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Influence of ferromagnetic substrate on the magnetoresistance of Cr film across a nonmagnetic insulating layer

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(5 nm) Cr/(x nm) MgO/Mn_{0.52}Zn_{0.48}Fe₂O₄ (MnZn spinel) substrate (1<x<7 nm), as well as (5 nm) Cr/(7 nm) MgO/glass substrate structures have been grown using molecular beam epitaxy. The influence of the MnZn spinel on the in-plane transport and magnetotransport properties of the Cr layer were studied. The existence of pinholes in the MgO layer was explored by evaluating resistance versus temperature, R(T), dependencies. A hump was observed on the R(T) curves for the MgO layer thickness ($t_{\rm MgO}$) less than 5 nm. This results from the electrical coupling between the Cr film and the MnZn spinel substrate through the pinholes in the MgO layer. A reversal of the magnetoresistance (MR) sign in Cr film was observed when $t_{\rm MgO}$ is less than 7 nm. It is thought that the effect of the electrical coupling through the pinholes and the magnetostatic coupling are not the reasons for the observed negative MR. A model is proposed which suggests that the magnetic structure in the Cr film adjoining a pinhole is distorted because of the exchange coupling. Such areas in the Cr film are thought to contribute to the negative MR. © 2003 American Institute of Physics. [DOI: 10.1063/1.1606520]

I. INTRODUCTION

The spin-dependent tunneling between two ferromagnetic (FM) films across an insulator (I) has significant potential for applications in digital storage and magnetic sensor technologies. ¹⁻⁴ Magnetoresistance (MR) greater than 20% at room temperature has been reported in spin tunnel junction. ⁵⁻⁷ The magnitude of the tunneling MR (TMR) at low temperatures closely agrees with the prediction of Julliere's model. This model is based on the difference in the density of states for the two spin directions at E_F of the itinerant electrons in the FM. A large TMR is predicted when a FM electrode with a high spin polarization around E_F is employed. Magnetite (Fe₃O₄), due to its half-metallic nature, has attracted much attention as a potential material for magnetic electrode layers in tunneling junctions. ¹¹⁻¹³

For tunnel junction devices based on a magnetite electrode, MgO has potential applications as an insulating layer because of the small lattice mismatch (0.3%) between MgO and Fe₃O₄. Some experimental results for the epitaxial growth of MgO on Fe₃O₄ have been reported. It is important to understand the magnetic interlayer coupling between FM electrodes across the MgO layer for application of this dielectric in tunnel junctions. There are several reports on investigations of interlayer coupling across the MgO layer. Slonczeswski proposed a theoretical model according to which spin-polarized conduction electrons of one- or two-band metallic semi-infinite magnetic layers tunnel from one layer to another across a nonmagnetic insulating interlayer. As a result of this spin-polarized tunneling, an effective Heisenberg-like interlayer coupling between the magnetiza-

tions of the magnetic layers across a nonmagnetic insulator was predicted. The coupling is either ferromagnetic or antiferromagnetic and its strength decreases rapidly with increasing interlayer thickness. The Brillouin light scattering spectra of Fe-whisker/5 ML MgO/25 ML Fe/24 ML Au (100) structure shows no evidence of exchange coupling between the Fe film and the Fe-whisker substrate. ¹⁸ van der Zaag *et al.* report a strong interlayer coupling between Fe₃O₄ electrodes when the MgO spacer layer thickness is less than 1.2 nm. ¹⁹ The aim of the present work is to investigate the influence of the ferromagnetic substrate on the MR of the Cr film through the MgO spacer layer and to establish how this influence is affected by the thickness of the MgO layer.

The present work was carried out using (5 nm) Cr film/ $(1-7 \text{ nm}) \text{ MgO/Mn}_{0.52}\text{Zn}_{0.48}\text{Fe}_2\text{O}_4$ spinel substrate structure. The MnZn spinel, $Mn_{1-x}Zn_xFe_2O_4$, could be considered as a solid solution of two spinels: MnFe₂O₄ and ZnFe₂O₄. The divalent Zn ions take up the A sites (tetrahedral sites) displacing an Fe^{3+} ion into the B sites (octahedron sites) to take up the vacated Mn²⁺ ion position, forming the structure $[(1-x)\text{Fe}^{3+}x\text{Zn}^{2+}]_A[(1-x)\text{Mn}^{2+}(1+x)\text{Fe}^{3+}]_B\text{O}_4$. The MnZn spinel is a ferrimagnetic material. It was selected as a substrate for the present study because it shows a relatively high conductivity ($\sim 5000 \,\Omega\,\mathrm{cm}^{-1}$) at room temperature and a very high resistivity at low temperature. These characteristics make it possible to investigate the influence of interlayer coupling on the transport properties of the Cr film when no current flows through the substrate at low temperature. They also make it possible to explore the existence of pinholes in the MgO layer by evaluating the resistance versus temperature dependencies of the specimens. The Cr film was employed because of its low MR value. Therefore, the expectation was that any influence of the substrate through the nonmagnetic MgO layer should be readily observed.

