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High-field magnetization steps and the nearest-neighbor exchange constant in $Cd_{1-x}Mn_xS$, $Cd_{1-x}Mn_xTe$, and $Zn_{1-x}Mn_xSe$

Y. Shapira
Francis Bitter National Magnet Laboratory and the Department of Physics, Massachusetts Institute of Technology,
Cambridge, Massachusetts 02139

N. F. Oliveira, Jr.
Francis Bitter National Magnet Laboratory, Massachusetts Institute of Technology,
Cambridge, Massachusetts 02139
and Instituto de Física, Universidade de São Paulo, Caixa Postal 20516, 05508 São Paulo, Brazil

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High-field magnetization steps due to energy-level crossings for pairs of nearest-neighbor Mn spins were observed in $Cd_{1-x}Mn_xS$, $Cd_{1-x}Mn_xTe$, and $Zn_{1-x}Mn_xSe$. For $Cd_{1-x}Mn_xTe$, two steps were observed. The difference H_2-H_1 between the magnetic fields at the centers of these steps gives $J_{NN}/k_B=-6.1\pm0.3$ K for the exchange constant between nearest-neighbor Mn spins. For $Cd_{1-x}Mn_xS$ and $Zn_{1-x}Mn_xSe$, only the first step at H_1 was observed. The values of H_1 give the estimates $J_{NN}/k_B\cong-10.5$ K for $Cd_{1-x}Mn_xS$, and $J_{NN}/k_B\cong-12.6$ K for $Zn_{1-x}Mn_xSe$. Our results for J_{NN} in the three materials are in good agreement with other recent determinations. The magnitudes of the magnetization steps in all three materials are consistent with those expected from a random distribution of Mn ions over the cation sites in the crystals.

I. INTRODUCTION

High-field magnetization steps in dilute magnetic semiconductors (DMS's) were discovered several years ago.[1,2] The magnetization steps arise from energy-level crossings for pairs of nearest-neighbor (NN) magnetic ions.[1-4] The steps are observed at low temperatures in materials in which the concentration x of magnetic ions is equal to several percent, typically. Most of the work, until now, has been on steps which arise from NN pairs of Mn ions in II-VI DMS's. The magnetization steps in these materials yielded the exchange constant J_{NN} for NN Mn spins, and they also gave the percentage of Mn ions which are in NN pairs. The latter percentage agreed with that calculated on the assumption that Mn ions are randomly distributed over the cation sites.

Most studies of the magnetization steps employed one of the following two methods: direct measurements of the magnetization[1-3,5] and reflectivity measurements which yielded the magnetization indirectly.[2,4,6] In the present work we used direct magnetization measurements to observe the steps in three materials: (1) $Cd_{1-x}Mn_xS$, in which the steps have never been observed before, (2) $Cd_{1-x}Mn_xTe$, in which the magnetization steps have been observed by the reflectivity method,[4] but not in direct magnetization measurements, and (3) $Zn_{1-x}Mn_xSe$, in which a magnetization step has been studied earlier,[1] but with a lower precision. The higher precision of the present measurements is largely due to the use of new magnetometers. These magnetometers measure the magnetic force on the sample (in a magnetic field gradient) by a sensitive capacitance technique. This method, unlike the one used earlier,[1,3] yields a continuous trace of the magnetization M as a function of magnetic field H, and it is also adaptable for use in ^{3}He temperatures where the magnetization steps are sharper than in ^{4}He temperatures. The data which were obtained were used to determine the exchange constant J_{NN} in the three DMS's.

The original model for the magnetization steps[1-4] ignored all exchange interactions other than between NN's. This simple model leads to five magnetization steps due to NN pairs, which occur at fields H_r given by

$$g\mu_B H_r = 2\,|\,J_{NN}\,|\,r\ , \tag{1}$$

where $r=1$, 2, 3, 4, 5, g is the g factor, and μ_B is the Bohr magneton. For Mn ions in the present three DMS's the difference between g and 2.000 is less than 0.4%.[7] The exchange constant J_{NN} which appears in the present paper corresponds to an interaction $-2J_{NN}\mathbf{S}_1\cdot\mathbf{S}_2$ between NN spins $\mathbf{S}_1$ and $\mathbf{S}_2$. (Other conventions for J_{NN}, which differ by a minus sign and/or by a factor of 2, are sometimes used in the literature.)

