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To cite this article: R H Rodriguez *et al* 2014 *J. Phys.: Conf. Ser.* **568** 042031

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240th ECS Meeting ORLANDO, FL

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Multiple First Order Transitions in Cr Thin Films

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Abstract. The incommensurate spin density wave (SDW) in metallic Cr forms below $T_N = 311$ K and is characterized by a temperature dependent wave-vector \mathbf{Q} along the $\langle 100 \rangle$ direction. The wavelength varies from 78 Å at 300 K to 60 Å at 150 K. In thin films, the \mathbf{Q} -vector orients perpendicular to the film surface and the SDW is forced to satisfy boundary conditions. As a consequence of the confinement, the SDW wavelength is quantized in a set of available modes, each one having n nodes. As the temperature decreases the wavelength does not follow a continuous bulk-type variation but jumps between different allowed values. These jumps occur at certain determined temperatures T_n as first order type transitions which are accompanied with a decrease (n to $n + 1$ nodes) or an increase ($n + 1$ to n nodes) of the resistance. In this work, we present transport measurements on Cr thin films showing hysteretic behavior in the resistance as a function of temperature in cooling-warming cycles. The hysteresis loops are thickness dependent and present structures which we associate with the possible multiple transitions that may occur as temperature is swept.

1. Introduction

In recent experiments attention was drawn back to the antiferromagnetic ground state of Cr system [1, 2]. The study of the spin density wave (SDW) was very important in the understanding of giant magnetoresistance and spin dependent transport (spintronics) in CrFe superlattices [3, 4, 5, 6] and the SDW ground state in Cr films [7, 8] became an active research area with experimental and theoretical studies involving different magnetic materials [9, 10, 11]. It was realized that confined geometries could induce non-trivial modifications in the magnetic and transport properties of these multilayered systems [12, 13]. Kummamuru's recent work [1] reopened the interest in this system showing how transport properties are modified by the antiferromagnetic state of Cr when the SDW is confined in very thin films. As further explored in other works [2], confinement leads to quantization of the wavelength and to the formation of antiferromagnetic domains. Transport properties are strongly influenced by the presence of these domains and their evolution with temperature, and since antiferromagnetic spintronics is a well considered candidate for future applications [14, 15], different ideas about the control and manipulation of these domains and domain-walls are now being rehearsed [16]. In Cr thin films domain structures emerge in a very natural way and it is possible to design basic experiments in several directions: *i*) it is clear that the domain structure depends on the films roughness, but how much this affects transport properties has not yet been addressed. *ii*) The average



dimension and nature of domains is still an open question and also *iii*) it is interesting to study how sensitive are transport properties to individual domain nucleation, growing or switching.

In this work we present experimental results on transport properties in thin (~ 400 Å) Cr films grown by molecular beam epitaxy (MBE). In thin films, the Q-vector of the SDW has been found to be perpendicular to the films surface in the whole range of studied temperatures (from 330 K to 30 K) [7, 10] and it has been observed that the strong boundary conditions emerging from the confinement within the film thickness produce a thickness-dependent hysteretic behavior of the transport properties [2]. In previous reports epitaxial thin films grown by sputtering have been investigated. In those cases, the observed phenomena are governed by the confinement of the SDW within the film thickness L with an uncertainty given mainly by the mean roughness ~ 20 Å. The results of the present article may give some answers to the three points stated above. In first place, our films grown by MBE have a mean roughness of ~ 5 Å and the effect of the roughness reduction can be directly analyzed. As shown in Fig. 1A), our experimental setup allows us to measure simultaneously different parts of the sample (with different dimensions) and thus to gain some insight into the size (and number) of domains, although this can be inferred only roughly. Finally, the hysteresis in resistance is an evidence of the motion of domains walls, i.e. growing or shrinking of domains and for that reason, a detailed study of its structure gives some information of the sensitivity to individual processes.

2. Experimental Method

Cr thin films were grown on MgO $\langle 100 \rangle$ substrates by molecular beam epitaxy following a similar procedure as reported for sputtering deposition. In the latter, the resulting films showed a very good degree of epitaxy [1, 2]. We characterized our films with X-ray diffraction and as shown in Fig. 1B), the spectrum shows a well defined peak at $2\theta = 64.6^\circ$ corresponding to the $\langle 002 \rangle$ Cr characteristic peak and an angular dispersion of 0.5° (FWHM from rocking curve). The other peaks correspond to the substrate. From small angle reflectometry, shown in Fig. 1C), we obtained the thickness of the sample $L = 370$ Å. The films surface were characterized by AFM (see Fig. 1D)) and the mean roughness found was ~ 5 Å. This is approximately four times better than what was found in epitaxial films grown by sputtering (~ 20 Å).

