Effect of doping with Co^{2+} ions on the resonant and static magnetic properties of hematite

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Spontaneous crystallization from a molten solution is used to grow α-$Fe_{2-x}Co_{x/2}Si_{x/2}O_3$ ($x = 0$–0.2) single crystals. The effect of the Co^{2+} ion on the AFMR parameters and on the magnetization is investigated. Compared with other impurities, the Co^{2+} ion exerts the strongest influence on the Morin transition temperature T_M. The concentration $x = 0.007$ is critical and at a larger impurity content the hematite remains weakly ferromagnetic down to $T = 0$ K. The contribution of the Co^{2+} ion to the single-ion anisotropy constant is estimated at 3.1×10^{-15} erg/ion at $T = 0$ K. The temperature dependences of the AFMR parameters of samples with $x = 0.008$–0.015 are investigated in the interval 4.2–300 K. At temperatures below 200 K the contribution of the Co^{2+} ion to the hexagonal anisotropy in the basal plane of the crystal becomes noticeable. At lower temperatures (below 40 K) anomalous behaviors of the hexagonal anisotropy, of the line width, and of the energy gap in the AFMR spectrum are observed. These anomalies are possibly due to freezing of the electron jumps.

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INTRODUCTION

Hematite α-Fe_2O_3 is a rhombohedral antiferromagnet (space group $D_{3d}{}^6$) with Néel temperature $T_N = 948$ K. At the Morin temperature $T_M = 263$ K a transition takes place in the hematite from the weakly ferromagnetic easy-plane state ($T > T_M$) to the easy-axis antiferromagnetic state ($T < T_M$). It is known from investigations of the magnetic properties of hematite by various methods[1–7] that when even relatively small (~ 1 mol.%) metal impurities (Al, Ga, In, Cr, Ti, Sn, Rh) is introduced into the crystal the Morin temperature can change substantially, and all the listed impurities, except Rh, lower T_M. The strongest influence on T_M is that of Sn^{4+} ion impurities.[2,5,6] This influence is due to the appearance in the crystal of charge-compensation Fe^{2+} ions, which have a strong spin-lattice interaction and influence strongly the single-ion anisotropy. The presence of Fe^{2+} ions explains also other effects observed in Ref. 5: the anomalous growth of the isotropic gap in the antiferromagnetic-resonance (AFMR) spectrum and of the hexagonal anisotropy in the basal plane when the temperature is lowered, as well as the appearance of frozen uniaxial anisotropy in the basal plane.

The present paper is devoted to the study of resonant and magnetic properties of hematite doped with Co^{2+} ions having a stronger spin-order interaction that the Fe^{2+} ions. The spin-orbit interaction constants for the free ions Co^{2+} and Fe^{2+} are respectively -180 and -100 cm^{-1}, respectively.

SAMPLES AND EXPERIMENTAL TECHNIQUE

Single crystals of α-$Fe_{2-x}Co_{x/2}Si_{x/2}O_3$ with $x = 0$–0.02 were grown by spontaneous crystallization from a molten solution by a technology similar to that of Ref. 9. To preserve the electroneutrality of the crystals, silicon oxide SiO_2 was added in the initial batch in an amount such that the numbers of Co^{2+} ions and of the charge-compensating Si^{4+} ions were equal. The chemical purity grades of the reagents used were: Fe_2O_3, SiO_2, Bi_2O_3—"specially pure," CoO—"pure for analysis," and Na_2CO_3—"chemically pure." Programmed control of the furnace temperature was provided by an RTP-3 regulator accurate to ± 0.2 °C. The obtained isometric hematite single crystal had pinacoid and rhombohedron faces with the basal planes of the crystals parallel to the pinacoid faces. The linear dimensions of the individual isometric crystals reached ~ 1.5 cm, and the mass ~ 5 g. Plates grown on the surface of the melt usually have a smooth mirror surface that coincides with the basal plane of the crystal. The linear dimensions of the plates reach 3 cm at a thickness 1–2 mm.

