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Morphology and Stress Investigations of Surface and Subsurface Regions of Plasma Hydrogenated and Annealed Czochralski Silicon

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Hydrogen plasma treatments applied on Czochralski silicon (Cz Si) wafers cause a structuring of the surface regions down to the sub-100 nm scale [1)]. The morphological properties and evolution of these 'nano-structures' and their impact on the subsurface regions of the wafers were studied in dependence on the process conditions on p- and n-type Cz Si wafers. The H-plasma treatments were applied for 1 hour at ~ 260 °C in a PECVD-setup operating at 13.56 MHz frequency (plasma power: 10 – 50 W). Investigations were done by scanning electron microscopy (SEM), atomic force microscopy (AFM), and µ-Raman spectroscopy (µRS).

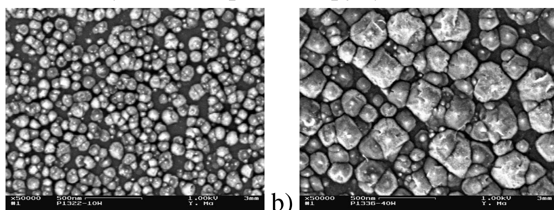

Fig. 1: SEM pictures of as plasma treated p-type Cz Si; power: a) 10 W, b) 40 W; area size: 2.28×1.65 µm^2.

SEM and AFM analysis elucidate the mechanism for the structuring of the Si surface layer by H-plasma exposure. Two reactions might occur, i.e. (i) etching of the Si surface by hydrogen and (ii) redeposition of Si 'nano-crystals' during the plasma exposure. SEM studies emphasize that both reactions occur, but redeposition seems to be more important concerning the morphology of the 'nano-structures'. If the wafer is immersed in a H-plasma, Si atoms are released from the surface (etching), and the plasma also contains a significant concentration of Si atoms, which can be redeposited onto the wafer surface again. Applying a low plasma power (10 W) the redeposited Si 'nano-crystals' are very small and do not cover the entire surface of the underlying wafer (Fig. 1a). If the plasma power is higher the diameter of the 'nano-structures' is larger (Fig. 1b). AFM analysis shows that the surface roughness parameters do not systematically change in dependence on the applied plasma power, i.e. it can be concluded that lateral growth of the redeposited 'nano-crystallites' is faster than vertical growth.

The morphological variations of the 'nano-structured' surface layers and the following appearance of stress in the surface/sub-surface layers of the hydrogenated wafers were analysed in dependence on annealing up to 1200 °C in vacuum for 2 hours. Up to 700 °C annealing the 'nano-structures' at the wafer surface are not changed (SEM). But annealing at $T_{ann} \geq 800$ °C causes their dissolution walking along with the reconstruction of a smooth wafer surface. But this process is not optimally executed, since the perfect single crystalline structure of the (100)-oriented wafer is not completely reconstructed again, since the reconstruction starts in parallel at numerous nucleation points over the entire wafer surface, and therefore a lot of grains are formed. At high temperature annealing (≥ 1000 °C) the grains grow together, but extended disturbed regions with strong mechanical stress remain. Stress can be verified by µRS, since the Raman shift depends on the elastic material properties. The relation between the components of the strain tensor and the Raman frequency can be calculated. The results are simple for uniaxial or biaxial stress. Tensile stress causes a decrease of the Raman mode ($\Delta\omega < 0$) and compressive stress an increase ($\Delta\omega > 0$). In Fig. 2 the Si Raman lines of plasma hydrogenated p- and-n-type Cz Si are shown in dependence on the annealing temperatures. Above 800 °C strong tensile stress occurs (1200 °C: ~ 3.0 GPa, ~ 1.8 GPa for p-, n-type Cz Si, respectively), as can be estimated from the shift of the Raman lines in Fig. 2.

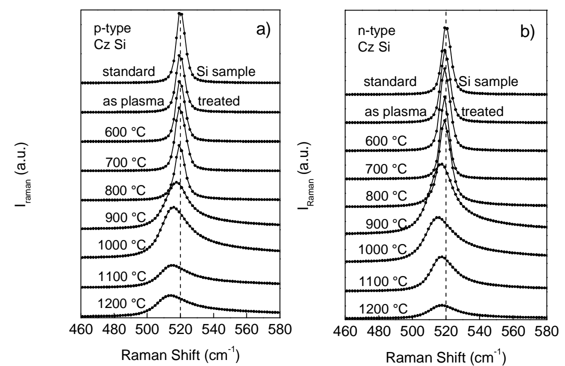

Fig. 2: Si optical phonon line measured by µ-Raman spectroscopy on plasma treated and annealed p- and n-type Cz Si, annealing temperature: 600 – 1200 °C.

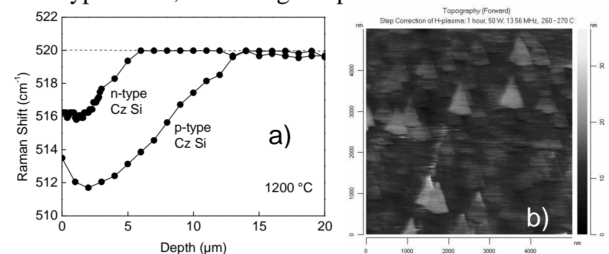

Fig. 3: Left: Si Raman shift in dependence on the wafer depth (broken line: Raman shift of the standard sample); right: AFM profile after 900 °C annealing.

AFM analysis and depth resolved µRS provide more insight. Fig. 3a shows the Raman modes of p- and n-type Cz Si measured in dependence on the wafer depth for samples with post-hydrogenation annealing at 1200 °C. Although the H-plasma caused 'nano-structured' surface layer is very thin (~ 150 nm) [1)], the stressed subsurface regions of the annealed samples are quite extended, e.g. up to ~ 14 µm depth for p-type Cz Si. An elucidation of the appearance stress in plasma treated and annealed Cz Si was found with atomic force microscopy (AFM). In Fig. 3b the surface roughness mapping for an n-type sample treated by H-plasma and annealing at 900 °C is shown. The elevated triangular structures indicate that the rebuilt grains on the wafer surface are (111)-oriented, in contrast to the (100)-orientation of the underlying wafer. With this regard the appearance of stress is evident.

The presented results might be important for various applications in semiconductor technology. For example, the H-plasma exposure could be done in local areas near electrically active regions on the forefront of the wafer. After appropriate annealing, stressed local regions for external getter purposes can be created. Such a method might especially be useful for the formation of external getter regions on SOI substrates. Experimental studies concerning surface modifications of H-plasma treated Cz Si can be also essential for the development of improved technological methods for a Si layer exfoliation walking along with wafer bonding (i.e. for a H-plasma supported Si layer exfoliation at low dose implantion, "Soft-Cut").

[1)] *R. Job, A. G. Ulyashin, W. R. Fahrner, M. F. Beaufort, J. F. Barbot, The European Physical Journal - Applied Physics (EPJ AP) 23, 25 (2003).*