survey elemental analysis of the initial fullerite C_{60} and of the samples subjected to high pressures and temperatures has shown that the general content of magnetic impurities in these materials does not exceed 0.003 at. %. According to the estimates done in Ref. [12], such an impurity content leads to a saturation magnetization value 30 times smaller than that observed in the experiment. It was also noted that pieces of ferromagnetic polymer samples that had undergone depolymerization as a result of heating to temperatures above 650 K completely give up their ferromagnetic properties. It was these two facts and the fact that the appearance of ferromagnetic states has been observed only in the isobaric cross section corresponding to the transformation of the rhombohedral phase and only within a narrow temperature range (outside this range such states have never been observed) that forced the researchers to search for an explanation of the ferromagnetism in the very nature of these high-pressure states. In Ref. [12], which summarized the results of the first stage of the investigations, it was suggested that the appearance of constant magnetic moments in the system is related to the presence of topological defects in the polymer C_{60} layers, which results in some of the electrons becoming unpaired. There it was also noted that only samples fabricated at the limit of thermal stability of the rhombohedral phase exhibited pronounced magnetic properties. Thus, although according to the results of X-ray phase analysis the rhombohedral phase is predominant in the ferromagnetic samples, the detected magnetic effects are, to all appearances, related to the deformation and partial destruction of C_{60} clusters, since in samples of a purely rhombohedral phase fabricated at lower temperatures, 873 or 973 K, ferromagnetic properties have never been observed. For the same reason, a defect structure based on the rhombohedral phase is, strictly speaking, not the only candidate for the role of the state that determines the ferromagnetic behavior of the samples. Other possible candidates are various ferromagnetic structures based on sp^2 and sp^3 hybridized states of carbon such as those proposed by Ovchinnikov and Shamovsky [14]. According to calculations of the band structure of some carbon materials and the related isostructural materials that are formed in a three-component C – B – N system, magnetically ordered states can also arise when nanometer-sized fragments of graphene planes with 'zigzag' edges appear in the system [15, 16].

At the same time it is obvious that until the origin of the ferromagnetic behavior of samples obtained by subjecting fullerite C_{60} to high pressures has been established beyond all doubt, the possibility cannot be excluded that the observed phenomena are related to an uncontrolled inclusion of metallic impurities in the samples. The probability of such a situation occurring is finite, since we are speaking of monitoring an impurity content that amounts to several thousandths of one percent, which requires using special technological methods corresponding, in essence, to the requirements adopted in the semiconductor industry. This, strictly speaking, was not done in the process of producing the different high-pressure states.

At present, in order to independently verify the results of Makarova et al. [12], we are carrying out a detailed investigation of a new series of ferromagnetic samples together with researchers in a number of laboratories in Russia and in foreign countries. The results of these investigations show, among other things, that there is a significant inhomogeneity in the distribution of the ferromagnetic component over a sample, which is probably related to the gradients in the distribution of pressure and temperature that exist in modern high-pressure chambers. The presence of such inhomogeneities opens up the possibility to take a new look at the results of the measurements of spin concentrations in magnetic samples obtained by Makarova et al. [12]. Previously, the value of the spin concentration (5×10^{18} cm^{-3}), determined as the concentration averaged over the entire sample and corresponding to roughly one spin per three hundred C_{60} molecules, could hardly be explained within the scope of the usual notions about the structure of the exchange interaction, since it implied that the average distance between separate spins is extremely large. If inhomogeneities are present, the local spin concentration in the ferromagnetic regions of the sample may really be much higher than the average value.

The discovery of inhomogeneities in magnetic samples determined the main area of research in this field — local methods of studying the samples. We believe that in conjunction with cutting the samples into separate fragments and separating these fragments magnetically, with the purpose of extracting the ferromagnetic fragments in a more concentrated form, these studies will make it possible to determine without a doubt the nature of the emerging magnetically ordered states and will provide the final answer to a question that always arises in studies of the magnetic properties of organic or carbon compounds: is the observed ferromagnetism merely an artifact or is it indeed related to the specific features of the structure of pure carbon states?

