Temperature dependence of blistering in hydrogen implanted Si and Ge

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D.J. Pyke [a], R.G. Elliman [a,*], J.C. McCallum [b]

[a] Department of Electronic Materials Engineering, Research School of Physics and Engineering, The Australian National University, Canberra, ACT 0200, Australia
[b] Microanalytical Research Centre, School of Physics, University of Melbourne, VIC 3010, Australia

ABSTRACT

The temperature dependence of hydrogen blistering rates are measured in (100) Si, (111) Si and (100) Ge substrates implanted with 40 keV H- ions to a fluence of 6×10^{16} H cm^{-2} using real-time imaging of samples during annealing. The time taken for blisters to form was found to exhibit Arrhenius behaviour and to be characterised by a single activation energy over the temperature range examined (375–650 °C for Si and 300–600 °C for Ge). The extracted activation energies, which are believed to be the sum of a hydrogen-complex (H–X) dissociation energy and a hydrogen migration energy, were found to be 2.28 ± 0.03 eV for (100) and (111) Si and 1.4 ± 0.03 eV for (100) Ge. These results are discussed with reference to a model for blister formation and compared with previously reported measurements.

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1. Introduction

Silicon-on-insulator (SOI) wafers are routinely employed for the fabrication of integrated electronic circuits, photonic devices and structures and micro-electro-mechanical systems (MEMS), and are most commonly fabricated by a hydrogen-induced cleavage technique in which ion-implanted hydrogen is employed to initiate and propagate cracks in a plane parallel to the silicon surface [1]. Considerable research effort has been devoted to understanding the cleavage process but several fundamental issues remain unclear, including the role of lattice strain on hydrogen platelet alignment [2,3], and details of the bond-breaking processes associated with crack propagation (i.e. stress induced or hydrogen-mediated) [4,5]. There is also interest in extending the process to other semiconductor materials, such as Ge [6] and III–V [7] semiconductors.

Accurate modelling of hydrogen cleavage [5,8,9] is essential for process optimisation and the development of new or novel approaches [10]. This requires a detailed understanding of the thermodynamics of hydrogen trapping, diffusion and aggregation, and of the fracture mechanics underpinning crack propagation. Central to this understanding is knowledge about the temperature and time evolution of hydrogen blistering. Han and Yu [5] have developed a thermodynamic model for hydrogen blistering that provides an explicit expression for the time dependence of blistering. In this study we use an in situ observation technique to measure the time dependence of blistering and compare the results with this model.

2. Experimental method

Three different materials were investigated for this study: (100) Si, (p-type, B-doped with resistivity $\rho = 10$–$20\ \Omega$ cm); (111) Si (p-type, B-doped with $\rho = 7$–$15\ \Omega$ cm); and (100) Ge (undoped with $\rho > 40\ \Omega$ cm). Each of these materials was implanted with 40 keV H ions to a fluence of 6×10^{16} H cm^{-2} at room temperature. During implantation the ion beam current was kept between 1 and 3 μA to minimise beam heating effects.

Implanted samples were cleaved into pieces approximately 3×3 mm in size, and annealed at temperatures in the range $T = 300$–650 °C. Annealing was performed in air by placing samples on a heated sample stage incorporating a simple vacuum chuck to ensure good thermal contact between the sample and the stage. A video monitoring system was used to record hydrogen-induced blistering during annealing, with a commercially available digital camera capturing images at a frame rate of 30 fps, and with a 1280×720 resolution. Images were subsequently analysed to determine the number of blisters as a function of time and to provide statistics on the blister dimensions. Selected samples were also examined using a Wyko NT9100 optical surface profiler to determine the size and fracture depth of the resulting craters.

3. Results and discussion

Fig. 1a and b show optical images of a hydrogen-implanted (100) Si sample during annealing at 525 °C. These images, which were taken after 16 and 400 s, clearly demonstrate the increase in non-specular reflection from blistered areas. This was used to measure the blistered area as a function of time during annealing.

* Corresponding author. Tel.: +61 2 6125 0521; fax: +61 2 6125 0511.
  E-mail address: rob.elliman@anu.edu.au (R.G. Elliman).
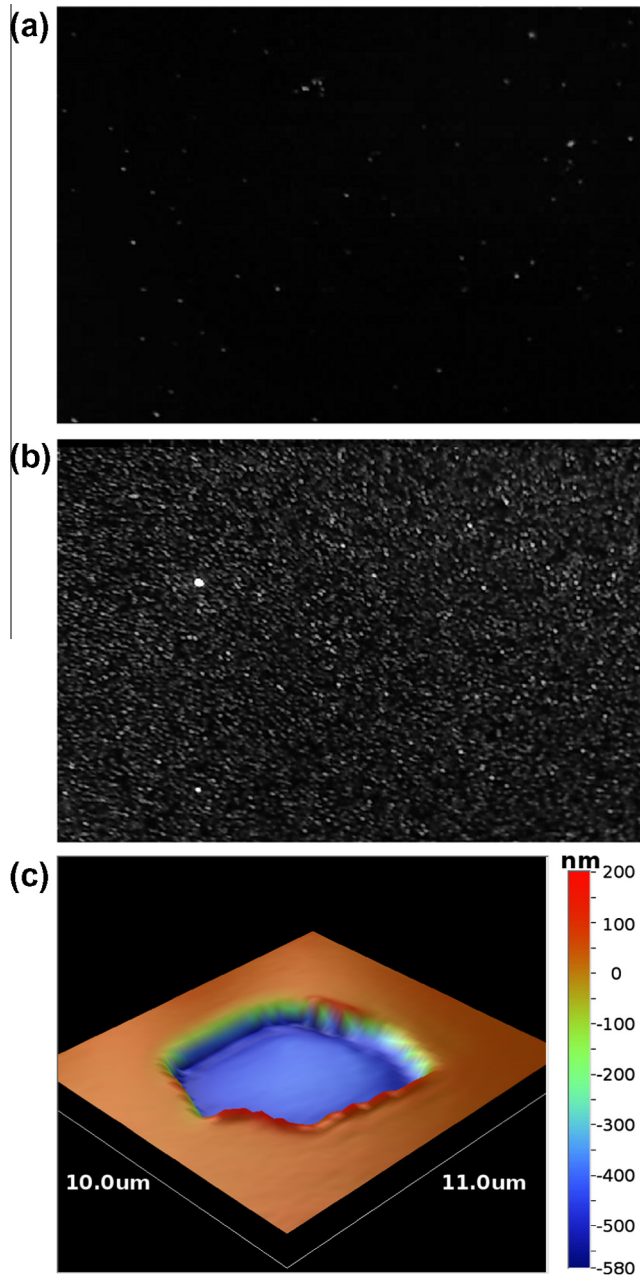
Fig. 1. Images recorded during in situ monitoring of a (100) Si sample annealed at 525 °C: (a) 16 s, (b) 400 s, and (c) an optical profiler image of a blister formed on the surface, showing a cleavage depth of 439 nm. Optical image area is 1.2 × 1.0 mm.

