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Engineering the size and density of silicon agglomerates by controlling the initial surface carbonated contamination

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ABSTRACT

Actually, thermally induced thin-films dewetting silicon in the silicon-on-insulator is a way to obtain silicon agglomerates with a size and a density fixed by the silicon film thickness. In this paper we report a new method to monitor both the size and the density of the Si agglomerates thanks to the deposition of a carbon-like layer. We show that using a 5-nm thick layer of silicon and additional ≤1-nm carbonated layer; we obtain agglomerates sizes ranging from 35 nm to 60 nm with respectively an agglomerate density ranging from 38 μm^{-2} to 18 μm^{-2}. Additionally, for the case of strained silicon films an alternative dewetting mechanism can be induced by monitoring the chemical composition of the sample surface.

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1. Introduction

Dewetting of ultrathin silicon-on-insulator (SOI) under high temperature is a promising method to obtain crystalline silicon agglomerates organized and tunable in size [1,2]. As pointed out by Nuryadi et al. [3], such silicon islands are self-organized along the crystallographic directions of the Si layer after dewetting [4]. This observation has fostered further studies of thermal agglomeration of single-crystalline silicon. For example, strained-silicon layer [6] and/or thinner silicon layer [3,7] have been used to control the size of the silicon agglomerates. Moreover, it has been reported that the presence of impurities on the sample surface can greatly influence the morphology of silicon agglomerates [8,9]. To describe SOI agglomeration, Danielson et al. [5] proposes a kinetic model, based on a surface-energy-driven dewetting phenomenon. This model consists of five steps: (i) critical void formation; (ii) void edge thickening; (iii) void edge breakdown; (iv) void finger formation and growth; (v) islands formation. In this paper we report a new method to monitor both the size and the density of the Si agglomerates thanks to presence of native carbon or the deposition of a carbon-like layer. In addition, for the case of strain silicon film, we show that an alternative dewetting mechanism can be induced by monitoring the chemical composition of the surface of the sample. The dewetting mechanism is studied by atomic force microscopy (AFM).

2. Experimental technique

In this work, thin monocrystalline silicon films of SOI and strained silicon on insulator (sSOI) samples from SOITEC are used. These films are prepared using the Smart-cut® process with a 140-nm thick buried silicon oxide and around 15-nm silicon film [19]. As for the sSOI sample, the silicon film is bi-axially strained at 1.6%. [10]

As the kinetic of the dewetting strongly depends on the silicon film thickness [20], thin silicon layers are used to reduce the dewetting time. For this purpose the upper silicon layer of both SOI and sSOI are thinned by forming a silicon oxide at high temperature and then by etching this oxide away using hydrofluoric acid (HF). The resulting thicknesses of the silicon films are estimated by ellipsometry with an accuracy of ±0.25 nm; they are equal to 5 nm for the SOI sample and 11 nm for the sSOI one. Arguably this thickness difference comes from the dependence of the etching rate on the strain in the silicon film. Nevertheless, this difference in silicon thickness has no effect on the results as the study of the surface carbonated contamination effect on dewetting are conducted independently on SOI and on sSOI.

The control of the surface carbonated contamination is obtained using two methods. The first method consists in reducing the content of native silicon dioxide and carbon contamination by successively etching away the organic contamination using a piranha

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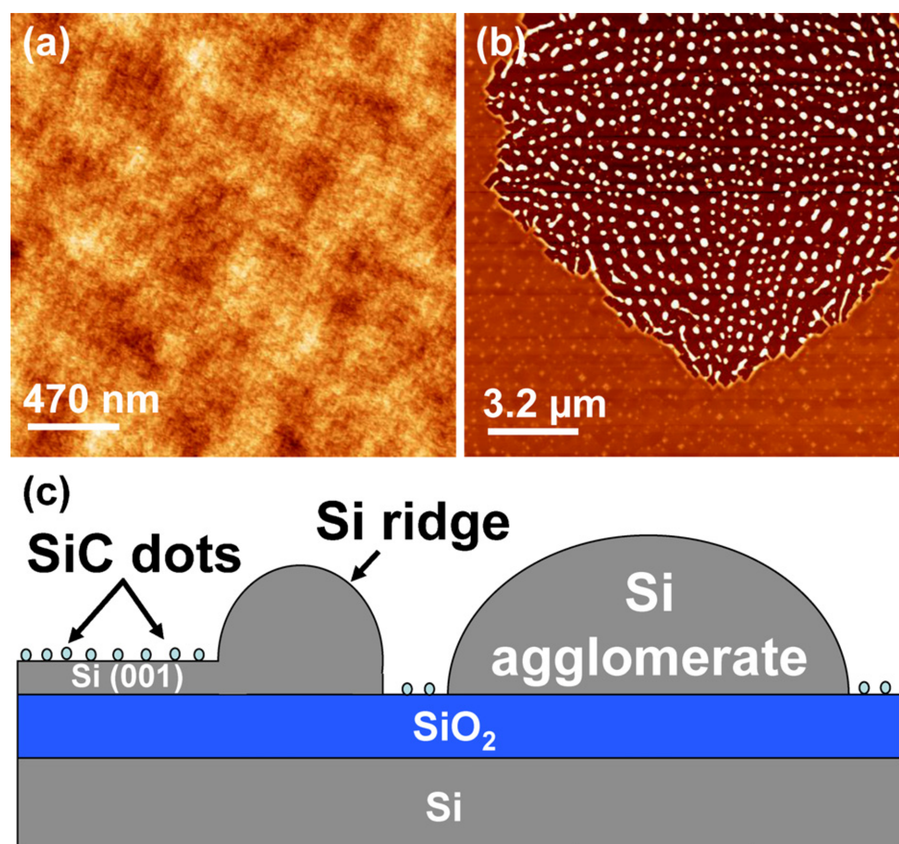