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Local magnetic states and hyperfine interactions in magnetic superlattices and spin-tunnel junctions

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We have carried out a comprehensive study of the structure and magnetic and electrical properties of, and hyperfine magnetic fields in, the magnetic Fe/Al, Fe/Ti, Fe/Ta, Fe/Pd and Fe/Mo superlattices (MSLs) and in spin-tunnel junctions based on half-metallic Ni–Mn–Sb Heusler alloys. In MSLs we observed intense oscillations of the magnetic parameters caused by variations in the thicknesses of the nonmagnetic layers with a period of 8–10 Å, which may be due to variations in the indirect exchange interaction. In some MSLs the spontaneous magnetization was found to be much higher than that in bulk iron. In Ni–Mn–Sb alloys in which systems of oriented microcracks were produced, huge tunneling magnetoresistance was found, as high as 230% at 35 K in a 50 Oe magnetic field.

1. Magnetic superlattices comprise one of the most studied and, from the practical viewpoint, important classes of nanometer-sized heterostructure magnetic systems. Researchers have been focusing on problems associated with the technical applications of these materials (primarily, the problem of giant magnetoresistance, or GMR), while, from the fundamental viewpoint, focusing on the problem of oscillations of the main magnetic parameters caused by variations in the thicknesses of the MSL layers and on finding a physical explanation for these phenomena. MSL studies have revealed far less information about the various features of the local states of magnetic ions in MSLs, although the theory demonstrated [1] that there are considerable variations in the local atomic moments of magnetically active ions and the corresponding hyperfine magnetic fields at the nuclei of these ions.

Spin tunneling junctions constitute the second important class of magnetic nanometer-size heterostructure systems. Quantum tunneling in such junctions creates the possibility for GMR, especially if one uses half-metallic ferromagnets, in which the spin polarization of delocalized electrons may be very high.

Here, we present the results of experimental studies of the structure and the magnetic properties of, and hyperfine fields at, ^{57}Fe nuclei in Fe/Al, Fe/Ti, Fe/Pd, Fe/Ta, and Fe/Mo MSLs and of studies of the magnetic properties, nuclear spin echo, and transport and magnetotransport properties of spin-tunnel junctions based on half-metallic Ni–Mn–Sb Heusler alloys.

2. To synthesize MSLs, we developed a new method involving a discharge with oscillating electrons. The magnetic properties were measured by a fully computerized vibrating-sample magnetometer with a sensitivity of 2×10^{-6} emu, and the Mössbauer spectra on ^{57}Fe nuclei were measured by an automatic spectrometer with a ^{57}Co source in Rh in the absorption mode. To measure the nuclear spin-echo spectra, we used a spectrometer operating in the 70–700 MHz range in hyperfine magnetic fields.

Our studies of the crystalline structure of MSLs by X-ray diffraction methods revealed that at the minimum layer thicknesses (6–9 Å), only the interfaces corresponding to the components involved exist, and that as the layers become thicker, reflections corresponding to bulk components and their satellites caused by a superlattice-type periodicity emerge.

The magnetic hysteresis curves of the MSLs suggest a rich variety of magnetization reversal processes in these systems and the possible existence of indirect exchange interactions of an antiferromagnetic type between the layers. For instance, in the case of Fe/Ta MSLs, we observed hysteresis loops characteristic of metamagnetic phase transitions, while in the case of Fe/Al MSLs, we observed a 'reversed' hysteresis loop when the magnetizing field was perpendicular to the MSL plane, meaning that, as the field strength is reduced within a certain range, the magnetic susceptibility changes sign and becomes negative. We also observed a substantial magnetic anisotropy in the film plane, related to the orientation of the magnetic field during film deposition.

