Magnetic and transport properties of epitaxial and polycrystalline chromium dioxide thin films (invited)

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The magnetic and transport properties of epitaxial and polycrystalline chromium dioxide (CrO_2) thin films have been investigated. They are grown epitaxially on single crystal TiO_2 (100) substrates, and with multiple grain orientations on polycrystalline TiO_2 substrates, by chemical vapor deposition. The films have a Curie temperature (T_C) of 390–395 K, with the epitaxially grown CrO_2 (100) films exhibiting in-plane uniaxial magnetic anisotropy. While the epitaxial samples display metallic characteristics, the polycrystalline films are semiconducting with a dominant grain boundary contribution to the resistance at low temperatures. The magnetoresistance (MR) properties have also been measured with the magnetic field applied in the plane. For the epitaxial films, the MR is negative at temperatures near T_C and is positive at low temperatures. A negative MR is also observed near T_C for the polycrystalline samples. However, unlike the epitaxial films, the MR is found to be negative also at low temperatures, with a significant low field component. The latter is attributed to spin-polarized transport of electrons across grain boundaries.
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I. INTRODUCTION

Magnetic oxide materials possessing a high degree of spin polarization exhibit enhanced spin-dependent transport properties.[1] In particular, spin-dependent conductance has been observed in transport across a macroscopic interface between two ferromagnetic oxide elements by controlling the relative orientation of the elements in a relatively small magnetic field. One simple approach to probe for spin-dependent conductance has been to study the magnetoresistance (MR) properties of polycrystalline samples, both in the form of thin films and bulk ceramics.[1] Measurements on these samples have shown that disruption in the crystalline order at the grain boundaries influences the electrical conduction and results in a large low-field MR component, especially at low temperatures.

Chromium dioxide (CrO_2)is a metallic ferromagnetic oxide that has been widely utilized as a particulate medium in magnetic tapes.[2] The chromium ions in CrO_2 are in the Cr^{+4} state with a magnetic moment of $2\mu_B$ per ion. Band structure calculations have predicted CrO_2 to be half metallic, with almost complete spin polarization at the Fermi level.[3–5] The two $3d$ electrons occupy spin-split t_{2g} subbands, one localized and the other in a half-filled band. They are strongly coupled by the on-site exchange interaction $J_H \approx 1$ eV.[5] Spin-polarized photoemission experiments have confirmed the presence of nearly complete spin polarization at $\sim$2 eV binding energy.[6] However, no spectral weight is observed for both spin electrons at the Fermi energy, E_F. This is rather surprising considering the metallic nature of CrO_2. More consistent with theoretical prediction, Soulen *et al.* have recently obtained a value of 90%$\pm$3.6% for the spin polarization at E_F from superconducting point contact measurements on CrO_2 films.[7] It seems likely that surface segregation effects during surface cleaning may have influenced the photoemission results.

Low-field MR has been reported in polycrystalline films[8,9] and powder compacts[10,11] of CrO_2. Hwang and Cheong have studied the properties of polycrystalline CrO_2 films grown by high-pressure thermal decomposition of CrO_3 on $SrTiO_3$ substrates.[8] In as-grown films, they observe a negative MR of 10% at 5 K in 2 T field. When the films are subsequently heated to higher temperatures, the resistivity increases by about three orders of magnitude at low temperatures and the MR is enhanced to about 24%. It has been suggested that the increase in MR in the postannealed films is a result of modification of the effective intergrain-tunneling barrier through surface decomposition of CrO_2 into insulating Cr_2O_3.

