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Spontaneous surface magnetisation of single crystal MnF_2 in the antiferromagnetic state

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Abstract. Spontaneous magnetisation of (100) and (010) surfaces of single crystal MnF_2 in the antiferromagnetic state has been discovered. The sign of the surface magnetisation is determined by the difference in dielectric constants ε of MnF_2 and ambient matter: magnetisation is directed to the substance with smaller ε.

PACS. 75.30.Pd Surface magnetism – 75.25.+z Spin arrangements in magnetically ordered materials (including neutron and spin-polarized electron studies, synchrotron-source X-ray scattering, etc.) – 75.50.Ee Antiferromagnetics

1 Introduction

During recent years several interesting results were obtained from investigations of antiferromagnets in small magnetic fields. In works [1–4] a remanent magnetisation along the easy-axis of disordered antiferromagnets ($Mn_{1-x}Zn_xF_2$, $Fe_{1-x}Zn_xF_2$, $K_2Fe_{1-x}In_xCl_5{\cdot}H_2O$ and $Rb_2Fe_{1-x}In_xCl_5{\cdot}\ H_2O$) was reported. Unfortunately industrial SQUID magnetometers equipped with big superconducting magnets were used that led to problems in obtaining very small (< 0.1 A/m) axial magnetic fields. Besides, uncontrolled and much larger (> 1 A/m) transverse magnetic fields existed.

The requirements of "clean" magnetic conditions were satisfied with home-built SQUID magnetometers, and using these, crystal Cr_2O_3 was investigated [5–7]. In these measurements the quadrupole magnetic moment was found below T_N. The existence of such a quadrupole moment is possible because rhombohedral Cr_2O_3 belongs to the magnetic class symmetry $D_{3d}(D_3)$ that has operations of inversion (I) and time reversal (R) in combination IR [8]. The same condition is necessary for the magnetoelectric effect to be observed.

In the present work we report the results obtained for clean MnF_2 single crystal. This is an easy-axis antiferromagnet with $T_\mathrm{N} \approx 68$ K. It belongs to the magnetic class $D_{4h}(D_{2h})$ which does not permit weak ferromagnetism (spontaneous volume dipole moment [9]) nor quadrupole magnetic moment to occur. Surprisingly we have observed a signal from both dipole and quadrupole magnetic moments below T_N. These moments resulted from spontaneous magnetisation of surfaces (100) and (010) of the sample. The magnetisation was always perpendicular to the easy-axis [001] and its sign was set by the conditions at the sample surface. When the sample was in contact with gaseous helium then the magnetisation was directed outward from the sample. When the sample was coated with a gold film then the magnetisation was directed inward. We believe that this behaviour is connected with the difference in dielectric constant values of MnF_2 ($\varepsilon \approx 6.5$), gas ($\varepsilon \approx 1$) and metal ($\varepsilon \to \infty$).

Because most of the experimental results follow from analysis of the shape of the signals registered with a SQUID magnetometer when the sample was moved along the coil axis, below we present formulae for the magnetic field and flux produced by a quadrupole (coefficients given in [8] are not correct). Generally a guadrupole consists of two opposite dipoles $\mathbf{m}$, $-\mathbf{m}$ displaced by vector $\mathbf{d}$. In the dipole approximation the magnetic field of the quadrupole can be written as

$$4\pi\mathbf{H} = \frac{3(\mathbf{mr})\mathbf{r}}{r^5} - \frac{\mathbf{m}}{r^3} - \frac{3(\mathbf{m}(\mathbf{r}+\mathbf{d}))(\mathbf{r}+\mathbf{d})}{|\mathbf{r}+\mathbf{d}|^5} + \frac{\mathbf{m}}{|\mathbf{r}+\mathbf{d}|^3}\,. \tag{1}$$

We can find the radial component of $\mathbf{H}$ by multiplying (1) by $\mathbf{r}/r$ and neglecting terms with d^2:

$$4\pi H_r = \frac{3}{r^6}[3(\mathbf{mr})(\mathbf{dr}) - (\mathbf{md})r^2]. \tag{2}$$

For the case when $\mathbf{m}\|\mathbf{d}$, we have

$$4\pi H_r = \frac{3(\mathbf{md})}{r^4}(3\cos^2\vartheta - 1) \tag{3}$$

where ϑ is the angle between the quadrupole axis $\mathbf{d}$ and $\mathbf{r}$.

