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Concentrational ferro-antiferromagnetic transitions in systems based on Fe

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The method of small-angle magnetic scattering of neutrons is used to study the magnetic states that occur during concentrational ferro-antiferromagnetic transitions. Subcritical neutron scattering, caused by the fluctuations of spin density that accompany the concentrational transitions, is observed. The concentration dependences of the magnetic transition temperatures and of the scattering cross sections are found, and the critical concentrations of the transitions are also determined. A cluster model of the transition is proposed; it enables one to calculate the concentration and the mean total value of the fluctuations of the spin density. It is shown that the production of an antiferromagnetic state in a ferromagnetic matrix is due to the γ-Fe atoms.

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INTRODUCTION

The deviation of the values of the mean magnetic moment $\bar{\mu}$, in systems based on Fe, from the Slater-Pauling concentration dependence is interpreted by many investigators[1-3] as a decrease of $\bar{\mu}$ caused by formation of an antiferromagnetic state in a ferromagnetic matrix. This implies the existence of a concentrational ferro-antiferromagnetic transition at some critical concentration c_0, where the Curie temperature $T_c = 0$ K. A complete concentrational ferro-antiferromagnetic transition, with replacement of long-range ferromagnetic order by long-range antiferromagnetic, is observed in the system $Fe_{65}(Ni_{1-x}Mn_x)_{35}$.[1] Here the magnetic state near c_0 is found to be two-phase: antiferromagnetic clusters in a ferromagnetic matrix for $c_{Ni} > c_0$ (Ref. 4) and ferromagnetic clusters in an antiferromagnetic matrix for $c_{Ni} < c_0$.[5] But the reasons for production of an antiferromagnetic state and the mechanism of the change of magnetic order still remain unclear. Furthermore, near c_0 and on the periphery of the clusters, where a change of sign of the exchange interaction occurs, the conditions arise for formation of a "spin glass," which has recently been the object of intensive study.[6]

In the system Fe–Ni, investigation of the magnetic properties near the postulated values c_0 is made difficult by the martensitic transformation, which occurs below 77 K when $c_{Ni} < 34\%$. Nevertheless, the method of small-angle critical scattering of neutrons enables one to study the magnetic structure of the γ phase, which remains after the martensitic transformation. In the present paper, this method is used to investigate the magnetic states of the systems Fe–Ni and $Fe_{65}(Ni_{1-x}Me_x)_{35}$ (Me = Mn, Cr, V) and to determine the parameters of the concentrational transitions and the mechanism and causes of the formation of an antiferromagnetic state.

Earlier,[7,8] diagrams of the magnetic states were constructed for the trinary systems Fe–Ni–Mn and Fe–Ni–Cr over the whole range of concentrations of the γ phase. In these papers, the concentration dependence of the small-angle neutron scattering was studied, but its temperature dependence was not studied, and therefore the origin of the subcritical scattering[4] during formation of the new magnetic phase and the basic physical parameters of the concentrational transitions remained unclarified.

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I. TECHNIQUE OF THE INVESTIGATIONS, AND SPECIMENS

The temperature dependence of the intensity I of small-angle scattering of neutrons was measured, over the temperature range 4.2–500 K, on a neutron diffractometer with wavelength $\lambda = 1.59$ Å. The procedure for obtaining the magnetic-scattering cross section $(d\sigma/d\Omega)_0$ at angle $\theta = 0°$ and the spin-correlation parameter $\varkappa$ at 4.2 K was described in a previous paper.[3]

The measurements were made on cylindrical polycrystals of diameter 8 mm and height 70 mm. In order to obtain a sufficient amount of the γ phase, the specimens of the Fe–Ni system were subjected to a stabilizing heat treatment. The volume fraction of the γ phase obtained after the final quenching to liquid helium was from 20 to 70% in various specimens, and it did not change over the measured temperature range. The smallest Ni content at which it was possible to obtain the required amount of the γ phase in the specimen was 16%. In specimens of the systems $Fe_{65}(Ni_{1-x}Me_x)_{35}$ (everywhere at.%), except for the composition with 14.8% V, the martensitic transition does not occur at 4.2 K.

