SPIN ORDERING AND MAGNETOCRYSTALLINE ANISOTROPY IN SINGLE CRYSTALS OF $BaCo_xFe_{18-x}O_{27}$ *FERRITES*

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Submitted to JETP editor October 25, 1965

J. Exptl. Theoret. Phys. (U.S.S.R.) **50**, 595-604 (March, 1966)

The neutron diffraction method was used to investigate the influence of cobalt ions on the positions of the spin ordering axes in crystals of hexagonal ferrites $BaCo_xFe_{18-x}O_{27}$ in the temperature range from 77° K to the Curie temperature. The temperature dependence of the magnetic anisotropy constants was investigated in the same range of temperatures and compared with the theory. The results of the investigation of the magnetic anisotropy were compared with the neutron diffraction results. It was established that the spin directions coincided with the directions of the total magnetization vector of the crystals.

INTRODUCTION

HEXAGONAL ferrites of the $BaCo_xFe_{18-x}O_{27}$ system (Co_xW, with the W type structure) have the interesting property that, depending on the cobalt content and on temperature, they exhibit various types of magnetic anisotropy, characterized by an easy magnetization axis, a cone of easy magnetization, or a plane of easy magnetization.[1,2] Moreover, at low temperatures the Co_xW ferrites exhibit a strong anisotropy in the basal plane.[1] These properties of the magnetic anisotropy of crystals of the Co_xW system have naturally attracted attention to the problem of how changes in the direction of the total magnetic moment, found from measurements of the magnetic anisotropy, are related to the directions of elementary magnetic moments. In view of this interest, an investigation was made of the magnetic structure of these ferrites by the neutron diffraction method. The results of this investigation are presented in the first section of the present paper.

In the second section, we present new data (compared with [1]) on the magnetic anisotropy energy of the Co_xW system. In discussing the magnetic anisotropy properties we have used the data obtained in the neutron diffraction studies. Particular attention has been paid to the results of an investigation of the temperature dependence of the magnetic anisotropy constants and comparison of the experimental data with the theory.

We investigated crystals grown by the Verneuil method and used in [1] (from $x = 0$ to $x = 1.5$), as well as crystals with $x = 1.5$, 1.65, and 1.75, grown from a molten solution.

1. NEUTRON DIFFRACTION STUDY OF THE MAGNETIC STRUCTURE

The atomic structure of type W hexagonal ferrites was investigated by Braun,[3] who studied the ferrite Fe_2W. He determined the unit cell dimensions ($a = 5.88$ Å, $c = 32.84$ Å), the space group ($P6_3/mmc$), and established the general nature of the distribution of the 92 ions in a unit cell.

There are as yet no published x-ray diffraction data on type W ferrites in which iron is partly replaced with cobalt. E. N. Belova of the Crystallography Institute obtained x-ray diffraction patterns of Co_xW ferrites containing various amounts of cobalt and found that, when iron was replaced with cobalt, the structure type W, the space group $P6_3/mmc$, and the cell parameters remained practically unchanged, and equal to the Braun values, up to $x = 1.75$.

From his considerations of the dependence of the magnitude of the exchange interaction on the Me–O–Me distances and the corresponding angles between bonds, Gorter[4] proposed a configuration of the mutual spin orientations associated with ions at various crystallographic positions, which explained satisfactorily the macroscopic magnetic properties of the Fe_2W ferrite. According to this configuration, the magnetic Fe^{3+} and Fe^{2+} ions form two sublattices, in each of which the spins are parallel while the moments of the two sublattices are respectively antiparallel and parallel to the c axis. However, the results of an investigation of the magnetic anisotropy of ferrites of the Co_xW series[1,2] indicated that this model was acceptable only for the ferrite Fe_2W.

To determine the spin ordering configuration in the Co_xW ferrites, we recorded the powder neutron diffraction patterns and investigated the temperature dependence of the intensities of some diffraction reflections for single-crystal samples. To obtain the neutron diffraction patterns of single crystals, we used the same samples as in the investigation of the magnetic anisotropy. To prepare polycrystalline samples we used a powder obtained by the mechanical pulverization of single crystals. The measurements were carried out using a neutron diffractometer[5] in the temperature range 77–770° K at $\lambda = 1.05$ Å. To isolate the nuclear contribution to the diffraction pattern, we recorded it in a magnetic field up to 20,000 Oe, parallel to the scattering vector ϵ.