In order to perform transport measurements we patterned the films by means of optical lithography and chemical etching in a hallbar-like configuration with several terminals (see Fig. 1A)). We contacted the samples with 25 microns gold wires glued with Epo-tek epoxy to different pads being thus able to measure four regions of the hallbar (four independent resistances) simultaneously (i.e. during the same cooling-warming cycle). Samples were thermally anchored to the cold plate of a commercial close cycle cryocooler and temperature cycles were performed from 330 K to 20 K with a step of 1 K and a temperature stabilization protocol that assures fluctuation of the temperature lower than 20 mK.

3. Results and discussion

Resistivity as a function of temperature $\rho(T)$ (see Fig. 2) shows an hysteretic behavior in cooling-warming cycles. This effect is rather small (0.1 %) and is consistent with the findings of Kumamuru *et al.* [1] and Osquiguil *et al.* [2] for thin Cr films grown by sputtering. A better look at the effect of the SDW in transport properties can be obtained from the hysteresis loop: the difference $\Delta\rho(T) = \rho_c(T) - \rho_w(T)$ between the resistivity when cooling $\rho_c(T)$ and warming $\rho_w(T)$ is shown in Fig. 2B). The hysteresis loop presents some remarkable features: *i*) first of all, hysteresis is different from zero only in the temperature window ranging from 60 to 260 K. *ii*) It is recognizable the presence of two regions where the hysteresis is maximum but also *iii*) a very sharp decrease is observed around 180 K.

The origin of the hysteresis in thin Cr films has been recently discussed and so far understood in terms of the confinement of the SDW within the film thickness [1, 2, 14, 17]. Assuming that

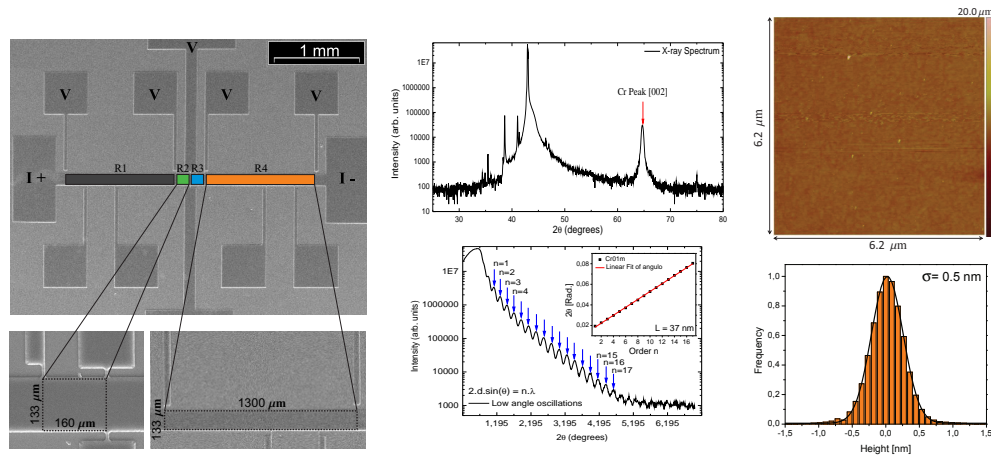


Figure 1. Sample fabrication and characterization: A) Cr thin film patterned as a hallbar with several terminals allowing different configuration measurements. The low panels show a zoom to two sample regions. B) X-ray diffraction spectrum showing the $\langle 002 \rangle$ characteristic Cr peak. The other peaks correspond to the substrate. C) Small angle X-ray reflectometry spectrum from which the film thickness can be obtained. D) AFM topography map and height histogram (inferior panel) showing a mean roughness of $\sim 5 \text{ \AA}$.

