

# Temperature dependence of magnetization in arrays of submicrometer Fe disks

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We investigate the magnetization in the arrays of submicrometer-size disks fabricated from an epitaxial Fe layer grown on GaAs(001). The Fe disks having a layer thickness of 20 nm undergo a transition between multiple and single magnetic domains when their diameter is about 100 nm. Remarkable temperature dependence is found in the characteristics of the magnetization. (1) The saturation magnetization increases with decreasing temperature. While the low-temperature increase is more pronounced for smaller disk diameters, it is independent of the magnetic-domain state in the disks. (2) Magnetic hysteresis exhibits two-component-like behavior at low temperatures for ultrasmall disks. The additional component that emerges with lowering temperature gives rise to slow saturation of the magnetization, and is possibly responsible for the low-temperature increase of the saturation magnetization. © 2005 American Institute of Physics. [DOI: 10.1063/1.2123373]

## I. INTRODUCTION

In the aim of increasing the capacity of magnetic storages, we are experiencing a dramatic shrinkage of the size of magnetic media to record a single bit. The characteristics of ferromagnetic materials are dramatically altered in reduced dimensions. For instance, Fe films having a thickness of several tens of nanometer can be treated, in practice, as consisting of a single magnetic domain because of the extremely slow relaxation of the magnetization. Upon reducing their size to be on the order of 1  $\mu\text{m}$ , however, the magnetic domain in the absence of an external magnetic field changes to a closure-flux-type multidomain state<sup>1-4</sup> due to the magnetostatic effect.<sup>5,6</sup> The magnetostatic energy becomes lower than the domain-wall energy when the diameter is further reduced.<sup>7</sup> As a consequence, the disks undergo a reverse transition from the multiple domain to a single domain.<sup>8,9</sup> The latter transition originates from the fact that the thickness of the domain walls is independent of the diameter of the disks, i.e., it is no longer negligibly small for such small disks. Below the critical size for the single-domain formation, magnetic disks are regarded as “nanomagnets” even in the demagnetized state. The magnetization reversal processes of such submicron ferromagnetic disks have attracted considerable interest in recent years.<sup>1-3,6,8,9</sup>

In this paper, we examine the variation of the magnetization induced by an in-plane external magnetic field in the arrays of submicron-large disks fabricated from an epitaxial Fe layer on GaAs(001). Our attention is focused on the temperature dependence of the magnetization. As the magnetic energies become comparable to or less than the thermal energy in the nanometer-scale disks, the thermal effects play an important role in the magnetization reversal processes. We observe two remarkable features in the temperature dependence of the magnetization traces. Both of them grow stronger with decreasing the disk diameter.

## II. SAMPLE PREPARATION

The magnetization is investigated in the arrays of Fe disks prepared from an epitaxially grown film. Epitaxial films provide an ideal experimental system due to their unmatched uniformity and material purity. A high quality bcc Fe(001) layer was grown on an As-terminated GaAs(001)-(2 $\times$ 1) surface at 50 °C by molecular-beam epitaxy (MBE).<sup>10,11</sup> The growth rate was 0.24 nm/min at a base pressure of  $1 \times 10^{-10}$  Torr. The thickness  $d$  of the layer is 20 nm. Although the Fe layer was uncapped, its natural oxidation was considerably small and was restricted to the surface due to the high purity of the MBE-grown Fe. X-ray reflectivity measurements suggested that the thickness of the native surface oxides was not more than a few nanometers. The inset of Fig. 1 shows the double-crystal x-ray diffraction rocking curve obtained from a “control” film grown under identical conditions. Apart from the peaks associated with the substrate and the Fe layer, no spurious peak is present,

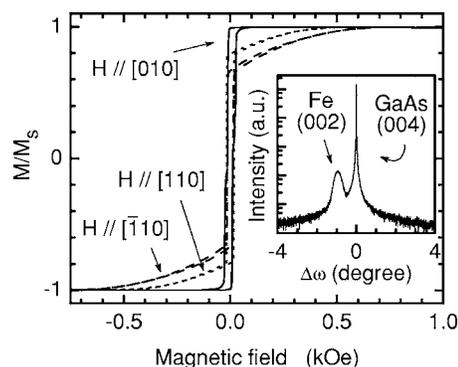


FIG. 1. Magnetization  $M$  normalized by the saturation magnetization  $M_s$  in a 20-nm-thick Fe film on GaAs(001) at temperature  $T=300$  K. The film was grown under conditions identical to those used for the growth of the film from which the disks were fabricated. The external magnetic field  $H$  is applied along the [010],  $[\bar{1}10]$ , and [110] directions of the substrate for the solid, dashed, and dotted lines, respectively. The inset shows the double-crystal x-ray diffraction curve ( $\omega-2\theta$  scan) of the film. The (004) peak from the substrate and the (002) peak from the epitaxial layer are indicated.