The neutron diffraction patterns of the powders were recorded at fixed positions separated by intervals of 5 angular minutes, obtaining a statistical count at each position for 10 min and reducing the number of counted pulses to a standard number measured with a monitor counter. The intensities were made absolute by reference to a NaCl standard. The integrated intensity of the diffraction maxima was determined (by weighting) as the area enclosed between the peak profile and the background level. The total error in the determination of the intensity did not exceed ±8%. The patterns were recorded at 77 and 293° K.

In the case of single-crystal samples, the intensities of the diffraction maxima were recorded on a chart of an electronic potentiometer when the sample was slowly rotated so as to pass through the reflecting position (the neutron detector was kept fixed). We measured the integrated intensity of the diffraction maxima 00·6 and 11·0 when the sample temperature was varied from 77 to 770° K. The error in these measurements did not exceed ±10%.

The neutron diffraction patterns of the polycrystalline samples were characterized in general by well-resolved basal reflections 00·4 and 00·6, a very strong peak, which was mainly of magnetic origin and due to closely-spaced reflections 10·0 + 10·1 + 10·2, and a number of weaker overlapping reflections.

By way of illustration, Fig. 1 shows the powder diffraction patterns of $Co_{1.75}W$ and $Fe_{2.0}W$, recorded at room temperature, and the neutron diffraction pattern of $Fe_{2.0}W$, recorded in a magnetic field parallel to ϵ. It is evident from Fig. 1 that, in the neutron diffraction pattern of Fe_2W, the reflections from the planes parallel to the basal plane are solely due to the nuclear scattering, which indicates that the probable orientations of

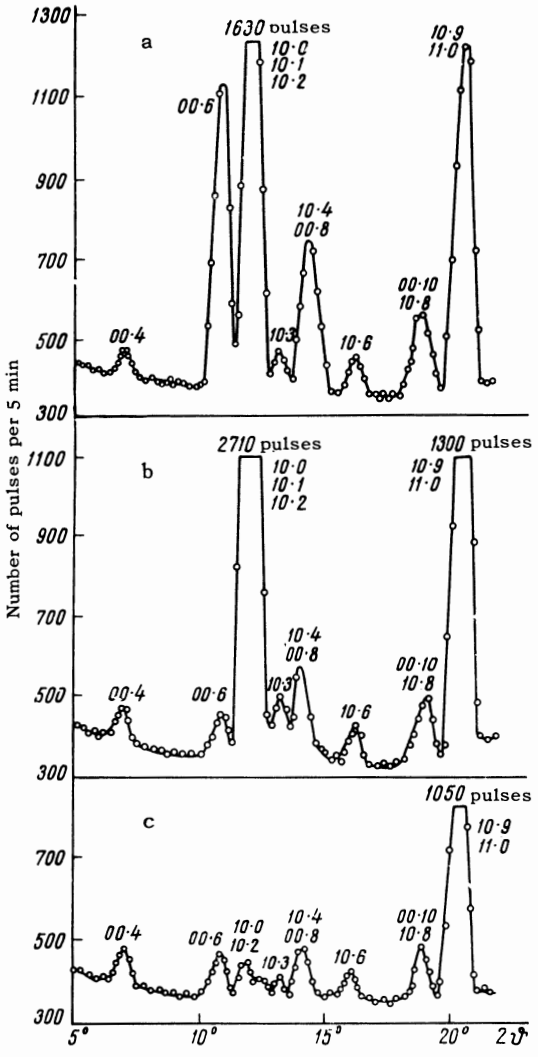

FIG. 1. Neutron diffraction patterns of: a) $Co_{1.75}W$; b) Fe_2W; c) Fe_2W in a magnetic field of 20 000 Oe, directed parallel to the scattering vector ϵ, at T = 293°K.

the spin magnetic moment are perpendicular to the basal plane, i.e., along the c axis. Bearing in mind that the absolute value of the saturation magnetization of Fe_2W and its temperature dependence were, as mentioned earlier, in satisfactory agreement with Gorter's configuration, we assumed that it was valid also in our case.

The basal reflections of $Co_{1.75}W$ include a considerable magnetic contribution. It can be explained by deviations of the spins from the c axis. The values of the nuclear contribution to the basal reflections of this ferrite differ from the corresponding values for Fe_2W, due to the considerable difference between the nuclear scattering amplitudes of iron and cobalt. This has made it possible to refine the positions of the divalent cobalt ions, which were ascribed the position 12k with the coordinate z = 0.1499.

