ESRF	**Experiment title: Oxidation and reconstruction of the CoGa(100) single crystal surface studied with surface sensitive x-ray diffraction**	**Experiment number**: SI-445
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Shifts: 18	**Local contact(s)**: Luc Ortega	*Received at ESRF:*

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Report: The aim of our experiment was the structural characterization of the clean CoGa(100) surface and the study of the initial oxidation at different temperatures. The experiment can be devided in a more standard part with crystal truncation rod (CTR) measurements form the clean and the oxidized surface and in a non-standard part during which a CCD camera was used to do time-resolved measurements during oxidation.
After cleaning the surface by cycles of sputtering and heating in the UHV surface diffraction chamber the (4*2) reconstruction indicative for the clean surface was observed. In Figure 1 a) curve (1) the structure factor of the (11) CTR is plotted as a function of the reciprocal lattice co-ordinate L perpendicular to the surface, together with a calculation for the bulk terminated surface (line). At L=1 the superstructure reflection of the CsCl structure can be found, whereas at L=2 the fundamental reflection. A tremendous deviation is observed indicative for surface relaxations and the reconstruction. Fig. 1 b) curve (1) and (2) show the (0.5,0) and (0.75,0.25) superstructure rods. In the second rod a modulation is present,

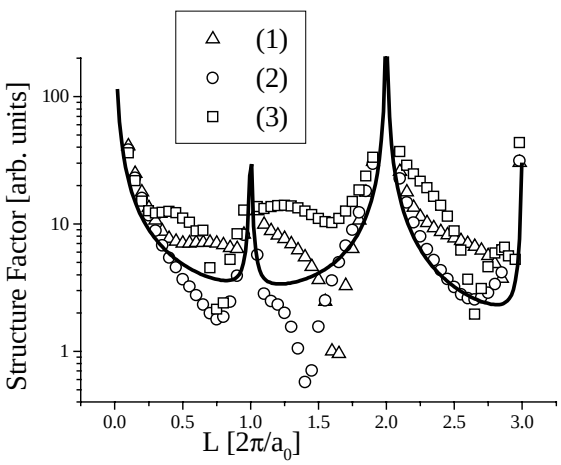


Fig. 1

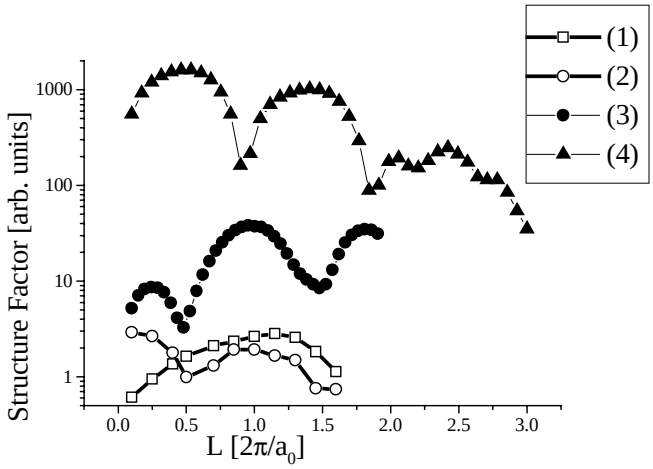
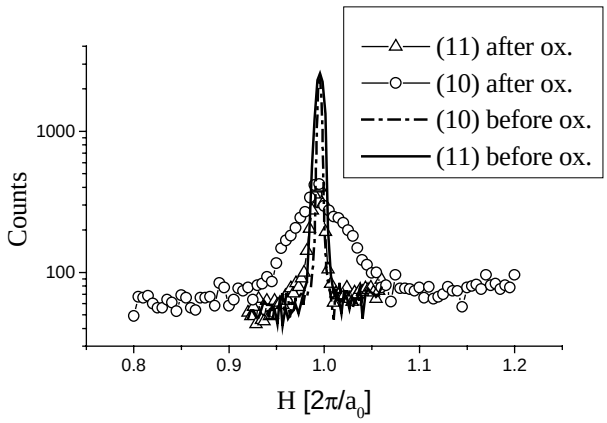