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attesting the high quality of the epitaxial film.<sup>10</sup> Moreover, an abrupt interface and no intermixing reaction at the Fe/GaAs heterojunction grown under the present optimum conditions have been demonstrated by transmission electron microscopy.<sup>11</sup>

For the present layer thickness, a fourfold magnetocrystalline anisotropy is dominant, while the Fe layer retains a weak uniaxial anisotropy. The latter becomes significant in thin layers.<sup>11–13</sup> In Fig. 1, we show the magnetization traces when an external magnetic field  $H$  was applied along high-symmetry directions of the substrate in the control film. The easy axes of the cubic anisotropy are oriented along the  $[100]$  and  $[010]$  directions of the GaAs substrate. The preferential magnetization direction due to the uniaxial anisotropy is along the  $[110]$  direction of the substrate.

We have processed the epitaxial layer to Fe disks using microfabrication technologies. An etch mask of Ti or NiCr was prepared on the Fe surface by electron-beam lithography and the lift-off technique. The Fe layer outside the etch mask was then removed by Ar milling at an acceleration voltage of 150 V. The metal mask was left on top of the Fe disks during the measurements using a superconducting quantum interference device magnetometer (SQUID) [Quantum Design MPMS XL]. The thickness of the mask was reduced to be well below 10 nm at the completion of the microfabrication process. In particular, the mask became virtually nonexistent for the device in which NiCr was employed. The magnetic moment originating from the mask metal is thus negligibly small and stress arising from the mask is not anticipated to affect the magnetic properties of the Fe disks. The magneto-crystalline anisotropy in Fe layers is rather weak, and so the shape anisotropy is typically the major origin of an anisotropy for the disks. For this reason, the disks were designed to be circularly shaped in order to avoid geometry effects.

We show scanning electron micrographs of the disks in the insets of Fig. 2. The disks were assembled as either regular or random arrays. The structural parameters of the arrays are summarized in Table I. For the disordered positioning, the disks were shifted from the lattice sites of a square array with random displacements. The amount of the displacements along the axes of the array was chosen to be less than the period of the array. Therefore, there exists a long-range order that only one disk is present in a unit cell of the square lattice. The axes of the square lattices were aligned along the  $[110]$  and  $[\bar{1}10]$  directions of the substrate. The linear dimension of the square arrays was  $N=164$ . For each device, several hundreds of the arrays containing  $N \times N$  disks were put together in a closely packed manner to strengthen the signal for the SQUID measurements. The dipole-dipole interaction is negligible for the interdisk spacings employed in our experiment. The difference in the arrangements of the disks thus produces no influence on the magnetic properties. The magnetization we examine below is equivalent to the statistically averaged one of a single disk.

### III. MAGNETIC-DOMAIN STRUCTURE IN DISKS

We compare in Fig. 2 the magnetization traces (filled circles) at a temperature  $T=2$  K in the Fe film, Fig. 2(a), and

