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Transient-Enhanced Surface Diffusion on Natural-Oxide-Covered Si(001) Templates during Vacuum Annealing

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We report on the transient-enhanced shape transformation of nano-structured Si(001) surfaces upon *in vacuo* annealing at relatively low temperatures of 900 – 950 °C for a few minutes. We find dramatic surface mass transport concomitant with the development of low-energy facets on surfaces that are covered by native oxide. The enhanced surface mass transport ceases after the oxide is completely desorbed, and it is not observed on surfaces where the native oxide had been removed by HF before annealing.

Introduction

Commercial ultra-large-scale integrated circuits have reached physical gate lengths well below 100 nm, and self-organization phenomena are explored as an alternative route toward the fabrication of even smaller device structures. However, the fabrication of such small structures is only one precondition for shrinking device dimensions. It is as important to preserve their size, shape and electronic properties during subsequent device processing. Here we concentrate on the shape stability of Si nanostructures during vacuum annealing at around 900 °C for a few minutes. Such thermal steps are typically employed for native oxide desorption prior to epitaxial growth, but similar thermal budgets are frequently required during device processing, e.g. after ion implantation.

While the shape evolution of structured Si surfaces is well described [1], [2], most of the experimental studies employed long-term, high-temperature anneals. Also, quite often exotic annealing procedures (e.g. a flash to 1200 °C [3]) and direct current heating (prone to electro-migration artifacts [4]) were used. Here we employed only cleaning and annealing procedures adapted from standard Si device processes.

Experimental

Sample Preparation

Our studies were concentrated on samples consisting of periodic wire arrays of rectangular cross section fabricated by holographic lithography and subsequent reactive ion etching on Si(001) substrates (Fig. 1 (a)). Periods were varied between 400 – 2000 nm at etch depths of typically 250 nm. After photoresist stripping, the samples underwent an RCA clean with or without a final HF-dip immediately before transfer into the UHV environment of an MBE system.

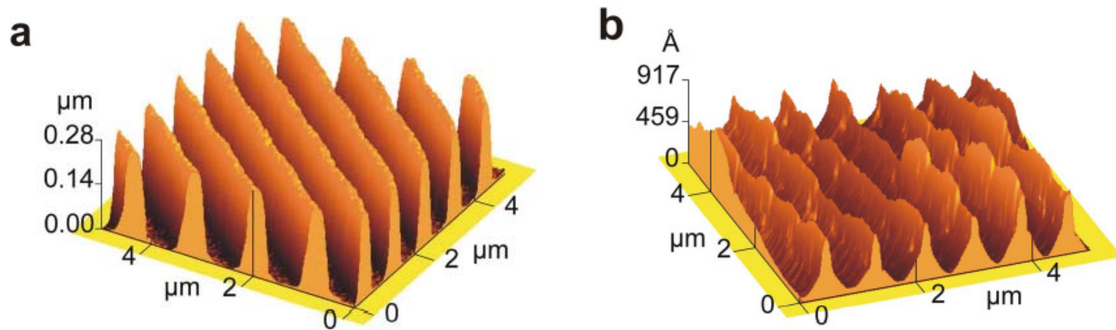

Fig. 1: 3D-AFM images of rectangular wire-template as processed (a) and after 4 min annealing @ 950°C without previous HF-dip (b).

Measurements

In the UHV environment of the MBE system the samples were radiatively annealed for 1 – 5 min at 900 – 950 °C, which corresponds to the standard oxide desorption step prior to epitaxial growth [5], [6]. On all samples covered by native oxide the originally rectangular profiles were transformed into {311}-faceted trapezoids concomitant with a loss of up to 80% of the peak-to-valley modulation (Figs. 1 (b), 2 “RCA”). In contrast, samples that had the native oxide removed by HF showed no significant morphological changes (Fig. 2 “HF”).

