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X-ray Magnetic Dichroism and Giant Magneto-Resistance study of the magnetic phase in epitaxial CrVO_x nanoclusters

Loïc Joly^{1,2}, Fabrice Scheurer^{1,2}, Philippe Ohresser², Brice Kengni-Zanguim¹, Jean-François Dayen¹, Pierre Seneor³, Bruno Dlubak³, Florian Godel³ and David Halley^{1*}

1-Université de Strasbourg, CNRS, Institut de Physique et Chimie des Matériaux de Strasbourg, UMR 7504, F-67000 Strasbourg, France

2- Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP 48, F-91192 Gif-sur-Yvette, France

3- Unité Mixte de Physique CNRS Thales, Université Paris-Saclay Palaiseau, France

Abstract

Epitaxial clusters of chromium and chromium-vanadium oxides are studied by giant magneto-resistivity measurements, X-ray absorption spectrometry and circular magnetic circular dichroism. They turn out to carry a small magnetic moment that follows a superparamagnetic behavior. The chromium ion contribution to this magnetization is mainly due to an original magnetic Cr_2O_3 -like phase, whereas usual Cr_2O_3 is known to be antiferromagnetic in the bulk. For mixed clusters, vanadium ions also contribute to the total magnetization and they are coupled to the chromium ion spins. By measuring the dichroic signal at different temperatures we get insight into the possible spin configurations of vanadium and chromium ions: we propose that the magnetic dipoles observed in the clusters assembly could be related to ionic spins that couple at a very short range, as for instance in short one-dimensional spins chains.

Text

Chromium and vanadium oxides attract a large interest due to their multifunctional properties: for instance, vanadium oxides VO₂ and V₂O₃ are correlated systems exhibiting a metal-insulator transition [MOR59, AET13, SCH04, McLE17], whereas Cr_2O_3 is an archetypal magneto-electric material [AST60, MOST10, HEX11]. Nevertheless the net magnetization in those materials – in the absence of applied electric field- is equal to zero since they are bulk antiferromagnets; this prevents their use for magnetic and spintronic applications. We recently demonstrated that Cr_2O_3 [HALL14, 15, 16, 17, 19] and VO_x

[HALL20] physically grown as strained epitaxial clusters in a MgO matrix develop interesting magnetic properties with the presence of a free permanent magnetic moment in the clusters and subsequent magneto-resistive effects.

The origin of the magnetization in these oxides was attributed either to spin canting relative to a perfect anti-ferromagnetic ordering or to non-compensation of the spins on different non-equivalent ionic sites [HALL16]: for instance, in the case of CrO_x clusters, the large epitaxial strain could lead to inequivalent Cr^{3+} sites between which the magnetic dipoles would not perfectly compensate, *i.e.* to a ferrimagnetic order or to a canted spin configuration. Slight changes in the spin on the different sublattices, induced by electric field, might then explain the huge magneto-electric effect that we observed in those systems [HALL14]. In order to unveil the spin configuration of adjacent Cr^{3+} ions and the subsequent magneto-electric mechanism, it is therefore appealing to introduce other 3dmagnetic ions, as vanadium ions, which may modify the imbalance between neighbor ionic sites. Furthermore, vanadium oxide doped with 10 to 20 percent chromium [WEST08, PIP10] was shown by West et al. to be ferromagnetic, even at room temperature, which raises the question of the spin coupling between chromium and vanadium ions in CrVO_x oxides [MAR72, RUPP18]. For these reasons, we here study oxidized alloyed CrV clusters that are achieved in the same way as in our previous reports [Hall 14, ZANG 20], the oxidation being favored by the proximity with MgO layers. Through magnetoresistivity measurements and X-Ray Magnetic Circular Dichroic measurements (XMCD), we address the question of the magnetization carried by those nanometric epitaxial CrV oxides clusters.