In our analysis of the intensities of the magnetic

reflections we found that, if the Gorter model of two sublattices with oppositely directed moments[4] was assumed for the Co_xW composition and these moments were taken to be oriented along a new axis forming some angle ϑ_0 with the c axis, the correct values were obtained for the contribution of the magnetization to the diffraction pattern and for the absolute value of the magnetization.

Having assumed this magnetic ordering, we calculated the values of the angles ϑ_0, using the intensities of the observed magnetic reflections 00·4 and 00·6, by means of the formula

$$I_{\text{mag}}^{\text{obs}} = K_{hkl} F_{hkl}^2 \sin^2 \vartheta_0,$$

where K_{hkl} is a constant for a given reflection, including the instrumental constant, geometrical factors, the absorption factor, etc.; F_{hkl} is the structure factor. The theoretical intensities of the other reflections were calculated using these values of ϑ_0.

Table I lists the calculated and measured values of the intensities of the magnetic reflections and of the angle ϑ_0 for the powder diffraction patterns of the investigated compositions. The values of the form factors of Fe^{3+} were taken from [6] and those of Fe^{2+} and Co^{2+} from [7]. The temperature correction was introduced using Braun's data.[3] The value of $[\sigma/\sigma_0]^2$ was found from the magnetization curve given in Fig. 7. The value of $\langle q^2 \rangle$ was calculated following Shirane.[8]

The close agreement between the calculated and measured intensities proved the correctness of the proposed magnetic ordering configuration. Table I shows also that the compositions containing cobalt shared a uniaxial spin ordering model at high temperatures and a tendency of the spin

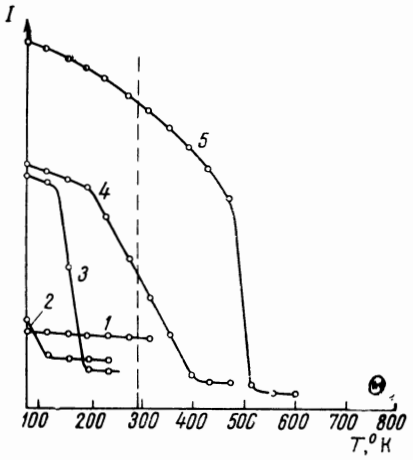

FIG. 2. Temperature dependence of the intensity of the 00·6 reflection for single crystals of: 1) Fe_2W; 2) $Co_{0.5}W$; 3) $Co_{1.0}W$; 4) $Co_{1.5}W$; 5) $Co_{1.75}W$.

axes to reorientate parallel to the basal plane when temperature was reduced. In the compositions with high cobalt content (x = 1.65, 1.75) the spin axes were already in the basal plane at room temperature. In the compositions with low cobalt content these axes made a large angle with the c axis only at the temperature of liquid nitrogen. An interesting property was exhibited by the ferrite $Co_{1.65}W$. In this ferrite the spins were oriented parallel to the basal plane at room temperature, but at 77° K they deviated again from this plane. This was manifested in the magnetic anisotropy by the appearance of a cone of easy magnetization at low temperatures (cf. Sec. 2, Fig. 4).

The information which could be obtained by an analysis of the neutron diffraction patterns of the polycrystalline samples was restricted to the general features of the spin ordering and of the angle between the spin axes and the c axis. Additional

Table I. Experimental (I_e) and calculated (I_c) intensities of magnetic reflections and the angles ϑ_0 for the compositions Co_xW *)

Composition	T,°K	10·0 + 10·1 + 10·2		10·4 + 00·8		10·9 + 11·0		00·6	ϑ_0, deg
		I_c	I_e	I_c	I_e	I_c	I_e	I_e	
Fe_2W	293	18230	17530	1270	1200	1880	1976	0	0
	77	22800	21920	1590	1520	2380	2210	0	0
$Co_{0.5}W$	293	17950	17050	1250	1160	1850	1960	0	0
	77	22600	21760	1790	1730	2370	2246	403	13
$Co_{1.0}W$	293	17600	16930	1240	1360	1815	1720	0	0
	77	17540	16480	2870	2630	1950	2030	3465	45
$Co_{1.5}W$	293	14240	13610	2000	1840	1490	1580	2120	39
	77	17190	16360	2830	2980	1900	1776	3480	45
$Co_{1.65}W$	293	9520	8900	3140	3010	1120	1148	5290	90
	77	12470	11770	3900	3760	1510	1400	6310	75
$Co_{1.75}W$	293	9640	9000	3140	2910	1110	1180	5310	90
	77	12080	11660	4020	3800	1460	1300	6620	90

*) Maximum error in the angle was ±5%.