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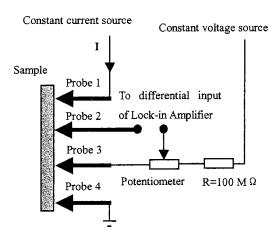


FIG. 1. Schematic drawing of electrical compensator.

II. EXPERIMENT

The Cr and MgO films were prepared in a molecular beam epitaxy system (DCA Instruments Oy, Turku, Finland) from bulk Cr and MgO with a 99.995% purity, using a multipocket electron-beam gun. The base pressure was lower than 5×10^{-10} mbar. The deposition rate of Cr and MgO was 0.1 and 0.02 Å/s, respectively. The growth rate was controlled using an INFICON IC-5 process controller and quartz thickness monitors. The composition of the MnZn spinel was determined as $Mn_{0.52}Zn_{0.48}Fe_2O_4$ by using an energy dispersive x-ray analysis system.

The MnZn spinel substrate was mechanically polished down to 1 μ m grain size, annealed at 250 °C for 30 min in a vacuum prior to deposition and held at 100 °C during deposition. The low substrate temperature during the deposition was selected to prevent film-substrate interdiffusion. (5 nm) $Cr/(x \text{ nm}) MgO/MnZn \text{ spinel substrate } (0 \le x \le 7 \text{ nm}), \text{ as}$ well as (5 nm) Cr/(7 nm) MgO/glass substrate structures were grown and their transport and magnetotransport properties were measured. Four gold strips with a separation of 2 mm and a thickness of 80 nm were deposited onto the Cr film as electrodes. A variable external magnetic field (0–2 T) was applied in the plane of the thin film and parallel to the current direction. The four-probe method was employed in the resistance and magnetoresistance measurements. An electrical compensator, as shown in Fig. 1, was employed to improve measurement resolution. Before cooling down the sample or applying the external magnetic field, V was set to 0 by adjusting the potentiometer. The change in resistance for the sample (ΔR) between probes 2 and 3 can be presented as $\Delta R = \Delta V/I$, where ΔV is the potential change which is measured by a lock-in amplifier (Stanford Research System SR 830) and I is the current flowing through the sample. An ac current (74 μ A) with a frequency of 187 Hz was employed in the present work. X-ray powder diffraction measurements were performed using a Siemens DACO_MP diffractometer. Hysteresis loop and magnetic moment measurement were carried out using a MicroMag 3900 vibration sample magnetometer (VSM) at room temperature. Surface roughness measurements were performed done using a Digital Instrument MMAFM-2 atomic force microscope (AFM).

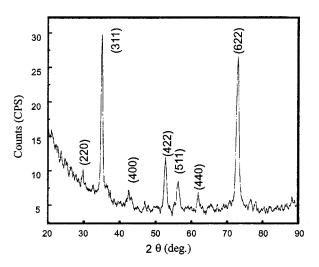


FIG. 2. θ –2 θ x-ray diffraction pattern of the MnZn spinel substrate.