The samples were grown on MgO [001] substrates by *e*-beam molecular epitaxy in a 10^{-10} torr base pressure chamber: A 20 nm MgO layer was deposited at 500°C followed by chromium and vanadium at 200°C which formed epitaxial CrV clusters [KOD11, ROM06], as observed by reflection high energy electron diffraction with a (001)[100]MgO//(001)[110]CrV epitaxial relationship. A subsequent 3 nm MgO layer was deposited at 200°C, under an oxygen partial pressure in the high 10^{-8} torr range contributing to the metal oxidation. The stack was then annealed at 500°C to favor the complete oxidation of CrV clusters, with the diffusion of oxygen ions from MgO to the clusters. We focus here on three samples: sample A, used as a reference without vanadium (the nominal thickness of deposited chromium is 0.3 nm); sample B, an alloy of 66% Cr and 33% V (the nominal thickness of deposited chromium is 0.3 nm, and 0.15 nm for vanadium); sample C, an alloy of 50% Cr and 50% V (the nominal thickness of deposited chromium is 0.3 nm, and 0.3 nm for vanadium). Magneto-transport measurements were done on a sample similar to sample C, but grown on top of a bottom electrode made of 10 nm epitaxial Fe on the 20 nm MgO layer.

Atomic Force Microscopy (AFM) pictures (Fig. 1 (a) and (b)) confirm what was already reported concerning CrO_x and VO_x samples, *i.e.* a dewetting phenomenon during deposition of the metal (Cr or V) into islands deposited on top of MgO. In the case of pure Cr islands (sample A), the density of islands is close to 150 per μ m² and the average cluster height equal to 2-3 nm. In the presence of vanadium (sample B), the density of islands is slightly lower (90 per μ m²) and the typical height of the islands increases to 5-6 nm.

Sample C (on a Fe bottom electrode) was processed into vertical magnetic tunnel junctions through the nano-indentation technique [BOU 03]: a resist, deposited on the surface of the MgO tunnel barrier, is locally indented using an AFM tip, and exposed to an oxygen plasma opening, thus creating a 100 nm nano-hole in the resist. The metallization of the surface of the sample with Ti and Au ends the nano-contact for electron injection. The size of this top electrode should correspond to less than ten buried oxide clusters if we refer to AFM pictures (Fig.1(b)). The resistance R of the vertical stack was measured as a function of an in-plane applied magnetic field *H* along the [100] easy axis of the Fe film - which is along the [110] direction of MgO since Fe obeys a (001)[100]MgO//(001)[110] Fe epitaxial relationship-. The R(H) curve, though noisy, shows a giant magneto-resistance (GMR) effect at 10K (Fig. 1(d)) reaching up to 2% under a 7 T magnetic field: electrons tunnel through the MgO double tunnel barrier via the clusters into the Fe magnetic bottom electrode. This GMR effect is thus a probe of the magnetization of the CrVO_x clusters, since the Fe magnetization remains along the magnetic field for *H* values above the Fe magnetic coercive field (about 5mT [HALL14]). The continuous change of resistance and the absence of discontinuities in the R(H) curve indicate that we do not observe individual cluster magnetization reversal. This GMR curve proves that the oxide clusters carry a non-zero magnetization as in the case of CrO_x and VO_x clusters and behave as in superparamagnetic state [HALL14, ZANG20].

X-Ray Absorption Spectroscopy (XAS) measurements were performed at the SOLEIL synchrotron on the DEIMOS beamline [OHRE14], at the V and Cr L_{2,3} edges. On the three samples, XAS spectra at the Cr edge (Fig. 2) can be fitted [HALL14] to the sum of reference experimental curves related to Cr^{4+} (CrO₂-like signal) and Cr^{3+} . This last contribution comes from a corundum α -Cr₂O₃ (anti-ferromagnetic) phase [GAU06] but also from a magnetic Cr₂O₃-like phase in which Cr³⁺ ions are close to the hexagonal

environment of Cr^{3+} diluted in ruby α -Al₂O₃ [GAU06]. As no additional Cr metal contribution is required in the fit, we deduce that chromium is fully oxidized. Concerning vanadium ions, the reference XAS spectra of the metallic state [HUT03] are too close to spectra related to ionic states to discriminate between metal and oxide.

