ESRF	**Experiment title:** Description of a new mechanism of strain relaxation in Buffer layers by means of Reciprocal Space Maps	**Experiment number:** HS-267
Beamline: BM 05	**Date of experiment:** from: 15avr-97 to: 24-avr-97	**Date of report:** ***28-8-97***
Shifts: 21	**Local contact(s):** Souvorov Alexei	***Received at ESRF:*** 1 SEP. 1997

Names and affiliations of applicants (* indicates experimentalists):

Filippo Romanato: Dip. di Fisica "G.Galilei" Via Marzolo 8, 35131 Padova
Marco Natali: Dip. di Fisica "G.Galilei" Via Matzo10 8,35131 Padova
Davide de Salvador: Dip. di Fisica "G.Galilei" Via Marzolo 8,35131 Padova
Sara Gennari CNR-MASPEC Via Chiavari 18/A, 43100 Parma

Report: In the context of the production of electronic and opto-electronic semiconductor devices considerable attention is paid to the development of buffer layers interposed between the substrate and the device layers when the latter two have widely differing lattice parameters. These buffer layers are needed to confine the extended cristallografic defects - which may form as a consequence of the lattice mismatch - into regions far away from the active device layers. Buffer layers with a graded composition profile seem to give the best performances. The comprehension of the meccanism of strain-relaxation in buffer layers is crucial to further develop these structures. In this respect it was found useful to measure, besides of the biaxial strain in the buffer layers, also eventually occuring rotations between the crystal planes of the buffer and the substrate (1). In previous investigations we have found for the first time evidence that this rotation, which we will call tilt, may change in a regular manner from point to point on the sample surface. To understand the origin of this fenomenon it is first of all necessary to characterize it as completely as possible. Therefore measurements of the complete strain tensor and of the rotation matrix as a function of the in plane position and the depth below the surface were carried out by measuring reciprocal-space-maps (RSMs) using X-ray diffractrion. The samples were InGaAs buffers grown on (001) GaAs substrates by MBE at a temperature of 500°C with a As4 source. The 2.4 µm thick buffers had a step-graded composition profile with different number of steps for each sample (3 steps = B3, 6 steps = B6, 9 steps = B9). The nominal Indium fraction of 35% at the surface was approached by increasing the Indium content by equal finite jumps at regular distances from the film-substrate interface giving rise to distinct layers within the buffer. Reciprocal-space maps were collected with the three-axis diffractometer at the open bending magnet beamline BM05 with a horizontal diffraction plane. Monochromatic radiation of wavelength $\lambda=1.49976$ produced by a Si(lll) monochromator was collimated by slits producing a spot of footprint lmm x lmm on the sample surface. The RSMs of the (004) symmetric reflection were recorded using a NaI scintillation counter prefaced by a Si(111) two bounce analyzer crystal. For the RSMs of (335) asymmetric reflections at grazing incidence the analyzer was replaced by slits in front of the detector. All reflections were measured with azimuths both along [llo] and [l-lo] directions and at different points on the sample surface.

The presence of a regularly changing tilt was confirmed as is shown in figure 1 for the buffer B3 and in figure 2 for buffer B6. For buffer B9 only 3 points were measured. The figures show the tilt direction and magnitude from layers 1 through N at different measuring point (circles) lying on a grid with 4mm steps both long x=[llo] and long **y=[1-10]**. Layer 1 is the one closest to the substrate. For convenience of representation the reported tilts are referred not to the substrate **but** to the underlying layer.

