Effect of Metal-Assisted Catalytic Etching (MACE) on Single-Crystal Si Wafers With Faceted Macropores

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The applications for silicon span a wide variety of fields including electronics, drug delivery, and energy storage. In particular, porous silicon (por-Si) is of great interest in optoelectronic and energy-harvesting applications due to the ability to absorb and scatter light. Additionally, Si possesses the greatest specific capacity to alloy with lithium making this element favorable for battery applications [1]. A common method used to create por-Si is metal-assisted catalytic etching (MACE), which exploits the enhanced kinetics of electron transfer at electrolyte/metal interfaces [2]. This method promotes both local and remote etching that are dependent on reaction conditions such as the HF:H_2O_2 ratio, the catalyst, and the geometry [3,4].

Here we consider Si wafer substrates in three crystallographic orientations: (100), (110), and (111). Prime grade silicon wafers of 500 μm thickness were ablated using a Spectra-Physics Quanta Ray INDI-HG- 205 Nd:YAG laser. Following ablation, wafers were chemically etched using KOH(aq) solution to create crystallographically-defined macropores. MACE was achieved using Ag nanoparticles as the catalyst with a HF + H_2O_2 solution as the etchant. The wafers were examined in two conditions: after chemical etching and after subsequent MACE etching. These studies were used to reveal the size distribution, morphology and crystallographic orientations of the etch track pores.

Figures 1A-1F are secondary electron SEM images that show the overall morphology of the chemically-etched macropores, and the fibrous topography of the MACE-etched surfaces. These latter surfaces are consistent with the interconnected Si nanowire morphologies reported elsewhere [5]. The details of these structures were investigated using FIB-cut cross-sectional TEM specimens prepared in a dual-beam FEI Helios NanoLab 460F1 Ga^+ FIB-SEM. Sequential e-beam and ion-beam Pt deposition was used to protect the delicate porous surface features. Examples of BF and HAADF STEM data obtained for one such specimen from a MACE-etched (110) Si-wafer are shown in Figures 2. These data were obtained using a Talos F200X operating at an accelerating voltage of 200kV. Such images reveal the orientations of the etch-track pores and the presence of Ag nanoparticles at the tips of the etch tracks.

Reference:

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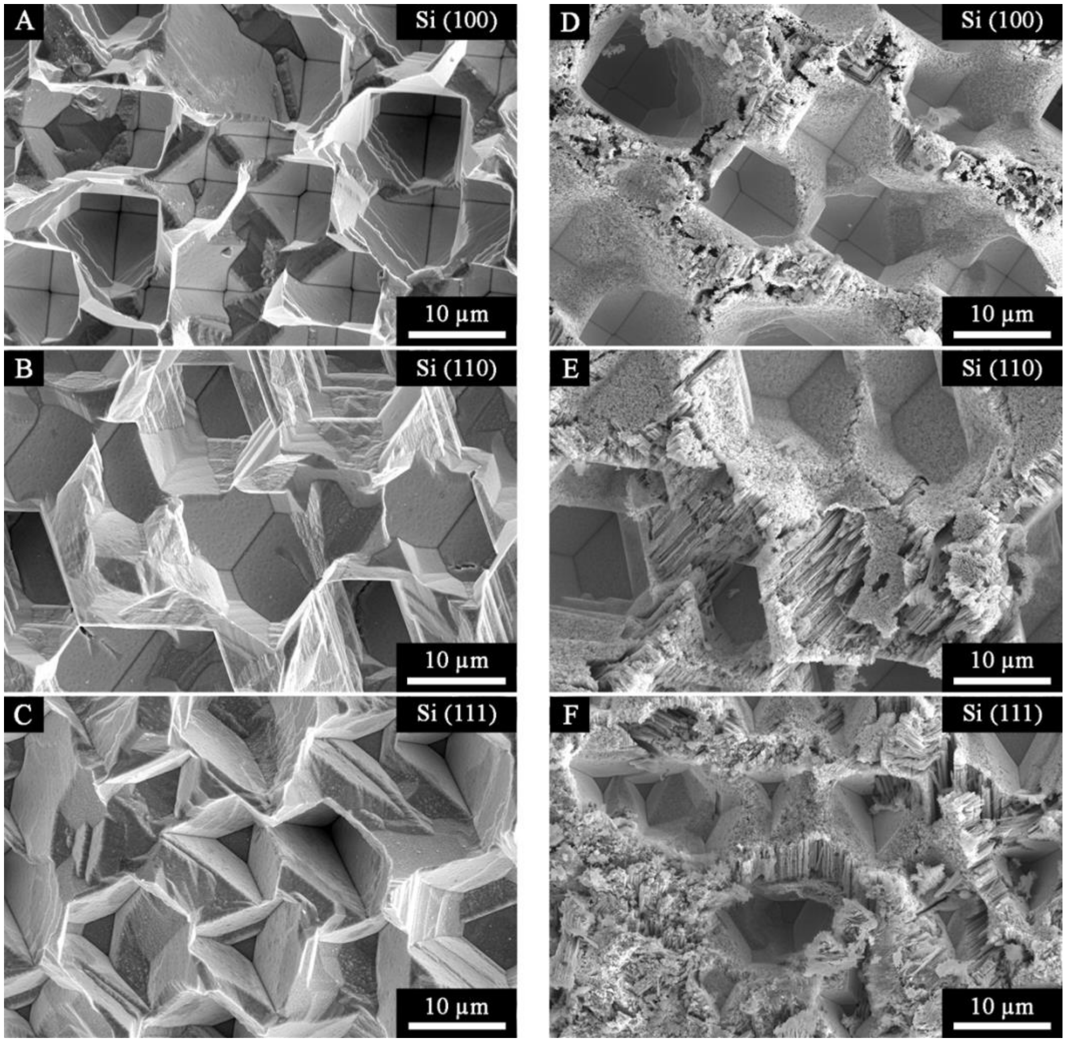