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Now we may calculate the flux through a turn. If the quadrupole is placed at a turn axis at a distance z from its centre, and φ is the angle between quadrupole and turn axes we have

$$\Phi_q(z,\varphi) = \frac{3}{4}a^2(\mathbf{md})\frac{z}{(z^2+a^2)^{5/2}}(3\cos^2\varphi - 1) \qquad (4)$$

where a is the turn radius.

For completeness we give an expression for flux from a dipole $\mathbf{m}$ placed at a turn axis

$$\Phi_d(z) = \frac{1}{2}m_z\frac{a^2}{(z^2+a^2)^{3/2}}\cdot \qquad (5)$$

If the dipole is shifted from the axis at a distance $\mathbf{r}$ then it produces quadrupolar signal

$$\Phi_q(z) = -\frac{3}{8}a^2(\mathbf{mr})\frac{z}{(z^2+a^2)^{5/2}}\cdot \qquad (6)$$

2 Experimental

Measurements were done with a home-built SQUID magnetometer. Our device [6,7] consisted of a pick-up coil (two turns from Nb-Ti wire joined in opposite) connected to the DC SQUID sensor. The distance between the turns was 4 cm and their radius a = 1.2 cm. The coil was placed outside a dewar-insert made from glass. Inside it a tube glued from aluminium foil and paper with bifilar heater and Si-diode thermometer was mounted. The main cryostat was made from fibreglass and shielded with three thin-wall permalloy tubes. The middle tube has a longitudinal demagnetising coil, the inner one – longitudinal and toroidal demagnetising coils. No superconducting shield was used, even for the SQUID sensor. Both components of residual magnetic field at the sample position, measured with a superconducting lead sphere, did not exceed 1.5×10^{-2} A/m (transverse component was estimated using Eq. (6)). The sample was placed in a basket made from thin 1 mm wide paper tape and suspended with a cotton thread 65 cm in length. No glue was used in the preparation of this sample holder. All these precautions permitted us to avoid parasitic magnetic signals. A stepper motor was used to move the sample along the coil axis. Calibration of the device was performed with known magnetic flux produced by dipole and quadrupole coils. Axial and transversal magnetic fields were produced by a thin solenoid and saddle-shaped coil respectively wound with 0.1 mm copper wire.

Registered dependences of the magnetic flux on the position of the sample along the coil axis were fitted by an expression

$$\begin{aligned}\Phi(z) = \Phi_d(z) - \Phi_d(z+4) + \Phi_q(z,0)\\ - \Phi_q(z+4) + \text{linear term}\end{aligned} \qquad (7)$$

where linear contribution results from the movement of a stepper motor. From this fit, values of dipole and quadrupole moments as well as their positions respective to the coil were obtained. Signs of the dipole moment and applied magnetic field were determined with respect to the chosen positive direction along the coil axis (from the bottom to the top of the cryostat). To get the real position of the sample centre respective to the coil, the measurements in positive and negative axial magnetic field with the same value (usually 8 A/m) were performed. From fitting the difference of these two curves, which corresponds to the volume susceptibility of the sample, the position of the sample centre was determined with an accuracy of about 0.1 mm.

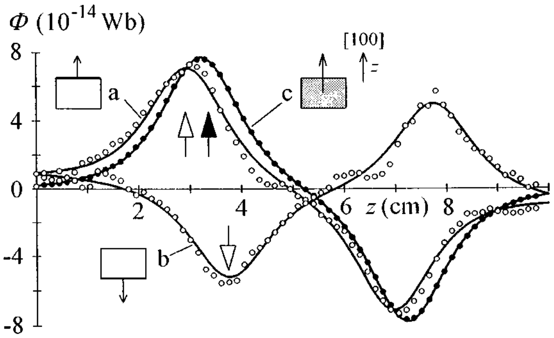

Fig. 1. Signals $\Phi(z)$ measured along [100] at T = 4.2 K: (○) in zero magnetic field, (●) H = 15.5 A/m; signal in field is multiplied by 0.05. Curve (a) was obtained after quick cooling ($m_z = 1.34 \times 10^{-9}$ J/T), curve (b) - after slow cooling ($m_z = -1.07 \times 10^{-9}$ J/T). For curve (c) $m_z = 3.05 \times 10^{-8}$ J/T. Big arrows show the positions of extremes.

