Fabrication of photonic crystals using a spin-coated hydrogen silsesquioxane hard mask*

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We present a method for creating a hard mask for the dry etching of microphotonic structures and devices. We demonstrate that spin-on glass [hydrogen silsesquioxane (HSQ)] has sufficient dry etch resistance to allow the creation of high-quality, deeply etched photonic crystals. Furthermore, HSQ is a more favorable hard mask for the creation of active devices than plasma-enhanced chemical-vapor deposition (PECVD) silica, as less damage is incurred. It is also an economic and convenient replacement for PEVCD in photonic crystal fabrication. We examine this method and show that it can create photonic crystals of equivalent quality to those created using PEVCD masking. © *2006 American Vacuum Society.* [DOI: 10.1116/1.2164850]

I. INTRODUCTION

Compact active devices are essential components of microphotonic circuits. Tunability on a micron-size footprint, for example,[1–3] gives advantages in terms of power consumption and tuning speed, as parasitic capacitances can be kept low. Microphotonic gain elements, such as ultrasmall lasers[4,5] and amplifiers, are other examples for devices that combine challenging fabrication with the need for electrical contacts on a small scale. III-V materials are the prime candidates for the realization of such devices as they offer both gain and efficient tuning (via both plasma and band-filling effects). One of the key requirements on electrical contacts is for their resistance to be low; otherwise, Ohmic heating reduces the efficiency of gain elements, and causes undesirable thermal tuning. Ohmic heating is especially emphasized by the small device size. The fabrication process should therefore not compromise the electrical injection properties of the material.

The second aspect addressed in this article is the complexity of the fabrication process, since the fabrication of microphotonic devices in III-V semiconductors typically requires several processes—hard mask deposition [typically SiO_2 or SiN deposited using sputtering or plasma-enhanced chemical-vapor deposition (PECVD)], pattern definition [e.g., using electron-beam lithography with polymethylmethacrylate (PMMA)], pattern transfer into the hard mask [usually by reactive ion etching (RIE) with fluorine chemistry], and finally the deep etch [using RIE, chemically assisted ion-beam etching (CAIBE), or inductively coupled plasma (ICP) etching]. Each of these steps can be a complex process requiring expensive machinery. Any simplification of this process offers advantages in both versatility and cost, particularly for the relatively small scale production typical in research institutes. Here, we introduce and characterize a process step that offers advantages in both aspects. Spin-on glass, in particular, hydrogen silsesquioxane (HSQ), sourced from Dow Corning as "flowable oxide" FOx-12, is shown to provide a durable hard mask for the high-aspect-ratio etching of photonic crystal patterns that also results in lower contact resistances than a comparable mask deposited by PECVD. Furthermore, it offers the flexibility of direct pattern generation by e-beam lithography as it acts as a negative resist for electron exposure[6] and produces very high resolution features.[7,8]

The PEVCD process used for the hard mask deposition usually results in damage to the surface of the material. This damage increases the electrical resistance of the finished device and generates additional heating during operation. In this article, we report on a method of masking which avoids this problem. This method uses HSQ that is applied through spin coating, thus avoiding the use of the PEVCD and its consequent drawbacks.

II. BACKGROUND

Hydrogen silsesquioxane consists of a molecular network of H–Si–O bonds.[9] As might be expected, the molecular structure has a very strong influence on its physical properties. Depending on the curing temperature, it may have a cage structure (low temperature ~100 °C) or a network structure (when the temperature is sufficient, ~400 °C, to start dissociating the Si–H bonds).[10] The ratio of Si–O to Si–H bonds has been shown to be the critical factor governing the hardness, which increases with increasing Si–O fraction. This ratio, in turn, is proportional to the curing temperature.[11] The ambient atmosphere is also important—for low O_2 levels; the dissociation rate of Si–H bonds is reduced.

III. FABRICATION

A commercially available HSQ resist (FOx-12, flowable oxide from Dow Corning) was used to create the masks. The sample is spin coated at 5000 rpm, prebaked at 100 °C for 1 min in order to drive off solvents, and then baked at

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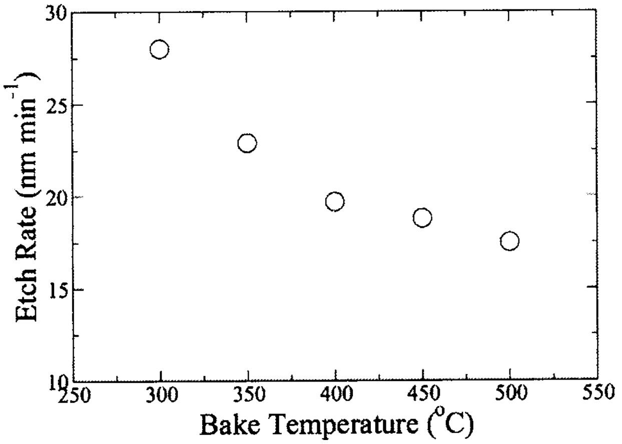

FIG. 1. Etch rate of HSQ silica as a function of bake temperature. The transformation to silica goes further for high temperature. Each sample was spun with a single layer of FOx-12, prebaked at 100 °C for 1 min, baked at 300 °C for 5 min, and finally cured at different temperatures for 1 h each. (The CAIBE conditions were those optimized for etching photonic crystals in GaAs.)