Figure 1 shows the hysteresis loop for an [Fe(7 Å)/Ti(8.4 Å)] × 400 MSL: (a) the full loop; (b) the central part of the loop. The loop is highly constricted, which suggests that for given Fe/Ti MSL layer thicknesses the indirect exchange between the magnetic layers is antiferromagnetic. We calculated the main magnetic parameters of these MSLs, with the values of the spontaneous magnetization calculated by the law of approaching saturation. Figure 2 depicts the dependence of the saturation magnetization and the coercive force of an Fe/Ti MSL on the Ti-layer thickness. Especially evident were the pronounced oscillations of these parameters. For instance, for Fe/Al MSLs, a change of one monolayer (~ 3 Å) in the Al-layer thickness changes the spontaneous magnetization by more than two orders of magnitude. The period of oscillation caused by variations in the thicknesses of the nonmagnetic layers amounts to 8–10 Å, which may be due to the oscillatory nature of the indirect exchange interaction between the ferromagnetic layers and allows interpretation in terms of the RKKY model (to name only one possible interpretation).

In some MSLs, e.g., in Fe/Pd, oscillations of the magnetic parameters were also observed when the Fe-layer thickness was changed. In this case, however, it is improbable that the oscillations of the magnetic parameters are caused by

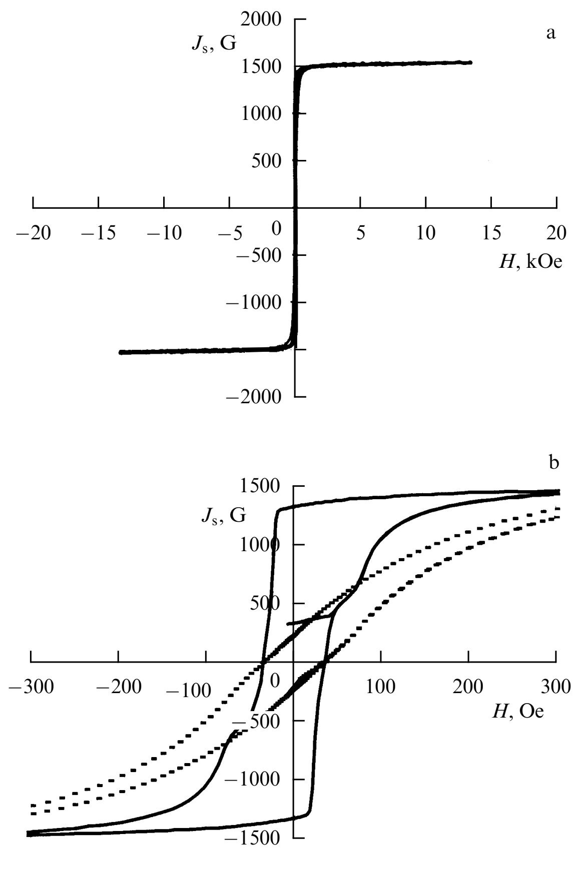

Figure 1. Hysteresis loop for an [Fe(7 Å)/Ti(8.4 Å)] × 400 MSL with an external field parallel to the film plane: (a) hysteresis loops in the field of maximum strength; (b) the central part of the same loops.

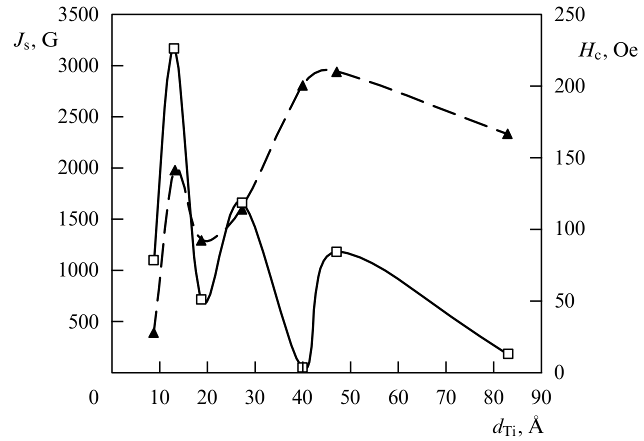

Figure 2. Spontaneous magnetization J_s (□) and coercive force H_c (▲) of an Fe/Ti MSL as functions of the Ti-layer thickness. The Fe- layer thickness is ~6 Å.

