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Microstructure and nanomechanical properties of Fe^+ implanted silicon

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ABSTRACT

Silicon wafers were implanted with iron ions at different fluences (from 5×10^{15} up to 2×10^{17} cm^{-2}), followed by annealing treatments at temperatures from 550 °C to 1000 °C, aiming at evaluating the nanomechanical response of the samples and its relation with the microstructural features and characteristics of the modified layer. After implantation, a homogeneous amorphous layer with a thickness between 200 nm and 270 nm is formed, without damaging the surface smoothness neither introducing surface defects. After annealing, recrystallization and formation of nanometric precipitates of iron silicides is observed, with the corresponding changes in the hardness and stiffness of the modified layer. These results indicate that ion implantation of silicon followed by annealing at proper temperatures, can be an alternative route to be deeper explored in what concerns the precise control of the microstructure and, thus, the improvement of nanomechanical properties of silicon.

1. Introduction

Crystalline silicon (c-Si) is certainly one of the most used materials in the microelectronics industry due essentially to three factors: its semi-conductor properties, the relatively low production costs and, nowadays, the well-established Si-based technology and the know-how for the production of submilimetric devices. This Si-based technology (and its continuous development, namely in what concerns micro and nanotechnology) is undoubtedly the basis of the extraordinary development of microelectronics in the last decades. As a consequence of the application of Si in electronics, its use has been naturally extended to the production of micro and nano-electromechanical systems (MEMS/NEMS). However, Si as a structural material, even to be used as base material for micro and nano-electronic devices, like MEMS and NEMS, presents poor mechanical and tribological properties [1–4]. Being this a major drawback to the use of Si for the production of MEMS and NEMS, several solutions to improve its mechanical properties at the surface contacting scale have been attempt. Namely, implantation of Si with different ions has been widely used as a modifying root for the surface properties, and different authors have reported results on the implantation of Si with Cr [5], C [6–8], N [6,7], Ne [8,9] and Ar [8,10], among other elements. Although some authors have reported the implantation of Si with Fe^+ [11,12], the main concern of these previous works has been the influence of Fe in the electronic behavior of Si. As far as the authors know, no attempts to characterize the nanomechanical surface properties of Si implanted with Fe^+, and its relation with the formed microstructure, has yet been made, in spite of the fact that the implantation of Si with Fe ions can be potentially beneficial due to the possibility of formation of hard and stiff iron silicides. Hence, in this work, it is presented an analysis of the influence of Fe^+ ion implantation on the nanomechanical properties of Si surfaces.

Silicon wafers were implanted with Fe^+ at different fluences (from 5×10^{15} up to 2×10^{17} cm^{-2}), followed by annealing treatments at temperatures from 550 °C to 1000 °C. The samples were mechanically characterized by depth sensing ultramicroindentation tests and the structures of the modified Si layer was characterized by scanning electron microscopy, X-ray diffraction and Rutherford backscattering spectrometry. It is shown that, in the as-implanted state, implantation with Fe^+ in the used conditions leads to a flat surface, without defects, formed by a supersaturated amorphous Si layer with a depth between 200 and 270 nm. The supersaturated amorphous microstructure that result from the implantation leads to a decrease of hardness and stiffness as compared with the un-implanted samples. Conversely the annealing treatments between 550° and 800 °C, leads to the highest values of the mechanical properties, which is a result of the recrystallization of silicon and precipitation of nanocrystallites of β-$FeSi_2$. The

