Isomeric shift and magnetic hyperfine interaction for Sn in Ni in the critical temperature region

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The Mössbauer effect was used to investigate, in the critical temperature region, the temperature dependences of the magnetic hyperfine interaction and of the electron density of impurity ^{119}Sn atoms in the vicinity of the nucleus in a nickel matrix. The existence of oscillations of the isomer shift of the resonance lines of ^{119}Sn in the Ni-Cr alloy, observed by us in 1974, is confirmed. The possible causes of the abrupt changes of the electron density following small changes of the temperature in the vicinity of T_c are discussed. The temperature-dependent broadening of the resonance line at temperatures above T_c, which can be attributed to critical fluctuations of the magnetic moment of the matrix, is obtained. For the sample Ni+0.95 at.% Sn in the temperature range $0.9678 \leq T/T_c \leq 0.9978$, the magnetic hyperfine field is proportional to $(1-T/T_c)^{\beta}$, where $\beta = 0.625 \pm 0.040$. No singularities whatever were obtained in the temperature dependence of the Mössbauer-effect probability in the vicinity of T_c.

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1. INTRODUCTION

In the critical temperature region, many properties of ferromagnetic matter depend strongly on the temperature. Measurements of the magnetization, of the magnetic susceptibility, of the specific heat, and of other quantities reflect the collective behavior of the spin system near the phase-transition temperature, but at present it is difficult to predict how the electronic structure of the ferromagnet is altered thereby. It is to be expected nevertheless that the electronic wave functions of a ferromagnetic crystal do not remain constant as the temperature varies in the critical region. For example, for a metallic ferromagnet, within the framework of the band model, the dependence of the electronic wave functions on the temperature can be regarded as a natural consequence of the shift of the Fermi level as the exchange splitting is rapidly decreased near the Curie perature (T_c). Interest attaches therefore to experiments in which the temperature dependence of the electronic wave functions can appear in explicit form. The corresponding information can be obtained with the aid of the Mössbauer effect, since the isomer shift (IS) that determines the position of the Mössbauer resonance line in the energy scale is directly proportional to the electron density (i.e., to the square of the electronic wave function) in the region of the nucleus.

Preston, Hanna, and Heberle have observed in 1962 [1] an abrupt change in the shift of the Mössbauer resonance line at T_c in metallic iron. Preston [2] investigated this phenomenon in detail and has shown that the change of the shift takes place in a narrow temperature range not exceeding 0.3°. It was shown in [1,3] that the rapid change of the position of the line near T_c should be connected indeed with the change of the IS, i.e., with the difference between the electron densities in the region of the nucleus for the ferromagnetic and paramagnetic state, but the cause of such a sharp "jumplike" change of the IS remained unclear. Calculations [3] predict only a smooth (proportional to the magnetization) change of the electronic wave functions under the influence of the exchange interaction, and consequently, as noted by Preston [2], the IS discontinuity at T_c seems to contradict the premise that the ferromagnetic phase transition is of second order.

We have recently observed [4] in an investigation of the temperature dependence of the hyperfine structures of Mössbauer spectra of impurity Sn atoms in an Ni-Cr alloy an unexpected phenomenon—"oscillations" of the shift of the resonance line in the vicinity of T_c. The temperature dependence of the shift had a complicated structure consisting of narrow maxima and minima, and in some case the abrupt change of the line shift was observed when the temperature was altered by 0.1° or less. The hypothesis was advanced that the onset of this structure is due to the influence exerted on the electronic wave functions by the exchange interaction, which should vary rapidly with temperature near T_c.

In the present study we investigated this phenomenon for Sn impurity atoms in a matrix of pure Ni, using an experimental technique greatly improved over that previously employed. [4] We have also obtained data on the behavior of the hyperfine magnetic field and of the width of the resonance line in the critical temperature region.

2. EXPERIMENTAL TECHNIQUE

The main requirements that the experimental procedure must satisfy are to ensure high stability of the sample temperature and eliminate the effect exerted on the measurement results by the instability of the Mössbauer spectrometer.

The sample temperature was stabilized with the aid of an electronic regulator, that stabilized the temperature within not more than ±0.01° for at least 8 hours. (The measurement time at a fixed temperature varied with the activity of the source and the magnitude of the resonant-absorption effect, and ranged from 3 to 5 hours.) The temperature was measured with a chromel-alumel thermocouple; a similar thermocouple was used in the feedback circuit of the regulator. There was no exact absolute calibration of the thermocouples, so that the absolute temperature was known with an approximate accuracy 0.3°.