The five magnetization steps are superimposed on the magnetization due to spins which are not in NN pairs. A schematic of the total magnetization M in the field region up to the second step is shown in Fig. 1. This schematic is based on a model which includes NN interactions only,[3] but it is not changed qualitatively when other exchange interactions (assumed to be much weaker) are included. In many experiments the first two magnetization steps are superimposed on a nearly constant magnetization due to spins which are not in NN pairs. The constant value is referred to as the technical saturation value M_s.[1,3] However, in some experiments (depending on the material and the operating temperature) the "background" magnetization on which the steps are superimposed is still varying appreciably in the region of the first magnetization step.

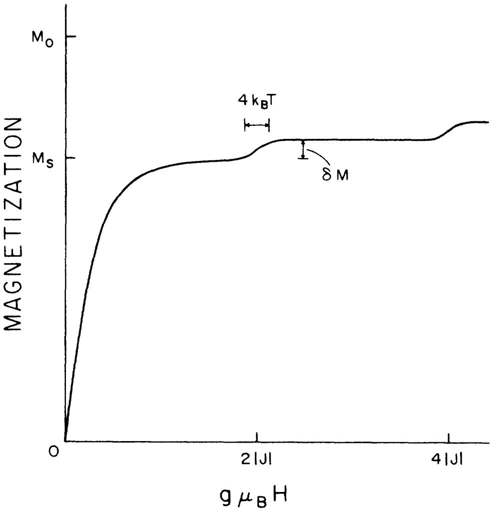

FIG. 1. Sketch of the predicted magnetization curve at low temperatures for a dilute magnetic semiconductor with a low x. Here, H is the magnetic field, M_s is the technical saturation value, M_0 is the true saturation value in the limit $H \rightarrow \infty$, and J is the nearest-neighbor exchange constant J_{NN}. This sketch is based on a model in which only nearest-neighbor interactions are included (after Ref. 3).

Following the early model for the magnetization steps,[1-4] a more refined model was developed by Larson *et al.*[8] This model includes both NN exchange interactions and interactions between more distant neighbors. The main new result is that interactions between more distant neighbors leads to a shift of H_r. This shift, Δ, is the same for all five steps, i.e.,

$$g\mu_BH_r = 2\,|J_{NN}|\,r + \Delta \ . \qquad (2)$$

The exchange constant J_{NN}, which is negative (antiferromagnetic) for all known II-VI DMS's, is therefore related to the difference between the fields at two successive steps, e.g.,

$$g\mu_B(H_2 - H_1) = 2\,|J_{NN}| \ . \qquad (3)$$

This result can be used to obtain J_{NN} when at least two magnetization steps are observed. However, often the available magnetic-field range permits the observation of only the first step. In that case an *estimate* for J_{NN} can be obtained by ignoring Δ, which leads to the relation

$$g\mu_BH_1 \cong 2\,|J_{NN}| \ . \qquad (4)$$

The shift Δ is a function of the Mn concentration x and the exchange interactions between distant neighbors. For low x, Δ decreases with decreasing x, and it vanishes in the limit $x \rightarrow 0$. The available information for the exchange constants in wide-gap II-VI DMS's suggests that for $x \leq 0.03$, Eq. (4) overestimates J_{NN} by no more than 10%.[3,8] For the $Cd_{1-x}Mn_xTe$ sample which was used in the present experiments, with $x = 0.05$, the data indicate that Eq. (4) overestimates J_{NN} by about 15%. However, it is believed that in many other II-VI DMS's with the same value of x, Eq. (4) would lead to a better estimate for J_{NN}.[3,8]

In summary, when two (or more) magnetization steps are observed, an accurate value for J_{NN} can be obtained from Eq. (3). When only one step is observed, an estimate for J_{NN} can be obtained from Eq. (4). For $x \leq 0.03$ this estimate is expected to be better than 10% in most cases.

The theory for the magnetization steps also makes predictions regarding the magnitude and shape of each step. These will be discussed in connection with the data analysis.