Similar MR results have been reported for pressed powder compacts of CrO_2.[10,11] The compacts have resistivity about three orders of magnitude higher than the value in single crystals, which is attributed to the resistance of the interparticle contact. Hysteretic MR is observed with a MR ratio of 29% at 5 K and 5.5 T field. On increasing the temperature, the MR decreases rapidly and at room temperature the MR is less than 0.1%. Coey *et al.* have also observed that CrO_2 powder diluted with 75% insulating antiferromagnetic Cr_2O_3 powder is near the percolation threshold and further increases the ρ by about three orders of magnitude.[10] At the same time, the MR ratio at 5 K reaches almost 50%. The MR has been ascribed to tunneling between contiguous ferromagnetic particles along a critical path with a spin-dependent Coulomb gap. Using a model for weak link conduction in a critical percolation path in half-metallic particles, the maximum MR predicted for a random orientation of magnetiza-

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tion in the particles is 60%.[10] The observed MR for the diluted CrO_2 compact is thus close to this theoretical prediction. The rapid decrease in the MR with increasing temperate has been attributed to a combination of the exchange contribution to the Coulomb gap and spin-flip scattering processes effective at higher temperature.

In this article we report on the magnetic and resistive behavior of intentionally fabricated polycrystalline CrO_2 films and compare them with their epitaxial counterpart. In addition, we have studied the magnetotransport characteristics of the samples. To provide a direct comparison, both the polycrystalline and epitaxial films are grown under identical process conditions on the same substrate material (TiO_2). This is similar to the approach we have followed in our previous study of manganite films grown on $SrTiO_3$ substrate.[12,13]

II. EXPERIMENT

Chromium dioxide thin films have been grown on (100)-oriented single crystal and polycrystalline TiO_2 substrates by chemical vapor deposition (CVD) using CrO_3 as a precursor. The polycrystalline TiO_2 substrates have an average grain size of about 5 μm and are obtained by cutting and polishing sintered pellets (~99% density) of the material. We have also grown finer-grain polycrystalline CrO_2 films on a seed layer of TiO_2 deposited on oxidized silicon substrates. For this purpose, a 500 Å Ti film is sputter deposited on a SiO_2-covered Si wafer and is oxidized at 400 °C prior to growth of the CrO_2 films. Besides TiO_2, we have also deposited films on Al_2O_3 (0001) substrate. Here, the CrO_2 grows highly oriented, but with multiple domains in the plane because of the sixfold symmetry of the substrate.[14]

Details of the CVD of CrO_2 films have been reported previously.[14–16] In brief, oxygen is used as a carried gas in a two-zone furnace to transport sublimed chromium trioxide (CrO_3) precursor from the source region to the reaction zone where it decomposes on the substrate to form CrO_2 with the evolution of O_2. The phase purity and morphology of the films is dependent on the surface and source temperature and the oxygen flow rate. Single-phase films have been obtained at substrate temperatures of 390–450 °C, with a source temperature of around 280 °C, and oxygen flow rate of ~100 sccm.

We have also developed a method for the selective-area growth of CrO_2 films.[16] This is based on our initial observation that CrO_2 grows readily on a clean TiO_2 surface, but not on amorphous SiO_2. The selective-area growth process has been employed to directly deposit patterned CrO_2 stripes for transport measurements. For these samples, patterned SiO_2 is obtained on the single crystal and polycrystalline TiO_2 substrates using conventional photolithography and lift-off using a ~1000-Å-thick SiO_2 film deposited by rf magnetron sputtering. The CrO_2 grows selectively within the stripe window openings on TiO_2, but not on the adjoining SiO_2 surface. Similarly, in the case of the CrO_2 polycrystalline films grown on the TiO_2 seed layer, the Ti film deposited on top of the SiO_2 is patterned by wet etching prior to oxidation in order to induce selective growth in these regions.

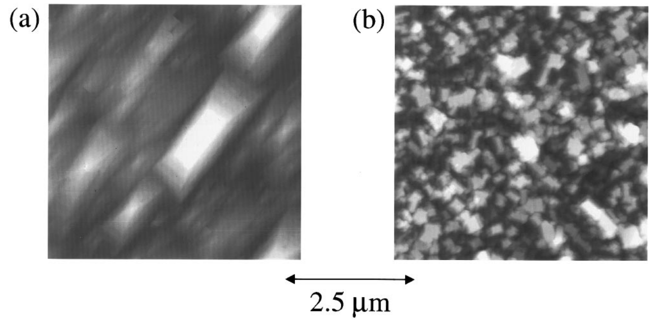

FIG. 1. Surface morphology of CrO_2 thin films ~4000-Å-thick imaged with an atomic force microscope. (a) Epitaxial film grown on a TiO_2 (100) substrate and (b) polycrystalline film grown on a TiO_2 seed layer on oxidized silicon wafer.

