Investigation of the effect of a random field in $Mn_xZn_{1-x}F_2$ by the neutron elastic scattering method

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Neutron diffraction was used to investigate the influence of a magnetic field on the phase transition from the paramagnetic to the antiferromagnetic state in $Mn_{0.47}Zn_{0.53}F_2$. A field of 21 kOe lowered the ordering temperature by 1.5 ± 0.1 K. An increase in the intensity of the magnetic reflection was observed as a result of cooling in the presence of a field, indicating formation of a domain structure which relaxed rapidly when the external field was reduced.

INTRODUCTION

Many theoretical and experimental investigations of phase transitions in dilute antiferromagnets in an external field have been published recently. These investigations have been stimulated by the theoretical paper of Fishman and Aharony[1] who showed that the properties of an easy-axis antiferromagnet diluted by nonmagnetic impurities and subjected to a homogeneous external magnetic field parallel to the easy axis are similar to the properties of a ferromagnet in a random magnetic field, i.e., in a field with its sign alternating at random from one lattice site to another.

The behavior of a dilute antiferromagnet in a homogeneous magnetic field differs qualitatively from that of the original undiluted system. Any random field (no matter how small) destroys the long-range order of a two-dimensional Ising magnetic system, which then splits (for energy reasons) into antiferromagnetic domains which decrease in size as the random field increases. According to the current ideas, the ground state of a three-dimensional antiferromagnet should be a single domain. However, many experiments and recent theoretical investigations demonstrate that a low-temperature state of such an antiferromagnet subjected to a magnetic field may depend strongly on its previous history. When a sample is cooled in a magnetic field, it may split into a set of domains which vary in size very slowly (if at all) with time. A random field alters also the phase diagram of a dilute antiferromagnet and it reduces strongly the ordering temperature.

The unusual behavior of dilute antiferromagnets in an external field has recently stimulated many experiments. The random field effect is being studied by a variety of methods: using the specific heat and magnetostriction, changes in the birefringence of light, diffraction of thermal neutrons, magnetic susceptibility, and absorption of ultrasound (for a review see, for example, Ref. 2). We investigated the random field effect in an $Mn_{0.47}Zn_{0.53}F_2$ crystal by the neutron elastic scattering method. Preliminary results were published earlier.[3]

By analogy with MnF_2, paramagnetic Mn ions in antiferromagnets of the $Mn_xZn_{1-x}F_2$ system form a body-centered tetragonal lattice. An investigation of the phase transition in compounds of this type by the neutron scattering method is convenient because the (100) plane may exhibit only the magnetic reflection, whereas the nuclear reflection at the same Bragg angle is observed only for the (200) plane. In zero magnetic field the phase transition in these compounds belongs to the Ising class, so that we can apply the theoretical predictions on Ising magnetic materials.

DESCRIPTION OF THE EXPERIMENTS

a) Neutron source

Our experiments were carried out using a pulsed neutron source based on a 30-MeV microtron at the Institute of Physics Problems of the USSR Academy of Sciences.[4] Electrons accelerated in the microtron traversed a thin (0.15 mm) stainless-steel window and entered a transport channel in which a focusing triplet of quadrupole lenses was located. The effective diameter of the electron beam at the exit was 20 mm. A water-cooled deceleration target made of Ta, 6 mm thick, was placed outside the exit window (the range of 30-MeV electrons in Ta was 6 mm) and this was followed by a neutron lead converter with a diameter of 50 mm and 50 mm long (Fig. 1). Determination of the total charge of the electrons which reached this target during an experiment provided one of the methods for monitoring the neutron flux reaching a sample. The (γ,n) reaction in Ta and Pb generated fast neutrons with an average energy of ≈ 1.5 MeV. A collimated neutron beam was moderated in high-pressure