variations in the indirect exchange integrals. More plausible is the idea of the importance of interference effects that emerge when the electron waves are reflected from the interfaces in the MSL and lead to the formation of quantum wells. The oscillation period in these cases was ~10 Å.

The high values of J_s normalized to iron content, which were observed in some MSLs in the Fe/Ti, Fe/Ta, and Fe/Mo systems, attracted much interest. In some cases these values exceed the value for pure bulk iron by a factor greater than three. The possible physical reasons for such a large increase in J_s are the increase in the spin moments of Fe ions when these ions are in low-dimension states in near-surface layers and interfaces, the contribution to J_s from the polarization of the delocalized electrons in the Ti, Ta, and Mo layers, and the increase in the atomic magnetic moments of iron due to the increase in orbital contributions. The last factor may be the dominating one. For instance, studies of the anomalous Hall effect have shown [2] that for Fe and Co atoms at the surface or in the bulk of a Cs film the local atomic magnetic moments amount to $7\,\mu_B$ and $8\,\mu_B$, respectively. First-principles theoretical calculations [3, 4] have shown that, with allowance for the intraatomic orbital polarization [5], the orbital contributions to the atomic magnetic moments of Fe and Co on the surface or in the bulk of the alkali metals K, Cs, and Rb may amount to $2.2-3.0\,\mu_B$. For the Ag and Au hosts, these contributions may amount to $2.2-2.5\,\mu_B$. These results also suggest the presence of substantial orbital contributions to J_s in the case of Fe/Ti, Fe/Ta, and Fe/Mo MSLs. High values of J_s were also observed in Fe/Pd MSLs, but in this case it is probably the contribution from the polarization of delocalized electrons with a large effective mass in Pd layers that plays the main role.

To study the local magnetic states of iron ions in MSLs, we measured the Mössbauer spectra on ^{57}Fe nuclei and, on the basis of this study, restored the distribution function for the hyperfine magnetic fields at these nuclei, $P(H_{hf})$. Figure 3 shows the Mössbauer spectrum measured in the transmission mode for an $[\mathrm{Fe}(5.2\ \text{Å})/\mathrm{Ti}(47\ \text{Å})] \times 550$ MSL at room temperature (Fig. 3a) and the corresponding distribution function $P(H_{hf})$ (Fig. 3b).

As is seen from the distribution function $P(H_{hf})$ curve, the probability peaks for the hyperfine fields are located within three groups corresponding to three nonequivalent local