The sample was cut using a wire saw from an ingot prepared at the Kapitza Institute for Physical Problems. At the beginning the sample had dimensions $5.5 \times 4.4 \times 3.7$ mm^3. Before measurements the sample was etched in 1 : 1 HCl for 1/2 hour (surface layer 5−10 μm was removed) and carefully washed in distilled water and pure acetone.

3 Results

In some magnetisation measurements of the MnF$_2$ sample in small magnetic fields we have observed that, for directions [100] and [010], magnetisation does not saturate below T_N. Measurements without magnetic field revealed the existence of spontaneous magnetisation in these directions. Its temperature dependence resembles that of a magnetic sublattice. If one subtracts this spontaneous magnetisation from magnetisation measured in a magnetic field then one obtains a constant value below T_N, as is to be expected.

The sign and the value of spontaneous magnetisation were occasionally unstable, that is illustrated in Figure 1. Curve (a) was measured after a quick (about 20 s) cooling of the sample from 300 K to 4.2 K. Repeating the scans at several elevated temperatures the change of the dipole sign was observed in the temperature range of 40−50 K. Curve (b) was obtained after a slow (about 10 min) cooling from 80 K to 4.2 K. Subsequent slow coolings have

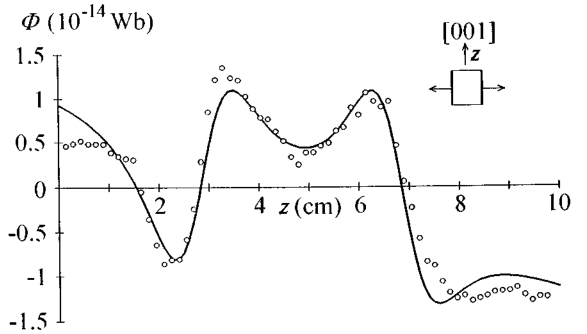

Fig. 2. Signal $\Phi(z)$ measured along [001] in zero magnetic field at $T = 4.2$ K. Solid line is the least-squares fit with $m_z = -8.4 \times 10^{-11}$ J/T, $(\mathbf{md}) = -5.36 \times 10^{-12}$ (J/T) m.

no influence on the sign or value of the spontaneous magnetisation. It is important to note that the sign and the position of the dipole have changed simultaneously, and that the dipole position did not coincide with the sample centre, which was determined from measurement in a magnetic field (curve c). From these results we may conclude that in both cases spontaneous surface magnetisation was directed outward from the sample.

There was no regular dependence of the sign and the position of the dipole on the cooling rate – those changes were sometimes detected and sometimes not. But when the dipole was observed it was always directed outward from the sample.

Spontaneous magnetisation was definitively observed for [100] and [010] directions. Along the tetragonal axis [001] the dipole signal was at least an order of magnitude smaller and may result from inclination between the sample and the coil axes. If this dipole signal was sufficiently small then a quadrupolar signal was observed, Figure 2. Note that this quadrupolar signal may be explained by spontaneous magnetisation of (100) and (010) faces of the sample with magnetisation directed outward. A similar signal could, in principal, be observed if the sample is magnetised in (001) plane and if it is moved not along the coil axis exactly, see equation (6). In order to check this we have made the same experiment in a perpendicular magnetic field. Indeed, changing sign and value of the field it was possible to obtain various quadrupolar signals, but due to chaotic changes in radial position of the sample these curves were so noisy and irregular that we have no doubt that the quadrupolar signal depicted in Figure 2 is really caused by surface magnetisation of the sample.