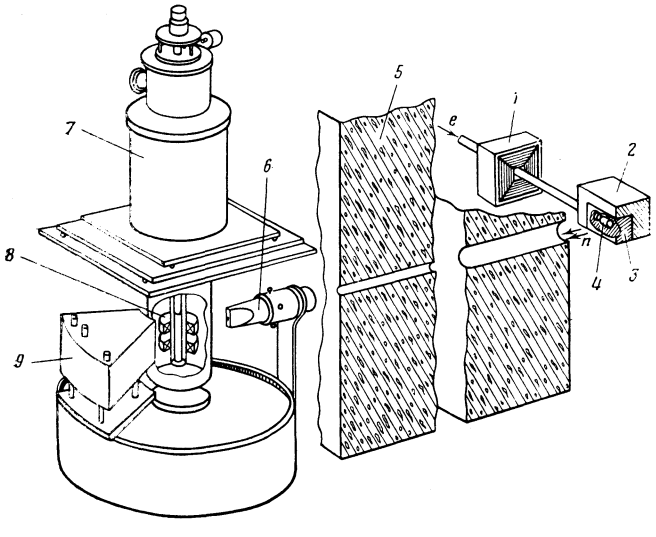

FIG. 1. General layout: 1) quadrupole lens; 2) beryllium reflector; 3) polyethylene moderator; 4) retardation target and neutron converter; 5) biological shield; 6) collimator; 7) cryostat; 8) superconducting magnet; 9) movable detector array.

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polyethylene and passed through channels in a heavy concrete biological shield 2.5 m thick into the laboratory. Immediately behind the fast neutron source on the side opposite to the channel was a beryllium reflector 30 cm thick, which enhanced the thermal neutron flux by a factor of about 1.5 (Ref. 5). The moderator was a stack of polyethylene plates 1 mm thick between which a cadmium plate 1 mm thick could be located arbitrarily (this plate caused heterogeneous poisoning of the moderator), which reduced the duration of the thermal neutron pulse and thus increased the energy resolution of the apparatus. The neutron channel was oriented at an angle of 90° to the electron beam in order to reduce the intensity of bremsstrahlung radiation in the direction of detectors.

The electron pulse length was 2.5–3.0 μsec and the repetition frequency could be varied from 50 to 800 Hz. When the moderator was not poisoned, the duration of the neutron pulse of the $\lambda = 1.0$ Å wavelength was 80 μsec (Ref. 5). The thermal neutron flux reaching a sample at a distance of 4 m from an unpoisoned source was about 5×10^3 neutrons $\cdot\sec^{-1}\cdot\text{cm}^{-2}$. The neutron beam divergence in the horizontal direction was $\approx 1.5°$.

b) Cryostat

Low-temperature neutron investigations were made in a special metal cryostat. All the parts of the cryostat in the neutron path (apart from a thin-walled inner can containing the sample) were made of aluminum. This made it possible to minimize the background representing the neutrons scattered by the cryostat. A superconducting magnet creating a vertical field up to 30 kOe in a sample was placed inside a helium bath. Two coaxial stainless-steel tubes were located inside the magnet (Fig. 2). An aluminum holder was fitted tightly to one end of the inner tube and a sample was bonded to this holder. Experience showed that bonding of samples with BG-2 or BF-4 adhesives resulted in cracking of a part of a sample at low temperatures and this produced uncontrolled rotation of the sample by angles $\approx 0.1°$. Such cracking was avoided when crystals were attached to the holder using vacuum grease or adhesive No. 88.

The temperature of a sample was measured by an uncased semiconductor thermometer. The crystal and thermometer were inside a thin-walled brass can on which two heater coils were wound bifilarly; an indium spacer provided a vacuum seal between the can and the inner tube. This tube and the sample could be rotated remotely about a vertical axis and the angle could be set to within 20′. A sample was cooled by heat transfer via gaseous helium admitted to both tubes. The temperature of the sample was stable to within 0.1 K and it could be varied from 4.2 to 300 K. Special experiments showed that the temperature drop along the sample was less than 0.1 K.

c) System for detection of scattered neutrons

The diffractometer used in our experiments was in many respects similar to the diffractometer at the Kurchatov Institute of Atomic Energy.[6] A more effective utilization of neutrons in the time-of-flight experiments was ensured by the use of multidetector systems for recording the scattered radiation. In our first experiments[3] we used detector arrays with bulk iron collimators. It was found that the high fringing fields of the superconducting magnet deflected the magnet and the sample from the vertical axis. A detector array specially designed for the study of tetragonal crystals was constructed entirely from nonmagnetizable materials and in this case there was no rotation of a crystal when a magnetic field was applied. The detector array made it possible to record simultaneously the reflections from the (100, 200) (120), and (410) planes at the scattering angles of 18, 38, and 47°, respectively. Neutron detectors were SNM-16 ^{3}He counters surrounded by boron-filled polyethylene with collimation channels. The distance between a sample and the detectors was 25 cm. The detector array could be rotated remotely as a whole and the angle could be set to within 20′. The scattered neutron background was reduced by surrounding the cryostat and the detector array with boron-filled polyethylene and cadmium shields. The whole diffractometer was placed on a steel plate supported by an air cushion which made it possible to align the diffractometer relative to the neutron channel axis. The neutron flux was monitored also with a ^{3}He counter placed in the direct beam or in some other neutron channel.

