Reentrant phenomena in Invar $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys

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Cooling of $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys with alloying element concentrations close to the critical value for the appearance of the ferromagnetic order produced the following sequence of magnetic transitions: paramagnet–collinear ferromagnet–canted ferromagnet (asperomagnet)–reentrant spin glass. The Invar alloy $Fe_{65}Ni_{35}$ exhibited an asperomagnetic state at temperatures below 20 K.

1. INTRODUCTION

It is now firmly established that in the case of fcc Invar FeNi alloys the interaction between the iron atoms separated by the shortest distances is antiferromagnetic (AFM) whereas the nearest-neighbor Fe–Ni and Ni–Ni interactions are ferromagnetic (FM).[1,2] In other words, these alloys belong to a class of systems with competing exchange interactions. It is therefore necessary to determine their ground magnetic state. However, the available experimental results are contradictory. For example, it is reported in Refs. 3 and 4 that Invar FeNi alloys exhibit the long-range AFM order at $T = 4.2$ K. However, neutron-diffraction investigations of a single crystal of $Fe_{65}Ni_{35}$ have failed to confirm the long-range AFM order.[5] Other authors (see, for example, the review in Ref. 6) are of the opinion that the low-temperature magnetic structure of Invar FeNi alloys can be regarded as collinear ferromagnets with randomly distributed AFM regions of dimensions of the order of several lattice constants. Finally, the authors of Ref. 7 report observation of a reentrant temperature-induced ferromagnetic–spin glass (FM–SS) transition in Invar FeNi alloys at temperatures $T \lesssim 30$ K. However, the occurrence of a reentrant spin glass state in these alloys is rejected in Ref. 8.

It follows from this account that there is as yet no generally accepted point of view on the nature of the ground magnetic state of Invar FeNi alloys.

Our aim was to investigate reentrant temperature-induced PM–FM–SS transitions (PM stands for a ferromagnet) in alloys corresponding to the quasibinary $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ tie-line, which includes also the classical Invar alloy $Fe_{65}Ni_{35}$, and to determine the nature of their low-temperature magnetic state.

2. EXPERIMENTAL METHOD

We used cylindrical samples with the height-to-diameter ratio of ~ 10, which were quenched from 1200 K in water before measurements. The static magnetization was determined in the temperature range 4.2–300 K using a vibrating-sample magnetometer. The temperature dependences of the spontaneous magnetization were obtained by the kink method[9] and by the Arrott–Belov method.[10] In both cases the results were qualitatively similar.

The real (χ_0') and imaginary (χ_0'') parts of the dynamic magnetic susceptibility in magnetization-reversing fields $h_0 = 0.3$–10.0 Oe were determined at temperatures 1.4–300 K using apparatus described in Ref. 11. The vertical component of the magnetic field of the earth was compensated to within $\pm 10\%$.

3. EXPERIMENTAL RESULTS AND DISCUSSION

We shall first consider the FM alloys with the alloying element concentrations close to the critical $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ corresponding to the appearance of the long-range ferromagnetic order.

3.1. Temperature dependence of the spontaneous magnetization

Figure 1 shows, by way of example, the temperature dependence of the spontaneous magnetization I_s of the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloy with $x = 0.2$ and it demonstrates that the spontaneous moment (which is the FM order parameter) appeared at the Curie point $T_c \approx 165$ K and it increased as a result of cooling. However, at a certain temperature $T_A < T_c$ the $I_s(T)$ curve deviated from the quasi-Brillouin dependence (dashed curve) toward lower values. This effect was particularly noticeable in the alloy with $x = 0.2$, which was closest to the critical concentration x_0. Enhancement of the FM exchange (reduction in x) weakened this anomaly so that, for example, it was no longer observed (within the limits of the experimental error) at $x = 0.14$.

