Apparent critical behaviour of sputter-deposited magnetoelectric antiferromagnetic Cr$_2$O$_3$ films near Néel temperature

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Abstract
Chromium(III) oxide is a collinear antiferromagnet with a linear magnetoelectric effect. We are presenting the measurements of the magnetoelectric susceptibility $\alpha$ of a sputter-deposited 500 nm film and a bulk single-crystal substrate of Cr$_2$O$_3$. We investigated the magnetic phase-transition and the critical exponent $\beta$ of the sublattice magnetization near Néel temperature. For the film, an exponent of 0.49(1) was found below 293 K, and changed to 1.06(4) near the Néel temperature of 298 K. For the bulk substrate, the exponent was constant at 0.324(4). We investigated the reversal probability of antiferromagnetic domains during magnetoelectric field cooling. For the sputtered films, reversal probability was zero above 298 K and stabilized only below 293 K. We attribute this behaviour to formation of grains during film growth, which gives different intergrain and intragrain exchange-coupling energies. The reversal probability dependence on the magnitude of cooling magnetic field could be explained by a phenomenological model. For the bulk substrate, reversal probability was stabilized immediately at the Néel temperature of 307.6 K.

Keywords: magnetoelectric, critical exponent, phase transition, chromium oxide

(Some figures may appear in colour only in the online journal)
as predicted by Dzyaloshinskii [1], and confirmed experimentally by Astrov [17]. There are two 180°-related domains \( L^+ \), which are stabilized depending on the relative orientation of magnetic and electric fields during field cooling (MEFC). Brown et al [18] determined that after a +MEFC (−MEFC), the Cr spins are pointing towards (away from) the shared smaller triangle between the CrO6 octahedra (shaded area in figure 1(a)).

The behaviour of the second-order phase-transition of AFMs and ferromagnets are the same, except that the staggered-moment vector (Néel vector) is the order parameter instead of the macroscopic magnetization. The critical exponent \( \beta \) of order-parameter of Cr2O3 films was reported before [19, 20]. Inferences from the exchange-bias effect on a prototypical ferromagnet [19], and the magnetization from uncompensated surface spins [20] were used to determine \( \beta \) at the surface of Cr2O3 near Néel temperature \( T_N \). It was argued that Cr2O3 have the character of 2D surface magnetism near \( T_N \) [19, 20], due to a cross-over from a 3D-bulk behaviour to a 2D-surface one [21, 22].

In this report, we measured the magnetoelectric susceptibility \( \alpha \) during the transition to the paramagnetic (PM) phase near \( T_N \) and used it to determine \( \beta \). In a single layer of Cr2O3 between two electrode layers, we measured \( \beta \) near \( T_N \). We found a cross-over of \( \beta \) and magnitudes different from a bulk-substrate sample. We investigated the relation between the temperature at which \( \beta \) changed in magnitude, and the domain reversal during field cooling. Finally, we analysed the dependence of the domain reversal probability on the magnitude of magnetic cooling field.