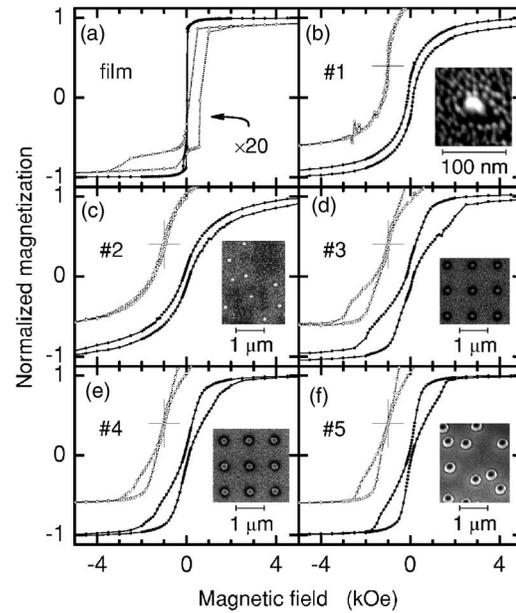


FIG. 2. Magnetization  $M$  normalized by the saturation magnetization  $M_s$  in an epitaxial Fe film on GaAs(001), (a), and in the disk arrays fabricated from the film, (b)–(f), at temperature  $T=2$  K. The axes of the square arrays are along the  $[110]$  and  $[\bar{1}10]$  directions of the GaAs substrate. The external magnetic field  $H$ , which is applied along the  $[110]$  GaAs direction, is varied between  $-9$  and  $9$  kOe. The diamagnetic contributions due to GaAs have been subtracted. The magnetization trace is expanded horizontally by a factor of 20 for the dotted curve in (a). The open circles in (b)–(f) show  $M$  at  $T=300$  K. These symbols are offset for clarity. (The thin lines indicate  $H=M=0$ .) The insets show scanning electron micrographs of the Fe disks. The images in (b) and (f) were taken from an inclined angle.

the arrays consisting of Fe disks having various diameters, Figs. 2(b)–2(f). Here, the diamagnetic contributions arising from the GaAs substrate have been subtracted. For these measurements, the magnetic field was swept between  $-9$  and  $9$  kOe. (The up and down traces do not meet in Fig. 2 as the data are plotted there for a restricted magnetic-field range.) Except for a few cases we state below, the magnetization of the Fe disks saturated at the highest magnetic field, thereby allowing us to quantify the diamagnetic contribution. The magnetization  $M$  is normalized by its saturation value  $M_s$  at high magnetic fields. The external field  $H$  was applied along one of the axes of the square arrays, the  $[110]$  direction of the GaAs substrate.

The saturation magnetization in the film is  $M_s=1.7 \times 10^9$  emu/m<sup>3</sup>, which compares well with the value of bulk Fe ( $1.75 \times 10^9$  emu/m<sup>3</sup>). The hysteresis loop of the film, Fig. 2(a), is not antisymmetric with respect to  $H=0$ , suggesting the presence of the exchange biasing effect<sup>14,15</sup> due to the

TABLE I. Structural parameters of the array samples fabricated from an epitaxial Fe film grown on GaAs(001).

Sample	Diameter $D$ (nm)	Period (nm)	Order	Mask
1	50	950	regular	NiCr
2	100	(920)	random	Ti
3	120	860	regular	Ti
4	200	870	regular	Ti
5	300	(950)	random	Ti

antiferromagnetic surface oxide layer. The estimation of the above-mentioned saturation magnetization in the film was made by ignoring the oxide layer. The agreement with the bulk value indicates that the oxide layer was very thin. In accordance with a relatively weak antiferromagnetic coupling, the exchange biasing effect vanished at high temperatures.

Submicrometer disks undergo a transition from a multi-domain state to a single-domain state when their diameter is reduced. We first identify the critical diameter for the single-domain regime. It is well understood that the difference in the domain state manifests itself in the response of the magnetization to the external magnetic field.<sup>8</sup>

The magnetization curves for the disk diameter  $D = 200$  nm, Fig. 2(e), and 300 nm, Fig. 2(f), are almost identical.<sup>16</sup> This type of hysteresis is typical when the magnetic properties are dominated by the creation and annihilation of magnetic-domain walls in the disks<sup>1,8</sup> and/or the pinning of domain walls.<sup>17</sup> There are a number of reports<sup>1-4</sup> in which magnetic-domain structures with a fourfold symmetry compatible with the magnetocrystalline anisotropy have been observed in circular disks. For the  $d=20$  nm layer, the disks in Figs. 2(e) and 2(f) are sufficiently large to contain multiple magnetic domains at  $H \sim 0$ . The multidomain state becomes energetically costly with increasing  $|H|$  as the magnetic moments of the domains are unanimously tilted to be aligned along the external field. At a certain field, the domain walls suddenly vanish, leading to an abrupt full polarization of the magnetic moments. In lowering the external field, the same situation basically takes place in the reversed order. However, the domain walls are created at a lower critical field as compared with that for the annihilation of the domain walls,<sup>8</sup> resulting in the pronounced hysteretic behavior at medium magnetic fields.

