Experimental Report

Proposal title: Looking for the charge density wave in chromium islands
Proposal number: 20211641 (ESRF A02-2-875)
Beamline: D2AM (ESRF BM02)
Shifts: 15
Date(s) of experiment: from: 08.04.2022 to: 11.04.2022
Date of report: 09.11.2022

- Objective & expected results (less than 10 lines): **-**

We have grown epitaxial Chromium islands of nanometric to microscopic size, depending on the growth parameters. Bulk Chromium is known to host a spin density wave (SDW) with propagation vector Q=(0,0,1+/-tau) r.l.u., tau~0.05, and a charge density wave (CDW) at 2Q, below 311 K. These density waves are known to be strongly dependent on strain. Here we want look for, and characterise, these density waves in our epitaxial islands. These samples could be model systems to study the interplay between SDW/CDW with crystal defects by Bragg coherent diffraction imaging.

- Justification and comments about the use of beam time (5 lines max.): **-**

All the allocated time (15 shifts) was used, without major beam loss.

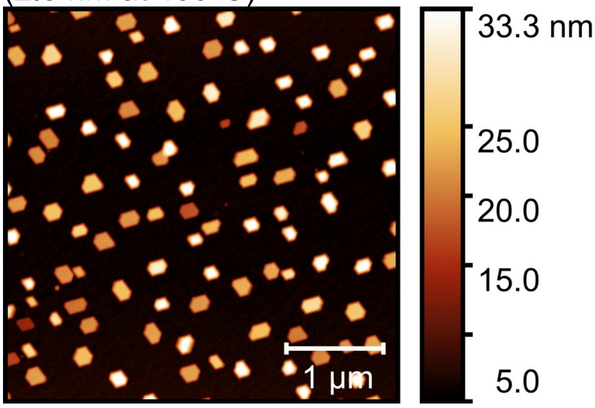
- Publication(s): **-**

We plan to publish a paper on these results

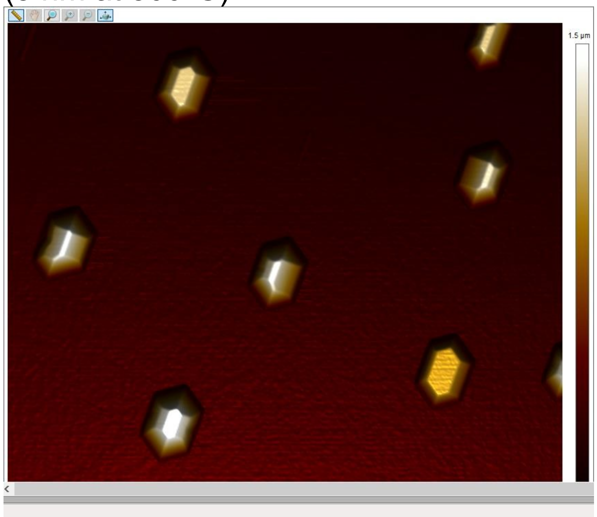
- Results and the conclusions of the study (main part): **-**

We studied 4 samples grown with different parameters, hence different morphologies, as seen by Atomic Force Microscopy in the Figure below:

« DPCr2 »
(2.5 nm at 400°C)

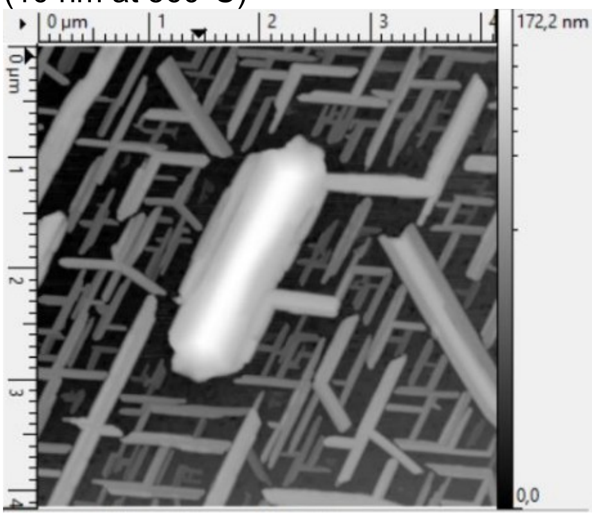


« DPCr3 »
(5 nm at 500°C)

