Porous Silicon Filters: Fabrication and Metallization

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Introduction

Porous silicon (PS) films formed during the anodic etching of silicon in HF have applications in a myriad of fields ranging from electronics to sensors, microreactors, and photovoltaics.

The fabrication of PS-based films involves the controlled removal of a PS layer from an etched silicon surface in a manner similar to the anodization and removal of alumina films. Depending on the etch conditions, the etching process in PS can produce a much broader range of distinct pore sizes and film thicknesses than can be obtained with alumina films. PS films thus present an extremely versatile material that can be used as operable filtration devices at high temperatures where polymers rapidly degrade. We have demonstrated here the creation of PS films with thicknesses in the range 4 – 70 μm and pore diameters ranging from one to two microns. Further, we also demonstrate a method for the metallization and reactive coating of these devices.

Procedure

PS films were created using monocrystalline boron-doped (p-type) silicon wafers with resistivites between 14 and 22 Ωcm. In preparation for the etch, the wafers were cleaned with HF, rinsed with methanol to remove the HF, and rinsed with trichloroethylene to remove organics. The back of the wafer was coated with conductive spray paint to reduce contact resistance.

The etching took place in an electrochemical cell consisting of a carved block of high-density polyethylene with a platinum cathode. The cell contained 49% aqueous HF (with volumetric concentration between 1.5 and 4.5%) in DMF. A constant current etch density between 6 and 10 mA/cm^2 was applied for times up to one hour.

Later, an electroless platinum plating of the PS films was performed. A bath consisting of disodium hexahydroxyplatinate (10g/L), sodium hydroxide (5g/L), ethylene diamine (10g/L), and hydrazine monohydrate (1g/L) was used. Some of the samples were not cleaned; others were soaked in dilute HF (30 minutes) or dilute HCl (3 hours) and then cleaned with methanol. The filters were then submerged in the electroless platinum bath for times ranging from 25 seconds to five minutes.

Results and Discussion

During the etching process we found that pore diameter and porosity depended greatly on anodization conditions. The pore diameter is affected by both the etch solution and the doping of the silicon wafer. For wafers of 14-22 Ωcm resistivity, pore diameters are approximately 1μm in diameter, varying little with the current density. Porosity can be increased by increasing the current density or by using high concentrations of HF (>10%). The filters that are separated successfully from the substrate all have pore diameters of ~1 μm and a porosity of ~30%, although their thicknesses vary with change in etching conditions.

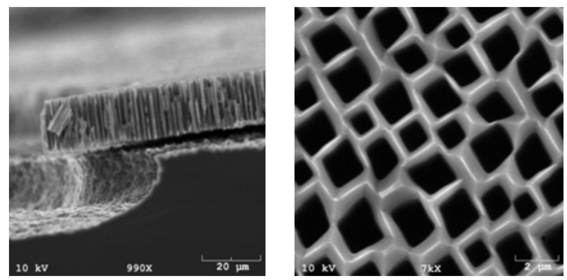

Figure 1: (Left) SEM image of a filter cross-section. (Right) Back view of the PS filter showing the pyramidal structure of the pores

Filter lift-off was first obtained using an etch cell with a $1cm^2$ etch area and 2cm electrode spacing, an HF concentration of 3%, and a current density of $6mA/cm^2$. When the etch was performed in a larger cell ($4cm^2$ etch area, 1.25cm electrode spacing), decreasing the HF to 1.5% was required to achieve lift-off. However, increasing the electrode spacing to 3.25cm allowed lift-off with HF concentrations up to 4.5% at $10mA/cm^2$, indicating that the separation process depends on electrode separation. Films are separated from the substrate by a thick white foam. An EDAX scan of this foam indicates that it is a silicon oxyfluoride-based polymer. The formation of this foam appears to somehow aid the separation process.

An EDAX spectrum of the PS filters after metallization indicates that there can be in excess of 2% by weight platinum incorporated into the films. It still remains to be established if cleaning the samples before they are coated affects the coating process.

Conclusion

We have demonstrated the potential for the fabrication of PS filters with a huge variety of pore shapes and sizes that can be operated at elevated temperatures with potential applications in microreactors, photovoltaic technology, and other areas. Subsequent experimentation will focus on expanding the range of pore diameters that can be produced and fine-tuning the metallization process.