The Mössbauer resonance-absorption spectra were measured with a double electrodynamic spectrometer, which could measure simultaneously both the spectrum of the investigated sample and the spectrum of the "standard" absorber (relative to which the shift of the resonance line was determined). The "standard" (just

as in [4]) was the alloy Pd + 0.5 at.% Sn, the shift for which is very close to the line shift in dilute Ni-Sn alloys. The measurements were made with two ^{119m}Sn sources, made of the compound $CaSnO_3$, with activity of several dozen mCi. In all the measurements, the sources (as well as the "standard" absorber) were at room temperature. The spectrometer operated in the constant acceleration regime (with a triangular time dependence of the velocity). It is important that the spectra of the Ni-Sn samples were measured in the moving-source regime (with the sample immobile and placed in an electric oven), and the spectra of the "standard" were measured in the moving absorber regime (with the source immobile). To effect this measurement method, one of the sources was connected to one end of a rigid rod fastened to the coils of the electrodynamic vibrators, and the light holder of the "standard" absorber was connected to the other end. As a result, the spectrum of the investigated sample and the spectrum of the "standard" were measured simultaneously, during the same section of the time scan. We believe that this procedure eliminates almost completely any effect of the spectrometer null drift and instability of the value of the analyzer channel on the measurement results. The pulses from the two counting channels corresponding to the spectrum of the sample and to the spectrum of the "standard" were registered in even and odd channels of an NTA-512 analyzer, respectively. Since the time dependence of the velocity was triangular, we measured simultaneously two spectra for the sample and two for the "standard"; the results of these two independent measurements were averaged.

To determine the center x_0 of the resonance line we calculated the areas $S_R(i)$ and $S_L(i)$ of the two halves of the line (to the right and left of the chosen channel number i); by definition, the center of the line corresponds to vanishing of the difference $Y(i) = S_R(i) - S_L(i)$. At small variations of the number i of the channel near the true center of the line ($\pm 2-3$ channels), the function Y(i) is linear with good accuracy, making it possible to determine x_0 by simple arithmetic operations. The calculations connected with the reduction of the spectra were performed with a computer. The rms statistical error of x_0 (expressed in analyzer channels) is given, when this method is used, by the formula

$$\sigma(x_0) \approx (2n/N\epsilon^2)^{1/2},$$

where n is the number of channels taken into account in the calculation of each of the areas S_R and S_L, N is the average number of counts per channel, and ϵ is the magnitude of the resonant-absorption effect. The measurement time was chosen such that $\sigma(x_0)$ for the sample spectrum did not exceed 0.1 channel. (The error in the determination of x_0 for the "standard" was negligible because of the large value of ϵ). At n = 15 and ϵ = 0.1 the condition $\sigma(x_0) = 0.1$ corresponds to $N \approx 3 \times 10^5$ counts. The value of each channel corresponded to 0.031 mm/sec.

The absorption spectra were measured for two Ni-Sn samples containing 0.95 and 0.55 at.% Sn. (These two samples will henceforth be referred to as Ni 95 and Ni 55.) The samples were prepared by melting in vacuum metallic Ni (99.98% pure) and metallic Sn enriched with ^{119}Sn to 85.2%. No chemical analysis of the samples was carried out, but the losses in melting were negligibly small. The ingots were homogenized at 1000°C for 30 hours. The ingots were powdered and the powder was annealed at 500 and 350°C. We note that prolonged annealing at a temperature close to T_c did not lead to noticeable changes of the form of the absorption spectra. The powders were deposited on an aluminum foil and then covered also with aluminum foil and sealed. The use of finely powdered absorbers eliminates the need to take into account, in the analysis of the unresolved hyperfine structure, the preferred domain orientation that always arises in foils of ferromagnetic metals. The spectra for the Ni 95 sample were measured in the temperature range 580–610°K. The measurements with the Ni 55 sample were regarded as a control; since the results for the two samples did not differ noticeably, the Ni 55 sample was investigated only in several temperature intervals near T_c.