The content of the cobalt impurity on the surfaces of the crystals was determined by x-ray-fluorescence analysis using the Spark-1 apparatus, with a 5% error. It turned out that the concentration of the cobalt in the crystal was lower than in the initial batch, and this difference increased with increasing impurity content. It was also observed that the final growth layers contain less Co than the initial ones. The total error of the average concentration x does not exceed 30% and is determined mainly by the inhomogeneity of the Co ion distribution over the sample.

The lattice parameters are independent, within the limits of the measurement error, of the amount of cobalt impurity and are equal to $a = 5.036 \pm 0.001$ Å, $c = 13.747 \pm 0.008$ Å. The high quality of the samples is characterized by the values, measured at room temperature, of the AFMR line width and of the resistivity: $\Delta H = 80$ Oe (in the 12 mm range) and $\rho = 2.4 \cdot 10^{10} \Omega \cdot$cm at $x = 0$.

The magnetostatic characteristics of the hematite were measured with a pendulum magnetometer in the range 4.2–300 K in fields up to 10 kOe. The resonant parameters were measured with an AFMR spectrometer with double modu-

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lation of the field. The frequency range 24–45 GHz was covered by interchangeable Gunn oscillators. The frequency-measurement accuracy 1×10^{-5} was ensured by a Ch5-13 frequency converter. Magnetic fields up to 10 kOe were measured accurate 0.1% with an Sh1-1 magnetic-induction meter. The sample temperature was stabilized with a PIT-3 regulator in the interval 4.2–300 K. The temperature maintained constant within ± 0.2 K.

EXPERIMENTAL RESULTS AND THEIR DISCUSSION

1. Effect of the Co^{2+} impurity on the static magnetic properties and on the Morin transition

In the weakly ferromagnetic state, the dependence of the hematite magnetization σ on the magnetic field $\mathbf{H}$ applied to the basal plane is of the usual form[10]

$$\sigma=\sigma_0+\chi_\perp \mathbf{H}, \tag{1}$$

where σ_0 is the spontaneous moment due to the Dzyaloshinskiĭ interaction and $\chi_\perp$ is the perpendicular susceptibility. It was observed that the values of σ_0 and $\chi_\perp$ obtained from the field dependence of the magnetization, viz., $\sigma_0 = (0.38 \pm 0.02)$ G·cm/g and $\chi_\perp = (1.88 \pm 0.09)\cdot10^{-5}$ cm/g do not depend on the Co^{2+} impurity content and agree within the limits of measurement error with the data for pure hematite.[1,6] The experimental data were used to estimate the value of the exchange field H_E and of the Dzyaloshinskiĭ field H_D at room temperature, namely $H_E = 9.3\cdot10^6$ Oe and $H_D = 2.0\cdot10^4$ Oe.

The Morin-transition temperature T_M for samples with different Co^{2+} contents was determined from the temperature dependences of the magnetization as well as from the AFMR intensity I and line width ΔH. The temperature dependences of the magnetization are shown for three samples in Fig. 1, and typical temperature dependence of the AFMR intensity and line width for one of the sample are shown in Fig. 2. The resonant and static data indicate that the Morin transition is stretched out in a certain interval ΔT_M, and this interval is substantially larger for substitutional samples than for pure hematite, where $\Delta T_M \approx 1$ K. The presence of this interval is due mainly to the inhomogeneous distribution of the Co^{2+} impurity in the sample. Annealing at 1200 °C for 24 hours decreases ΔT_M (see Fig. 2). It appears that this annealing leads to a more uniform impurity distribution in the sample.

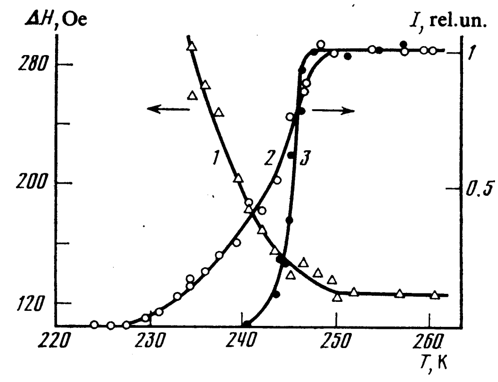

FIG. 2. Temperature dependences of the AFMR line width (1) and intensity I prior to annealing (2) and after annealing (3). $x = 0.0026$; $\nu = 24500$ MHz.

