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SOLIDS
Structure

Incommensurate Magnetic Structure in Copper Metaborate

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Abstract—Based on the experimental data on copper metaborate single crystals obtained in X-ray and neutron diffraction studies and heat capacity, magnetic susceptibility, and muon spin relaxation measurements, a phenomenological theory of the incommensurate magnetic structure of this crystal was developed. Considering the space group of the crystal, $I\bar{4}2d$, Lifshits invariants were included into its thermodynamic potential. An analysis showed that magnetic structure formation at 10–20 K was dominated by the subsystem of copper spins in 4*b* unit cell sites. Below 10 K, the role played by the magnetic subsystem of copper spins in 8*d* unit cell sites in the formation of the magnetic structure of copper metaborate substantially increased. This caused a sharp increase in the wave vector of the incommensurate structure as temperature lowered. Numerical simulation of the temperature dependence of the wave vector of the helix and the heat capacity of the crystal gave a satisfactory description of the experimental data. This simulation was used to estimate the parameters of the phenomenological thermodynamic potential of the magnetic system of copper metaborate. © *2001 MAIK "Nauka/Interperiodica".*

1. INTRODUCTION

Modulated (incommensurate) magnetic structures are known to appear most often as a result of competition of exchange interactions [1]. The crystal structure then does not impose any limitations on the possibility of the formation of such structures. Less frequently, incommensurate structures are formed as a result of relativistic interactions. Dzyaloshinskii [2] was the first to mention such a possibility. The physical reason for the appearance of incommensurate structures is then the so-called Dzyaloshinskii–Moria antisymmetric exchange interaction. Formally, incommensurate structures of the relativistic origin can be described by introducing the Lifshits invariant [1], which linearly contains the first derivatives with respect to the two-component order parameter coordinates, into the thermodynamic potential. Note that an important limitation is then imposed on crystal symmetry. Namely, the Lifshits invariant can only be included into the thermodynamic potentials of crystals that have no center of symmetry. The magnetic state of a system with the Lifshits invariant is generally a lattice of magnetic solitons. In the simplest case, if magnetic crystallographic anisotropy effects are ignored, the distribution of magnetic moments obeys a simple sine law. The smallness of relativistic interactions implies the smallness of the wave vector of incommensurate magnetic structures. Close to the transition of a magnetic system into an incommensurate phase, strong diffuse neutron scattering should be observed [1]. The soliton character of magnetic ordering leads to a complex structure of magnetic satellites in the neutron diffraction pattern.

As modulated magnetic structures of the relativistic origin are rare, their detailed study, especially performed for high-quality single crystals, is of great interest. Copper metaborate CuB_2O_4 single crystals were for the first time synthesized and studied in [3–6]. Neutron diffraction data on this crystal were reported in [7]. In this work, we suggest a theoretical interpretation of the magnetic state of the spin system in copper metaborate at various temperatures; our interpretation is based on analyzing the whole set of available experimental data.

2. EXPERIMENTAL DATA

A procedure for growing high-quality large copper metaborate single crystals was described in [4]. According to X-ray and neutron diffraction studies [7], CuB_2O_4 forms tetragonal crystals, space group $I\bar{4}2d$ (D_{2d}^{12}) with lattice parameters a = 11.528 Å and c = 5.607 Å. The unit cell contains 12 formula units. Cop-

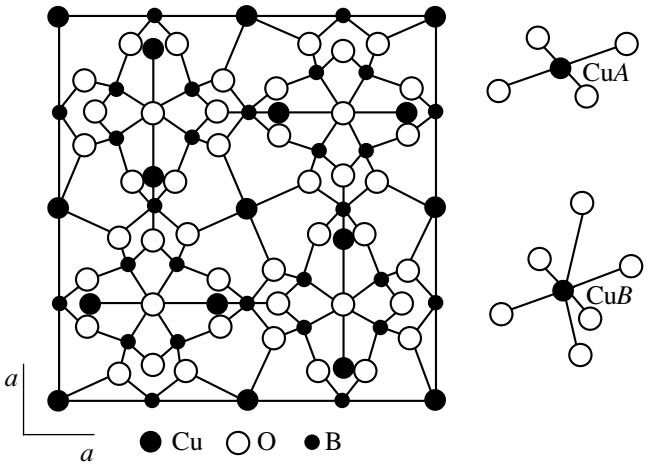

Fig. 1. Crystal structure of copper metaborate.

