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Phase diagram and magnetic properties of the diluted fcc system $Ni_pMg_{1-p}O$

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Starting from Ni and Mg nitrates, about 20 samples of $Ni_pMg_{1-p}O$ samples (with $0.06 \le p \le 0.86$) were prepared and x-ray-diffraction studies showed the samples to have the NaCl structure with the lattice constant fitting the expression $a(p) = 4.2115 - 0.0340p$ Å. Temperature-dependent magnetic-susceptibility (χ) studies of the samples were carried out between 1.8 and 600 K using a superconducting-quantum-interference-device magnetometer and Néel temperatures T_N's were determined from the peak in $\partial(\chi T)/\partial T$. The variation of $t = T_N(p)/T_N(1)$ vs p is compared with that of $Co_pMg_{1-p}O$. For both systems, the variations for $p > 0.31$ are found to fit the predicted values for a simple-cubic Heisenberg antiferromagnet and a theoretical basis for this anomalous result is advanced. The experimental percolation threshold $p_c = 0.15 \pm 0.01$ and for $p_c < p < 0.31$, χ below T_N shows irreversible behavior for the zero-field-cooled and field-cooled cases, suggestive of spin-glass-like behavior also observed in other diluted fcc antiferromagnets, such as $Co_pMg_{1-p}O$ and $Eu_pSr_{1-p}Te$. It is suggested that the differences in the t-vs-p variations for $p < 0.31$ in $Ni_pMg_{1-p}O$, $Co_pMg_{1-p}O$, and $Eu_pSr_{1-p}Te$ may be related to the differences in the ratio of the next-nearest-neighbor to nearest-neighbor exchange constants in these systems.

I. INTRODUCTION

The nature of magnetic ordering in systems in which magnetic ions occupy sites on a face-centered-cubic (fcc) lattice depend on the relative signs and strengths of J_1 and J_2, the nearest-neighbor (NN) and next-nearest-neighbor (NNN) exchange constants respectively.[1,2] A number of such experimental systems with their magnetic structures and J_1 and J_2 values are listed in Ref. 2. Random site dilution of such systems presents interesting examples of the percolation phenomenon and, depending on the ratio J_2/J_1, a very different kind of magnetic behavior is expected as a function of dilution although only a few limited cases of J_2/J_1 have been attempted theoretically.[3,4] One of the well-studied cases is that of $Eu_pSr_{1-p}S$ (EuS is a ferromagnet) in which, for a certain range of p values, the system first orders ferromagnetically upon cooling but at lower temperatures exhibits a second transition to a spin-glass state,[3,5] presumably because of competing interactions ($J_1 = -0.24$ K, $J_2 = 0.12$ K) and because $|J_2/J_1| < 1$ (we use the sign convention of the exchange constants $J > 0$ for antiferromagnetic interactions and $J < 0$ for ferromagnetic interactions).

Among the diluted fcc antiferromagnets, the two systems which have been studied extensively in recent years have been $Eu_pSr_{1-p}Te$ (Ref. 6) and $Co_pMg_{1-p}O$,[7] where $J_1 = -0.06$ K and $J_2 = 0.20$ K for EuTe and $J_1 = 5.5$ K and $J_2 = 27.4$ K for CoO.[2] Experimentally, one of the important considerations is that, upon magnetic dilution, there should be no change in crystal structure and no significant change in the lattice constant (< 1% in both $Eu_pSr_{1-p}Te$ and $Co_pMg_{1-p}O$ between $p = 0$ and 1) so that J_1 and J_2 are not significantly affected. The magnetic phase diagrams of $Eu_pSr_{1-p}Te$ and $Co_pMg_{1-p}O$ were found to be rather similar in that, upon cooling, only a single transition from a paramagnetic-like to antiferromagnetic-like state is observed for all p, the percolation threshold $p_c \simeq 0.13$ in agreement with the theoretical prediction, and for $p_c < p < 0.45$, the transition is to a spin-glass-like state.[8] In $Co_pMg_{1-p}O$, the nature of the magnetic ordering has also been investigated by neutron scattering[9] and it is found that a transition from long-range order (LRO) to short-range order (SRO) occurs as p is reduced below 0.45, i.e., in systems for $p < 0.45$, transition upon cooling is to a state with SRO in that the correlation length is finite and becomes smaller as p is reduced below 0.45. A Monte Carlo simulation reproduces this LRO→SRO transition,[10] although the physical basis for this observation is not yet clear.