2. Experimental details

Two samples were used: a \( 4 \times 4 \times 0.5 \) mm\(^3\) commercial 99.9%-purity Cr2O3 substrate made by the Verneuil process from Furuuchi Chemical Corporation, and a sputter-deposited Cr2O3 film. The presented results are from a single sample for each, and they were qualitatively reproducible for other ones. The structure of the sputtered film was as follows: c-Al2O3 (0001) substrate/Pt (25 nm, 773 K)/Cr2O3 (500 nm, 773 K)/Pt (25 nm, 423 K), where temperature values are the substrate temperatures during deposition. The optimized growth conditions for previous reports were used [10, 23]. The thickness values were determined from calibrated deposition rates of 4.6 and 3.8 nm min\(^{-1}\) for Pt and Cr2O3, respectively. The choice of Cr2O3 thickness was to minimize the leakage current during measurement. The bottom Pt electrode was prepared by sputtering through a slit in a stencil mask, then the mask was removed in air for subsequent deposition. After that, the bottom electrode was heated inside the chamber for 30 min before the deposition of the subsequent layers to remove water and other adsorbates. Cr2O3 was deposited from a 99.99%-pure Cr-metal target by reactive radio-frequency magnetron sputtering, with the oxygen flow controlled by a plasma emission monitor. An orthogonal top Pt electrode was made by photolithography and ion etching. The electric field was applied at the cross-junction and the magnetic field was applied uniformly on the whole substrate. The area of crossing was designed at 8 mm\(^2\) and was measured more accurately by an optical microscope. A schematic of the device geometry and field directions is shown in figure 1(b).
The linear ME ac susceptibility $\alpha = \frac{dM}{dE}$ was measured from the magnetization induced by an ac electric field, using a commercial Quantum Design MPMS-XL SQUID magnetometer (figure 1(c)) [23, 24]. The electric field was applied by an external ac source connected through a sample holder equipped with electrical connections. The induced ac magnetization was detected by a phase-sensitive detection of the flux-locked-loop output using a dedicated lock-in amplifier. However, a part of the detected magnetization comes from the leakage current flowing in the wire loop made by the sample and connections. The leakage current was simultaneously measured by a low-noise transimpedance amplifier for background correction. The simultaneous measurement of the current was used to compensate for the background with a single proportionality constant. This compensation factor was chosen to zero the detected magnetization above $T_N$, and a single value could be used during the span of experiments. This scheme allowed keeping the sample at the center of the gradiometer pick-up coil for weeks, without the need to move the holder for nulling at null-response points [24], and no drift was observed even after two weeks of various experiments. The excitation frequency was determined by searching the spectrum for the least noise while keeping the capacitive current at a minimum, and was fixed at 9 Hz. Peak-to-peak noise was less than $5 \times 10^{-6}$ emu at a zero magnetic field. Sample centering was done at a high field by the substrate’s diamagnetic response.

In the first experiment, the temperature dependence of $\alpha$ was measured after MEFC at a freezing magnetic field $H_{fr}$ and a freezing dc voltage $V_{fr}$ from 315 K to 30 K. The measurement of temperature dependence was under a zero magnetic field, an ac peak-to-peak voltage of 8.5 V, and a 0.5 K min$^{-1}$ heating rate. In a second experiment, we explored the relation between the domain-reversal probability and the transition region of 293–298 K around $T_N$. The effect on reversal probability by the sign of MEFC and at which temperature $T^*$ MEFC is stopped was investigated. Ideally, the average domain state should be measured directly after setting magnetic and electrical fields to zero at $T^*$. However, the amplitude of $\alpha$ is very small near $T_N$. So after MEFC from 315 K to $T^*$, the magnetic and electric fields are set to zero, then the domain state is zero-field-cooled to the peak temperature of $\alpha$. At the peak temperature, $\alpha_{peak}$ was measured for 3 min. A schematic of the procedure is shown in figure 1(d). Same procedures were followed for the Cr$_2$O$_3$ single-crystal substrate, except MEFC was from 330 K and measurement of $\alpha_{peak}$ was at 270 K with a sense voltage of 200 V peak-to-peak. We minimized the temperature under-shoot at $T^*$ to <0.25 K during MEFC from a high temperature. The zeroing of magnetic field was confirmed by a Hall probe positioned at the center of the measurement bore to be <2 Oe. In a third experiment, we investigated the relation between the domain-reversal probability and the amplitude of $H_{fr}$. We interpret the results based on a phenomenological model that will be introduced based on the conclusions of the previous two experiments and insights from the structural properties.