both, vacuum at the surface and non-magnetic MgO at the bottom interface define strong boundary conditions for the magnetic state of Cr, a discrete set of wavelengths Λ_n would be stable. As in bulk samples the SDW wavelength changes with the temperature, from 78 \AA at 300 K to 60 \AA at 150 K [18], it is reasonable to assume that the hysteresis in thin films is the result of the discontinuous jumps between a high-temperature wavelength Λ_n to a smaller low-temperature one Λ_{n+1} . Here n indicates the number of nodes inside the film and, as shown by the hysteresis loops in Hall measurements [1], in the low-temperature phase a larger number of effective carriers is present (thus a lower resistance) than in the high temperature phase. Indeed, as shown in the inset of Fig. 2A), resistivity when warming ρ_w is always lower than when cooling ρ_c and hysteresis loop is positive. The jumps that lead to the hysteretic behavior occur as a result of a competition between the energy cost of deforming the SDW (a different wavelength with respect to that expected from bulk, continuously decreasing with temperature, $\Lambda_{SDW}(T)$) and the surface energy related to the boundary conditions (imposing $\Lambda_n = 2L/n$). The change from n to $n + 1$ occurs as a first order type transition at a temperature that is determined by this energetic balance, close to a commensurability condition $L \sim (n + 1)\Lambda_{SDW}(T)/2$, here we recall, L is the film thickness [2].

From the derivative of the resistivity $d\rho(T)/dT$ some other features of the hysteretic behavior become apparent. As shown in Fig. 2C), there is a slope change at 300 K , then a wide minimum and a maximum. The nucleation of the SDW, due to the nesting of electron and hole sheets, is accompanied with a partial destruction of the Fermi surface (through the opening of a gap). As a consequence, resistivity increases at the transition temperature T_N and a change in the slope is thus expected. The combined effect with electron-phonon scattering makes the resistivity decrease once again with temperature and this is the observed minimum. In general, the Néel temperature is defined at the minimum of $d\rho/dT$, however in the present case it is more appropriate define T_N as the temperature at which the slope changes. The former case would be 240 K and the latter, more consistent with the literature, 300 K . The reason why there is so much difference between the position of the minimum in MBE vs sputtering grown Cr thin films is not clear to us at the moment. What is nevertheless more notorious is the fact that cooling

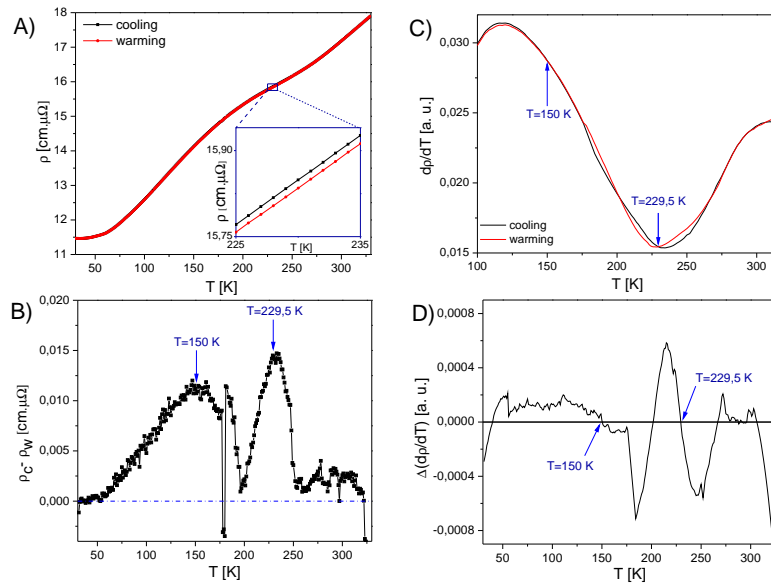


Figure 2. Hysteretic Behavior: A) Resistivity as a function of temperature when cooling (black curve) and warming (red curve). A zoom is shown in the inset to better appreciate the hysteresis. B) Hysteresis loop: difference between resistivity when cooling and warming. The maximum of hysteresis is observed at 150 K and 230 K (indicated with arrows). C) Derivative of the resistivity as a function of temperature showing crossing at the same temperatures where the hysteresis maximum occur. D) Difference between the derivatives of the resistivity when cooling and warming.

and warming derivatives curves have their minimum occurring at different temperatures and as shown in Fig. 2C), they cross at the temperatures at which the maxima of $\Delta\rho$ in the hysteresis loop are found, $T = 230$ K and $T = 150$ K as indicated in the figure.