Like CoO, NiO is a type-II antiferromagnet, but with $T_N = 523$ K (compared to $T_N \simeq 289$ K for CoO) and $J_1 \simeq 34$ K and $J_2 = 202$ K.[2] We have been able to prepare solid solutions of $Ni_pMg_{1-p}O$ in the whole composition range and the observed change in the lattice constant for $p = 0$ to 1 is only 0.7%. Therefore, it is of interest to compare the phase diagrams and magnetic properties of $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$ systems. In this paper, we report the results of these studies involving crystal preparations and x-ray characterizations, and temperature-dependent magnetic susceptibility studies. Preliminary results of some of these studies have been reported at a recent conference.[11]

II. EXPERIMENTAL PROCEDURES

About 20 samples of $Ni_pMg_{1-p}O$ in the composition range of $0.06 \le p \le 0.86$ were prepared in the powdered form similar to the procedure used for the $Co_pMg_{1-p}O$ system.[7] Starting with the appropriate amounts of $Ni(NO_3)_2 \cdot 6H_2O$ and $Mg(NO_3)_2 \cdot 6H_2O$ (puratronic grades from Johnson Matthey), the mixture is warmed in a beaker in air to form a clear solution and then heated further to obtain dry flakes of mixed nitrates. The flakes are powdered thoroughly with a mortar and pestle, put in a alumina-silica boat and sintered at 500 °C for 2 h in flowing air using a Lindberg furnace to remove NO_2. The residual powder of mixed oxides is powdered again. A part of it is pressed into a cylindrical pellet (pressure$\simeq$15 000 lbs) of 5 mm diameter for eventual magnetic susceptibility studies. The remaining powder and the pellet are then sintered for about 16 h in air at 1200 °C for homogenization, followed by rapid quenching in liquid nitrogen. Under these conditions, the resulting samples were single-phase fcc systems as determined by x-ray diffraction using a diffractometer (Rigaku/DMax), see Fig. 1. Lower temperatures or less time for sintering usually produces multiphase samples. The lattice constant $a(p)$ was determined for each composition using the standard extrapolation procedure.[7] A part of each powder sample was then sent to the Galbraith Laboratories (Knoxville, TN) for an independent determination of the Ni concentration (using inductively coupled plasma technique). In all cases, the Ni concentration so determined was within 4% of the nominal composition. These experimentally determined p values are the ones used in the analysis of our data.

The temperature dependence of the dc magnetic susceptibility χ was measured using a superconducting-quantum-interference-device (SQUID) magnetometer (Quantum Design Model MPMS) in either the ZFC (zero-field-cooled) or FC (field-cooled) conditions. Under ZFC, the sample is cooled to the lowest temperature under zero field, a measuring field of 50 Oe is then turned on and the data is then taken with increasing temperatures. Under FC conditions, the sample was cooled in the measuring field (50 Oe) to the lowest temperature, followed by measurements of the magnetization with increasing temperature. The data above 400 K were taken using the high-temperature probe of the SQUID.

