

Nanofabrication by Metal Assisted Chemical Etching of Silicon in Gas Phase

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High aspect ratio nanostructuring requires high precision pattern transfer with highly directional etching. In this work, we demonstrate the fabrication of structures with unprecedented ultra-high aspect ratios up to 10'000:1 (see Fig.1) in the nanoscale regime (down to 10 nm) by metal assisted chemical etching (MacEtch) of silicon in gas phase.

The etching gas is created by a vapor of the water diluted hydrofluoric acid and a continuous air flow,¹ which works both as an oxidizer and as a gas carrier for reactive species. The high reactivity of platinum as catalyst and the formation of platinum silicide to improve the stability of the catalyst pattern allow a controlled etching. The method has been successfully applied to produce straight nanowires with section size in the range of 10-100 nm and length of hundreds of micrometers. X-ray optical elements with feature sizes down to 10 nm and etching depth in the range of tens of micrometers were successfully fabricated, too. Since it is a “dry” process, it can be used for stiction sensitive applications without requiring a supercritical drying steps. In this work, random nanowires were produced (Fig.1) by MacEtch of direct self-assembly holes array in thin Pt film (8-20 nm thick).² Patterned structures were instead produced by e-beam lithography or projection photolithography. Micro-structures such as high aspect ratio linear grating used for X-ray interferometry and Fresnel zone plates (Fig.2) have been etched in low resistivity N-type Si (0.001-0.01 Ωcm) with a reduced porosity and less defects compared to the wet MacEtch process.

The method is an interesting and promising low-cost technology for producing high aspect ratio nanostructures bypassing the nanoscale limits of reactive ion etching. It has high potential as nano- and micro-fabrication technique for applications, where silicon very high aspect ratio nanostructures and high precision of pattern transfer are required, for example X-ray optics, photonics and MEMS.

¹ L. Romano, M. Kagias, J. Vila-Comamala, K. Jefimovs, L.-T. Tseng, V. A. Guzenko, and M. Stampanoni, *Nanoscale Horizons* **5**, 869 (2020).

² L. Romano, M. Kagias, K. Jefimovs, and M. Stampanoni, *RSC Advances* **6**, 16025 (2016).

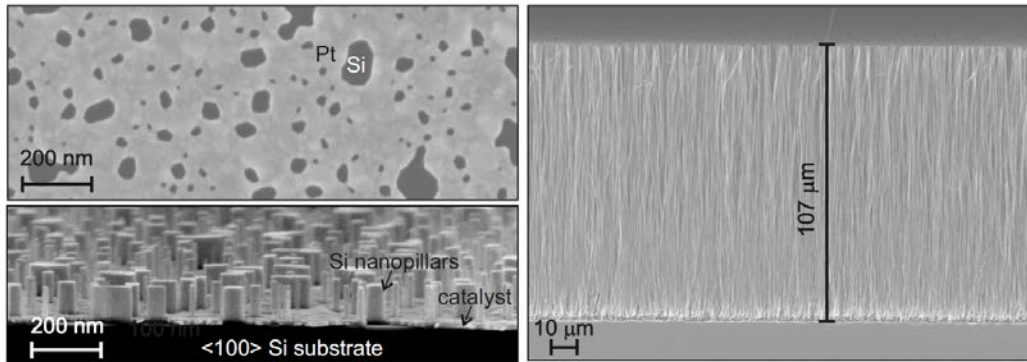


Figure 1: Plan-view SEM image (left up) of Pt dewetting pattern (10 nm Pt annealed at 450°C) used to etch Si nanowires; cross-section SEM images of the sample after 10 min (left bottom) and 4 hours (right) of MacEtch in gas phase at 40°C. The Pt dewetting pattern is exposed to the etching atmosphere that is a mixture of HF and air. The Pt catalytically decomposes O₂ present in the flowing air, HF removes the oxidized Si so that the Pt layer progressively deepens into the Si substrate producing Si nanostructures with feature size of the original Pt pattern.

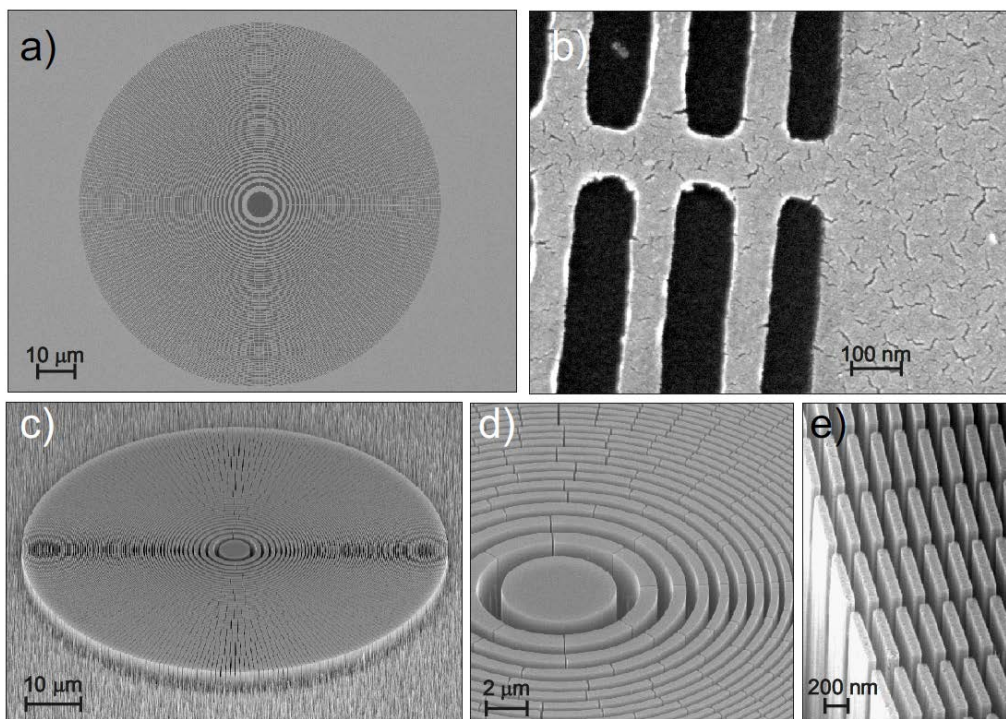


Figure 2: Fresnel zone plate: plan-view SEM images (a, b) of metal pattern after lift-off and annealing at 250°C, details of outmost zone, the cracks in the metal layers are consequence of dewetting and generate nanowires during MacEtch, producing a uniform movement on the metal pattern inside the Si substrate. Tilted view SEM images (c-e) after MacEtch in gas phase. The example shows the capability of uniform etching rate despite the feature size variation from center to border, with the outermost zone width decreasing down to 100nm (e).