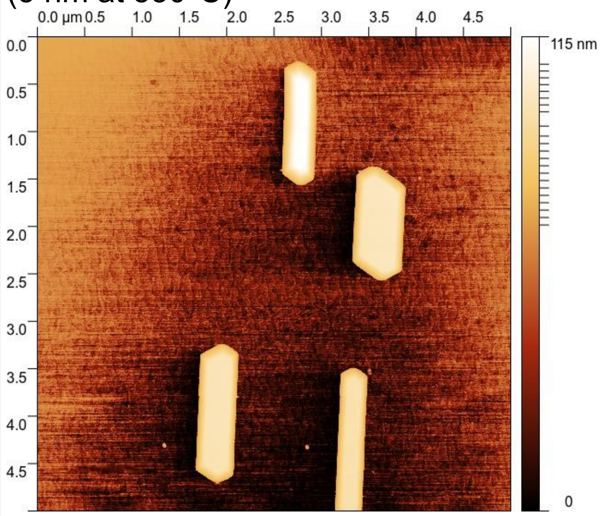


(image width : 4 µm)

« DPCr4 »
(10 nm at 560°C)



« DPCr7 »
(5 nm at 530°C)



The Cr islands grow epitaxially with their (110) perpendicular to the surface of the substrate layer. We explored therefore the reciprocal space in the vicinity of the (110) reflection in Bragg geometry, using the diffractometer in vertical 4-circle mode (3 sample axes + 1 detector axis). A schematic of the reciprocal space around the (110) reflection in bulk Cr is shown in Figure 1. The energy of the X-rays was 8.34 keV. The samples were enclosed in the cryostat and the temperature was varied between ~6 K and room temperature.

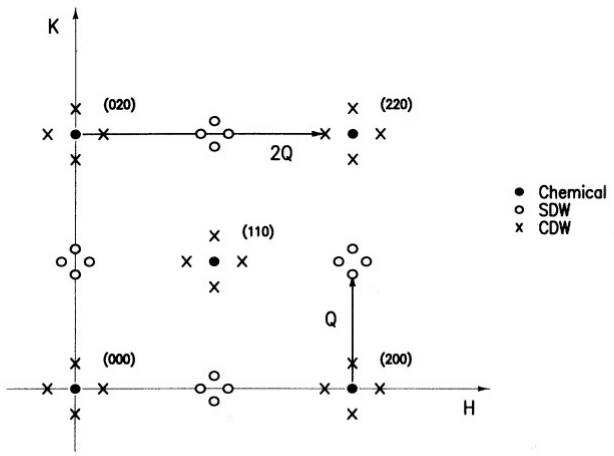


Figure 1: Schematic of the (H,K,0) plane of reciprocal space [Hill et al, Phys. Rev. B 51, 10336 (1995)].

Sample DPCr7

We detail first the results for sample DPCr7, which yielded the stronger intensity and for which could carry a full characterisation.

First, let us note the temperature dependence of the out-of-plane lattice parameter (Figure 2), as measured on the (110) reflection, which clearly shows a phase transition between 300 K and 320 K, with a saturation around 100 K. The overall shape of the curve is very similar to temperature dependence of the order parameter shown by Hill *et al* for bulk chromium [Phys. Rev. B 10, 10336 (1995)]. This suggest a magnetostrictive effect (antiferromagnetic ordering appears simultaneously with CDW and SDW).