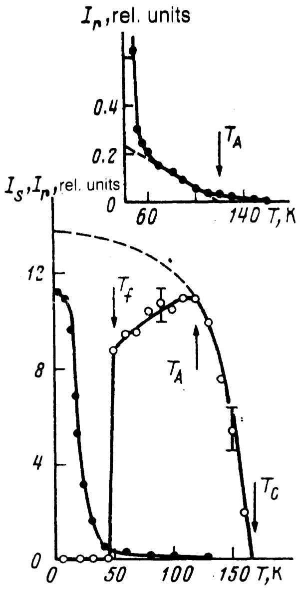

FIG. 1. Temperature dependences of the spontaneous I_s (open circles) and thermoremanent I_r (black dots) magnetizations of the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloy with $x = 0.2$.

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It is natural to attribute this effect, predicted in Ref. 12, to the establishment of a noncollinear FM (asperomagnetic) state in the alloys. Let us assume that in the FM state (at temperatures $T_A < T < T_c$) the direction of the spontaneous magnetization vector $\mathbf{I}_s$ coincides with the z axis. The transition to the asperomagnetic (ASM) state results in the appearance of randomly frozen x and y spin projections,[12] which is equivalent to an effective reduction in the z projection $\mathbf{I}_s(T)$. This is precisely the situation which occurs in reality (Fig. 1), since cooling of the alloy below T_A makes the $I_s(T)$ curve "lag" the quasi-Brillouin dependence.

We found that stronger cooling of this alloy reduced steeply the spontaneous magnetization to zero at $T_f = 50$ K (Fig. 1). Hence, the alloy assumed the reentrant SS state in which there was no long-range ferromagnetic order. This conclusion was confirmed by the results of recent investigations of low-angle neutron scattering reported for alloys of the same class, but with somewhat different compositions and x close to x_0 (Ref. 13). It should also be stressed that this result conflicted with the predictions of the molecular field theory for Heisenberg disordered ferromagnets[14,15] according to which a degenerate SS coexists with the long-range ferromagnetic order at temperatures $T < T_f$.

It therefore follows that the experimental results plotted in Fig. 1 demonstrate that, firstly, at temperatures $T_A < T_c$ there is a change in the state of a disordered ferromagnet which can be treated as a transition from the collinear to the noncollinear FM (ASM) state. Secondly, at even lower temperatures T_f there is a phase transition to the reentrant SS state accompanied by the loss of the long-range magnetic order. Additional arguments in support of these conclusions will be given below.

3.2. Thermoremanent magnetizations and magnetization-reversal loops

In addition to the spontaneous magnetization, Fig. 1 shows the temperature dependence of the thermoremanent magnetization I_r of the alloy under consideration. It follows from the results obtained that below T_f the dependence $I_r(T)$ is extremely steep. This is evidence of the appearance of strong longitudinal irreversibilities at $T < T_f$. At higher temperatures $T_f < R < T_A$ we observed clearly weaker longitudinal irreversibilities (inset in Fig. 1).

This behavior of $I_r(T)$ is in qualitative agreement with the predictions of the replica theory of reentrant FM–SS transitions,[14,15] according to which the Gabay–Toulouse line (the line labeled T_A in the x–T plane) represents random freezing of the spin components transverse relative to the direction of $\mathbf{I}_s$. The replica symmetry is lost along this line, i.e., a nonergodic "transverse" SS appears and is characterized by the presence of strong transverse irreversibilities. This can be identified as the asperomagnetic state. Moreover, according to Refs. 14 and 15, freezing of the longitudinal components of the spins occurs at lower temperatures in the region of T_f and this gives rise to strong longitudinal irreversibilities.

It should be stressed that when the longitudinal magnetization of an isotropic Heisenberg ferromagnet is determined, it is fundamentally impossible to record the transverse irreversibilities. Moreover, in this case a finite transverse order parameter of a spin glass $q^{\perp}$ cannot coexist with $I_s \neq 0$ (Ref. 16), which is a consequence of an invariance of the exchange Hamiltonian under rotation. However, the presence of anisotropic interactions which cause breaking of the macroscopic symmetry of the system has the effect that the transverse fluctuations of the magnetic moment do not destroy the ASM state.[16] For example, the existence of a random anisotropy results in the "interaction" of the transverse $q^{\perp}$ and longitudinal $q^{\parallel}$ order parameters of a spin glass and this makes it possible to detect a weak longitudinal irreversibility of the magnetization which originates from strong transverse irreversibilities. It follows that the temperature dependences of the thermoremanent magnetization given in Fig. 1 confirm the hypothesis of the appearance, in the investigated alloy, of the ASM state in the investigated range $T_f < T < T_c$ and of the reentrant spin glass state at $T < T_f$.