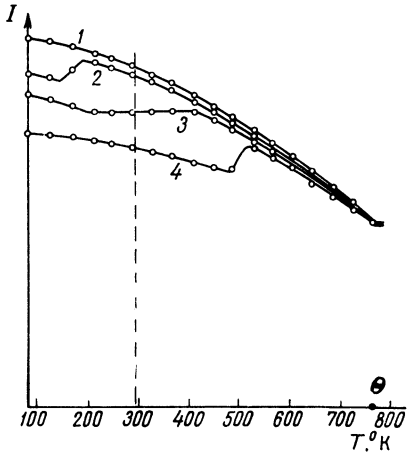

FIG. 3. Temperature dependence of the intensity of the 11·0 reflection for single crystals of: 1) Fe_2W; 2) $Co_{1.0}W$; 3) $Co_{1.5}W$; 4) $Co_{1.75}W$.

information was obtained from observations of the temperature dependence of the intensity of the 00·6 and 11·0 reflections of single-crystal samples (Figs. 2 and 3). From these figures, it is evident that each composition, except Fe_2W, had characteristic ranges of temperature in which the spin deviated from the c axis.

This was particularly clear in the case of $Co_{1.75}W$. Above 520° K, the 00·6 reflection of this composition consisted only of the nuclear contribution (spins parallel to the c axis). In the temperature range 520–480° K, the 00·6 reflection intensity rose rapidly, indicating a reorientation of the spins away from the c axis and toward the basal plane. When the temperature was reduced further, the intensity of the 00·6 reflection continued to increase slowly, repeating the magnetization curve (cf. Fig. 7 in the next section).

A rapid variation of the intensity in the range 520–480° K was observed also for the 11·0 reflection. Above 758° K, the intensity of this reflection was practically constant and equal to the nuclear contribution. As the temperature fell the intensity repeated the magnetization curve, but at 520–480° K it rapidly decreased. This drop in the intensity was the same for all six reflections from the (11·0) plane, recorded by rotating a sample about its c axis. The only explanation of this observation is the transformation of a single-domain single crystal, for which $\vartheta_0 = 0$, into a polydomain crystal ($\vartheta_0 \neq 0$), in which each domain has its own spin axis. A similar effect was observed in a neutron diffraction investigation of hexagonal cobalt.[9] The neutron diffraction pattern showed that, in a certain range of temperatures, the magnetic structure changed and the spins deviated from the direction parallel to the c axis toward the basal plane, and a magnetically uniaxial (single-domain) crystal split into domains whose spin axes lay in the basal plane, making an angle of 120° with one another.

The existence of a similar mechanism of splitting of Co_xW single crystals into domains was confirmed by the observation of a system of six easy magnetization axes, whose projections on the basal plane made angles of 60° with one another. The measurements of the positions of these axes, carried out with a neutron diffractometer, gave the results listed in Table II.

Table II. Positions of spin axes

Composition	Plane in which spin axis lies and angle	
	at 77° K	at 293° K
Fe_2W	$\parallel c$	$\parallel c$
$Co_{0.5}W$	(10·0), 13°	$\parallel c$
$Co_{1.0}W$	(10·0), 45°	$\parallel c$
$Co_{1.5}W$	(10·0), 45°	(11·0), 39°
$Co_{1.65}W$	(10·0), 75°	(11·0), 90°
$Co_{1.75}W$	(10·0), 90°	(11·0), 90°

It is clear from Table II that the characteristic feature of all the cobalt-substituted compositions is that at 77° K the easy magnetization axes lie in the (10·0) planes making an angle with the c axis, which increases as the cobalt content increases. At higher temperatures (293° K), the spin axes of the compositions with $x \geq 1.5$ lie in the (11·0) planes. In the case of the composition with $x = 1.65$, whose spin axes are in the basal plane at room temperature, these axes leave the basal plane again at 77° K making an angle of 75° with the c axis.