The growth characterization of Cr$_2$O$_3$ over a Pt buffer was reported in more details elsewhere [15, 23, 25], and $c$-axis-oriented epitaxial growth was confirmed. However, Cr$_2$O$_3$ is strained and $30^\circ$-rotated domains are embossed from the Pt buffer. The in-plane strain was quantified at 0.65%, but the out-of-plane strain was unknown due to the overlap of Pt (111) and Cr$_2$O$_3$ (0006) x-ray-diffraction peaks. Transmission electron micrographs of the bottom Pt/Cr$_2$O$_3$ interface show that Cr$_2$O$_3$ exhibits a textured growth with boundary lines that run perpendicular to the interface (figure 2(a)). The starting mode of the growth was investigated by topography scans of the surface of a 1.5 nm Cr$_2$O$_3$ layer grown over Pt using an atomic force microscope (figure 2(b)). The growth is initiated by nucleation sites and small islands that are 60–100 nm in diameter. After the growth progresses, the grains merge and a flat surface results, as seen from the surface of a 500 nm Cr$_2$O$_3$ layer over Pt (figure 2(c)). The mating at the observed grain boundaries should give different properties for inter-grain and intra-grain interactions.

3. Magnetoelastic susceptibility and critical exponent crossover

The temperature dependence of the leakage current (figure 3(a)) showed an increase with increasing temperature, indicating a semiconducting character. Still, the resistance was high (4 MΩ) and a uniform electric field can be assumed along the thickness of the film. The voltage measured by the four-wire method (figure 1(b)) was equal to the applied voltage up to 10 V. Thus, the electric field distribution is uniform across the area of the film.

The wide-range temperature dependence of magnetoelastic susceptibility $\alpha$ after $\pm$MEFC is shown in figure 3(a). The measurements without correction for leakage background and the ones with correction are denoted by a square and a triangle in figure 3(a), respectively. After correction, $\alpha$-T dependence after positive and negative MEFC became symmetric. The maximum peak value of $\alpha$ in the film sample $\alpha_{max}$ of 3.6 ps m$^{-1}$ is the same as the bulk value [24], within errors in sample misalignment and centring. A similar result was recently reported [23], and indicates that the ME properties of the bulk crystals and sputtered films are similar.

The positive (negative) MEFC at +10 kOe and +10 V (−10 V) resulted in the saturation of $\alpha_{peak}$ at $+\alpha_{max}$ (−$\alpha_{max}$), indicating the formation of a single $L^+$ ($L^-$) AFM domain state. MEFC at 0 V and +10 V were identical, showing that the AFM domains in sputter-deposited Cr$_2$O$_3$ films are coupled to the applied magnetic field [13, 23, 26, 27]. The relation and applications of the coupling to magnetic field will be reported elsewhere [28]. The temperature dependence of $|\alpha|$ was identical for $L^+$ and $L^-$ states (figure 3(b)). The average of four temperature sweeps after MEFC at different $H_{fr}$’s and $V_{fr}$’s was used for the subsequent fitting.

The temperature dependence of $\alpha = \chi_\parallel(M_{sub})$ is governed by the volume-averaged sublattice magnetization $\langle M_{sub} \rangle$ and the magnetic susceptibility along the $c$-axis $\chi_\parallel$ [29, 30]. The temperature dependence of $\chi_\parallel$ of Cr$_2$O$_3$ bulk crystal, and AFMs in general, is continuous near $T_N$ with no power-law divergence (lower curve in figure 3(c)) [31]. The smoothness is
The value of $\beta$ determined from $\alpha$ and $\alpha/\chi$ differed by only $<0.005$ for the bulk crystal. Therefore, for the determination of $\beta$ critical exponent we can consider that $\alpha \propto (M_{sub})$.

The sublattice magnetization's critical exponent $\beta$ of the film sample was determined by fitting the magnetoelectric susceptibility $\alpha$ at different temperature ranges near $T_N$ to a power law of the following form:

