Observations of flow in In$_x$Ga$_{1-x}$As multilayers

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Abstract. Work elsewhere has shown that the hardnesses of coherent In$_x$Ga$_{1-x}$As multilayer structures in which the misfit stresses are controlled by varying the indium content in each layer are influenced by the thicknesses and the coherency strains in the layers. These results have been interpreted in terms of the length-scale for flow being greater than the layer thickness and associated with deformation size effects. However, nanoindentation of In$_x$Ga$_{1-x}$As suggests an influence of the flow stress of the individual layers on the overall multilayer flow stress. Consequently, initiation of flow in the weaker layer is expected. A deformed multilayer has been characterized by measurement of the lattice rotations measured from energy-filtered convergent beam electron diffraction (CBED) patterns recorded in scanning transmission electron microscopy (STEM) mode. Based on the movement of Kikuchi lines as a result of rotations of the lattice the local orientation of the crystal can be extracted, allowing the local orientations to be estimated. The small probe-size in CBED of ~1 nm used here ensures the region sampled is smaller than the thickness of the individual layers. These measurements have been used to construct a map of the axes of rotation in the lattice which demonstrates the ability to distinguish the individual layers.

1. Introduction

There has been considerable interest recently in the mechanical behaviour of coherently strained superlattices, in particular as model systems in which the coherency strains can be modified by varying the concentration of indium within the layers. Based on experiments in which the thickness and coherency strains in the layers were systematically varied, the latter by varying the concentration of In, previous work on In$_x$Ga$_{1-x}$As [1] has concluded that there exists a minimum volume required for flow to occur. This work has assumed that the flow stress is not influenced by the In concentration, x. However pure GaAs and InAs have microhardnesses of 7.5 and 3.5 GPa respectively [2], and measurements by Vigdorovich and Patriarche [3, 4] suggest a non-linear change in hardness with composition, with a maximum hardness at an atom fraction of In of 0.2 (In$_{0.2}$Ga$_{0.8}$As). The aim of this paper is to investigate whether such effects occur here and whether they might influence the earlier conclusions.
2. Experimental methods

2.1 Materials

2 µm thick monolithic In$_x$Ga$_{1-x}$As layers were grown by molecular beam epitaxy (MBE) on GaAs substrates to measure the variation of yield pressure and hardness with $x$. In$_x$Ga$_{1-x}$As multilayers were grown by MBE on InP substrates, with individual layer thicknesses of 50 nm to a total thickness of 2.5 µm. Coherency strains are introduced in such structures by varying the In concentration from $x = 0.532$, which is the composition with the same lattice parameter as InP. This is described fully elsewhere [1]. Layers of equal tensile and compressive strain were alternated to achieve approximately zero net strain of the multilayer. All samples were characterized by XRD to determine misfit strains, compositions and degrees of relaxation. The monolithic layers were relaxed and more detail on the multilayers is given in table 1.

| Sample | $\varepsilon_T$ (%) | $\varepsilon_C$ (%) | $x_T$ | $x_C$ | Relaxation     |
|--------|---------------------|---------------------|-------|-------|----------------|
| ML0.0  | 0.0                 | 0.0                 | 0.532 | 0.532 | Fully strained |
| ML0.2  | 0.2                 | 0.2                 | 0.503 | 0.561 | Fully strained |
| ML0.4  | 0.4                 | 0.4                 | 0.474 | 0.590 | Fully strained |
| ML0.6  | 0.6                 | 0.6                 | 0.445 | 0.619 | Fully strained |
| ML0.8  | 0.76                | 0.88                | 0.422 | 0.66  | Fully strained |

2.2 Indentation

Spherical indentations were made using a tip radius of 10 µm (Micromaterials NanoTest 600). Multiple partial unloading was used with 50 unloading cycles to 75% load and a total depth of 500 nm at a loading and unloading rate of 1 mN s$^{-1}$. Indentation stress and strain were calculated as described by Field and Swain [5] and the yield pressure was defined as the first detectable deviation from linear elasticity.

