

³⁾We note that expression (17), which was derived here for an axially symmetrical inhomogeneity, remains valid, apart for a factor of the order of unity, also in the case of a planar inhomogeneity.

⁴⁾In the case opposite to (18), the heat intensity was calculated in ^[9].

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Neutron-diffraction investigation of the magnetic state of $Ni_{1-x}S$, $Ni_{1-x}S_{1-y}O_y$, and $Ni_{1-x}S_{1-z}Se_z$ in the metal-semiconductor phase transition region

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A neutron-diffraction investigation of the effect of oxygen and selenium on the variation of the magnetic state of hexagonal $Ni_{1-x}S$ has been carried out in the temperature range from 4.2 to 293 K, which includes the critical temperature of the phase transition from the antiferromagnetic to the paramagnetic state. It is found that the doping elements do not change the magnetic structure and the magnitude of the magnetic moment of the nickel ions. From the independence of the diffuse scattering of temperature it is concluded that, in the metallic state, all the solid solutions are Pauli paramagnets.

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The existence of correlation between the nature of the variation of the binding forces in a number of 3d-transition metal sulfides, selenides, and tellurides and the type of conduction of these compounds, which was pointed out in Refs. 1 and 2, has led the authors of these papers to infer the possibility of a directional variation of the electrophysical properties through the variation of the relation between the ionic and homopolar components of the binding forces. The latter is realized by doping these compounds with anions with electronegativity different from the electronegativity of

the anions of the original compound. It was assumed that the variation of the difference between the mean electronegativities of the anions and cations can significantly change the degree of ionicity of the phases, on which quantity clearly depends the degree of localization of the valence electrons.

Experiments with pure and doped $Ni_{1-x}S$, in which the intermediate character of the electrophysical properties manifests itself most clearly, ^[1,2] confirmed the correctness of the stated hypothesis. It was shown

that the partial substitution of sulfur in Ni_{1-x}S by the more electronegative oxygen leads to the stabilization of the semiconducting state, i. e., the transition into the metallic state occurs at a higher temperature than in the undoped sulfide, while the activation energy for the intrinsic conductivity in the semiconducting phase increases. On the other hand, the substitution of sulfur in Ni_{1-x}S by the less electronegative selenium leads to the lowering of the critical temperature of the transition into the metallic state and to a decrease in the activation energy for the intrinsic conductivity of the semiconducting state.

These results were obtained on the basis of an analysis of the behavior of the temperature dependence of the thermo-e. m. f. coefficient $\alpha(T)$ and the magnetic susceptibility $\chi(T)$ of the doped and undoped nickel sulfides. It was also found that a change in the vacancy concentration in the metal sublattice of the original sulfide can lead to a significant change in the dependence of the magnetic susceptibility on the concentration of the doping elements.^[3] The necessity for a careful neutron-diffraction investigation of pure nickel sulfide and of the sulfide doped with the metalloids oxygen and selenium was demonstrated in this connection. Investigations of the scattering of neutrons on these objects should help resolve the question of the influence of doping on the magnetic parameters—the magnetostructural characteristics—connected with the electrophysical properties, and provide additional information about the nature of the magnetic state in the metallic phase. It can be expected that such experiments will also facilitate the elucidation of the nature of the semiconductor-metal phase transition, about which opinions differ in the literature.

Sample preparation and the experimental procedure

Using the technology described in Ref. 1, we prepared samples in the form of powder and with the compositions indicated in Table I. The measurements were performed on a neutron-diffraction apparatus located in one of the horizontal channels of the IVV-2 reactor. The wavelength, λ , of the neutrons was 1.075 Å. The sample was poured into a vanadium container with an inside diameter of 8 mm and a height of 50 mm. The angular distribution of the scattered neutrons was studied at room, liquid-nitrogen, and liquid-helium temperatures. Intermediate temperatures in the 293–77-K range were obtained in a liquid-nitrogen cryostat with a special heater located between the container and the

TABLE I. Concentration dependence of the magnetic moment of the nickel ion.