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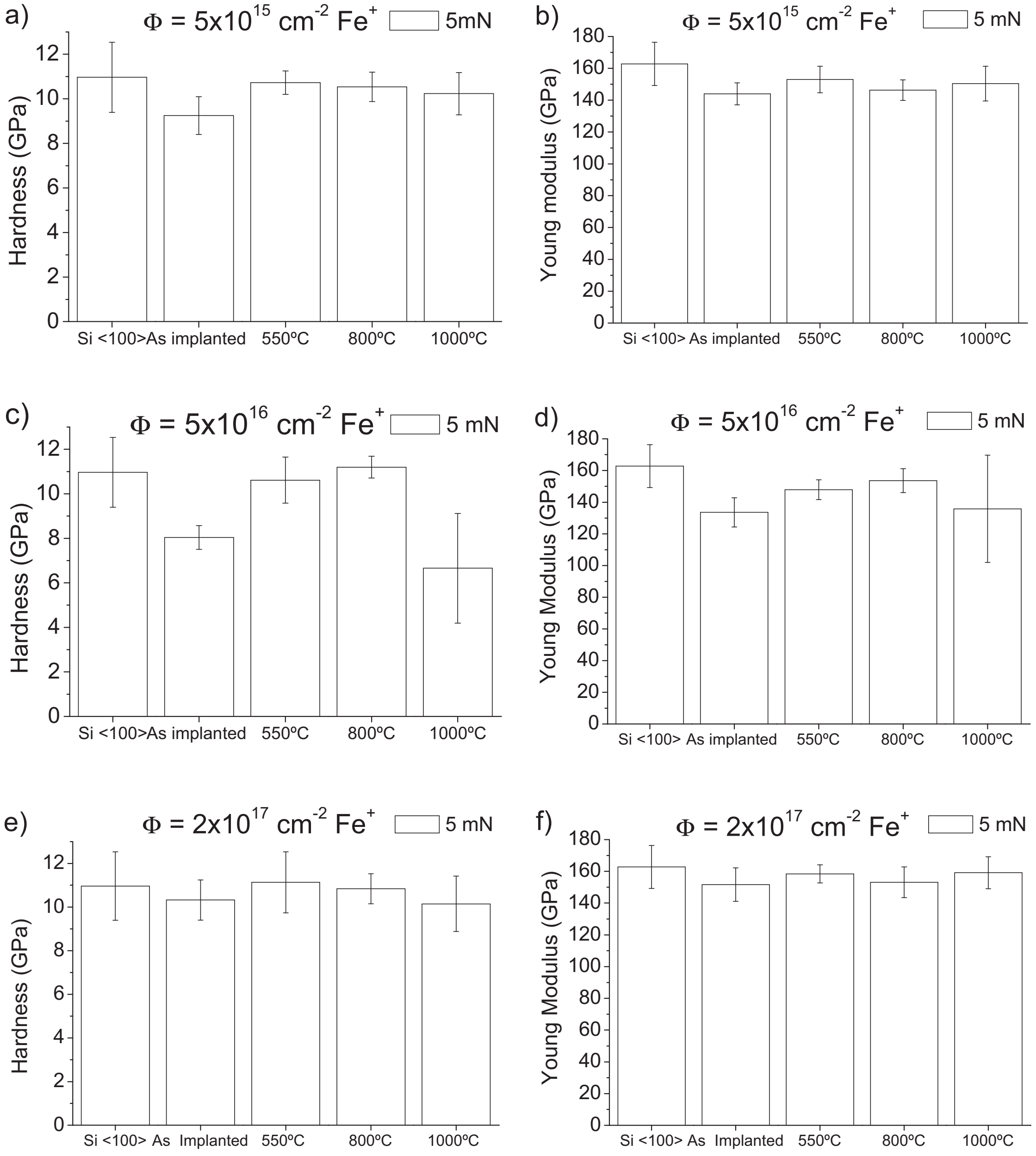

Fig. 1. Hardness and Young's modulus of 150 keV Fe$^+$ ion implanted Si(1 0 0) with a fluence of 5×10^{15} cm^{-2} (a and b); 5×10^{16} cm^{-2} (c and d); and 2×10^{17} cm^{-2} (e and f) measured with 5 mN normal load, for both the as-implanted state and all annealing temperatures. Si(1 0 0) is used as reference.

mechanical properties decrease again when the annealing is made at 1000 °C, due to the formation of α-FeSi$_2$.

2. Experimental

2.1. Materials and methods

Single crystal 50–100 (Ω m)$^{-1}$, *p*-type (1 0 0) silicon wafers were implanted with Fe$^+$ at 150 keV using a high fluence implanter Danfysik 1090. The ion fluences used were 5×10^{15}, 5×10^{16} and 2×10^{17} cm^{-2} (from this point forward the samples will be designated as low, medium and high fluence samples, respectively, for simplification). The ion implantation was done at room temperature with normal incidence of the ion beam. The beam current was kept below 0.5 μA/cm^2 in order to avoid heating effects. After the implantation the samples were annealed for 30 min at 550, 800 and 1000 °C under vacuum (<10^{-6} mbar). A preliminary report on this type of implantation was published elsewhere [13].

2.2. Characterization techniques

The nanomechanical properties of the surface were evaluated by depth sensing ultramicro-indentation. The measurements were performed at room temperature with a Shimadzu DHU-211S apparatus using a Berkovich indenter with 115° between faces. The maximum load was chosen to obtain in all the samples an indentation depth smaller than 100 nm. In this way, after preliminary tests, the samples were indented at a constant rate of 0.2926 mN/s until a maximum load of 5 mN. The maximum load was kept for 20 s. The hardness and Young modulus determination was based on Oliver and Pharr method [14].

The microstructure of the samples was observed with a JEOL JSM-7001F field emission gun scanning electron microscope