Micron 58 (2014) 32–37

Contents lists available at ScienceDirect

Micron

journal homepage: www.elsevier.com/locate/micron

Study of vertical Si/SiO_2 interface using laser-assisted atom probe tomography and transmission electron microscopy

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ARTICLE INFO

Article history:
Received 5 August 2013
Received in revised form
13 November 2013
Accepted 16 November 2013

Keywords:
Atom probe
Shallow trench isolation
Preferential evaporation
Interface reaction

ABSTRACT

Laser-assisted atom probe tomography has opened the way to three-dimensional visualization of nanostructures. However, many questions related to the laser–matter interaction remain unresolved. We demonstrate that the interface reaction can be activated by laser-assisted field evaporation and affects the quantification of the interfacial composition. At a vertical interface between Si and SiO_2, a SiO_2 molecule tends to combine with a Si atom and evaporate as a SiO molecule, reducing the evaporation field. The features of the reaction depend on the direction of the laser illumination and the inner structure of tip. A high concentration of SiO is observed at a vertical interface between Si and SiO_2 when the Si column is positioned at the center of the tip, whereas no significant SiO is detected when the SiO_2 layer is at the center. The difference in the interfacial compositions of two samples was due to preferential evaporation of the Si layer. This was explained using transmission electron microscopy observations before and after atom probe experiments.

1. Introduction

The quality of the interface in semiconductor devices critically influences the device performance (Cavin et al., 2000; Thompson and Parthasarathy, 2006), so there is great interest in interpreting the interfacial composition at the atomic scale. Atom probe tomography (APT) has received a great deal of attention from semiconductor researchers for its three-dimensional visualization of nanostructures and its ppm-level sensitivity (Kelly et al., 2007); it is easily adaptable to the analysis of semiconductor devices, which are entering the 10-nm-level design rule regime and are beginning to employ 3D structures. However, despite the recent application of femtosecond pulsed lasers (Gilbert et al., 2011), care must be taken to ensure that the interpretation of the interfacial composition is reliable. Heterogeneous structures reportedly raise unavoidable artifacts in the reconstruction of the AP data, and various reasons for these artifacts have been suggested (Oberdorfer and Schmitz, 2011; Vurpillot et al., 2000; Larson et al., 2010). First, exposing the structure to a laser pulse to assist field evaporation causes asymmetric evaporation of atoms with respect to the laser direction (Marquis et al., 2011a; Sha et al., 2008). When the thermal diffusion length is thinner than the tip diameter, the thermal distribution of the tip becomes anisotropic, which induces an alteration in the shape of the tip (Bunton et al., 2007). In oxides, which have lower thermal diffusivity than metals, severe preferential evaporation occurs on the side illuminated by the laser (Müller et al., 2011). Moreover, the laser exposure changes the nature of multiple events, so the compositional information varies with the position with respect to the laser (Müller et al., 2012). Second, the differences in the evaporation field among the layers also cause the surface curvatures of layers to change from the initial statuses (Marquis et al., 2011a). This continuous evolution of the tip shape results in local magnification and disordering of the field evaporation sequence (Oberdorfer and Schmitz, 2011). As a result, the atomic concentration across the interface cannot be quantitatively measured when the evaporation field differs greatly. Third, the dependence of the ion trajectory on the crystallographic orientation has been widely recognized since field ion microscopy was initially used (Gault et al., 2010a; Miller et al., 1989). The difference in the evaporation field depending on the crystallographic orientation leads to local changes in the radius of curvature of the specimen and to local magnification (Gault et al., 2010a). These phenomena can be observed by transmission electron microscopy (TEM). When a SiO_2 layer on a Si substrate is evaporating, the lower Si substrate evaporates before the upper SiO_2 layer owing to the low evaporation field of Si. Fig. 1(a)–(c) are TEM images taken after laser-assisted field evaporation. In Fig. 1(a), the red dotted areas are preferentially evaporated regions of the

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http://dx.doi.org/10.1016/j.micron.2013.11.003

