

**Report on experiment n° 2223 :]**  
**“Statics and dynamics of steps at the (111) surface of a copper  
single crystal studied by x-ray coherent diffraction”.**

The aim of the experiment was the observation of surface steps from constructive/destructive interferences between the amplitudes scattered by various terraces. This needs a careful observation of the intensity variations in the vicinity of the “antibragg” (AB) positions, specifically here the  $(1/2 \ 1/2 \ 1/2)$  position of the  $(\vec{G} = (111))$  crystal truncation rod (CTR) for a  $[111]$  vicinal surface copper crystal.

For the observation of surface defects in the vicinity of the AB position, the crystal irradiated by the coherent X-ray beam must be free from structure defects like dislocations, subgrain boundaries. As the beamsize was close to  $10\mu\text{m}$ , a careful sample annealing had to be carried out. After a few hours in the 720-750C temperature range, regions of high crystalline quality were obtained, as shown in Fig. 1.

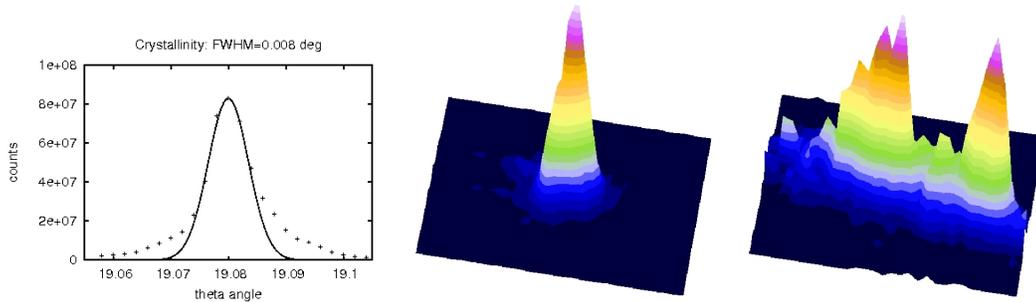


Figure 1: *The Bragg peak width (left figure) is  $0.008^\circ$ , close to the Darwin width of copper ( $0.006^\circ$ ) and a single peak (middle) is observed for the  $(0.85 \ 0.085 \ 0.85)$  position, showing the absence of crystal defects. Speckles are observed (right figure) close to the AB position, imaging a step in the irradiated area*

2d images of the diffraction in the vicinity of various  $\vec{Q}$  value along the CTR were obtained. After annealing, an unique peak was observed for small

values of  $x = |\vec{Q} - \vec{G}|/|\vec{G}|$  ( $x=0.15$  in Fig 1, center), and speckles were observed in the vicinity of the AB position. This means that the observed speckel modulations are connected to surface defects, with a small number of steps and terraces in the irradiated region. ( $x \simeq 0.5$  in Fig. 1, right).

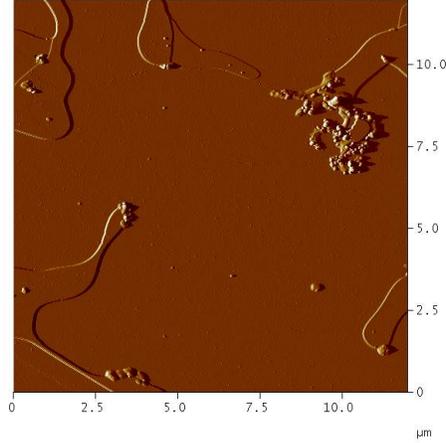


Figure 2: *An AFM map of a  $12 \times 12 \mu\text{m}^2$  sample area after 10 hours annealing at  $720\text{C}$  showing an essentially flat surface with steps of a few nanometer height. This sample was held in air, and some post experiment oxide is observed along the steps*

Our sample was vicinal with a small miscut angle ( $< 0.1^\circ$ ), and after annealing, step bunching produced essentially flat terraces with a small amount of steps of height of the order of 10 nm, as shown in Fig. 2. This could be checked from the small number of speckles observed for  $x < 0.3$  in our diffraction figures.

In this experiment, low intensity in the AB region (2-3 x-rays/s at the AB position above a flat background) made surface dynamics study difficult. As some hundreds of seconds were necessary to obtain a significant statistics, the surface fluctuations were too fast to be observed at  $700\text{C}$ . Improvements in beam intensity can be obtained if the focussing KB mirrors provide a smaller spot and if the beamsizes can be better optimized. This needs to carefully measure the beam dimensions at the sample, and we think that large significant improvements can be obtained.