The deposited CrO_2 films are ~4000 Å thick, as determined using Rutherford backscattering spectroscopy (RBS) and surface profilometry measurements (for the selectively grown films). They have been structurally characterized using x-ray diffraction, and the crystallinity of the epitaxial films evaluated using RBS channeling measurements. The morphology of the films has been studied using atomic force microscopy (AFM), scanning electron microscopy, and optical Nomarski microscope. Standard four-probe dc method has been used for resistivity and magnetotransport measurements on patterned lines (50 μm wide and 200 μm long) obtained by selective growth. Contacts are made by wire bonding on Au deposited by evaporation on lithographically opened contact pads. The magnetic field for these measurements is applied in the plane of the substrate and aligned either parallel or perpendicular to the current flow direction. The magnetic measurements have been carried out using a superconducting quantum interference device SQUID magnetometer.

III. RESULTS AND DISCUSSION

The surface morphology of the epitaxial and polycrystalline films has been characterized using AFM. Figure 1 shows the AFM images for 4000-Å-thick CrO_2 films grown on a TiO_2 (100) substrate and polycrystalline TiO_2 seed layer on oxidized silicon wafer, respectively. The film grown on TiO_2 (100) is epitaxial [Fig. 1(a)], exhibiting platelet-like features elongated along the ⟨010⟩ direction and has a surface roughness rms value of about 40 Å. The film grown on the TiO_2 seed layer is polycrystalline [Fig. 1(b)], with an average grain size of 2000–3000 Å and a rms roughness of 400 Å. Larger grain films have been grown on a polycrystalline TiO_2 substrate, where one observes a repilation of the polycrystalline morphology of the substrate with an average grain size of ~5 μm (not shown).

The epitaxial and polycrystalline nature of the respective films has been confirmed using x-ray diffraction. We have previously reported on the x-ray characterization results for the epitaxial films.[15,16] In the case of the polycrystalline films, we find that the films grown on the polycrystalline TiO_2 substrate have a completely random orientation, whereas the ones grown on TiO_2 seed layer display some degree of (110) texture.

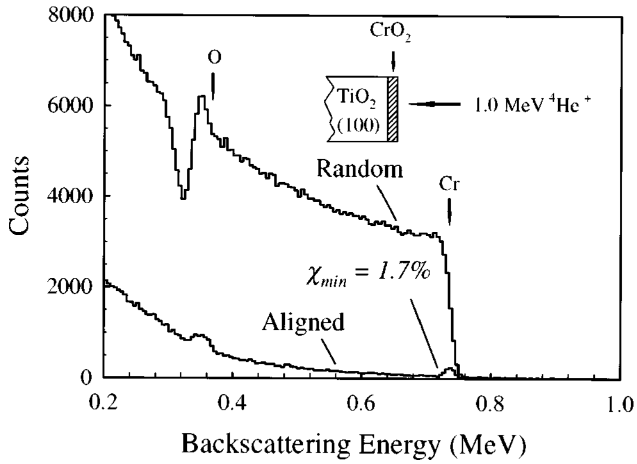

FIG. 2. Rutherford backscattering spectrum of intensity vs energy for both channeling and random 1.0 MeV $^4He^+$ ions backscattered from a 4000-Å-thick epitaxial CrO_2 film on a $TiO_2(100)$ substrate. The low value of χ_{min} suggests a very high crystalline quality of the film.

