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SOLID-STATE ELECTRONICS

Nanooxidation of $n$-$\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ with an Atomic Force Microscope

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Abstract—It is shown that the features formed when an InGaAs layer lattice-matched with the InP substrate is modified by an atomic force microscope are of the oxide nature. Various models of InGaAs nanooxidation by an atomic force microscope are considered. The conventional Cabrera–Mott model refined by introducing the space charge into the oxide is taken as the basic model of nanooxidation by an atomic force microscope. © 2002 MAIK “Nauka/Interperiodica”.

INTRODUCTION

The knowledge of local oxidation mechanisms and kinetics is of crucial importance for optimizing the oxidation process by means of scanning probe microscopes, in particular, atomic force microscopes (AFMs). AFM oxidation is used in the production of electron devices with feature sizes as small as tens of nanometers. Also, an understanding of the local oxidation mechanism and kinetics would provide the reliable control of the device performance.

In many works on AFM oxidation (see, e.g., [1–3]), it has been reported that the geometric parameters of the resulting structures depend on the AFM probe size. This means that the electric field has an effect on the process. The high voltage increases the height and the width of the oxide lines because of enhanced anodizing or ionic diffusion.

A number of mechanisms are responsible for nanooxidating the substrate surface with an AFM. They depend on the polarity of the applied voltage and the process environment. For silicon, the AFM surface modification can be described within two models. One is based on the hypothesis that the oxidation near the probe–surface contact is facilitated by the electric field in the presence of oxygen [4, 5]. In this case, the probe is positively biased. The other model considers the anodizing of the surface covered by a thin water film under the action of the negatively biased probe [6]. Anodizing of semiconductors may proceed in either an oxygen plasma [7] or an electrolytic solution [8, 9].

At present, researchers suggest two mechanisms of local oxidation with the negatively biased probe that work in parallel. These are anodizing through a thin water film adsorbed on the surface [10] and electric-field-enhanced oxidation, where the electric field promotes the diffusion of ionized water molecules through the native oxide [3].

Attempts [11] to treat probe oxidation in terms of the Cabrera–Mott field model have failed [12], since the actual oxide growth rates much differ from the values predicted for anodizing. Yet, the Cabrera–Mott model shows the best correlation with the experiment and the discrepancy between the growth rates can be viewed as resulting from nonperfect oxidation conditions due to factors not taken into account. Such factors may be (1) mechanical stresses arising in the oxide because of a large difference in the oxide and silicon volumes [1], (2) space charge induced immediately at the point of potential application, or (3) a change in the field strength in response to a change in the shape and/or in the conductivity of the probe. These changes may be related to the plasticity of conductive coatings covering the probe and/or to the oxidation of the probe material itself.

Comparative studies of the effect of the probe polarity on the local oxidation of silicon [4] and chromium [13] have shown that the oxidized lines (features) are distinct, i.e., high, if the AFM probe is negatively biased. This has played a crucial role in deciding on the probe polarity for the formation of oxides more than 5 nm high. Such a height is necessary for the direct patterning of tunnel barriers in quantum devices.

The need for a water film in local oxidation has been demonstrated [14] in support of the fact that local oxidation has much in common with electrochemical anodizing. The optimization of the air humidity to produce oxides of a height of more than 5 nm has been reported [15].

An important parameter that characterizes the oxide growth rate is the norm of oxide growth, or the increment of the oxide thickness per volt. For a number of modifiable materials, the growth norm has been determined from experiments on anodizing at the macro-
level, so that it may indicate to what extent the nanoprocess and the macroprocess correlate.

EXPERIMENT

The subject of investigation was the $n$-$\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ layer ($n = 5 \times 10^{18} \text{ cm}^{-3}$) MBE-grown on the semi-insulating InP substrate. The surface conditioning procedure and the experimental setup used in the experiments were described in [15].

First, we tested the potentialities for oxidizing the $n$-$\text{InGaAs}$ surface by applying a potential $V_{\text{tip}} = -5 \text{ V}$ to the tip when scanning a surface area of $100 \times 100 \text{ nm}^2$ in size. The scanning frequency and pitch were 4.3 Hz and 1 nm, respectively. After oxide dots and lines have been formed under various conditions, we studied the characteristics of AFM oxidation. The oxide dots were produced by applying a negative voltage from 1 to 10 V to the fixed tip for $t_{\text{ox}} = 60 \text{ s}$. The lines were produced by scanning a 0.2- to 2.0-$\mu\text{m}$-long segment with a negative voltage $V_{\text{tip}} = 5$–30 V applied to the tip. The scanning rate was 10–260 nm/s. The identification of the structures obtained was performed immediately after the oxidation by imaging them with the same oxidizing tips. We also tested the feasibility of transferring the oxidized pattern into the $\text{InGaAs}$ layer by etching the sample in an HF solution.

To reveal the basic laws of local AFM-assisted $\text{InGaAs}$ oxidation, the same sample was totally oxidized (anodized) in a glycol–water solution (GWS) of tartaric acid. The anodizing solution and the associated setup were described in [16]. A transparent glass test tube was filled with the GWS, and a positive (relative to the Pt electrode) voltage of 15–30 V was applied to the sample. The sample was illuminated by an incandescent lamp to saturate the semiconductor surface by charge carriers.

The solution was prepared as follows. Assuming that the thin water film covering the sample is a neutral medium, a 3% water solution of tartaric acid ($\text{C}_4\text{H}_6\text{O}_6$) was neutralized by ammonium hydroxide to provide pH 6. The resulting solution was then mixed with propylene glycol so that the glycol-to-water volume ratio was 1 : 2.

RESULTS AND DISCUSSION

1. AFM Oxidation and Etching

Figure 1a shows the AFM image of the oxidized $100 \times 100 \text{ nm}$ surface area when the tip was under a negative voltage of 5 V. The cross section $A$–$A'$ is depicted in Fig. 1b. The direction of the slow scanning was from left to right and from top down, as indicated by arrows in Fig. 1a. The size of the oxide island exceeds that of the scan area. The base of the island is 60 nm longer than the scan length. Moreover, the island is pentagonal (Fig. 1a) rather than rectangular, although the rectangular area was scanned. Finally, the height (thickness) of the oxide smoothly varies over the former half of the scan area, remaining almost constant (about 1.8 nm) over the latter half.

In the contact mode of AFM operation (Fig. 2), the range of the threshold voltage (2–11 V) was close to that (5–10 V) observed for the AFM oxidation of GaAs—the material that is similar to $\text{InGaAs}$ in composition [17, 18]. Such a spread in the threshold voltage can be related to one more factor essential for AFM oxidation: the condition of the tip during the oxidation. As follows from the experiments, the lowest threshold value, 2 V, corresponds to the early stage of scanning. After several tens of cycles including dot formation and imaging, the threshold voltage of oxidation rises to 10 V or higher. The stripping of the conductive layer from the AFM tip was corroborated by observing it in a scanning electron microscope (Fig. 3).

Figure 4a shows four lines oxidized by 15-fold scanning of the straight segment at the fixed frequency. In all the cases, the negative tip voltage was 5 V. The scan length was 0.2, 0.5, 1, and 2 $\mu\text{m}$ from right to left. In