Table III compares the experimental and calculated intensities of some reflections with the highest magnetic contribution in the case of single crystals of the investigated compositions. The value of the coefficient of the primary extinction was calculated in the same way as in [10]. The expression for $\langle q^2 \rangle$ of a single crystal was obtained by averaging over the angles η between the normal to the reflecting plane and the spin axes of domains, whose projections on the basal plane made an angle of 120° with one another. (For this spin axis configuration, $\langle q^2 \rangle_{\text{single-cryst.}} = \langle q^2 \rangle_{\text{polycryst.}}$)

It must be mentioned that in an investigation of Co_1W single crystals, grown by the Verneuil method, it was found that the neutron diffraction pattern included two superstructure peaks located symmetrically with respect to the structure re-

Table III. Ratio of calculated and experimental intensities of neutron diffraction in Co_xW crystals at temperatures of 77 and 293° K

Composition	$hkl = 00{\cdot}6$		$hkl = 11{\cdot}0$	
	$I_c^m E_p^m / I_c^n E_p^n$	I_e^m / I_e^n	$I_c^m E_p^m / I_c^n E_p^n$	I_e^m / I_e^n
$Co_{0.5}W$	1.9	1.95 ±0.2	1.06	1.04 ± 0.06
$Co_{1.0}W$	6.1	6.2 ±0.4	1.14	1.07 ± 0.06
$Co_{1.5}W$	1.37	1.40 ±0.1	1.11	1.12 ± 0.07
$Co_{1.75}W$	1.25	1.18 ± 0,05	1.10	1.10 ± 0.07

Superscripts m and n refer to 77 and 293°K, respectively.

flection 10·0. Measurements carried out at high and low temperatures in magnetic fields showed that these peaks were antiferromagnetic in nature. Their appearance may be associated with the existence of a magnetic structure in the form of a conical spiral whose axis is parallel to the c axis and spins that lie along the cone generatrices. A magnetic structure of this type may be considered as an assembly of interpenetrating sublattices, containing magnetic ions, with antiferromagnetic ionic spin coupling between the sublattices. The spiral extended in the direction [10·0] and its period was ≈ 62 Å. The superstructure was found in the temperature range 170–340 ° K.

The intensity of the "satellites" of some samples was comparable with the intensity of the nuclear structure reflections, while in other samples the satellite intensity was at the limit of the sensitivity of the apparatus. No superstructure reflections were found for single crystals grown from a molten solution. We could assume that the appearance of such reflections was associated with imperfections of the cation distribution in samples prepared by the Verneuil method.

2. MAGNETOCRYSTALLINE ANISOTROPY

The investigation was carried out, in the same way as in [1], by the method of rotating moments. The equilibrium orientation of the magnetization vector, represented by the angle ϑ_0 between the c axis and the easy magnetization direction, in crystals of various compositions in the Co_xW system is given in Fig. 4 as a function of temperature. The Fe_2W crystals (x = 0) had, over the whole temperature range from 77 ° K to the Curie temperature, an easy magnetization axis coinciding with the c axis (this case is not shown in Fig. 4). When $Co_{0.5}W$ (x = 0.5) and Co_1W (x = 1) crystals were cooled from the Curie point to the temperature of liquid nitrogen, the easy magnetization direction gradually turned away from the c axis ($\vartheta_0 = 0$) to a direction lying on the circular cone whose vertex angle was $\vartheta_0 = 10°$ and $\vartheta_0 = 45°$ for $Co_{0.5}W$ and Co_1W, respectively. In a $Co_{1.75}W$ (x = 1.75) crystal, the direction of easy magnetization changed from the c axis to the basal plane at about 470 ° K. The composition $Co_{1.65}W$ (x = 1.65) behaved in a very interesting way. Above 470 ° K, the easy magnetization direction changed from the c axis to a cone whose vertex angle increased when temperature was reduced. Near 330 ° K, the cone opened up into a plane. As the temperature dropped further, the nature of the anisotropy was first conserved, but below 170 ° K a cone appeared again and the vertex angle of this cone decreased when the temperature was reduced.

In Fig. 4, the black dots show the angles between the c axis and the directions of the spin moments in crystals, which were obtained in the neutron diffraction investigation (cf. Table II). The spin directions coincide with the easy magnetization direction, i.e., with the position of the total magnetization vector of a crystal.