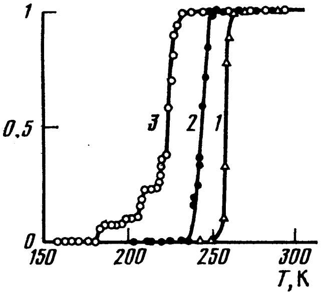

FIG. 1. Temperature dependences of the magnetization of samples of α-Fe_2O_3:Co:1 — $x = 0$; 2 — $x = 0.0026$; 3 — $x = 0.0040$. They were measured in a field $H = 4.5$ kOe.

Figure 3 shows the concentration dependence of T_M, obtained from resonant and static measurements. The square shows schematically the concentration $x = 0.0077$ for which no Morin transition is observed all the way to $T = 4.2$ K. It is known (see, e.g., Ref. 1) that the anisotropy in hematite is determined by competition of two contributions, dipolar and single-ion. These contributions are close in absolute value but of opposite sign (for pure hematite at 0 K we have $H_{\text{dip}} = 10.040$ and $H_{\text{s.i.}} = -10.26$ kOe, Ref. 1). Owing to some difference between the temperature dependences of H_{dip} and $H_{\text{s.i.}}$, these contributions become equal at a certain temperature approximately close to T_M. Owing to the change of the sign of the total anisotropy H_a, a spin reorientation, called the Morin transition,[1)] takes place. Introduction of impurities change the ratio of the dipolar and single-ion contributions to the anisotropy and leads in the upshot to a change of T_M. The strongest effect on T_M can be expected from doping with ions having a strong spin-lattice bond and which alter strongly the single-ion contribution to the anisotropy. Comparison with the data of Refs. 1–7 shows that the effect of the Co^{2+} impurity is stronger by tens of times than that of diamagnetic impurities, and by 3–4 times than that of divalent iron Fe^{2+}. It can be assumed that the entire change of the single-ion anisotropy field by the doping is due only to the influence of the Co^{2+} ion. The contribution

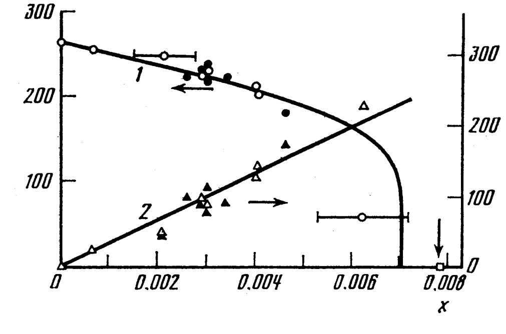

FIG. 3. Concentration dependence of the Morin temperature T_M (1) and of the contribution of Co^{2+} to the single-ion anisotropy at $T = 0$ K(2):○, △—resonance measurement data; ●, ▲—static-measurement data.

of Co^{2+} to the single-ion anisotropy is therefore

$$\delta H_{\text{s.i.}}^{\text{Co}}(x)=H_{\text{s.i.}}(x)-H_{\text{s.i.}}(0), \tag{2}$$

where $H_{\text{s.i.}}(x)$ and $H_{\text{s.i.}}(0)$ are the single-ion anisotropy fields of doped and pure hematite, respectively. The values of $H_{\text{s.i.}}(x)$ and $H_{\text{s.i.}}(0)$ were determined from the concentration dependence of T_M. Use was made of the fact that at $T=T_M$ the effective field $H_a{}^*$ reverses sign. The temperature dependences of the corresponding anisotropy fields are given by the expressions[12]

$$\begin{gathered}H_{\text{dip}}(T)=H_{\text{dip}}(0)m(T),\\ H_{\text{s.i.}}(T)=H_{\text{s.i.}}(0)\hat{I}_{5/2}[\mathcal{L}^{-1}(m)]/m(T).\end{gathered} \tag{3}$$

Here $m(T)=M_0(T)/M_0(0)$ was the reduced temperature dependence of the sublattice magnetization, $I_{5/2}$ is the reduced hyperbolic Bessel function normalized to unity at $T=0$, and $\mathcal{L}^{-1}(m)$ is the inverse Langevin function. The concentration dependence of $H_{\text{dip}}(x)$ was taken into account in analogy with Ref. 1.