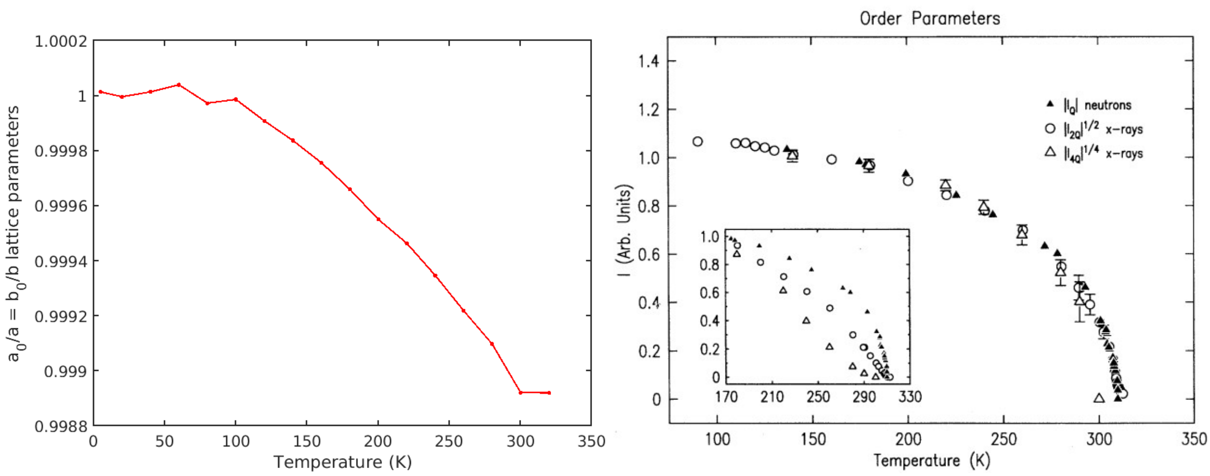


Figure 2: Left : Temperature dependence of the out-of-plane lattice parameter, measured on the (110) reflection. The inverse lattice parameter, normalised by its room temperature value, is plotted. Right : Temperature-dependence of the order parameter in bulk Cr [Hill et al, Phys. Rev. B 51, 10336 (1995)].

We could observe the (1,1±2τ,0) and (1±2τ,1,0) satellites, but not the (1,1,±2τ) satellites, as shown in Figure 3. We believe that the epitaxial strain favours the out-of-plane propagation vectors over the pure in-plane one.

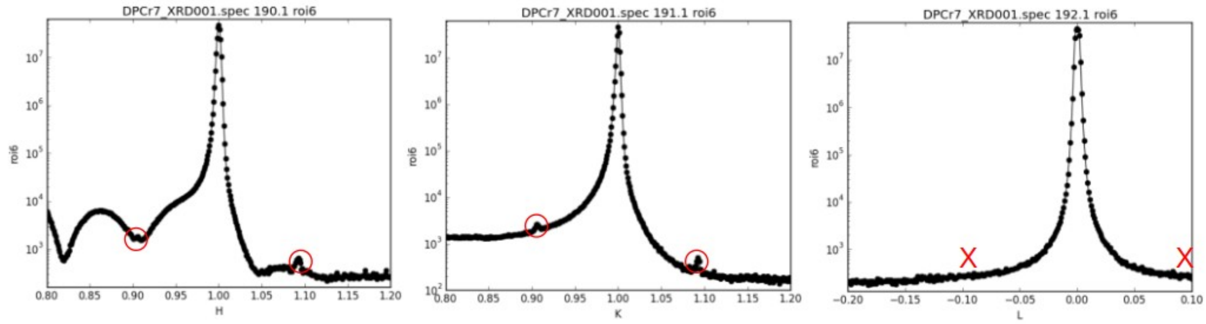


Figure 3: H, K and L scans around the (110) reflection at 6 K).

The intensity is 5 orders of magnitude than the (110) reflection, to be compared with a ratio of 4 orders of magnitude in the bulk, as reported by Hill *et al*. Again, epitaxial strain could be the explanation for this observation: it is possible that CDW form only in the most relaxed parts of the islands, i.e. away from the interface, and possibly away from the corners, edges and a free facets, hence in a much smaller volume than the main lattice.

We followed the temperature dependence of the (1,1+2τ,0) and (1+2τ,1,0) satellites (Figure 4). Their intensity and the value of the propagation vector follows a similar trend as order parameter reported in the bulk.