Figure 1 shows the temperature dependence of I for several speciments of the systems $Fe_{65}(Ni_{1-x}Me_x)_{35}$ at angle $2\theta = 45'$. Similar curves were obtained in investigation of all specimens. In curves 1 and 2, maxima of the critical neutron scattering are evident; their positions determine T_c. The decrease of I when $T_0 < T < T_c$ reflects an increase of the degree of long-range ferromagnetic order, which decreases the magnetic nonuniformity of the alloy. At the same time, there is observed on curves 1 and 2 at $T < T_0$ an increase of I that is due to subcritical scattering and indicates an increase of fluctuations of the spin density clear down to 4.2 K.[4] Thus at T_0 a process begins that prevents the establishment of complete ferromagnetic order at 4.2 K.

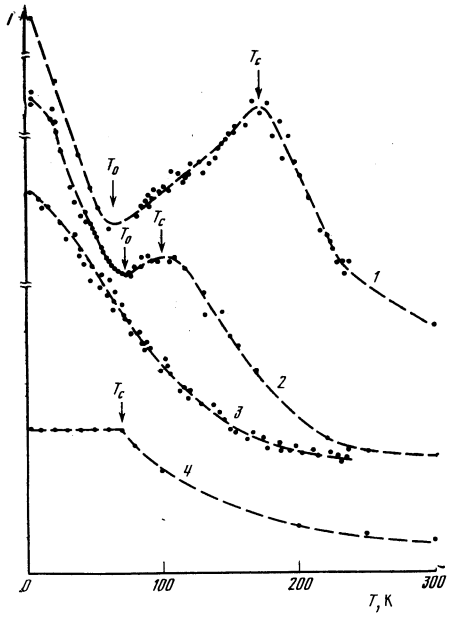

FIG. 1. Temperature variation of the intensity I of neutron scattering at angle $2\theta = 45'$. 1, 7.5% V; 2, 10.3% Cr; 3, 10.9% Cr; 4, 9.8%Mn.

Comparison of curves 1 and 2 and the results of a previous paper[4] show that decrease of c_{Ni} leads to a drop of T_c and a rise of T_0. These changes are accompanied by an increase of the values of I at 4.2 K as compared with the values of I at T_c; this indicates a concentrational increase $\Delta\rho$ of the fluctuations of spin density. In an alloy in which T_c and T_0 disappear ($T_0 = T_c$), the fluctuations prevent altogether the formation of long-range ferromagnetic order; and from the concentration dependence of the values of $T_c - T_0$ one can determine the value of c_0. The temperature dependence of I for such an alloy is shown by curve 3; the value of T_c is 0 K.

When $c_{Ni} < c_0$, in the system containing Mn, formation of long-range antiferromagnetic order is accompanied by a range of decrease of the small-angle scattering,[4] while the temperature variation of I acquires a "step" form (curve 4). Here T_c characterizes the local or short-range ferromagnetic order in regions that remain in the antiferromagnetic matrix after the transition.

Since the para-antiferromagnetic transition is not accompanied by a change of I,[9] it is difficult to identify T_0 directly with the temperature of establishment of short-range antiferromagnetic order. The values of T_0 are above the lower limit of the values of Néel temperature T_N obtained from data on exchange anisotropy.[10,11] Therefore the values of T_0 may be regarded as an upper bound for the existence of short-range antiferromagnetic order. Moreover, at the periphery of fluctuations of the spin density there occurs a change of sign of the exchange interaction, and then the values of T_0 may reflect the beginning of a "freezing" of the spin glass. Thus Fig. 1 demonstrates a systematic change of the magnetic states during concentrational ferro-antiferromagnetic transitions.

II. LOW-TEMPERATURE MAGNETIC STATES

1. *The system* $Fe_{65}(Ni_{1-x}Mn_x)_{35}$. Figure 2 shows the concentration dependence of T_c, T_0, and T_N. From the concentration dependence of the values of the difference $(T_c - T_0)$ we find $c_0 = 27\%$; the minimum value of T_c observed in this system is 150 K. The concentration dependence of T_N plotted from the data of Refs. 1 and 5 and extrapolation of the values of T_N to $T = 0$ K give a

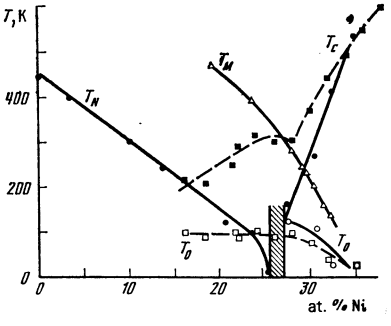

FIG. 2. Concentration variation of T_c, T_O, T_N, and T_M for the systems: ● and ○, containing Mn; ■, □, and △, Fe–Ni.