II. EXPERIMENTAL TECHNIQUES

A. Magnetometer

The magnetometer measured the magnetic force F_m on the sample. The sample was located on the axis of the bore of a Bitter magnet, but at some distance d from the magnet's center. The force F_m, which attracted the sample to the magnet's center, was proportional to the product of the magnetization M and the field gradient $\partial H/\partial z$ along the bore's axis. For the magnets which were used, $\partial H/\partial z$ (at a given d) was proportional to H. Thus, the ratio F_m/H was proportional to M. The proportionality constant was not determined in the present experiment. All the results for M are therefore in arbitrary units.

The force F_m was measured by a capacitance method, which was a modification of the one used earlier by Brooks *et al.*[9] The sample was mounted on a thin (0.1-mm) plate of fused quartz which was separated by a 0.1-mm gap from a thick (1-mm) plate also of fused quartz. The separation between the plates was maintained by two crescent-shaped slivers of fused quartz which were inserted between the plates on two opposite sides. The slivers were attached to the plates by G.E. 7031 varnish. With this arrangement the separation between the plates in the region between the two slivers was a function of the magnetic force F_m. That is, the magnetic force caused the thin quartz plate to bend, which changed the average separation between the two plates. The opposing surfaces of the two plates were gold plated, and formed a capacitor. Under the action of the magnetic force F_m, the capacitance C changed by ΔC. The change ΔC in these experiments was smaller than 1% of C. It was assumed that for these small changes, ΔC was proportional to F_m. The data for ΔC versus H were acquired automatically, and the ratio $\Delta C/H$ was obtained using a minicomputer. In all the experiments the capacitor was immersed in liquid helium (^4He or ^3He), so that liquid He was present between the two plates. No problems due to the possible bubbling of liquid ^3He were encountered (the operation in ^4He was below the λ point, where no bubbling occurs). One problem which was encountered was the deterioration of the bonds formed by the G. E. varnish on repeated thermal cycling from room temperature to liquid helium. Usually, these bonds were stable only for a few thermal cycles, but this was adequate for our purpose.

Ideally the ratio $\Delta C/H$ should be proportional to the magnetization M of the sample. In practice, however, a small background was present. This background was, at least in part, due to the diamagnetism of the thin quartz plate and the rubber cement which held the sample to this plate. In addition, the diamagnetism associated with the lattice of the sample should have also contributed to F_m, so that the magnetic force was not solely due to the magnetization of the Mn spins. In all cases, however, the small background did not obscure the magnetization steps, and it produced a negligible error in the determination of the field H_r at the center of the rth step.

The magnetic field $H_0 \equiv H(d=0)$ at the center of the magnet was known to an accuracy of 0.25%. To determine the field H_P at the sample position P (which was at a distance d from the magnet's center) we used two different methods. The first was based on the fact that the magnetic force vanishes at the magnet's center, and it reverses its sign as the sample is moved from above to below the center. By monitoring the signal as a function of sample position, the position which corresponded to $d=0$ was located to within 2 mm. The distance d at any other sample position was then known to the same accuracy. Using the known field profile of the magnet (measured independently), the ratio H_P/H_0 was determined.

The second method utilized the magnetoresistance of a small Allen-Bradley carbon resistor which was placed at a fixed known distance above the sample. The magnetoresistance of this resistor was first measured at 4.2 K after the resistor was moved to the sample position P. These measurements were performed for a set of values I_j of the current through the magnet. Subsequently, the resistor was moved to the magnet's center, and the same values of the magnetoresistance (at the same temperature) were reproduced, but with different currents I_j' through the magnet. With this procedure $H_P(I_j)=H_0(I_j')$. For the magnets which we used, $H_0(I_j')$ was proportional to I_j'. Therefore, for a given current through the magnet, $H_P/H_0=I_j'/I_j$. The measured ratios I_j'/I_j for a given sample position, were always independent of I_j to within the experimental accuracy. Moreover, they were always in agreement with the ratio obtained by the first method of determining H_P/H_0. As a result, the ratio H_P/H_0 was known to within 0.5%, so that the total uncertainty in H_P was less than 0.75%.

