 360°K $\sigma_0(111)$ (σ_0 in (111) plane) is a maximum value while $\sigma_0[111]$ (σ_0 along [111] direction) is almost zero. When the temperature decreases from 360°K to 250°K, $\sigma_0(111)$ decreased from maximum to zero while $\sigma_0[111]$ increases from zero to maximum. Below 250°K, $\sigma_0(111)$ remains zero while $\sigma_0[111]$ remains almost maximum.

5. From the statement (4) it is indicated that the ferromagnetism along the ternary axis at low temperature and that in the basal plane at high temperature seem to have the same nature and origin.

Néel's[2] interpretation that the weak ferromagnetism comes from ferromagnetic impurities such as Fe_3O_4, and Li's and Jacobs and Bean's interpretation that it comes from the unbalanced antiferromagnetic domain walls, do not seem to agree with the above mentioned five phenomena. Dzyaloshinsky[8] and Vonsovsky[9] have proposed that the magnetic moments of the sublattices are not exactly antiparallel but turn toward each other in the basal plane above transition. Then there is a net magnetic moment which is perpendicular to the antiferromagnetic axis. The proposal explains the weak ferromagnetism in the basal plane above transition very well. Below transition, the spin orientation is along the [111] direction (as confirmed by the neutron diffraction[4]) and the sublattice moments are almost completely antiparallel (as indicated by our experiment that the ferromagnetism in the basal plane is practically zero), therefore, there should be no ferromagnetism along the [111] direction below transition. These may be the cases with the synthetic αFe_2O_3. On the contrary, however, some other natural single crystals, such as those from Elba, show weak ferromagnetism along the ternary axis below transition as in our case and in the case of others. One possibility of explaining this phenomenon is to extend the idea of Dzyaloshinsky and Vonsovsky by assuming a general model of a canted antiferromagnetism with unequal sublattice moments; i. e., the magnetic moment of the sublattices may tilt slightly toward each other above and in the transition region and at the same time their magnitudes may differ slightly. In order to explain the high value of $\sigma_0(111)$ in the basal plane above transition and zero value below transition it is assumed that the cant is maximum above transition. When the temperature decreases through transition the sublattice moments turn from the basal plane toward the ternary axis and at the same time the canted sublattice moments are gradually straightened out to become antiparallel below transition.

With this model, all the experimental data can be explained qualitatively. The complementary curves of σ_0-T, Fig. 4, may be explained as follows: Above 360°K the antiferromagnetic axis lies in the basal plane. When an external field (higher than 60 oe from Anderson[17]) is applied along any direction in this plane, the antiferromagnetic sublattice moments orient themselves almost perpendicular to the field (indicated by curve 1, Fig. 3, that χ is independent of temperature) and the net weak ferromagnetic moment from the canted antiferromagnetism is almost along the external field (indicated by Smith's experiment[1] which will be described near the end of this section). When the temperature decreases from 360°K to 250°K the antiferromagnetic axis (the direction of the difference of the sublattice moments) turns itself from the basal plane toward the ternary axis in such a way that it always keeps itself perpendicular to the field direction and at the same time the canted angle is continuously decreased. The component of the ferromagnetism in the basal plane then continuously decreases from the maximum value to zero while the component along the ternary axis continuously increases from zero to maximum. At 250°K (the conventional transition temperature) the orientation is almost completed and the antiferromagnetic axis and the unbalanced sublattice moments are all along the ternary axis, therefore, $\sigma_0(111)$ is zero and $\sigma_0[111]$ is maximum.

The difference in magnitude of σ_0 in the basal plane at high temperature and along the ternary axis at low temperature can also be explained. In the basal plane the ferromagnetism is due to both the effect of the cant of the sublattice moments and the difference in magnitude of the moments. Along the ternary axis the sublattice moments orient antiparallel and the ferromagnetism is due to the difference in magnitude only.

The above proposal of the magnetic behavior of hematite may help to understand the peculiar forms of Fig. 2. Well below the transition, the magnetization vector is along the ternary axis so that the corresponding isotherms of Fig. 2 are independent of the magnetic field. At a certain temperature above 250°K the magnetization vector is kept at a certain direction between the ternary axis and the basal plane by the anisotropy field. When the external field along the ternary axis is high enough (but less than the critical field) the magnetization is linear with the field; i. e., saturated at that temperature. If the field reaches a critical value which is comparable with the anisotropy field, the magnetization vector begins to turn toward the field direction, and the isotherms begin to bend away from the field axis. The turning of the magnetization vector toward the ternary axis is accompanied by the following three events which govern the form of the magnetization isotherms: (1) the magnitude of the ferromagnetic moment decreases, (The canted sublattice moments are gradually straightened out to become antiparallel below transition. This event will be more clear in the next paper), (2) the component of the ferromagnetic moment along the field direction

(the ternary axis) increases, and (3) the antiferromagnetic susceptibility decreases. Therefore the form of isotherms depends upon these three competitive factors.

We would like to discuss a little more about the weak ferromagnetism below transition. In general the intensity of weak ferromagnetism below transition is lower than that above transition and the intensity may vary with samples from different sources. We have interpreted this ferromagnetism along the ternary axis as the result of the canted antiferromagnetism of sublattice moments of unequal magnitude. If this unequal magnitude is interpreted as due to the lattice defect or imperfection of the crystal the variation of intensity with different samples is permissable. However, it should be noted that if there is only inequality of the sublattice moments without canting mechanism, the phenomena would be different. For this case, the ferromagnetic moment must be along the direction of the antiferromagnetic axis instead of perpendicular to it. According to Smith's[1] experiment, the hematite sphere, hung in a horizontal magnetic field of 1500 oersteds with the [111] axis vertical, could stay anywhere when the sphere is rotated in the field; i.e., the ferromagnetism is always coincident with the external field when applied along any direction in the basal plane above transition. Then the magnetic moment, observed in this plane, must be the sum of the total ferromagnetic moment and the moment due to the parallel antiferromagnetic susceptibility instead of the perpendicular one. The susceptibility would decrease with decreasing temperature. On the contrary, from Curve 1, Fig. 3, it is almost constant throughout the temperature range except in the immediate neighborhood of transition. This means that the antiferromagnetic axis is perpendicular to the external field. We have just noticed that the ferromagnetism is along the external field, and therefore these two phenomena combine to indicate that the origin of the weak ferromagnetism is due to the canted antiferromagnetism.

Haigh[18] made an extensive experiment on remanent magnetization of hematite powder. He found that the anisotropic component of the ferromagnetism recovered about 70% after his sample was cooled to −75°C and warmed to room temperature again. According to Néel's interpretation, below the transition the anisotropic component of the remanence completely disappeared. As Haigh pointed out, since there was no directive field when the sample was warmed to room temperature, the anisotropic moment would have recovered randomly in the basal plane; i.e., there would have been no recovery. From our argument, the anisotropic remanence below transition still existed, orienting along the ternary axis. According to our experiment, at each temperature including the transition region, the remanent magnetization vector of the anisotropic ferromagnetism would have a more or less definite magnitude and orientation between the [111] direction and the basal plane. (This point will be more clear from the remanence measurements which will appear in the next paper). Since Haigh's sample was not in a demagnetized state when it was cooled, it should comply with its previous remanence-temperature relationship without the help of any field when it was heated again. The recovery of the anisotropic component at room temperature was a natural consequence. Hence, Haigh's data may be considered as a confirmation of the present proposal.

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[18] G. Haigh, Phil. Mag. **48**, 905 (1957); and **48**, 877 (1957).