Journal of
MATERIALS RESEARCH

Scanning electron microscopy observations of misfit dislocations in epitaxial $In_{0.25}Ga_{0.75}As$ on GaAs(001)

R. R. Keller and J. M. Phelps
National Institute of Standards and Technology, Materials Reliability Division, 325 Broadway, Boulder, Colorado 80303

(Received 20 January 1995; accepted 3 November 1995)

Dislocations in the misfit epitaxial film system $In_{0.25}Ga_{0.75}As$ on GaAs(001) were imaged using a modified electron channeling contrast technique in a LaB_6 SEM. We obtained images at an incident beam energy of 30 keV, a beam divergence of less than 1 mrad and a specimen tilt of 70° in conjunction with a movable scintillator detector mounted at a takeoff angle of approximately 3° to 5°. We achieved a spatial resolution of approximately 80 to 100 nm with this technique. Such resolution allowed rapid imaging of clusters consisting of only a few closely spaced dislocations in a 55 nm thick film. At such small film thicknesses, we did not require accurate knowledge of the incident beam direction in order to obtain sufficiently strong channeling contrast for qualitative characterization. The observed defect arrangements included features that we believe represent clustered threading segments.

Scanning electron microscopy (SEM) for the investigation of crystal defects has been rarely used over the years, despite several theoretical and experimental demonstrations of its utility.[1–6] The localized elastic distortions associated with the core region of an isolated dislocation or a small cluster of dislocations in an otherwise perfect crystal can provide enough of a perturbation in the lattice to result in a detectable variation in the backscattered electron (BSE) intensity, provided the crystal is oriented in a channeling condition. Pitaval *et al.*[4] and Morin *et al.*[5] obtained the first dislocation images from bulk materials, using a field-emission SEM and a retarding-field energy filter. The filter removed the noise-producing low energy electrons from the signal. By incorporating high specimen tilts and low detector takeoff angles, the low-loss portion of the BSE signal can be naturally enhanced without an energy filter.[7] This decreases electron penetration and therefore the chances for inelastic scattering, leaving a high energy signal which contains crystallographic information. Wells[8] showed that for a specimen tilted 60°, approximately half of the detected electrons leave the specimen with at least 95% of their initial energies, when incorporating a detector takeoff angle of about 4° to 5° off the specimen surface tangent. Wilkinson *et al.*[9] showed for the first time that a field emission source was not required for the observation of dislocations using electron channeling contrast. We demonstrate in this communication another application of dislocation imaging from bulk specimens in a LaB_6-equipped SEM, with only minor modifications to the instrument. Films of $In_{0.25}Ga_{0.75}As$ on GaAs(001), containing a high density of misfit dislocations, were investigated. Channeling contrast images of dislocation clusters are presented. We show evidence of threading segments in these images.

$In_{0.25}Ga_{0.75}As$ was grown by molecular beam epitaxy onto a GaAs(001) substrate under the following conditions. A 100-nm film of GaAs was deposited onto an oxide-free GaAs substrate at 580 °C, with an As/Ga ratio of about 3. The desired 25% indium mole fraction was set while growing InGaAs at 450 °C, followed by evaporation of this film at 700 °C. Approximately 300 layers of GaAs were then grown as a buffer at 580 °C and annealed to produce a sharp (2×4) reflection high energy electron diffraction (RHEED) pattern. The sample was cooled to 450 °C and a 55-nm film of $In_{0.25}Ga_{0.75}As$ was grown at 0.25 layer per second. The lattice constant mismatch was determined by Vegard's law to be 1.8%. A 2-nm GaAs layer was then evaporated at this temperature as a cap; such a thin layer had a negligible effect on the images since channeling contrast information arises from volumes over an order of magnitude deeper. No three-dimensional features were seen in the RHEED patterns during growth. The surface lattice constant for this alloy has been observed to almost fully relax, implying the formation of a high density of misfit-relieving dislocations.

We observed pieces of the as-grown specimen in a LaB_6-equipped SEM at 30 kV using probe currents of 0.1 to 1.0 nA. A tilt/rotation specimen stage was used for imaging under various orientation conditions at working distances of 28 to 39 mm and a specimen tilt of 70°. A 50 μm diameter objective lens aperture resulted in a beam divergence of 0.6 to 0.9 mrad. We used a scintillator/light-pipe/photomultiplier detector mounted in a variable low takeoff angle position. The scintillator was standard P47 material and had a diameter of approximately 12 mm. The detector was positioned at 15° to 20° off the incident beam direction, and approximately 80 mm away from the point of intersection between the incident beam and the specimen. This configuration resulted in takeoff angles of 3° to 5° from the specimen surface tangent and a solid collection angle of approximately 0.07 sr. The primary reason for using a small collection angle was to maximize the average energy of detected electrons, based on the approach of Wells.[8] We found that channeling contrast decreased strongly for takeoff angles less than 3° and greater than 5°, as determined by comparing image contrast as a function of detector position for a 70° specimen tilt. All observations were made at room temperature, and a cold trap was used to reduce contamination from within the specimen chamber. A Kalman frame-averaging routine was used to improve the signal-to-noise ratio of the images by feeding the detected signal into a workstation which had direct control of the electron beam. Up to ten $512 \times 512 \times 8$-bit frames were averaged, using a 6800 ns beam dwell time per pixel. No additional image processing was performed.