The concentration dependence of the contribution of the Co^{2+} ion to the single-ion anisotropy, referred to $T=0$ K, is shown in Fig. 3. The linear character of the dependence makes it possible to calculate the single-ion contribution to the uniaxial anisotropy constant $b=H_aM_0$ at $T=0$ K:

$$\Delta b_{\text{s.i.}}^{\text{Co}}=3.1\cdot 10^{-15}\ \text{erg/ion}.$$

We cite for comparison the single-ion contribution of Co^{2+} to the uniaxial anisotropy constant in MnO (Ref. 13):

$$|\Delta b|=6.5\cdot 10^{-15}\ \text{erg/ion}.$$

The data obtained were used to calculate the temperature and concentration dependences of the effective field $H_a{}^*=H_a+H_D{}^2/2H_E$ (Fig. 4). The Morin transition takes place at the abscissa intercept of the temperature dependence of $H_a{}^*$ for the corresponding impurity concentration. The solid line in Fig. 3 shows the theoretical concentration dependence of $T_M(x)$. It can be seen from Figs. 3 and 4 that the concentration $x_{\text{cr}}=0.007$ is critical; at a Co^{2+} content higher than $x_{\text{cr}}/2$ the hematite remains weakly ferromagnetic in the entire temperature region down to $T=0$ K.

2. Low-frequency AFMR branch

The frequency and field dependences of the AFMR were measured at 300, 77, and 4.2 K for samples with different Co^{2+} contents. The most general form of the expression for the low-frequency AFMR branch is[1]

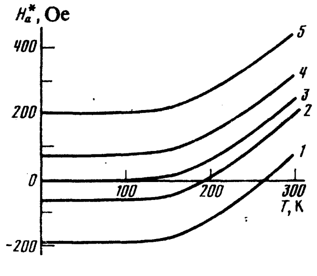

FIG. 4. Temperature dependence of the effective field $H_a{}^*$. Impurity content x: 1—0; 2—0.005; 3—0.007; 4—0.01; 5—0.015.

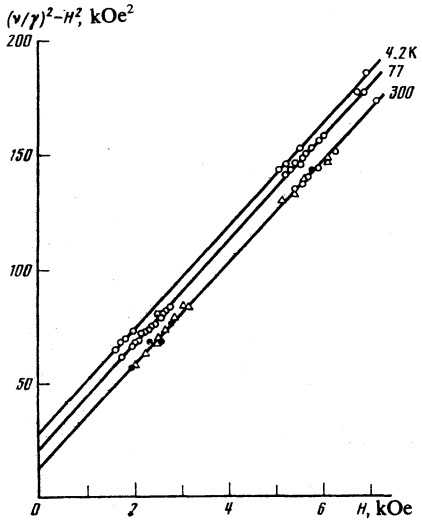

FIG. 5. Frequency and field dependences of AFMR in α-Fe_2O_3: Co. △—$x=0$; ●—$x=0.005$; ○—$x=0.015$.

$$(\nu/\gamma)^2=H(H+H_D)+H_\Delta{}^2+36H_EH_{a6}\cos 6\varphi_H{}'+2H_EH_{a2}\cos 2\varphi_H{}''. \tag{4}$$

Here H is the external magnetic field applied in the basal plane, H_Δ^2 is the isotropic energy gap, H_{a2} and H_{a6} are the effective fields of the uniaxial and hexagonal magnetic anisotropy in the basal plane, φ_H' and φ_H'' are the azimuthal angles between the external magnetic field H and the corresponding preferred direction. The last term in (4) was added to describe the experimentally observer uniaxial anisotropy in the basal plane. Typical frequency and field dependences are shown in Fig. 5. Reduction of the plots by least squares yields the effective field values listed in Table I. Analysis of the data shows that the Dzyaloshinskiĭ field is practically independent of both the concentration and the temperature in the interval 4.2–300 K.