The crystalline quality of the epitaxial films has been further investigated by RBS ion channeling. Figure 2 shows the aligned ⟨100⟩ and random RBS spectra (1.0 MeV $^4He^+$ ions) for a 4000 Å CrO_2 film on TiO_2 (100). The ratio (χ_{min}) of the backscattered yield for CrO_2 along ⟨100⟩ to that in a random direction is 1.7%. This is consistent with the previously reported x-ray diffraction results on epitaxial films, suggesting a very high degree of crystalline perfection.[15,16]

We have characterized the magnetic properties of the films from the SQUID measurements. From plots of the spontaneous magnetization as a function of temperature, a sharp magnetic transition is observed for both the polycrystalline and epitaxial films, with a Curie temperature (T_C) of 390–395 K. The value of the saturation magnetization at low temperatures is ~650 emu/cm^3, corresponding closely to the full theoretical moment of 2 μ_B per Cr ion observed in the bulk. Figures 3(a) and 3(b) display the hysteresis loops at 5 K for an epitaxial and polycrystalline CrO_2 film, respectively, with the field applied parallel to the plane. For the epitaxial film, with plots displayed for the field applied along the ⟨001⟩ and ⟨010⟩ directions, it is clear that the magnetic easy axis lies along ⟨001⟩ (*c* axis) with close to ideal 100% remanence. The ⟨010⟩ (*b* axis) is the magnetic hard axis direction exhibiting a reversible behavior. At 5 K, the coercive (H_c) and anisotropy (H_K) fields are determined to be 15 and ~1350 Oe, respectively.[15] The polycrystalline film [Fig. 3(b)] exhibits a much more gradual and rounded hysteresis loop with a higher value for H_c (~40 Oe). This is to be expected for the average of a random orientation of grains.

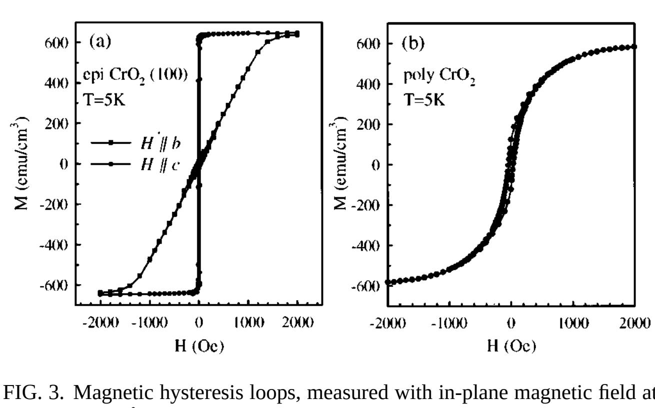

FIG. 3. Magnetic hysteresis loops, measured with in-plane magnetic field at 5 K, for 4000-Å-thick CrO_2 films grown on: (a) $TiO_2(100)$ and (b) polycrystalline TiO_2 substrate. Hysteresis loops are shown for the epitaxial film with the field aligned either along the *b*- or *c*-axis direction, wherea the field is set in an arbitrary direction for measurement of the polycrystalline film.