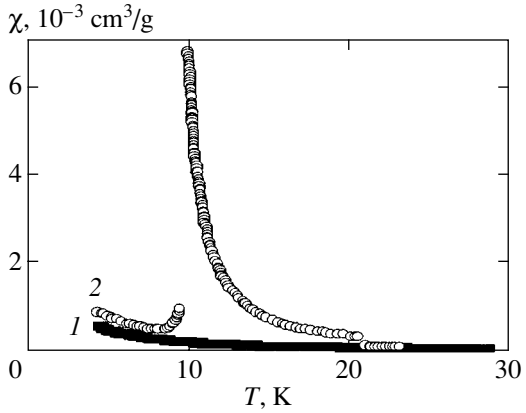

Fig. 2. Temperature dependence of magnetic susceptibility of single crystalline copper metaborate: (*1*) magnetic field is parallel to the tetragonal crystal axis and (*2*) magnetic field is parallel to the basal plane.

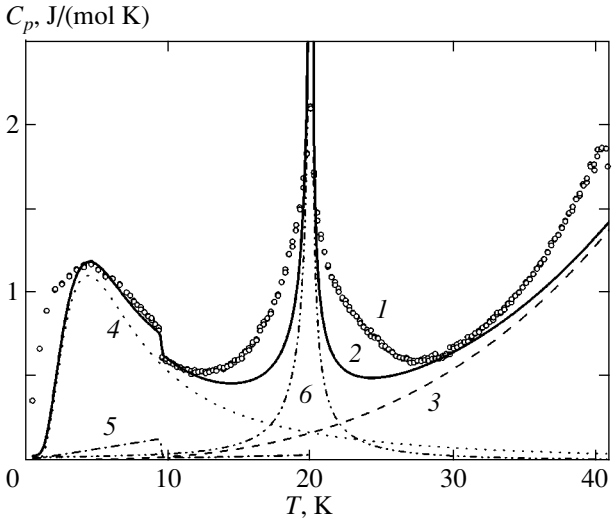

Fig. 3. Temperature dependence of the heat capacity of single crystalline copper metaborate: (*1*) experimental [3], (*2*) simulated, (*3*) Debye contribution, (*4*) Schottky anomaly, (*5*) Landau contribution, and (*6*) Ginzburg estimate.

per Cu^{2+} ions are situated in two nonequivalent positions, namely, Cu*A*, site 4*b*, point symmetry group S_4 (0, 0, 0.5) and Cu*B*, site 8*d*, point symmetry group C_2 (0.0815, 0.25, 0.125). Each Cu*A* ion occupies the center of a square formed by four oxygen ions. All Cu*B* ions are surrounded by six oxygen atoms in vertices of distorted octahedra (Fig. 1). The special high-resolution neutron diffractions study [7] showed that, up to 1.5 K, the crystal did not experience structural phase transitions of any kind. The magnetic reflections at 12 K corresponded to the Bragg commensurate phase positions. The observation of forbidden reflections of the (110) or (002) type at this temperature shows that the magnetic structure is antiferromagnetic. The magnetic and crystal chemical unit cells coincide, and the magnetic structure is described by the $\mathbf{q} = 0$ propagation vector.

Magnetic measurements on single crystals showed that sharp singularities appeared in the magnetic susceptibility curve at $T_A = 21$ K and $T_B = 10$ K. The temperature dependences of magnetic susceptibility obtained on a SQUID magnetometer for magnetic field orientations parallel and perpendicular to the tetragonal crystal axis are shown in Fig. 2 [6]. Attention is caught by the sharp anisotropy of susceptibility. For a magnetic field applied in the basal plane of the crystal, a susceptibility jump is observed at 21 K; susceptibility then rapidly increases as temperature decreases. At 10 K, susceptibility decreases in a jump by approximately one order of magnitude and then monotonically increases down to 4.2 K. For a magnetic field applied parallel to the tetragonal axis of the crystal, the temperature dependence of susceptibility is smooth in the whole temperature range. The Néel paramagnetic temperature and the effective copper ion moment determined from the high-temperature magnetic susceptibility portion equal $\Theta_N = -9.5$ K and $\mu_{\text{eff}} = 1.77\mu_B$, respectively.