The temperature dependences of the AFMR parameters (of the anisotropy in the basal plane, of the energy gap H_Δ^2, and of the line width ΔH) were measured in the interval 4.2–300 K for samples with the following impurity contents: $x=0.008$ (sample No. 1), $x=0.009$ (No. 2), $x=0.011$ (No. 3) and $x=0.015$ (No. 4). The samples were disks 2.0–2.8 mm in diameter and 0.5–0.8 mm thick, and their planes coincided with the basal plane. All the samples were annealed beforehand at $T=1200$ °C.

The anisotropy of the resonant field in the basal plane was determined by the joint influence of the hexagonal and uniaxial anisotropies. To separate the contributions, the experimental angular dependence was reduced in accord with Eq. (4). By way of example, the thin lines in Fig. 6 show the angular dependences of the corresponding contributions δH_6 and δH_2 to the resonance field for sample No. 3 at room temperature ($H_{a6}=-0.34\cdot 10^{-2}$ Oe; $H_{a2}=7.1\cdot 10^{-2}$ Oe). The thick line shows the theoretical angular dependence of the resonance field with allowance for these contributions. The angular dependence of the resonance field at $T=77$ K

TABLE I. Effective α-$Fe_{2-x}Co_{x/2}Si_{x/2}O_3$, parameters obtained from the AFMR frequency and field dependences.

	T, K	H_D, kOe	H_Δ^2, kOe^2	H_{a6}, 10^{-2}Oe	H_{a2}, 10^{-2}Oe	ΔH, Oe*
$x = 0$ sample № 1	300	21.7±0.2	14.7±0.3	0	0	80
$x = 0$ sample № 2	300	21.8±0.2	14.6±0.3	0	0	72
$x = 0.005$	300	22.0±0.2	15.4±0.4	—	—	250
$x = 0.015$	300	22.4±0.2	11.6±0.5	—0.1	1.7	230
	77	22.5±0.3	11.1±0.8	2.5	2.0	130
	4.2	22.2±0.3	22.5±0.8	—0.5	—	350

*At a frequency $\nu = 24500$ MHz.

shows that the uniaxial anisotropy is practically unchanged but its sign is reversed.

Typical temperature dependences of the AFMR parameters are shown in Figs. 7 and 8. For all four samples, the hexagonal anisotropy at room temperature is small and depends weakly on the temperature down to $T \approx 200$ K. With further cooling of the sample, the sign of H_{a6} is reversed and the weakly ferromagnetic moment σ_0 is reoriented from the mirror plane to the twofold axis (at $T = 160$–175 K for the different samples), and the absolute value of the hexagonal anisotropy increases considerably. At temperatures below 40 K the hexagonal anisotropy again behaves anomalously, causing one more spin-flip transition (at $T = 5$–10 K for different samples). The temperature of the transition and the absolute change of the anisotropy field H_{a6} vary in this case from sample to sample.

It is known[10,11] that the hexagonal anisotropy in the basal plane is determined by a combination of the anisotropy constants:

$$H_{a6} = \frac{1}{2M_0}\left(e - \frac{q^2}{4b}\right), \tag{5}$$

where b, q, and e are the anisotropy constants at the invariants of second, fourth and sixth orders in the components of the antiferromagnetism vector. In pure hematite the temperature dependence of the constant b leads to a steep rise of the hexagonal anisotropy in the region of T_M (Refs. 14, 15). For the substitutional samples investigated by us, however, with $x \approx 0.01$, the value of $1/b$ changes little in the interval 4.2–200 K (by an approximate factor of two). Therefore the growth of the anisotropy is connected mainly with the temperature dependence of the constants e and q.