We furthermore observe a clear XMCD signal at the chromium and vanadium $L_{2,3}$ edges (see Fig. 2 and 3) confirming that we are not dealing with perfect antiferromagnetic oxides, but with magnetic clusters, exhibiting magnetization at 6.5 T. In order to fit our experimental XMCD observations at the Cr edge to reference experimental spectra we distinguish between the magnetic contributions of Cr^{4+} (CrO₂ like phase) and of Cr^{3+} (magnetic Cr_2O_3 -like phase). The α - Cr_2O_3 phase being nonmagnetic, this phase does not contribute to the XMCD signal. Fitting simultaneously XAS and XMCD spectra at the Cr $L_{2,3}$ edges (see Table I) yields an estimate of the ionic species composition as already done in Ref. [Hall14]. The obtained fitting parameters (Table I) are fully consistent with our previous report [Hall14] of a dominating magnetic contribution from a Cr₂O₃ -like phase (about 70 % of the magnetic signal in the present study and 80% in Ref. [Hall14]) with a smaller contribution due to a CrO₂-like phase [GOE02]. We should remember that CrO_2 is ferromagnetic in the bulk [GOE02]: in our case, it is likely that this magnetic oxygen-rich phase locates at the cluster boundaries, closer to the MgO interface. The presence of vanadium in sample B and C does not noticeably modify these features: the Cr^{3+} magnetic contribution remains close to 70% in presence of vanadium.

Concerning vanadium ions, XAS curves (Fig. 3 (a)) do not enable us to determine their configuration in the oxide: as underlined in Ref [HAl 20] several V_nO_{2n-1} Magneli phases can indeed be observed in vanadium oxides [SCH04] with slight variation of the expected XAS spectra. Furthermore, overlapping L₂ and L₃ edges [THOL92, ZANG20] make any quantitative analysis very difficult. We nevertheless observe a non-zero XMCD signal at the V edge (Fig. 3 (b)) which clearly proves that the vanadium ion assembly also contributes to the magnetic signal.

The moderate influence of temperature on the magnetization of chromium and vanadium ions is illustrated in Figs. 4 (a) and (b): on the different XMCD spectra recorded at the Cr and V $L_{2,3}$ edges on sample B, the XMCD signal only slowly decreases when T increases, proving a relatively small influence of temperature on the magnetization carried by the chromium and vanadium ions assembly. This is not in

contradiction with the superparamagnetic behavior since all these measurements were performed under a high magnetic field (6.5 T) close to saturation of the magnetization.

It would be tempting to apply sum rules [CHEN95] to XMCD spectra in order to extract the spin and orbital magnetic contributions of the different ions. Unfortunately, it is not possible to give values of these moments with real signification since Cr appears in three different structural phases, and two magnetic phases, possibly with different spin and orbital moments. The problem is the same in the case of vanadium, with possibly different phases in the oxide. Moreover, as stated above, vanadium has strongly overlapping L_2 and L_3 edges preventing from sum rules application [THOL92].

Nevertheless, in the case of chromium, we can rely on the relative maximum intensities of XMCD signals of CrO_2 and magnetic Cr_2O_3 phases (this ratio is close to 1:4 in Figs. 2 (b) and 2 (d)) to obtain an order of magnitude of the moment carried by chromium in the magnetic Cr_2O_3 phase. Indeed, as evaluated above (Table I) the ratio of involved ions in the two magnetic phases is close to 2.5:1. Since chromium ions in the ferromagnetic phase of CrO_2 carry a moment of about 1.2 µB [GOE2] a rough proportional evaluation would yield about 1.9 µB for chromium ions in the Cr_2O_3 magnetic phase, if we suppose that the maximum XMCD signal varies proportionally to the ionic magnetic moment. We insist that this is only an order of magnitude, but this will be useful below to rule out the hypothesis of very low magnetic moments in the magnetic Cr_2O_3 phase.

The XMCD signal that we exploited above is obtained at high magnetic field (6.5 T) for which the magnetization is close to saturation (Fig.4). In order to get information concerning the collective magnetic behavior of the system at lower magnetic field, we recorded the dichroic signal at its maximum for the V and Cr edges as a function of magnetic field between +6.5T and -6.5T. The curves (see Fig. 4 (c)) are S-shaped, without evidence of hysteresis, and consistent with the shape of GMR curves shown in Fig 1 (d). Theses curves can be successfully fitted to Langevin functions ($\mathcal{L}(x)=1/tanh(x)-1/x$) with $x = m\mu_0H/k_BT$ where *m* is the "free" magnetic moment of a superparamagnetic assembly, which results from the collective coupling of ionic spins in the clusters. We focus on the Cr signal, which can be observed on both A and B samples: From the fits we can extract the *m* value, for instance at 10K, *m*=3.9µB (sample A) or *m*=11µB (sample B). These values, though relatively small, exceed a single atom contribution. We have shown above through a rough evaluation that the magnetic moment carried by chromium ions in CrO₂