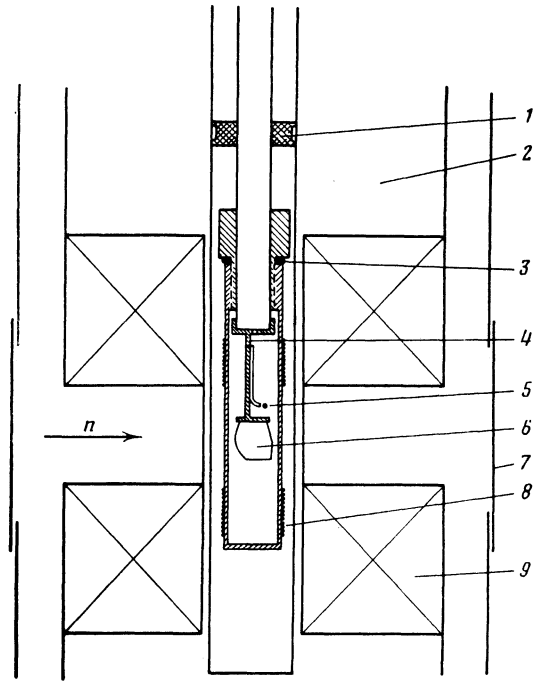

FIG. 2. Low-temperature part of the cryostat with a sample: 1) Teflon guide; 2) liquid helium chamber; 3) indium spacer; 4) aluminum sample holder; 5) semiconductor thermometer; 6) sample; 7) nitrogen screen (aluminum part); 8) heater; 9) superconducting magnet.

Pulse preamplifiers were located in the direct vicinity of neutron detectors; after amplification and discrimination, the pulses then entered a NOKIA LP 4900 multichannel analyzer. Synchronization of the operation of the electronic apparatus was provided by the same pulse which triggered the accelerator. The apparatus was protected from overloads resulting from a strong γ-ray flash by blocking the analyzer for a time of ≈ 20 μsec.

d) Sample

The initial materials used to grow single crystals were the fluorides MnF_2 and ZnF_2 formed by melting powders. The melts were prepared in a periodically replaced atmosphere (pumping out alternated with vacuum evaporation of

HF), which ensured efficient desorption of moisture from the surface and thus the purity of the initial material, and made it possible to avoid subsequently the loss of components during evacuation of the apparatus and to reduce the sorption surface of the fluorides.

The molten fluorides MnF_2 and ZnF_2 taken in suitable proportions were loaded into a conical thin-walled platinum crucible and when an external furnace was heated from room temperature to 500 °C the working chamber was evacuated periodically and filled with dry helium from a portable Dewar flask. The initial temperature for the growth of a single crystal was $\approx$1000 °C. This was followed by slow cooling at a rate of 2–3 K/h for the first 10 h and then, as the crucible left (by raising the furnace) the zone with a higher gradient, the cooling rate increased to 15 K/h. The whole process of growth of single crystals lasted approximately one day.

EXPERIMENTAL RESULTS AND DISCUSSION

1. Reduction of the ordering temperature by a random field

The antiferromagnetic ordering temperature of an $Mn_xZn_{1-x}F_2$ crystal was determined by recording the temperature dependence of a diffraction maximum representing the reflection of neutrons from the (100) plane. This dependence is shown in Fig. 3. The black symbols represent the values obtained in a magnetic field 20 kOe applied along the antiferromagnetic axis of a crystal and the open symbols are the reflection intensities observed in the absence of a field. Knowing the Néel temperature deduced from these measurements, we could find more accurately the Mn concentration x in the investigated sample. The Néel temperature of $Mn_xZn_{1-x}F_2$ compounds was described satisfactorily by the following expression obtained in the coherent potential approximation[7]:

$$T_N(x)=1{,}25T_N(1)\,(x-0.2). \qquad (1)$$

In the case of pure MnF_2 we found that $T_N(1) = 67.3$ K, whereas for our crystals the Néel temperature was $T_N(x) = 23$ K, whence we deduced that $x = 0.47$, which agreed (within the limits of the experimental error) with the concentration of the initial components used to grow a single crystal (which was 0.5).