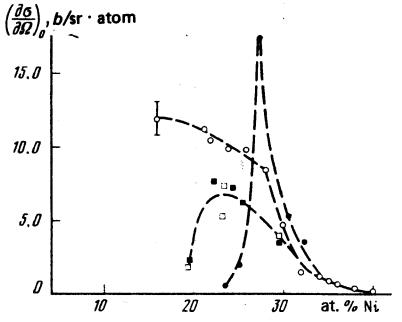

FIG. 3. Concentration variation of magnetic-scattering cross sections $(d\sigma/d\Omega)_0$ for the systems: ●, containing Mn; ○, Fe-Ni; □, containing Cr; ■, containing V.

second value, $c_0 = 25.6\%$. Thus is determined in Fig. 2 a critical range of concentrations (cross-hatched) where there is neither ferro- nor antiferromagnetic long-range order. In Ref. 7 the bounds of such a region were determined less definitely, and the region itself was called a region of "micromagnetism." For $c_{Ni} = 25.2\%$, the temperature for formation of local ferromagnetic order in an antiferromagnetic matrix was given (curve 4 in Fig. 1).

Figure 3 shows the concentration dependence of $(d\sigma/d\Omega)_0$, which reflects the change $\Delta\rho$ during the ferro-antiferromagnetic transition. It is seen that the transition is accompanied by a peak in the concentrational critical scattering. In the critical range (Fig. 2), the specimens have maximum magnetic nonuniformity, which decreases the discontinuity upon appearance of long-range antiferromagnetic order.

The cross section $(d\sigma/d\Omega)_0$ can be expressed in terms of the concentration C of spin-density fluctuations and of $\Delta\rho$ (Ref. 3):

$$(d\sigma/d\Omega)_0 = 0.0486\, C(1-C)\,[M(0)]^2, \qquad (1)$$

where $M(0) = \int_V \Delta\rho\, dr$. Therefore the increase of the values of $(d\sigma/d\Omega)_0$ as $c_{Ni} \to c_0$ in Fig. 3 is dependent on the increase of C and of $|M(0)|$. The values of $|M(0)|$ increase with increase of the spin-fluctuation dimensions $1/\varkappa$, and the concentration dependence of $\varkappa$ at 4.2 K is shown in Fig. 4. It is seen that the values of $1/\varkappa$ increase as $c_{Ni} \to c_0$ and have a maximum value in the critical range. Therefore at the critical values of C and $1/\varkappa$ there occurs a "flow" of spin-density fluctuations to large volumes, and a destruction of the long-range order.

Since the long-range antiferromagnetic order observed[5] at $c_{Ni} < 25.6\%$ is formed from spin-density fluctuations that are increased in size and accumulated in the ferromagnetic matrix as $c_{Ni} \to c_0$, the magnetic state

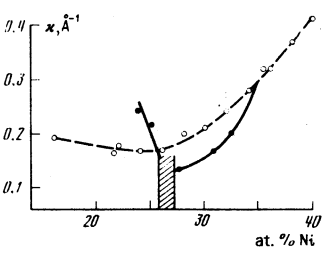

FIG. 4. Concentration variation of spin-correlation parameters $\varkappa$ for the systems: ●, containing Mn; ○, Fe–Ni.

of such fluctuations must be recognized as short-range antiferromagnetic order. Then the range of coexistence of the short-range orders in Fig. 2 is the range in which the conditions for formation of a spin glass are most fully realized.[6]

From Figs. 2 and 3 it follows that the concentrational ferro-antiferromagnetic transition is a phase transition of the first kind.