$$ A \cdot [(1 - T/T_N) \cdot \alpha(1 - T/T_N)]^\beta, $$

where $\alpha(x)$ is the Heaviside step function, which is used to remove the singularity in fitting at or above $T_N$. The fitting
could not be done on the whole temperature range using a single pair of $\beta$ and $T_N$ values. They changed from 0.486(11) and 292.6(2) K at a fitting range of 275–290 K, to 1.061(35) and 297.4(2) K in the range of 290–312 K (figure 3(b)). The change of $\beta$ occurred near 293 K with a global $T_N \approx 298$ K. The adjusted coefficients of determination $R^2$ were 0.990 and 0.984 for each fitting range, respectively. The same qualitative feature of having a higher value of $\beta \approx 0.5$ at 275–290 K was found for various sputtered-film samples, regardless of the top electrode being Pt or Co, the oxygen’s partial pressure, deposition position, and/or dopant material. For the bulk-substrate sample, $\beta$ was constant at 0.324(4) with $T_N$ at 307.6 K. The effect of $T_N$ inhomogeneity on the fitted value of $\beta \approx 0.5$ is minimal. We modelled the effect of $T_N$ inhomogeneity by convolving equation (1) and a Gaussian distribution of $T_N$. The error was <1% for a distribution of a 2 K standard deviation. The higher value of $\beta$ in the sputtered films also correlates well with the shift of the peak temperature of $\alpha$ from 267 K to 236 K for the bulk substrate and the films, respectively. This peak shift of 30 K is more than the 10 K decrease of $T_N$, supporting the explanation of the increase of $\beta$ in the sputtered films. In the following paragraphs, we will give separate discussions to the value of $\beta \approx 0.5$ in the sputtered sample, and the change of $\beta$ from $\approx 0.5$ to $\approx 1.0$. As a reminder to the reader, Ising, XY, and Heisenberg models rely on the sole presence of near-neighbour interactions. In the three-dimensional case, they have $\beta$ values ranging 0.32–0.38.

In a report based on measurements of uncompensated surface magnetization of sputtered films, values of surface $\beta = 0.5$–1.0 were found [20]. They were linked to the surface nature of the boundary magnetization in Cr$_2$O$_3$ [33], based on the assumption that $\beta$ will increase to 0.78(2) at the surface of a 3D-Ising magnetic system [22, 34]. We obtained similar values of $\beta$, but we should emphasize that $\alpha$ is a volume response and the surface contribution is negligible. According to the Ginzburg criterion [35], the true critical exponents are only valid in a range very close to the critical temperature. For a 3D system, that temperature range scales with the characteristic length $L$ as $L^{-\alpha}$. Away from the critical point, the critical exponents may show a mean-field behaviour. The strain is a long-range interaction over a few hundreds of nanometres, meaning that the true exponents are present only within a few millikelvins from $T_N$. Therefore, strained films should show a mean-field behaviour in the experimentally-accessible temperature resolutions [36]. The presented $\beta = 0.49(1)$ is close to the mean-field value. Thus, we think that care should be taken in deducing the relation between $\beta$ and phase-transition classification for grown films of Cr$_2$O$_3$.

In a report based on measurements of exchange bias on a proximate ferromagnetic layer, a cross-over of exchange-bias’ critical exponent from 0.2–0.3 to 0.7–0.8 was found and it was argued to be related to the critical exponent of surface spins [19]. The exchange bias measured on a ferromagnet is a surface phenomenon, so it should infer the surface order-parameter of the AFM. However, due to the low anisotropy of Cr$_2$O$_3$, the exchange biases change into an enhancement of coercivity at a temperature lower than $T_N$. The results of [19] seem to be near the blocking temperature of exchange bias, not $T_N$. On the other hand, we have an alternative explanation to the change of $\beta$ near $T_N$ from 0.49 to 1.06 in our results. We should consider the in-plane random placement of 30°-rotated grains during sputter growth [15, 23, 25]. Two possible effects can result. The first is an inhomogeneous distribution of $T_N$ across the film area with a normal distribution spanning 293–298 K range. The superposition of the power-law contributions with different $T_N$’s can give a tail above the average $T_N$ and an apparent cross-over of $\beta$ [37]. The second possibility is lower exchange energies at grain boundaries compared to the inside of grains, with the corresponding equivalent $T_N$’s of $\approx 293$ K and $298$ K, respectively. Then, the effective domain volume will suddenly decrease above $\approx 293$ K to the size of grains, the thermal energy will overcome the anisotropy energy, and the fluctuations of AFM domains become dominant (i.e. superparamagnet-like). Within the finite-sized clusters, the order parameter is highly correlated, but the ensemble’s order parameter is fluctuating in a superparamagnetic manner [38]. The phase-transition should resemble a percolation process between magnetic particles in a paramagnetic matrix. The critical exponent of the order parameter of the percolation process equals exactly 1.0 in the case of a very large number of dimensions (i.e. mean-field-like) [39]. Determining the more likely mechanism is discussed in the next section by investigating the field-cooling process.