An important experimental condition was to reduce to a minimum the temperature gradient over the sample. To this end, the sample in the oven was covered on both sides with several layers of aluminum foils and with beryllium disks. By direct measurements, we were able to demonstrate only that the temperature gradient between the center of the sample and its periphery does not exceed 0.1°. Actually the effective temperature gradient was much lower, since we observed in each experiment the singularities in the changes of the isomeric shift, the widths of which did not exceed 0.03° in a number of cases. The absorption spectra were measured at 700 different values of the temperature for the Ni 95 sample and 150 different temperatures for the Ni 55 sample.

3. MEASUREMENT RESULTS AND THEIR DISCUSSION

3.1. Temperature Dependence of Hyperfine Magnetic Field

The magnetic field acting on the nucleus of an impurity Sn atom in an Ni matrix is small even at low temperatures, and therefore we always observed in the spectra single lines with unresolved hyperfine structure. To determine the hyperfine field at temperatures below T_c, the measured absorption spectra were compared with the theoretically calculated ones. The forms of the spectra were analyzed under the following assumptions: 1) the hyperfine field was the same for all the Sn atoms, and 2) all the hyperfine-structure components in the ferromagnetic phase had the same width, equal to the line width in the paramagnetic phase at sufficiently high temperatures (see Sec. 3.2). The correctness of the first of these assumptions is obvious, since the concentration of the impurity atoms is low enough. The validity of the second assumption is difficult to verify directly, but the differences in the widths of the individual components of the hyperfine structure cannot be appreciable, since the hyperfine field near T_c is very weak. According to our estimates, the possible deviations from accuracy of this assumption should not greatly influence the determined hyperfine field.

The measured temperature dependence of the magnetic hyperfine field H for Sn and Ni was compared with the formula

$$H(T) = H(0)D(1-T/T_c)^{\beta}, \quad (1)$$

where H(0) is the field at T = 0, and D and β are empirical constants. Formula (1) is the analog of the relation customarily used to describe the temperature dependence of the magnetization in the critical region. The applicability of formula (1) in this case is not obvious, since the hyperfine field for Sn in Ni is known not

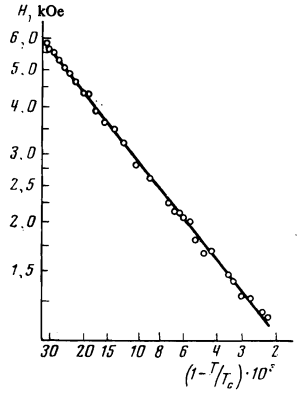

FIG. 1. Plot of H against $\ln(1-T/T_C)$ for Sn in the alloy Ni+0.95 at. % Sn in the critical temperature region. The straight line corresponds to formula (1) with $\beta=0.625$.

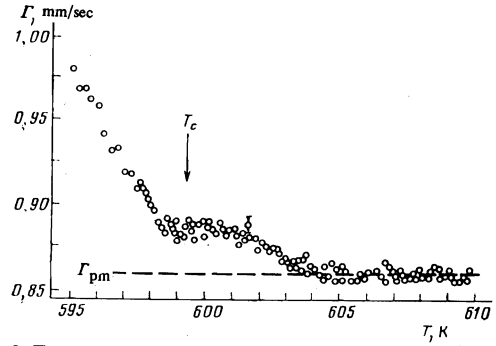

FIG. 2. Temperature dependence of the width Γ of the resonance line for the sample Ni+0.95 at. % Sn.

to be proportional to the magnetization of Ni in a wide temperature range.[5,6] It turned out nonetheless that formula (1) agrees quite well with the experimental data for temperatures close enough to T_c. To determine the parameters H(0) D and β, we used Heller's method.[7] For sample Ni 95, good agreement between the experimental data and formula 1 in the temperature range $0.9678 \leq T/T_c \leq 0.9978$ was obtained at the following values of the parameters:

$$\beta=0.625\pm0.040,\ H(0)\,D=(51.11\pm0.40)\,\text{kOe},\ T_c=(599.3\pm0.3)\ \text{K}. \quad (2)$$

A comparison of the experimental data with (1) at these values of the parameters is shown in Fig. 1.