Figure 3 is interesting because of the extremely large reduction in the transition temperature amounting to $\Delta T = T_N - T_c = 1.5 \pm 0.1$ K caused by application of a magnetic field. In the case of dilute MnF_2 the shift of T_N due to the same field was only 0.063 K. Fishman and Aharony[1], who were the first to consider the behavior of a dilute Ising antiferromagnet in an external magnetic field applied along the easy axis, showed that the boundary separating the antiferromagnetic and paramagnetic phases in the H-T diagram at low values of H should be described by the equation

$$T_c-T_N=-AH^2-BH^{2/\varphi}. \qquad (2)$$

The first term in Eq. (2), which is $-AH^2$, exists also in the case of a nondilute antiferromagnet[8] and, as already reported, in the case of MnF_2 in a field of 21 kOe it amounts to 0.063 K. The second term, $-BH^{2/\phi}$, is due to the random field and appears only for a dilute magnetic material. The coefficients A and B depend on the concentration x of the magnetic ions. In the mean-field approximation[9] the coefficient A is inversely proportional to this concentration:

$$A(x)=A(1)/x. \qquad (3)$$

Therefore, the temperature shift due to the term quadratic in the field is 0.13 K. The second term for a random field in our case is an order of magnitude higher. The measured value $\Delta T = 1.5 \pm 0.1$ K for $x = 0.47$ can be compared with recent results on thermal expansion and magnetostriction obtained by Shapira *et al.*[10] for a single crystal of $Mn_{0.75}Zn_{0.25}F_2$. In a field of 20 kOe they found that the transition temperature decreased by 0.50 K and they measured the critical index $\phi = 1.25 \pm 0.07$. Belanger *et al.*[11] reported that $\Delta T = 0.81$ K for $x = 0.65$ in the same magnetic field. However, measurements of the refractive index indicated that $\phi = 1.4 \pm 0.1$. These results were obtained for samples with high concentrations of the Mn ions and they could be extrapolated to $x = 0.47$ using the theory of Cardy[12] who calculated in the mean-field approximation how the tem-

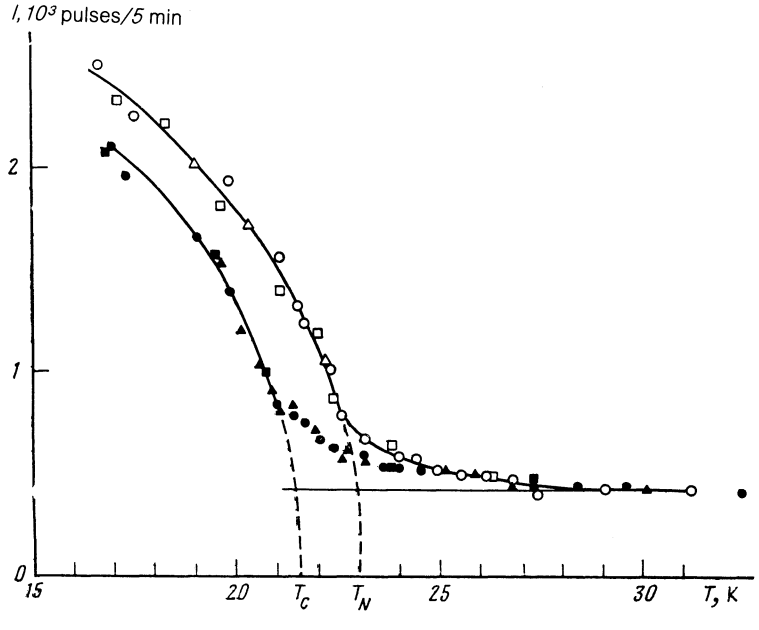

FIG. 3. Temperature dependences of the intensity of reflection from the (100) plane in a field of 21 kOe (filled symbols) and in the absence of the field (open symbols); the different shapes of the symbols represent different series of measurements.