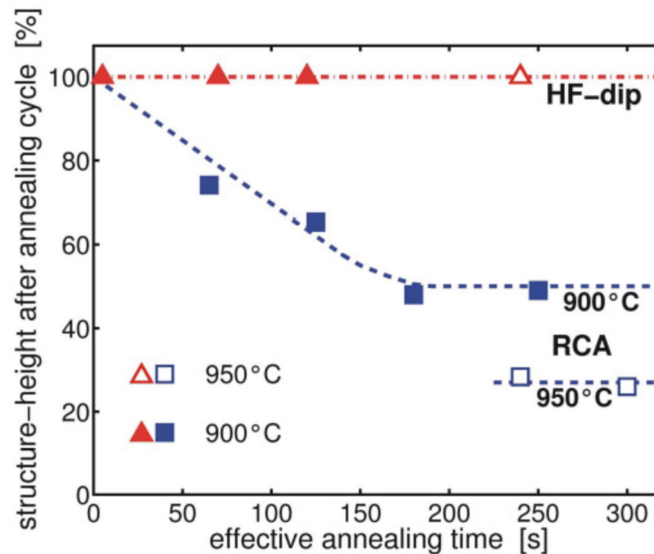

Fig. 2: Reduction of structure-height after different annealing cycles.

We followed the kinetics of faceting as a function of the annealing conditions by high-resolution XTEM imaging (Fig. 3), and compared the results to Monte-Carlo-type simulations that used the surface energies of the {111} and {311} facets as the only input parameters. We found good agreement, which is indicative of a shape transformation that behaves as expected near thermal equilibrium [1], [7], despite the fact that our experiments were conducted 500 °C below the melting point of Si.

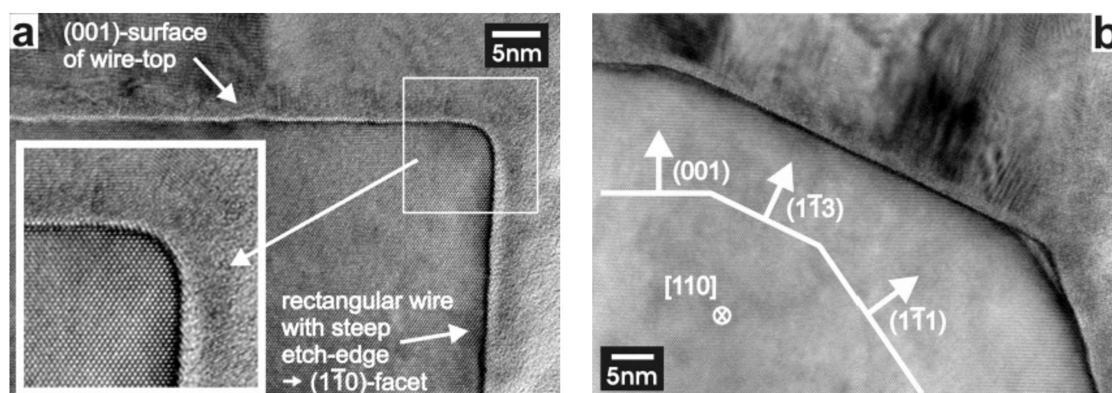

Fig. 3: High-resolution XTEM-images of rectangular and faceted wire-edges, as processed (a) and after 2 min annealing @ 900°C without previous HF-dip (b).

Since only oxide-covered structures reveal the transient enhanced shape transformation, the reaction [8] Si + $SiO_2 \rightarrow SiO\uparrow$, on which thermal oxide desorption at 900 °C is based, is most likely involved in this phenomena. This reaction takes place everywhere at the Si/SiO_2 interface, but it has been found that the oxide desorption reaction occurs mainly at the periphery of voids, which form in the early stages of thermal decomposition in the oxide layer [9]. No correlations between void nucleation and shallow structures at the Si surface have been found yet. However, it is not clear, whether this applies to our nanostructures, which provide almost atomically sharp intersections between two low-index planes (Fig. 3 (a)).