The magnetic susceptibility anomalies described above are accompanied by singularities of the temperature dependence of heat capacity [3]. The results of heat capacity measurements in the temperature range 2–40 K are shown in Fig. 3. Two anomalies at the temperatures that coincide with those of magnetic susceptibility anomalies are quite manifest. In addition, the $C_p(T)$ curve contains an anomaly in the form of a broad maximum near 4 K.

Muon spin relaxation (μSR) data were reported in [3]. These data lend support to the conclusion of magnetic transformations at 21 and 10 K. More recent measurements at temperatures down to 1 K [8] revealed the occurrence of an additional magnetic transformation close to 1 K. This leads us to suggest that a new rearrangement occurs at this temperature in the spin subsystem of copper metaborate.

Neutron diffraction studies of copper metaborate were performed for a single crystal that contained the ^{11}B isotope to decrease absorption of neutrons [7]. The magnetic structure in the temperature range 10–21 K was found to be commensurate and antiferromagnetic.

This structure was described as a noncollinear arrangement of the Cu*A* and Cu*B* ion spins along the diagonals of the tetragonal plane of the crystal [7]. The magnetic moment of the Cu*A* ion was found to be about $1.3\mu_B$ at 12 K. Its component along the tetragonal *c* axis of the crystal was small, $\mu_z = 0.25\mu_B$, which corresponded to a 14° deviation from the *ab* plane. The Cu*B* ion spins lied in the *ab* plane and had a small magnetic moment of about $0.25\mu_B$ at $T = 12$ K. The magnetic moment of the Cu*B* ion rapidly increased as the temperature lowered below 10 K and equaled $0.7\mu_B$ at $T = 2$ K.

At temperatures below $T_B = 10$ K, two magnetic satellites situated symmetrically with respect to reciprocal lattice points of the commensurate phase appeared (Fig. 4). The magnetic structure of copper metaborate turned incommensurate along the tetragonal axis of the crystal and was described by a spin density wave with phase modulation [7]. The spin modulation period continuously increased from $\mathbf{q} \approx 0$ at 10 K to $\mathbf{q} = (0, 0, 0.15)$ at 1.8 K. At this temperature, spin structure modulation was characterized by a $c/0.15 \approx 40$ Å period. The temperature dependence of the wave vector of the incommensurate spin structure phase obeyed the power law

$$q(T) = A(T - T_B)^{0.48}. \quad (1)$$

As is shown in Fig. 5, Eq. (1) well described the temperature dependence of the incommensurate phase wave vector in the whole temperature range of measurements.

Wave vector **q** decreases virtually to zero as temperature increases, and the period of the incommensurate spin structure at T_B becomes large compared with the lattice parameter. In addition, strong diffuse neutron scattering is superimposed on the Bragg peaks for the $\mathbf{Q}_0$ neutron scattering vector along the [001] crystallographic direction. The intensity of diffuse scattering increases as temperature rises from 1.8 K and reaches a maximum close to T_B [7]. Diffuse scattering is observed even at the lowest temperature attained in experiments. This is sharply different from usual behavior of the spin subsystem of 3D-magnets with localized spins, for which critical fluctuations are limited by a narrow temperature region in the vicinity of the phase transitions. Adjustment of the magnetic structure at 2 K gives the best fit for a simple helix with a $0.7\mu_B$ for Cu*B*.

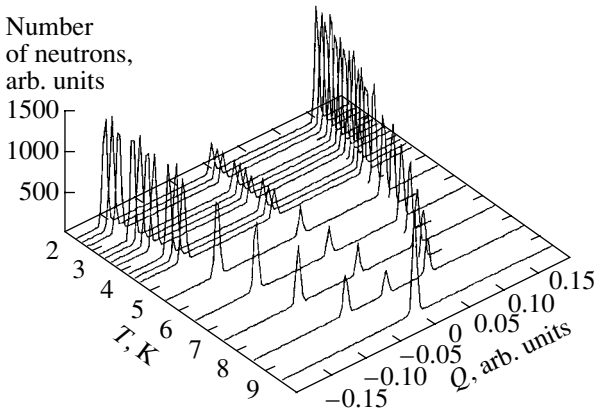

Fig. 4. Temperature dependence of propagation vector **q** = (3, 3, *Q*).