Sample number	Composition	Magnetic moment of Ni^{2+} (in μ_B) at 4.2 K
1	NiS	1.2±0.1
2	$\text{Ni}_{0.985}\text{S}$	1.2±0.1
3	$\text{Ni}_{0.96}\text{S}$	0.0±0.1
4	$\text{Ni}_{0.985}\text{S}_{0.995}\text{O}_{0.005}$	1.2±0.1
5	$\text{Ni}_{0.985}\text{S}_{0.985}\text{O}_{0.015}$	1.2±0.1
6	$\text{Ni}_{0.96}\text{S}_{0.975}\text{O}_{0.025}$	1.0±0.1
7	$\text{Ni}_{0.985}\text{S}_{0.98}\text{Se}_{0.02}$	1.0±0.1
8	$\text{Ni}_{0.985}\text{S}_{0.98}\text{Se}_{0.02}$	0.0±0.1
9	$\text{Ni}_{0.96}\text{S}_{0.97}\text{Se}_{0.03}$	0.0±0.1

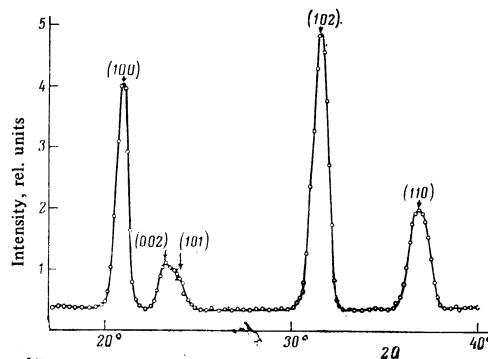


FIG. 1. Neutron-diffraction pattern for $(\text{Ni}_{0.985}\text{S})_{0.995}(\text{NiO})_{0.005}$ at a temperature of 293 K.

point where the container was fixed to the bottom part of the nitrogen tank of the cryostat. A copper-constantan and a copper-iron-copper thermocouple were used. The form factor value for the computation of the magnetic moment was taken from Ref. 4.

RESULTS OF THE INVESTIGATIONS

For all the nine samples we obtained neutron-scattering patterns in the range of the angle 2θ from 0 to 45° at the three temperatures (293; 77; 4.2 K). Figure 1 shows the neutron-diffraction pattern for the sample $(\text{Ni}_{0.985}\text{S})_{0.995}(\text{NiO})_{0.005}$. The neutron-diffraction patterns for all the rest of the samples have the same form at room temperature. In the indicated angle range they contain (100), (002), (101), (102), and (110) reflections. The relative intensities of these reflections are in good agreement with the computed values, obtained under the assumption that all the investigated compounds have the NiAs-type structure, that the vacancies in the cation sublattice and the doping atoms in the anion sublattice are distributed statistically, and that the magnetic moment of nickel is equal to zero (the neutron scattering amplitudes are: $b_{\text{Ni}^{2+}} = 1.03C$, $b_{\text{S}^{2-}} = 0.78C$, $b_{\text{O}^{2-}} = 0.27C$, and $b_0 = 0.575C$, where $C = 10^{-12}$ cm/atom^[5]). At room temperature coherent scattering, which would have indicated the presence of magnetic long-range order, was absent in all the compounds. The unit cell of pure Ni_{1-x}S coincides with the unit cells of the doped compounds, but in the latter case the (1/3, 2/3, 1/4) and (2/3, 1/3, 3/4) sites can be occupied by oxygen or selenium with a probability proportional to their concentration in the compound.

Figure 2 shows the neutron-diffraction pattern, measured at 4.2 K, for the compound $(\text{Ni}_{0.985}\text{S})_{0.995}(\text{NiO})_{0.005}$. (similar neutron-diffraction patterns are obtainable at 4.2 and 80 K for the compounds 1, 2, 4–7, whose compositions are indicated in Table I.) This neutron-diffraction pattern differs from the one shown in Fig. 1 only in the magnitude of the (101) reflection, which is roughly two times more intense than in the first case. The most likely cause of the appearance of the additional scattering is, apparently, the appearance in the material of long-range magnetic order connected with magnetic moments localized on the nickel ions.^[1] It is precisely such a conclusion that was arrived at by Sparks and Komoto^[7] as a result of a

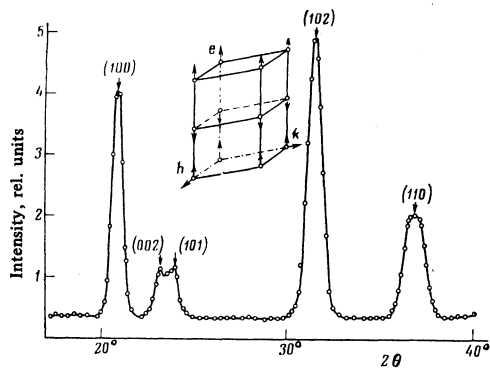


FIG. 2. Neutron-diffraction pattern for $(\text{Ni}_{0.985}\text{S})_{0.995}(\text{NiO})_{0.005}$ at a temperature of 4.2 K.

neutron-diffraction investigation of pure nickel sulfide. Indeed, the pattern of scattering from the magnetic structure, whose unit cell is shown in Fig. 2, should contain only the (101) magnetic reflection, which coincides in position with the (101) nuclear reflection. A calculation shows that diffraction on such a magnetic structure should give another (111) magnetic reflection, but it is located in the $38\text{--}42^\circ$ angle range, where the form factor for the nickel ion is small, in consequence of which the reflection does not show up.

