TEM Characterization of Etched Si Nanopillars Prepared by Small Angle Cleavage Technique

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Introduction

Silicon nanowires are promising candidates for future device applications, e.g. in single-electron devices. Shrinkage of nanopillars by subsequent dry thermal oxidation enables the fabrication of silicon nanostructures in the sub-10 nm regime. Such structure sizes are required for semiconductor devices, which make use of quantization effects. The electrical properties of these nanostructures are strongly affected by etching-created defects and by their surface properties.

To characterize the defect structure of such nanopillars they have to be studied by means of transmission electron microscopy (TEM) in cross-sectional view. The problem here is that the traditional cross-section sample preparation method is difficult to apply (see below). The aim of this paper is to test the Small Angle Cleavage Technique (SACT) for preparation of artefact free TEM studies on silicon nanopillars.

Fabrication of Silicon nanopillars

Silicon nanopillars were fabricated by an CF$_4$ based highly anisotropic reactive ion etching (RIE) process. This process uses a lithography technique utilizing gold colloidal particles (or citrate Au sol [1]) with 50nm diameter as etching mask. The gold particles are chemically bonded to the substrate using a aminofunctional silane [3-(2-Aminoethylamino) propyltrimethoxysilane] (APTS) as a coupling agent. The gold particles are statistically ditributed on the substrate surface [2] (Fig. 1).The deposition process and the RIE etching process are described in detail in [3] and [4], respectively.

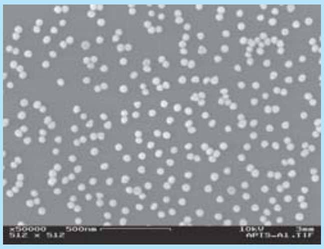


Fig. 1: SEM picture of 50 nm colloidal gold particles on silicon substrate

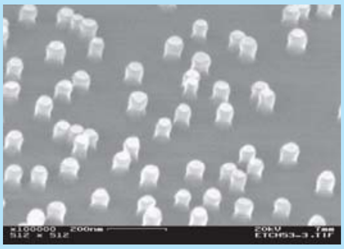


Fig. 2: SEM picture of etched Si nanopillars. On top of the pillars the gold particles

Experimental

TEM sample preparation by the Small Angle Cleavage Technique

5 Steps to prepare a XTEM sample by SACT (described in detail in [5], [6], [7]):

1) Mount sample surface down on a platen and polish at a small angle to the edge of the sample or to a cleavage plane (if you have an oriented sample) to a final thickness of about 100 µm. Polishing will generate grooves, wich act as orientation for scribing in the next step.

2) Scribe the sample along polishing induced grooves and remove from the platen. After short cleaning in acetone bath, cleave the sample along the sribes (see Fig. 3). For cleaving place the sample on a glass slide and align the scratch marks along the edge.

Fig. 3: Charting of first sample cleaving

3) Take one of the cleaved stripes with a smooth fracture edge and place it on a glass slide. Scribe parallel to the normal cleaving direction of sample and cleave a little wedge (Fig. 4).

4) To get a good TEM sample it is necessary to preinvestigate the wedges using a light microscope. The sharper the apex of the wedge, the better the sample (Fig. 5).

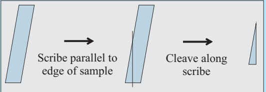


Fig. 4: Charting of second sample cleaving

5) Mount the wedge on a 30 µm thick copper ring. Because of the difficult use of these thin rings the ring is stabilized by small pieces of silicon (100µm thickness) and another copper ring which are stick to the ring with epoxy glue. The sample is fixed between the silicon pieces, the surface in vertical direction. The second copper ring is cut as seen in the Fig. 6a to be able to take up the sample (Fig. 6). Overlapping parts of the wedge are removed after baking.

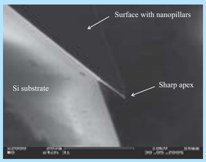


Fig. 5: SEM picture of apex of cleaved wedge

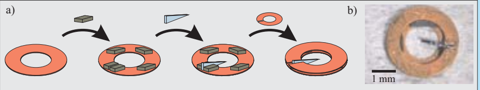
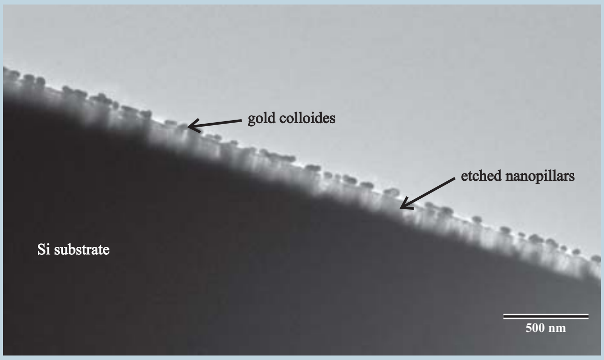


Fig. 6: a) Charting of mounting the cleaved wedge on copper rings.
b) Picture of the ready sample (real sample).

[1] N. Uyeda, M. Nishino, E. Suito, J. Colloid Interface Sci. **43**, 181 (1973), [2] P. A. Lewis, H. Ahmed, T. Sato, J. Vac. Sci. Technol. B **16(6)**, 2938 (1998), [3] T. Sato, D. G. Hasko, H. Ahmed, J. Vac. Sci. Technol. B **15(1)**, 45 (1997), [4] G. S. Oehrlein, J. F. Rembetski, IBM J. Res. Develop. **36(2)**, 140 (1992), [5] J. P. McCaffrey, Ultramicroscopy **38**, 149 (1991), [6] J. P. McCaffrey, Microscopy Research and Technique **24**, 180 (1993), [7] S. D. Walck, J. P. McCaffrey, Mat. Res. Soc. Symp. Proc. **480**, 149 (1997)

Results

SACT

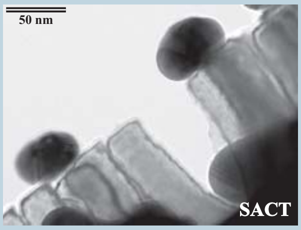


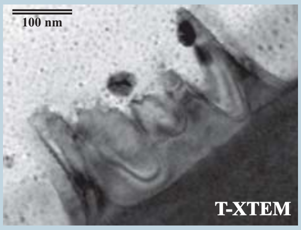
Overview of a cross-section of Si nanopillars. Below Si subtrate, in the middle the etched Si nanopillars with gold colloides on top. Note the wide range of electron transparency.

Good structural transference is clearly seen.

Thin amorphous layer around nanopillars.

COMPARISON





SACT to Traditional Cross-Section TEM (T-XTEM) Sample Preparation

Using the T-XTEM sample preparation method, artefacts mainly by argon ion beam thinning and face to face glueing are generated.

Conclusion

It was found :
Using the traditional cross-sectional sample preparation method, a number of artefacts were produced, which unable a structural investigation of the nanopillars.

The small angle cleavage technique was found to be an excellent and fast method to prepare cross-sectional TEM samples of nanopillars without artefacts. It was found that the nanopillars are crystalline.The interface between nanopillar and Au particle was resolved. An amorphous layer with a thickness of 2 nm to 4 nm around the nanopillars could be clearly identified.

We already applied the method to GaAs substrates successfully.

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