The magnetization exhibits a fundamentally different switching behavior when  $D$  is as small as 50 nm, Fig. 2(b), or 100 nm, Fig. 2(c). The magnetization traces are S shaped, i.e., approximately antisymmetric with respect to  $H=0$ . The curves for the up and down magnetic-field sweeps are almost identical except for a small displacement that gives rise to a hysteresis. We conclude that the Fe disks retain the single-magnetic-domain state for the entire magnetic-field range when  $D \leq 100$  nm.<sup>3,8</sup> In this regime, the gradual tilt of a magnetic moment having a fixed magnitude is the mechanism for the magnetization reversal. We do not find any remarkable differences in the magnetization traces between the regular and random arrangements of the disks. The hysteresis loop for the random array, Fig. 2(c), is smaller than that for the regular array, Fig. 2(b), although the opposite is expected when dipole-dipole interaction is strong.<sup>18</sup> The interaction is hence confirmed to be insignificant in our devices.

The diameter of the disks shown in Fig. 2(d), 120 nm, is around the transition value between the two mechanisms of the magnetization reversal processes. The hysteresis observed for this array is, in principle, of the domain-wall-type. However, higher magnetic fields are required to polarize the array and the saturation of the magnetization is less clearly established compared to the large disks. We speculate that the array consists of both single- and multidomain disks at

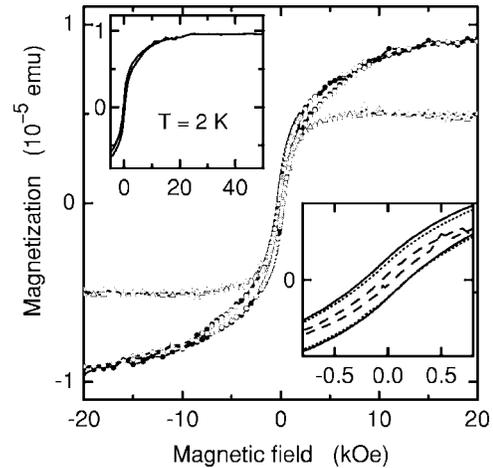


FIG. 3. Magnetization of  $D=100$  nm disks (sample 2) at room temperature (triangles) and 2 K (circles). The external magnetic field is applied along the  $[110]$  direction of the substrate. The diamagnetic contributions due to GaAs have been subtracted. The sample was cooled in the absence of magnetic field for the open circles, whereas an external magnetic field of 20 kOe was present during the cooling for the filled circles. The magnetization traces are shown with expanded scales in the lower-right inset. The solid and dotted lines correspond to the field and zero-field coolings, respectively. The dashed line shows the magnetization at room temperature. The upper-left inset demonstrates the saturation of the magnetization in high magnetic fields at  $T=2$  K.

this marginal size. The magnetization trace is thus given as a mixture of those associated with the two types. We note that the breakdown of the single-domain behavior is known to take place at smaller diameters when the layer thickness increases.<sup>7-9</sup>

#### IV. TEMPERATURE DEPENDENCE OF MAGNETIZATION

The first of the two remarkable features we observe for small disks is the strong temperature dependence of the saturation magnetization  $M_s$ . In Fig. 3, we show the magnetization traces obtained from sample 2 ( $D=100$  nm). The circles and triangles indicate  $M$  at  $T=2$  and 300 K, respectively. The saturation magnetization at room temperature is only about a half of that at 2 K.<sup>19</sup> (We have confirmed the saturation of the magnetization at 2 K as shown in the upper-left inset of Fig. 3.) The diamagnetization in GaAs has a negligibly small temperature dependence. (The temperature variation is 1.0% between 2 and 300 K.<sup>20</sup>) Moreover, as the diamagnetic correction was evaluated for each measurement temperature, the temperature dependence of the substrate contribution cannot account for the temperature dependence of  $M_s$ . We note that the correction attributed to the substrate was indeed only weakly temperature dependent (a few percent decrease when  $T$  was increased from 2 to 300 K).

