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Surface blistering of low temperature annealed hydrogen and helium co-implanted germanium and its application to splitting of bonded wafer substrates

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ABSTRACT

Applications involving transfer of germanium layers to silicon-based substrates often require a process involving a restricted thermal budget. The use of relatively low temperatures has a major advantage in reducing stresses when thermal splitting of implanted germanium wafers bonded to silicon-based substrates is used to create germanium-on-oxide (GeOI) layers. The present study investigates the phenomenon of blistering of hydrogen and helium co-implanted germanium over the temperature range 250–400 °C, optical microscopy being used to detect the initial appearance of the blisters. Results showed that plots of Ln(time) vs. blister initiation temperature consisted of several straight-line regions yielding an activation energy for each region. The plots showed similarities to those observed in previous work with silicon co-implanted and annealed under similar conditions. At temperatures below the blister initiation temperature, transmission electron microscopy (TEM), revealed the presence of spherical bubbles at a depth below the surface estimated to be approximately that of the hydrogen implant projected range. GeOI layers were produced by thermal splitting of co-implanted germanium wafers bonded to oxide-coated silicon substrates wafers at a temperature of 300 °C. The RMS roughness of the split germanium surface measured by atomic force microscopy (AFM) was about 11 nm averaged over the wafer surface. In addition there were isolated and randomly distributed regions of 27 nm roughness covering about 20% of the total surface area of the wafer.

1. Introduction

Transfer of germanium layers onto silicon-based substrates is of considerable interest for the development of improved performance in micro-electronics [1], solar energy [2] and near infra-red optics [3]. For optical telecommunications the integration of light detectors in the near infra-red, for example in the form of arrays and CMOS based circuitry, has major benefits in terms of cost and efficiency [4,5]. Several approaches have been tried involving deposition of Ge [6–8] or SiGe [9] on silicon or silicon-oxide substrates. Whilst these technologies continue to improve, the use of bulk germanium in wafer form as a primary source offers freedom from dislocations, improved performance from the high crystalline quality starting material and economical use of material. Transfer of Ge can be made effectively using thermal splitting of a hydrogen implanted substrate [2], although the difference in expansion coefficients entails the risk of stress fracture during annealing leading other workers to use mechanical cleavage [10]. We have found that this problem can be avoided by limiting processing temperatures to 300 °C. Thermal splitting of hydrogen implanted silicon substrates is normally carried out at temperatures between 400 °C and 500 °C [11], but can also be achieved at lower temperatures with longer annealing times [12]. The technique is particularly applicable to materials such as GaAs or SiGe [13,14]. An alternative approach is to co-implant with hydrogen and helium and this has been found to be effective for co-implanted silicon bonded to oxide-coated substrates and subsequently annealed at 280 °C [15]. In the present studies we have investigated the effect of annealing on the production of surface blisters using germanium samples implanted with both hydrogen and helium in the temperature range 250–400 °C and report the results of low temperature thermal splitting of implanted germanium wafers bonded to oxide-coated silicon wafers.

2. Experimental

Experiments were made on commercially available <100> p-type, Ga doped, germanium 100 mm diameter wafer substrates.

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The substrates were cleaned in dilute HF solution and rinsed in de-ionised water (DIW). For thermal splitting tests an oxide layer of about 100 nm was deposited on the surface of the germanium wafer to be implanted and a 600 nm layer thermally grown on the silicon handle wafer by wet oxidation. The germanium substrates were then implanted with a fluence of 3×10^{16} ions/cm^2 of H_2 at 120 keV followed by 10^{16} ions/cm^2 of He at 68 keV. The fluence for implanted H was calculated on the basis that H_2 splits on impact producing twice the dose at half the energy [12] (H_2 fluences are given in this text).

For blister tests, germanium substrates without any oxide coating were implanted using the conditions as given above and then broken into small samples of about 0.5–1 cm^2. A given sample was annealed by placing in a heated furnace set at a fixed temperature for a pre-determined time in the range from 250 °C to 400 °C, and then removed and examined for the appearance of surface blisters using a Reichert-Jung Polyvar Met optical microscope fitted with a Nomarski differential interference contrast objective. The technique is similar to those adopted by other authors, the blisters having a similar appearance to those observed with implanted SiGe [14] and silicon [16]. For the annealing process, the sample was placed on a pre-heated 5 mm thick flat quartz support sitting in turn directly over a measuring thermocouple which was used to control the sample temperature whose variation during the first few minutes of stabilisation was controlled to ±2 °C. The control system did not allow the temperature to exceed 2 °C above the set-point at any time and once the temperature had stabilised, deviations were less than ±0.5 °C.

If the result of a test showed no optically visible blister formation, another sample (from the same wafer substrate), was annealed for an increased time. The process was repeated until blisters were observed under the microscope. The method of investigation used here yielded two points of interest on a time–temperature graph at each fixed temperature: a time for no-blister formation and an increased time for the optical discernment of recognisable blisters. Hence the actual critical time for the first appearance of blisters would be defined by these two limits and lie somewhere in between.