The magnetic anisotropy energy was expanded in [1] into a power series:

$$E = K_1 \sin^2\vartheta + K_2 \sin^4\vartheta + \ldots + K_3 \sin^6\vartheta \cos 6\varphi, \quad (1)$$

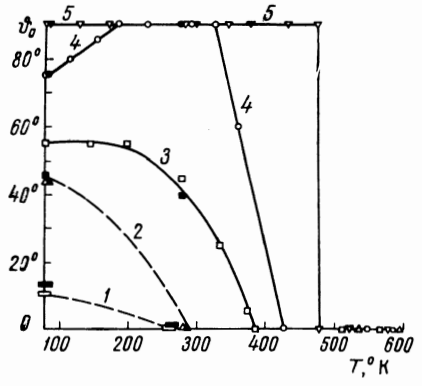

FIG. 4. Temperature dependence of the value of the angle ϑ_0: 1) x = 0.5; 2) x = 1.0; 3) x = 1.5; 4) x = 1.65; 5) x = 1.75.

where the angle ϑ gives the orientation of the magnetization vector with respect to the c axis, and the angle φ is measured in the basal plane. However, analysis of the temperature dependence of the constant K_1 showed that this constant had a discontinuity at a certain temperature. It seemed more useful to use an expression for the magnetic anisotropy energy in the form of an expansion in terms of harmonic polynomials. As particularly stressed by Turov,[11] such an expansion is the most rational for a correct analysis of the experimental data and in a comparison with a theory.

Following Bickford,[2] this expansion may be written in the form

$$E = S_0 + S_2(\sin^2\vartheta - {}^2/_3) + S_4(\sin^4\vartheta - {}^8/_7\sin^2\vartheta + {}^8/_{35}) + S_6(\sin^6\vartheta - {}^{18}/_{11}\sin^4\vartheta + {}^8/_{11}\sin^2\vartheta - {}^{16}/_{231}) + S_6'\sin^6\vartheta\cos 6\varphi. \quad (2)$$

The dependence of the constants S_{2n} on the composition is shown in Fig. 5 for temperatures of 290 and 77° K. A characteristic feature of this dependence is the approximately additive variation of the constant S_2 when the cobalt content is increased.

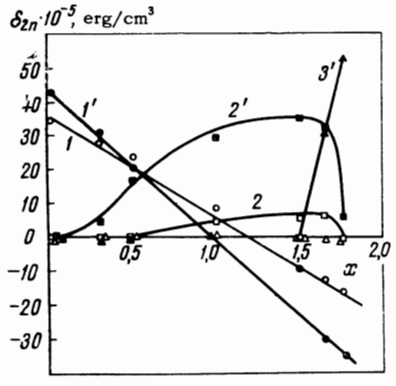

FIG. 5. Dependence of the magnetic anisotropy constants on the composition: 1) S_2 at 293°K; 2) S_4 at 293°K; 3) S_6 at 293°K; 1') S_2 at 77°K; 2') S_4 at 77°K; 3') S_6 at 77°K.

At low temperatures and high cobalt contents, higher-order anisotropy constants S_4 and S_6 begin to play an important role.

For $x > 0$ and low temperatures, we observe a strong magnetic anisotropy in the basal plane (the constant S_6' becomes comparable with constants of lower order). In the case of crystals of composition $x \geq 1.65$, this anisotropy appears also at room temperature. Figure 6 shows the temperature dependence of the constant S_6' for the composition $x = 1.65$. At 220° K, the sign of S_6' changes. It should be mentioned that in the case of non-zero values of S_6' the concept of a cone of easy magnetization requires refinement because the anisotropy energy then depends on the angle φ in the basal plane.

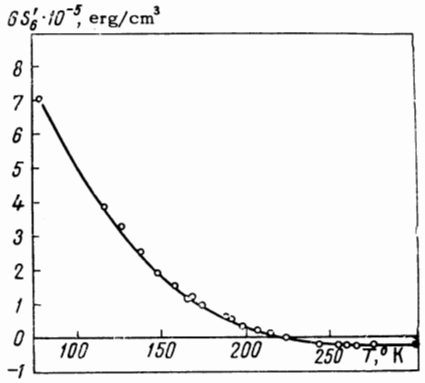

FIG. 6. Temperature dependence of the "basal" anisotropy constant S_6' of $Co_{1.65}W$.

