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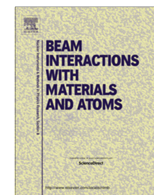

Silicon exfoliation by hydrogen implantation: Actual nature of precursor defects

Pauline Sylvia Pokam Kuisseu [a,*], Timothée Pingault [a], Esidor Ntsoenzok [a], Gabrielle Regula [b], Frédéric Mazen [c], Audrey Sauldubois [d], Caroline Andreazza [e]

[a] CEMHTI-CNRS, 3A, rue de la férollerie, 45071 Orléans, France
[b] IM2NP-CNRS-Université d'Aix-Marseille, Avenue Escadrille Normandie Niemen, 13397 Marseille, France
[c] CEA-Leti, MINATEC campus, 17, rue des Martyrs, 38054 Grenoble Cedex 9, France
[d] Université d'Orléans, rue de Chartres – Collegium ST, 45067 Orléans, France
[e] ICMN-CNRS-Université d'Orléans, 1b rue de la férollerie, 45071 Orléans, France

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ABSTRACT

MeV energy hydrogen implantation in silicon followed by a thermal annealing is a very smart way to produce high crystalline quality silicon substrates, much thinner than what can be obtained by diamond disk or wire sawing. Using this kerf-less approach, ultra-thin substrates with thicknesses between 15 μm and 100 μm, compatible with microelectronic and photovoltaic applications are reported. But, despite the benefits of this approach, there is still a lack of fundamental studies at this implantation energy range. However, if very few papers have addressed the MeV energy range, a lot of works have been carried out in the keV implantation energy range, which is the one used in the smart-cut® technology. In order to check if the nature and the growth mechanism of extended defects reported in the widely studied keV implantation energy range could be extrapolated in the MeV range, the thermal evolution of extended defects formed after MeV hydrogen implantation in (100) Si was investigated in this study. Samples were implanted at 1 MeV with different fluences ranging from 6×10^{16} H/cm^2 to 2×10^{17} H/cm^2 and annealed at temperatures up to 873 K. By cross-section transmission electron microscopy, we found that the nature of extended defects in the MeV range is quite different of what is observed in the keV range. In fact, in our implantation conditions, the generated extended defects are some kinds of planar clusters of gas-filled lenses, instead of platelets as commonly reported in the keV energy range. This result underlines that hydrogen behaves differently when it is introduced in silicon at high or low implantation energy. The activation energy of the growth of these extended defects is independent of the chosen fluence and is between (0.5–0.6) eV, which is very close to the activation energy reported for atomic hydrogen diffusion in a perfect silicon crystal.

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1. Introduction

Hydrogen implantation at high enough fluence and moderate energy (i.e. 20 keV–250 keV) in silicon is known to introduce several types of defects in a damaged zone, which will precipitate in extended defects upon thermal annealing [1]. These extended defects can be used as fracture precursors to achieve material splitting at the implanted depth. Commonly called "ion-cut" process, this ability to induce fracture into materials is widely used in microelectronics, through the smart-cut® technique, to produce SOI substrates [2]. Recently, it has been demonstrated that hydrogen implantation can be used to exfoliate free-standing thick layers up to 100 μm by using MeV implantation energy range, instead of keV implantation energy range as used for smart-cut® technique [3–4]. These free-standing layers find their potentials and emerging applications in many fields, including micro-electro-mechanical systems [5], power devices [6], and photovoltaic solar cells [7].

Since extended defects play the key role in the ion cutting process, their nucleation and their growth in hydrogen-implanted silicon in the keV range have been extensively studied [8–10]. The standard process is commonly articulated as follow: hydrogen implantation at high fluence generates in silicon a damaged zone with various defect types, including vacancy-hydrogen complexes, silicon interstitials, and extended planar defects, the so-called

* Corresponding author.
E-mail address: pauline-sylvia.pokam-kuisseu@cnrs-orleans.fr (P.S.P. Kuisseu).

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"platelets", distributed around the maximum hydrogen concentration (*Rp*) [11,12]. It has been reported that the damaged zone induced by the implantation creates an in-plane compressive stress in the plane parallel to the substrate surface and hence a lattice strain of crystalline planes in the z direction [13]. This material modification drives the orientation of platelets preferentially parallel to the surface. This is why platelets formed after hydrogen implantation at low energies are mostly parallel to the substrate surface [14]. However, when the in-plane compressive stress is too weak, platelets are expected to lie in {111}, which are the planes having the lowest surface energy in silicon [15]. The idea is supported by the fact that preferred orientation of platelets along {111} is usually observed after plasma hydrogenation of silicon with little or no induced-strain [16–17]. Under thermal annealing, hydrogen diluted in the lattice diffuses into the platelets and promotes their growth. The growth of platelets is mainly attributed to the Ostwald ripening mechanism [12], which involves the hydrogen diffusion from smaller to bigger platelets. Consequently, bigger platelets grow while smaller ones dissolve in the lattice. Since the growth of platelets is mainly due to the exchange of hydrogen, it is assumed that platelets are highly pressurized by H_2 molecules [18]. For further annealing in time or temperature, platelets highly pressurized further grow in size, and at a critical anneal duration or temperature, they coalesce to generate the propagation of a crack into the material [14].

