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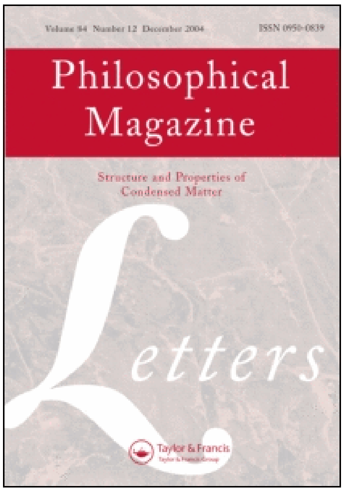

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Plastic deformation under microindentations in GaAs/AlAs superlattices

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ABSTRACT

Presented is a high-resolution scanning electron microscopy study of the mechanical behaviour of GaAs/AlAs superlattices that have been deformed through microindentation. Transverse cleavage through the indentation sites reveals the pattern of the deformed multilayers, showing in particular debonding between the GaAs and AlAs layers, regions of compressive strains of up to 30% and substantial slip on the {111} planes.

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§ 1. INTRODUCTION

The elastic–plastic response of materials to microindentation is not well understood in either an experimental or theoretical context, nor is there enough experimental information available to support proposed models (Chiang, Marshall and Evans 1982, Yoffe 1982) or computer simulations (for example Laursen and Simo (1992)). Advances in indentation testing (Pethica, Hutchings and Oliver 1983) and surface microscopy mean that the deformation of very small volumes can be examined through techniques such as scanning tunnelling microscopy (Castell, Walls and Howie 1992, Walls, Chaudhri and Tang 1992) and high-resolution scanning electron microscopy (SEM). Indentation studies provide a convenient and well controlled method for testing the mechanical properties of surface layers, but little subsurface information is gained directly unless additional sample preparation such as thinning for transmission electron microscopy is undertaken (Page, Oliver and McHargue 1992). A popular approach for metals and other plastic materials has been to pattern split specimens before reassembly and subsequent indentation. A resulting change in the mapping grid is then observed (Atkins and Tabor 1965), but this technique is not really satisfactory because stresses cannot cross the boundary at the split; similar work by Sebastian and Biswas (1991) using wedge indenters also suffers from this restriction. Another method has been to cut through large indentations of millimetre dimensions and to etch the exposed surface. Using this method, Chaudhri's (1993) recent work on mild steel containing parallel sheets of pearlite grains has revealed the shape of the deformed volume under various indenter geometries and these results may be compared with work by Samuels and Mulhearn (1957) on 70–30 brass.

To map the deformation at much higher spatial resolution we use single-crystal superlattices that are made up of GaAs/AlAs layers and cleave through the indentation sites. The deformed layers are imaged with in-lens SEM equipped with a field emission gun which is capable of spatial resolutions of less than 1 nm. These superlattice structures which have periodicities that would normally be beyond the resolution of conventional SEM can now be imaged directly using secondary electrons (SEs) or

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backscattered electrons (Ogura 1991). In our study we have used the way that the SE signal provides contrast due to atomic number differences as well as the topographic structure of the surfaces of the cleaved multilayers.

§2. EXPERIMENTAL PROCEDURES

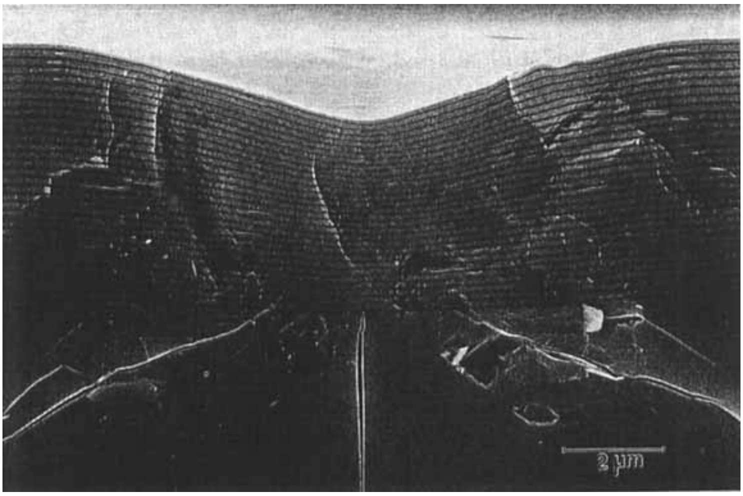
The materials used were fabricated by molecular beam epitaxy (MBE) in a VG Semicon V80H machine where the GaAs/AlAs superlattices were grown on buffers 500 nm thick on GaAs (001) substrates. The periodicities of the structures were subsequently characterized using double-crystal X-ray diffraction and compared with SE images taken in a Hitachi S900 scanning electron microscope; the X-ray data (where available) and SEM measurements corresponded to within 5% of the layer periodicity specifications.