Data were usually taken at several sample positions, with distances $d \leq 4$ cm from the magnet's center. The results at the different positions were in good agreement with each other. Because the field gradient increased with d, a better signal-to-noise ratio was obtained at a larger d. On the other hand, the maximum field which could be obtained with a given magnet decreased with increasing d.

B. Samples

All the samples were single crystals grown by the Bridgman method. The $Cd_{1-x}Mn_xTe$ sample was from the same boule as the sample in Ref. 4. This boule was kindly provided by R. R. Galazka. The nominal Mn concentration in this boule was $x=0.05$. Atomic absorption analysis[10] of a portion of the sample which we used gave $x=0.047$. The mass of this sample was 104 mg.

The $Zn_{1-x}Mn_xSe$ and $Cd_{1-x}Mn_xS$ samples were cut from boules obtained from Eagle-Picher Industries. The $Zn_{1-x}Mn_xSe$ sample was the same as that in Ref. 1. The Mn concentration (from atomic absorption) was $x=0.033$, and the mass was 225 mg. The $Cd_{1-x}Mn_xS$ sample was cut from the same boule which was used in Ref. 11. The mass of the sample was 377 mg. Atomic absorption analysis of a portion of this sample a gave $x=0.023$, which agrees with the earlier result.[11]

III. RESULTS AND DISCUSSION

A. $Cd_{1-x}Mn_xS$

The magnetization of $Cd_{1-x}Mn_xS$, with $x=0.023$, was measured at 1.25 K. The first magnetization step was observed clearly. This is illustrated by the data in Fig. 2, which shows the upper portion of one of the magnetization traces. Note the slight decrease of the measured magnetization in fields just below the magnetization step. This decrease is due to the small background discussed in Sec. II.

The field H_1 at the center of the first magnetization step was obtained by two methods. First, the field where the step reached half of its total size was obtained by inspecting the upper portion of the magnetization curve. This determination was accurate to within 3 kOe, typically. The second method was based on the derivative $\partial M/\partial H$, which was obtained by a numerical differentiation of the data. The field H at the peak of this derivative was identified as H_1. The accuracy of this determination of H_1 was also ± 3 kOe, typically. For each trace of the magnetization curve the two methods of determining H_1 agreed to within the experimental accuracy. The average of the two values was then used. The same method of analysis was applied to the 9 magnetization traces which were taken. Based on the overall average, and taking into account the uncertainty in the field H_P at the position of the sample, we obtained $H_1=157\pm 4$ kOe. Using Eq. (4) we then obtained $J_{NN}/k_B=-10.5\pm 0.3$ K, where k_B is the Boltzmann constant. As explained in Sec. I this value of J_{NN} is only an estimate, because it ignores exchange interactions between distant neighbors. These exchange interactions are known to be predominantly antiferromagnetic,[12] so that the true magnitude of J_{NN} should be slightly lower.[8] The difference between the true value of J_{NN} and the estimate we obtained from Eq. (4) is expected to be smaller than 8%. The last statement is based on the discussion in Sec. I and the value of x in this particular sample. Recent Raman scattering experiments[13] gave $J_{NN}/k_B=-10.6\pm 0.2$ K, which is very close to our value. Thus, the estimated uncertainty of 8% quoted above is probably overconservative (see Note added in proof).

The magnitude of the magnetization step will be designated as δM (see Fig. 1). This δM was obtained from several magnetization traces. The small background, observed in fields below the step, was taken into account by extrapolation. The results indicate that the ratio between δM and the value of the magnetization in the region immediately preceding the step is 0.052 ± 0.006. In the present case, the magnetization just before the onset of the step corresponds to the technical saturation value M_s (see Fig. 1). Thus, $\delta M/M_s=(5.2\pm0.6)\times10^{-2}$. The theoretical prediction for $\delta M/M_s$ is contained in Eqs. (8) and (10) of Ref. 3, which express $\delta M/M_s$ in terms of the probabilities of finding a Mn ion in various types of clusters. Using the probabilities which follow from the assumption that the Mn ions are randomly distributed over the cation sites, these equations give $\delta M/M_s=4.7\times10^{-2}$ for $x=0.023$. This prediction agrees with the experimental result to within the experimental error. It is noteworthy that the theory in Ref. 3 does not take into account exchange interactions between distant neighbors. However, the prediction for $\delta M/M_s$ remains unchanged when these interactions are included.