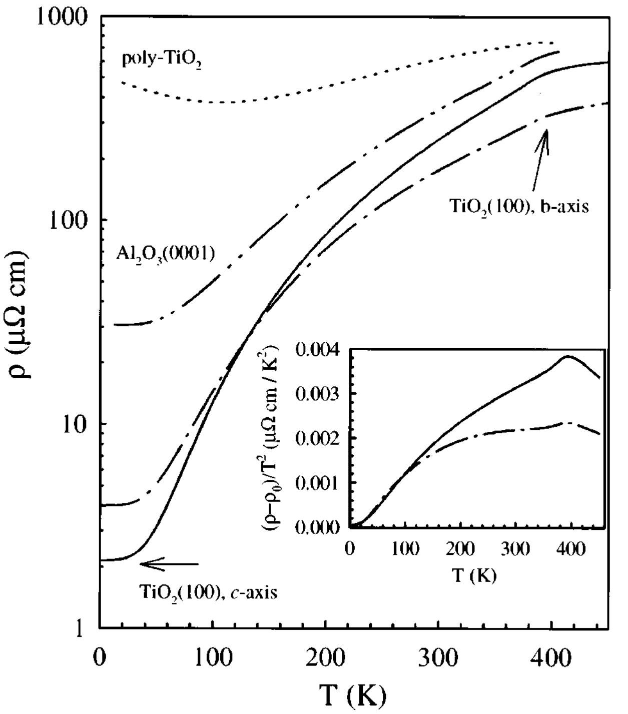

FIG. 4. Resistivity of CrO_2 films grown on TiO_2 (100), Al_2O_3 (0001), and polycrystalline TiO_2 substrates. The resistivity is measured with the current direction either along the *c* axis or the *b* axis for the CrO_2 film on TiO_2 (100) and in an arbitrary direction for the CrO_2 films on Al_2O_3 (0001) and polycrystalline TiO_2 substrates. A plot of $(\rho-\rho_0)/T^2$ vs T for the epitaxial film is shown in the inset.

Patterned films for the resistivity measurements have been deposited using the selective growth method described earlier. Figure 4 plots the resistivity, ρ, as a function of temperature (T) for the epitaxial and polycrystalline film grown on TiO_2. The resistivity of a film grown on a single crystal Al_2O_3 (0001) substrate is also included for comparison. The resistivity of the epitaxial film on TiO_2 (100) clearly exhibits metallic behavior and is anisotropic in the plane, with a small change in slope around the Curie temperature. While the ρ at room temperature along the *c*-axis direction is about 250 $\mu\Omega$ cm and decreases to 2.15 $\mu\Omega$ cm at 2 K, the ρ along the *b* axis is somewhat lower at room temperature (177 $\mu\Omega$ cm), but decreases to a higher value of ~4 $\mu\Omega$ cm at 2 K. The residual ρ at low temperatures is very sensitive to the structural disorder, as suggested from the data of the CrO_2 film grown on Al_2O_3 (0001). Here a much higher residual ρ is observed presumably because of the growth of multiple domains.[14] The resistivity of the polycrystalline film, plotted as a dotted curve in Fig. 4, is significantly higher than the epitaxial film prepared under the same condition. The difference is particularly striking at low temperatures, where the grain boundary resistance dominates for the polycrystalline film.

Annealing the polycrystalline sample in air at a temperature as low as 400 K—which is much below the decomposition temperature of CrO_2—dramatically increases its resistance. On the other hand, no noticeable effect is observed on the resistance of the epitaxial film annealed under the same condition. This provides additional confirmation that grain boundaries are responsible for the increased resistance and that annealing can easily modify the stoichiometry of the grain boundary region. Hwang and Cheong have reported a similar finding in their study of polycrystalline CrO_2 films deposited on a $SrTiO_3$ substrate that are annealed at a somewhat higher temperature.[8] As noted previously, the increased resistance after annealing is most likely a result of partial conversion of CrO_2 to Cr_2O_3 in the grain boundary regions.

In order to examine the temperature dependence of the resistivity for the epitaxial film, we have plotted $(\rho-\rho_0)/T^2$ vs T for the *c*- and *b*-axis directions. This is shown as an inset in Fig. 4, where ρ_0 is the residual resistivity. The slopes for the two plots are similar at temperatures below 100 K, but deviate at higher temperatures. We find that at low temperatures ($\leqslant$40 K), the data is best fitted with $\rho(T)\cong\rho_0+AT^{\alpha}$, with $\alpha\approx3$. In conventional itinerant weak ferromagnets, a $\rho\propto T^2$ dependence is usually observed at low temperatures due to one-magnon scattering process.[17,18] For a perfect half-metal, one-magnon scattering is prohibited in the ground state, and a $\rho\propto T^{4.5}$ dependence has been proposed for two-magnon processes based on a rigid band model.[19] Furukawa has recently argued that unconventional one-magnon scattering process can still be important in a half metal if one takes into account the nonrigid band behavior of the minority band due to spin fluctuations at finite temperatures.[20] Considering a nonrigid band behavior, a $\rho\propto T^3$ dependence has been derived, which is consistent with our observation for CrO_2. Previously, Barry *et al.* have used a phenomenological expression: $\rho(T)\cong\rho_0+AT^2e^{-\Delta/T}$, to fit the resistivity data of epitaxial CrO_2 films grown by high pressure thermal decomposition.[21] While the fit to our data using this expression is reasonable, it is not as good as the T^3 fit, particularly at low temperatures.