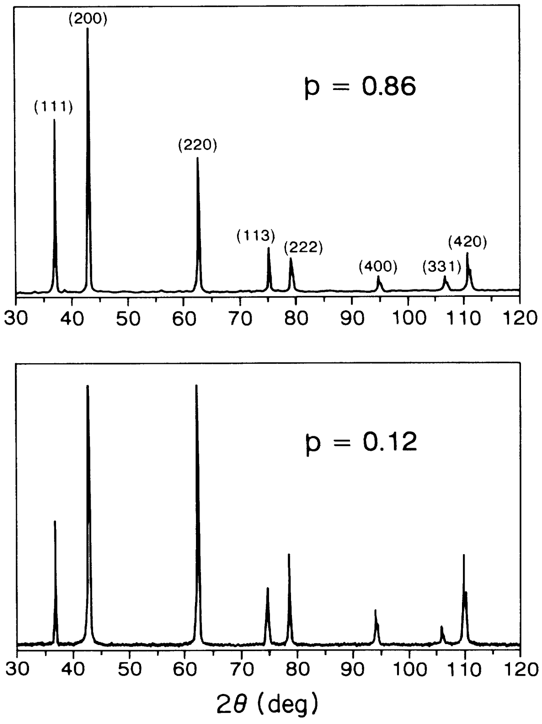

FIG. 1. X-ray diffractograms of two samples of $Ni_pMg_{1-p}O$ ($p=0.86$ and 0.12). The splitting of the lines for higher angles θ is due to differences in the Cu $K\alpha_1$ and $K\alpha_2$ wavelengths used in the experiments. The (hkl) indices are those of the NaCl structure.

III. HIGH-TEMPERATURE PARAMETERS FOR A DILUTED ANTIFERROMAGNET

The results of the recent calculations of Spálek *et al.*[12] for the high-temperature ($T > T_N$) properties of a diluted antiferromagnet are outlined below in order to ease the discussion of our experimental results. In the high-temperature limit, the Curie-Weiss law is still valid, i.e.,

$$\chi_M(p)=\frac{C_M(p)}{T+\Theta(1)p} , \tag{1}$$

but the molar Curie constant $C_M(p)=C_M(1)p$ and Curie-Weiss temperature $\Theta(p)=\Theta(1)p$ scale with p. From the high-temperature plot of $1/\chi_M(p)$ versus T, both $C_M(1)$ and $\Theta(1)$ can be deduced. In the molecular field theory for type-II antiferromagnets,[1] one gets the following equations for Θ and T_N:

$$3T_N=J_2Z_2S(S+1) , \tag{2}$$

$$3\Theta=S(S+1)(J_1Z_1+J_2Z_2) . \tag{3}$$

In the above equations Z_1 and Z_2 are, respectively, the number of NN and NNN and J_1 and J_2 are expressed in degrees K.

IV. RESULTS AND DISCUSSION

A. Lattice constant

The lattice constant $a(p)$ for various compositions was measured by x-ray diffraction (Fig. 1) and the plot of $a(p)$ versus p is given in Fig. 2. From this work, $a(p)$ varies between 4.1795 ± 0.0011 Å for $p=1$ (NiO) to 4.2113 ± 0.0015 Å for $p=0$ (MgO), the total variation being about 0.7%. The solid line is a least-squares fit to the data yielding the equation

$$a(p)=4.2115-0.0340p \text{ Å} . \tag{4}$$

It is noted in $Co_pMg_{1-p}O$ the experimental results[7] follow the corresponding equation $a(p)=4.2139+0.0476p$, leading to the observation that, compared to MgO, the lattice constant of NiO is smaller whereas that of CoO is larger. The ionic sizes of Mg^{2+}, Ni^{2+}, and Co^{2+} are, respectively, 0.86, 0.83, and 0.89 Å so that the above results are quite understandable. Because changes in $a(p)$ upon dilution in both $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$ are less than 1%, the corresponding changes in J_1 and J_2 should be negligible. Hence, both systems are crystallographically ideal for studying the effects of magnetic dilution on the magnetic properties.

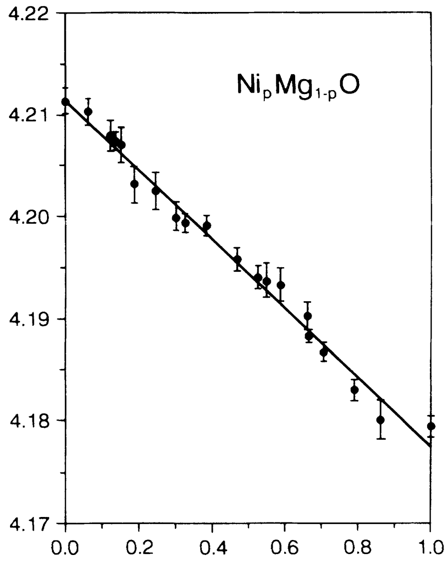

FIG. 2. The lattice constant $a(p)$ vs p for $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$. The solid line is the least-squares fit yielding Eq. (6).

