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Characterization and Optimization of Semiconductor Specimen Preparation for QHREM

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The quality of TEM specimen is one of the crucial factors of successful electron microscopy, especially in the case of quantitative high-resolution electron microscopy (QHREM) on an atomic scale. The amorphous over-layers, the thickness of which is related to the conditions of preparation, limit the accuracy of the determination of the image maximum positions. In the present work the conditions of low-energy ion (LE) polishing on the basis of optimized specimen preparation of III-V semiconductors has been studied. The quality of the LE-polished specimens has been evaluated by QHREM using an image processing package LADIA (LAttice DIstortion Analysis).[1-3]

GaAs {110} cross-sectional specimens are prepared with an optimized process. This involves not only suitable parameters for ion-milling (3kV 10-12 μA before perforation) and ion-polishing (1.7kV 40min) with GATAN PIPS 691, but also precise knowledge of the specimen thickness before ion-milling, as well as perfect specimen surface finishing by multi-step grinding, dimpling, and polishing. [Higher gun-voltage, e.g. 3.5kV in the case of specimen D, without any ion-polishing leads to thick amorphous over-layers and defect formation, as pointed by an arrow in Fig.1(a), due to ion damage.] After HREM imaging, the specimens are further LE-polished with various ion energies and time duration (Table 1) using Technoorg Linda.

Fig.1 compares the HREM image taken after LE-polishing [Fig.1(b)] with that before LE-polishing [Fig.1(a)] of the same specimen D. Defects and thick amorphous layers are successfully removed, and the image quality is higher. Fig. 2 demonstrates HREM images of specimen C before and after LE-polishing of medium voltage and duration. Specimen quality is evaluated by QHREM. The distances between intensity maximum positions along [001] direction, d, are determined with a sampling rate of 70 pixel/nm [Fig.3(a)]. The width of the distribution of d (2σ) is used to estimate the accuracy of the measurements.[4] Fig.3(b) shows the histogram of d measured from Fig.2. The distribution obtained after LE-polishing is much narrower, indicating an improvement of the accuracy of the measurements. In the Table 1, the results from specimens LE-polished at various conditions are summarized and compared with those obtained before LE-polishing. The quality of specimens is markedly improved by choosing suitable parameters of LE-polishing. The standard deviation σ is reduced by a factor two and three in specimen C and D respectively. It should also be noted that for well ion-milled specimens (e.g., sample A) further LE-polishing might lead to degradation, and is therefore not necessary.

In summary, the preparation of cross-sectional TEM specimens of III-V semiconductors is optimized to meet the need of HREM. Further LE-polishing with proper parameters improves the results of QHREM up to a factor of two, when routine ion-milling alone is not satisfactory.

References

[1] N.Y. Jin-Phillipp and F. Phillipp, J. Microscopy 194 (1999) 161
[2] N.Y. Jin-Phillipp and F. Phillipp, J. Appl. Phys. 88 (2000) 710
[3] K. Du et al, J. Mat. Sci. Technol. 18 (2002), in press
[4] H. Seitz, et al, J. Microscopy 190 (1998) 184

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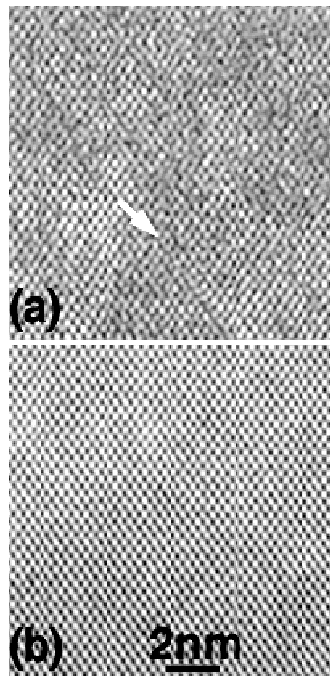
Table 1 Parameters and summary of QHREM results of LE-polishing

Specimen	Voltage (kV)	Duration (min)	σ_0 (nm)	σ_e (nm)	Result
A	0.25	20	-	-	more amorphous
B	0.25	40	0.0240	0.0188	fair
C	0.35	40	0.0207	0.0112	good
D	0.5	30	0.0407^+	0.0125	good
E*	0.5	60	0.0229	0.0222	fair

$^+$ Thick amorphous layers and defects due to ion-damage during ion-milling with 3.5kV in PIPS.
* Specimen E was polished with a reconstructed Baltec RES 010
σ_0 : standard deviation before LE-polishing, σ_e : standard deviation after LE-polishing.



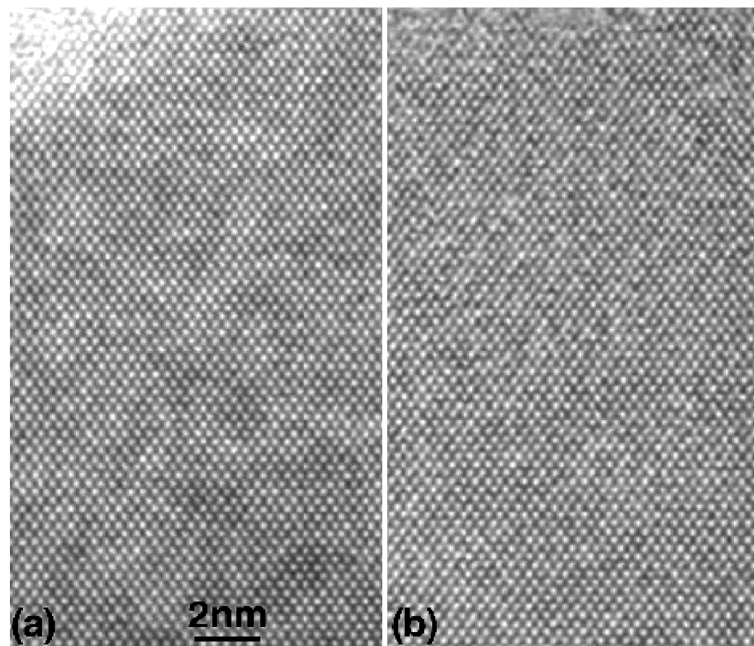


Fig1. HREM from specimen D. Fig.2 Images of specimen C before (a) and after (b) LE-polishing for QHREM.

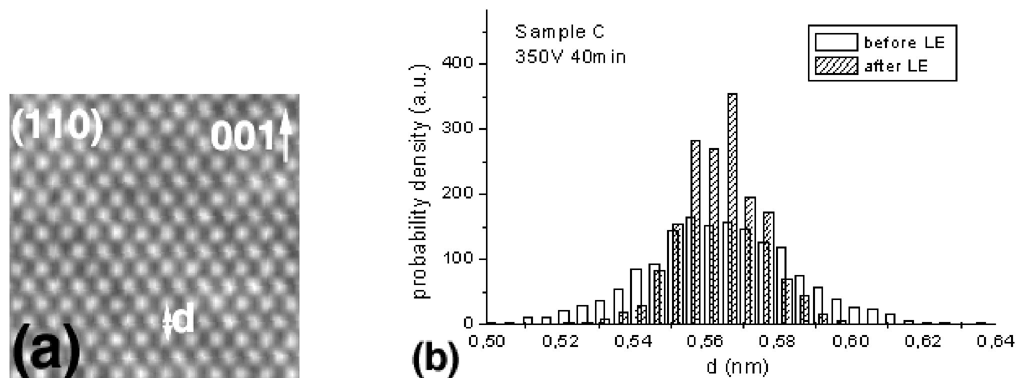


Fig.3 (a) Schematic of QHREM, (b) Distribution of *d* measured from specimen C.

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