perature of the antiferromagnetic transition should vary with x for a material with randomly substituted sites (Fishman and Aharony obtained the same dependence[1] for the problem of randomly broken bonds, which is not encountered in practice). According to Cardy, near the transition temperature, i.e., for $T \approx T_N(x) = xT_N$, we have

$$BH^{2/\Phi} \propto x[(1-x)/x^3]^{1/\Phi}. \quad (4)$$

Extrapolating the above values of ΔT from Refs. 10 and 11 in accordance with Eqs. (2)–(4) we found that the temperature shift in our single crystal should be 1.5–1.6 K, in excellent agreement with the experimental value.

It should be pointed out that the results obtained cannot be used to draw a definite conclusion about the value of the critical exponent ϕ. The difference between the behavior of the experimental field dependence for $\phi = 1.25$ and $\phi = 1.4$ was of the order of the experimental error.

2. Hysteresis in a system with a random magnetic field

Imry and Ma[13] were the first to use simple phenomenological considerations to show that a two-dimensional Ising ferromagnet should split into domains in a random magnetic field no matter how weak. On the other hand, a three-dimensional ferromagnet remains stable in the presence of a random field, i.e., the long-range order is not destroyed. The model of Imry and Ma has been considered theoretically by many authors employing more elaborate methods. Some of them reached the conclusion that d_l is the lower critical dimensionality (dimensionality of the space above which a long-range magnetic order should appear in a random field) is 3, whereas other authors confirmed the results of Imry and Ma that $d_l = 2$ (see, for example, a review in Ref. 14).

The first neutron diffraction experiments[15] carried out on a three-dimensional Ising antiferromagnet $Co_{0.3}Zn_{0.7}F_2$ demonstrated destruction of the long-range order and it was therefore concluded that $d_l = 3$. However, the subsequent experiments on dilute Fe and Mn fluorides demonstrated that the situation is more complicated. It was found that when a magnetic field was applied to a sample at a low temperature, the state of the sample depended strongly on its previous history. If a sample was cooled from the paramagnetic phase in a magnetic field (cooling in a field), then the frozen-in state was of the polydomain type as demonstrated by broadening of the diffraction peaks, but if cooling took place in the absence of a field (zero-field cooling) and the field was applied below T_N, the long-range order was not destroyed.

Recent theoretical investigations[16] suggest that $d_l = 2$, i.e., that the ground state can be of the single-domain type and that the state reached by cooling in a field may be metastable.[17] The average domain size is in this case $L \propto H^{-2}$ and it rises logarithmically with time:

$$L \propto \ln(t/\tau), \quad (5)$$

where $\tau \approx 10^{-11}$ sec.

In our experiments the diffractometer resolution was two orders of magnitude poorer than in the cited experiments carried out using a high-flux reactor in Brookhaven and, therefore, we observed no broadening of the diffraction peaks. On the other hand, a determination of the intensity of the (100) magnetic reflection alone enabled us to follow the appearance and relaxation of magnetic domains in an $Mn_{0.47}Zn_{0.53}F_2$ single crystal when the external magnetic field was varied.