B. Temperature dependence of magnetic susceptibility

The temperature dependence of the normalized susceptibility $\chi_g^* = \chi_g/p$, where χ_g is the measured susceptibility per gram for each composition of $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ is plotted against temperature in Figs. 3–5. For $p \leq 0.59$, the χ values were measured for the FC and ZFC conditions since, in the corresponding $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ system, an irreversible behavior of χ for $p < 0.50$ was evident.[7] For each composition, the χ_g values have been corrected for the diamagnetic (χ_d) and the Van Vleck (χ_{VV}) susceptibilities using the following guidelines. For MgO, $\chi_d = -0.5 \times 10^{-6}$ emu/g (Ref. 13), whereas for NiO, $\chi_{\mathrm{VV}} + \chi_d = 3.2 \times 10^{-6}$ emu/g.[14] Therefore, for $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$, we have used the correction $\chi_{\mathrm{VV}} + \chi_d = p(3.2) - (1-p)(0.5)$ in units of 10^{-6} emu/g. This temperature-independent correction to χ_g does not affect T_N, but does influence the magnitudes of the Curie constant and the Curie-Weiss temperature which are obtained from plotting $1/\chi_g$ versus T as described later.

For $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ samples with $p = 0.59$, 0.55, 0.53, 0.47, and 0.38, the temperature behavior of χ_g for the ZFC and FC cases is nearly identical, although a few percentage differences in their magnitudes below T_N (approximately given by the temperature at which χ_g peaks) is usually observed. In contrast, for samples with $p \leq 0.33$, the temperature dependence for the ZFC and FC curves below T_N is really different, similar to the observations in the $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ system for $p \leq 0.47$. For samples with $p = 0.25$, 0.19, and 0.15, we took data down to 1.8 K and for the $p = 0.15$ sample, no ordering down to 1.8 K was observed. Note that for samples with $p = 0.79$ and 0.86, we took data up to 600 K in order to observe T_N (Fig. 3).

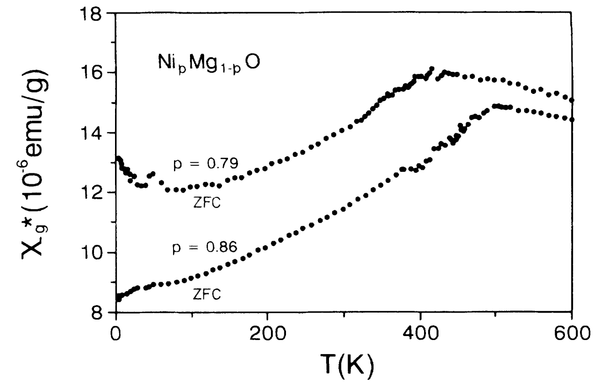

FIG. 3. Temperature dependence of $\chi_g^* = \chi_g/p$ for two samples of $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ for $p = 0.86$ and 0.79.

C. Effects of magnetic dilution

In antiferromagnets, χT near T_N represents magnetic energy. Hence, T_N is accurately determined by the peak position of $\partial(\chi T)/\partial T$ rather than by the peak in χ versus T.[15] In most cases, the T_N values so determined are few percent lower than the position of the maximum in χ versus T. We used a computer program to fit the χT versus T data near T_N to a second-order polynomial in order to determine $\partial(\chi T)/\partial T$. The T_N values so determined are then used to plot the reduced Néel temperature $t = T_N(p)/T_N(1)$. The plot of t versus p is shown in Fig. 6 in which we have also included the available data of Arkhipov[16] for several compositions of $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ and the data on $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ from Ref. 7.