Fig. 1. (a) Topography of SOI sample before dewetting (3 nm *z*-scale). (b) AFM image (100 nm *z*-scale) and (c) scheme of partially dewetted SOI film showing the Si agglomerates and SiC dots.

solution [H_2SO_4/H_2O_2 mixture (3 to 2)] during 20 min at 140 °C and then, by etching away the silicon dioxide using a buffered oxide etch (BOE) solution [HF/NH_4F mixture (6 to 1)] at room temperature during 20 s. The second method consists in forming, in a scanning electron microscope (SEM), a carbon layer through an organic contamination decomposition induced by the SEM's electron beam. A beam current of 0.58 μA and a beam voltage of 15 keV are used. Four different carbon layer thicknesses are obtained by irradiating four 10 μm × 10 μm areas of the silicon surface during respectively: 30 s, 60 s, 120 s and 300 s.

The dewetting of samples is thermally induced under ultrahigh vacuum (UHV) condition (1×10^{-10} mbar) in an Omicron system. The sample size (5 mm × 10 mm) is large enough, compared to the silicon film thickness, to avoid sample edge effects. Dewetting is obtained by heating the samples during typically 10 min at a temperature ranging between 700 °C and 1000 °C. The temperature has been calibrated on a bulk silicon surface using both a pyrometer and a Pt/Rh thermocouple. Dewetting kinetics are temperature controlled: the higher the temperature the shorter the dewetting time.

Morphological characterization is done using AFM after dewetting, at room temperature. The AFM observations are performed using a Multimode microscope from Bruker operating in Tapping^{TM} mode [11] with a tip apex <10 nm (Tap300Al-G from Budget Sensors).

3. Results and discussion

As reported in the literature [1,3,7,15–17], the silicon agglomerates formation is induced by the thermal instability of the silicon film owing to a high surface tension. As an example, typical images SOI sample before and after dewetting under UHV are presented in Fig. 1(a) and (b). As shown, two kinds of structures are clearly visible after the dewetting process: (i) silicon agglomerates on SiO_2 induced by the dewetting process and (ii) small dots observable in the entire image. These small dots have already been observed by other groups [5,7] however, no information is given about their chemical composition and their role in the dewetting process. The presence of silicon carbide in the dots is confirmed by scanning Auger microscopy measurement. Due to the shape of the Auger C KLL peak (not shown here, see ref. [14]) the chemical nature of these dots can be ascribed to silicon carbide (SiC). The formation of SiC dots occurs, through the reaction [12,13] $SiO_2 + 3C \rightarrow SiC + 2CO$ between the native oxide and the carbon contamination present on the silicon surface. As a result, Fig. 1(c) illustrates the resulting silicon film dewetting in presence of carbonated contamination.

In the following, the effect of the SiC dots, induced by the presence of carbon, on the dewetting process is presented using the two protocols previously described. Firstly, dewetting on reduced carbon contaminated SOI and sSOI surfaces is studied; secondly, dewetting on controlled carbon contaminated SOI surfaces is presented.

3.1. *Chemical treatment on SOI surface*

The influence of the chemical cleaning treatment on the SOI surface morphology after heating is presented in Fig. 2(a) and (b). Without chemical treatment, the surface density of SiC dots is typically less than 1 μm^{-2}, with a height from 5 nm to 20 nm. After chemical treatment, the surface density of dots is about 500 μm^{-2}, with a height ranging from 1 nm to 4 nm. The chemical composition of dots observed after chemical treatment does not have importance in this study and needs to be more investigated. As dots are always observed, we conclude that the chemical recipe

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