We have used the selective-area grown films for MR measurements with the field applied parallel to the film plane. Figure 5 plots the MR as a function of field (0–40 kOe) for the epitaxial film at temperatures of 5 and 380 K. Both the longitudinal and transverse MR, with the current flowing parallel to the *c* axis, are shown. At $T=5$K, the MR is positive with a H^2 field dependence, reaching a value of 25% in the transverse geometry where $I\|c$ and $H\|b$. For the longitudinal geometry ($I\|c$ and $H\|c$), the MR is only about 2% at 40 kOe. At $T=380$ K—a temperature somewhat lower than T_C—the MR is negative and has a value of about 7% at $H=40$ kOe.

The positive MR at low temperatures is attributed to the Lorentz force effect, with the MR being enhanced because of the relatively low resistivity in our high quality grown epitaxial films. This is similar to the MR effect observed in a number of metals at low temperatures.[22] The transverse MR exhibits a change in slope below 1400 Oe, corresponding to

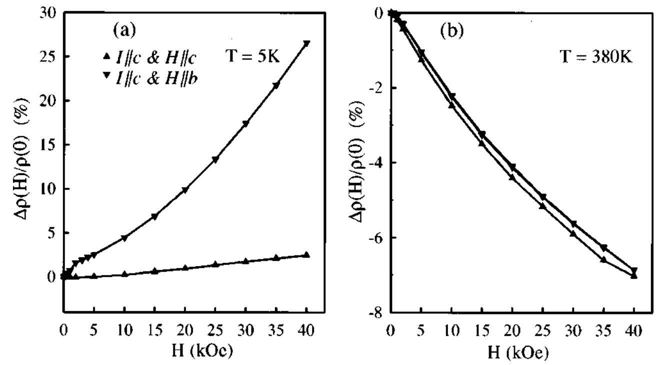

FIG. 5. Longitudinal and transverse MR (0–40 kOe) of a 4000-Å-thick epitaxial CrO_2 film measured at: (a) 5 and (b) 380 K. The measurements are with the current applied along the *c*-axis direction.

the anisotropy field for rotation of the magnetic moment from the *c*-axis to the *b*-axis direction. The magnitude of the MR at a particular field decreases gradually with increasing temperature, switching sign at temperatures above 100 K. The negative MR at high temperatures results from magnetic field suppression of the spin disorder scattering, which reaches a maximum value near T_C.[18,23]

We have further investigated the correlation between the magnetization and the field-induced negative MR for the epitaxial film around the Curie temperature. At 400 K—a temperature slightly above T_C—the low-field MR scales with the square of the ratio of the field-induced magnetization to the saturation magnetization[18]

$$\Delta\rho/\rho=C(M/M_{\mathrm{sat}})^2,$$

with the scaling factor $C=0.437$ in the low magnetization range. Using a simple Born scattering model, Mazumdar and Littlewood have shown that, for a wide range of ferromagnetic metals and doped semiconductors, $C\approx x^{-2/3}$, where x is the number of charge carriers per magnetic unit cell.[24] The manganites are a notable exception to this model. Based on our Hall measurement of 0.3 holes/f.u. for the carrier concentration,[14] the determined value of C for CrO_2 fits the observed trend quite well.

