Damage Layers in Si vs. Ion Dose during 30 keV FIB Milling

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Focused ion beam (FIB) systems have been widely used over the last two decades for crosssectional and plan-view TEM specimen preparation. However, an inevitable result of the FIB preparation process is the formation of the damage layers on the sides of the final thin electron transparent membrane, which reduces the quality of the TEM imaging and limits the minimal useful specimen thickness [1]. The knowledge of the thickness of the damage layers is critical because it allows to estimate the value of undamaged material in the FIB prepared samples. In many works [2-5] the damage in the FIB prepared samples is attributed to the amorphous layers created during the FIB preparation process and its thickness was estimated on the basis of popular TRIM program [6]. Using TRIM the projected range (R_p) of implanted ions can be calculated for the particular type of ions, ion energy, target material and incident angle. The value of R_p is commonly used for the estimation of the thickness of the amorphous damage layer in the FIB prepared TEM samples [2-4]. However, the experimentally obtained thickness of the amorphous damage layers in the silicon and other materials [2, 5] was nearly twice larger than the TRIM calculated data for projected range.

In this work the process of the damage formation during 30 keV FIB milling in silicon samples was studied as a function of ion dose delivered to the sample at incident angles 4° and 90° at room temperature. According to the TRIM simulation the projective ranges for these angles are 7 and 28 nm respectively. Cross-sectional TEM samples containing implanted areas were prepared using liftoff technique [4]. First isolated amorphous clusters were observed at implantation dose 1.0E+14 ions/cm². This value indicates the amorphisation threshold for 30 keV Ga ions in silicon. The average distance from the surface to these clusters was found to be 19 nm which correlates with peak position of the damage profile obtained from TRIM simulations. The cross-sectional images of the damage layers for incident angles 4° and 90° and higher implantation doses are shown on Fig1. The relationship between the thickness of the amorphous damage layer and implantation dose is shown on Fig.2. Initially, for both implantation angles the thickness of the amorphous damage layer was growing very fast and then saturated with the dose around 2.0E+16 ions/cm² achieving 22 and 64 nm respectively. The interaction between the ion beam and the Si target results in two competitive processes – damage formation and sputtering and can be described by simple equation. From this equation the maximum ion dose accumulated by the sample can be found as D_{max}= $0.5 \times R_p \times Y^{-1} = 2.3E + 16$ ions/cm² (Y- sputter yield) which correlates well with saturation dose obtained experimentally.

It can be also noted on Fig. 1 that there are dark layers below the amorphous damage layers. The concentration of point defects there is below the amorphisation threshold but high enough to cause small lattice distortion. The thickness of these layers was found to be ~ 5 nm for 4° incident angle. The thicker the amorphous damage layer the more electrons will be absorbed and scattered in arbitrary directions. These will lead to a rise in intensity of background noise on TEM images and significant contrast reduction [7]. The presence of the heavily damaged crystalline layers (second type of damage) in FIB prepared samples results in un-sharpening of atomic columns during high resolution imaging and drastic reduction in the quality of the lattice images.



Fig1.Amorphous damage layers in silicon for different implantation doses: 2.0E+14 ions/cm²(a,d), 5.7E+14 ions/cm² (b,e), 0.9E+15 ions/cm²(c-f) and incident angles $90^{\circ}(a-c)$ and 4° (d-f).





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