and magnetic Cr_2O_3 is in the range of one Bohr magneton. The free magnetic moment leading to the super-paramagnetic behavior should therefore involve only a very few atoms (less than ten). This result clearly rules out the possibility that a free magnetic moment could correspond to the magnetic dipole of a whole cluster. Indeed, $CrVO_x$ clusters, whose size is determined from Fig. 1, contain at least thousands of atoms. This clearly proves that the spin coupling takes place at a very short range, here in the order of ten ions or less. This behavior could for instance be consistent, in the Cr_2O_3 magnetic phase, with spin canting along one-dimensional ion chains, which are well-known in the case of V_nO_{2n-1} Magneli bulk oxides [SCH04, L116,Hall20].

From the experimental hysteresis curves, we can furthermore draw different conclusions concerning the spin configuration:

- When comparing samples A, B and C, we observe that the doping of CrO_x with vanadium does not drastically modify the Cr-related magnetic signal. Either vanadium and chromium are not homogeneously alloyed and form, for instance, a core-shell structure before oxidation, or the effect of substitution of vanadium in the magnetic configuration of the chromium ion spins is limited. This last case would make the hypothesis of a ferrimagnetic-like magnetic behavior very unlikely.
- In sample B, at different temperatures, *m*, the average free dipole values extracted from Langevin fits at the V and Cr edges are very close (Fig. 4 (d)) to each other. This is also illustrated in Fig. 4 (c) where the normalized hysteresis curves at the chromium and vanadium edges almost superpose. This result proves that a magnetic coupling takes place between V and Cr magnetic atomic spins which all contribute to a larger "free dipole" and follow the same behavior as a function of the applied magnetic field. Taking into consideration the fact that the "free dipole" corresponds to a few atoms as demonstrated above, this result rules out the hypothesis of non-alloyed chromium and vanadium ions.
- In both A and B samples, the extracted *m* values (at the Cr edge, and at the V edge in the case of sample B) seem to be proportional to the temperature (Fig. 4 (d)). We already observed this phenomenon in the case of pure chromium oxide clusters [Hall nanotech] through magneto-resistive measurements: the magnetic free dipole was indeed shown to vary linearly with T and could be extrapolated to zero at T=0.

This last point seems contradictory with the relatively low change of the total magnetization of Cr and V ions with T that was observed above on XMCD spectra (Fig. 4 (a) and (b)): if the magnetization tended to zero as T tends to zero, we should observe a very low XMCD signal as T decreases which is not the case.

We will consider different hypotheses that could tentatively explain this linear decrease of m with T:

- A nearly zero magnetization close to T=0 and a dramatic increase of the magnetization with T was for instance reported in [ALIA01] in Co/Cu granular systems: in such systems the mutual magnetic interaction between magnetic dipoles was shown to strongly modify the total magnetization by favoring an antiferromagnetic order between magnetic free dipoles; this therefore leads to a zero-magnetization of the cluster assembly at very low temperature. Nevertheless, if the magnetic interaction between dipoles induced in our case a decrease of the total magnetization at low temperature, the XMCD signal should also tend to zero, which is not observed here.
- A freezing of the largest cluster magnetization at low temperature (due for instance to a non-negligible magnetic anisotropy) could lead to an apparent decrease of the *m* value, since only the smallest clusters, that are not blocked, would contribute to the Langevin curves at low temperature. This should nevertheless cause hysteretic behaviors in M(H) curves, that are not observed, and this should also make that the total XMCD signal vanishes at low temperature, contrary to what we observe.
- We wonder whether the temperature could influence the size of the "free dipole" involved in the super-paramagnetic behavior, for instance the length of the hypothetical coupled-spins chains in the oxide. By analogy with VO_x Magneli phases [SCH04] in which chains of anti-ferromagnetically coupled spins are observed, whose length depends on the *x* value, we could suppose that the free magnetic dipole that are evaluated through Langevin curve fits could correspond here to temperature-dependent canted spin chains. If increasing the temperature increased the number of spins included and mutually coupled in a given chain, we could explain the surprising increase of *m* (T) with temperature.