The value H(0) = (20.2 ± 0.5) kOe was obtained by extrapolating the data obtained in the 77–300°K range, using the results of[5]. Using this value of H(0), we obtain D = 2.53 ± 0.11. The hyperfine field at room temperature was found to be 18.6 ± 0.6 kOe, in good agreement with recent measurements by Hembree and Price[8] for an alloy containing 1 at. % Sn.

A similar analysis of the experimental data (in the temperature range $0.9774 \leq T/T_c \leq 0.9962$) yielded the parameter values $\beta = 0.63 \pm 0.06$, H(0)D = (52.3 ± 1.0) kOe and T_c = (610.8 ± 0.5) K. Thus, within the limits of the measurement errors, the values of the constants H(0)D and β are the same for both samples.

The obtained value of the critical exponent β for the hyperfine field greatly exceeds the values of this parameter, characterizing the magnetization of Ni in a hyperfine field for the magnetic impurity atoms in Ni ($\beta \approx 0.38$[9]). This difference, generally speaking, is not surprising, since the temperature dependence of the hyperfine field for Sn in metallic ferromagnets is usually anomalous and does not follow the temperature dependence of the matrix magnetization. A direct comparison of the two values of the critical exponent is hardly possible at the present time: the causes of the anomalous behavior of H(T) are still unclear and there is no quantitative theory to connect the H(T) dependence for nonmagnetic impurity atoms of the type Sn with the magnetization of the ferromagnetic matrix.

3.2. Line Width and Probability of Resonant Absorption

In each individual measurement, the resonance line width Γ (at half height) was determined on the average with approximate accuracy 0.01 mm/sec. However, within the limits of each 0.1° temperature interval the absorption spectrum was measured several times (from 4 to 12 individual measurements), and within the limits of any of these intervals we did not observe any temperature variations of Γ exceeding the statistical error. This enabled us, in the analysis of the temperature dependence of Γ, to average the results of individual measurements for each interval of 0.1°. The error of such an averaged value of Γ does not exceed 0.006 mm/sec.

The measured dependence of Γ on the temperature for the Ni 95 sample is shown in Fig. 2. At $T < T_c$ an increase of temperature is accompanied by a decrease of Γ, due to the decrease of the hyperfine field. At $T \approx T_c$, a characteristic break is observed in the Γ(T) plot, but with further rise in temperature Γ(T) becomes anomalous: it remains approximately constant in the interval 599–601°K and only at $T > 602$°K does it decrease slowly to a value Γ_{pm} = 0.86 mm/sec. This anomaly can obviously not be attributed to a smearing of the phase-transition point because of the statistical inhomogeneity of the alloy, since such an inhomogeneity can lead only to a smearing of the break in the plot of Γ(T) near T_c and to a monotonic decrease of Γ. It is reasonable to assume that the anomaly of Γ(T) near T_c is connected with relaxation phenomena in the critical region. The critical fluctuations of the magnetization can have a sufficiently large effective lifetime, and this leads to the appearance of a nonzero time-averaged hyperfine field at temperatures above T_c. An influence of such a mechanism on the width of the Mössbauer line was observed for EuO in[10] and for Fe in Ni in[11]. In these cases the temperature dependence of the excess width due to the critical fluctuations agreed well with a power law with a critical exponent close to −1. Our data for Sn in Ni are not described, however, by such a power law. It is possible that this is due to the fact that Sn is a nonmagnetic impurity in the Ni matrix, and the influence of the fluctuations of the magnetic moment on the width manifests itself in this case differently than for atoms having their own magnetic moment. In addition, the hyperfine field for Sn in Ni, even under saturation conditions, is much smaller than for Fe in Ni, and this should lead to an appreciable change of the ratio of the correlation time of the fluctuations to the nuclear Larmor precession frequency.

In the investigated temperature range, we observed no singularities whatever in the behavior of the probability of the resonant absorption: the area under the resonance line remained constant with a relative accuracy not worse than 3%. In particular, no noticeable changes were observed in the resonant absorption on going through T_c. Thus, the amplitudes of the Mössbauer-atom oscillations in the critical temperature region are practically independent of the temperature. That the probability of the Mössbauer effect is constant on going