Although our SEM did not have electron channeling pattern (ECP) capabilities, we nonetheless had little difficulty obtaining strong contrast from these samples. The use of electron channeling contrast to image dislocations requires precise setting of the incident beam direction along a channeling-in direction, for cases where the strain field about the dislocations varies only very slightly with distance away from the dislocation line.[9] The distortion field then scatters electrons out where the Bragg condition is not locally satisfied. However, in the case of our films, where the defects are very close to the surface, the need for an accurate setting of a channeling-in condition is relaxed. This is possible since the lattice tilt due to the local strain field decreases inversely with distance away from the core. Hence the distortion should be greater and should show more variation with distance from the core in a thinner overlayer, along a direction parallel to the film surface. Such an effect was modeled quantitatively in Ref. 9. Therefore, in practice, only an approximate setting of the Bragg condition results in establishment of a channeling condition somewhere near the core. Strong contrast then becomes easy to attain and immediate qualitative information is at hand in cases where ECP's are not available.

A typical electron channeling contrast image from the $In_{0.25}Ga_{0.75}As$/GaAs(001) specimen is shown in Fig. 1. Note the alternating bright/dark bands. The different scale markers are due to the specimen tilt. This image shows clearly the nonuniform nature of the defect distribution. TEM observations on pieces of the same and similar thin film samples[10] indicated the presence of clusters consisting of up to three to five dislocations, spaced on average a few tens of nanometers apart. The dislocation lines were observed to lie nominally along the orthogonal $\langle 110 \rangle$ directions. The channeling contrast result, showing bright bands of maximum width 200 nm, also lying along $\langle 110 \rangle$ directions, is consistent with the maximum size of such clusters as seen by TEM. More often, narrower features were observed, with 80 to 100 nm a repeatable minimum observable image width. Such a width corresponds to the presence of perhaps two to three dislocations.

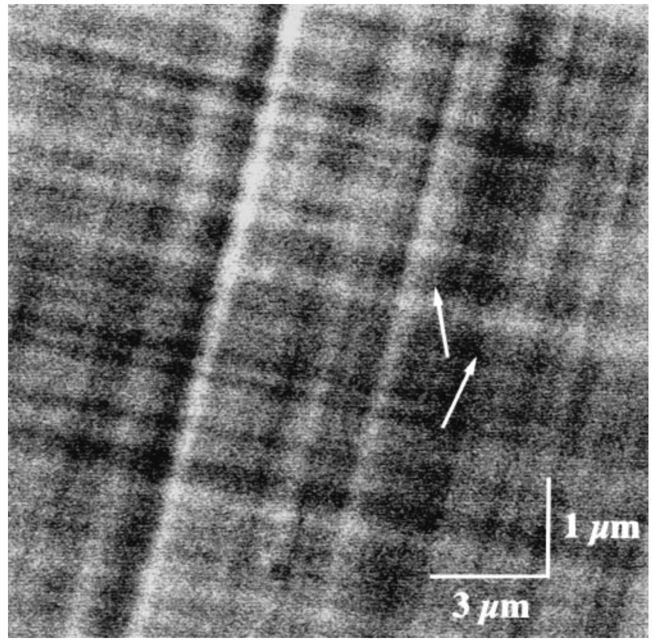

FIG. 1. Electron channeling contrast image showing clusters of possible threading segments, where dislocation line directions locally deviate from $\langle 110 \rangle$, as indicated by the arrows. Incident beam energy 30 keV, specimen tilt 70°.

