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Regular surface patterns by local swelling induced by He implantation into silicon through nanosphere lithography masks

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ABSTRACT

Nanopatterning of silicon surfaces by means of He^+ ion implantation through self-organized colloidal masks is reported for the first time. Nanosphere lithography (NSL) masks with mask openings of 46–230 nm width were deposited on Si(100) wafers. He^+ ions were implanted through these masks in order to induce a local cavity formation and Si surface swelling. The surface morphology and the subsurface structure were studied using atomic force microscopy (AFM) and cross-sectional transmission electron microscopy (XTEM), respectively, as a function of mask and implantation parameters. It is demonstrated that regular arrays of both individual hillocks and trough-like circular rings can be generated.

1. Introduction

The implantation of light ions such as He^+ or H^+ is known to lead to the formation of bubbles and cavities in numerous materials, which upon coalescence frequently results in the exfoliation of thin surface layers [1]. In semiconductors, this effect is exploited to split off thin surface layers for subsequent transfer of the surface film on other materials substrates, whereby the thickness of the transferred film can be well controlled by adjusting the ion energy.

It is generally accepted that the cavity formation process is initiated by the capture of He atoms at vacancies, leading to the nucleation and growth of He bubbles and subsequent He release at a later stage, depending on the thermal history during the process. It is obvious that the formation of large cavities must be accompanied by (a) the emission Si self-interstitials Si_I and (b) by the swelling of the Si surface. Although the cavity formation process has been studied in much detail, relatively little is known about the accompanying swelling process [2,3].

In this paper, it is investigated whether a local swelling of a silicon surface can be achieved if the implantation of He is performed in surface areas restricted to a few ten nanometers. To this end, He^+ implantations are performed into silicon wafers covered with a self-organized nanosphere lithography (NSL) mask exhibiting a regular array of nanometric mask openings. This should enable the formation of regular morphological patterns on the Si surface consisting of individual hillocks, the size and position of which is controlled by the NSL mask openings and with heights determined by the implantation parameters. Such nanopatterned surfaces could be useful as substrates on which various small features can be created by subsequent processing steps.

2. Experimental

Czrochalski-grown Si(100) wafers were cleaned by an RCA type cleaning process and subsequently covered with NSL nanomasks consisting of 200, 600 or 1000 nm diameter polystyrene (PS) spheres, which arrange themselves in a hexagonally close-packed monolayer on the Si surface. This is achieved by the controlled drying of a droplet of a commercially available aqueous suspension of PS beads spilled on an inclined Si surface using a technique similar to the one described in [4]. The resulting monolayers are up to 1 cm^2 in size and thus contain up to $\sim 5 \times 10^9$ mask openings between each triple of adjoining beads. The mask openings have the shape of regular triangles with concave sides. Owing to deviations in the monomodal size distribution of the colloid beads the

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monolayer masks contain some defects, mostly in form of line defects.

He^+ implantations were performed with a medium current ion implanter at energies between 8 and 48 keV in a dose range of 1–50 × 10^{16} He/cm^2 from a 7° off-normal direction with the substrates kept at room temperature. The discussion here will be restricted to the use of 8 keV He^+ ions. Surfaces were inspected by scanning electron microscopy (SEM) prior and after removal of the PS masks. Mask removal was done by ultrasonification of samples in organic solvents or using adhesive tape or a combination of both. The revealed surfaces were studied by atomic force microscopy (AFM) in tapping mode. Selected samples were examined by cross-sectional transmission electron microscopy (XTEM) using a Jeol JEM2100F operated at 200 keV in conventional, high-resolution and analytical modes. In order to protect the surface of samples during XTEM specimen preparation a film of 100 nm Co was electron beam evaporated prior to preparation.

3. Results and discussion

Colloidal PS nanomasks are surprisingly stable against ion irradiation even at high doses. However, some changes of the mask geometry occur, as detailed in [5]. Linear mask defects widen up into elongated mask openings most likely as a result of surface charging effects. Ion beam induced sintering (IBSI) at the contact points between spheres leads to strong necks between the spheres, similar to what has been observed in NSL masks consisting of SiO_2 spheres [6]. The neck formation in PS masks is even more pronounced than in masks made from SiO_2 as PS shrinks upon irradiation with ions.