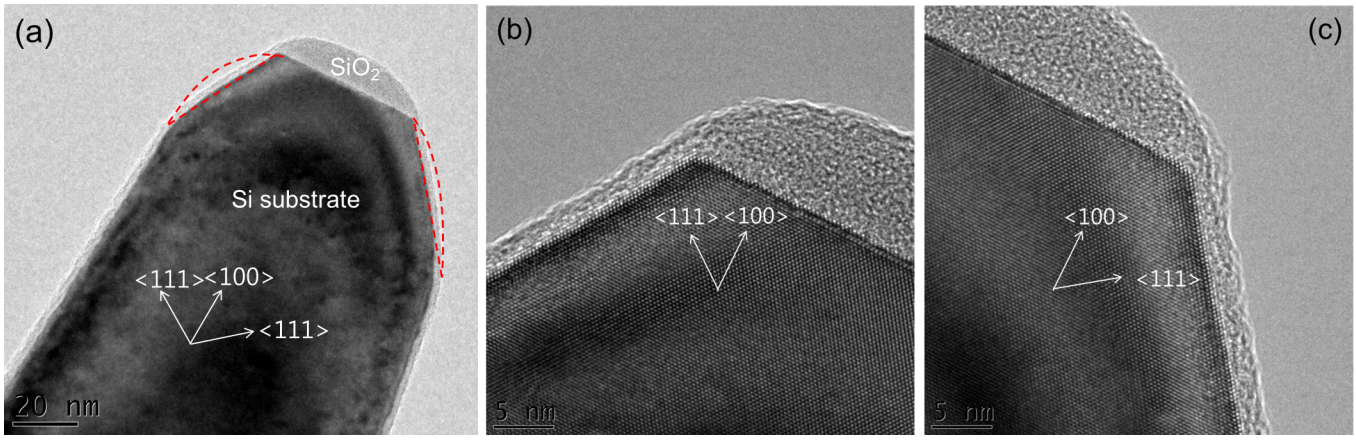

Fig. 1. (a) TEM image of Si/SiO_2 (100 nm) multilayered sample after AP experiment. TEM image was recorded during the (temporarily paused) AP experiment. Pulsed laser illumination came from the right side. The red dotted area represents the preferentially evaporated Si area, which formed a flat surface in the (1 1 1) plane (the close-packed plane). High-resolution images of (b) left and (c) right sides. The native oxide was grown on the Si surface because of the strong electron beam and vacuum breaking. The preferentially evaporated Si surface formed a (1 1 1) facet. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Si substrate. The laser illumination came from the right side of the tip in the TEM image, and this is evident from a comparison of the radii of curvature on the left and right sides. Because the right side was exposed directly to the pulsed laser, the radius of curvature there is larger than that on the left side. Additionally, the facet of the (1 1 1) surface exposed at the Si layer means that the field evaporation is strongly related to the crystallographic orientation.

Fourth, surface migration of the atoms on the tip surface degrades the lateral resolution (Kellogg et al., 1978; Gault et al., 2010b). Early research demonstrated the wandering of surface atoms under nanosecond laser pulses (Gault et al., 2006). If surface migration is possible in laser-assisted APT (Gault et al., 2012; Cerezo et al., 2007), we can assume that chemical reactions requiring low activation energy could also be activated. Because these reactions would also disturb the compositional quantification, this aspect of the treatment should be considered. In this paper, we will demonstrate the interface reaction at the Si/SiO_2 interface and show that it depends on the inner structure of the tip by using TEM observations.

2. Experimental

Shallow trench isolation (STI) was chosen for the interface analysis [Fig. 2(a)]. The STI structure is commonly used to isolate neighboring cells. The STI region is filled with SiO_2 by chemical vapor deposition after a dry etch process. The width of the Si column and the depth of the STI are 25 nm and 250 nm, respectively. Two types of sample from the same structure were prepared using a focused ion beam. The Si column is centered in sample A, and the SiO_2 STI is centered in sample B, as shown in Fig. 2(a). JEM-2100F was used for TEM observation. The APT analysis was performed by LAWATAP (laser-assisted wide-angle tomographic atom probe) developed by CAMECA. A 343-nm, 100-kHz UV laser was set to 0.09 μJ/pulse at 30 K (spot size ~30 μm). The UV laser was illuminated perpendicular to the Si column [from the left or right direction in Fig. 2(a)]. The standing voltage was controlled automatically with respect to the detection flux in order to acquire AP data at the AP image. It provides three reconstruction algorithms: the voltage-based approximation, cone-shape approximation, and tip-shape based approximation. Among them, the tip-shape-based

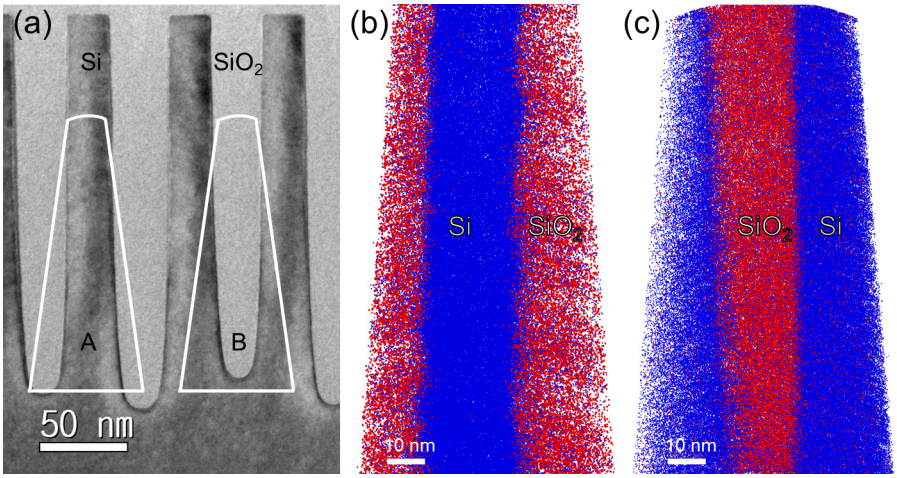

Fig. 2. (a) TEM image of shallow trench isolation (STI). The width of the SiO_2 layer is around 30 nm, and the depth of the STI is around 250 nm. (b) Side view of AP image from area A in (a). (c) Side view of AP image from area B in (a). Blue indicates Si; red indicates SiO_2. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)