2. *The system* Fe-Ni. The concentration dependences of T_c and T_0, with inclusion of values of T_c for compositions $c_{Ni} > 32\%$,[3] are shown in Fig. 2. The value of T_0 for $c_{Ni} = 35\%$ is taken from measurements of the exchange anisotropy.[12] It is seen from Fig. 2 that for $c_{Ni} \leq 28\%$ the values of T_0 are constant, while the concentration variation of T_c deviates from the variation that occurs at $c_{Ni} > 28\%$. Similar "breaks" of smooth curves are observed also in the concentration dependences of $(d\sigma/d\Omega)_0$ (Fig. 3) and of $\varkappa$ (Fig. 4).

A trivial cause of the "breaks" might be a change of the content of γ and α phases in alloys with $c_{Ni} < 28\%$, occurring as a result of the stabilizing heat treatment. Then the value of c_{Ni} in the γ phase of alloys with $c_{Ni} \leq 25\%$ would increase by 4–10%. But a micro-x-ray spectral analysis of the phase content, carried out on "Comeca MS-46" apparatus, showed that after this heat treatment the γ and α phase contents differ in each specimen by no more than ±2%, which is within the limits of liquation of the content.

Comparison of the concentration dependences of T_c and of T_M (where T_M is the temperature of the martensitic transformations[13]) in Fig. 2 shows that the deviation of the curves occurs at compositions at which $T_c \leq T_M$ and a change of the kinetics of the martensitic transformation occurs.[14] Therefore a possible reason for them may be the influence of the martensitic transformation, intensifying the ferromagnetic state of the residual γ phase.

Figure 5 shows the temperature variations of I obtained, under strictly identical conditions, on the alloy with $c_{Ni} = 30\%$ before the martensitic transition (curve 1) and after the stabilizing heat treatment (curve 2). It is seen that after the stabilizing heat treatment the peak of the critical scattering was somewhat smeared out, and the value of T_c was increased by 25°. A still larger effect in the increase of T_c is observed on the alloy

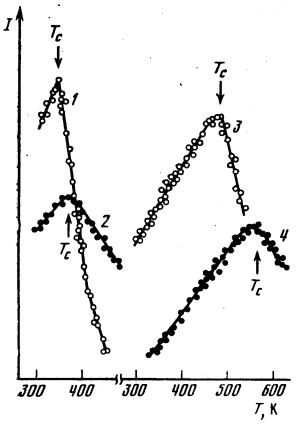

FIG. 5. Critical scattering of neutrons in the alloys $Fe_{70}Ni_{30}$ (1,2) and $Fe_{65}Ni_{30}Cu_5$ (3,4): 1 and 3, before the martensitic transformation; 2 and 4, after the martensitic transformation.

$Fe_{65}Ni_{30}Cu_5$ (Fig. 5), where curves 3 and 4 were obtained, respectively, before and after the martensitic transformation, without stabilizing heat treatment. The broadening of the peaks of critical scattering after the martensitic transformation indicates intensification of the dispersion of the values of T_c, averaging of which apparently gives an increased value of T_c in the residual γ phase. Thus the martensitic transformation actually increases the T_c of the γ phase.

It is impossible by this method to follow experimentally the increase of the values of T_c in the alloys with $c_{Ni} \leq 28\%$, since for them $T_c < T_M$. But in these alloys also there is observed a broadening of the peaks of the critical scattering as compared with curves 1 and 3; the broadening is the more pronounced, the smaller the volume fraction of the residual γ phase in the specimen. Apparently in these alloys the increase of the values of T_c in Fig. 2 is also due to the influence of the martensitic transformation.

Consequently, in the Fe–Ni system, because of the influence of the martensitic transformation, it is not possible to produce an alloy in which $T_c = T_0$ (Fig. 2), and the transition to long-range antiferromagnetic order is absent. Linear extrapolation of the values of $T_c - T_0$ to zero for the compositions with $c_{Ni} \geq 28\%$ leads to a value close to $c_0 = 25\%$, which agrees with the value of c_0 from Ref. 15 and is a virtual value.