Cooling the sample from $T > T_N$ in magnetic field has minor influence on the spontaneous magnetisation. In Figure 3 we present the temperature dependences of spontaneous magnetisation measured in zero magnetic field after cooling the sample from 85 K in zero magnetic field and in fields up to 3100 A/m. This result is a strong evidence against ferromagnetic impurities, weak ferromagnetism or a piezomagnetic [10] origin of the spontaneous magnetisation because in all these cases the direction of the moment should coincide with the direction of the magnetic field in which the sample was cooled down. Note, that in work [11] a magnetic field, an order of magnitude smaller than used by us was sufficient to change the sign of piezomagnetic moment of FeF_2. (From our measurements it follows that an even smaller magnetic field of 8 A/m is sufficient to change the sign of piezomagnetic moment of FeF_2.)

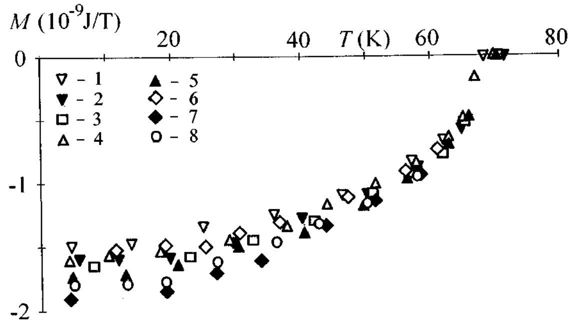

Fig. 3. Temperature dependences of spontaneous magnetisation measured along [100] in zero magnetic field after cooling the sample from 85 K in different magnetic fields in the following sequence: 1) $H = +320$ A/m; 2) $H = -320$ A/m; 3) $H = 0$; 4) $H = +800$ A/m; 5) $H = -800$ A/m; 6) $H = +3100$ A/m; 7) $H = -3100$ A/m; 8) $H = 0$.

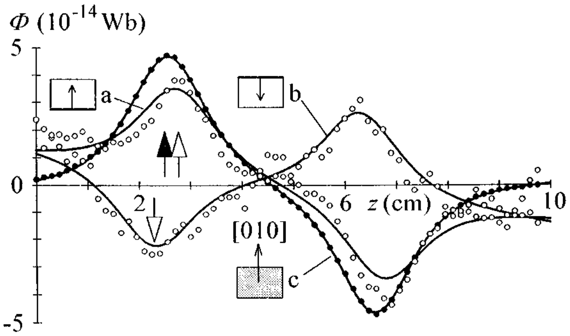

Fig. 4. Signals $\Phi(z)$ measured on the gold coated sample along [010] at $T = 4.2$ K: (○) in zero magnetic field, (●) $H = 8$ A/m; signal in field is multiplied by 0.1. Curve (a) was obtained after slow cooling ($m_z = 5.91 \times 10^{-10}$ J/T), curve (b) - after quick cooling ($m_z = -5.88 \times 10^{-10}$ J/T). For curve (c) $m_z = 9.27 \times 10^{-9}$ J/T. Big arrows show the positions of extremes.

To alter the conditions at the sample surface we have evaporated the gold film over the whole surface of the sample. We observed that the spontaneous magnetisation has changed its sign. In Figure 4 the results measured in the [010] direction at $T = 4.2$ K after slow (curve a) and quick (curve b) coolings are shown. The centre of the sample was determined from measurement in a magnetic field (curve c). In scan (a) positive magnetic moment is placed at the lower face of the sample, in scan (b) negative magnetic moment is placed at its upper face. The same change of the dipole sign was sometimes observed for the [100] direction. Again, as for a clean surface, there was no regular dependence of the sign and the position of the dipole on the cooling rate, but with gold film at the surface the magnetisation was always directed inward to the sample.