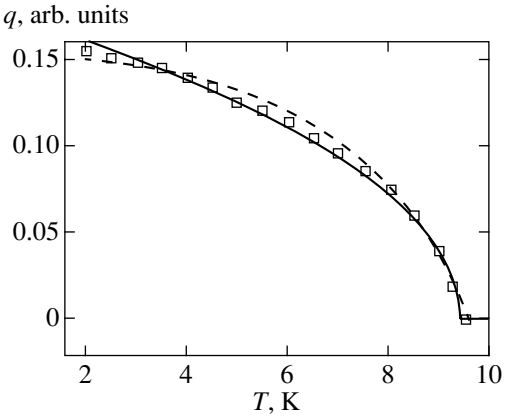

Fig. 5. Simulation of the temperature dependence of propagation vector *q* by Eqs. (1) (solid line) and (11) (dashed line).

3. DISCUSSION

The combined experimental data described above show that the spin subsystem of copper metaborate experiences rearrangements at 21 and 10 K. In addition, it may well be that one more rearrangement of the magnetic structure occurs near 1 K. In the commensurate phase, the magnetic structure is antiferromagnetic and possesses a spontaneous magnetic moment [6]. The magnetic and crystal chemical unit cells coincide, accordingly, the propagation vector **q** is zero. As the *I* symmetry operation of the lattice is also a magnetic transformation if $\mathbf{q} = 0$, the corresponding irreducible representations of the magnetic structure are the representations of the $\bar{4}2m$ point symmetry group. This group comprises eight symmetry elements and has five irreducible representations [9]. Four of these (Γ_1, Γ_2, Γ_3, and Γ_4) are one-dimensional, and one (Γ_5) is two-dimensional. The decomposition of representations gives

$$\Gamma_{4b} = \Gamma_3 + \Gamma_4 + 2\Gamma_5,$$

$$\Gamma_{8d} = \Gamma_1 + 2\Gamma_2 + \Gamma_3 + 2\Gamma_4 + 3\Gamma_5.$$

The Γ_3 and Γ_4 magnetic modes of site 4*b* correspond to collinear ferromagnetic and antiferromagnetic orderings along the *c* axis, respectively. The modes related to the Γ_5 representation describe a noncollinear magnetic structure. Similar magnetic modes for the 8*d* site can also be obtained from group theory. An analysis of the neutron diffraction pattern containing 25 purely mag-

netic peaks [7] shows that the magnetic structure of CuB_2O_4 copper metaborate can be described as a noncollinear arrangement of spins of both Cu*A* and Cu*B* along the diagonals of the tetragonal plane with out-of-plane displacements of Cu*A* magnetic moments. A symmetry analysis of the structure of copper metaborate shows that Cu*A* spins may be involved in Dzyaloshinskii–Moria interactions. Therefore, these interactions favor a noncollinear arrangement of spins, which is observed experimentally. In the incommensurate magnetic order region, helical ordering is observed [7].

It follows from the aforesaid that the whole spin system of copper metaborate comprises two subsystems, namely, subsystem *A* formed by Cu*A* ions, where the distribution of spins corresponds to a mixture of the Γ_4 and Γ_5 irreducible representations (axial antiferromagnetic and noncollinear planar configurations), and subsystem *B* formed by Cu*B* ion spins. The distribution of spins in this subsystem corresponds to the Γ_5 irreducible representation (a noncollinear planar configuration). It follows that, in writing the phenomenological thermodynamic potential of the spin system of copper metaborate, we must take into account two two-component order parameters corresponding to the Γ_5[Cu*A*] and Γ_5[Cu*B*] representations and one order parameter corresponding to the Γ_4[Cu*A*] representation. It is also important that crystal symmetry allows Lifshits invariants to be introduced for both subsystems.

Let the one-component order parameter be denoted by η, and two two-component order parameters, by (η_{A1}, η_{A2}) and (η_{B1}, η_{B2}) for subsystems *A* and *B*, respectively. The thermodynamic potential of the whole spin system can then be written as