The behavior of the concentration dependence of $(d\sigma/d\Omega)_0$ and $\varkappa$ in Figs. 3 and 4 attests to the influence of the martensitic transformation on the static fluctuations of the spin density in the γ phase. Thus the values of $(d\sigma/d\Omega)_0$ do not reach the values characteristic of the critical range of compositions in the system containing Mn, and the peak in the concentrational critical scattering is absent. The values of $\varkappa$, in turn, despite the increase of the values of C, do not insure "flow" of the fluctuations and formation of long-range antiferromagnetic order, no reflections of which are in fact observed in the neutron-diffraction patterns of the specimens at 4.2 K.

According to Fig. 3 and equation (1), the values of C and $|M(0)|$ increase with decrease of c_{Ni}, and consequently it is the Fe atoms that are responsible for the statistical fluctuations of the spin density. The generality of the concentration dependences in Figs. 2, 3, and 7 for the Mn-containing and Fe–Ni systems shows that even in the latter, the fluctuations of the spin density are due to short-range antiferromagnetic order. Therefore the magnetic structure of the γ phase of the Fe–Ni system at $c_{Ni} < 40\%$ is inhomogeneous and is represented by a ferromagnetic matrix in which there exist regions of short-range antiferromagnetic order.

3. *Systems containing* Cr *and containing* V. Figure 6 shows the concentration dependences of T_c and T_0 for both these systems, which differ practically only at $c_{Ni} < 24\%$. In the system containing Cr, $c_0 = 23.2\%$, whereas in the system containing V, $c_0 = 18.4\%$. Since in these systems the concentration of Fe atoms remains constant, the increase of T_0 (Fig. 6) and of $(d\sigma/d\Omega)_0$ in Fig. 3 is effected by spin-density fluctuations that include the Cr and V atoms. The values of T_c and of $(d\sigma/d\Omega)_0$ in the system containing Cr agree well with the data of Ref. 8, although in it the value of c_0 estimated from magnetic measurements was 21.6%. As Figs. 3 and 6 indicate the transition to long-range antiferromagnetic order is not realized. This means that, in contrast to the system containing Mn, not all the spin-density fluctuations take part in the development of antiferromagnetic order, but some of them represent paramagnetic states. Although according to Fig. 6 a concentrational ferro-paramagnetic transition formally occurs in the system containing Cr, in specimens with $c_{Ni} < 23.2\%$, when antiferro- and ferromagnetic short-range orders coexist, formation of a spin glass is possible.

In the system containing V, the maximum of inhomogeneity in Fig. 3 does not coincide with c_0, as is observed for the systems containing Mn and Cr. It is possible that this is also affected by a martensitic transformation, which is observed in the specimen with 14.8% V.

III. CLUSTER MODEL OF A FERRO-ANTIFERROMAGNETIC TRANSITION

In order to explain the nature of the production of the antiferromagnetic state in a ferromagnetic matrix, we shall determine C and $M(0)$ by using the experimental concentration dependence of $\bar{\mu}$, plotted from the results of Refs. 1, 2, and 16 (Fig. 7). From the definition of C and $M(0)$, Eq. (1) and from Fig. 7 it follows that they can be connected with $\bar{\mu}$ by the relation

$$\mu_0 - \bar{\mu} = C|M(0)|, \tag{2}$$

on the assumption that there is no overlapping of the

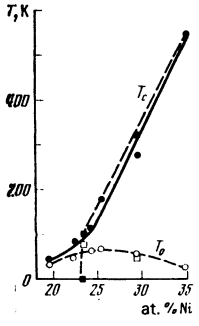

FIG. 6. Concentration dependence of T_c and T_0 for the systems: ■ and □, containing Cr; ● and ○, containing V.

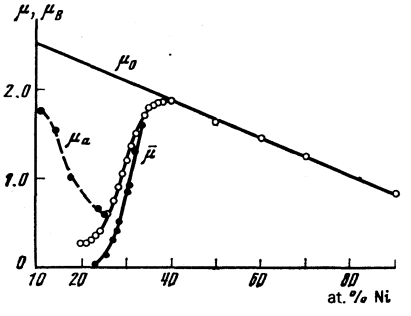

FIG. 7. Concentration dependence of magnetic moments of the systems: ●, containing Mn; ○, Fe–Ni.