Two known mechanisms contribute to the width ΔH of the magnetization step. First, there is a thermal broadening $\Delta H=4k_BT/g\mu_B$, corresponding to the field interval where the magnetization (relative to the value before the onset of the step) rises from $0.12\delta M$ to $0.88\delta M$. This is discussed in Ref. 3, and is shown in Fig. 1. The detailed shape of the magnetization step, assuming thermal broadening only, is given by Eq. (12) of Ref. 3. The second broadening mechanism is due to exchange interactions with distant Mn neighbors.[8] These interactions lead to a distribution of fields at which the energy levels of different NN pairs cross. This second source of step broadening is more readily observed at very low temperatures, where the thermal broadening is very small.

An analysis of the shape of the magnetization step in Fig. 2 indicates that the width of the magnetization step at this (relatively high) temperature is dominated by thermal broadening. A least-squares fit which assumed thermal broadening only, but with an effective temperature T_{eff} instead of the true temperature $T=1.25$ K, gave $T_{\text{eff}}=1.67$ K. In this fit, the small background (observed in fields below the magnetization step) was taken into account by extrapolation. If this background were neglected, T_{eff} would have been much closer to the actual temperature T.

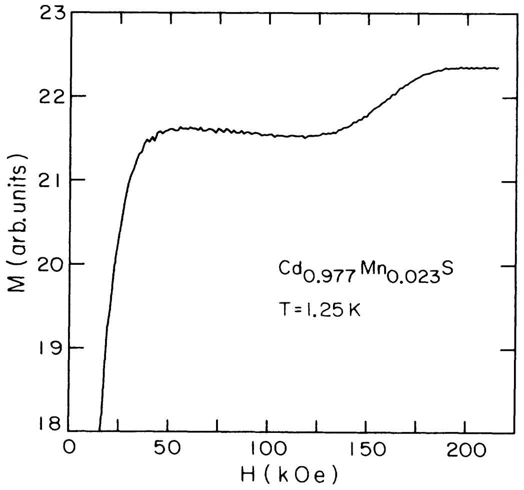

FIG. 2. Upper portion of the magnetization curve of $Cd_{0.977}Mn_{0.023}S$.

B. $Cd_{1-x}Mn_xTe$

Our first measurements on $Cd_{1-x}Mn_xTe$, with $x=0.047$, were carried out at 1.28 K. The results showed the existence of two magnetization steps at high magnetic fields. An example of these data is shown in Fig. 3. The determination of the field H_1 (at the center of the first step) was somewhat difficult in this case because the first step was superimposed on a field-dependent magnetization due to spins in clusters other than NN pairs. This situation arose primarily because the field H_1 in $Cd_{1-x}Mn_xTe$ is unusually low, as compared with other II-VI DMS's with Mn. To overcome this problem, the fields H_1 and H_2 were determined only from the peaks in the derivative $\partial M/\partial H$, obtained by a numerical differentiation of the data. The contribution of the "background" magnetization (due to clusters other than NN pairs) to this derivative was small, and it was estimated and subtracted. The fields H_1 and H_2 were obtained after this subtraction. The results, averaged over several magnetization traces, gave $H_1=104.6\pm4$ kOe and $H_2=194\pm4$ kOe.

The earlier magnetoreflectance work on the steps in the same crystal[4] gave $H_1=115\pm5$ kOe and $H_2=195\pm10$ kOe. Because this earlier value for H_1 was somewhat higher than the one we obtained from the data at 1.28 K, additional magnetization data were taken between 0.43 and 0.47 K. At these lower temperatures the steps were sharper than at 1.28 K, and the "background" magnetization was more nearly saturated by the time the field H_1 was reached. An example of the low-temperature magnetization data is shown in Fig. 4. From several magnetization traces between 0.43 and 0.47 K we obtained $H_1=104.0\pm3$ kOe, and $H_2=195.5\pm4$ kOe. These values were based on the derivative $\partial M/\partial H$ (after background subtraction), but were also consistent with estimates of the middles of the magnetization steps.