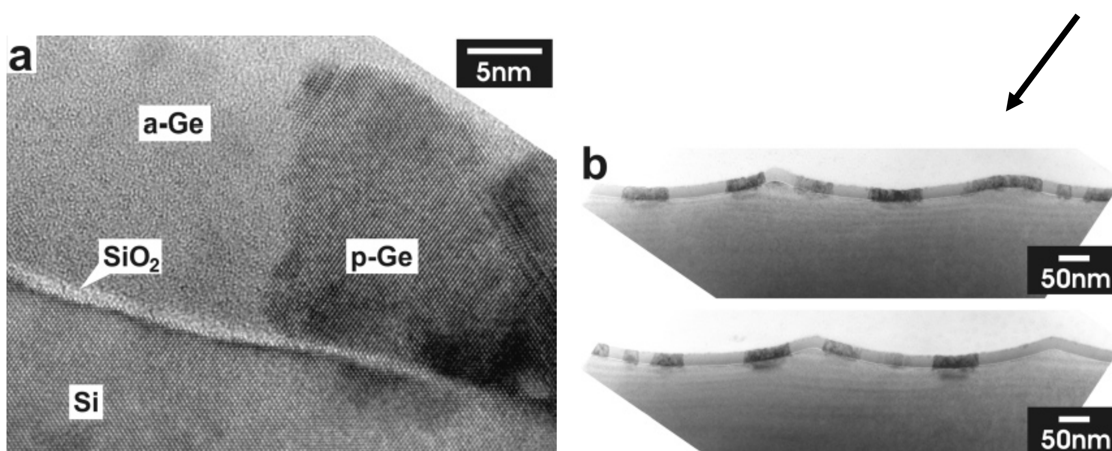

Fig. 4: (a) High-resolution XTEM-image of wire-template with a period of 400 nm after 1 min annealing @ 900 °C with posterior Ge-overgrowth at temperatures below 175 °C. On the SiO_2 covered Si structure amorphous Ge is formed, whereas polycrystalline Ge indicates regions of oxide-desorbed surface parts.
(b) Low resolution TEM images indicate that oxide break-up shows no correlation with the wire template.

In order to distinguish whether the shape transformation is linked to void formation or occurs beneath a still existing layer of oxide, we have developed an XTEM decoration technique. Covering annealed (1 min @ 900 °C) wire templates in situ with low temperature Ge leads to poly-Ge growing in the substrate-exposing voids, and to amorphous Ge on areas that are still covered by native oxide (Fig. 4 (a)). The p-Ge areas can easily be identified in XTEM images because the strain contrast makes them ap-

pear darker (Fig. 4 (b)). In addition, the mass contrast allows a straightforward identification of the SiO_2 layer, which appears as alight stripe.

Figure 4 (b) shows that no correlation whatsoever exists between void formation and the template structure: Most of the remnants of the wires are still buried underneath a continuous SiO_2 film. That means, the shape transformation takes place predominantly below the oxide, and the oxide follows this transformation. The voids do have some influence: The one ridge structure that accidentally coincides with a void in the oxide (arrow in Fig. 4) appears even flatter than the oxide-covered ridges. This effect is most likely responsible for the roughness overall in Fig. 1 (b).

Conclusion

Shrinking the dimensions of semiconductor devices structures to the nanometer range, the preparation and conservation of small morphological features becomes increasingly relevant. The surface free energy of a structured heterosystem is determined by composition, crystal orientation, and strain. The interplay between these parameters is widely exploited for the fabrication of self-organized nanostructures. Pre-patterned substrates can enhance ordering of these sub-micron structures. Here we report on the morphological integrity of such templates under chemical and thermal treatments typically employed during device processing. The results show that even proven and supposedly uncritical process steps can drastically affect the morphology of nanostructures.

Acknowledgements

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References

[1] J. M. Bermond, J. J. Métois, X. Egéa, F. Floret; Surf. Sci. **330** (1995), 48 and references therein

[2] T. Suzuki, J. J. Métois, K. Yagi; Surf. Sci. **339** (1995), 105

[3] S. Tanaka, C. C. Umbach, J. M. Blakely, R. M. Tromp, M. Mankos; J. Vac. Sci. Technol. **A15** (1997), 1345

[4] L. V. Litvin, A. B. Krasilnikov, A. V. Latyshev; Surf. Sci. Lett. **244** (1991), L121

[5] E. Kasper, M. Bauer, M. Oehme; Thin Solid Films **321** (1998), 148

[6] E. A. Gulbransen, S. A. Jansson; Oxid. Metals **4** (1972), 181

[7] M. E. Keeffe, C. C. Umbach, J. M. Blakely; J. Phys. Chem. Solids **55** (1994), 965

[8] R. E. Walkup, S. I. Raider; Appl. Phys. Lett. **53** (1988), 888

[9] D. Jones, V. Palermo; Appl. Phys. Lett. **80** (2002), 673