III. RESULTS AND DISCUSSION

The θ -2 θ x-ray diffraction patterns indicated that the MnZn spinel substrates were polycrystalline (Fig. 2). As expected the structure of the substrates was that of the spinel with a lattice constant of 8.5090 Å. As shown in Fig. 3, VSM results proved that the substrates were soft ferromagnets having a saturation magnetization and coercivity of 27.1 emu/g and 0.37 Oe, respectively, consistent with the expected values for Mn_{0.52}Zn_{0.48}Fe₂O₄. Figure 4 shows the resistivity and MR of the MnZn spinel substrate, at 2 T, as a function of temperature. In the present work, MR is defined as [R(H)]-R(0)]/R(0). An exponential increase in resistivity R(T)as temperature decreases is observed, which is consistent with typical insulator behavior. The activation energy, U= 0.059 eV, was determined by fitting the data to a thermally activated resistivity curve $\rho = \rho_0 \exp(-U/kT)$. A negative MR is observed and its absolute value increases with decreasing temperature. Our results are consistent with earlier studies of the magnetoresistance of $Mn_{1-x}Zn_xFe_2O_4$ (0<x <0.6) ceramics.²¹ For spin-flip scattering, the following temperature dependency of the magnetoresistance is expected: $MR(T) = MR(0)(T_0/T)^2$. Figure 5 shows measured MR values as a function of $T^{-1/2}$. The temperature depen-

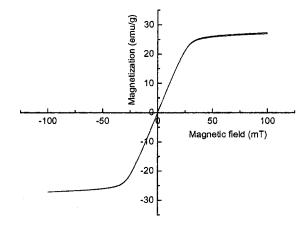


FIG. 3. Magnetization vs magnetic field (M-H) curves for the MnZn spinel substrate at room temperature.

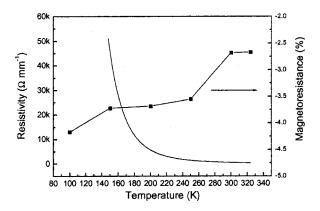


FIG. 4. Resistivity and magnetoresistance of the MnZn spinel substrate at 2 T as a function of temperature.

dency of the MR is found to be in agreement with the prediction of above equation when the temperature is lower than 250 K. A deviation from this dependency is observed at higher temperatures. As shown in the inlet of Fig. 5, a Curie temperature of $T_c = 350 \, \mathrm{K}$ was determined. Therefore, the deviation from the magnetoresistance scaling curve begins at a temperature close to T_c .

In this study, we focus on the influence on the magnetoresistance of the Cr film caused by the ferromagnetic substrate across a nonmagnetic insulating MgO layer. We realized that the MR of Cr may also be affected by the possible formation of an alloy caused by interdiffusion at the Cr-MgO interface. To exclude this possibility, we performed comparative studies on two systems: Cr/MgO/MnZn spinel and Cr/MgO/glass. The two systems were grown under identical conditions, at the same substrate temperature and the same deposition rate. We found that the resistance and magnetoresistance for (5 nm) Cr/MgO/glass does not depend on the thickness of the MgO layer. The representative results for the transport and magnetotransport properties of the Cr film in the Cr/MgO/glass structure are shown in Fig. 6. It was found that the resistivity of the Cr film decreases with decreasing temperature, thus showing a metallic behavior. In contrast to the reported values for bulk Cr crystals, a positive

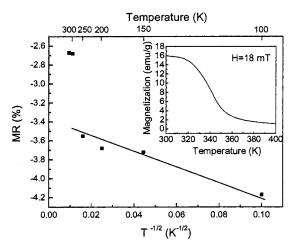


FIG. 5. MR of the MnZn spinel substrate as a function of $T^{-1/2}$. The solid line is a guide for the eye. The dependence of the magnetization on temperature is shown in the inset.

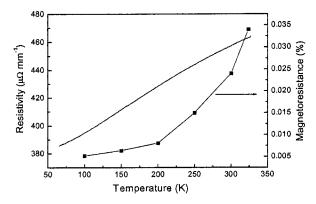


FIG. 6. Resistivity and magnetoresistance of 5 nm Cr thin film at 2 T as a function of temperature. The film is part of the structure (5 nm) Cr/(7 nm) MgO/glass.

MR for the Cr film was observed. Abdul-Razzaq and Amoruso²² reported that Cr film shows antiferromagnetic order when the film thickness is greater than 50 nm and a positive MR has been observed on a Cr film with a thickness of 30 nm.