In Fig. 4,  $M$  at  $H \approx \pm 9$  kOe is plotted with a normalization by its value at room temperature. Except for the ultrasmall disks at low temperatures, the magnetization is fully saturated at  $|H|=9$  kOe. That is, the saturation is achieved in all the samples at room temperature. Figure 4 thus presents the lower bound of the low-temperature increase of  $M_s$ .<sup>21</sup> The variation of  $M_s$  between 2 and 300 K is only 2.5% for

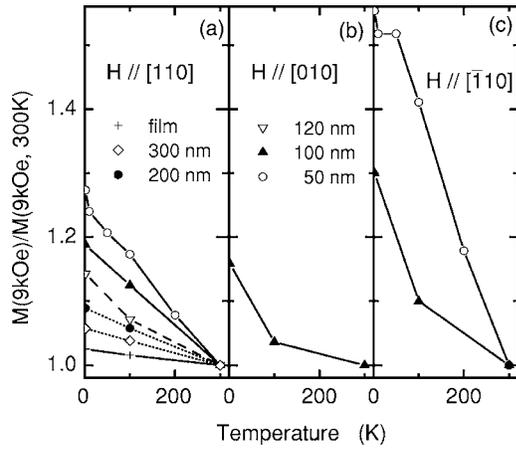


FIG. 4. Magnetization  $M$  at  $|H| \approx 9$  kOe normalized by its value at room temperature. The external field is applied along the  $[110]$ ,  $[010]$ , and  $[\bar{1}10]$  directions of the GaAs substrate in (a), (b), and (c), respectively. The diameter of the disks ranges between 50 and 300 nm.

the film. The saturation magnetization in the low-temperature region is known to obey the Bloch law<sup>5</sup>

$$M_s(T) = M_s(0)[1 - (T/T_M)^{3/2}], \quad (1)$$

where  $T_M = 4.3 \times 10^3$  K for Fe. Equation (1) predicts a 2% reduction of  $M_s$  at room temperature, in agreement with the observation in our film. With reducing the disk size, the enhancement of  $M_s$  at low temperatures grows stronger, see Fig. 4(a). The transition in the magnetic-domain state in the disks does not appear to influence the temperature dependence of  $M_s$ . The external magnetic field is applied along the  $[110]$  direction of the substrate in Fig. 4(a). In Figs. 4(b) and 4(c), the field is applied along the  $[010]$  and  $[\bar{1}10]$  directions of the substrate, respectively. The low-temperature enhancement of  $M_s$  takes place irrespective of the direction of the field.

The mechanism for the strong temperature dependence is not understood at present. It is well known that nanometer-scale ferromagnetic particles exhibit superparamagnetism as their magnetic moment is easily randomized by thermal fluctuations. As one finds in Fig. 4, however, the magnetization traces reveal clear saturation behavior, especially at high temperatures. That is, the magnetic moments of the disks are fully oriented along the external field.<sup>22</sup> We thus rule out the effects of the thermal disorientation of the magnetic moments as the origin of the small  $M_s$  at high temperatures. It does not seem likely that the Curie temperature is altered by reducing the size as the diameter is still considerably larger than the layer thickness even for the smallest disks. Out-of-plane components of the magnetic moments may emerge when the disk diameter approaches the layer thickness. However, there is no reason that the effect should be strong at high temperatures.

The antiferromagnetic coupling due to the surface oxide may give rise to an unusual temperature dependence. However, we have found so far no evidence for the influence of the antiferromagnetic coupling in the disks. The filled circles in Fig. 3 show  $M$  when the sample was cooled in the presence of an external magnetic field (20 kOe). As shown by the

plot with expanded scales in the lower-right inset of Fig. 3, the exchange biasing effect<sup>23–25</sup> is less than the experimental noise. (The solid and dotted lines correspond to the field and zero-field coolings, respectively.) We have neither observed any unusual field dependencies of  $M$  at high temperatures. The energy associated with the antiferromagnetic coupling is suggested to have scaled down to be less than  $k_B T$  in the small disks. The antiferromagnetic order in the oxide layer was dwarfed by the thermal fluctuations, similar to the fact that the exchange biasing effect in the film was not observable at room temperature. Although we may not be able to completely ignore the antiferromagnetic coupling as the exchange biasing effect is evident in the magnetization trace of the film, the factor of  $\sim 2$  change of  $M_s$  in Fig. 3 is, nevertheless, too large to be caused by the antiferromagnetic coupling considering the small fraction (less than 10%) of the native oxides in comparison to the volume of Fe.<sup>26</sup> The side walls are additionally exposed in the disks. However, the condition  $D \gg d$  implies that the surface effects in the present disks are not much different from those in the film.