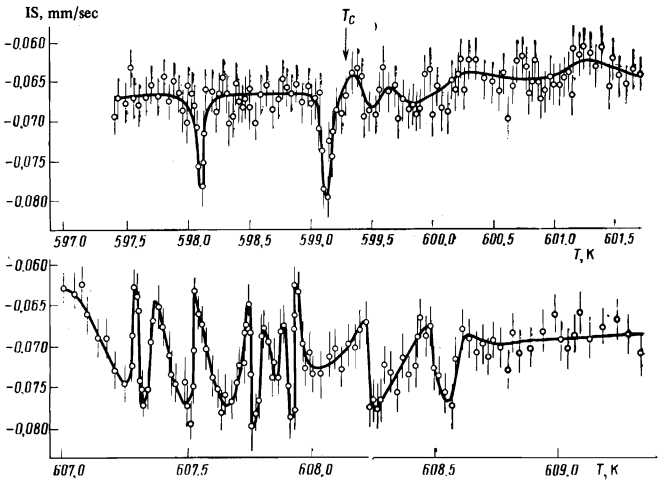

FIG. 3. Temperature dependence of the isomer shift (IS) for Ni+ 0.95 at. % Sn in the ranges 597.4–601.7°K (upper figure) and 607.0–609.3°K (lower figure). The solid lines are drawn through the experimental points.

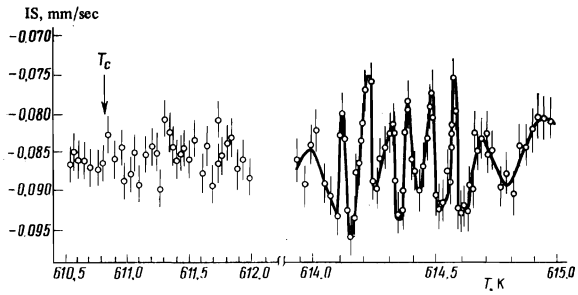

FIG. 4. Temperature dependence of the isomer shift (IS) for the Ni+ 0.55 at. % sample in the ranges 610.5–612.0°K and 614–615°K. The solid line is drawn through the experimental points.

through T_c was established earlier for Fe atoms in an Ni matrix.[17] No effect of the magnetic ordering on the dynamics of the lattice was observed also in a neutron-spectroscopy investigation of pure Ni.[13] We regard these results as sufficient basis for expecting no singularities whatever also in the temperature dependence of the thermal shift of the Mössbauer line. This conclusion follows, for example, from an analysis of the relation between the change of the probability of the effect and the thermal shift, carried out (within the framework of the Debye model) by Belova and Nikolaev.[14] At sufficiently high temperatures, when the singularities of the phonon spectrum are of little importance, the corresponding conclusions of[14] should be valid for any model of the dynamics of the atoms in a crystal. Therefore in the discussion of the experimental data on the temperature variations of the shift of the Mössbauer line (see Sec. 3.3) we shall take into consideration only the temperature dependence of the IS, i.e., the changes of the electron density in the region of the nucleus.

3.3. Temperature variations of the isomeric shift

The temperature dependence of the resonance-line shift was measured for the Ni 95 sample in the temperature range 597–610°K in average steps of 0.05° in intervals where the shift changes relatively smoothly, and in steps of 0.01° in regions where the shift varies rapidly. (At temperatures below 597°K the shift was not measured, since the rapid increase of the line width in the ferromagnetic phase decreased the accuracy with which the shift was determined.) Analogous measurements on the Ni 55 sample were made in individual temperature intervals; the results were qualitatively the same for both samples. We have confirmed the existence of temperature "oscillations" of the IS, observed earlier for Sn in the Ni-Cr alloy,[4] and investigated certain qualitative regularities connected with the temperature dependence of the IS. At this stage of the work it was not our purpose to study in detail the forms of the individual singularities in the temperature dependence of the IS, since this would call for an increase in the statistical accuracy of the measurements by at least a factor of two and would increase excessively the duration of the experiment. The minimum amplitude of the "oscillations" of the line shift for Sn in Ni is 0.02 mm/sec, which is approximately seven times larger than the rms statistical error of an individual measurement.