Indentations in the load range 15–100 gf were made using an Ernst Leitz Miniload hardness tester that was fitted with a diamond Vickers indenter. Our experimental procedure involved aligning the diagonals of the indenter with the [110] sample direction and then making a line of indentations in the [110] direction. The sample was then cleaved along the indentations which produced a cross-sectional $(1\bar{1}0)$ plane through the volume of the deformed material. Without additional preparation, samples were transferred to the ultra-high-resolution scanning electron microscope which was typically operated at accelerating voltages of 20 kV.

§3. RESULTS AND DISCUSSION

The SE micrograph in fig. 1 shows the $(1\bar{1}0)$ cleavage plane which lies at right angles to the surface and has produced a cross-section through a 50 gf indentation made into a GaAs/AlAs superlattice of 160 nm periodicity. The exposed material appears dark and light corresponding to the AlAs and GaAs layers respectively and the boundary of the deformed volume can be readily identified. A comparison of the layer positions and

Fig. 1



Cross-section of a 50 gf indentation produced by cleavage through a GaAs/AlAs superlattice with 160 nm periodicity. Extensive compression and bending of the layers is evident as well as characteristic cracks which start at the GaAs–superlattice interface.

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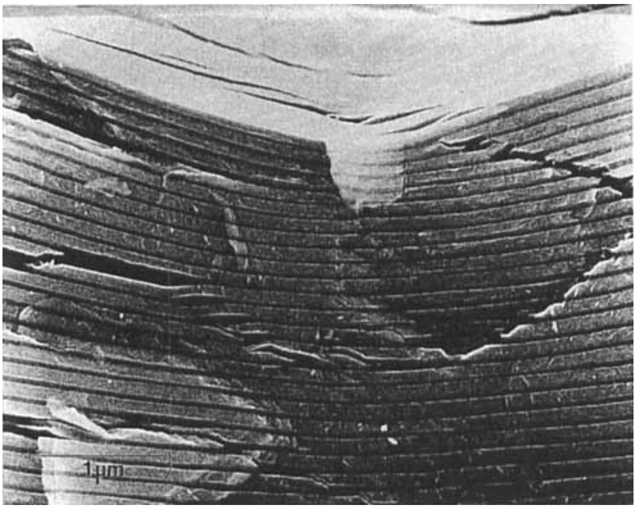
separations directly under the bottom of the indentation with those that are far away indicates a compressive strain of about 30%, a value which drops off to about 10% near the superlattice–GaAs interface. This strain implies a large amount of plastic shear flow within the layers. Furthermore, the displacements of the layers necessitated by this deformation appear to take place by continuous bending rather than by a step process (as in fig. 3(*b*)), and, as the interfaces are weaker than the layers themselves (discussed later), there must have been a relatively large amount of strain in the interfaces. An accumulation of dislocations at the interfaces would also be necessary to produce the bending. Characteristic compressive cracks, which were almost certainly formed during the loading phase of the indentation test, originate at the superlattice–GaAs buffer interface; this is probably because stresses in the unlayered GaAs can no longer be accommodated through shear in the weak interfaces of the superlattice which results in the visible cracking.

Figure 2 shows a close-up of the region under a 100 gf indentation which was made into the same material as that in fig. 1. In this micrograph, deformation mechanisms other than just compression and shear of the layers become apparent and, in contrast with fig. 1, the cracks were probably formed during the unloading stage of the indentation test. Noteworthy are the apparent differences in the mechanical properties of the individual compounds which are shown in some areas through cracking across the layers; the GaAs layers have fractured, whereas the thinner AlAs layers have undergone extensive bending but are still intact, although the effect of the different layer thickness might be significant here. Many areas in this region of the sample also show layer delamination which suggests that the layer interfaces are weaker than the materials themselves. This effect will allow the stress field to fall off faster with depth than would be the case in the unlayered materials.