$$\Phi = \int\left\{\frac{\alpha}{2}\eta^2 + \frac{\beta}{4}\eta^4 + \frac{\alpha_A}{2}\eta_A^2 + \frac{\beta_A}{4}\eta_A^4 + \frac{\gamma_A}{4}\eta_A^4\cos(4\varphi_A)\right.$$
$$+ \frac{\delta_A}{2}[(\nabla\eta_A)^2 + \eta_A^2(\nabla\varphi_A)^2] - \sigma_A\eta_A^2\varphi_A' + \frac{\alpha_B}{2}\eta_B^2 + \frac{\beta_B}{4}\eta_B^4$$
$$+ \frac{\gamma_B}{4}\eta_B^4\cos(4\varphi_B) \qquad (2)$$
$$+ \frac{\delta_B}{2}[(\nabla\eta_B)^2 + \eta_B^2(\nabla\varphi_A)^2] - \sigma_B\eta_B^2\varphi_B'$$
$$\left. + \kappa\eta_A\eta_B\cos(\varphi_A - \varphi_B) + \frac{\kappa_A}{2}\eta^2\eta_A^2 + \frac{\kappa_B}{2}\eta^2\eta_B^2\right\}dV,$$

where

$$\alpha_A = \alpha_{A0}(T - T_A), \quad \alpha_B = \alpha_{B0}(T - T_B),$$
$$\eta_A^2 = \eta_{A1}^2 + \eta_{A2}^2, \quad \eta_B^2 = \eta_{B1}^2 + \eta_{B2}^2,$$
$$\varphi_A = \arctan(\eta_{A2}/\eta_{A1}), \quad \varphi_B = \arctan(\eta_{B2}/\eta_{B1}),$$

$\alpha_{A0} > 0$, $\alpha_{B0} > 0$, $\beta_A > 0$, $\beta_B > 0$, $\delta_A > 0$, $\delta_B > 0$, ∇ is the nabla operator, and $f' \equiv df/dz$. The relation between subsystems *A* and *B* described by the invariant with coefficient κ in (2) results in the appearance of a helicoid in both subsystems at the same temperature.

The η one-component order parameter describes the axial component of Cu*A* spins and is of no importance for analyzing the incommensurate structure of the simple helix type with the helicoid axis along the tetragonal crystal axis. For this reason, thermodynamic potential (2) can be reduced to

$$\Phi = \int\left\{\frac{\alpha_A}{2}\eta_A^2 + \frac{\beta_A}{4}\eta_A^4 + \frac{\gamma_A}{4}\eta_A^4\cos(4\varphi_A)\right.$$
$$+ \frac{\delta_A}{2}[(\nabla\eta_A)^2 + \eta_A^2(\nabla\varphi_A)^2] - \sigma_A\eta_A^2\varphi_A'$$
$$+ \frac{\alpha_B}{2}\eta_B^2 + \frac{\beta_B}{4}\eta_B^4 + \frac{\gamma_B}{4}\eta_B^4\cos(4\varphi_B) \qquad (3)$$
$$+ \frac{\delta_B}{2}[(\nabla\eta_B)^2 + \eta_B^2(\nabla\varphi_A)^2] - \sigma_B\eta_B^2\varphi_B'$$
$$\left. + \kappa\eta_A\eta_B\cos(\varphi_A - \varphi_B)\right\}dV.$$

The Φ extremum conditions with respect to the order parameters corresponding to the equilibrium state of the system have the form

$$\frac{\delta\Phi}{\delta\eta_A} = \alpha_A\eta_A + \beta_A\eta_A^3 + \gamma_A\eta_A^3\cos(4\varphi_A) + \delta_A\eta_A(\nabla\varphi_A)^2 \qquad (4)$$
$$- 2\sigma_A\eta_A\varphi_A' - \delta_A\Delta\eta_A + \kappa\eta_B\cos(\varphi_A - \varphi_B) = 0,$$
$$\frac{\delta\Phi}{\delta\varphi_A} = -\gamma_A\eta_A^4\sin(4\varphi_A) - \delta_A\nabla(\eta_A^2\nabla\varphi_A) \qquad (5)$$
$$+ \sigma_A(\eta_A^2)' - \kappa\eta_A\eta_B\sin(\varphi_A - \varphi_B) = 0,$$
$$\frac{\delta\Phi}{\delta\eta_B} = \alpha_B\eta_B + \beta_B\eta_B^3 + \gamma_B\eta_B^3\cos(4\varphi_B) + \delta_B\eta_B(\nabla\varphi_B)^2 \qquad (6)$$
$$- 2\sigma_B\eta_B\varphi_B' - \delta_B\Delta\eta_B + \kappa\eta_A\cos(\varphi_A - \varphi_B) = 0,$$
$$\frac{\delta\Phi}{\delta\varphi_B} = -\gamma_B\eta_B^4\sin(4\varphi_B) - \delta_B\nabla(\eta_B^2\nabla\varphi_B) \qquad (7)$$
$$+ \sigma_B(\eta_B^2)' + \kappa\eta_A\eta_B\sin(\varphi_A - \varphi_B) = 0,$$

where Δ is the Laplase operator. As the perturbation of the homogeneous magnetic system state by the Lifshits invariant is one-dimensional, and deviations transverse to the *z* axis in the equilibrium state are excluded by positive δ_A and δ_B values, the nabla operator in these conditions should be replaced by single differentiation

with respect to z, and the Laplase operator, by double differentiation.