Since the compounds 1, 2, 4–7 (see Table I) give identical magnetic-diffraction patterns, we can assume that the same type of antiferromagnetic ordering exists in these compounds at low temperatures. The (002) and (101) reflections are poorly resolved in the neutron-diffraction patterns. Therefore, the magnetic component of the (101) reflection was determined as the difference between the total intensities of the (002) and (101) reflections measured at 4.2 and 293 K. In Table I we give the values, computed from these data, of the magnetic moment of the nickel ion. As the reference reflection, we chose the (100) pure nuclear reflection. The expression for the determination of the magnetic moment of the nickel ion has the form

$$\mu_{\text{eff}}(\text{Ni}^{2+}) = [I_{\text{n.e.}}^{(101)} I_{\text{n.c.}}^{(100)} P_{(101)} / (I_{\text{n.e.}}^{(100)} \bar{q}_{(101)}^2 j_{(101)})]^{1/2} \times [0.2695 \cdot 10^{-12} F(\lambda^{-1} \sin \theta_{(101)})]^{-1},$$

where $F(\lambda^{-1} \sin \theta_{(101)})$ is the magnetic-scattering form factor of the nickel ion for the (101) reflection, $j_{(101)}$ is the repetition factor for the (101) plane, $\bar{q}_{(101)}^2$ is the mean value of the square of the magnetic-interaction vector for the (101) plane, $I_{\text{n.e.}}^{(101)}$ is the experimental value of the intensity of the (101) magnetic reflection, $I_{\text{n.e.}}^{(100)}$ is the experimental value of the intensity of the (100) nuclear reflection, $I_{\text{n.c.}}^{(100)}$ is the computed value of the intensity of the (100) nuclear reflection, and $P_{(101)}$ is the integratedness factor for the (101) reflection.

For the compounds 1, 2, 4–7 we obtained virtually the same values for the magnetic moments of the nickel ion (see Table I). A similar result is obtained in the computation of the moment for the undoped nickel sulfide from Sparks and Komoto's experimental data, although these authors give a significantly different value: $1.6 \mu_B$. The disagreement is due, apparently, to

the use of form factors taken from different sources.

A detailed investigation of the temperature dependence of the intensity of the (101) magnetic reflection was carried out on the compounds 1, 2, 4–7 in the temperature range 80–300 K. In the case of some of the compounds the region of investigation was extended down to 4.2 K. Typical curves are shown in Fig. 3. It can be seen that doping with selenium shifts the temperature at which magnetic scattering appears toward by oxygen leads to the elevation of this temperature. In this case the width of the interval in which the magnetic transition, which manifests itself in a sharp change in the intensity of the magnetic coherent scattering, occurs depends on the concentration of the doping component.

A comparison of the $I_{\text{n.e.}}^{(101)}(T)$ curves with the dependences $\rho^{-1}(T)$, $\alpha(T)$, and $\chi(T)$ obtained in Refs. 1 and 2 shows that, in both pure nickel sulfide and nickel sulfide doped with metalloids, magnetic scattering arises and increases, as the temperature is lowered, simultaneously with the transition to semiconducting properties. It can be asserted that the antiferromagnetism and the semiconducting properties are most clearly related here and are, apparently, interconditional.

Indeed, the compounds $(\text{Ni}_{0.985}\text{S})_{0.9}\text{Se}_{0.2}$ and $(\text{Ni}_{0.98}\text{S})_{0.97}\text{Se}_{0.03}$, for example, do not give magnetic scattering right down to 4.2 K. But, in accordance with the foregoing, this was to be expected, since, according to the data of Ref. 1, the metal-nonmetal transition does not occur in these compounds; they possess metallic conductivity in the entire investigated temperature region. Another example: the doping with oxygen of the sulfide of composition $\text{Ni}_{0.98}\text{S}$, in which the metal-semiconductor phase transition does not occur and there are no signs of long-range magnetic order, leads to the simultaneous appearance in the low-temperature region of semiconducting conductivity and antiferromagnetic properties.