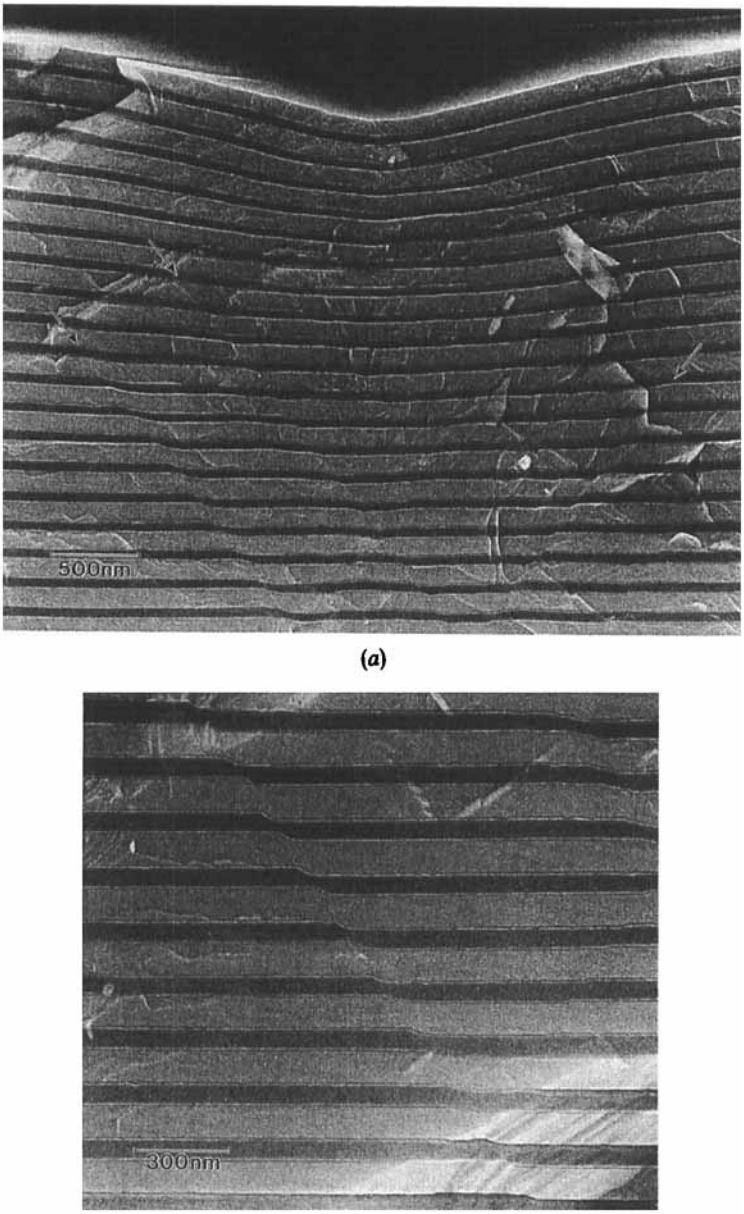
Studies of indentations made at lower loads than those discussed above, reveal that the effects of fracture and delamination are greatly reduced. Figure 3(*a*) shows an

Fig. 2



Close-up of the region at the bottom of a 100 gf indentation showing cracking and delamination between the layers. The bright GaAs layers seem more prone to fracture than the dark AlAs layers.

Fig. 3



(*b*)

(*a*) A 15 gf indentation where there is little cracking or delamination. Two (111) slip planes and one $(\bar{1}11)$ slip plane are indicated in the micrograph. (*b*) A close-up of (111) slip planes where about 80 individual slip processes have occurred on approximately 70 planes.

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indentation made at 15 gf where the deformed volume is easily identified, but not much cracking or delamination is evident. The region below the indentation does, however, show clear evidence of slip planes which make an angle of approximately 56° with the layers. A (111) plane slipping in the $[10\bar{1}]$ or $[01\bar{1}]$ directions and a $(\bar{1}\bar{1}1)$ plane slipping in the $[0\bar{1}\bar{1}]$ or $[\bar{1}0\bar{1}]$ directions would both have an angle of 54·73° between the slip planes and the layers. Our experimental evidence certainly suggests that these slip systems have been activated in the deformed material seen in the micrographs. The offset of the layers can be measured from the micrographs and thus an estimate made of how many slip processes have occurred in one region. For the slip plane shown in fig. 3(*b*), found under a 15 gf indentation, the offset perpendicular to the layers is approximately 23 nm which corresponds to about 80 individual slip processes on the (111) planes slipping in the $[10\bar{1}]$ or $[01\bar{1}]$ directions. From the micrograph, one can also measure that the number of planes that have slipped is of the order of 70, which might lead one to the conclusion that each (111) plane has slipped approximately once.

§4. CONCLUSIONS

This indentation study has highlighted the usefulness of layered structures in studying the plasticity of materials and has shown some of the mechanisms that are active in the deformation of semiconductor superlattices. It would also be possible to study the mechanical properties of other semiconductors by using similar layers as inert markers; this research is currently in progress. As our samples are brittle materials, elastic effects are significant during the indentation process and we would hope in future to describe the deformation using Yoffe's (1982) model or that proposed by Chiang *et al.* (1982). Using information from fig. 1 and assuming isotropic flow and no densification of the material, one could calculate the precise flow pattern of the material under the indenter.

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