Finding the equilibrium state of the system from (4)–(7) is a complex problem. We will use the approximation of constant order parameter moduli, $\eta_A \neq \eta_A(z)$ and $\eta_B \neq \eta_B(z)$ [2]. As follows from (4) and (6), such an approximation is admissible if the Lifshits, anisotropy, and intersubsystem interaction invariants are small compared with the other invariants. As far as interaction between subsystems is concerned, it is only required that the dependence of the difference between helicoid phases in subsystems A and B on the z coordinate be negligibly small. In this approximation, thermodynamic potential (3) for the equilibrium state decomposes into two parts,

$$\Phi = \Phi_\eta + \Phi_\varphi,$$

$$\Phi_\eta = V\left\{\frac{\alpha_A}{2}\eta_A^2 + \frac{\beta_A}{4}\eta_A^4 + \frac{\alpha_B}{2}\eta_B^2 + \frac{\beta_B}{4}\eta_B^4 - |\kappa|\eta_A\eta_B\right\},$$

$$\Phi_\varphi = \int\left\{\frac{\gamma}{2}\cos(4\varphi) + \frac{\delta}{2}\varphi'^2 - \sigma\varphi'\right\}dV,$$

where $\varphi = \varphi_A = \varphi_B + \pi\theta(\kappa)$, $\theta(\kappa)$ is the Heaviside function,

$$\gamma = \gamma_A\eta_A^4 + \gamma_B\eta_B^4, \quad \delta = \delta_A\eta_A^2 + \delta_B\eta_B^2,$$

$$\sigma = \sigma_A\eta_A^2 + \sigma_B\eta_B^2.$$

Accordingly, the equilibrium conditions take the form

$$\begin{aligned}\alpha_A\eta_A + \beta_A\eta_A^3 - |\kappa|\eta_B &= 0,\\ \alpha_B\eta_B + \beta_B\eta_B^3 - |\kappa|\eta_A &= 0,\\ \delta\varphi'' + \gamma\sin(4\varphi) &= 0,\end{aligned} \tag{8}$$

where $f'' \equiv d^2f/dz^2$.

The first two equations of system (8) determine the temperature dependences of order parameter η_A and η_B moduli. Physically, the absence of phase $\varphi(z)$-dependent terms in these equations is justifiable, because η_A and η_B are almost fully determined by exchange interactions in the spin system of copper metaborate.

The third equation in (8) determines the dependence of the helicoid phase on the z coordinate along the tetragonal axis and on order parameter moduli. Its solution is the Jacobi amplitude function [10]

$$\varphi(z) = am(2q_0[z + \Delta z], k_1)/2, \tag{9}$$

where $q_0 = \sqrt{\gamma/\delta}/k_1$, and k_1 and Δz are the integration constants. The Δz constant corresponds to arbitrariness of selecting the origin along the tetragonal axis and is further set equal to zero. The k_1 constant is determined by the minimization of Φ_φ after substituting (9) into the equation for Φ_φ,

$$\begin{aligned}\Phi_\varphi &= \frac{V}{\lambda}\int_0^\lambda\left\{\frac{\gamma}{4}\cos(4\varphi) + \frac{\delta}{2}\varphi'^2 - \delta\varphi'\right\}dz\\ &= V\left\{\frac{4q_0\delta E(k_1) - 2\pi\sigma}{\lambda} + \frac{\gamma(1-2k_1^{-2})}{4}\right\},\end{aligned} \tag{10}$$

where

$$\lambda = \int_0^\lambda dz = \int_0^{2\pi}\frac{d\varphi}{|\varphi'|} = \frac{4K(k_1)}{q_0}, \tag{11}$$

is the helicoid period length, and

$$K(k_1) = \int_0^{\pi/2}\frac{du}{\sqrt{1 - k_1^2\sin^2(u)}}$$

and

$$E(k_1) = \int_0^{\pi/2}\sqrt{1 - k_1^2\sin^2(u)}\,du$$

are the total elliptical integrals of the first and second kind, respectively. The minimization of (10) with respect to k_1 gives

