ESRF	**Experiment title:** Topotactical reactions between ZnO and Al2O3: identification of reaction products and intermediates	**Experiment number**: CH-2814
Beamline: ID03	**Date of experiment**: from:13/05/2009 to:16/05/2009	**Date of report**: 31/08/2009
Shifts: 9	**Local contact(s)**: Dr. Olivier Balmes	*Received at ESRF:*

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Report:

A 20 nm thick ZnO thin film has been obtained by RF-magnetron sputtering of ZnO (Aldrich, 99.99 %) onto Al_2O_3 (0 0 0 1) oriented single crystals (MaTecK) at room temperature. After the deposition the film has been treated in sequence at 800 °C for 120 minutes. According to previous work by our group [1], the topotaxy for the film before the heating treatment is $(0\ 0\ 0\ 1)_{ZnO} \parallel (0\ 0\ 0\ 1)_{sapphire}$. At the firing temperature and after this firing time, for a film 20 nm thick, a solid state reaction takes place to form an unknown compound with an unknown crystal structure: the morphology of the reacted interface is shown in Fig. 1. To obtain structural information, the sample was mounted on the z-axis diffractometer of ID03 (EH1) and surface diffraction patterns have been acquired. We have been able to obtain diffraction effects from the nanocrystal on the sapphire surface. As an example, Fig. 2 shows an *l* scan for h = 1, k = 1 (black line) and h = 1, k = -1 (red line) where the Miller indexes are referred to the sapphire unit cell. In addition to the peaks of Al_2O_3, peaks at non integer values of *l* (2.6, 3.28 and 5.65) are apparent and are attributed to the nanocrystals. Analogously, Fig. 3 shows *h* and *k* scans for *l* = 5.65. We have been able to obtain 25 reflections without doubt due to the nanocrystals. These reflections could be indexed in the hexagonal system with lattice constants *a* = 4.767(10) Å and *c* = 27.614(57) Å. It should be noted that the hexagonal symmetry could be due to a topotactic relatinship between the sapphire and the nanoscristals. However, the value of the *c* lattice constant is quite definite, and it's large value points towards a very complex crystal structure.

In conclusion, we have been able to obtain significant (although incomplete) structural data for the unknown compund that is formed when a ZnO thin film is recated with the (0 0 0 1) sapphire surface. Additional beamtime would be necessary in order to conclude this work.

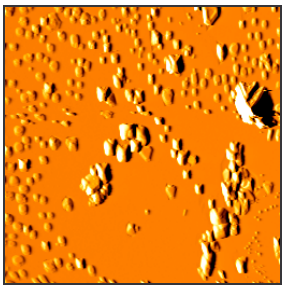

Fig. 1 - AFM image, of the sample investigated in this experiment, showing the morphology of the $(0\ 0\ 0\ 1)_{ZnO} \parallel (0\ 0\ 0\ 1)_{sapphire}$ interface, after firing for 2 hours at 800 °C. The lateral dimension of the image is 20 μm.

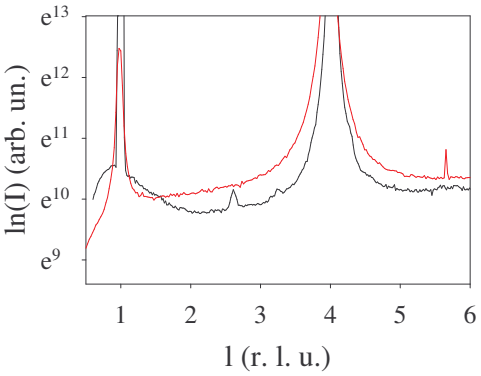

Fig. 2 – l scan for h = 1, k = 1 (black line) and h = 1, k = -1 (red line); in addition to the peaks of Al_2O^3, peaks at non integer l values (2.6, 3.28 and 5.65) are evident.

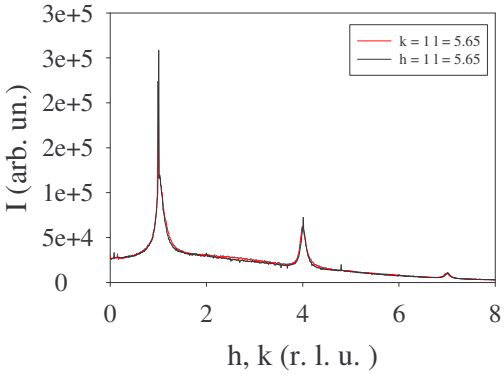

Fig. 3 – h scan (red line) and k scan (black line) for l = 5.65 and k(h) = 1, respectively.

References

[1] - S. Pin, P. Ghigna, G. Spinolo, E. Quartarone, P. Mustarelli, F. D'Acapito, A. Migliori, G. Calestani, Journal of Solid State Chemistry 182, 1291 (2009).