There is a remarkable similarity in the variation of t versus p for $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ and $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ except for $p < 0.33$ where the values of t are a bit higher for

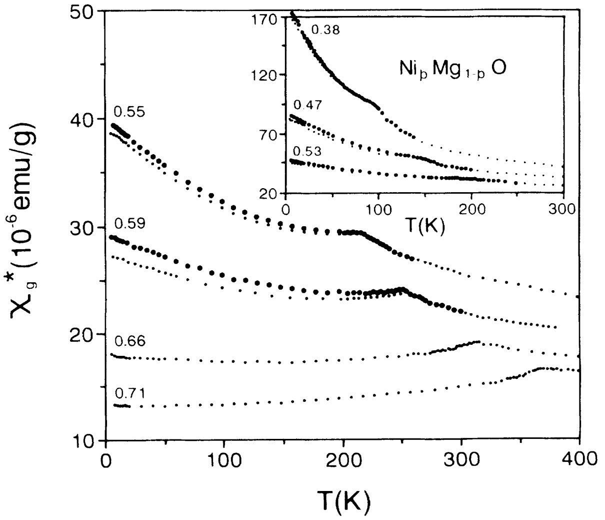

FIG. 4. Temperature dependence of $\chi_g^* = \chi_g/p$ for $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ samples with p values indicated on graphs. For $p \leq 0.59$, the lower sets are for the ZFC (zero-field-cooled) and the upper set for the FC (field-cooled) cases. The units of the ordinate for the inset are the same as on the main figure.

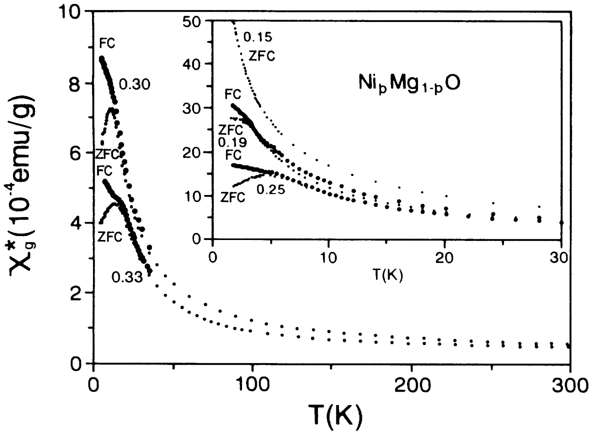

FIG. 5. Same as Fig. 4 except for samples with $p \leq 0.33$.

$Co_pMg_{1-p}O$ for a given p than those for $Ni_pMg_{1-p}O$. We note that, in comparing the phase diagrams of $Co_pMg_{1-p}O$ and $Eu_pSr_{1-p}Te$, a similar behavior was observed in that, for the lower p values, the t values for $Eu_pSr_{1-p}Te$ were now higher than those for $Co_pMg_{1-p}O$.[8] We will return to the discussion of this point later.

The theoretically predicted variations of t versus p for a simple-cubic (sc) Heisenberg and Ising systems are also shown in Fig. 6.[4] It is noted that a calculation of t versus p for a diluted fcc system, with both NN and NNN interactions included, is not yet available although the theoretical percolation threshold $p_c = 0.136$ is well established.[17] For the sc system with NN interaction only, $p_c \simeq 0.31$.[4] Surprisingly, the observed t versus p variations for both $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$ systems for $p > 0.32$ fits rather well with the prediction for a sc Heisenberg antiferromagnet with NN interaction only. Our interpretation for this surprising result is as follows.