The temperature dependence of H_{a6} can be represented formally as a sum of two contributions. For sample No. 4 these contributions are shown by the dashed and dash dot lines in Fig. 7. Comparison with the temperature dependences of the first and second anisotropy constants of cobalt-containing ferrites (see e.g., Refs. 8 and 16) allows us to conclude that the positive contribution to H_{a6} is apparently due to the Co^{2+} ions, and the comparison with these anisotropy constants makes sense, since they are likewise coefficients of the invariants of fourth and sixth order in the magnetization components.

It is difficult at present to explain unambiguously the origin of the negative contribution to H_{a6}, and particularly its low-temperature anomalous growth. It is interesting that a similar behavior of the hexagonal anisotropy with an equally abrupt increase of the negative contribution to H_{a6} at low temperatures was observed in $FeBO_3$ single crystals.[17] The appearance of this contribution was attributed in that reference to uncontrollable impurities, the most probable of which is apparently Fe^{2+}.

It was observed in Ref. 5 that in a hematite single crystal in which an Fe^{2+} impurity was specially produced, an

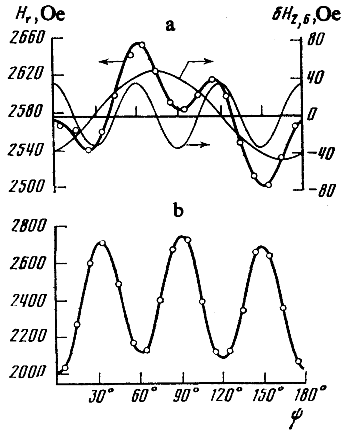

FIG. 6. Angular dependences of the resonance field H_r in sample No. 3 $x = 0.011$): a—$T = 295$ K, $\nu = 24395$ MHz; b—$T = 77$ K, $\nu = 24490$ MHz.

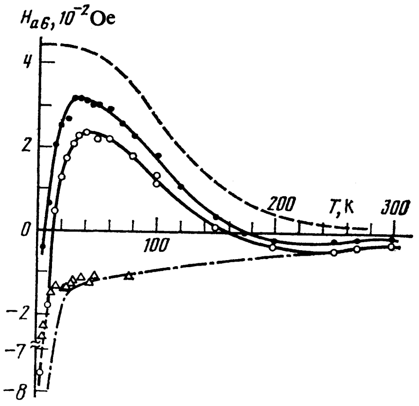

FIG. 7. Temperature dependences of the effective field of the hexagonal anisotropy H_{a6}: ○ – $x = 0.009$; ● – $x = 0.015$; Δ was calculated from the data of Ref. 5.

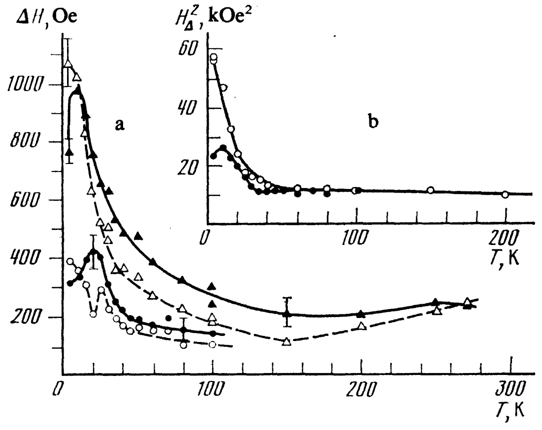

FIG. 8. Temperature dependence of the AFMR parameters. a) Linewidth ΔH when the external magnetic field is oriented in the mirror plane (dark circles) and along a twofold axis (light circles): △, ▲ — $x = 0.009$; ○, ● — $x = 0.015$, b) Energy gap $H_\Delta{}^2$: ○ — $x = 0.009$; ● — $x = 0.015$.