The longitudinal MR ($H\|I$) of the polycrystalline CrO_2 films grown on polycrystalline TiO_2 substrate and TiO_2 seed layer has also been measured. The MR as a function of field (0–40 kOe) for a few different temperatures is shown in Fig. 6 for a film grown on a polycrystalline TiO_2 substrate. Measurements have also been made for a polycrystalline CrO_2 film on TiO_2 seed layer, and are very similar (not shown). Unlike the positive MR observed at low temperatures in the epitaxial film, the MR for the polycrystalline film is negative, with a value of 15% at 4.2 K in a field of 40 kOe. This is consistent with the observation of Hwang and Cheong for polycrystalline films on $SrTiO_3$ substrate.[8]

It is interesting to note that while the total magnetization of the polycrystalline film at 10 kOe approaches 95% of the saturation value, the MR reaches only about 60% of the value obtained at 40 kOe. Moreover, even at 40 kOe, the MR is far from saturation. It thus appears that the high-field MR is not correlated with the magnetization of the grains and is likely related only to the magnetization of the grain boundary

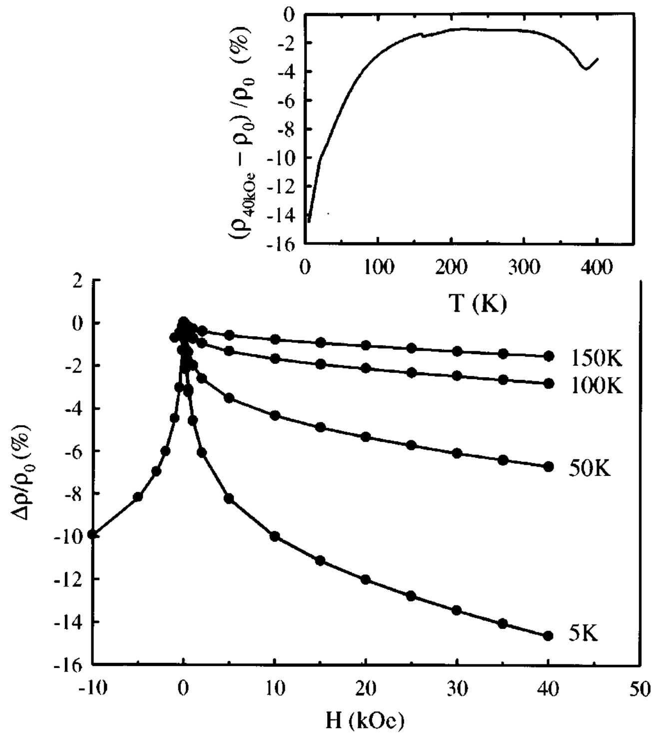

FIG. 6. High-field longitudinal MR at four different temperatures for a CrO_2 film grown on polycrystalline TiO_2 substrate. The temperature dependence of the MR at 40 kOe is shown in the inset. The small dip observed at ~150 K is an artifact of the measurement.

region. As in the case of the manganites, a very large magnetic field is needed to align the disordered spins in this region along the field direction. The MR decreases rapidly with increasing temperature, to only about 2% at 150 K. The inset in Fig. 6 plots the MR in the polycrystalline film at 40 kOe as a function of temperature. The decrease in MR at intermediate temperatures is followed by an increase again near T_C, with a maximum of about −4%. The MR behavior near T_C is similar to the behavior observed in the epitaxial film and appears to be intrinsic to the material.

The low-field MR has also been measured as a function of field (0–1500 Oe), and is shown in Fig. 7. A hysteretic

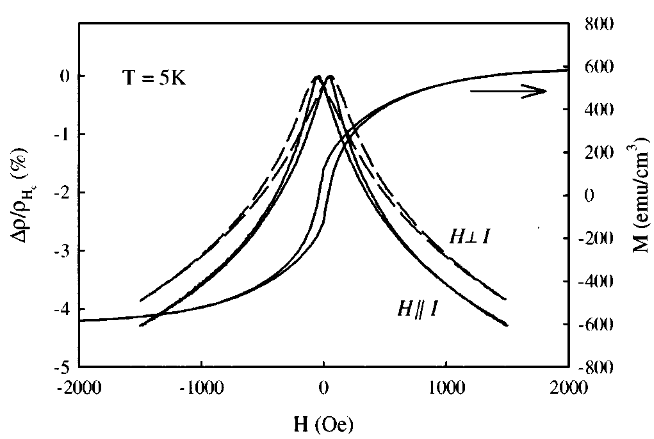

FIG. 7. Low-field MR and the corresponding magnetic hysteresis loop measured for the polycrystalline CrO_2film at 5 K. MR for both the field-parallel and field-perpendicular alignments are plotted.