TEM observations were made on several samples that had not produced blisters after annealing (but by reference to previous measurements were believed to be close to the blistering point), using a focused ion beam (FIB) to produce a cross-sectional slice for subsequent examination.

For thermal splitting tests, the 100 nm oxide deposited on the implanted germanium wafers was stripped with dilute HF solution and the substrate exposed to an RF oxygen plasma for 15 s at 20 W power, dipped in H_2O:HF 10:1 solution for 10 s, then rinsed in DIW and dried. These substrates were contact bonded in atmosphere to oxide-coated silicon wafers which had been exposed to an RF oxygen plasma for 15 s at 25 W and rinsed in DIW and dried. Bonded wafer pairs were annealed at temperatures in the range from 280 °C to 300 °C for times in the range 10–72 h and examined at intervals for thermal splitting. Thermally split samples were examined by AFM for surface topography and roughness.

3. Results

Fig. 1 shows the results of the blister tests plotted as a Ln(time) against temperature graph. The graph shows three regions that allow possible straight-line interpretations between 250 °C and 310 °C, 318 °C and 340 °C, and between 340 °C and 400 °C. Additional measurements in the region 308–318 °C verified the abrupt nature of the change in slope, and can be compared to the ‘dog-leg’ discontinuity previously observed with silicon substrates (at about 305 °C) and performed under similar conditions using the same techniques [15]. The measurements are re-plotted as Arrhenius plots in Fig. 2 together with the previous results for silicon blistering. In these plots, the mean of the two points, blister and no-blister, at a given temperature is used as the best estimate for the time for inception of blisters. The graphs may be interpreted in terms of a process activation energy for the straight-line regions and are shown in Table 1. Fig. 3 shows a TEM image of implanted germanium annealed at 300 °C for 30 min. A line of spherical bubbles 10–30 nm diameter is observed and centred at about 430 nm below the surface. Dark lines and shadows are present on the side of the bubbles furthest from the surface. Dark irregular-shaped regions of similar size can be seen distributed throughout the volume.

Co-implanted germanium wafers bonded to oxide-coated silicon substrates split after 24 h annealing at a temperature of 300 °C producing a pinhole-free GeOI layer of 400 nm measured thickness, significantly greater than the calculated hydrogen projected range of 348 nm (from the germanium surface after stripping the protective oxide). Fig. 4 shows a cross-section measured with an AFM of the split surface. Fig. 4(a) is typical of the surface as

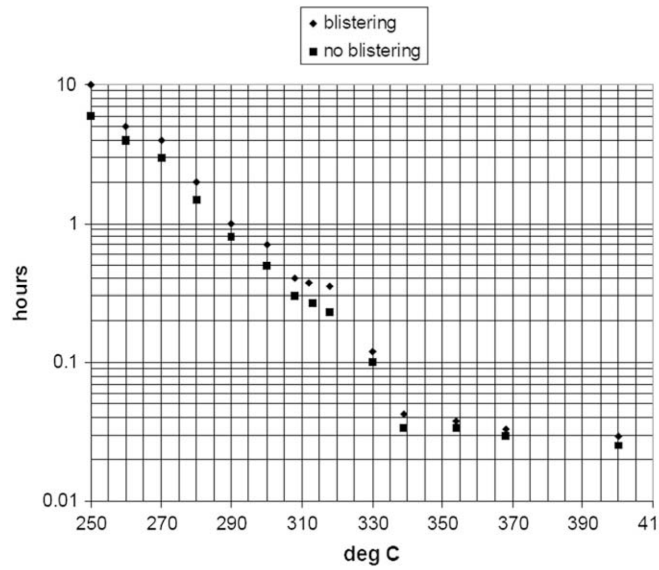

Fig. 1. Plot of annealing time required for optically detectable surface blisters against temperature for co-implanted germanium (3×10^{16} ions/cm^2 of H_2 at 120 keV followed by 10^{16} ions/cm^2 of He at 68 keV). Points for maximum time measured for no-blisters detected are also given.

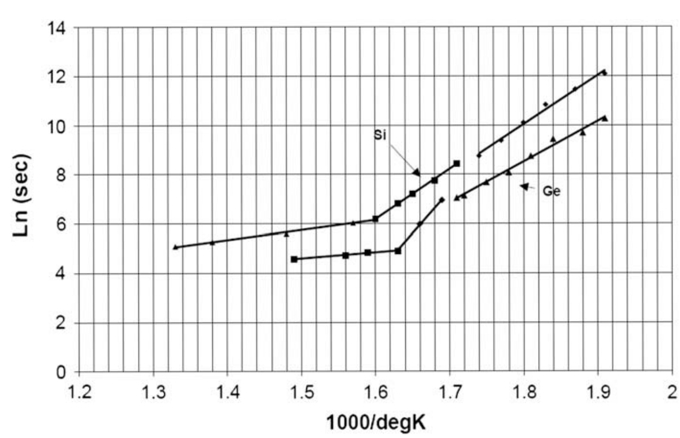

Fig. 2. Arrhenius plot, Ln(time) against 1000/K for data given for Ge in Fig. 1 and for Si processed under similar conditions [15]. Each point represents the mean of blister and no-blister points at a given temperature in Fig. 1.