This last scenario remains highly speculative, but would be, as far as we know, the only way to conciliate the observed decrease to zero of m(T) when T tends to zero with

the remaining high XMCD signal at low temperature: indeed m would decrease to zero as T tends to zero, but the total magnetization of Cr or V ions under a sufficiently high magnetic field (6.5 T) would remain constant since all spins would align along H, whatever the temperature.

In conclusion, strained epitaxial clusters of metallic chromium-vanadium alloys, oxidized during and after deposition, and embedded in MgO, exhibit a net magnetization at low temperature at least up to 20K. Spins of chromium and vanadium ions contribute to the magnetization and are coupled inside "free dipoles" of a few Bohr magnetons, which follow a superparamagnetic-like behavior. Compared to similar chromium oxide clusters, the influence of vanadium insertion appears limited: neither drastic changes of the Cr oxidation state, nor strong modifications of the cluster size, or increase in the "free dipole" values are observed which rules out the hypothesis of a ferri-magnetic system with non-equivalent ionic sites. We moreover showed that the observed free magnetic dipole, but are carried by a few coupled ionic spins.

The non-zero magnetization in these clusters is appealing since it could make possible the development of devices combining the well-known metal-insulator properties of these oxides with spintronics applications. The origin of the magnetic "free dipoles", is nevertheless still unclear, but our observations, especially as a function of temperature, suggest that it could correspond to the coupling of non-compensated ionic spins - for instance canted- on a length that is temperature-dependent. This hypothesis has to be confirmed by further experimentation, but seems all the more interesting as it could tentatively explain the huge magneto-electric effect [HALL14,15] observed in similar chromium-based oxide clusters, if the spin-coupling length were also electric-field dependent.

Figures:

Fig. 1:

AFM pictures of respectively (a) CrO_x and (b) $CrVO_x$ clusters that are not capped with MgO. Height profiles along the red lines are given in inset. (c) Scheme of the device devoted to magneto-transport measurements, which includes a Fe bottom electrode. The magnetizations of this Fe layer and of the oxide clusters are shown as green arrows. (d) GMR curve at 10K under an in-plane applied magnetic field and for a 400mV applied bias voltage. Red curve, as a guide to the eyes: magneto-resistance curve following a Langevin function, taken for a free magnetic dipole equal to 5μ B at 10K. In inset: I(V) curve measured on the same device at 10K.



Fig. 2:

XAS experimental spectra at the chromium $L_{2,3}$ edge for (a) sample A (4K) and (c) sample B (10K). (b) and (d) corresponding XMCD experimental spectra for the same samples, in percent of the XAS signal. The estimated contributions of the CrO₂-like phase, the α -Cr₂O₃ phase and the magnetic Cr₂O₃ phase are given, as well as the resulting total fit. Note that the XMCD curves have been normalized to the edge jump of the polarization-averaged total XAS. The incident X-Ray beam, parallel to the magnetic field, is 60° off-normal.



(a) XAS and (b) XMCD experimental spectra at the vanadium $L_{2,3}$ edge for sample B $(Cr_{66}V_{33}O_x)$ measured at 10K.



Influence of temperature on the cluster magnetic properties. (a) XMCD spectra at the chromium $L_{2,3}$ edge for sample B ($Cr_{66}V_{33}O_x$) at T= 4.2K, 10K and 20K. (b) XMCD spectra at the vanadium $L_{2,3}$ edge for sample B ($Cr_{66}V_{33}O_x$) at T= 4.2K and 10K. (c) Hysteresis curve: Intensity of the maximum XMCD signal measured on sample B as a function of the 60° off normal magnetic field at 4.2K at the chromium edge (blue curve), at the vanadium edge (green curve) and Langevin fit of the hysteresis measured at the chromium edge (red line). (d) "free magnetic dipole" *m* obtained from fits of the hysteresis curves at the Cr and V edges at different temperatures.



Table I

Estimation of the repartition of chromium ions into the three crystalline phases in the clusters, obtained from simultaneous fits of the XAS and XMCD data on samples A, B and C. The weight of the magnetic Cr_2O_3 -like phase among the two magnetic phases is given in parenthesis.

	CrO ₂ -like phase	Non-magnetic α -Cr ₂ O ₃ phase	Magnetic Cr ₂ O ₃ -like phase
Α	10	63	27 (72%)
В	6	80	13 (68%)
С	4	85	11 (73%)

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