However almost all studies made on hydrogen implantation in silicon have been carried out only at low or medium energy ranges (20 keV–250 keV). The extrapolation of the hydrogen behavior at high implantation energies in the MeV range is not yet systematically investigated, although implantation in this energy range does appear as a promising way to detach free-standing thin substrates required for applications cited above. Earlier investigation made by C. Braley et al. [19] on the splitting of (100) Si after MeV hydrogen implantation energy showed that unlike implantation at low energies, platelets formed after implantation at high energies are mostly oriented along {111} planes whatever the substrate orientation. Authors found that preferred platelets orientation along {111} is a consequence of the low in-plane compressive stress induced by MeV hydrogen implantation energy. In fact, as the implantation energy rises, the hydrogen level profile broadens. Due to this broadening, lattice is less distorted, which results in a lower in-plane compressive stress, unable to favor any other platelet orientation other than {111}. This interesting result was used to explain why implanted layer detachment is preferentially obtained in (111) Si compared to (100) Si. But numerous questions on the fracture mechanisms involved at the MeV energy range are still open. Hence, in this work, we propose to investigate the nature and the growth mechanism of extended defects in the MeV range where the in-plane stress is relatively low compared to the widely studied "ion-cut" conditions at considerably lower energy. Attention is focused on the shape, the spatial distribution and the thermal growth of extended defects in (100) Si after MeV hydrogen implantation. Induced damaged zone is about six times higher to that reported in the keV range. Extended defects involved in the fracture mechanism are some kind of planar clusters of gas-filled lenses, instead of platelets as commonly reported at low implantations energies. The activation energy of extended defect growth is found to be around 0.5 eV and independent of the hydrogen fluence.

2. Experimental procedure

Material investigated in this study is Cz p-type (100) Si with resistivity in the range of 8 to 14 Ω.cm, i.e. having a boron doping level of 1.6×10^{15} cm^{-3} to 9.4×10^{14} cm^{-3}. Samples are implanted with hydrogen at room temperature at 1 MeV, each with one of the following fluences: 6×10^{16} H/cm^2, 7×10^{16} H/cm^2 and 2×10^{17} H/cm^2. The first fluence is expected to induce blistering, while the two other ones allow the wafer splitting. During implantations, the ion beam current is kept at a few μA and samples are tilted at approximately 5° away from normal incidence to avoid channeling effects. According to SRIM simulations [20], the projected range *Rp* is located at (16.8 ± 0.2) μm below the sample surface. Implantations are carried out with a pelletron accelerator located at CEMHTI-CNRS in Orléans, France. After implantation, samples are cleaved in small pieces and annealed into a tubular furnace under atmospheric conditions at 573 K, 738 K, and 773 K during 1800 s, and at 873 K during 3600 s. Subsequently, annealed samples are glued for cross-section observations and thinned by standard mechanical polishing procedure with a tripod, followed by Ar^+ beam milling at 5 keV until perforation of the sample. Cross sectional transmission electron microscopy (XTEM) images are obtained using a Philips CM20 operating at 200 kV to study induced-defects of both as-implanted and annealing samples. The best conditions to visualize extended defects are "out-of-Bragg" conditions, with a tilt of sample of few degrees away from the zone axis. The images are taken in bright field mode, and small tilt angles between 20° and 30° had to be reached to better figure out shapes and arrangements of induced extended defects.

3. Results and discussion

3.1. Induced crystalline-damaged zone

Fig. 1 shows typical XTEM images of a 7×10^{16} H/cm^2 as-implanted sample. A damaged zone of (1.3 ± 0.3) μm induced by the ion ballistics is observed. No extended defects are observed in this area. A closer observation of a small area taken in this damaged zone (see Fig. 1b) reveals the presence of a strong strain contrast due to a high density of point defects. The electron diffraction pattern taken in this zone reveals that it remains crystalline after implantation. Note that the same kinds of images are obtained after implantation at 2×10^{17} H/cm^2 (not shown). This means that under our experimental conditions, no extended defects are created during the implantation step. Indeed, for the same range of hydrogen fluence, the width of the damaged zone reported in the keV range is about six times smaller, and typical extended defects such as platelets are commonly observed in as-implanted samples [14,21]. Hence, the absence of extended defects under our implantation conditions is likely a consequence of the wider dimension of the damaged zone which results in a lower local hydrogen concentration at Rp

Fig. 2 shows thermal evolution of damaged zones of samples implanted at 7×10^{16} H/cm^2 and 2×10^{17} H/cm^2 and annealed at 738 K during 1800 s. Compared to as-implanted sample, it is obvious that the thickness of the detectable damaged zone has significantly increased upon thermal annealing with a high density of extended defects. For both fluences, the width of the damaged zone is very similar, and we can conclude that the thickness of the damaged zone is independent of the hydrogen fluence for the same used energy. A closer observation of the damaged zones after annealing reveals that, for the lowest fluence (7×10^{16} H/cm^2) most of extended defects are oriented along {111} (see Fig. 3a), while for the highest fluence (2×10^{17} H/cm^2) most of them are oriented along {100} (see Fig. 3b). This observation can be explained in term of level of in-plane compressive strain induced during the implantation, as previously described in the introduction section. Indeed, for the two lowest fluence, the in-plane compressive strain is weak and then unable to influence the orientation of extended defects. That is why extended defects are oriented

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