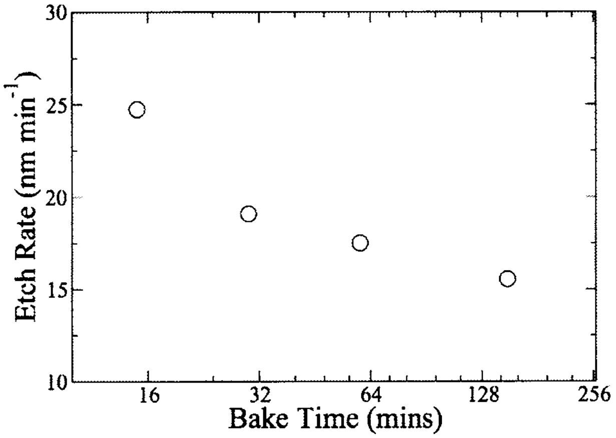

FIG. 2. Etch rate of HSQ silica as a function of bake duration. The duration of the final bake (at 500 °C) was varied between 15 and 150 min. (The CAIBE conditions were those optimized for etching photonic crystals in GaAs.)

300 °C for 5 min. This is repeated three times so as to build a silica mask of the thickness required for deep etching (typically approximately 300 nm). It is finally baked at 300–500 °C for 1 h. This last step cures the resist, dissociating the Si–H bonds, largely transforming the film into silica.

In order to compare the surface damage caused by the spun-on HSQ mask and the PEVCD silica mask, we examined the electrical resistance of samples created using the two techniques. This, we believe, is a valid indication of the damage caused. Three samples were examined; one sample with a HSQ mask (cured at 400 °C for 1 h, one sample with a PEVCD silica mask, and one control sample (without any mask). Using a HF wet chemical etch the respective masks were removed (where necessary). Using photolithography and a lift-off resist, the desired contact pads were defined. The samples were then deoxidized in HCl and Ni/Au contacts were deposited in an electron-beam evaporator. No annealing was carried out, since in some situations this can be detrimental to photonic crystal devices.[12]

The photonic crystals shown here were defined in PMMA using a Leo 1530/Raith Elphy Plus electron-beam writing system. The pattern was then transferred into the HSQ/silica mask using RIE with fluorine chemistry and finally deeply etched using CAIBE.

IV. RESULTS

The most important characteristic of an etch mask is its physical resistance (hardness).

To examine this, a number of samples were made up. The above steps were followed except for that both the temperature and duration of the final bake were varied.

The etch resistance has a strong dependence on the temperature of the high-temperature bake (see Fig. 1). This is in agreement with Ref. 11, which reports a linear dependence of the hardness on curing temperature. The curing time is also important, as demonstrated by Fig. 2. After an initial rapid decrease with curing time, the etch rate asymptotically converges to a value that is presumably the etch rate of pure silica. The HSQ silica used in our etching is, thus, slightly less resistant to etching than conventional silica. By curing at even higher temperatures and/or for longer times, equivalent performance to PEVCD silica should be possible as all the S–H bonds break. However, the highest curing temperature that we have used is 500 °C, as we believe that curing at higher temperatures in an oxygen-rich atmosphere may compromise device performance.

In order to determine the contact resistances of the respective samples, the transmission line measurement method[13] was used. Contact pads with varying separations were created on each sample for this purpose. The resistance between two adjacent pads is then given by

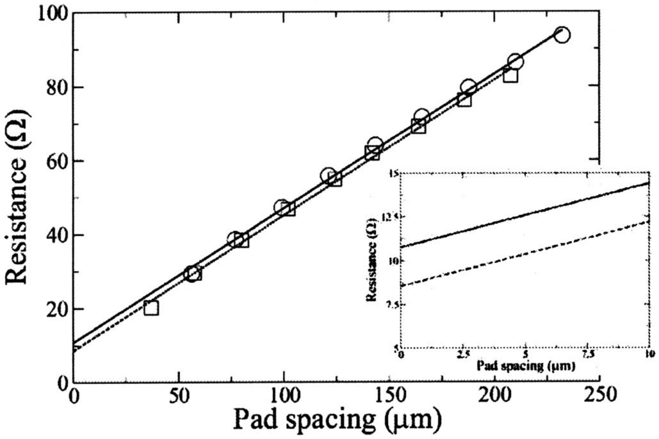

FIG. 3. TLM measurement of the contact resistance of PEVCD silica masked device (circles with the fit as a solid line) and that of HSQ silica masked device (squares with the fit as a dashed line). The contact resistance can be determined from the y-axis intercept (see inset).

