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Composition and morphology of fluorinated anodic oxides on InAs (1 1 1)A surface

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ABSTRACT

The composition and morphology of fluorinated anodic oxide (FAO) films grown on InAs (1 1 1)A in alkaline aqueous (pH 11.5) and acid waterless (pH 1.5) electrolytes are studied by means of X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and high resolution transmission electron microscopy (HRTEM) in order to reveal the passivation mechanism of fluorine on the FAO/InAs(1 1 1)A interface. The formation of the highest oxidation form of As^{+5} and passivation of defects in the FAO layers during the fluorination process explain the reduction of the density of surface states and unpinning of the Fermi level on the fluorinated AO/InAs(1 1 1)A interface.

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1. Introduction

The formation of III–V semiconductor/SiO_2 interface with low density of surface states plays a crucial role in device fabrication based on the metal-insulator-semiconductor (MIS) structures. The native oxide of complex composition between semiconductor and SiO_2 leads to a large density of surface states (N_{ss}) at the interface and to the Fermi level pinning, which is not compatible with MIS device operation [1,2]. The improvement of the interface properties in the InAs MIS structures was achieved by the formation of fluorinated anodic oxide (FAO) layer on InAs (1 1 1)A [3]. The effective passivation of InAs by thin FAO layer (10–15 nm), before deposition of SiO_2 layers, allowed to fabricate the In_2O_3/SiO_2/InAs MIS structure with $N_{ss} \leq 5 \cdot 10^{10}$ cm^{-2} eV^{-1} [4]. The voltage of the flat band and the breakdown electric field were achieved 3–5 V and $\sim 6 \times 10^6$ V cm^{-1} at $T = 77$ K, respectively. The improvement of the interface electrical properties allowed to fabricate a medical thermovision camera with the high-contrast images of two-dimensional thermal patterns with a resolution up to 7 mK [5].

In spite of the technological success in the interface formation with low N_{ss}, the mechanism of passivation of anodic oxide/InAs(1 1 1)A interface by fluorine is still unknown. In this paper the surface morphology and the phase composition of anodic oxide (AO) films grown on InAs(1 1 1)A in alkaline aqueous (pH 11.5) and acid waterless (pH 1.5) electrolytes with addition of NH_4F were studied by atomic force microscopy and X-ray photoelectron spectroscopy in order to elucidate the mechanism of FAO/InAs interface passivation.

2. Experimental

Commercially available InAs(1 1 1)A epitaxial layers on n-type ($1–5 \times 10^{15}$ cm^{-3}) substrates were used. Before oxidation, the samples were degreased in toluene, then treated in the solution of monoethanolamine with hydrogen peroxide (MEA:H_2O_2 = 1:1), washed in deionised water and dried by pure argon. Anodic oxidation was performed at room temperature in a cell with two electrodes in galvanostatic mode at current density 0.25 mA/cm^2 for alkaline electrolyte (NH_4OH:ethylene glycol = 1:5) and 0.1 mA/cm^2 for acid electrolyte (H_3PO_4:isopropanol:glycerin = 5:65:30) adding NH_4F. Indium arsenide specimens were anodized to thickness ~5 and 10 nm measured by ellipsometry methods. The thickness of anodic films was determined by the formation voltage, which was

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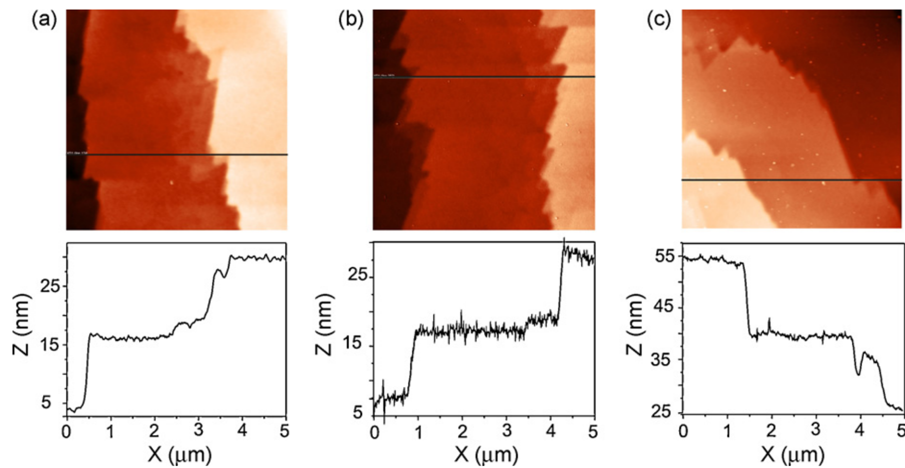

Fig. 1. AFM image of InAs(1 1 1)A surface before (a), after anodic oxidation in fluorinated alkaline electrolyte (b) and after removal of oxide layer in HCl-iPA solution (c).