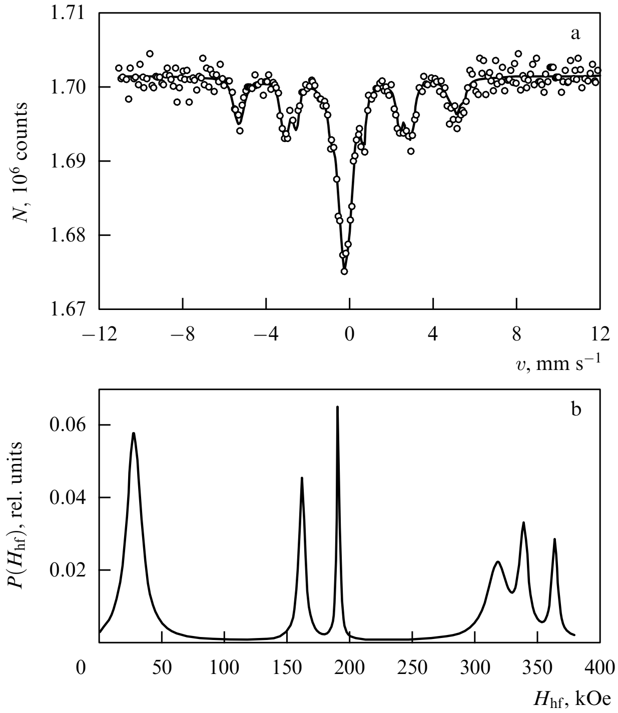

Figure 3. (a) Mössbauer spectrum for an $[\mathrm{Fe}(5.2\ \text{Å})/\mathrm{Ti}(47\ \text{Å})] \times 550$ MSL. (b) Distribution function for the probability density H_{hf} of the same MSL.

states of Fe ions. For one of these groups, the peak is at $H_{hf} = 30$ kOe, which suggests that in the given local states the iron ions have no intrinsic magnetic moment and that the value of H_{hf} is due to the polarization of the electron spin density on ^{57}Fe nuclei caused by the surrounding ions. For these states, the fact that Ti ions are the nearest neighbors is probably crucial. The group of peaks near the highest possible value of H_{hf} (up to 380 kOe) is due to Fe ions with maximum values of spin magnetic moments in near-surface regions and to Fe ions that have other Fe ions as nearest neighbors. Finally, the group of peaks at intermediate values of H_{hf} (150 – 200 kOe) probably corresponds to the states of Fe ions in interfaces with mixed surroundings consisting of Fe and Ti ions.

The results of our Mössbauer studies involving Fe/Ti MSLs agree well with those of measurements of the temperature dependence of the magnetization of these films in weak ac magnetic fields. The anomalies observed in the curves may be explained by the magnetic transitions of the Fe ions that are in the appropriate local states.

In the case of Fe/Pd MSLs, the distribution functions $P(H_{hf})$ have two peaks. The iron layers in these MSLs have an fcc structure and, accordingly, the observed peaks may be associated with the low- and high-spin states of the Fe ions in the fcc lattice.

3. Spin-tunnel junctions represent a system of two ferromagnetic electrodes separated by an insulating layer. Devices based on the materials in question have a magnetoresistance $[R(H)-R(0)]/R(0)$ that is usually no higher than 50%, and, often, to achieve such values one needs low temperatures and strong magnetic fields (several dozen kilooersteds). The mechanisms of formation of giant magnetoresistance are different for different heterostructures, but in each heterostructure, especially in spin-tunnel junctions, the degree of spin polarization of the conduction electrons plays a major role. In accordance with this, the use of half-metallic ferromagnets may prove a promising way of reaching maximum magnetoresistance [among these ferromagnets are some Heusler alloys with a stoichiometric composition XYZ and a crystal lattice of the $C1_b$ (MgAgAs) type]. As is known, theoretical calculations of the electronic structure of these ferromagnetic alloys [6] have shown that one of the exchange-split subbands in the alloys is of a typical metallic nature, while the second subband is of a typical semiconducting nature, with a gap near the Fermi level. This feature of the electronic structure of half-metallic ferromagnets determines the high degree of spin polarization of the conduction electrons (theoretically, up to 100%) in the metallic subband.

In our report, we present the results of a comprehensive study of half-metallic Ni – Mn – Sb Heusler alloys with small variations in the component concentrations with respect to the stoichiometric composition XYZ. The samples were fabricated through melting in an electric arc furnace in an atmosphere of purified argon with repeated remeltings followed by homogenization. X-ray studies of the structure revealed that the resulting alloys with the cubic lattice of the MgAgAs ($C1_b$) type were single-phase. We measured the magnetization curves and the hysteresis loops of the alloys with a vibrating-sample magnetometer at 300 – 1100 K in 15 kOe magnetic fields and also with a SQUID magnetometer at 5 – 300 K in ± 30 kOe magnetic fields. Measurements of the electrical resistance and the magnetoresistance were carried out at 5 – 300 K in magnetic fields of up to ± 30 kOe by the four-probe method. The spectra of nuclear spin echo on ^{55}Mn, ^{121}Sb, and ^{123}Sb nuclei were measured in hyperfine fields in a zero external field at 5 – 80 K in the 160 – 700 MHz frequency range.