Fig. 2

which is a first hint that the relaxation extends of 1-2 unit cells into the bulk. 4 bulk CTRs were measured and 24 superstructure rods, opening the possibility of a precise structural refinement of the reconstruction, not finally completed yet.
Next the surface was oxidized at 300 K. At this temperature the formation of a disordered oxide layer was observed with STM. Fig. 1 curve (2) shows the (11) CTR structure factor after RT oxidation. The modulation of the (4*2) reconstruction disappeared and the signal in the minima of the CTR decreased because of the increased surface disorder and roughness. The 4 measured bulk CTRs allow us to analyze the interfacial structure between the amorphous oxide layer and the CoGa(100) surface. Finally, we studied the surface structure after oxidation at 450^{o}C at p(O_2)=2x10^{-6} mbar for 250 s, the conditions for the formation of well ordered oxide layers. We observed a (2*1) superstructure, and the modulation due to the oxide film is present on the (11) CTR (Fig.1 curve (3)), as well as on the (2*1) (0.5,0) and (0.5,1) superstructure rods (Fig. 2, curves (3)+(4), offset for clarity; in total 17 independent (2*1) rods were measured). The oxide layer structure will be analysed starting with the monoclinic β-Ga_2O_3 structure, which is the only stable bulk phase of this oxide.
In the second part of the experiment we studied the line profile change due to oxidation to get insight into the surface mass transport processes during the oxide layer growth. In Fig. 3 curve (1) and (2) are depicted linescans through (1,1,1.4) in the (11) and (10) direction. Both scans show a gaussian profile. After oxidation 330^{o}C for 230 s at p(O_2)=3x10^{-8} mbar the (11) lineshape is unaffected, whereas the (10) lineshape has significantly broadened. At this oxidation stage the surface is partially covered with oxide islands, that give rise to the diffuse scattering. To observe the diffuse scattering distribution in reciprocal space we used a Photometrics

Fig. 3



CCD camera system, looking at the intersection of the CTR with the Ewald sphere. In Fig. 4 is plotted a part of a sequence of images taken during oxidation. The initially sharp spot from the CoGa surface smears out in the h and k direction, which corresponds to the formation of regular shaped oxide islands oriented in (10) and (01) directions. After complete oxidation the islands coalesce and the sharp component increases again.
A residual broadening of the peak is due to growth defects in the oxide layer. We conclude that the experiment was very successful in terms of quality and quantity of the results and that the system CoGa is a very good candidate for detailed studies of the initial oxide growth processes in real time using a fast CCD camera detection system.

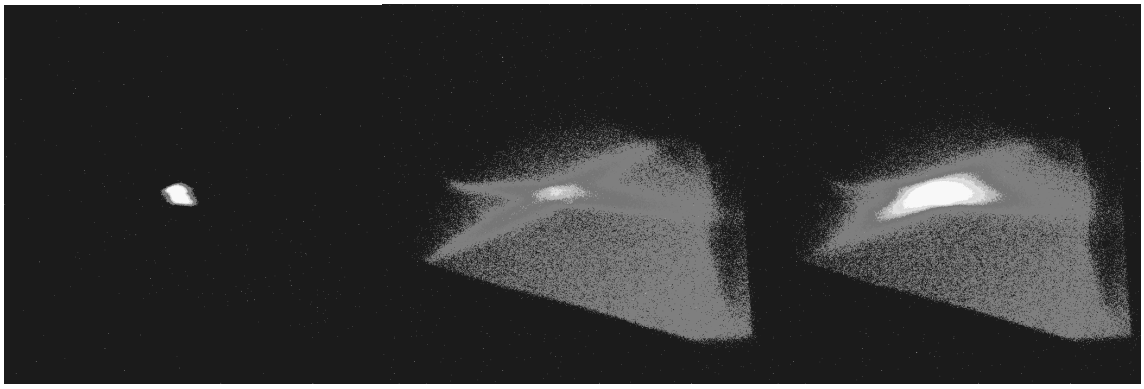


Fig.4 t=0 t=230 s complete oxidation