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Electron-Beam Induced Etching as a Key Process in Through-Vacuum Fabrication of GaAs-AlGaAs Nanoheterostructures

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Electron-beam (EB) induced etching of photo-oxidized GaAs layer by chlorine gas enables a novel *in situ* nanofabrication of GaAs-AlGaAs heterostructures where all the processings are performed in ultra-high vacuum (UHV) multichamber system. The features and underlying mechanism of this EBinduced etching are described.

1. Introduction

There has been a keen interest to impliment nm scale heterostructures in the future electronic and optoelectronic devices in order to improve the device performance substantially.1) For such purposes, the technology capable of fabricating fine structures in the nm scale and arbitrarily designed integrated structures is essential. A variety of attempts have been made to fabricate such nanostructures. For example, early in 80's Petroff et al. fabricated GaAs-AlGaAs quantum well-wire structures by the combination of the molecular beam epitaxy (MBE) and the photolithography²). Then, Kamon et al. reported the fabrication of the similar structures by selective-area organometallic vapor phase epitaxy.³⁾ Recently, many authors have tried to grow the quantum-wires or boxes using epitaxy on the prepatterned substrates with the aid of lithography.⁴⁾ That is, nm scale semiconductor heterostructures are fabricated by the combined use of the epitaxy controlled on the atomic scale and the nm scale lithography.

However, the materials to be dealt with here are compound semiconductors which are very sensitive to the atmosphere and the size accuracies demanded are in the order of nm. Therefore, it will be natural to consider the fabrication processes which are carried out under ultra-high vacuum (UHV) conditions without exposing specimens to the air in order to avoid the effects of the surface oxidation or contamination.⁵) Along this line, we have recently developed an electron-beam (EB)assisted lithographic method compatible with an all UHV processing of GaAs-AlGaAs system, which we call *in situ* electron-beam (EB) lithography.⁶)

In this paper, the electron-beam (EB) induced etching, the key process of the *in situ* EB-lithography, is described.

2. Through-Vacuum Processing

In *in situ* EB-lithography, an ultra-thin surface oxidized layer of GaAs is used as both the electron-sensitive resist film and the etching mask. This oxide layer can be patterned by the EB-induced etching and can be removed by heating. That is, the basic process of *in situ* EBlithography is the rocal removal of the surface oxide layer of GaAs by simultaneous irradiation of electron beam and chlorine gas.

As illustrated in Fig.1, the procedure of *in* situ EB-lithograhy comprises the following five steps: 1) Preparation of a clean GaAs by MBE. 2) Formation of the thin oxide layer as a resist film by photo-oxidation in the pure oxygen gas. 3) Patterning of the oxide layer by EB-induced etching and subsequent Cl₂ etching of GaAs as



Fig.1 Schematic illustration of in situ EB-lithography.

pattern transfer. 4) Removal of the surface oxide layer by heating under arsenic flux in the MBE chamber. 5) Overgrowth of epitaxial layers by MBE. All these processings are carried out thoroughly in a UHV system. This procedure can be repeated if necessary.

In this *in situ* EB-lithography, the key process is the EB-induced chlorine etching of GaAs oxide. In the following, features and underlying mechanism of EB-induced etching are reviewed.

3. EB-Induced Cl₂ Etching of GaAs Oxide

As reported previously, the properly formed GaAs oxide is resistant to the Cl2 gas etching. In Fig.2, an example of the etched depth by the simultaneous irradiation of electron-beam (EB) and chlorine gas is plotted against the electron dose. A steep rise of etching depth is seen around the electron dose of 3 x $10^{\overline{17}}$ cm⁻². This is the minimum EBdose to remove oxide layer completely. This nonlinear etching characteristics is favorable for practical lithography. Furthermore, it turned out that the electron-beam exposure and the chlorine etching can be separated. This means that the electron source can be installed in the separate chamber from the etching chamber. This will make easier the design of UHV system.

An example of the fine pattern of GaAs formed by this method is shown in Fig.3. The best resolution of patterns obtained up to now is about 20 nm, which is considered to be limited by the radius of the electron-beam used.

As the details of EB-induced patterning mechanism will be discussed in the following paper⁷), we will touch here only a phenomenon which some of the present authors have recently



Fig.2 Etching depth against the electron dose.

found to be responsible for etching. Ide and Yamada found by using x-ray photoelectron spectroscopy (XPS) that the chlorine gas adsorbs on the EB-irradiated photo-oxide of GaAs while only a very little adsorption of chlorine atoms is seen on the as-formed GaAs photo-oxides⁸). The electron-dose necessary for the adsorption of chlorine gas was in the same order of magnitude of the EB-dose for patterning. They found further that the irradiation of electron beam causes the change in the oxidized states of As and considered that the chlorine exposure triggers the preferential removal of the EB-induced As suboxides (or elemental As) in the forms of AsClx, and that this is the key of the patterning of the photooxide mask.

Next, we will discuss some practical aspects of the EB-induced etching. Fig.4 (a) and (b) compare the photoluminescence (PL) spectra of the *in situ* Cl₂ etched sample and the *ex situ* processed sample with the PL spectrum of an *in situ* grown reference sample. In

contrast to the ex situ processed sample, the in situ etched and regrown sample shows much better PL inensity, especially for PL intensities from the quantum-wells embedded near the etched surfaces. This fact demonstrates clearly the importance of the all UHV-processing or in situ processing.

As considerably high dose of electronbeam (EB) is used in the in situ EB-lithography, the damage generated during processing must be checked. The influence of the EB-irradiation was examined by photoluminescence (PL) measurements of the GaAs-AlGaAs quantumwells embedded at the different depth from the surfaces. The PL intensities did not change up to the dose level of 1×10^{18} electrons/cm² by the irradiation of 10 keV electron-beam. As seen previously the electron-dose necessary for in situ EB lithography is in the middle of 1 x 10^{17} electrons/cm². Thus the electron beam induced damage has been shown to be very small.

4. Summary

Electron-beam (EB) induced etching as a key process in an all ultra-high vacuum (UHV) processing of GaAs-AlGaAs system was described. Experimental results show that the EB-assisted in situ processing is a low-damage process and a promising candidate for the fabrication of nano-heterostructure in the future electronic and optoelectronic devices. However, as the nano-structures become smaller and smaller, the requirements for the interface properties will become more severe and more realistic characterizations will be needed.

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Fig.3 Examples of fine pattern fabricated by in situ EB-lithography.



Fig.4 PL spectra of MQW's grown on (a) ex situ and (b) in situ etched samples compared with a reference MQW sample.

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