Figure 4 shows the hysteresis loops of the stoichiometric alloy NiMnSb measured at 5 and 300 K. A characteristic feature of these loops is a virtually linear increase in the magnetization in the initial stage of the increase in the magnetic field strength and a steep kink at magnetic field strengths of roughly 1 kOe. Saturation is achieved at $H \approx 4.5$ kOe practically without paraprocess. This shape of the hysteresis loop suggests that rotation processes play the dominant role in the magnetization and magnetization reversal of the alloys. The saturation magnetic induction B_s at 5 K amounted to 90 emu g^{-1}. Note the small values of the remanent induction B_r and the coercive force H_c, which are 0.4 emu g^{-1} and 1 Oe, respectively. These characteristic features of the hysteresis loops of the half-metallic alloy NiMnSb do not change with variations in the composition with respect to the stoichiometric composition XYZ and when Sb ions are partially replaced by Sn ions.

The nuclear spin-echo spectrum for the NiMnSb alloy measured at 5 K in the 150 – 400-MHz range, where the main resonant-absorption regions are located, is shown in Fig. 5. The spectrum contains three characteristic resonance lines: a diffuse resonance band extending from 270 to 350 MHz attributed to ^{121}Sb nuclei, a broad resonance line in the 160 – 170 MHz range attributed to ^{123}Sb nuclei, and a sharp peak at 297.2 MHz attributed to ^{55}Mn nuclei of Mn ions at the Y sites of the $C1_b$ lattice. A characteristic feature of these crystallographic positions of the Mn ions is that in an atomically ordered state they have no magnetically active ions among their nearest atomic neighbors. Only in the third coordination shell is the Mn ion at a Y site surrounded by

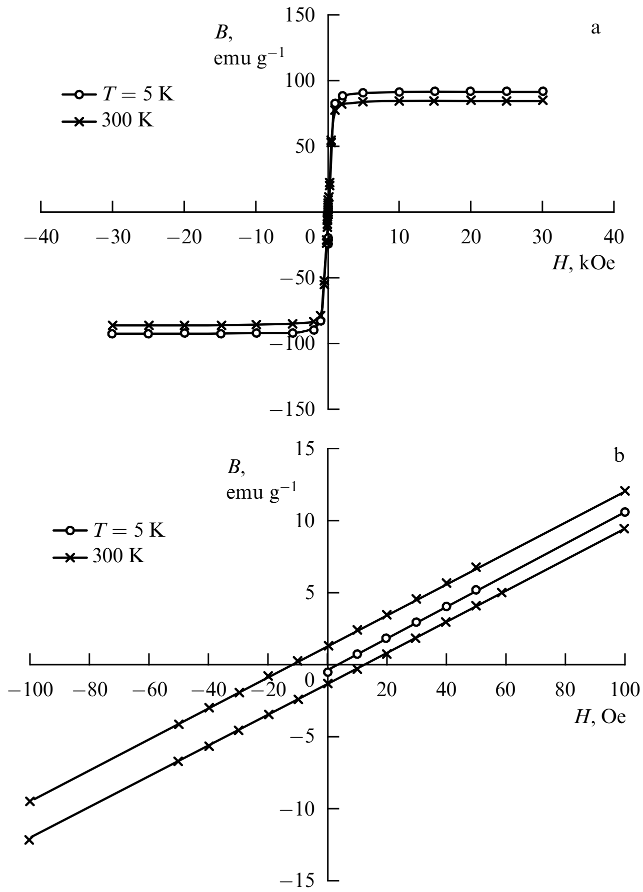

Figure 4. Hysteresis loop for the NiMnSb alloy: (a) the full loop and (b) the central part of the loop.