The temporal evolution of blisters as a function of temperature, as determined from the in situ image analysis, is shown in Fig. 2 for each substrate. This clearly shows the strong temperature dependence of blister formation and the order of magnitude difference in kinetics for Ge and Si. These curves can also readily be differentiated to provide the rate of blister formation as a function of time
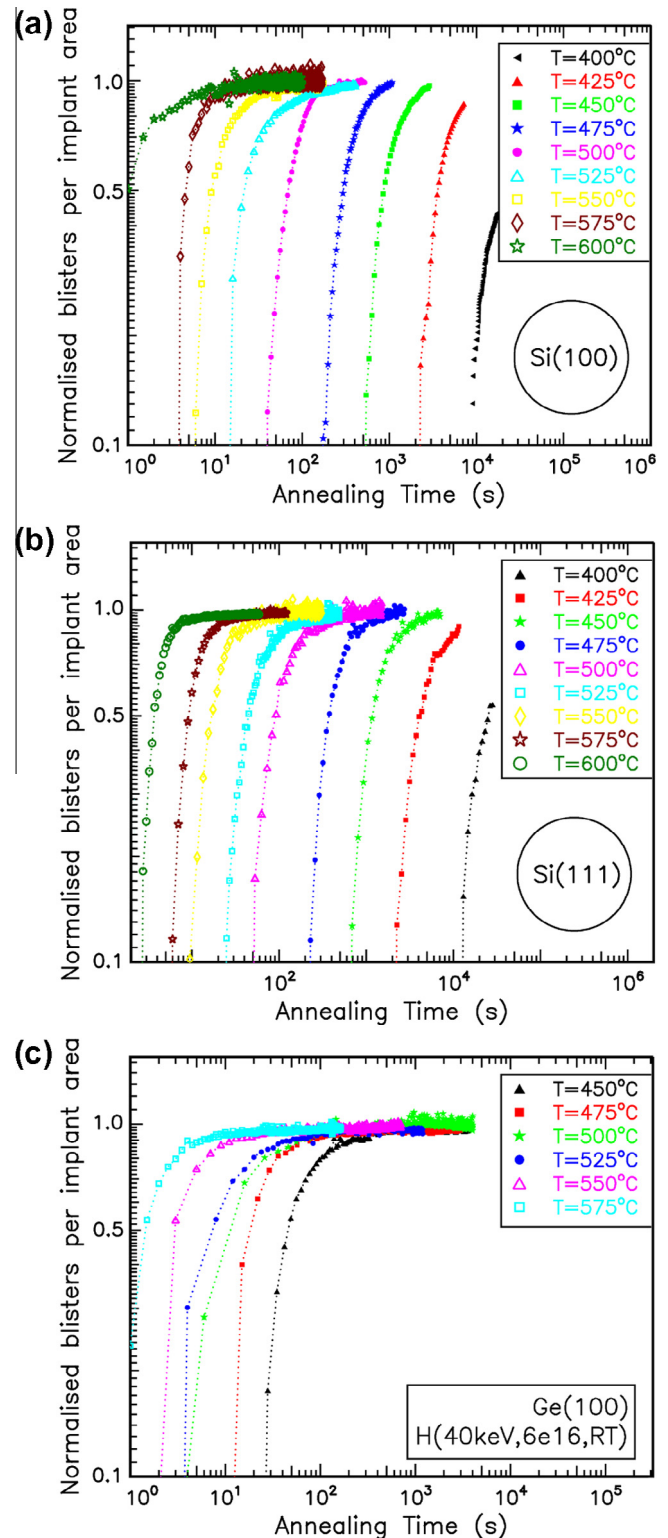
Fig. 2. Normalised image intensity (blister density) as a function of temperature and time for Si(100), Si(111) and Ge(100).

The as-implanted surface was used to calibrate the intensity without blisters, and the first few video frames were used to calibrate the relationship between blister number or area and the average image intensity. For very low blistering rates, where individual blisters were readily identified in each frame, manual counting was performed to provide a direct correlation between intensity and blister number. The typical dimensions and fracture depths of blisters formed on (100) Si during annealing at 525 °C are shown by the optical profiler image in Fig. 1c. For this particular case the blisters have lateral dimensions of order 5 μm and a well-defined fracture depth of 439 ± 6 nm. As blistering leads to the removal of the surface layer there is no overlap of blisters and no additional blisters form in the blister crater. This means that the measured surface reflectivity is a linear function of blister area up to complete surface coverage.