The Dzyaloshinskiĭ–Moriya anisotropy may play an important role in metallic systems: it is due to the spin–orbit interaction of a pair of spins $\mathbf{S}_1$ and $\mathbf{S}_2$ with the coordinates $\mathbf{R}_1$ and $\mathbf{R}_2$, via a third atom located at the origin of the coordinate system.[17] The energy $H_{\rm DM}$ of such an interaction can be represented in the form

$$H_{DM} = -\mathbf{D}(\mathbf{R}_1, \mathbf{R}_2)(\mathbf{S}_1, \mathbf{S}_2), \tag{1}$$

where $\mathbf{D}(\mathbf{R}_1, \mathbf{R}_2)$ is a constant.

The interaction described by Eq. (1) gives rise to a macroscopic anisotropy of unidirectional nature[18] and manifested by a shift of a hysteresis loop relative to the origin in the direction of negative magnetic fields after cooling of the system in a magnetic field. It is clear from Eq. (1) that the Dzyaloshinskiĭ–Moriya anisotropy does not appear in a collinear ferromagnet, but it should be manifested only in those magnetic structures in which spins are rotated relative to one another, i.e., in spin glass and ASM states. It follows from the above that the investigation of magnetization reversal curves can provide information on the nature of the magnetic phases and states which appear in the course of cooling of a disordered ferromagnet.

Figure 2 shows the magnetization-reversal curves (hysteresis loops) of the alloy $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ with $x = 0.2$ after it was cooled in a magnetic field from temperatures $T > T_c$ to three temperatures $T_A < T = 150$ K $< T_c$, T_f

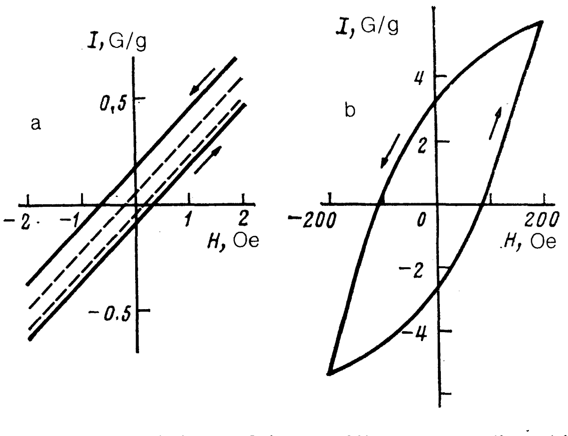

FIG. 2. Hysteresis loops of the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloy with $x = 0.2$ after cooling to a given temperature in a magnetic field: a) $T = 65$ K (continuous curve), $T = 150$ K (dashed curve); b) $T = 20$ K.

$< T = 65$ K $< T_A$, and $T = 20$ K $< T_f$; clearly, at 150 K the hysteresis loop was symmetric relative to the origin of the coordinate system (dashed line). Hence, we concluded that at this temperature the magnetic state of the investigated alloy was collinear FM. At a lower temperature (65 K) the hysteresis loop was shifted relative to the origin (continuous curve) indicating the existence of a canted (noncollinear) ASM state. It should also be stressed that the relative shift of the hysteresis loop along the x axis increased as a result of cooling. According to Eq. (1), this means that the angle of rotation of the spins relative to the initial direction of the spontaneous magnetization vector increased as a result of cooling.

At temperatures $T < T_f = 50$ K the hysteresis loop became considerably broader and its shift was much greater than in the ASM state (Fig. 2). This confirmed the conclusion of the appearance of an isotropic SS state with no long-range ferromagnetic order at temperatures $T < T_f$.