fluctuation ranges. Here μ_0 is the linear continuation of the Slater-Pauling relation to compositions with c_{Ni} <40%. The concentration dependence of C, obtained from the solutions of equations (1) and (2) for all the systems, are shown in Fig. 8. In view of the effect of the martensitic transformation, values of $(d\sigma/d\Omega)_0$ for the Fe-Ni system at $c_{Ni}<28\%$ were not included for estimation of C. The values of $\overline{\mu}$ for the systems containing Cr and V were interpolated according to the data of Ref. 17 and are estimates, and the values of C are illustrative. The solid curves in Fig. 8 show the calculated concentrations of clusters containing no Ni atom in the first coordination sphere around the Fe, Mn, Cr, and V atoms (curve 1) and containing only one Ni atom in the first sphere (curve 2). The calculation was made by the formulas of the binomial distribution of short-range atomic order in the Fe-Ni system for specimens with $c_{Ni}\geq 30\%$.[3]

From Fig. 8 it follows that only clusters consisting exclusively of Fe, Mn, Cr, and V atoms act as centers of static spin-density fluctuations. They are formed by fluctuations of composition in the first coordination sphere. The value of C corresponding to c_0 for the system containing Mn is 1.8%. This value, together with the value of $\varkappa$ in Fig. 4, insures flow of short-range antiferromagnetic order to large volumes and consequent formation of long-range order. In the Fe-Ni system, the virtual value $c_0=25\%$ corresponds to the value $C=2.4\%$, which is not attained in the compositions investigated in Fig. 8.

It is evident from Fig. 8 that the values of C in the systems containing Cr and V at $c_{Ni}<25\%$ exceed the value necessary for formation of long-range antiferromagnetic order. This means that not all the clusters containing Cr and V take part in the development of short-range antiferromagnetic order. As is mentioned in Ref. 8, for realization of a ferro-antiferromagnetic transition in the system containing Cr it is necessary to raise the Fe content. Thus for the composition $Fe_{70}Ni_{15}Cr_{15}$, long-range antiferromagnetic order is observed.[18]

The concentration dependence of $|M(0)|$ for the Mn-containing and Fe-Ni systems is shown in Fig. 9 (curve 1). The values of $|M(0)|$ increase with decrease of c_{Ni}, and therefore the similar increase of $(d\sigma/d\Omega)_0$ in Fig. 3

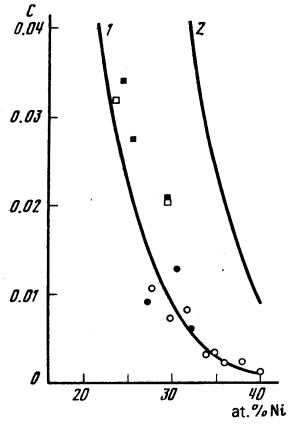

FIG. 8. Variation of the cluster concentration C with composition of the alloy for the systems: ●, containing Mn; ○, Fe–Ni; □, containing Cr; ■, containing V. Curves 1 and 2: calculated cluster concentrations.

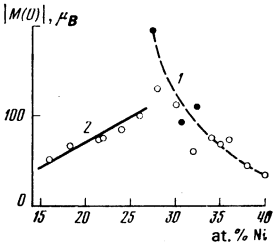

FIG. 9. Concentration dependence of total change of spin density $|M(0)|$ for the systems: ●, containing Mn; ○, Fe–Ni.

is caused both by increase of C and by increase of $|M(0)|$. In the systems containing Cr and V at $c_{Ni}>23\%$, the values of $|M(0)|$ remain constant and equal to $70\,\mu_B$; this again indicates slight development of short-range antiferromagnetic order in them.