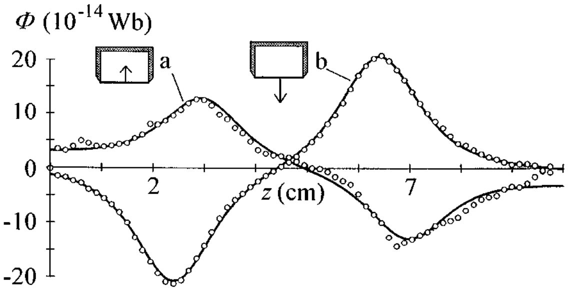

Fig. 5. Signals $\Phi(z)$ measured along [100] in zero magnetic field at $T = 4.2$ K when large jump in dielectric constant was only at one (100) face (all other faces were coated with alcohol solution of rosin). Curve (a): gold surface ($m_z = 2.26 \times 10^{-9}$ J/T); curve (b): clean surface ($m_z = -4.1 \times 10^{-9}$ J/T).

From the above experiments it became clear that the sign of spontaneous surface magnetisation is determined by the electric properties of a substance which contacts with the sample. Room temperature dielectric constant of our sample was equal to $\varepsilon \approx 6.5$ what is significantly different from $\varepsilon \approx 1$ of gaseous helium and very large ε of a metal. One of the main problems in interpretation of our experiments resulted from contributions of all sample faces to the measured signal. We tried to avoid this difficulty by adjusting appropriate conditions at the sample surface so that a large jump in dielectric constant was only at one face. For this purpose gold was mechanically removed by polishing on glass plate with tiny corundum powder from both faces (010), both faces (001) and one face (100). After that clean surfaces were covered with alcohol solution of rosin ($\varepsilon \approx 7$, this value is time-dependent due to the evaporation of alcohol). Then the sample was dried and mounted for measuring magnetisation along [100] (gold surface down). The obtained result is shown in Figure 5 by curve (a). Spontaneous magnetisation is directed inward. After removing gold, curve (b) was obtained. Spontaneous magnetisation is directed outward from the sample. In both cases the magnetic moment was placed about 2 mm below the centre of the sample (positions of extremes of the curves (a) and (b) do not coincide because of different lengths of the suspension thread). The smaller signal with gold cover probably results from some damage of the film continuity that was established visually. If we suppose that the whole surface is magnetised uniformly, then the magnetisation density for curve (b) will be about 2×10^{-4} J/T m^2.

In a separate experiment we have investigated the influence of water on spontaneous magnetisation. For this purpose all faces of the sample, except one (100) face, were covered with a thin layer of Stycast 1266 ($\varepsilon \approx 7.5$). Then magnetisation along the [100] axis was measured on clean and dry surface (100). The result is shown in Figure 6, curve (a). The magnetisation is directed outward. After wetting of the clean surface the magnetisation has changed its sign (curve b) and become directed inward. When the sample was dried again, the sign and the value of the magnetisation were restored (curve c, this measurement was done two days later). In all cases the magnetic moment was placed below the sample centre (scans in magnetic field are not shown for clarity). These results show that the influence of water ($\varepsilon \approx 80$) is the same as that of a metal – the spontaneous magnetisation becomes directed inward.

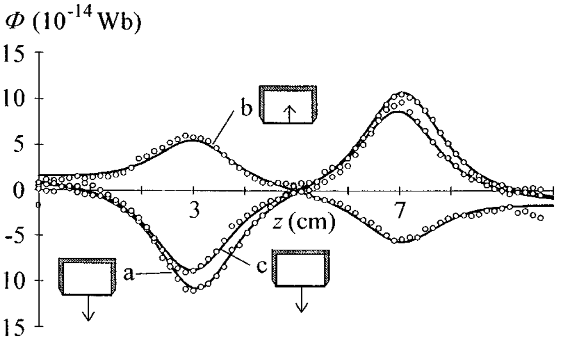

Fig. 6. Signals $\Phi(z)$ measured along [100] in zero magnetic field at $T = 4.2$ K when the large jump in dielectric constant was only at one (100) face (all other faces were coated with Stycast 1266). Curve (a): dry surface ($m_z = -2.21 \times 10^{-9}$ J/T); curve (b): wet surface ($m_z = 0.99 \times 10^{-9}$ J/T), curve (c): dry surface ($m_z = -1.82 \times 10^{-9}$ J/T).