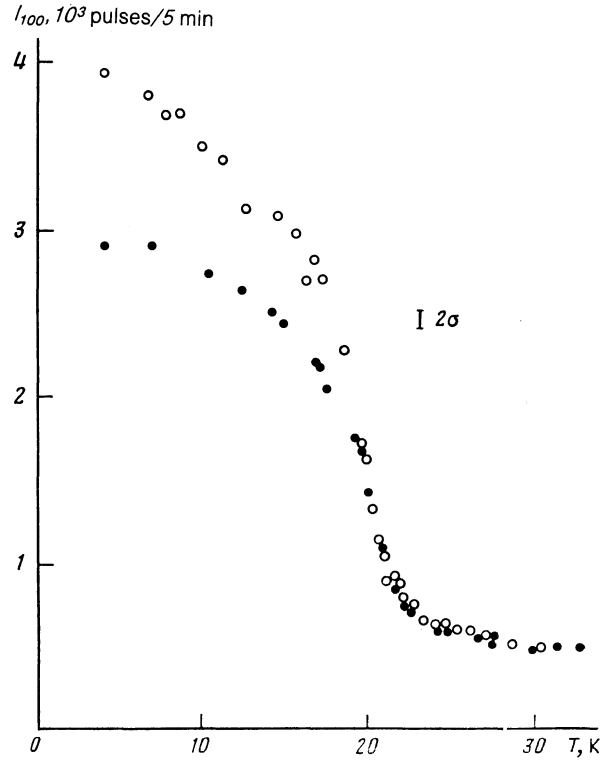

FIG. 4. Temperature dependences of the (100) reflection intensity in a field of 21 kOe obtained after cooling in a field (open symbols) and after zero-field cooling (filled symbols).

It was found that the intensity of the (100) reflection after cooling in a field was stronger than after zero-field cooling (Fig. 4) and the intensity of this reflection on reduction of the field to zero after cooling in a field was the same as after zero-field cooling. The (200) nuclear reflection was unaffected in all cases. A similar increase of the intensity of the magnetic reflection had been observed earlier for $Mn_xZn_{1-x}F_x$ compounds with $x = 0.65$ and 0.75 (Refs. 18 and 19). The intensity of the Bragg reflection by perfect crystals (those with a weak mosaic structure) was determined not only by the structure factor, but to a large extent also by the magnitude of the extinction which suppressed the reflection intensity. The extinction itself depended on the mosaic structure of the crystal, i.e., on the angular scatter of the orientations of crystalline blocks and on the size of these blocks. When a magnetic system split into domains which scattered neutrons incoherently, the extinction of the magnetic reflection should decrease and the intensity should rise. The intensity of the (200) nuclear reflection should not be affected.

Figure 5 shows the changes in the intensity of the (100) reflection on reduction of the magnetic field from 25.5 kOe to 0 (open symbols) and during the subsequent increase of the field (black symbols). The initial state was obtained by cooling a sample from the paramagnetic phase to 4.2 K in a field of 25.5 kOe. A reduction of the field caused domain growth as manifested by a reduction in the (100) reflection intensity. In $H = 0$ the structure rapidly relaxed (in a time less than 5 min, representing the time needed for one mea-

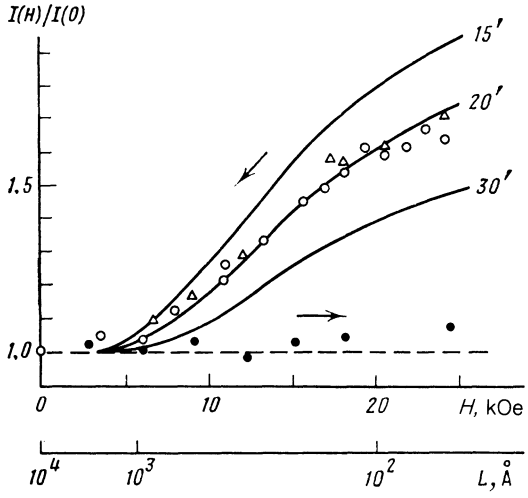

FIG. 5. Ratio of the (100) reflection intensity obtained after cooling in a field of 25.5 kOe from 35 to 4.2 K to the intensity of reflection in $H = 0$. The open symbols represent reduction of the field from 25.5 kOe and the filled symbols represent an increase from $H = 0$. The continuous curves are the calculated increments in the intensity due to a reduction of secondary extinction in crystals with mosaic angles 15′, 20′, and 30′. The abscissa gives also the domain size calculated from Eq. (6) for $L_0 = 10^4$. The different symbols represent different series of measurements.

surement) to the ground single-domain state. A subsequent increase of the field did not destroy the long-range order: the intensity of the (100) reflection remained constant. After the crystal had been cooled in zero field its state was not affected by variation of the field. It was interesting to note such a dependence of the low-temperature state of a magnetic material on the random field was exhibited only by some dilute systems. For example, the magnetic state of $Fe_xZn_{1-x}F_2$ cooled in a field remained as stable as the state produced by zero-field cooling. This difference was clearly due to the much greater energy gap in the spectrum of spin waves of $Fe_xZn_{1-x}F_2$ than that of $Mn_xZn_{1-x}F_2$ (in the case of MnF_2 it was an order of magnitude less than for FeF_2). Therefore, a sufficiently large number of low-energy excitations facilitated relaxation of domain walls in $Mn_xZn_{1-x}F_2$ even at $T = 4.2$ K. In the case of $Fe_xZn_{1-x}F_2$ the domains were practically frozen-in.