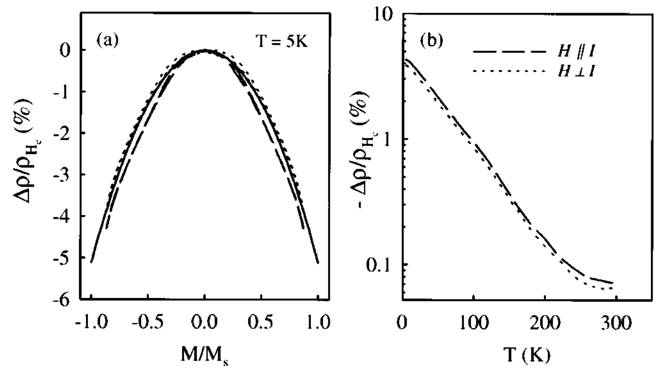

FIG. 8. (a) MR vs magnetization ratio, M/M_s, for the polycrystalline CrO_2 film. The solid curve is the $(M/M_s)^2$ fit to the data. (b) Temperature dependence of the MR measured in a field of 1500 Oe.

behavior is observed for the resistance, with the resistance peaks occurring at the coercive field of the sample. This indicates that the bulk magnetization of the grain is related to the switching behavior, similar to behavior observed in polycrystalline manganite films.[12,13]

In the models that have been proposed for grain-boundary transport, ρ is expected to be a maximum at the coercive field and decreases as the relative orientation of the magnetization between grains changes with the application of a field.[1] the MR hysteresis loops will therefore mirror the global magnetization, M, with the MR being proportional to $(M/M_s)^2$, where M_s is the saturation magnetization.[25–27] The MR at 5 K for the polycrystalline CrO_2 film is plotted against M/M_s in Fig. 8(a) for two different field orientations. The MR data is fit quite well by a $(M/M_s)^2$ dependence, as illustrated by the solid curve in the figure. A similar fit has also been noted for polycrystalline manganite films.[13]

We finally comment on the temperature dependence of the low field MR in polycrystalline CrO_2. Figure 8(b) plots the MR at 1500 Oe as a function of temperature for both the current parallel and perpendicular to the field orientations. The MR decreases almost exponentially with temperature. This behavior is unlike that observed for other spin-polarized oxides, including the manganites, where a much more gradual decrease in MR is observed. The difference in behavior from the other half-metallic oxides is possibly related to the nature of the grain boundary region in CrO_2 and needs to be further investigated.

IV. SUMMARY

In summary, we have studied the magnetic and resistive properties of epitaxial and polycrystalline CrO_2 films grown by CVD. The films have a Curie temperature of 390–395 K, with the ones grown epitaxially on TiO_2 (100) exhibiting a large magnetocrystalline anisotropy. Resistivity measurements show that the epitaxial films have a resistivity drop of about two orders upon cooling from room temperature to 2 K, with some anisotropy in the resistance along the *b*- and *c*-axis directions. In contrast, the change in resistance with temperature for the polycrystalline films is quite small and is dominated by the grain boundary resistance at low temperatures. We have also compared the MR properties of the films. The epitaxial films exhibit a large positive transverse

MR at low temperatures and high field. This can be attributed to the Lorentz force effect that is significant in these films because of their relatively low residual resistivity. The MR decreases with increasing temperature and changes sign, eventually reaching a maximum negative value near T_C, where spin disorder scattering is dominant. A similar negative MR is observed near T_C for the polycrystalline films. However, unlike the epitaxial films, the MR in these films remains negative at low temperatures, with a large low-field component. We attribute the MR at low fields to spin-polarized transport of electrons across grain boundaries.

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