Supporting Information for "Anomalous plastic deformation and sputtering of ion irradiated silicon nanowires"

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Evaluation of the sputter yield

Figure 1: Image analysis protocol. (a) The SEM images are cleaned up with a $3 \times 3$ median filter and a gaussian unsharp mask filter with $\sigma = 1 \, \text{px}$ weighted with 60% [1]. Next the Otsu thresholding [2] algorithm is applied to extract the wire area from the background (b). The wire was separated from non-contiguous pixel clusters by a particle analysis algorithm (c) [3,4]. The particle analysis algorithm is used to turn the wire upright (d). Finally, the gray values of the rows were summed to get a diameter profile shown in graph (e). To compare the wires after different fluences, the maximum at the base is aligned. Only the area in between the indicated lines is evaluated, since thresholding at the tip and bottom of the wire is inaccurate.

A large effort was made to minimize the influence of slightly different focal planes and contrast settings in the SEM analysis, as this is crucial to the evaluation of the sputter yield. The image analysis shown in figure 1 produces reliable profiles of the diameter of the nanowires over their height. It was performed after each irradiation step. If these profiles
are well aligned, the change in diameter per ion fluence can be used to calculate the sputter yield (SY) from \( d_1 \) and \( d_2 \), the local diameters before and after irradiation, using:

\[
SY_{(1 \rightarrow 2)} = \frac{\Delta V_{12}}{\Delta A_{12} \cdot \Phi_{12}} = \frac{\pi (d_1 - d_2) \cdot \rho_{Si}}{2 \cdot \sin(45^\circ) \cdot \Phi_{12}} , \tag{1}
\]

with \( \rho_{Si} \) the atomic density of silicon, \( \Delta V = \pi h (d^2_1 - d^2_2)/4 \) the sputtered volume, \( \Delta A = h \cdot \sin(45^\circ) \cdot (d_1 + d_2)/2 \) the corresponding irradiated area and \( \Phi_{12} \) the fluence. All results are plotted over the average diameter \( (d_1 + d_2)/2 \). As the difference in diameters after irradiation of \( 1 \times 10^{16} cm^{-2} \) was only sightly larger than the SEM resolution, only the two subsequent fluence steps of \( 2 \times 10^{16} cm^{-2} \) each were evaluated.

**Sputtering at 100 keV and 300 keV**

![Figure 2: Sputter yield of Si nanowires as a function of the diameter obtained by simulating the irradiation with 100 keV Ar\(^+\) and 300 keV Ar\(^+\) with trandina (black/red circles). The respective ion ranges of in Si-bulk at 45° are calculated with SRIM. The measured data-points (black/red triangles) correspond to the average of hundreds of individual measurements grouped together every 10 nm, the ‘error bars’ indicate the standard deviation. The outliers and discontinuities in the measured data curves correlate with a low number of evaluated nanowires for those diameters and the change from one nominal diameter on the samples to another.](image-url)
Crystallinity of the nanowires after irradiation

Figure 3: b), c) SEM images of the nanowires irradiated at 300°C and room temperature respectively. a) and d) EBSD images taken with the e-beam focussed on the spot indicated in b) and c). The clear Kikuchi-lines in a) show that the nanowire is crystalline. The black shadow at the bottom of this image is the shadow of the nanowire on the EBSD detector. The absence of Kikuchi-lines in d) shows that the wires irradiated at room temperature are amorphized.

SEM images of plastic flow

Figure 4: SEM images of Si nanowires after various fluences of 100 keV Ar⁺ irradiation at room temperature, rotated at 45° to the ion beam. The images were all take at 45° from the same set of wires. The wire shown in the main text is the first one from the top on the far right. Only wires that remained reasonably straight and were free of other debris could be evaluated.
Evaluation of the mass-transport-rate (MTR)