Three curves for crystals with different mosaic structures are plotted in Fig. 5 to compare the observed increase in the (100) reflection in a magnetic field with the calculated value of the extinction. The extinction coefficient was calculated using the Zachariasen formula[20] postulating a Gaussian distribution of mosaic blocks. In zero field when the samples were in the single-domain state, the extinction coefficient was governed by the block nature of the crystal structure, i.e., by the block size L and the mosaic scatter of the orientations which for real crystals ranged from 1° to 0.01°. The experiments of Cowley *et al.* on $Fe_{0.35}Zn_{0.65}F_2$ crystals[21] indicated that, in agreement with the theoretical predictions, the domain size decreased as H^{-2} and in high fields the extinction coefficient was governed by the domain dimensions. Therefore, a reasonable approximation for the effective size of a block L in the calculation of the extinction coefficient of the magnetic reflection as a function of the field was the expression

$$L = L_0/(1+H^2), \qquad (6)$$

where L is in units of the crystal lattice constant and H is in kilo-oersted. Equation (6) describes well the results of Aleksandrov *et al.*[20] obtained for $L_0 = 10^5$. Our results (Fig. 5) were described well by $L_0 = 10^4$ and a mosaic angle 20′. An increase in I_{100} by a factor of 1.7 in a field of 25 kOe could be obtained by calculation also on the assumption that $L_0 = 10^5$, as in Ref. 21, and that the mosaic angle was $\approx 5'$, but this provided a poorer description of the whole $I_{100}(H)$ curve. A tenfold increase in the intensity in such a field, reported in Ref. 21, was attributed to a very small mosaic angle of $\approx 0.01°$ of the crystal used by Cowley *et al.*[21]

In the case of our crystal the state of the magnetic structure was very stable both after cooling in a field and after zero-field cooling. We observed no changes in the reflection intensity in either case for 2 h in a fixed external magnetic field. Equation (5) could be used to estimate the change in the average domain size L in the time interval from 10 min to 2 h; it amounted to 7%. As can be seen from Fig. 5, such an increase in L could not result in a significant change of I_{100} in our experiments.

As pointed out already, it follows from recent theoretical investigations that the ground state of a dilute antiferromagnet in a random field should be of the single-domain nature, but there may be a large number of metastable states which should relax slowly (at a logarithmic rate) to the ground state. However, these theories fail to account for the origin of a metastable polydomain state observed experimentally. This is clearly due to the fact that all these theoretical investigations are based on the model of Imry and Ma of a ferromagnet in a random field and do not consider the real situation in the case of a dilute antiferromagnet. Yoshizawa and Belanger[22] carried out a numerical modeling and showed that in the case of a dilute antiferromagnet there is a narrow range of temperatures near T_c where the formation of a polydomain state is favored by the energy considerations. In a dilute antiferromagnet a domain wall passes through sites with the largest number of broken bonds. Therefore, cooling increases the magnetization at these sites grows more slowly than at the sites inside a domain and the average value of the entropy per spin in a domain wall is greater than per spin inside a domain. Consequently, the free energy of a polydomain state is less than the free energy of a single-domain state at temperatures $T_c < T < T_N$. When temperature is lowered still further, the reduction in the Zeeman energy cannot compensate for the increase in the energy of domain walls and a single-domain state becomes energetically preferable at $T = 0$.

CONCLUSIONS

The method of inelastic scattering of thermal neutrons was used to investigate the behavior of a dilute easy-axis antiferromagnet $Mn_{0.47}Zn_{0.53}F_2$ in a magnetic field applied parallel to the easy axis. The experimentally determined shift of the ordering temperature amounted to 1.5 ± 0.1 K in a field of 21 kOe. A major change in the intensity of the reflection by a magnetic structure was observed when a crystal was cooled from the paramagnetic to the antiferromagnetic phase in the presence of a magnetic field, which indicated formation of a domain structure. Estimates indicated that in a field of 25 kOe the effective size of such domains was

approximately 100 times less than the effective size of blocks in the original crystal.

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