Some of these necks can be clearly seen in the XTEM bright-field image in Fig. 1 of a Si wafer covered with a 200 nm diameter NSL mask and implanted with 8 keV He ions at a dose of 2 × 10^{17} He/cm^2. The mask is still partially covered with the Co protection layer. Co has also entered the mask openings and formed metallic nanodots. The size of these dots seems to vary. However, this impression results mainly from the fact that the XTEM specimen slab is not completely cut parallel to the axis connecting the spheres. The Co dots roughly mark the position of mask openings, where He^+ ions penetrated into the Si wafer. The projected range of 8 keV He^+ ions in PS is 125 nm according to SRIM [7] Monte-Carlo simulations, the range straggling 36 nm. Therefore, He^+ ions hitting the centre of a PS sphere are expected to be completely stopped in the PS spheres and He incorporation should take place in the silicon substrate only close to the centre of the mask openings. In fact, amorphization of the Si substrate is observed at the mask openings, visible by the vanishing contrasts in the regions directly underneath the Co dots. The amorphized regions contain numerous voids, as can be visualized either by defocusing the TEM (not shown) or in high-resolution mode (Fig. 2). These voids are considered as remainders of formerly formed He bubbles. As expected, the bubble/void formation has lead to a localized surface swelling, as is highlighted by the dashed line in Fig. 1 indicating the position of the original surface. In between the amorphized void containing regions, i.e. underneath the PS sphere centres, salt-and-pepper contrasts indicate the presence of a high density of point defect clusters. These defects cannot be due to ions impinging into these areas, as these areas are completely masked by the PS beads. Moreover, He ions entering the Si substrate at the mask opening have a lateral projected range of < 40 nm only. Therefore it is concluded that the point defect clusters largely result from Si_I atoms which are injected from the void containing amorphous regions under the mask openings. The amorphized regions (a-Si) exhibit frayed interfaces with the surrounding crystalline Si (c-Si). High-resolution XTEM images of these areas show that voids exist only in amorphous regions (Fig. 2). The structure appears similar to the one reported in [3] for a 20 keV He^+ implantation with 1 × 10^{17} He/cm^2 at 100 °C, which however also contained isolated crystalline inclusions throughout the buried cavity containing layer.

Taking into account the surface swelling, which can be simply read from XTEM images, one can easily show that the largest cavities (25 nm) are formed at the depth of maximum vacancy generation (60 nm), as calculated by SRIM, while smaller cavities (down to 4 nm) are observed at the periphery of the cavity containing volumes. In this respect, a hierarchical nanostructure is present here. The presence of largest cavities at the depth of maximum vacancy concentration supports the model [1] of He bubble nucleation at vacancies.

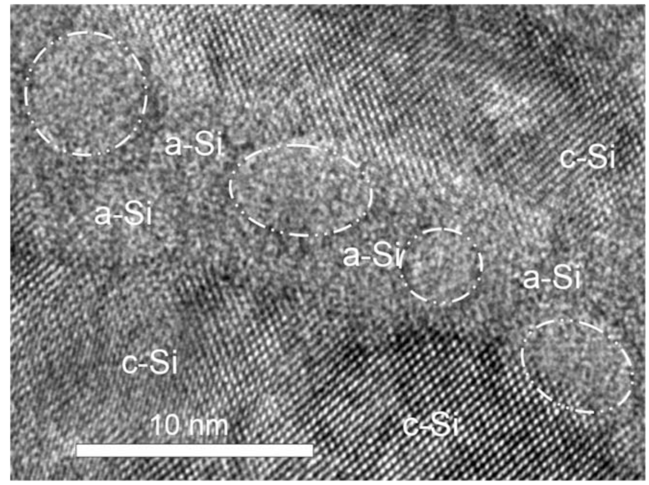

Fig. 2. High-resolution micrograph taken at the periphery of one of the amorphized regions (a-Si) of the sample in Fig. 1. The position of cavities is marked with dashed circles, as under the imaging conditions used their contrast is weak. Note that no cavities are present in crystalline Si (c-Si) regions.

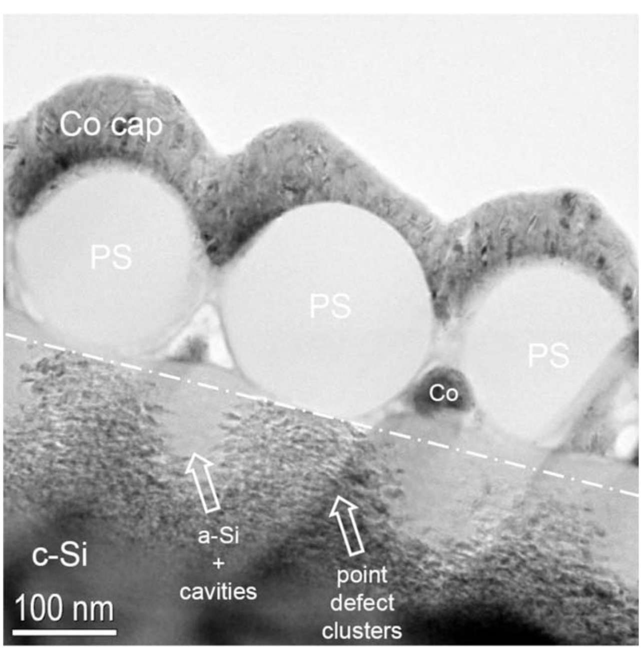

Fig. 1. Cross-sectional TEM bright-field image of a Si(1 0 0) wafer under a NSL mask consisting of 200 nm diameter polystyrene (PS) spheres after irradiation with 8 keV He^+ ions at a dose of 2 × 10^{17} He/cm^2. The Co protection layer as well as one of the Co dots created at the mask openings are marked. The dashed line indicates the position of the original surface and the local swelling which results from the He implantation at the mask openings.