### 4. Domain reversal probability during field-cooling

In this section, we present the effects of field-cooling conditions at $T_N$ on the AFM domains state at low temperatures. In the following experiment, we measured the effect of $T^*$, the temperature at which the field-cooling is stopped, on domains population in both of the film and bulk samples (figure 4). Then, we describe the field-cooling process based on a phenomenological model, and compare it to the experimental dependence of domain reversal on $H_f$ (figure 5).

In the first experiment, the average domain state ($L$) of a whole sample was estimated by normalizing the measured $\alpha_{peak}$ as in the following equation:

$$\langle L \rangle = \frac{\alpha_{peak}}{\alpha_{max}} \frac{\nu^+ - \nu^-}{\nu^+ + \nu^-},$$

where $\nu^\pm$ are the volumes of $L^\pm$ domains. Using the sputtered-film sample, MEFC was under $H_f = +10$ kOe and $V_f = 0$, $\pm 10$V. At $T^* > 298$ K, $\langle L(T = 240 K) \rangle$ was zero, as would be expected from a zero-field cooling. At $T^* < 293$ K, $\langle L \rangle$ was stable at $\pm 1$ as expected from $\pm$MEFC. The domain state after $+MEFC$ showed saturation at $T^* < 295$ K (blue diamonds of figure 4(a)). MEFC at $V_f = 0$ V (i.e. cooling with magnetic field only) showed the same behaviour as $+10$V (black squares of figure 4(a)). We expected that if the inhomogeneity in $T_N$ was the reason for the tail above 293 K, then $\langle L \rangle - T^*$ should be with a width of 293–298 K, not 295–298 K as we found. The switching after $-MEFC$ showed saturation at $T^* < 293$ K (red circles), with an anomalous positive pump at 296 K. If we consider that during MEFC at $H_f = +10$ kOe and $V_f = -10$ V there are two regions in the film: one is the
cross-junction area under a MEFC(+10 kOe, −10 V) preferring $L^-$ domains, and the other is the surrounding region with MEFC(+10 kOe, 0 V) preferring $L^+$ domains. Thus, when magnetic and electric fields are being removed at $T = 294–298$, $L^+$ domains can propagate inside the capacitor area giving a higher population of them. $L^-$ domains are stabilized by an increase of volume-anisotropy product below 293 K. When a negative weak field $H_f < H_T$ of $-20$ Oe was applied below $T$, the signs of an uncompensated magnetization and $\alpha$ change upon cooling in positive and negative $H_T$ [23]. To determine the possibility of switching after field cooling, $L$ can be found either from $M$ or $\alpha$ magnitudes at any specific temperature, when normalized by their maximum values at that temperature. The dominance of either domain is determined by the energy affecting the AFM at $T_N$ through the Zeeman energy of the uncompensated magnetization. The free energy per unit volume in CGS units is:

$$W = -M H_f \cos \theta$$

$$\equiv W_0 \cos \theta,$$

(3)

where $M$ is the uncompensated magnetization normalized to the volume of the sample, and $\theta$ is the angle of $M$ with the direction of $H_f$ applied along $c$-axis.

The model of field cooling process is based on the observation that domains are stabilized within 5 kelvins below $T_N$ by an anisotropy barrier. Just below $T_N$, we assume that a short-range order appears within a single-domain volume $V$ where all spins are aligned. This implies that the values of $M$ used in calculating $W_0$ are the low-temperature limits retrieved from magnetometry measurements. Furthermore, we assume that films are composed of a non-interacting ensemble of particles down to 5 K below $T_N$, then a Boltzmann distribution can be used to represent the normalized probability distribution of $\theta$:

$$P_0(\theta, V) \propto \sin \theta \exp\left(\frac{-W_0 V \cos \theta}{k_B T_N}\right),$$