The second temperature effect we report in this paper is found in the shape of the hysteresis loops. The magnetization traces of the single-magnetic-domain disks depend markedly on temperature. The open circles in Figs. 2(b)–2(f) show the magnetization reversal behavior at room temperature. (The symbols are offset for clarity.) The hysteresis becomes fairly small at the elevated temperature when  $D = 50$  and 100 nm: a consequence of random flipping of the magnetic moments due to thermal fluctuations. For the large disks, Figs. 2(e) and 2(f), the coercive field becomes similarly almost zero. In contrast, the pronounced hysteretic behavior at medium fields barely depends on temperature, supporting our interpretation of it in terms of the domain wall.

We analyze the temperature dependence of the hysteresis for  $D = 50$  nm in detail. In Fig. 5, we plot the difference of the magnetization between the down and up magnetic-field sweeps

$$\Delta M(H) = M(H:\text{down}) - M(H:\text{up}), \quad (2)$$

for various temperatures. The external field  $H$  was applied along the  $[110]$  and  $[\bar{1}10]$  directions of the substrate in Figs. 5(a) and 5(b), respectively. The following observations were made for both of the field orientations.  $\Delta M$  is identical within the experimental error for the positive and negative values of  $H$ , suggesting again the negligibly small exchange biasing effect for the disks. The area enclosed by the hysteresis loop grows larger with decreasing temperature. More importantly, the hysteresis loops at  $T = 10$  and 2 K appear to consist of two components having, possibly, different coercive fields, see Fig. 5. The ordinary component dominates the hysteresis for  $|H| < 1$  kOe. The additional component appears as a plateau-like feature in  $\Delta M$  for  $|H|$  between  $\approx 1$  and  $\approx 5$  kOe. The large-coercive-field component is responsible for the slow saturation of the magnetization in small disks. Its origin may be related to the low-temperature enhancement of  $M_s$ , as the deviation of the magnetization trace at  $T = 2$  K from that at  $T = 300$  K in Fig. 3 occurs when the

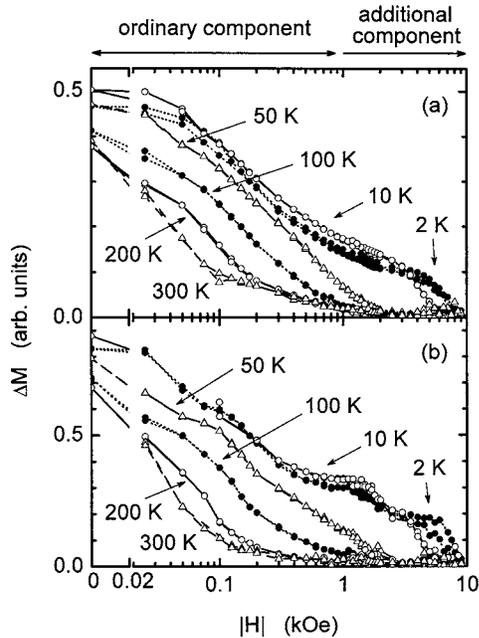


FIG. 5. Difference  $\Delta M$  of the magnetization in the Fe disks between the down and up sweeps of the external magnetic field  $H$  as a function of temperature. The disk diameter is 50 nm (sample 1). The field is applied along the (a)  $[110]$  and (b)  $[\bar{1}10]$  directions of the substrate.

component of large coercivity becomes dominant at large values of  $|H|$ .

## V. CONCLUSION

In conclusion, we have investigated the magnetization reversal processes in the arrays of disks microstructured from an epitaxial Fe layer. Remarkable temperature dependence of the magnetic properties, a low-temperature enhancement of the saturation magnetization, and an emergence of a magnetization component having a large coercivity at low temperatures, have been observed. Both of these features are pronounced when the disk diameter is reduced. The upper-most diameter for the disks to behave as nanomagnets, i.e., to maintain the single-magnetic-domain state, has been identified to be about 100 nm when the layer thickness is 20 nm. The transition in the magnetic-domain state in the disks appears to have no relevance to the strongly temperature-dependent behavior of the magnetization.

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