| ESRF | **Experiment title:**<br>Segregation and Ordering at a $Ni_{0.9}Al_{0.1}$(111) surface | **Experiment number**:<br>SI-443 |
|---|---|---|
| **Beamline**:<br>ID 03 | **Date of experiment**:<br>from: 4.2.1999 to: 11.2.1999 | **Date of report**:<br>24.2.1999 |
| **Shifts:**<br>18 | **Local contact(s)**:<br>Alvarez Jesus | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

**Harald Reichert, Oliver Klein, Herwig Requardt**

Max-Planck-Institut fuer Metallforschung
Heisenbergstrasse 1
D-70569 Stuttgart
Germany

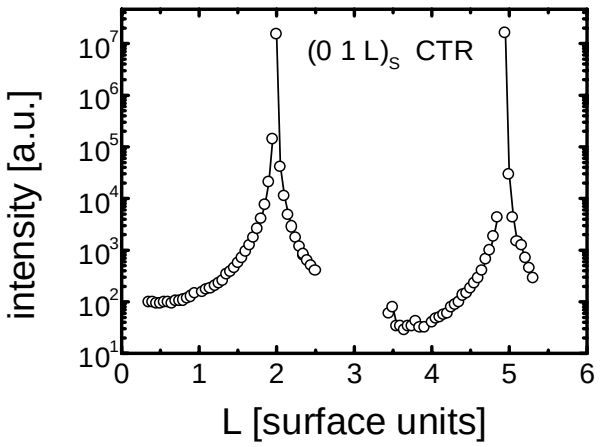
**Report:**

Up to now investigations of the surface structure of compoud systems have been performed mainly at ordering systems. In the this experiment we have tried to study the pure alloy surface of $Ni_{0.9}Al_{0.1}$(111). This system exhibits phase separation in the Ni-rich part of the phase diagram. Recent first-principles theories [1] predicted the formation of a 2-dim. ordered layer at the surface of this system, driven by surface segregation at high temperatures [2,3]. This leeds to the seemingly paradox situation of a long-range ordered surface on top of a phase separating bulk.

The single crystal surface was kept in a portable UHV chamber built at our home institution. In this chamber have prepared and characterized the sample before the actual synchrotron experiment. In a first step we have already removed residual contaminants in the bulk of the sample by repeated sputter and annealing cycles after polishing the sample. The clean $Ni_{0.9}Al_{0.1}$(111) sample surface was than transported to the ESRF under UHV conditions.

After aligning the instrument we have characterizd the structure of the sample in the phase separated state up to T=900K, which includes measurements of the scattered intensity around $L1_2$-superstructure peaks of the $Ni_3Al$ phase. Subsequent heating of the sample to 1270K enhanced the surface concentration of Al to $x_s$=0.25. In the next step we tried to characterize

the inplane structure of the proposed 2D ordered surface layer by measuring the diffracted intensity along the surface normal L starting at (2, -2,0) and (1,-1,0) and their symmetry equivalent positions. The following figure shows the $(01L)_S$ fundamental CTR revealing a smooth and unrelaxed surface structure after the surface preparation procedure.




During the experiment we faced a number of problems with the beamline and the machine:

(i) The optics of the beamline was not aligned correctly (mirror) so that we lost more than an order of magnitude in beam intensity. In addition, the beam was unstable and drifting. Following an injection we had to wait up to three hours to obtain a stable signal-to-monitor ratio.

(ii) The following statement is taken from the operation report of run 99-01/third week : “14 trips in one week was the worst figure experienced for a long time and lead to an MTBF of only 12 hours”. Combined with the stability problems described in (i) we lost the major part of the beamtime assigned for this project

Due to the problems noted above we were not able to confirm or reject the predicted formation of a 2D long-range ordered surface layer at $Ni_{0.9}Al_{0.1}(111)$ in this experiment. Since the expected intensities from the 2D ordered surface layer are very week a substantially longer period of undisturbed operation is required.

[1] T.C. Schulthess, R. Monnier, submitted

[2] M. Polak, J. Deng, L. Rubinovich, Phys.Rev.Lett. **78**, 1058 (1997)

[3] T. Schulthess, E. Wetli, M. Erbudak, Surf. Sci. 320, L95 (1994)