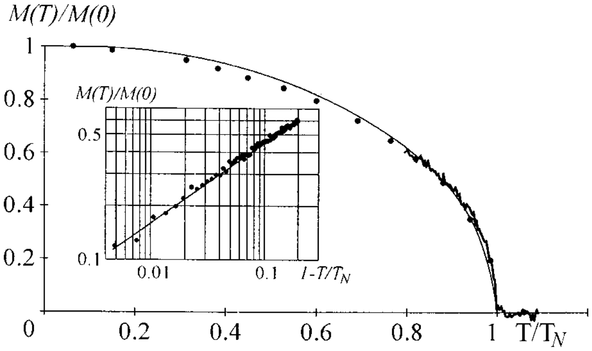

Fig. 7. Temperature dependence of spontaneous surface magnetisation measured along [100] when the large jump in dielectric constant was only at one (100) face (all other faces were coated with alcohol solution of rosin). Points were obtained at fixed temperatures on warming up the sample. Solid line is the record of SQUID output when the sample was slowly cooled down from $T > T_\mathrm{N}$. Thin line is the Brillouin functions for total spin $S = 5/2$. Insert: log-log plot of spontaneous magnetisation near T_N *versus* the reduced temperature $t = 1 - T/T_\mathrm{N}$. Straight line corresponds to the critical exponent $\beta_\mathrm{S} = 0.45$.

Unfortunately we had no possibility to control the state of the sample surface during the experiment. Oxygenation of the surface with time or due to mechanical and chemical treatment is very unlikely because $\mathrm{MnF_2}$ has more ionic character than manganese oxides (Pauli's electronegativity is 1.5 for manganese, 3.5 for oxygen and 4.0 for fluorine). Nevertheless the main experimental factor – the reproducibility of the results – was employed. Experiments with gold film, alcohol solution of rosin and water were repeated three times. Each time before changing the condition at the sample surface it was mechanically polished, etched in diluted HCl

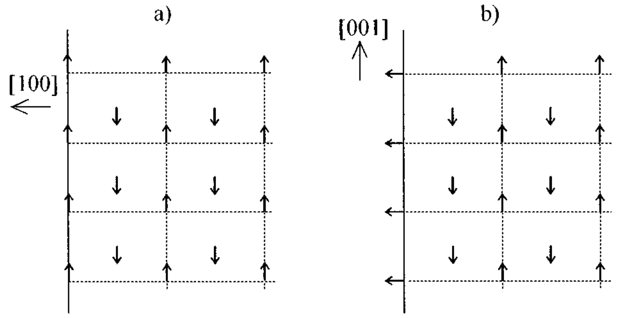

Fig. 8. Schematic representation of the arrangement of magnetic moments at the surface of the MnF_2 crystal. (a) ε (ambient matter) $= \varepsilon(MnF_2)$, (b) ε (ambient matter) $< \varepsilon(MnF_2)$.

and washed in water and acetone. Thus new inner parts of the sample came to the surface and its dimensions became gradually smaller. Obtained results were qualitatively the same as described before, only some changes in amplitude of the dipole signal were observed. In one case when five faces of the sample were covered with alcohol solution of rosin and the last (100) face was clean we obtained $m_z(4.2\ \text{K}) = 6.09 \times 10^{-9}$ J/T – the largest value we have ever observed (approximately the same moment has its volume in field 8 A/m). This value corresponds to the density of surface magnetisation 2.9×10^{-4} J/T m^2 or $5\mu_B$ per surface ion Mn^{2+}. This large signal permitted us to make accurate measurements of the temperature dependence of surface magnetisation presented in Figure 7. Obtained points are close to the Brillouin function for total spin $S = 5/2$. The signal above T_N was less than 10^{-11} J/T (the noise level). The insert in Figure 7 shows a log-log plot of spontaneous magnetisation *versus* the reduced temperature; solid line corresponds to the critical exponent $\beta_S = 0.45$. Critical exponents of spontaneous magnetisation obtained by us in other experiments are in the range $0.42-0.47$ that is significantly different from the critical exponent for the sublattice magnetisation obtained by the magnetic X-ray scattering measurements [12]. This supports our view that spontaneous magnetisation is not a volume effect.