Hardness measurements with a depth of 200 nm were made using a Berkovich tip at a strain rate of 0.05 s$^{-1}$. Continuous stiffness measurements (MTS Nanoindenter XP) showed no significant influence of the different substrates at this depth.

2.3 Transmission electron microscopy

A thin foil was prepared from under a spherical indentation in ML0.8 by focussed ion beam milling. Scanning transmission electron microscopy (STEM) was carried out using an accelerating voltage of 200 kV (FEI Tecnai F20). The convergent beam electron diffraction (CBED) patterns were collected using energy-filtered TEM (EFTEM) with a slit width of ±10 eV. The probe size was 1 nm and the convergence angle was 0.09°.

CBED patterns were collected over an area of 400 by 200 nm with a horizontal spacing of 20 nm and vertical spacing of 10 nm between the sampled areas. From two pairs of Kikuchi lines and the 000 beam the orientation of the crystal relative to a reference orientation can be determined at each point as described elsewhere [6]. The rotation of each point relative to the reference orientation can then be expressed as an angle of misorientation and a corresponding axis of rotation.

3. Results and Discussion

3.1 Indentation

Indentation showed that both the yield pressure, where yield starts, and the overall hardness vary with the concentration, $x$, of In, see figure 1. The yield pressure reaches a maximum where $x \approx 0.2$ and varies linearly over the range of compositions used in the multilayers. This is in good agreement with microindentation data published by Vigdorovich et al. [4].
The hardness of the multilayers on the other hand did not vary with misfit strain, see figure 2. Here the yield pressure is plotted against the deviation of the In concentration from the lattice-matched value, $x = 0.532$. The multilayer hardness therefore follows a rule of mixtures, which is in agreement with measurements on ceramic multilayers [7].

This is compared with the yield pressures measured by Jayaweera et al. [1] for a wider range of multilayer structures in figure 2. Here the net strains were not always zero, so the deviations from the lattice-matched composition were not always the same in the tensile and the compressive layers in these multilayers. Each multilayer is therefore represented by two points, one giving the composition of the tensile layer, the other that of the compressive layer. It can be seen that the yield pressures of the multilayers (as opposed to the hardnesses) tend to follow the yield pressures for the material with the higher In concentration (the weaker of the two layers), shown by the dotted line. The gradient of the yield pressure measured on the monolithic samples is lower compared to the multilayers, but this can be accounted for by the coherency strains in the respective layers. The implication is that at the onset of yield only the softer layer deforms, as observed elsewhere [7]. The hardness however appears to depend only on the average composition of the two layers, suggesting that as plastic flow develops both layers must deform. However if yielding can occur in a single layer, then the earlier conclusion [1] that deformation requires a minimum volume extending over both layers would be incorrect.

3.2 Observation of flow

To investigate this idea, TEM was used to study the flow patterns. At the edge of the plastic zone bowing dislocations have been observed, broadly similar to our earlier findings [8]. To obtain more quantitative measurements we have measured the rotations of the crystal lattice around the indentation. Deformation in individual layers is more quantitatively observable as lattice rotations, see figure 3. All misorientations are low at 0 - 3°. The crystal was found to rotate around axes distributed over 180° in
a half-plane indicated in the legend in figure 3. Towards the top left the axis of rotation is the same in both layers, as might be expected in a region closer to the indenter. However further away, there are differences in the rotation axes in the different layers, consistent with the idea that deformation occurs separately in the different layers. Further work is underway to confirm these differences.

**Figure 3.** Position of examined area relative to the indentation, high angle annular dark field image of the same area and map of axes of rotation. The axis of rotation changes within the marked half-plane.

### 4. Conclusions

Indentation experiments indicate the In concentration influences the flow stress of In$_x$Ga$_{1-x}$As and that the magnitude of this is consistent with the change observed in In$_x$Ga$_{1-x}$As multilayers, suggesting that flow can occur separately in both layers. If this is correct, the earlier conclusion [1] that initial yield requires a minimum volume extending over both layers would be incorrect. TEM showed a difference in lattice rotations between the layers, consistent with the idea that deformation in the two layers can occur separately.

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