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Defects Evaluation of Blue Light Emitting Materials by Wet Etching and Transmission Electron Microscopy

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Abstract

Evaluation of defects by etch-pit formation was studied. A NaOH(30 mol%) etchant was found useful for etch-pit development on ZnSe-based epilayers grown on (001) GaAs. And, a H_3PO_4(85 mol%) was used in order to develop etch-pits on GaN-based epilayers grown on (0001) Al_2O_3. After etch-pit formation on the surface, Transmission Electron Microscopy(TEM) was conducted. By etch-pit development and TEM observation, we could determine the defect types by etch-pit configurations and found origin of etch-pit in the case of ZnSe-based materials. Based upon these results, we can do defect identification by etch-pit test, simply. In the case of GaN-based materials, we could evaluate nanopipe density. However, high density of threading dislocations in GaN epilayers were not revealed by etch-pit development. Based upon these results, we can evaluate the nanopipe density which difficult to evaluate using TEM because of its small size(diameter). And at present status, direct matching of etch-pit density to dislocation density would make severe mistake.

KEYWORD: blue light emitting material, ZnSe, GaN, wet etching, etch-pit, defect, TEM.

1. Introduction

An etch-pit development test is simple and effective to assess the quality of an epilayer. This method is useful to determine the defect density. An etch-pit development test followed by TEM observation had been tried in order to resolve the origin of etch-pits and to determine the defect type. In this study, we studied the TEM observation of etch-pit developed samples and were able to investigate the origin of etch-pits of ZnSe-based and GaN-based blue light emitting semiconductor materials.

2. Experimental

A NaOH(30 mol%) solution was used in order to develop etch-pits on the ZnSe surface. The etching time was 10 - 15 seconds at the boiling temperature of the NaOH(30 mol%) solution, depending on the film thickness. The average etching rate was about 5.2 nm/sec. A H_3PO_4(85 mol%) solution was used in order to develop etch-pits on the GaN surface. The etching was done at 215 °C for 1 minute, and the average etching rate of GaN was about 4.5 nm/sec.

After etch-pit formation, we prepared plan-view samples for TEM observation. We carried out the TEM work with a Hitachi H-9000 operating at an acceleration voltage of 300kV.

3. Results and Discussion

3.1. ZnSe-based epilayers

The etch-pits have been formed in three different configurations; regularly paired etch-pits in the horizontal direction of the each ellipsoidal etch-pit, regularly paired etch-pits in the vertical direction of the each ellipsoidal etch-pit and an array of single etch-pits in the <110> and <100> directions which result from Frank-type stacking faults, Shockley-type stacking faults and threading dislocation segments, respectively[1). These results mean that we can do defect identification by etch-pit test.

For the Frank-type stacking faults, paired etch-pits had been formed on each of the two partial dislocations which bound the stacking fault not stacking fault plane. Our observation was that the etch-pits formed at the partial dislocations rather than at the stacking fault itself. This result appears reasonable, considering the low stacking fault energy in the ZnSe lattice. For the Shockley-type stacking faults, one etch-pit was formed at one set of partial dislocation and stacking fault plane. In this case, we can not determine whether the etch-pits form at the partial dislocation or at the stacking fault itself because the width of the stacking faulted area is too narrow compared with the etch-pit. For the threading dislocations, etch-pits were formed at end point of threading dislocation segment.

3.2. GaN-based epilayers

Hexagonal shaped etch-pits were formed at nanopies[2). However, in the case of high density of threading dislocations which regularly spaced and arranged in a granular shape, there were no etch-pits. These results mean that the evaluation of dislocation density by etch-pit would make mistake.

4. Conclusions

We could determine three different defect types of ZnSe-based epilayers grown on (001)GaAs by etch-pit configurations and found origin of etch-pit. Therefore, we could identify defect types by etch-pit test, simply.

In the case of GaN-based epilayers, we could evaluate the nanopipe density, accurately. And based upon our series of etch-pit experiments with various kinds of etchant[3), at present status, direct matching of the dislocation density to the etch-pit density would make severe mistakes in the case of GaN-based epilayers.

References

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