Figure 1. Comparison of surface topography before and after MACE on Si wafers (100), (110) and (111). A)-C) are chemically etched macropores. D)-F) are MACE etched macropores.

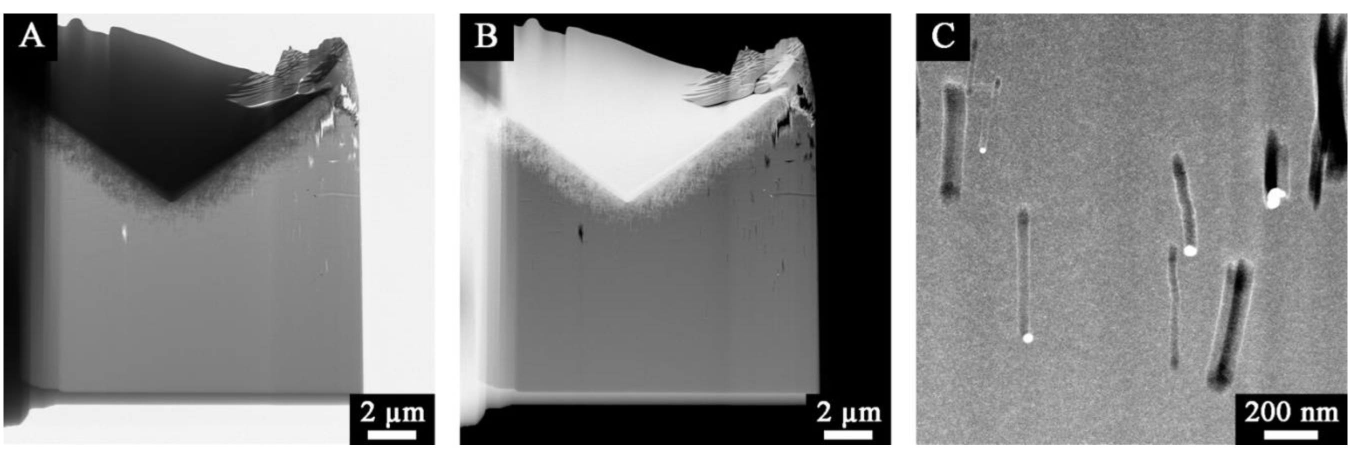

Figure 2. Cross-sectional STEM data from a MACE-etched (110) Si wafer with macropores. A) BF image. B,C) HAADF STEM images. The image in (C) reveals Ag nanoparticles at the tips of etch track pores.