anomalous growth of the hexagonal anisotropy is indeed observed and is due to the freezing of the electron jumps at temperatures below 20 K. It is unfortunately impossible to determine from the data of Ref. 5 the sign of the effective field H_{a6}. However, if it is assumed that the contribution of the Fe^{2+} ions is negative, the temperature dependence of H_{a6}, calculated from the data of Ref. 5 and shown by the triangles in Fig. 7, agrees qualitatively with the negative contribution. It is important to note that an anomalous behavior of the line width ΔH (Fig. 8a) and of the isotropic energy gap H_Δ^2 (Fig. 8b) are observed in exactly the same temperature region. Similar phenomena were observed in Refs. 5 and 17, and a comparison of the data for samples 1–4 shows that the larger the absolute change of the hexagonal anisotropy in the low-temperature region, the more strongly pronounced the anomalies of H and H_Δ^2. At the same time, in the region of the other phase transition ($T \approx 160$–175 K) no anomalies of ΔH and H_Δ^2 were observed at all. Summarizing all these facts, we can suggest that the anomalous behavior of the resonance parameters H_{a6}, ΔH, and H_Δ^2 at low temperatures have a common cause, possibly the freezing of the electron jumps. In our case, furthermore, a part can be played in the valence-exchange processes not only by the iron ions,[2] as in hematite with tin impurity, but also by cobalt ions.

When α-Fe_2O_3:Sn^{4+} samples were cooled in an external field, uniaxial anisotropy in the basal plane was noted and attributed in Ref. 5 to freezing, below 18 ± 1 K, of the Fe^{2+}-Fe^{3+} electron jumps. In our case the resonance parameter H_{a6}, ΔH and H_Δ^2 at $T = 4.2$ K had an insignificant dependence on the cooling method, and when the magnetic field was rotated in the basal plane they relaxed slowly to the equilibrium values shown in on the plots. The relaxation time was of the order of ten seconds. The freezing of the electron jumps seems only to start at temperatures close to that of liquid helium. This is possibly why the uniaxial induced anisotropy was not observed on cooling to $T = 4.2$ K in any of the samples. At the same time, in the basal plane uniaxial induced anisotropy exists already at room temperature and, as already noted, does not depend on the temperature. In addition, its value and its characteristic direction vary randomly from sample to sample. It appears that this induced anisotropy is due to ion-ordering processes that proceed via ion diffusion. The characteristic direction of such an ordering can be set by the internal stresses that arise when the crystals are grown, or by an external magnetic field. Understandably, this ordering is frozen already at room temperature.

6. CONCLUSIONS

1. From among all the known impurities, Co^{2+} ion exerts the strongest influence on the Morin temperature in hematite. This is due to the substantial change of the single-ion anisotropy on account of the strong spin-lattice coupling of the Co^{2+} ion in an octahedral position. The contribution of the Co^{2+} ion to the single-ion anisotropy constant at $T = 0$ K is 3.1×10^{-15} erg/ion.

2. The impurity concentration $x_{cr} = 0.007$ is critical. At a Co^{2+} content larger than $x_{cr}/2$ the hematite remains weakly ferromagnetic in the entire temperature region.

3. The exchange field and the Dzyaloshinskiĭ field are practically independent of the Co^{2+} impurity.

4. The temperature dependence of the effective hexagonal-anisotropy field offers evidence that in the regions 5–10 K and 160–175 K there exist two spin-flip transitions in the basal plane of the crystal. The contribution of the Co^{2+} ions to the hexagonal anisotropy becomes considerable at temperatures lower than 20 K. At low temperatures (<40 K) one observes an anomalous behavior of the hexagonal anisotropy, of the width of the AFMR line, and of the energy gap in the AFMR spectrum. These anomalies are possibly connected with the freezing of the electron jumps.

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[1] Strictly speaking, the Morin transition takes place when the expression $H_a{}^* = H_a + H_D{}^2/2H_E$ reverses sign,[11] where $H_a = H_{\rm dip} + H_{\rm s.i.}$

[2] A certain number of Fe^{2+} ions is always present in the crystals because of valency exchange with cobalt ions, an excess amount of Si^{4+} or other causes connected with the crystal-production technology.

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