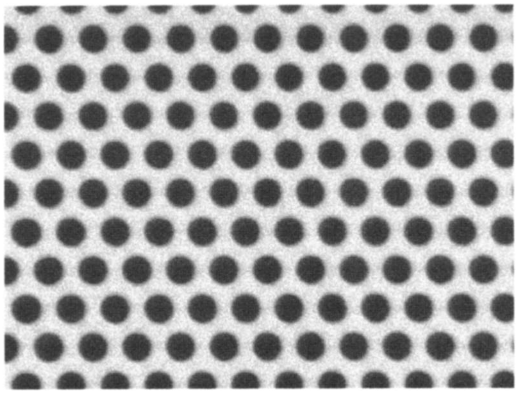

FIG. 4. Two-dimensional photonic crystal pattern created using silica. The period is 400 nm and the hole size is 310 nm.

$$R_T = \frac{R_s}{W} L + 2R_c, \tag{1}$$

where R_T is the measured resistance, R_s is the sheet resistance, W is the width of the contact pads, L is the separation, and R_c is the contact resistance. A four-probe measurement of the resistance for adjacent pairs of pads was made and the results are plotted in Fig. 3. The four-probe measurement is necessary in order to eliminate errors due to the resistance between the probes and contacts. The contact resistance can then be determined from the intercept [see Eq. (1)]. The slope of the plot gives the sheet resistance.

The PEVCD and HSQ samples had the same sheet resistance, as might be expected. For the PEVCD sample, a value for the contact resistance of 5.4 Ω was obtained. The HSQ sample had a resistance of 4.4 Ω—within the experimental error this was equal to that of the control sample. As the HSQ silica is spun on, it avoids the damage caused by the plasma present during PEVCD, resulting in very good surface quality and consequently a lower contact resistance. This change in contact resistance may be small, but can be significant.

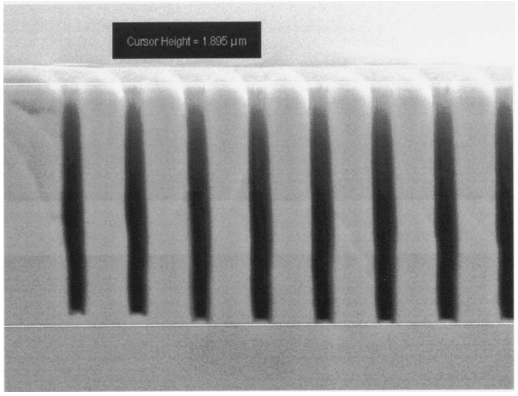

FIG. 5. One-dimensional photonic crystal etched in GaAs. A high-beam-voltage (1450 V) low-beam-current (12 mA) regime of CAIBE was used (Ref. 3). A significant amount of mask remains. The HSQ mask shown here was cured at 400 C.

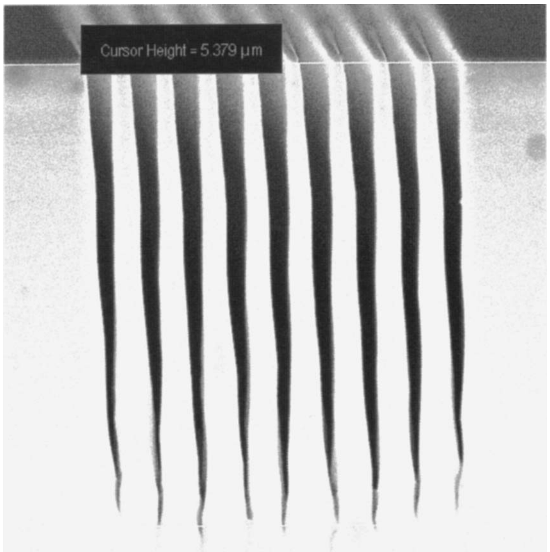

FIG. 6. One-dimensional photonic crystal in InP. Deep high verticality features are achieved using a 300-nm-thick spin-coated HSQ silica hard mask.

At the same time, we have successfully etched photonic crystals using this mask. The pattern may be transferred from the PMMA to the silica using RIE as normal, though with a slightly higher etch rate compared to PECVD SiO_2. No loss of resolution or increased edge roughness is observed relative to patterns created in conventionally deposited silica masks (see Fig. 4).