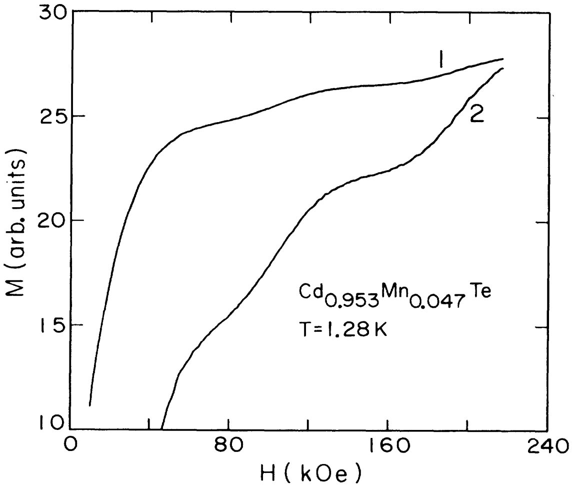

FIG. 3. Curve 1: Upper portion of the magnetization curve of $Cd_{0.953}Mn_{0.047}Te$ at 1.28 K. Curve 2: Expanded view of the magnetization steps. The ordinate scale applies only to curve 1.

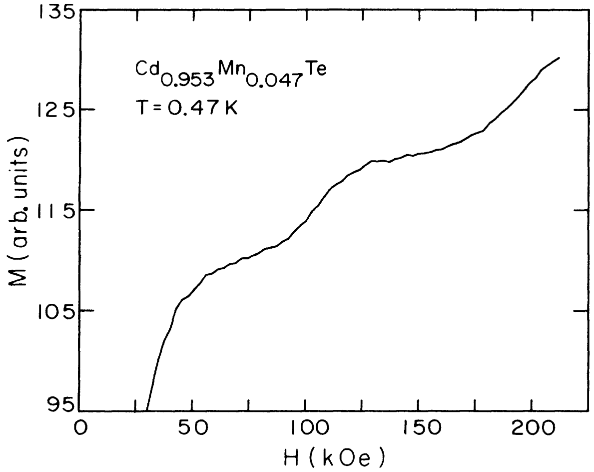

FIG. 4. Upper portion of the magnetization curve of $Cd_{0.953}Mn_{0.047}Te$ at 0.47 K.

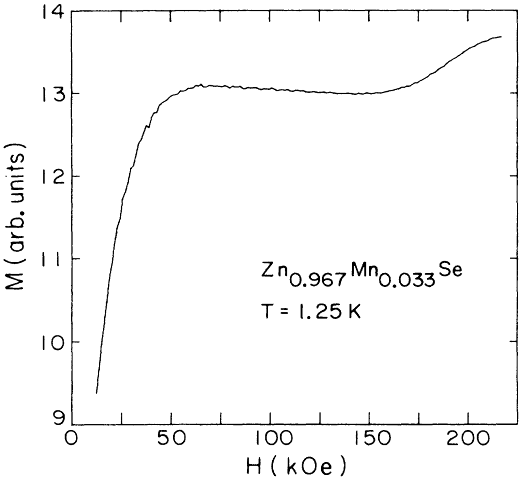

FIG. 5. Upper portion of the magnetization curve of $Zn_{0.967}Mn_{0.033}Se$.

The data at 1.28 K gave $H_2-H_1=89.4\pm6$ kOe, whereas those at ^{3}He temperatures gave $H_2-H_1=91.5\pm5$ kOe. Combining these values, $H_2-H_1=90.5\pm4$ kOe. Using Eq. (3) this leads to $J_{NN}/k_B=-6.1\pm0.3$ K. Because this value is based on the difference H_2-H_1, it is independent of the exchange interactions between distant neighbors. Other recent values for J_{NN}/k_B are -6 K from analysis of specific-heat and neutron diffraction data,[14] and -6.9 ± 0.15 K from the Curie-Weiss temperature (ignoring interactions between distant spins).[15] We believe that our result for J_{NN} is the most accurate to date.