(4)

where $k_B$ is the Boltzmann constant, and $T_N$ is taken to be 300 K. As the temperature is lowered to a measurement

Figure 4. The dependence of switched domain population on $T^*$ in (a) the film sample, and (b) the bulk substrate. The error bars in ($L$) denote the standard deviation of averaged measurements, and for temperature denote the undershoot during cooling towards $T^*$. Solid lines are eye-guides.
temperature $\ll T_N$, we can assume that any domain falling in the range $0 < \theta < \pi/2$ ($\pi/2 < \theta < \pi$) is pushed towards $L^-$ ($L^+$). Then, the value of $\langle L \rangle$ can be found as the expectation value of $g(\theta) = \text{sgn}(\theta - \pi/2)$:

$$\langle L \rangle = \int_0^\pi gP_\theta d\theta = \tanh \left( \frac{-W_0 V}{2k_B T_N} \right) \equiv \tanh(H_0/\lambda_H),$$

(5)

where $\lambda_H = 2k_B T_N/(M V_n)$, which can be used to estimate $V_n$ from a field cooling experiment. The actual $V_n$ near $T_N$ cannot be verified by any other direct experimental method. Therefore, the simplified image of uncorrelated particles in the narrow window near $T_N$ was used instead of accounting for a weak correlation. The fitted values of $V_n$ should be considered as indicative.

For the effect of grain-size distribution on switching probability, the probability distribution of $V$ can be considered as having a log-normal distribution of:

$$P_V(v) = \frac{1}{v_0\sqrt{2\pi} \sigma_V} \exp \left[ -\left( \frac{\ln(v/v_0)}{\sigma_V \sqrt{2}} \right)^2 \right],$$

(6)

where $V_0$ is the median volume, and $\sigma_V$ is the scaling parameter. The effect of the value of $\sigma_V$ on $\langle L \rangle$ was found from a numerical solution to:

$$\langle L \rangle = \int_0^{\infty} \int_0^\pi gP_\theta P_V d\theta dv.$$

(7)

Even at $\sigma_V = 0.5$, the maximum error between equations (5) and (7) was less than 4%. This small difference is because that for any grain with $V > V_0$ that has a steep switching probability, there would be a grain with $V < V_0$ that has a gradual switching probability, and the average would be the response of the median size. The experimental results of $\langle L \rangle - H_F$ dependence with fittings to equation (5) for three thicknesses are shown in figure 5. The experimental values of $M$ measured for each sample were used, which were $\approx0.2$ emu cm$^{-3}$. The hyperbolic tangent function shows a good qualitative description of $\langle L \rangle - H_F$ dependence. The median short-range-order volume is estimated at $100^3$ nm$^3$ independent of the thickness (inset of figure 5). This value of $V_n$ is consistent with the area estimated from the topography scans of figure 2(b). Thus, the picture of non-interacting, or weakly-interacting, single-domain grains just below $T_N$ is more likely. Finally, we would like to note that in a recent report by Kosub et al [13], a similar size of twinning grains were found for Cr$_2$O$_3$ grown over noble-metal buffers. According to the data shown by their supplementary data, the average grain area can be estimated at $130^2–150^2$ nm$^2$, which are comparable to our estimations.

5. Conclusions

We measured the temperature dependence of magnetoelectric susceptibility in a 500 nm Cr$_2$O$_3$ film and a bulk substrate. We found a crossover of the critical exponent $\beta$ near $T_N$ of the Cr$_2$O$_3$ film from 0.49 to 1.06, 5 K below Néel temperature. No such change was found in the bulk substrate. The magnetoelectric susceptibility is a volume measurement and cannot detect the surface component at the used thickness. Therefore, an explanation based on a change of dimensionality does not apply. Two possible explanations were considered. The more likely one is that the film has a $\beta$ value close to 0.5 due to strain resulting in a mean-field-like behaviour. Additionally, we found that probably a phase-transition occurs within 5 K below the global Néel temperature from a long-range-ordered AFM to a thermally-fluctuating short-range AFM, before becoming a paramagnet above Néel temperature.

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