Figures 3 and 4 show fragments of the temperature dependence of the shift of the Mössbauer line for the samples Ni 95 and Ni 55. It is seen that in certain temperature intervals there are observed abrupt changes of the shift at small changes of the temperature. As a rule, the widths of the maxima (or minima) of the shift did not exceed 0.1°, and in a number of cases they were much less. The distribution of these "oscillations" of the shift is not uniform over the temperature scale. For the Ni 95 sample, the maximum density of the "oscillations" was observed in the ranges 601.6–604.5°K and 606.3–608.7°K, whereas in other temperature ranges the shift varied relatively smoothly or remained constant. Figures 3 and 4 show sections with shift "oscillations," as well as sections where the shift is practically independent of temperature. We note that the Curie temperatures determined from the temperature dependence of the hyperfine field fall for both samples in intervals where there are no "oscillations" or where the latter are weakly pronounced. The mean value of the line shift in the entire investigated temperature range changes little; no isolated shift "jumps" that might correspond to a transition of the system from the ferromagnetic to the paramagnetic state (similar to those observed for metallic iron[1]) were observed. Attention can be called to the fact that some of the "oscillations" have a noticeably asymmetrical (sawtooth) shape; in individual cases an abrupt change of the shift was observed when the temperature changed by an amount on the order of 0.01°.

We were unable to observe in the shift-"oscillation" distribution any regularities that could be represented in mathematical form. The results of our work and of[4] are apparently still insufficient for a unique choice of the model of the new phenomenon, and we therefore confine ourselves below to a qualitative analysis of some of the possible explanations of the temperature oscillations of the electron density. Abrupt changes of the electron density in the region of the nucleus can occur either because of changes in the volume or for other reasons which are not connected directly with the change of volume. The scale of the volume variations of the electron density can be estimated from the results of measurements of the influence exerted on the IS by an external pressure. From these data (see, e.g.,[13]) it can be found that in metallic systems for ^{119}Sn the change of the IS and the relative change of the volume V are connected by the empirical relation

$$\Delta\,(\text{IS, mm/sec}) \leqslant \Delta V/V.$$

It follows from this relation that to explain the abrupt changes of the IS, which were observed in our experiment, it would be necessary to resort also to "oscillating" changes of the volume amounting to at least 1–2%. There are no data pointing to so large a change in the volume of Ni in the region of the magnetic phase transition. (We note, for example, that the magnetostriction in the case of Ni can lead to IS changes much smaller than those observed in our experiment.) It appears thus that the IS "oscillations," the amplitude of which reached 0.2 mm/sec, can not be attributed to volume effects.

In [4] we have proposed that the cause of the abrupt changes of the IS following small changes of the temperature is the influence exerted on the electronic wave functions by the exchange interaction, which changes rapidly with temperature in the vicinity of T_c. The dependence of the wave functions on the exchange interaction seems in itself to be natural and is discussed, for example in [3] in connection with an interpretation of the jump of the IS at T_c in metallic iron. In this case, however, it is necessary to explain the discrete character of the variations of the IS at very small changes of the temperature. It was assumed [4] that this discreteness can result from quantization of the electronic states under the influence of the effective exchange field. Small changes of temperature could then be expected to be accompanied by changes of the electron density on the Fermi surface, and this should manifest itself in corresponding changes of the IS. In this interpretation, the "oscillations" of the IS can be set in correspondence, for example with the oscillations of the Knight shift in an external magnetic field, or with the de Haas–van Alphen effect in magnetic metals. This explanation, however, calls for an additional assumption that there exist near T_c very large exchange fields, for otherwise, at temperatures on the order of 600°K, no influence of quantization of the electronic states on the IS can be observed.

In the discussion of the results of this paper, Yu. M. Kagan suggested another possible approach to the interpretation of the experimental data, based on considering the influence of critical fluctuations on the Friedel oscillations of the electron density, and in which account is taken of the fact that in the present experiment (just as in [4]) the variations of the electron density were observed for impurity atoms in a metallic ferromagnetic matrix. In this case the electron density in the vicinity of the nucleus depends on the sum of the distributions that oscillate with distance, and the period of the oscillations depends on the electronic wave vector on the Fermi surface, k_F. In the critical temperature region, k_F can vary very rapidly with temperature, owing to the change of the geometric dimensions of the fluctuations of the magnetic moment and of the magnetization in different regions of the crystal. If it is recognized that the sum of the oscillating functions for a given lattice site can be very sensitive to small changes of k_F, this mechanism should lead in principle to a sharp dependence of the IS on the temperature. In future experiments we propose to obtain new data on the behavior of the IS near T_c for other ferromagnetic and antiferromagnetic systems.

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