Thus, the low-temperature semiconducting phase is always antiferromagnetic, irrespective of the composition of the compound. A change in the content of the doping component affects in like manner the boundaries of the existence regions for the antiferromagnetism and the semiconducting properties, both transitions—the

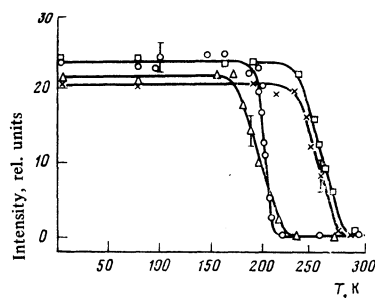


FIG. 3. Temperature dependence of the intensity of the (101) magnetic reflection for the compounds: \circ $\text{Ni}_{0.985}\text{S}$; \times $(\text{Ni}_{0.98}\text{S})_{0.975}(\text{NiO})_{0.025}$; Δ $(\text{Ni}_{0.985}\text{S})_{0.98}\text{Se}_{0.02}$; \square $(\text{Ni}_{0.985}\text{S})_{0.995}(\text{NiO})_{0.005}$.

electric and magnetic—always occurring simultaneously. It can be seen from Fig. 3 that, in the antiferromagnetic region, the intensity of the (101) reflection for the compound 1, 2, 4–7 does not significantly depend on temperature. Rough estimates of the Néel temperature from the temperature dependence of the intensity of the (101) reflection in this region yield a value lying within the limits 700–800 K. This result agrees with the computed Néel-point value obtained for pure NiS.^[8] The obtained experimental data do not reveal any influence of the doping components on the character of the temperature dependence of the intensity of the magnetic reflection in the antiferromagnetic region (Fig. 3).

Thus, the behavior of the (101) reflection indicates a weak dependence of the magnetization of the cation sublattice on temperature in the antiferromagnetic state.

In the region of variation of the conductivity mechanism, as the temperature is raised, there occurs an abrupt decrease of the intensity of the (101) magnetic reflection down to the complete disappearance of the reflection at the point of transition of the material into the metallic state (Fig. 3). The corresponding change in the sublattice magnetization can be explained in two different ways:

- 1) as a result of a change in the exchange interactions, the substance goes over into the paramagnetic state with disordered magnetic moments;
- 2) the magnetic moments localized on the nickel ions disappear completely during the transition (Pauli paramagnetism).

The transition of the material with long-range magnetic order into the paramagnetic, disordered state should have inevitably been accompanied by the appearance of incoherent diffuse neutron scattering, the intensity of which is connected with the magnetic moment of the scatterer by the well-known relation

$$d\sigma/d\Omega = \frac{2}{3} S(S+1) (r_0 \gamma)^2 F^2(\lambda^{-1} \sin \theta),$$

where $d\sigma/d\Omega$ is the cross section for paramagnetic scattering, S is the effective spin quantum number of the nickel ion, r_0 is the electromagnetic radius of the electron, γ is the magnitude of the magnetic moment of the neutron in nuclear magnetons, and $F(\lambda^{-1} \sin \theta)$ is the magnetic form factor of the nickel ion.^[5]

On the basis of the magnitude of the magnetic mo-

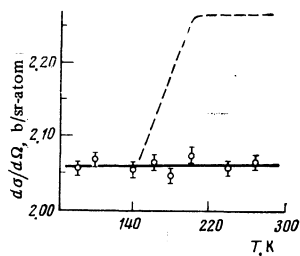
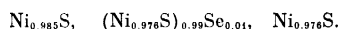


FIG. 4. Temperature dependence of the diffuse scattering (background) in the $\text{Ni}_{0.976}\text{S}$ sample ($2\theta = 10^\circ$).

ment on the nickel ion ($1.2 \mu_B$), determined by us for the magnetically ordered state, an increase of 0.265 b/sr-atom was to be expected in the scattering cross section upon the transition of the material from the semiconducting to the metallic state on account of the paramagnetic component (if we assume that the magnitude of the moment is conserved).

We investigated the temperature dependence of the diffuse, incoherent scattering in a temperature range encompassing the semiconductor-metal phase transition on three samples of the following compositions:



The neutron counter was set in a position corresponding to the angle $2\theta = 10^\circ$. As a standard we used a vanadium sample.