We have also made an experiment on the sample entirely covered with alcohol solution of rosin. No signal was observed in this case.

4 Discussion

Let us summarise our observations.

1. Spontaneous magnetisation of surfaces (100) and (010) of single crystal MnF_2 below T_N were discovered. The critical exponent for its temperature dependence $\beta_S = 0.45$ is significantly different from the critical exponent for the sublattice magnetisation.

2. The direction of the spontaneous magnetisation is determined by the difference in dielectric constants of the sample and the material it contacts with. The magnetisation is directed toward the substance with smaller dielectric constant.

4. Position of the observed spontaneous magnetic moment coincides with the surface under investigation (within our resolution).

5. Spontaneous magnetisation is absent when the sample is covered by a substance with the same value of dielectric constant.

6. Density of the surface magnetisation at $T = 4.2$ K reaches 2.9×10^{-4} J/T m^2 or $5\mu_B$ per one surface ion Mn^{2+} that corresponds to $S = 5/2$.

We must emphasise that these results were confirmed by observation of both dipolar and quadrupolar signals. Because magnetisations of opposite crystal faces have opposite directions the resulting dipole moment is strongly dependent on the distribution of magnetisation over the sample surface that itself is determined by the cleanness of the surface. The adsorption of moisture may lead to very confusing and non-reliable results. This circumstance explains the instability of the results and their dependence on cooling rate. Quadrupolar contribution was usually masked by a dipolar one, its observation was possible if magnetisations of opposite faces were almost equal.

Our results are radically different from the remanent magnetisation observed in disordered antiferromagnets [1–4]. First of all, direction of the remanent magnetisation coincides with the [001] axis whereas spontaneous magnetisation is perpendicular to it. Second, sign and value of the remanent magnetisation is determined by the magnetic field in which the sample is cooled down from $T > T_N$ even if this field is very low (~ 0.1 A/m). In the case of spontaneous magnetisation the effect of much higher magnetic field was negligible. A very important fact is the existence of a preferential direction of spontaneous magnetisation which is dependent on dielectric constant of ambient environment. This experimental observation clearly points to the *extrinsic* origin of the spontaneous surface magnetisation.

We attribute spontaneous magnetisation to the surface anisotropy. Physically this anisotropy is analogous to the single-ion anisotropy arising if the crystalline field is distorted from cubic symmetry [13]. In distinction from the single-ion anisotropy which for ions Mn^{2+} in bulk MnF_2 is *uniaxial*, the surface anisotropy is *unidirectional* and its sign is determined by the difference of the dielectric constants at the sample surface so that the spontaneous magnetisation always points toward the lower ε.

At first sight, unidirectional (ferromagnetic) ordering of the surface is very unlikely if the bulk is ordered antiferromagnetically. For example, the surface of antiferromagnetic NiO is ordered antiferromagnetically [14] with the critical exponent $\beta_S = 0.89$ [15]. But such a large value of β_S means that the surface exchange interaction J_S is equal or smaller than the bulk J [16]. The critical exponent measured by us for MnF_2 is much smaller, $\beta_S = 0.45$ (Fig. 7, insert). From comparison with the results of Monte Carlo simulations [16] we obtain $J_S/J \approx 1.35$. As this ratio is less than 1.6, so according to work [16], temperature of surface ordering should equal that of the bulk; in our case as the nearest-neighbour exchange is positive [17] it means ferromagnetic ordering of the surface and antiferromagnetic ordering of the bulk. This was confirmed by our simultaneous measurements of spontaneous magnetic moment and specific heat (it will be published elsewhere).

Figure 8 illustrates our observations. Spontaneous magnetisation arises at (100) or (010) surfaces if there is sufficient difference in dielectric constants of the sample and ambient matter. Its maximal value corresponds to exactly one monolayer of Mn^{2+} ions. In an idealised picture all these ions lie in the upper-most layer and occupy the same position in an elementary cell. Their magnetic moments are always parallel in the antiferromagnetic state (layer is ordered ferromagnetically). Only one difference between the bulk and surface layers is that magnetisation of the bulk layers is oriented along [001] whereas that of the surface layer is perpendicular to it.

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