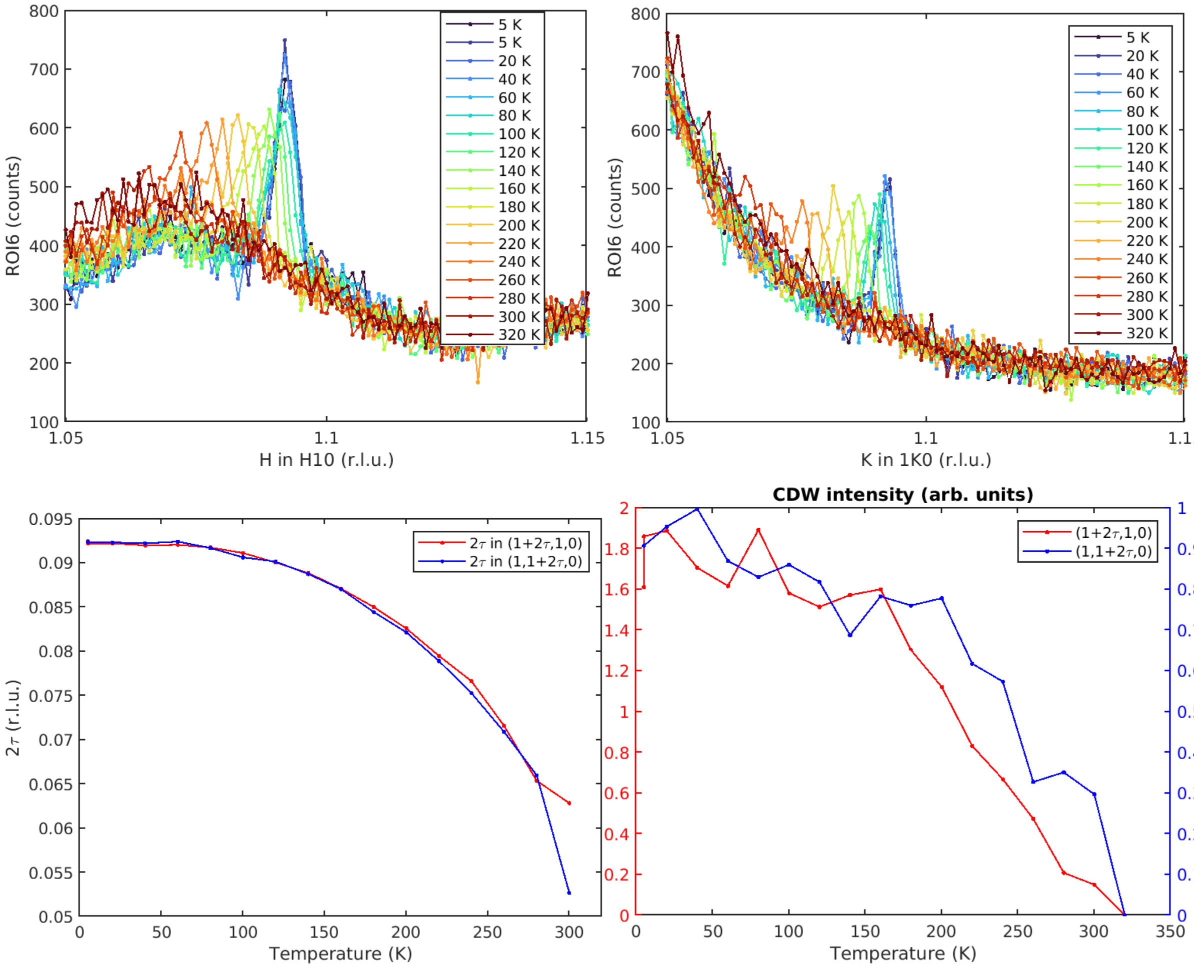


Figure 4: Temperature dependence of the (1,1+2τ,0) and (1+2τ,1,0) satellites.

Sample DPCr4

Similarly, in DPCr4, only the (1,1±2τ,0) and (1±2τ,1,0) satellites were observed (Figure 5), while the (1,1,±2τ) satellites were absent. We had time to record only a rough temperature dependence (Figure 6). The CDW satellites were seen at low temperature (at least up to 200 K), and not at room temperature. The global shape of these curves are very similar to the ones measured in DPCr7.

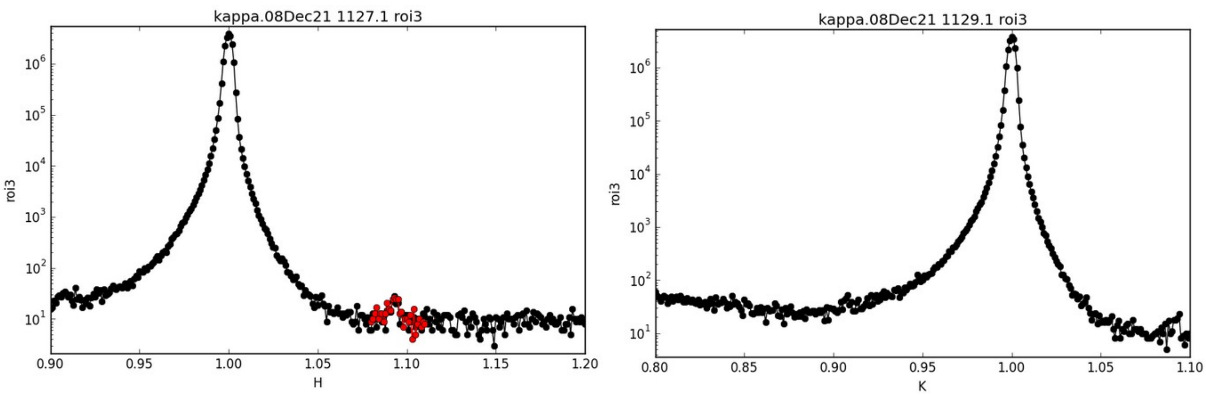


Figure 5: H and K scans around the (110) reflection at 6 K in sample DPCr4.