3.3. Dynamic magnetic susceptibility and low-angle neutron scattering

Figure 3 shows the temperature dependences of the real (χ_0') and imaginary (χ_0'') components of the dynamic magnetic susceptibility of the alloy $Fe_{65}(Ni_{1-x})Mn_{35}$ with $x = 0.2$, obtained in magnetization-reversing fields of different intensities h_0. The dependence χ_0' (T) exhibited two sharp anomalies very typical of the systems undergoing the reentrant PM–FM–SS transition. The high-temperature rise of χ_0' (T), limited by the rise of the demagnetization factor of the investigated sample, was associated with the appearance of the long-range ferromagnetic order in the alloy, whereas the steep low-temperature fall was due to the transition to the reentrants SS state (see above).

The behavior of χ_0'' (T) was more complex (Fig. 3). Near $T_c = 168$ K there was a sharp maximum of $\chi_0''(T)$ reflecting the development of critical FM fluctuations near T_c as a result of the PM–FM transition. At lower temperatures ($T_f = 54$ K) the anomaly of χ_0'' (T) was associated with the transition to the SS phase. A very important feature was that at temperatures $T_f < T < T_c$ there was one further anomaly of $\chi_0''(T)$ and, in contrast to T_c and T_f, the temperature T_A of the intermediate anomaly χ_0'' (T) depended strongly on the intensity of the magnetization-reversing field (Fig. 3).

These features of the temperature dependences of χ_0' (T) and χ_0'' (T) of the investigated alloy were very typical also of other crystalline [$Fe_xNi_{90-x}Cr_{20}$, $(Pd_{1-x}Fe_x)_{95}Mn_5$ (Ref. 19), $Au_{100-x}Fe_x$ (Ref. 20)] and amorphous [$(Fe_x Mn_{1-x})_{75}B_6P_{16}Al_3$ (Ref. 21)] systems exhibiting reentrant temperature-induced PM–FM–SS transitions. Possible reasons for the anomaly of χ_0'' at the temperature T_A and for the strong dependence of T_A on h_0 were considered in Ref. 20 using a model[22–24] of reentrant temperature-induced FM–SS transitions. According to this model, at temperatures $T_A < T_c$ the FM matrix exhibits ASM fluctuations with an effective size amounting to several lattice constants and these fluctuations appear near frustrated sites, i.e., near the atoms coupled to the nearest neighbors not only by the FM but also by the AFM interactions. Cooling increases the angle of rotation of the spins relative to the direction of the spontaneous mag-