Figure 7 shows temperature dependencies of the resistances of (5 nm) Cr/(1-7 nm) MgO/MnZn spinel structures normalized to their resistances at 324 K. Two different categories of R(T) curve were obtained: one with a hump and one without. The hump appears on the R(T) curve when the thickness of the MgO layer (t_{MgO}) is less than 5 nm. These curves can be readily explained by the existence of pinholes in the MgO layer. When the MgO layer contains pinholes, the R(T) behavior of the entire Cr film/MgO layer/MnZn spinel structure can be described as two resistors: one from the Cr film and the other from the MnZn spinel substrate, connected in parallel. As shown in Figs. 6 and 4, the resistances of the Cr film and the MnZn spinel substrate decrease and increase, respectively, with decreasing temperature. Based on the parallel resistors model, a maximum resistance should then appear during cooling. At low temperature, the resistance of the substrate is too high and the overall resistance of the structure behaves like that of the Cr film show-

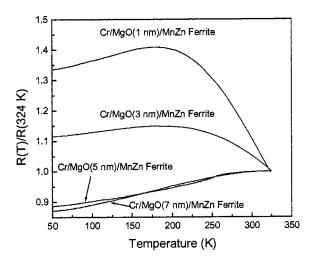


FIG. 7. Resistance of (5 nm) Cr/(0-7 nm) MgO/MnZn spinel structures vs temperature normalized to their respective resistance values at 324 K.

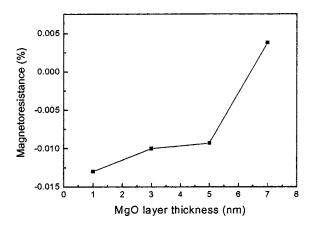


FIG. 8. Temperature dependence of MR at 2 T for (5 nm) Cr/(1-7 nm) MgO/MnZn spinel substrate structures at 100 K.

ing metallic dependence of resistance versus temperature. At high temperatures ($T>200\,\mathrm{K}$), the resistance of the substrate is rather low and therefore the resistance of the entire structure is determined by the substrate and has an insulator-like temperature dependence. We could conclude that the observed hump results from the electrical coupling between the Cr film and the MnZn spinel substrate through the pinholes in the MgO layer. The R(T) curve of the structure is identical to the ones for Cr film in Cr/MgO/glass structures when t_{MgO} exceeds 5 nm. This indicates the absence of electrical coupling through the pinholes at this MgO layer thickness.

Figure 8 shows the temperature dependence of the MR at 2 T for (5 nm) Cr/(1–7 nm) MgO/MnZn spinel substrate structures at 100 K. This is the central result of this publication. In contrast to the observed positive MR of Cr film grown on a MgO/glass substrate, a negative MR is observed when $t_{\rm MgO}{\le}5$ nm. A positive MR behavior coinciding with the one for Cr films grown on glass is observed when $t_{\rm MgO}$ = 7 nm. This implies that the influence of the substrate on the MR in Cr film exists when the thickness of the MgO layer is less than 7 nm and vanishes for values of $t_{\rm MgO}$ of 7 nm and greater. The MR of the Cr/MgO/MnZn spinel was linear for a field up to 2 T which is the maximum field available to us in these experiments.

It is remarkable that the influence of the ferromagnetic substrate on the MR in Cr extends across such a thick layer

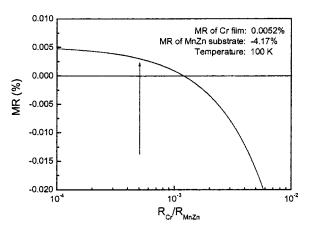


FIG. 9. Calculated MR of the Cr film–MnZn substrate system as a function of film to substrate resistance ratio $(R_{\rm Cr}/R_{\rm MnZn})$.

of MgO, e.g., thickness of 3 and 5 nm. Before starting the discussion of possible physical mechanisms for such a coupling, we must exclude simple electrical coupling through pinholes.

Considering that pinholes exist in the MgO layer when $t_{\text{MgO}} \leq 3 \text{ nm}$ and the negative characteristic MR in the MnZn spinel substrate, one may argue that the observed negative MR in the Cr/MgO/MnZn spinel structure could be attributed to the electrical bridge through the pinholes. We suggest that the electrical coupling through the pinholes is not the reason for the observed negative MR in the structure. First of all, as we have established, there is no electrical coupling through the pinholes when t_{MgO} is 5 nm and yet a negative MR still appears. Moreover, even though pinholes do appear for $t_{MgO} \le 3$ nm, electrical coupling through them still could not result in a negative MR in the entire Cr/MgO/MnZn spinel substrate system. We explain this point here in detail. The resistance of the Cr film and MnZn spinel substrate can be described $R_{\text{MnZn(Cr)}}(H,T) = R_{\text{MnZn(Cr)}}[1$ $+MR_{MnZn(Cr)}(H,T)$], where $R_{MnZn(Cr)}$ is the resistance at zero field and $MR_{MnZn(Cr)}(H,T)$ is the magnetoresistance coefficient of the Cr film and MnZn spinel substrate under the external magnetic field (H) at temperature (T), respectively. Based on the two parallel resistors model, the MR of the entire film-substrate system is