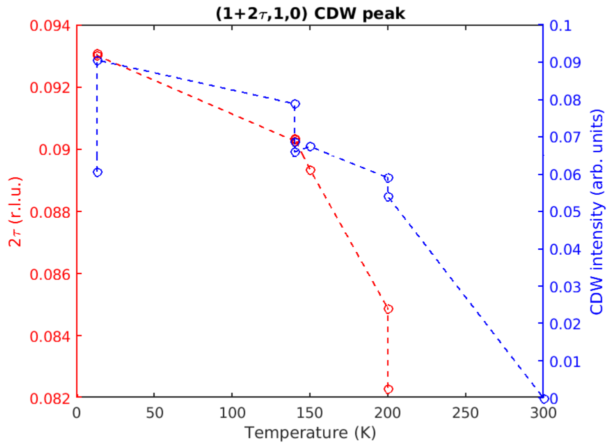


Figure 6: Temperature dependence of the propagation vector and intensity of the CDW peak in DPCr4.

Sample DPCr3

Similarly, in DPCr3, only the (1,1±2τ,0) and (1±2τ,1,0) satellites were observed (Figure 7), while the (1,1,±2τ) satellites were absent. We did not have time to record a full temperature dependence, but the CDW satellites were seen at low temperature (at least up to 120 K), and not at room temperature.

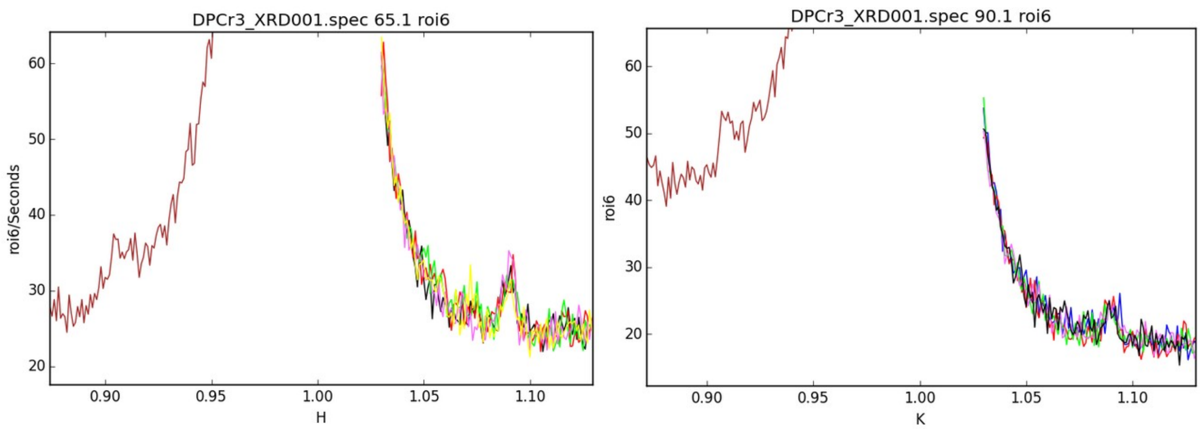


Figure 7: H and K scans around the (110) at low temperature (between 6 K and 80 K) in sample DPCr3.

Sample DPCr2

We were not able to see the CDW satellites in sample DPCr2. One possible reason is the total amount of deposited Cr, which is less than for other samples, leading to CDW satellites too weak to be measured with this set-up. Moreover, the nanoislands in this sample were smaller than in other samples, and it is possible that the epitaxial strain is too large in the entire volume of the islands to host CDW.

Summary

We were able to see CDW satellites in DPCr3, DPCr4 and DPCr7, but not in DPCr2. The intensity of the satellites (weaker than in the bulk as compared to the main reflections) and their absence in DPCr2, which displays the smallest islands, suggests that the epitaxial strain prevents the formation of the charge density waves in part of the crystal. Moreover, the systematic absence of the variant with in-plane propagation vector could also be a consequence of epitaxial strain.

Because of the weakness of the CDW satelittes, **we did not look for the SDW satellites**, which are much weaker in intensity.