The arrows in Fig. 1 show an interesting feature found fairly often in the channeling contrast images from dislocation clusters in $In_{0.25}Ga_{0.75}As$ on GaAs. Some of the lines locally deviate from the $\langle 110 \rangle$ type directions. The deviations occur over about 1 to 2 μm along the line directions and about 0.5 μm normal to the line directions. They are best viewed by looking at the figure along the direction of one set of lines, at a shallow angle. Such contrast implies that the displacement fields of this cluster of dislocations do not continue along the entire length of the sample. Two possible explanations include the presence of either large bowing dislocations in the film/substrate interface or clusters of threading segments. Bowing within the interface is not likely as the slip plane in the zincblende structure is not (001). Threading segment clusters are another possibility since these are often seen in relaxed alloys of this material.[11] We show in Fig. 2 schematics of two possible configurations of threading segment clusters. In both configurations, we

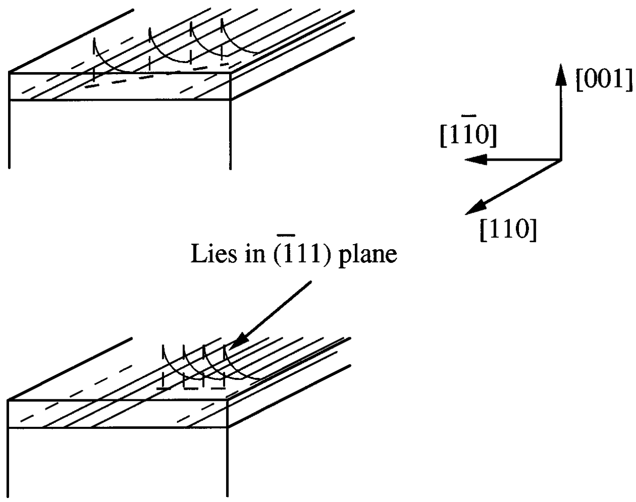

FIG. 2. Schematic illustrations of two possible threading segment clusters. The upper configuration would result in a more wedge-like region of bright intensity, as compared to the lower configuration.

consider clusters of four adjacent threading dislocations. We envision intensities as being bright where the lines and threading segments are situated. In the top case, the threading segments are displaced along the [110] direction, resulting in a wedge-shaped deviation from [110] as viewed along the [00$\bar{1}$] direction. The bottom case considers the four threading segments lined up perfectly along the [1$\bar{1}$0] direction, resulting in a more abrupt halt to the contrast along the [110] direction. Detailed transmission electron microscopy is necessary to confirm this.

In conclusion, electron channeling contrast has been used to image misfit dislocation strain fields in $In_{0.25}Ga_{0.75}As$ on GaAs(001) within a SEM, without further preparation of the as-grown films. A highly tilted specimen and a small solid angle of collection resulted in the detection of high spatial resolution contrast without the need for a field emission electron source. Dislocation displacement fields in very thin films require less stringent orientation conditions for producing strong contrast as compared to thicker films. We have observed features that seem to correspond to clusters of threading segments.

ACKNOWLEDGMENTS

The authors thank A. Dabiran for thin film preparation and J. Angelo, D. Joy, J. Mancuso, and D. Newbury for helpful discussion. Financial support was provided by the NIST Office of Microelectronics Programs and the National Research Council Post-Doctoral Research Associateship Program.

REFERENCES

1. G. R. Booker, A. M. B. Shaw, M. J. Whelan, and P. B. Hirsch, Philos. Mag. **16**, 1185 (1967).
2. D. R. Clarke, Philos. Mag. **24**, 973 (1971).
3. J. P. Spencer, C. J. Humphreys, and P. B. Hirsch, Philos. Mag. **26**, 193 (1972).
4. M. Pitaval, P. Morin, J. Baudry, E. Vicario, and G. Fontaine, in *SEM/1977: Proceedings of the Workshop on Analytical Electron Microscopy* (IITRI, Chicago, 1977), Vol. 1, p. 439.
5. P. Morin, M. Pitaval, D. Besnard, and G. Fontaine, Philos. Mag. A **40**, 511 (1979).
6. R. Sandström, J. F. Spencer, and C. J. Humphreys, J. Phys. D: Appl. Phys. **7**, 1030 (1974).
7. J. T. Czernuszka, N. J. Long, E. D. Boyes, and P. B. Hirsch, Philos. Mag. Lett. **62**, 227 (1990).
8. O. C. Wells, Scanning **2**, 199 (1979).
9. A. J. Wilkinson, G. R. Anstis, J. T. Czernuszka, N. J. Long, and P. B. Hirsch, Philos. Mag. A **68**, 59 (1993).
10. J. E. Angelo, Sandia Livermore National Laboratories, unpublished results.
11. K. H. Chang, R. Gibala, D. J. Srolovitz, P. K. Bhattacharya, and J. F. Mansfield in *Layered Structures—Heteroepitaxy, Superlattices, Strain, and Metastability,* edited by B. W. Dodson, L. J. Schowalter, J. E. Cunningham, and F. H. Pollak (Mater. Res. Soc. Symp. Proc. **160**, Pittsburgh, PA, 1990), p. 129.