The number of atoms per height can be calculated from the local radius assuming constant density. Weighting the local number of atoms with the height gives the center of mass. The effective mass-transport rate (in $\text{atoms} \cdot \text{nm/ion}$) required to account for deformation seen in the SEM images is equal to the movement of the center of mass per fluence. As in the sputter yield evaluation, the difference of the sum of all atoms in a nanowire was divided by the number of ions hitting the wire after the set fluence to determine the sputter yield. The sputtered atoms have to be discounted in the evaluation of the movement of the center of mass. The effective mass-transport rate ($MTR$) is thus calculated to:

$$MTR_{(1 \rightarrow 2)} = \left[ z_c \cdot \sum_i \pi r_i^2 h \cdot \rho \cdot z_i - z_c \cdot \sum_i \pi r_i^2 h \cdot \rho \cdot (z_i - z) \right]/N_{ion}. \quad (2)$$

The height of the center of mass $z_c$ can be calculated by first summing up the height weighted by the number of atoms $z_c \cdot N = \sum_i \pi r_i^2 h \cdot \rho \cdot z_i$ and dividing this by the number of atoms $N = \sum_i \pi r_i^2 h \cdot \rho$ in the nanowire. The sums are over all slices $i$ of height $h = 1 \text{ pixel}$ each. $N_{ion} = \sum_i (r_i + 2r_i) \cdot \sin(45^\circ) \cdot h \cdot \Phi_{12}$ is the number of ions that hit the nanowire in the irradiation of fluence $\Phi_{12}$ performed between making SEM images 1 and 2. The sputter yield could be calculated by $(N - 2N)/N_{ion}.$

As only few wires remained straight and could be evaluated, the values show a large spread that could not be mitigated by a large number of samples as was done in the evaluation of sputtering. The small diameters at the base of the wires contributed to the instability of the samples (see also the SEM images in the supplementary figure 4).
Figure 5: a) Illustration of the MRT calculation in equation 2). Moving $^1N$ atoms from their center of gravity $^1z_c$ to $^2z_c$ is equivalent to moving $\bar{N}$ atoms from their center of gravity $\hat{z}$ to $^2z_c$, subtracting sputtered atoms $^1N - ^2N$. b) Histogram of all results obtained by evaluating the plastic deformation as a mass-transport rate. The average mass-transport rate for all fluence-steps $\Phi_{12}$ (see main text) is plotted. Due to the large spread, there is neither a significant correlation between the mass-transport rate and the average diameter nor the ion energy (not shown).
Focussed ion beam dual-beam processing.

Figure 6: a) Schematic of picking up a nanowire of a growth sample with a micro-manipulator in a focused ion beam (FIB) System. The nanowire is glued to the micro-manipulator with e-beam deposited Pt and cut from the substrate with the focused Ga\(^+\) ion beam. To be able to orient and rotate the single nanowire in the ion irradiation chamber, the nanowire is glued to a gold microwire, also with e-beam deposited Pt. Finally the wire is cut from the micro-manipulator with the FIB. The SEM images in c) and d) show the respective situations sketched in a) and b). During this procedure there may be some Pt deposited on the wire, but as the temperatures are low, there will be very little intermixing of Pt and Si.

References

[1] B. Sankur, *Journal of Electronic Imaging* **2004**, *13* (1) 146. doi:10.1117/1.1631315.

[2] N. Otsu, *IEEE Transactions on Systems, Man, and Cybernetics* **1979**, *9* (1), 62-66. doi:10.1109/TSMC.1979.4310076.

[3] J. Schindelin; I. Arganda-Carreras; E. Frise; V. Kaynig; M. Longair; T. Pietzsch; S. Preibisch; C. Rueden; S. Saalfeld; B. Schmid; J.-Y. Tinevez; D. J. White; V. Hartenstein; K. Eliceiri; P. Tomancak; A. Cardona, Fiji: an open-source platform for biological-image analysis, *Nature Methods* **2012**, *9* (7), 676-682. doi:10.1038/nmeth.2019.

[4] D. Sage; D. Prodanov; J.-Y. Tinevez; J. Schindelin; Daniel Sage, in: ImageJ User & Developer Conference (IUDC’12) **2012**, Conference Proceedings.