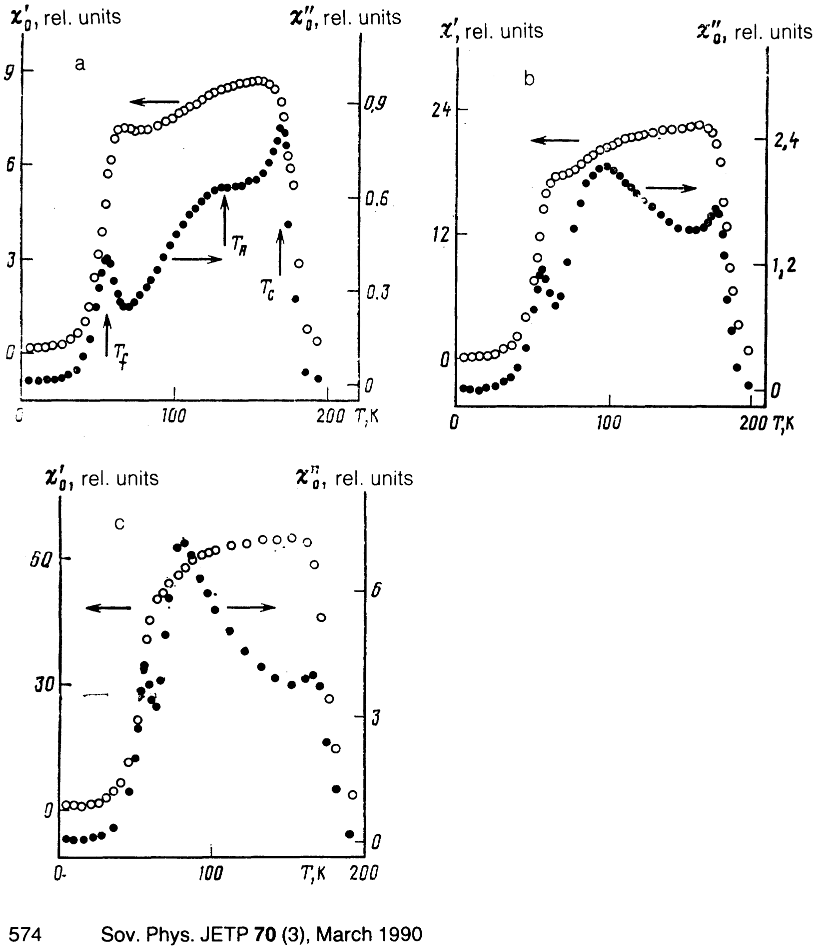

FIG. 3. Temperature dependences of the real (χ_0') and imaginary (χ_0'' components of the dynamic magnetic susceptibility of the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloy with $x = 0.2$ in magnetization-reversing fields of different intensities h_0: a) 0.3 Oe; b) 1.0 Oe; c) 3.0 Oe. Measurements were made at a frequency of 60 Hz.

netization within the limits of ASM fluctuations and at temperatures $T_f < T_A$ the SS state becomes frozen and this is accompanied by an abrupt reduction in I_s (Ref. 22).

Therefore, in accordance with Refs. 22–24, a canted (ASM) spin structure of local nature appears in a disordered frustrated ferromagnet at temperatures $T_f < T < T_c$. Nevertheless, the whole FM system can be described in this state by a single order parameter (noncollinearity or canting parameter)[22]

$$Q_{nc} = \Big[(2/N^2) \sum_{ij} |(\langle S_i \rangle \langle S_j \rangle)|^2 \Big]^{1/4}, \qquad (2)$$

where N is the number of the magnetic atoms and $\langle S_i \rangle$ is the average spin of the ith atom. Obviously, the noncollinearity parameter of the FM phase is $Q_{nc} = 0$, whereas in the case of the ASM phase we have $Q_{nc} \neq 0$ and the parameter rises as a result of cooling, assuming its highest values in the reentrant SS state. It follows from the above that the appearance of local ASM fluctuations in these alloys can be regarded formally as a transition of the whole alloy to the ASM state, which is characterized by the order parameter Q_{nc}. The appearance of this state is evidence of anomalies of χ_0'' at temperatures $T_f < T_A < T_c$ (Ref. 20).

It should be stressed once again that, in contrast to T_c and T_f, the value of T_A depends very strongly on the intensity of the magnetization-reversing field (Fig. 3). Therefore, the temperature T_A obtained in the lowest magnetization-reversing field, in our case $h_0 = 0.3$ Oe, is closest to the true temperature of the appearance of the ASM state.

We shall conclude by considering the dynamic magnetic susceptibility and note that the proposed model describes all the features of the temperature dependences (Fig. 1) and the magnetization-reversal processes in the investigated alloy at various temperatures (Fig. 2).

We shall now consider the temperature dependences of the cross section of low-angle neutron scattering (LANS) in $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys. Near the Curie temperature T_c there was a critical scattering maximum whereas at lower temperatures the LANS intensity increased (subcritical scattering).[25] This indicated that the magnetic inhomogeneities appeared in the FM matrix of the alloys. In fact, the LANS cross section $d\sigma/d\Omega$ of an FM material can be represented in the form[26]

$$d\sigma/d\Omega \propto M^2 C(1-C)|\delta M|^2, \qquad (3)$$

where C is the concentration of magnetic inhomogeneities and δM is the deviation of the density of the magnetic moment M from its average over a sample. It is clear from Eq. (3) that in the case of an FM sample where cooling induces magnetic inhomogeneities within which, for example, the magnetic moment is lowered ($|\delta M|^2 \neq 0$), we can expect subcritical neutron scattering.

