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Role of Pb for Ag growth on H-passivated Si(1 0 0) surfaces

S. Mathew, B. Satpati, B. Joseph, B.N. Dev*

Institute of Physics, Sachivalaya Marg, Bhubaneswar 751005, India

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Abstract

We have deposited Ag on hydrogen passivated Si(1 0 0) surfaces under high vacuum conditions at room temperature. The deposition, followed by annealing at 250 °C for 30 min, produced silver islands of an average lateral size 36 ± 14 nm. Depositing a small amount of Pb prior to Ag deposition reduced the average island size to 14 ± 5 nm. A small amount of Pb, initially present at the Ag–Si interface, is found to be segregating to the surface of Ag after annealing. Both these aspects, namely, reduction of the island size and Pb floating on the Ag surface conform to the surfactant action of Pb. Samples have been characterized by transmission electron microscopy (TEM) and Rutherford backscattering spectroscopy (RBS). A selective etching process that preferentially removes Pb, in conjunction with RBS, was used to detect surface segregation of Pb involving depth scales below the resolution of conventional RBS. The annealing and etching process leaves only smaller Ag islands on the surface with complete removal of Pb. Ag growth in the presence of Pb leads to smaller Ag islands with a narrower size distribution.

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1. Introduction

The study of growth of metal thin films on semiconductor substrates is important from the fundamental as well as the technological point of view. Semiconductor technology requires a multitude of metal films for contacts, diodes and interconnections. The structure and morphology of the film has a significant influence on the reliability of the devices.

There are many reports [1,2] in the literature on the growth of Ag on Si substrates. However, new interesting features are still being revealed. Recent observations of growth of islands of quantized heights for Ag-on-Si [2] and Pb-on-Si [3] have generated a renewed interest in these system. In most of the studies the final substrate cleaning is done by in situ thermal desorption of oxide from the Si surface under ultrahigh vacuum (UHV) condition. Some Ag growth

* Corresponding author. Tel.: +91 674 2301058; fax: +91 674 2300142.
E-mail address: bhupen@iopb.res.in (B.N. Dev).

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studies have been conducted on chemically passivated Si substrates, where chemical passivation has usually been performed by adsorbing monovalent species like H [4,5] and Br [6]. Chemically terminating the Si surface with hydrogen eliminates the impurity diffusion during substrate cleaning. Some desired impurity overlayers can promote epitaxial growth [7] by changing the arrangement of substrate atoms, mobility of deposited atoms or the energetics of the growth process. Growth via an impurity surface layer has been termed 'surfactant-mediated' growth. Thin (submonolayer to a monolayer) layers of some typical elements, e.g. Sb, Pb, As, Te, In, etc., have been used as surfactants in epitaxial growth [8].

There are several reports on the surfactant action of Pb for homoepitaxy, for example Si growth on Si [9], and GaAs growth on GaAs [10]. Scanning tunneling microscopy (STM) studies of Sleradzki et al. [11] indicated that layer-by-layer growth can be achieved for Ag films (up to 200 monolayer thickness) on Au(1 1 1) surfaces using one monolayer (ML) of Pb as a surfactant. Wang et al. showed that a ML of Pb promotes layer-by-layer growth of ultra thin films of Ag on Au(1 1 1) surface [12]. Here we present transmission electron microscopy (TEM) and Rutherford backscattering spectrometry (RBS) studies of Ag growth on hydrogen-passivated Si(1 0 0) [hereafter H–Si(1 0 0)] surfaces with and without depositing a small amount of Pb prior to Ag deposition. In our high-vacuum deposition experiment we explore two aspects of surfactant mediated growth, namely, surfactant material floating on the surface of the deposited layer and the alteration of island size via the modification of growth kinetics. The size distribution is studied by TEM. The segregation of Pb to the Ag surface is investigated by a chemical etching process along with RBS.

2. Experimental

Hydrogen passivation of a Si(1 0 0) surface involves the following steps [4,5]: First step involves degreasing the Si wafers in acetone, methanol and deionised water (18.2 M Ω cm). After degreasing, the native oxide is etched by buffered HF (7:1 mixture of NH_4F:HF) for 2 min. Next a uniform oxide is produced by dipping the sample in a 4:1:1 mixture of H_2O:H_2O_2:HCl at 80 °C for about 10 min. The final etching and termination of the dangling bonds with hydrogen is achieved by keeping the sample in 40% NH_4F solution for 6 min.

The passivated samples [H–Si(1 0 0)] were loaded into a high vacuum chamber and deposition was performed by evaporating the materials from tungsten baskets. The pressure during deposition was maintained at 4×10^{-6} mbar. A quartz thickness monitor was used to monitor the thickness of the deposited layer. A set of samples were prepared only with Ag deposition [Ag/H–Si(1 0 0)]. For the case of Ag/Pb/H–Si(1 0 0) samples, silver was deposited following lead deposition on H–Si(1 0 0), without breaking the vacuum of the high vacuum chamber, by using two separate tungsten baskets. The samples were taken out and annealing (250 °C for 30 min) was done in another chamber at a pressure of 6×10^{-7} mbar.

Plan view and cross-sectional TEM measurements were carried out using a 200 kV (JEOL 2010) high resolution transmission electron microscope (HRTEM) with point to point resolution of 0.19 nm and lattice resolution of 0.14 nm.

RBS measurements were made at the 3 MV 9SDH2 tandem Pelletron accelerator facility in our institute. A beam of 3 MeV He^{2+} ions was used in the present study. The backscattered ions were detected by a surface barrier detector kept at a scattering angle of 160°. During the experiment a pressure of 2×10^{-6} mbar was maintained in the scattering chamber. RBS spectra were simulated by GISA3.9 [13].

We performed the following chemical etching procedure to detect the amount of Pb segregated to the Ag film surface after annealing. The as-deposited and the annealed samples were dipped in 10% HCl solution for 30 s. Then the samples were kept in hot water (80 °C) for 1 min so that $PbCl_2$, formed with Pb at the surface of the Ag film or with otherwise exposed Pb, would be dissolved and removed from the surface. RBS measurements were performed before and after this chemical treatment to determine the loss of Pb.

3. Results and discussions

3.1. Ag/H–Si(1 0 0)

Ag deposition on H–Si(1 0 0) surfaces leads to the growth of Ag islands. A typical plan view TEM