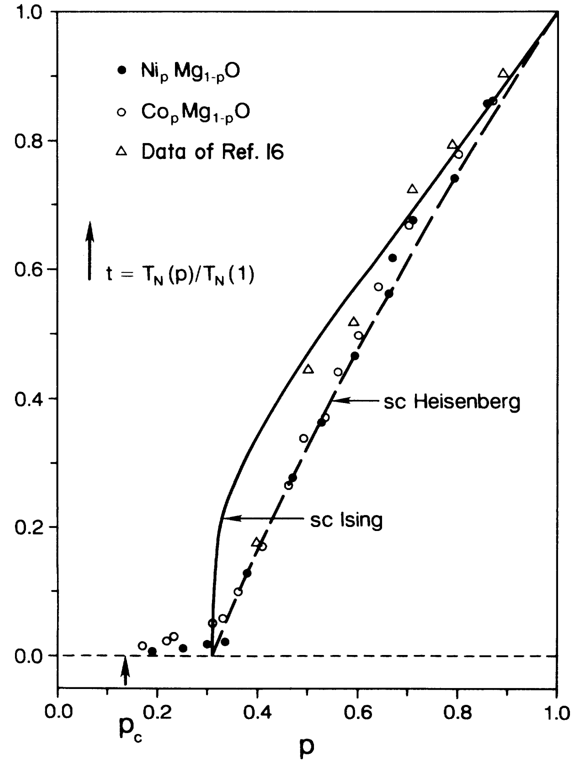

FIG. 6. Variation of the reduced Néel temperature $t = T_N(p)/T_N(1)$ vs p for $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$. The data of Arkhipov for $Ni_pMg_{1-p}O$ is from Ref. 16 and the data for $Co_pMg_{1-p}O$ is from Ref. 7. The theoretical curves for sc (simple-cubic) Ising and Heisenberg models are from Ref. 4.

As shown by Eq. (2), T_N for a type-II antiferromagnet in the molecular field approximation is determined by J_2, the NNN exchange interaction only. This lack of dependence of T_N on J_1 is due to the fact that, in type-II ordering, six of the NN of a given ion are oriented parallel and the remaining six NN are oriented antiparallel so that the total molecular field on a given ion due to 12 NN is cancelled. Although in the more accurate random-phase Green's-function theory of type-II antiferromagnetism,[18] some dependence of T_N on J_1 is recovered, the error at least in NiO is no more than 15%.[14] Therefore, it is proposed that, in initial dilutions ($p > 0.31$), the NN's play no significant role in determining t versus p and the systems therefore reduce to sc systems as far as the determination of T_N is concerned. Since the anisotropy in both NiO (Ref. 14) and CoO (Ref. 19) is much less than the exchange energy, the systems behave more like Heisenberg systems than Ising systems. Using the excellent fit obtained in Fig. 6, one may conclude that, for $p > 0.31$, type-II antiferromagnetism is maintained in both $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$ systems upon dilution.

A consequence of the above argument is that, for $p < 0.31$, J_1 must be playing a significant role or perhaps it is the ratio of J_2/J_1 which is significant. In $Eu_pSr_{1-p}S$, $|J_2/J_1| < 1$, whereas for the antiferromagnetic systems $Eu_pSr_{1-p}Te$, $Co_pMg_{1-p}O$ and here $Ni_pMg_{1-p}O$, $|J_2/J_1| > 1$ and this is perhaps the primary reason why the phase diagram of $Eu_pSr_{1-p}S$ for $p < 0.50$ is so different from those of the antiferromagnetic counterparts. The fact that, in this region, the phase boundaries separating the paramagnetic and spin-glass states for $Eu_pSr_{1-p}Te$, $Co_pMg_{1-p}O$, and $Ni_pMg_{1-p}O$ systems do not quite match perhaps is also the result of the different values of J_2/J_1.

The computed variations of dt/dp versus p for $Ni_pMg_{1-p}O$ and $Co_pMg_{1-p}O$ are shown in Fig. 7 along with the computation for the sc Heisenberg system. The dt/dp peaks at slightly different values of p for the two systems, demonstrating that there are indeed some noticeable differences for the two systems. (The curves in Fig. 7 are slopes of the smooth curves drawn through the data points of Fig. 6.)