The magnitude δM of the first magnetization step was estimated from the data at ^{3}He temperatures. The results, averaged over several magnetization traces taken in different experimental runs, gave $\delta M/M_s=(7\pm1.5)\times10^{-2}$. The large uncertainty is due to the background. The theoretical value for $x=0.047$, assuming a random distribution of Mn spins, is $\delta M/M_s=7.8\times10^{-2}$.

The widths of the magnetization steps at $0.43\leq T\leq0.47$ K were larger than those expected from thermal broadening alone. The shape of the first magnetization step at these temperatures, was compared with that expected from thermal broadening with an effective temperature $T_{\rm eff}$. The background on which the step was superimposed was estimated and subtracted. The results showed that $T_{\rm eff}$ was approximately twice the actual temperature.

C. $Zn_{1-x}Mn_xSe$

Magnetization data on $Zn_{0.967}Mn_{0.033}Se$ were taken at 1.25 K. The first magnetization step was observed clearly. An example of the data is shown in Fig. 5. The small decrease of the magnetization between 75 and 140 kOe is due to experimental background, as discussed in Sec. II. The field H_1 was determined from the peak in the derivative $\partial M/\partial H$. (The background magnetization, which is linear in H, has no effect on the position of the peak in the derivative.) The results, averaged over several sets of data, gave $H_1=187\pm4$ kOe. This value is more accurate than the value $H_1\cong190$ kOe obtained earlier for the same sample.[1] Using Eq. (4) we then obtain the estimate $J_{NN}/k_B=-12.6$ K. From the discussion in Sec. I we expect that this estimate for J_{NN} agrees with the true value of J_{NN} to within 10%. A recent determination of J_{NN} from inelastic neutron scattering[16] gave $J_{NN}/k_B=-12.3$ K. This result suggests that our estimate for J_{NN} is, in fact, much better than 10% (see Note added in proof).

The magnitude of the magnetization step was determined from several magnetization traces, after a background subtraction. The experimental result $\delta M/M_s=(6.4\pm1)\times10^{-2}$ agrees with the theoretical prediction $\delta M/M_s=6.2\times10^{-2}$ which assumes a random Mn distribution.

The width of the magnetization step was comparable to that expected from thermal broadening alone. A least-squares fit of the magnetization step in Fig. 5 to the line shape expected from thermal broadening with an effective temperature gave $T_{\rm eff}=1.57$ K. In this fit the small experimental background was subtracted. The result for $T_{\rm eff}$ should be compared with the actual temperature 1.25 K. The best values for the other variable parameters in the fit (H_1 and $\delta M/M_s$) agreed with those quoted above to within the uncertainties.

Note added in proof. Very recently Barilero *et al.*[17] proposed a method estimating the parameter Δ in Eq. (2) from the shape of the magnetization curve in fields well below the first step. The standard fit of the magnetization in this field range to the modified Brillouin function

$$M=M_sB_{5/2}[5\mu_BH/k_B(T+T_0)] \qquad (5)$$

yields the parameter T_0. According to Barilero *et al.*,

$$\Delta\cong2k_BT_0/(S+1)\ , \qquad (6)$$

where $S=5/2$ for Mn ions. When only the first magnetization step is observed, one may use the values of H_1 and T_0 together with Eqs. (2) and (6) to obtain a better estimate for J_{NN} than that obtained from Eq. (4). The values of T_0 for the three samples investigated here are known.[4,11,18] For $Cd_{0.977}Mn_{0.023}S$, Eqs. (2) and (6), with $H_1 = 157$ kOe and $T_0 = 0.65$ K, give $J_{NN}/k_B = -10.36$ K, as compared with -10.55 K from Eq. (4). For $Zn_{0.967}Mn_{0.033}Se$ ($H_1 = 187$ kOe, $T_0 = 1.1$ K), Eqs. (2) and (6) give $J_{NN}/k_B = -12.2$ K, as compared with -12.6 K from Eq. (4). For $Cd_{0.953}Mn_{0.047}Te$ ($H_1 = 104.3$ kOe, $T_0 = 2.04$ K), Eqs. (2) and (6) give $J_{NN}/k_B = -6.4$ K, which is an improvement on the value -7.0 K obtained from Eq. (4) but is not as accurate as -6.1 K obtained from Eq. (3).

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