From a comparison of Figs. 5 and 6, it follows that there is a plane of easy magnetization ($S_6' = 0$) only at 220° K. Above this temperature, there are six directions in the basal plane along which the anisotropy energy is less than that along other directions in the same plane. When the temperature is raised further, these directions leave the basal plane and become generatrices of a circular cone. The same applies below 220° K, when a crystal has six easy magnetization directions in the basal plane. Below 170° K, these directions become generatrices of a cone. The neutron diffraction investigation established that above 220° K the spin directions in the basal plane coincided with the directions [11·0] and below this temperature they coincided with [10·0].

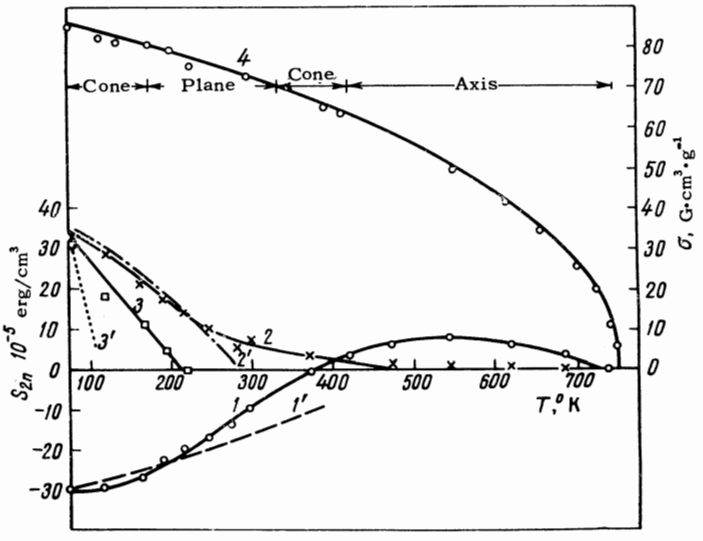

FIG. 7. Temperature dependence of the magnetic anisotropy constants S_{2n} and of the saturation magnetization σ_s $Co_{1.65}W$: 1) $S_2(T)$; 2) $S_4(T)$; 3) $S_6(T)$; 4) $\sigma_s(T)$. Curves 1', 2', and 3' were calculated using formula (3).

Figure 7 shows the temperature dependence of the magnetic anisotropy constants S_2, S_4, S_6 and of the specific saturation magnetization σ_S of the composition $Co_{1.65}W$. The value of σ_S was determined from the magnetization curves obtained with a vibration magnetometer. It should be mentioned that the value of σ_S varied little with the cobalt content x in a crystal. The Curie temperature, found to be $\Theta = 775 \pm 5°$ K from the magnetic measurements and $\Theta = 758 \pm 5°$ K from the neutron diffraction data, was also practically independent of x. A theoretical calculation of the saturation magnetization at absolute zero, carried out in the usual way allowing for the ion distribution, showed that σ_S decreased only by 5% from Fe_2W to Co_2W, i.e., the decrease was within the limits of our experimental error. The same figure shows the ranges of temperature in which a plane, a cone, and an axis of easy magnetization was obtained.

In a theoretical analysis of the temperature dependence of the magnetic anisotropy constants one can use Turov's formula[11] deduced from the phenomenological theory of spin waves:

$$\frac{S_{2n}(0) - S_{2n}(T)}{S_{2n}(0)} = n(2n+1)\frac{\sigma_s(0) - \sigma_s(T)}{\sigma_{(s)}(0)}. \qquad (3)$$

Figure 7 shows the theoretical curves of the temperature dependence of the magnetic anisotropy constants S_{2n} (curves 1′, 2′, 3′), plotted using this formula. Since formula (3) should be satisfied only at low temperatures and the determination of the coefficients S_{2n} from the experimental rotating-moment curves was not very accurate (particularly in the case of higher-order coefficients), the agreement between experiment and theory should be regarded as satisfactory.

The one-ion theory is usually employed in a theoretical analysis of the behavior of the magnetic anisotropy in ferrites. This theory is mainly applied to spinel ferrites. Bickford showed that the magnetic anisotropy of the Co_xW ferrites was satisfactorily accounted for by the one-ion theory developed by Slonczewski[12] for cobalt-substituted magnetite. This is explained by the fact that the hexagonal ferrite structure includes cubic packing layers, which are almost fully analogous to spinels. Our data indicate that the fully satisfactory agreement with the experimental results is also obtained by the use of formula (3), which follows from the general phenomenological theory without allowance for the structure model.

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