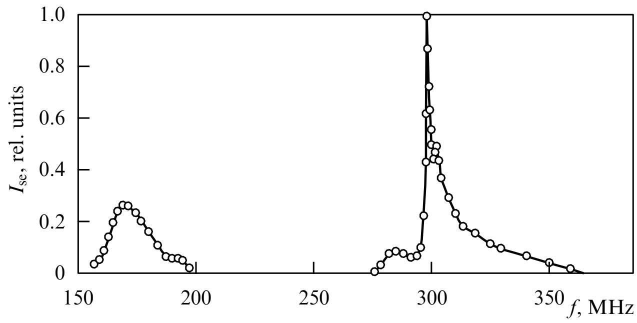

Figure 5. Nuclear spin-echo spectrum for the half-metallic NiMnSb Heusler alloy.

12 identical Mn ions at other Y sites. Hence, in accordance with the given structure, the hyperfine magnetic field at the ^{55}Mn nuclei at Y sites is formed primarily by the spin density localized within the given ion, and variations in the alloy composition near the stoichiometric composition XYZ do not alter the resonance frequency of this peak, while the high intensity and small width of this peak suggest a high degree of atomic ordering in the arrangement of the Mn ions among the Y sites of the $C1_b$ lattice.

In addition to this, variations in the alloy composition lead to significant changes in the intensities and positions of the resonance lines attributed to ^{121}Sb and ^{123}Sb nuclei, for which the hyperfine magnetic fields are formed by the spin density of the delocalized electrons polarized by the nearest-neighbor magnetic ions. This indicates a high degree of polarization of the delocalized electrons, which is characteristic of the half-metallic state of the NiMnSb alloy.

The temperature dependence of the electrical resistivity of the Ni–Mn–Sb alloy was measured at 30–300 K. A characteristic feature of the alloys in the initial state is a high electrical conductivity and a normal temperature dependence of electrical resistivity of the metallic type. The electrical resistivity was found to increase from 760 μΩ cm at 35 K to 1100 μΩ cm at 300 K.

We also measured the magnetoresistance of Ni–Mn–Sb alloys of different concentrations near the stoichiometric composition XYZ at different temperatures in magnetic fields of up to 40 kOe. The measurements were done both for parallel and perpendicular orientations of the external magnetic field with respect to the direction of the current. In the initial state, the value of $\Delta R/R$ at 35 K did not exceed 0.015; with variations in the alloy composition, the magnetoresistance does not exceed this value.

Our studies also involved the fabrication in Ni–Mn–Sb alloys of prototypes of spin-tunnel junctions, to which end we treated the alloys in a special way to produce in the sample one, two, or more microcracks with certain orientations with respect to the direction of the current. As a result of the treatment, the electrical and magnetic transport properties of the samples changed significantly. The electrical resistivity of the stoichiometric NiMnSb alloy at 50 K increased by a factor of greater than 50, while its temperature dependence took on a semiconducting nature. As the temperature increased from 30 to 300 K, the electrical resistivity of the sample dropped from 10^4 to 2.5×10^3 μΩ cm. The current–voltage curve of the NiMnSb alloy at 46 K has a characteristic nonlinear shape.

The most striking changes were found in the magnetoresistance of Ni–Mn–Sb alloys: after treatment, the value of $\Delta R/R$ changed by several orders of magnitude. For instance, for the $Ni_{30.6}Mn_{33.9}Sb_{20.6}Sn_{14.9}$ alloy the value of $\Delta R/R$ at 35 K amounted to 6%. The strongest effect was observed in the stoichiometric alloy NiMnSb after three parallel microcracks were formed in the alloy as a result of the above-mentioned treatment. Figure 6 shows the dependence of the magnetoresistance of this alloy at 35 K on the external magnetic field. The maximum value of $\Delta R/R$ was achieved in a weak magnetic field (50 Oe) and amounted to 230%.