$$MR_{f-s}(H) = \frac{MR_{Cr}(H,T)[1 + MR_{MnZn}(H,T)] + \frac{R_{Cr}}{R_{MnZn}}MR_{MnZn}(H,T)[1 + MR_{Cr}(H,T)]}{[1 + MR_{MnZn}(H,T)] + MR_{MnZn}(H,T)[1 + MR_{Cr}(H,T)]}.$$
(1)

The calculation result is shown in Fig. 9 as a function of the ratio of the film to substrate resistances. The measured values of the MR for bulk MnZn spinel and Cr film grown on MgO/glass substrate were employed during the calculation; these value are -4.17% and 0.0052%, respectively. This demonstrates that the MR of the film–substrate system is deter-

mined mainly by the MR of the Cr film provided the ratio $R_{\rm Cr}/R_{\rm MnZn}$ is small, even though the MnZn spinel substrate shows a much greater MR than the Cr film. At 100 K, the typical resistance of the Cr film is about 50 Ω and that of the MnZn spinel substrate is some 3 orders of magnitude greater, i.e., 100 k Ω . The MR corresponding to our experimental

TABLE I. Calculated coupling field based on "orange peel" mode.

t (nm)	H (mT)
1	0.161
3	0.155
5	0.148

value for the ratio $R_{\rm Cr}/R_{\rm MnZn}=5\times 10^{-4}$ is marked in Fig. 9 by an arrow. A positive MR was expected, which proved to be contrary to our experimental result. Thus, we conclude that the observed negative MR does not result from electrical coupling through the pinholes and therefore the MR behavior of the MnZn spinel substrate does not play a significant role in the MR of the entire film–substrate system.

The second obvious mechanism that could potentially result in coupling across a nonmagnetic layer is Néel or so-called "orange peel" magnetostatic coupling. This coupling results from the correlated interface roughness of the Cr film and substrate surface. The interlayer coupling between Fe₃O₄ layers separated by a MgO layer, with thickness in the range 0–45 nm, has been investigated. Coupling for the MgO layer thicker than 1.3 nm was attributed to the Néel magnetostatic coupling. The magnetic field due to Néel coupling depends on the surface roughness. The coupling field H could be modeled assuming that the surface has a two-dimensional sinusoidal waviness with amplitude h and wavelength ω^{25}

$$H = \frac{\pi^2 h}{\sqrt{2}\omega} M_s \exp\left(-\frac{2\pi\sqrt{2}}{\omega}t\right). \tag{2}$$

Here M_s is the saturation magnetization of the substrate and t is the thickness of the nonmagnetic spacer. To establish whether Néel coupling could be the reason for the observed effect, surface roughness measurements on the Cr/MgO/ MnZn spinel structures were performed using AFM. We measured the roughness of the bare MnZn spinel substrate, the roughness of the substrate with the layer of MgO deposited on it, and finally the roughness of Cr film deposited on top of MgO. Only a small change in the root-mean-square (rms) roughness value was observed after the deposition of the MgO layer. Typically the change was less than 0.15 nm, indicating that there was a substantial correlation between the lower surface of the Cr film and the surface of the substrate. Therefore, the basic assumption of the "orange peel" model was fulfilled for our films. Yet the rms roughness of the MnZn spinel substrate and the Cr film was rather small, typically in the range of 1.5-1.7 nm. The values of H obtained by substituting the measured values for M_s (5.25 emu/cm^3) and t are presented in Table I. The value $\omega = 412$ nm, obtained from the AFM measurement, was used in our calculation.