The determination of p_c (the percolation threshold below which magnetic ordering disappears) is considered next since, from Fig. 6, one can at best determine an extrapolated value. We follow the same procedure as used for $Co_pMg_{1-p}O$, i.e., of measuring magnetization M versus p at the lowest experimental temperature. Such a plot is shown in Fig. 8, where the data for the lower p was taken at both 1.8 and 5 K. As shown, M peaks at

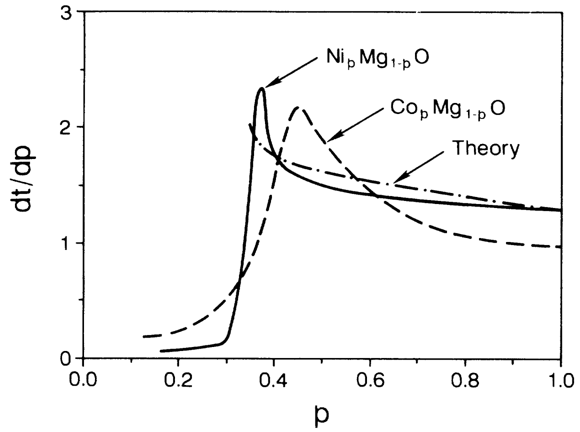

FIG. 7. Computed variations of dt/dp vs p for $Co_pMg_{1-p}O$, $Ni_pMg_{1-p}O$, and for the simple-cubic (sc) Heisenberg model (theoretical).

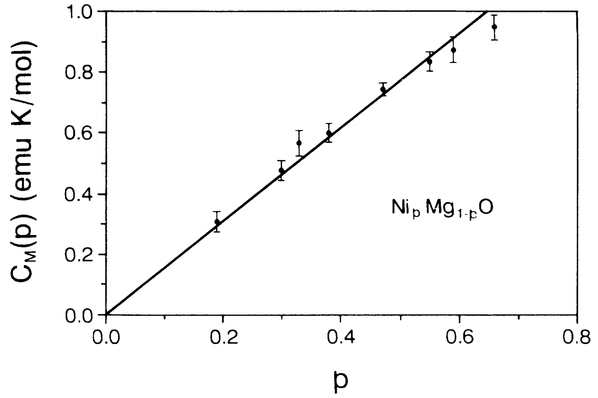

FIG. 9. Variation of the Curie constant $C_M(p)$ [Eq. (1)] vs p for $Ni_pMg_{1-p}O$. In drawing the line through the points, it is assumed that $C_M(0)=0$ and it yields $C_M(1)=1.5$ emu K/mol.

$p=0.145$ at 1.8 K and at $p=0.155$ at 5 K with uncertainties of about ± 0.01. Therefore, we infer that $p_c=0.15\pm0.01$, in good agreement with the theoretical estimate of $p_c=0.136$.[17] The argument for using this procedure is that, for $p>p_c$, the system consists of an infinite antiferromagnetic cluster and only a few isolated smaller uncompensated clusters whose size and number decreases as p increases. For $p<p_c$, the system consists of only isolated uncompensated clusters whose size and number increases as p approaches p_c, therefore resulting in a peak value of M at $p=p_c$.[20]

D. High-temperature properties

According to Eq. (1), a plot of $1/\chi_M(p)$ versus T can be used to determine $C_M(p)$ and $\Theta(p)$ using the data in the region $T \gg T_N$. The values of $C_M(p)$ and $\Theta(p)$ so determined are plotted against p in Figs. 9 and 10, respectively. Since the $T \gg T_N$ condition is difficult to attain experimentally for samples with higher p values because of their high T_N's, the data are limited to compositions between $p=0.2$ and 0.7. An extrapolation of the data to $p=1$ yields $C_M(1)=1.5$ and $\Theta(1)\simeq 1150$ K for NiO. For pure NiO, $\Theta \simeq 2000$ and 800 K have been estimated by Singer[21] and Arkhipov,[16] respectively, from the data of χ versus T for $T>T_N$. Our estimate is nearly an average of these two values. The magnitude of $C_M(1)$ corresponds to a moment $\mu=3.46\mu_B$ for Ni^{++}. This is to be compared with the known moment of $\mu=3.15$ determined from low-temperature properties (neglecting zero-spin deviations).[14] Our estimate is close to the value of $\mu=3.37\mu_B$ determined by Arkhipov[16] for NiO but much smaller than $\mu=4.6\mu_B$ reported by Singer.[21] It is possible that estimates of $\Theta(1)$ and $C_M(1)$ by Singer are seriously affected by the neglect of χ_{VV} whose magnitude is relatively large in NiO. Using $\Theta=1150$ K and $T_N=523$ K in Eqs. (2) and (3), we obtain $J_2=131$ K and $J_1=78$ K. These magnitudes are to be compared with $J_2=202$ K and $J_1=34$ K obtained by the use of random-phase-approximation Green's-function theory.[14] It is well known[18] that Eqs. (2) and (3) of the molecular field approximation give very poor results for estimating J_1 and J_2 for the oxides so that use of Θ and T_N in Eqs. (2) and (3) for determining J_1 and J_2 is not reliable. This point has been discussed in earlier publications.[14,18]