$$\frac{k_1}{E(k_1)} = \frac{2\sqrt{\gamma\delta}}{\pi\sigma}.$$

The corresponding Φ_φ value is

$$\Phi_\varphi = \frac{V\gamma(1-2k_1^{-2})}{4}.$$

Because of the absence of magnetic satellites in the spectra of inelastic neutron scattering on CuB_2O_4 in the temperature range 10–21 K, it can be suggested that $\sigma_A \ll \sigma_B$; therefore, σ_A can be ignored. The η_A order parameter at T_B is already not small, and anisotropy in subsystem A interferes with the appearance of a helicoid as a result of interaction between the subsystems. Therefore, the γ_A parameter is also assumed to be negligibly small in this work.

The relations obtained above allowed us to simulate the temperature dependences of η_A, η_B, and the $q = 2\pi/\lambda$ wave number and the temperature dependence of the specific heat capacity of the crystal, $C_p = -T\partial^2\Phi/\partial T^2$, with the use of the $C_p = -T\partial^2\Phi/\partial T^2$ relation for the following thermodynamic potential parameters (in kelvins):

$$\alpha_A = 1.6(T - 20), \quad \beta_A = 2.7, \quad \gamma_A = 0,$$

$$\delta_A = 0.16, \quad \sigma_A = 0, \quad \kappa = 0.01,$$

$$\alpha_B = 4(T - 9.6), \quad \beta_B = 2, \quad \gamma_B = 0.001,$$

$$\delta_B = 0.16, \quad \sigma_B = 0.1.$$

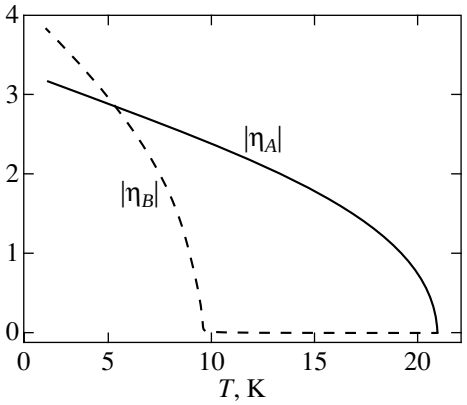

Fig. 6. Simulated temperature dependence of order parameter moduli.

Figure 6 shows that, in the temperature range 10–20 K, the η_B order parameter is small compared with η_A and rapidly increases at $T < 10$ K. A similar increase in the $q \equiv 2\pi/\lambda$ wave vector in Fig. 5 coincides with that observed experimentally (Figs. 4, 5) but qualitatively differs from the latter in that $q \neq 0$ already at temperatures below 20 K: in the temperature range of smallness of anisotropy invariants compared with the Lifshits invariants ($k_1 \ll 0$) we have

$$q \approx \sigma/\delta$$

and, at $\sigma_A = 0$, q variations are largely determined by η_B.

In addition to the jump at the transition point described by the Landau theory and the Ginzburg estimate of the contribution of order parameter thermal fluctuations, the contributions of acoustic phonons and Schottky-type anomalies were taken into account in heat capacity calculations. At 20 K, the fluctuation contribution characteristics of second-order phase transitions with a maximum at the transition point predominated. In the experimental curves (Fig. 3), the singularity at 9.6 K has the form of a step. This singularity is related to a rapid growth of the order parameter in the second spin subsystem. This parameter is already nonzero below 20 K because of bilinear interaction with the first subsystem. The field induced by this interaction suppresses thermal fluctuations in the second subsystem. The latter were therefore ignored in the calculations. Note that the step at 9.6 K is observed against the background of a broad maximum, which increases as temperature decreases. This maximum can be assigned to a Schottky-type anomaly that is not described by the phenomenological approach. The deviation of the experimental dependence from the calculation results at $T < 3.5$ K is caused by the approach to the supposed transition involving a low-temperature transformation of the magnetic structure near 1 K.

To summarize, we developed a phenomenological model of the magnetic subsystem of copper metaborate based on the experimental data on this compound. This model was applied to analyze the temperature dependence of the order parameter, the wave vector of magnetic structure modulation, and heat capacity. Further, we plan to study the properties of this crystal under strong magnetic field actions.

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