The exposure chosen in the measurement at each point enabled us to reliably establish a change in the cross section $d\sigma/d\Omega$ of 0.016 b/sr-atom, which would correspond to a magnetic moment of $0.3 \mu_B$ for the paramagnetic scatterer. In Fig. 4 we present the measurement results for the $\text{Ni}_{0.976}\text{S}$ sample. It can be seen that the background has the same magnitude to the left, as to the right, of the region of the semiconductor-metal phase transition, which, in the alloy under consideration, occurs in the temperature range 140–200 K. The dashed curve in the same figure represents the computed diffuse-scattering intensity versus temperature curve, obtained under the assumption that the magnitude of the magnetic moment localized on the nickel ion remains unchanged after the transition into the paramagnetic state. Similar results were obtained by us in the investigation of nickel sulfide doped with oxygen or selenium. Thus, these measurements demonstrate conclusively that, in the metal phase, there is no incoherent magnetic scattering (i. e., no paramagnetic diffuse background), which implies the absence of localized magnetic moments on the nickel ions. Consequently, in the transition region, as the temperature is raised, there occurs a change in the electronic structure, including the disappearance of the localized magnetic moments. This conclusion is in accord with Sparks and Komoto's conclusion,^[9,10] which was drawn on the basis of only an analysis of the angular dependence of the background in the diffraction pattern of NiS. In view of the necessity to make manifest the small paramagnetic-scattering effect ($\mu \approx 1 \mu_B$) in a large incoherent-nuclear-scattering background, it was necessary to carry out additional experiments based on a different procedural approach that ensured substantially higher sensitivity and measurement accuracy.

Thus, it can be regarded as conclusively established that, in the metallic state, pure nickel sulfide and nickel sulfide doped with metalloids are Pauli paramagnets. This conclusion agrees with the data obtained in susceptibility measurements performed on the compounds under consideration and with the results obtained in a study of the Mössbauer effect in pure nickel sulfide.^[11]

¹Doubt has been expressed in the literature about the existence of localized moments in the low-temperature phase of NiS. However a detailed thermodynamic analysis, carried out by Polovov *et al.*,⁶¹ attests to their existence.

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Absorption of sound in helium II

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A theory is developed describing the propagation of high-frequency sound in helium II at low temperatures ($T < 0.6$ K) and high pressures ($P > 16$ atm), when the energy spectrum of the phonons becomes stable. The absorption coefficient and the sound dispersion are calculated under these conditions. The dependence of the velocity of second sound on the frequency is determined. The resonance properties of the obtained solution are discussed.

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INTRODUCTION

There has recently appeared a large number of works devoted to the study of kinetic phenomena in helium II at low ($T < 0.6$ K) temperatures (see the review of Ref. 1). Interest in this region has been raised by the assumption of Maris and Massey^[2] that the phonon portion of the dispersion curve in He II can be unstable, i. e., the dependence of the phonon energy ε on the momentum p has the form

$$\varepsilon = cp(1 + \gamma p^2 - \delta p^4) \quad (1)$$

(c is the velocity of sound, $\gamma, \delta > 0$).

Three-phonon processes become possible in such dispersion, and the effect of these processes on the kinetics and propagation of sound has been studied by Maris.^[3] One of the results of this investigation has been the conclusion that, in addition to the ordinary second sound in He II at low temperatures, propagation of "new" sounds is possible. Also, it turned out that the velocity of ordinary second sound increases upon increase in the frequency, from the hydrodynamic value $c/\sqrt{3}$ to c . The qualitative explanation of this phenomenon has been given by Maris within the framework of the theory of Mandel'shtam-Leontovich for sound propagation in relaxing media.

The appearance of "new" sounds, i. e., weakly

damped solutions of the dispersion equation, has been connected with the emergence of "quasiconserved" quantities, i. e., quantities which undergo almost no change within a time of the order of the period of the sound wave.

Such a situation was possible because $\omega t_{\parallel} \ll 1$ in the equilibrium regime (ω is the sound frequency, t_{\parallel} is the time of establishment of equilibrium for phonons moving in a single direction) the transition from the region of hydrodynamics $\omega t_2 \ll 1$ (t_2 is the relaxation time of the second harmonic of the distribution function, expanded in Legendre polynomials) to the high frequency region $\omega t_2 \gg 1$ is described not by a single relaxation t_2 , but by a whole series of times t_i , each of which corresponds to the relaxation of the harmonic $P_l(\cos\theta)$. For small l , a dependence $t_i^{-1} \sim l^4$ was obtained in Refs. 3-5. Therefore, with increase in frequency a situation becomes possible in which some of the harmonics are "conserved," and for them $\omega t_i \gg 1$, while others are in the hydrodynamic regime $\omega t_i \ll 1$.

Kinetic phenomena in a gas of phonons for the case of four-phonon processes (under the assumption that $\gamma < 0$ in (1)) was considered by Khalatnikov and Chernikova.^[6] They assumed that the transition from the hydrodynamic region to the high-frequency region is described by a single relaxation time τ_2 .

In the present paper it will be shown that even in the