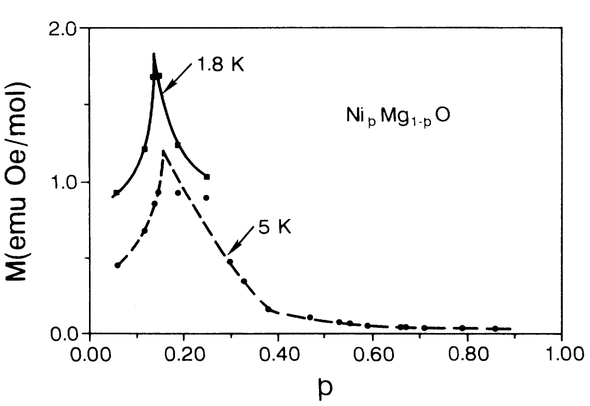

FIG. 8. Variation of the low-temperature magnetization M for ZFC samples measured at 50 Oe. The data at 1.8 and 5 K are shown. The lines are drawn through the points for visual aid.

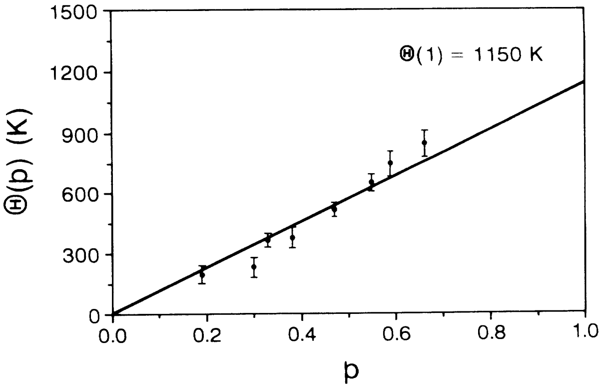

FIG. 10. Variation of the Curie-Weiss constant $\Theta(p)$ of Eq. (1) vs p for $Ni_pMg_{1-p}O$. Assuming $\Theta(0)=0$, the solid line drawn through the points leads to $\Theta(1)\simeq 1150$ K.

V. CONCLUDING REMARKS

In this work, accurate values of T_N versus p for $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ for $p_c \leq p < 1$ have been presented and it is found that, by and large, the phase diagram of $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ is similar to that of $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ except for $p < 0.31$, where some differences are noted. Second, it is shown that, for $p > 0.31$, the variation of $t = t_N(p)/T_N(1)$ versus p for both $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ and $\mathrm{Ni}_p\mathrm{Mg}_{1-p}\mathrm{O}$ fit the predictions for a sc Heisenberg antiferromagnet and a theoretical justification for this seemingly anomalous result has been presented. What is needed is a calculation of t versus p for diluted fcc antiferromagnets for the case of $J_2/J_1 > 1$ to compare with the experimental results presented here.

Finally, we comment on the spin-glass-like behavior of χ versus T for the ZFC and FC cases in samples with $p < 0.3$. Neutron-diffraction studies[10] and studies of the nonlinear susceptibilities[22] for $p < 0.3$ are now underway to determine the nature of magnetic order in this region and to compare these observations with the LRO→SRO transition observed in the $\mathrm{Co}_p\mathrm{Mg}_{1-p}\mathrm{O}$ system.[9] Details of these results will be published in the near future.

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