The photonic crystals were etched by CAIBE using a regime of operation, described in Ref. 14, favorable for high-aspect-ratio features. Figure 5 shows a one-dimensional photonic crystal etched in a GaAs laser material using HSQ silica resulting in deep slots. A laser incorporating such a mirror will, thus, have a lower contact resistance than the one using the traditional mask. This is an important development in quantum well and quantum dot lasers incorporating photonic crystals as poor contact resistances can degrade laser performance, particularly in applications that require multiple silica deposition and removal steps.[15]

It is well known that two-dimensional photonic crystals are more difficult to etch than the one-dimensional ones;

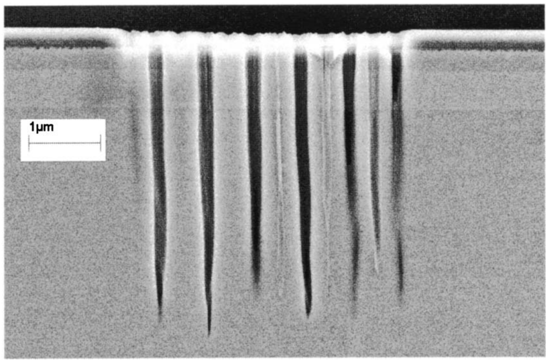

FIG. 7. Two-dimensional photonic crystal in InP. An etch depth of 4.1 μm is achieved. The hole diameter is 200 nm.

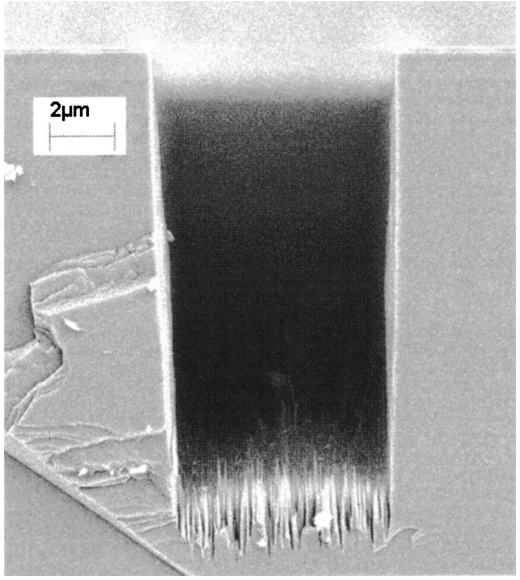

FIG. 8. Trench etched to a depth of 12 μm in InP using a 600-nm-thick HSQ mask.

however, we demonstrate that HSQ is a suitable mask for these crystals also. Figures 6 and 7 show one- and two-dimensional photonic crystals, respectively, etched in the traditionally problematic InP. This etching is comparable with the results of Ref. 14, and is superior to etch depths reported elsewhere, including those etched using ICP.[16,17] InP is the material of choice for active elements and lasers at the telecommunication wavelengths due to its potential to provide gain and its low surface recombination velocity. As for GaAs lasers, attention should always be paid to the contact resistance in such devices.[1–3] We have also used HSQ hard masking to etch extremely deep large features. Figure 8 shows a wide (7 μm) trench etched to a depth of 10+ μm. This was created using a thick (600 nm) film of hard baked (500 °C) HSQ.

The photonic crystals, shown here, were etched using HSQ cured at 400 and 500 °C; by curing at higher temperatures, superior etch resistance should be possible (see Fig. 4). However, the effects of such a temperature on the device need to be considered.

Finally, the etch rate of HSQ used as an electron-beam resist was examined. The resist was spun on to the sample and baked at 220 °C for 1 min and then exposed in an electron-beam writer and developed (Shipley MF319). The exposed regions undergo a reaction that partially transforms the resist into silica, making these areas insoluble in the developer. The etch rate of this mask was found to be approximately. 30 nm/min under the standard CAIBE conditions used here. The use of a postexposure bake improves this resistance by further completing the transformation, giving an etch rate of 18 nm/min. This, as might be expected, is comparable to that of HSQ that is hard baked at this temperature without exposure.

V. CONCLUSION

We have used a spin-on glass (HSQ) as a mask for high-aspect-ratio etching of photonic crystals. We have examined its etch resistance when prepared using different conditions. For the optimum conditions, no loss of performance is observed in the resultant etching compared to conventional PECVD SiO_2.

This mask is more favorable for use in the creation of active devices than the commonly used silica mask deposited by PEVCD. HSQ masking shows no degradation in the contact resistance relative to an unmasked control sample. This gives a greater than 15% improvement compared with PEVCD silica masking. This is important for the creation of high-efficiency active photonic crystal devices. Furthermore, this method cuts out an expensive step in the fabrication of photonic crystals, requiring no more than a spinner and hot plate.

We have used this technique, elsewhere, to produce high-quality active[18] and passive devices.[19]

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