We can easily see that this model of the magnetic structure of a frustrated ferromagnet accounts qualitatively for the subcritical scattering in $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys reported in Ref. 25. In fact, in this case the ASM fluctuations, in which the density of the magnetic moment is less than in the surrounding FM matrix, act as magnetic inhomogeneities. Cooling of a sample increases the angle of rotation of the spins within ASM fluctuations and this is equivalent to an increase in $|\delta M|^2$. Consequently, the temperature at which subcritical neutron scattering appears in a ferromagnet as a result of cooling is the temperature of the formation of the ASM state. It is important to note that in the case of $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys this temperature agrees well with the values of T_A obtained above from the temperature dependences of I_s, I_r, and χ_0'' (Sec. 3.5).

3.4. Asperomagnetism of Invar Fe_xNi_{100-x} alloys

We shall now consider the classical Invar alloy $Fe_{65}Ni_{35}$ which belongs to the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ system of interest to us. Figure 4 shows the temperature dependences of the real (χ_0') and imaginary (χ_0'') components of the dynamic magnetic susceptibility. It is clear from the results obtained that, as in the case discussed in the preceding section, there is a kink in the dependence $\chi_0'(T)$ and an ASM maximum of $\chi_0''(T)$ at a temperature which depends strongly on h_0. It is important to note that, in contrast to ternary $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys (Fig. 3), in the case of $Fe_{65}Ni_{35}$ there is no spin-glass anomaly of $\chi_0''(T)$ right down to $T = 1.4$ K. Hence, it follows that the ground magnetic state of the Invar alloy $Fe_{65}Ni_{35}$ cannot be the reentrant SS state and even less so the AFM state. The latter can be explained by an insufficient number of the AFM bonds compared with the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys characterized by $x \neq 0$, where (in addition to the interactions between the Fe–Fe nearest neighbors) also the exchange Mn–Mn interactions are negative.

It thus follows from our experimental results that the ground (at $T \approx 0$ K) magnetic state of the binary Invar $Fe_{65}Ni_{35}$ alloy is the ASM state. It should be stressed that the asperomagnetism is typical also of Invar FeNi alloys of other compositions for which the fcc structure is stable right down to $T = 0$ K (Ref. 27).

It follows from the ideas put forward above that the asperomagnetism of Invar FeNi alloys can account for other low-temperature properties. In particular, we can easily explain the appearance of the low-temperature anomalies of the high-field[28] and low-field static[6] and dynamic[7] magnetic susceptibilities, as well as the occurrence of the subcritical neutron scattering,[29] unidirectional anisotropy,[30] etc.

3.5. Magnetic phase diagram

The results of our investigation can be used to construct the magnetic phase diagram of $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys in

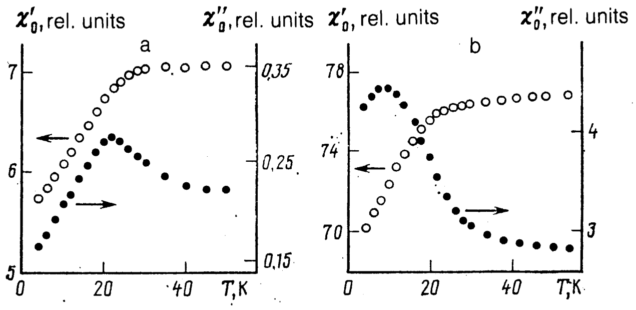

FIG. 4. Temperature dependences of the real (χ_0') and imaginary (χ_0'') components of the dynamic magnetic susceptibility of the alloy $Fe_{65}Ni_{35}$ in magnetization-reversing fields of different intensities: a) $h_0 = 0.3$ Oe; b) $h_0 = 3$ Oe. Measurements were made at a frequency of 36 Hz.