The result showed the additional field created in the Cr film due to "orange peel" effect was negligibly small in comparison with the 2 T field at which the MR of the Cr/MgO/MnZn spinel was measured. Therefore, as the magnetic moment of the MnZn substrate was typically saturated in a field of less than 50 mT (Fig. 3), the additional correction

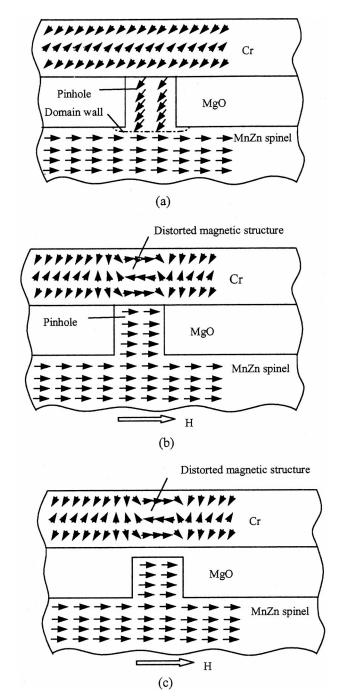


FIG. 10. (a) Schematic diagram of a pinhole. The material in the pinhole forms a domain coupled to the Cr film by exchange interaction. The external field is zero. (b) External magnetic field is applied. This rotates the magnetic moments in the MnZn spinel in the pinhole area and also spins in the Cr coupled by exchange with the MnZn spinel. This creates an area with a distorted magnetic structure where the electrons traveling in the Cr film undergo less scattering. (c) The case when a pinhole is not completely open, i.e., there is a thin layer of MgO preventing electric contact between the Cr and MnZn substrate but still placing the two in close proximity.

due to the "orange peel" field from the substrate seen by the Cr film in the field ranging from 50 mT to 2 T is negligibly small and cannot explain the reversal of MR of the system.

We suggest that the negative MR is the result of the direct magnetic coupling through the pinholes. It is known that the spin structure of Cr film with a thickness below 4.5 nm is commensurate antiferromagnetic with magnetic mo-

ments in the (001) plane. ²⁶ The Cr films grown for our study were polycrystalline. According to our model, the magnetic structure in Cr in the direct vicinity of the pinhole is distorted through an exchange interaction with the MnZn spinel substrate. This is schematically shown in Figs. 10(a) and 10(b). Such a distortion is affected and modified when an external magnetic field is applied. Our model suggests that electrons undergo less scattering in such an area with a distorted spin structure. This is thought to be the possible reason for the negative MR. We further suggest that the distortion of magnetic the structure in Cr takes place even if the pinhole is not completely open, i.e., there is a thin layer of MgO preventing electric contact between the Cr and the MnZn substrate but still placing the two in close proximity, as shown in Fig. 10(c). From our experimental data, we can estimate the thickness (λ_c) of MgO layer separating the Cr from the MnZn substrate at which the coupling between the latter two is still formed. Our results suggest that the 5 nm thick MgO layer does not contain electric pinholes. However as the 3 nm layer does contain such pinholes, we can conclude that the 5 nm MgO film contains "near" pinholes, i.e., areas where the MgO thickness is reduced to 1-2 nm. Therefore, as such films with a 5 nm MgO layer still have a negative MR, we can conclude that λ_c is in the range of 1–2 nm. Reduction in the absolute value of MR with increasing thickness of the MgO layer in the range 1-3 nm can then be understood as follows: as the fraction of the interface covered by pinholes decreases with increasing MgO layer thickness, the number of areas in Cr with a distorted magnetic structure decreases, and each of them leads to negative contribution to the MR. This value of λ_c suggests that such coupling is due to exchange interaction between the Cr film and the MnZn spinel and is in agreement with results of Ref. 19. Further work on the characterization of the pinhole size and density in the samples is ongoing.

IV. CONCLUSIONS

The influence of the MnZn spinel on the in-plane transport and magnetotransport properties of the Cr layer was investigated by using (5 nm) Cr/ (1–7 nm) MgO/MnZn spinel substrate as well as (5 nm) Cr/(7 nm) MgO/glass substrate structures. A distinct influence of the MnZn spinel substrate on the magnetoresistance of Cr film was observed when the thickness of the MgO spacer layer ($t_{\rm MgO}$) was less than 5 nm. The influence is so noticeable that the sign of MR is reversed, resulting in a negative magnetoresistance. We have established that the pinholes exist when $t_{\rm MgO}$ is less

than 3 nm by evaluating the resistance versus temperature dependencies of specimens. The effects of electrical coupling through the pinholes and magnetostatic coupling are excluded as possible reasons for the observed negative MR. A model is proposed which suggests that the Cr film adjoining the pinholes has a distorted magnetic structure because of the exchange coupling with the MnZn spinel substrate. Such areas in the Cr film are thought to contribute to the negative MR.

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