The hysteresis loop has been shown to be thickness dependent, being this related to the fact the commensurability condition $L \sim (n + 1)\Lambda_{SDW}(T)/2$ involves the thickness L [2, 19]. For different values of L , it is possible to obtain several corresponding sets of available modes with $\Lambda_n \in [60, 78]$ Å and for that reason, depending on the case, more than one transition may be expected. For a 370 Å film, two transitions, the first one $n = 10 \rightarrow 11$ and the second one $n = 11 \rightarrow 12$, may be predicted simply from the strong boundary conditions. Whether these transitions can be resolved is another problem. Each of the first order transitions does not occur in the whole sample all at once, at a given temperature. On the contrary, as temperature is swept there is region of coexistence. An antiferromagnetic domains structure develops consisting on regions of SDW's with different n (i.e. different wavelength, neither orientation nor polarization). The hysteresis loop is a direct consequence of this coexistence and the temperature at the maximum can be taken as the mean (or more probable) transition temperature. The width of the hysteresis loop and the temperature separation between consecutive commensurability conditions determine whether more than one transition can be resolved as in the case presented in Fig. 2. The interesting point to be addressed is that up to now it had not been reported so clearly the observation of multiple phase transitions in Cr thin films. We believe that the reduction of the roughness by growing our films by MBE was crucial for our finding.

We have observed that the hysteresis loops all together with the noise patterns vary little

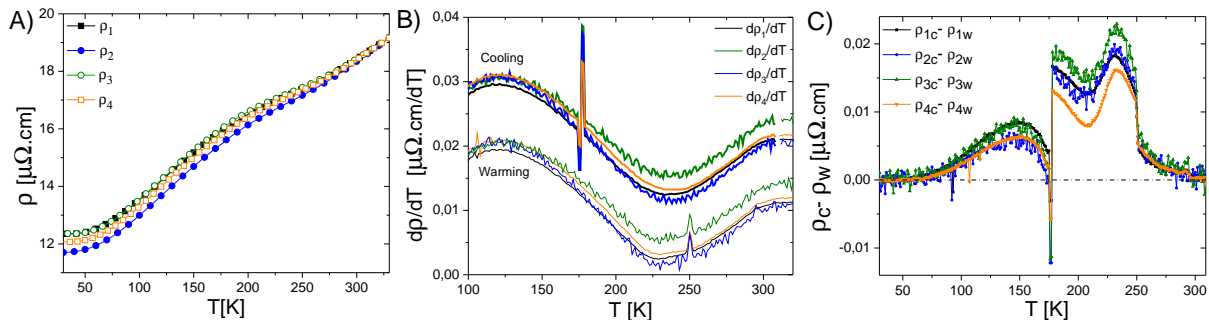


Figure 3. Size effects: A) Resistivities as a function of temperature when cooling for four different regions of the sample identified as 1-4. B) Derivatives of the resistivities as a function of temperature when cooling and warming with an arbitrary offset. C) Hysteresis loops for 1-4.

from run to run but also, rescaling appropriately with the respective dimensions, the hysteresis loops from the different parts of the sample (see Fig. 1A)) can be reproduced with the same parameters. In Fig. 3A) we show the resistivities of these parts, designated 1-4, measured in a cooling-warming cycle with a better than 5 mK temperature stabilization. They coincide at high temperatures above T_N and then follow a similar temperature dependence. The derivatives are shown in Fig. 3B). In all the sample parts there is a strong peak at 180 K when cooling and another peak at 250 K when warming. From a careful look at the resistivities we observe that nothing happens during the cooling at 250 K and similarly, no change occurs in the resistivity at 180 K when warming. The sudden decrease of the resistivity at 180 K when cooling and the sudden increase at 250 K when warming generate a big distortion of the hysteresis loop as shown in Fig. 3C). We do not have an explanation for the origin of these jumps which we think are not directly related to the hysteresis. We believe that an hysteretic transition from a transversal to a longitudinal SDW [7] may be the cause of such behavior. The fact that it seems to occur in the whole sample at once (and considering our AFM characterization) makes us disregard a possible step pinning the SDW and thus creating a big hysteron.

It is also remarkable that the loops are almost the same independently from the size of the sample (there is a factor ten between 1-4 and 2-3, see Fig. 1A)). We believe this is a consequence of the domains being always much smaller than the sample dimensions even for the parts 2 and 3 (whose length is 130 μm). It also tells us that the number of hysterons (related indirectly to the number of domains) scales proportionally to the length of the sample.

L.T. has a scholarship from CONICET and R.H.R has a scholarship from CNEA. Work partially supported by PICT 1060 ANPCYT.

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