calculated from the growth rate constant (2.5 and 3.6 nm/V for alkaline and acid electrolytes, respectively). Thickness measurements were performed using ellipsometer LEF-3 M with 632.8 nm wavelength at incidence angle equal to 55 degree. The thickness of AO films was calculated in the model of uniform isotropic film on a substrate with complex refractive index N_{sub} = 3.898–0.683j. Surface morphology was studied by AFM (Solver P-47H) in semi-contact topographic regime. TEM investigations were carried out on JEM-4000EX transmission electron microscope at 200 kV accelerating voltage. XPS spectra were recorded using SPECS spectrometer with PHOIBOS-150-MCD-9 hemispherical energy analyzer and X-ray monochromator FOCUS-500 (AlKα irradiation, $h\nu$ = 1486.74 eV, 200 W). Binding energy scale was preliminarily calibrated by the position of the peaks of Au4$f_{7/2}$ (84.0 eV) and Cu2$p_{3/2}$ (932.67 eV) core levels. The binding energy (BE) and a full width at half maximum (FWHM) are reported with accuracy of 0.05 eV. The spectral regions of In4d, As3d, C1s, In3d, O1s, F1s and As2p have been recorded. The XPS spectra of the studied samples were compared to XPS spectra of reference samples. The ratio of surface atomic concentrations of the elements was calculated from the integral intensities of photoelectron peaks corrected by corresponding atomic sensitivity factors (ASF). Depth profiling of the samples was carried out using an argon ion gun (SPECS model IQE 11/35). The energy of Ar^+ ions for depth profiling, the current density, the angle of sputter erosion were 2, 5 keV, 8 μA/cm^2 and 45°, respectively.

3. Results and discussion

3.1. Morphology and structure of the anodic oxide/InAs(1 1 1)A interface

AFM studies of the InAs(1 1 1)A epitaxial layer surfaces show that anodic oxidation preserved the flatness of the surface, keeping the mean roughness on the terraces at a level of approximately 0.3–0.5 nm (Fig. 1a and b). To determine the roughness of the interface between anodic oxide layer and InAs(1 1 1)A surface, the AO was removed by the treatment in HCl-isopropanol (iPA) solution. This treatment removes oxides without etching of InAs itself and covers the surface by several monolayers of elemental arsenic [6]. It is seen that morphology does not change after removal of AO layer by HCl-iPA solution (Fig. 1c). HRTEM demonstrates that the oxide layer is amorphous and the interface roughness is of the order of several monolayers (Fig. 2).

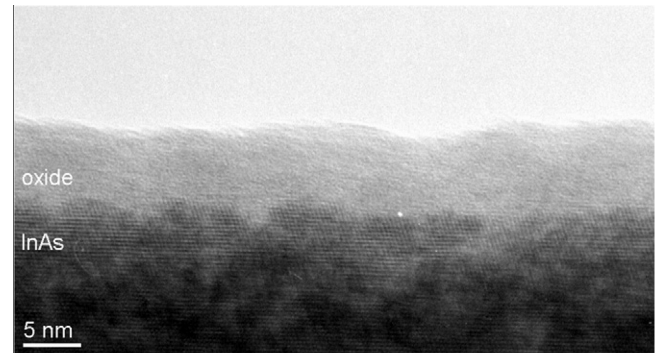

Fig. 2. Cross section of InAs(1 1 1)A/fluorinated alkaline oxide interface measured by HRTEM.

3.2. Composition of anodic oxide/InAs(1 1 1)A interface

Fig. 3 shows the XPS spectra of the bulk sensitive In4d and As3d core level spectra of AO layers grown on InAs(1 1 1)A surface by anodic oxidation in fluorinated acid (a) and acid electrolytes (b), anodic oxidation in fluorinated alkaline (c) and alkaline electrolytes (d), and clean InAs(1 1 1)A surface prepared by ion treatment (e).

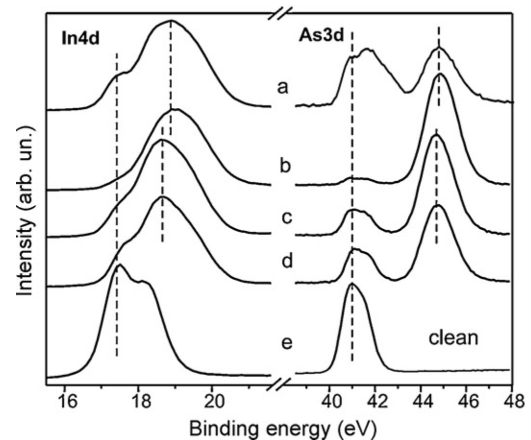

Fig. 3. XPS spectra of the bulk sensitive In4d and As3d CLS of AO layers grown on InAs (1 1 1)A surfaces by anodic oxidation in fluorinated acid (a) and acid (b) electrolytes, fluorinated alkaline (c) and alkaline (d) electrolytes, and clean InAs(1 1 1)A surface prepared by ion treatment (e).

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