Since the value of C depends only on c_{Ni}, its value in alloys of the Fe–Ni system, where an influence of the martensitic transformation has been noted, can be determined from curve 1 of Fig. 8. The concentration dependence of $|M(0)|$ in this case is found from the concentration dependence of $(d\sigma/d\Omega)_0$, Fig. 3, and is also shown in Fig. 9 (curve 2). For these alloys, the increase of C with decrease of c_{Ni} is accompanied by a decrease of $|M(0)|$, which correlates with some decrease of $1/\varkappa$ according to Fig. 4. Thus the slowing down of the increase of $(d\sigma/d\Omega)_0$ at $c_{Ni}<28\%$ in Fig. 3 is connected with a decrease of $|M(0)|$, and the influence of the martensitic transformation on the static spin-density fluctuations manifests itself in a decrease of their size and dimensions. This fact leads to a decrease of the magnetic inhomogeneity of the γ phase and impedes the transition to long-range antiferromagnetic order, despite the increase of the values of C. It is natural to suppose that the decrease of the magnetic inhomogeneity, in turn, is the cause of the slowing down of the drop of T_c and rise of T_0 in Fig. 2.

These results do not reveal the mechanism of the influence of the martensitic transformation on the antiferromagnetic state of the magnetic structure of the residual γ phase, and the ambiguity and insufficient conclusiveness of the possible mechanisms prevent the making of even a qualitative choice.

The relations in Figs. 7–9 indicate that the antiferromagnetic state of the clusters is determined by the value of the magnetic moments μ_{Fe} of the Fe atoms that form a cluster. Figure 7 shows the concentration dependence of the mean antiferromagnetic moments μ_a of specimens of the Mn-containing system at $c_{Ni}<27\%$, plotted from the results of Refs. 4 and 19. Comparison of the values of μ_a with the value $1.85\,\mu_B$ for the composition $Fe_{65}Mn_{35}$,[4] where $\mu_{Fe}=1\,\mu_B$,[20] and with the value $\mu_{Fe}=0.7\,\mu_B$ for pure γ-Fe[21] shows that in specimens with long-range antiferromagnetic order, $\mu_{Fe}\leq 0.7\,\mu_B$. Hence it may be supposed that in the Fe–Ni system also, short-range antiferromagnetic order is formed by clusters of γ–Fe with $\mu_{Fe}=0.7\,\mu_B$.

IV. CONCLUSION

Study of concentrational ferro-antiferromagnetic transitions has shown that the production of an antiferromagnetic state in a ferromagnetic matrix occurs as a

result of a fluctuation of composition in the first coordination sphere. Fluctuations of concentration produce spin-density fluctuations, which develop into long-range antiferromagnetic order with increase of the Fe content. The basic nuclei of the antiferromagnetic state are clusters composed solely of Fe atoms. These results agree well with Sedov's paper,[22] where it was shown that the magnetic state of the Fe atom in the γ phase is determined by its nearest environment and that for certain neighborhoods the value of μ_{Fe} tends to zero.

Atoms of Mn promote the development of the antiferromagnetic state and accelerate the ferro-antiferromagnetic transition. But only a part of the Cr and V atoms participate in the formation of clusters with short-range antiferromagnetic order, and this leads, at insufficient concentrations of Fe, to a concentrational ferro-paramagnetic transition.

The martensitic transformation that occurs in the Fe–Ni system affects the antiferromagnetic state in the residual γ phase and slows down the ferro-antiferromagnetic transition. Since the production of an antiferromagnetic state begins in the Invar range of compositions, it causes so-called Invar anomalies of the properties, one of which (a decrease of $\bar{\mu}$) is described by the proposed cluster model.

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Dilatometric investigation of critical phenomena in the ferroelectric phase transition in antimony sulfoiodide SbSI

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A capacitive dilatometer is used to investigate in detail the ferroelectric phase transition in single crystal antimony sulfoiodide SbSI at pressure up to 5 kbar. The coordinates of the polycritical point were found to be $T = 233 \pm 5$ K and $P = 1430 \pm 100$ bar. An analysis of the experimental data yielded within the limits of the experimental accuracy a value 0.5 for the exponent α, in agreement with the Landau theory for the case of the tricritical point.

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At first shown by Landau,[1] the line of first-order phase transitions can terminate at a critical point of a singular type, wherein the phase does not vanish at all, as in the case of the critical point on the evaporation curve, but becomes a second-order phase transition. The possibility of the existence of critical points of this type follows from the Landau expansion

$$\Phi = \Phi_0 + A\eta^2 + B\eta^4 + D\eta^6 + \ldots, \quad (1)$$

a second-order phase transition occurs at $B > 0$, a first order transition at $B < 0$, and the critical point

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