figure 1

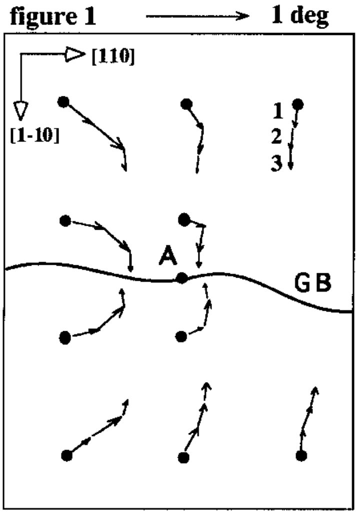


figure 2

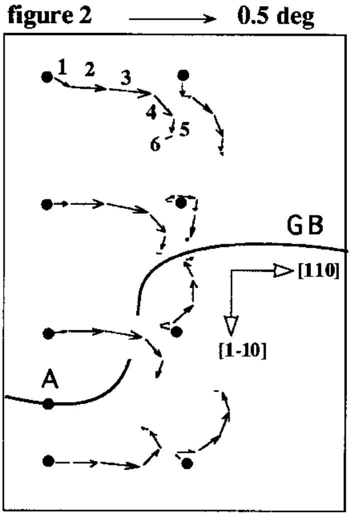
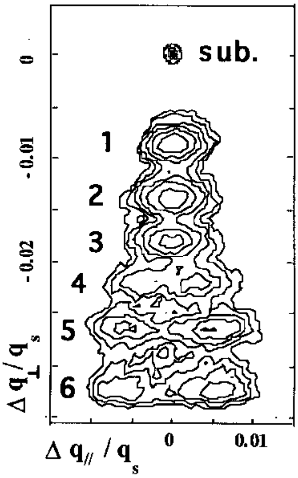


figure 3



In a very qualitative way we might describe the behaviour of the tilt as follows: (A) The tilt vectors $\tau=(\tau_x,\tau_y)$ converge to some region contained within the sample area. (B) The magnitude of the individual layer tilts initially increases and then decreases. The final decrease is due to the decreasing strain relaxation of the last few layers. Indeed the individual-layer strain-relaxation rates (in %) are: from layer 1 through N for B3: 93,93,63 for B6: 94,94,99,94,73,43, for B9: 95,89,96,100,93,90,88,69,57. (C) When either of the the curvatures $\chi_x=d\tau_x/dx$ or $\chi_y=d\tau_y/dy$ exceeds a critical value a small-angle grain boundary (GB) develops in the perpendicular direction, dividing the sample area into two large macroscopic grains. For the buffer B3 the grain boundary forms after layer 1 (verified at point A of figure 1). Similarly for the buffer B6 the grain boundary forms after layer 3. Indeed as can be seen in figure 3, which shows a (004) RSM along [l-lo] at point A of figure 2, the peaks of layers 4, 5 6 are doubled due to the presence of two grains within the lmm^2 of the X-ray spot. (D) Within each grain the curvatures χ_x, χ are constant and $\chi_x > \chi_y$. ,This was verified indipendently by previous measurements of the surface tilt by channeling-RBS on a tighter grid of points. (E) As the ordinal number of the layer increases the tilt direction rotates in the same sense within each grain, passing through direction [llo] and eventually returning to direction [llo]. There is however an interesting exception: we observe that passing from layer 1 to layer 2 of buffer B6 in figure 2 there is a very large rotation of the tilts in the righthand column of points, whereas there is only a large increase in magnitude of the tilts in the lefthand column. Furthermore initially the tilts in the two columns are directed oppositely. These observations suggest some hind of "repulsion" between oppositely oriented tilts acting over macroscopic distances. A further indication for such a repulsion might be seen in the evolution of the tilts of layers 5,6 at the two points neighbouring A in figure2. With this idea in mind one might think of the evolution of tilts in the following way: The sample border somehow polarizes the tilts and this polarization is transmitted across the whole area of the sample. If the borders produce a convergent tilt pattern, probably somewhere on the sample a grain boundary will be produced. Some words on the shear strain: considering the angles α,β,γ of the unit cell, with γ characterizing the (001) face parallel to the film surface, values of α,β differing from 90deg by about 150+30 arscec were found for all buffer layers. Such large triclinic distortions to our knowledge are not observed in InGaAs/GaAs single-layers without tilt and therefore they should be connected with the long-range force which leads to the formation of the observed regularly varying tilts in our graded buffer layers.

(1) LeGoues, BSMeyerson, J.F.Morar, P.D Kirchner J.Appl.Phys 71 (9) 1992, 4230