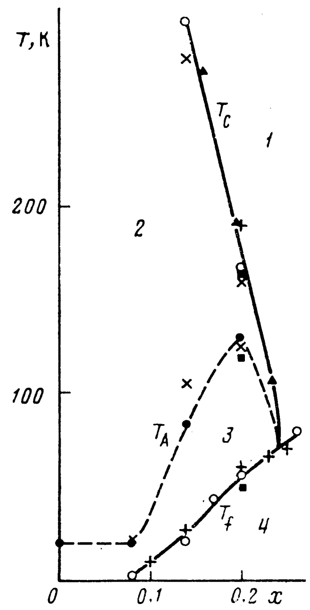

FIG. 5. Magnetic phase diagram of the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ system of alloys: 1) paramagnetic; 2) ferromagnetic; 3) asperomagnetic; 4) spin glass regions. Source of data: (×) Ref. 25; (+), (▲) Ref. 32; our data were obtained from the dependences $\chi_0'(T)$ and $\chi_0''(T)$ represented by ○ and ● and from the dependences $I_s(T)$ and $I_r(T)$ represented by ■.

the ferromagnetic range of compositions (Fig. 5).

The line of the Curie temperatures T_c represents the phase transition from the PM state (region 1) to the collinear FM state (region 2). At lower temperatures a canted FM (ASM) state appears in these alloys (region 3). The T_A line represents the appearance of asperomagnetism and it is deduced from the temperature of the corresponding anomaly of the dependence $\chi_0''(T)$ at minimum values of the magnetization-reversing field h_0.

It is pointed out above that T_A depends strongly on h_0. A similar result is reported in Ref. 31 for amorphous $Pd_{80-x}Fe_xSi_{20}$ alloys. Hence, it follows that the T_A line in the phase diagram (Fig. 5), obtained from the dynamic magnetic susceptibility data in the lowest magnetic fields, is not strictly speaking a phase-transition line. However, there is no doubt that the state of the alloys in this part of the phase diagram differs greatly from the collinear ferromagnetic state and can be identified with asperomagnetism. This conclusion is supported by the observation that the experimental data obtained by other methods on the temperatures of the appearance of the subcritical neutron scattering,[25] the temperatures of deviations of the dependence $I_s(T)$ from the quasi-Brillouin curve, and the temperature dependences of the appearance of weak longitudinal irreversibilities (Fig. 1) are all in good agreement with the values of T_A obtained from the dependences $\chi_0''(T)$ (Fig. 5). Moreover, it is in this part of the phase diagram that we have the Dzyaloshinskiĭ–Moriya anisotropy (Fig. 2), which is observed in frozen noncollinear magnetic structures.

It is shown above that in the case of $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys with x close to the critical concentration x_0 at the temperature T_f there is a phase transition to the state of a reentrant SS (region 4 in Fig. 5) characterized by the absence of the long-range ferromagnetic order. Consequently, in this case the $T_f(x)$ line can quite properly be plotted in the phase diagram.

Figure 5 does not separate the regions in which the SS state appears from the PM and ASM states. This allows for the fact that in the case of the alloy with $x = 0.2$ the SS state does not exhibit the long-range ferromagnetic order. Consequently, in the case of this system of alloys the difference between the reentrant SS phase and the SS phase which appears as a result of the PM–SS transition is manifested only in the magnitude of the FM correlations over regions of finite size. In all other respects the two states are clearly identical.

4. CONCLUSIONS

Our results thus demonstrate that cooling of $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ alloys with compositions in the range $x<0.24$ gives rise to the following sequence of magnetic states: PM–FM–ASM–SS. The asperomagnetic state can be regarded as a superposition of the FM order along a selected direction and spin-glass ordering of the transverse components of the spins. This is the magnetic structure typical of Invar FeNi alloys at helium temperatures.

Alloys belonging to the $Fe_{65}(Ni_{1-x}Mn_x)_{35}$ system with compositions in the range $0.08<x<0.24$ undergo, at even lower temperatures, a transition from the ASM to the reentrant SS state without the long-range ferromagnetic order, but with FM correlations over larger or smaller regions, depending on the difference between x and the critical concentration x_0.

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