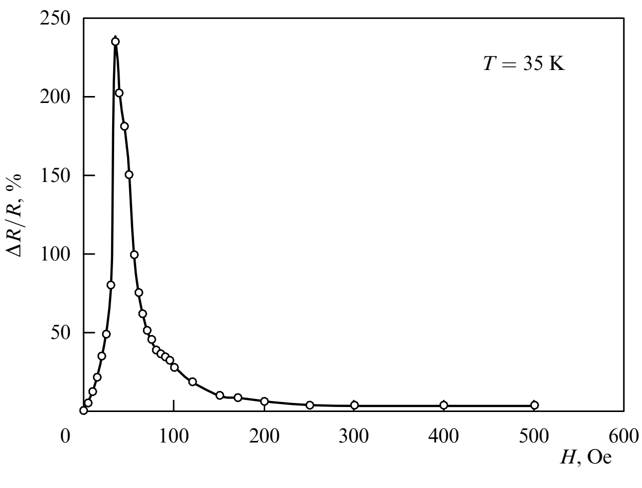

Figure 6. Magnetoresistance $\Delta R/R$ of the half-metallic NiMnSb Heusler alloy plotted against the magnetic field strength at 35 K.

To give a physical interpretation of this result, one must bear in mind that the state of the sample of the alloy with three parallel microcracks passing through the entire sample and oriented perpendicular to the direction of the current is analogous to three series-connected spin-tunnel junctions. The cleavage surface is very smooth, with only a slight roughness, and the air gaps in the microcracks play the role of insulating layers in spin-tunnel junctions. Mathon [7] theoretically calculated the electrical conductivity σ for quantum tunneling between Co electrodes through a vacuum gap. The calculations were based on numerical estimates made by the Kubo–Landauer method [8] using the band structure of Co in the tight-binding approximation. The calculations also made it possible to model the vacuum gap between the Co electrodes by using hopping matrices in the tight-binding approximation. This model was shown to lead to the same values of tunneling magnetoresistance as the model of quantum tunneling through a barrier with a strong insulator. The dependence of the spin polarization of the tunneling electrons $P = (\sigma^{\uparrow} - \sigma^{\downarrow})/(\sigma^{\uparrow} + \sigma^{\downarrow})$ on the width of the vacuum gap (expressed in terms of the number of reciprocal hopping parameters) was constructed, and the calculated value of the tunneling magnetoresistance was found to be $\Delta R/R = 65\%$. In our case, such high values of $\Delta R/R$ are probably caused by the high degree of polarization of the tunneling electrons in the half-metallic ferromagnetic alloy NiMnSb, which makes the spin-tunnel junctions highly effective, and by the above-noted special features of the magnetic hysteresis loops of this alloy, namely, by the small values of coercive force and remanent induction, at which the state of the sample with microcracks is fairly stable. The very fact that in our case the change in orientation of magnetization in the contact pair of the NiMnSb electrodes occurs in a weak magnetic field (50 Oe at 35 K) can be explained by the anisotropic shape of these electrodes: one is shaped like a parallelepiped with its major axis directed along the magnetizing field, and the other is a thin plate oriented perpendicularly to the magnetizing field (this plate has a large demagnetizing factor). In accordance with this, as the external field becomes stronger, the long electrode with the small demagnetizing factor is the first to undergo magnetization (or magnetization reversal), and only then the planar electrode with a high demagnetizing factor undergoes magnetization (or magnetization reversal). This explains the presence of a sharp and relatively narrow peak in the dependence of the magnetoresistance on the external magnetic field strength of the NiMnSb alloy at 35 K.

Our results open up the possibility of making extensive use of new magnetic materials — half-metallic ferromagnetic Heusler alloys — in devices of spin electronics with record values of tunneling magnetoresistance.

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