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Anisotropic high aspect ratio etch for perfluorcyclobutyl polymers with stress relief technique

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The authors have developed an anisotropic, high aspect ratio (18:1) etch for perfluorocyclobutyl (PFCB) polymers with trenches as narrow as 800 nm using a CO/O_2 etch chemistry in an inductively coupled plasma reactive ion etcher. Anisotropy is achieved by carbon sidewall passivation. The motivation for this etch development is to use the air trenches as very compact waveguide splitters [S. Kim *et al.*, Opt. Eng. **45**, 054602 (2006)]. The authors report a new trench widening mechanism due to tensile stress of the PFCB films and a method of avoiding this widening through the use of additional stress relief trenches on both sides of the desired trench. © 2006 American Vacuum Society. [DOI: 10.1116/1.2382945]

I. INTRODUCTION

Polymers have long been of interest as a platform for planar lightwave circuits (PLCs).¹⁻⁶ Fluorinated polymers have attracted particular attention because of their typically low propagation loss at telecommunication wavelengths. Perfluorocyclobutyl^{7,8} (PFCB) (Tetramer Technologies, Inc.) comprises a family of partially fluorinated copolymers with selectable refractive index based on copolymer composition, and glass transition temperatures as high as 350 °C (PFCB chemical structure is shown in Fig. 1).9 Waveguides fabricated with such materials typically have low refractive index contrast (1%-3%) between the core and clad. Similar to low refractive index contrast silica waveguides, this results in a minimum waveguide bend radius on the order of several millimeters or larger, which is a limiting factor in decreasing the size of waveguide elements in PLCs to enable higher levels of integration.

We recently proposed the use of air trenches to create small area bends and splitters in materials with low refractive index (i.e., on the order of 1.5, as opposed to higher index materials such as silicon nitride and semiconductors), which permits the size of these waveguide elements to be essentially independent of the core/clad refractive index contrast.¹⁰ We have demonstrated high efficiency bends in PFCB,⁴ and have designed air trench splitters suitable for compact ring resonators and Mach-Zehnder interferometers (see, for example, Fig. 2).⁵ However, fabrication of air trench splitters is challenging since they require narrow (800 nm), high aspect ratio (18:1) trenches.

Anisotropic etching of polymers has been reported in the literature with different degrees of achieved anisotropy and aspect ratio. For example, a high aspect ratio etch of features as narrow as 150 nm has been developed for SiLKTM using a high density plasma source and in which anisotropy is a result of passivation with polymer graphitization.¹¹ The

maximum achieved aspect ratio is ~6:1 in N₂/O₂ and N₂/H₂ plasmas. A deep reactive ion etch (RIE) with vertical sidewalls has likewise been achieved in polymethylmethacrylate (PMMA) using Ar and O₂.¹² Etch depths of up to 85 μ m were achieved for features ~65 μ m and larger.

As reported in Ref. 4, we have developed an anisotropic etch for PFCB using a He/O₂ etch chemistry in an inductively coupled plasma reactive ion etcher (ICP RIE) (Surface Technology Systems) with SiO₂ as an etch mask.⁴ Etched trenches 20 μ m wide and 14 μ m deep with vertical sidewalls have been fabricated. However, this etch has over 1 μ m lateral widening, which makes it unsuitable for the 800 nm features needed for air trench beam splitters.

Schuppert *et al.* developed a cryocooled RIE etch process for PFCB using a SF_6/O_2 etch chemistry and a substrate temperature of $-50 \,^{\circ}C.^{13}$ The purpose of the low temperature is to reduce the kinetics of spontaneous etching on the sidewalls. This etch has a smaller lateral widening compared to room temperature etches, but the achieved aspect ratio is only 1:1 and the sidewall angle is 85° . In contrast, we need to achieve a sidewall angle of $90^{\circ} \pm 1^{\circ}$ to achieve high optical efficiency.



FIG. 1. PFCB chemical structure showing the polymer unit cell and the two chemical structures contained in the copolymers supplied to us by Tetramer Technologies, Inc. (Ref. 7).

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FIG. 2. Two-dimensional finite difference time domain simulation results at a wavelength of 1550 nm for an 800 nm wide air trench as a 50:50 waveguide beam splitter. Two-dimensional simulation predicts a total optical efficiency of 98% (Ref. 5).

While oxygen is typically used as the primary gas in plasma etching of polymers due to its high reactivity, ion induced lateral spontaneous etching is also present,^{14,15} which usually results in linewidth broadening and nonvertical sidewalls, particularly for etches conducted at room temperature or above. To reduce or eliminate this lateral etching, a gas can be added to the plasma to create a protective passivation layer on the sidewalls.^{11,13,14,16} In this article we report the successful development of a PFCB etch using a CO/O₂ etch chemistry in an ICP RIE. We achieve deep (~14 μ m), narrow (800 nm) trenches with vertical sidewalls for an etch aspect ratio of 18:1. The addition of CO promotes the development of a thin passivation layer on the sidewalls to suppress spontaneous chemical etching by oxygen and thereby prevents excessive trench widening.

We have also found another source of trench widening that can dramatically affect the realization of submicron high aspect ratio features. Since the PFCB films in our samples are under tensile stress, this stress tends to pull apart the edges of the trench to create a wider trench than would otherwise result if the film were unstressed. We report a method of avoiding this stress induced broadening by etching narrow stress-relieving trenches on both sides of the main trench.

This article is organized as follows. In Sec. II we discuss initial etch development with optically patterned features using a contact mask aligner. However, since fabrication of high efficiency splitters requires precise air trench-towaveguide alignment that cannot be achieved in a mask aligner, we finalized our etch process development with electron beam lithography (EBL) performed in a scanning electron microscope (SEM). This not only permitted accurate alignment, but also had the benefit of being much more repeatable in defining the trench width for submicron features. The EBL etch process is discussed in Sec. III. In Sec. IV we



FIG. 3. SEM images of fully etched optically written trenches: (a) cross sectional view showing 14 μ m depth and vertical sidewalls and (b) top view showing ~800 nm width.

explore the effects of PFCB film stress on trench widening and show how this can be avoided with extra stress-relieving trenches.

II. ETCH DEVELOPMENT WITH OPTICALLY PATTERNED FEATURES

Initial PFCB etch development using a CO/O_2 chemistry was done with an optically patterned array of trenches ranging in width from 0.5 to 20 μ m. Etch samples were prepared by sequentially spin coating and curing two 11 μ m thick PFCB films on 3 in. Si wafers, which corresponds to the final thickness of eventual fully processed waveguide samples. A HCl-based adhesion promoter was applied to the Si substrate prior to spin coating the first film. Curing was performed in a nitrogen atmosphere with samples beginning at room temperature and then ramped up to 190 °C at a rate of 1 °C/min followed by a dwell time of 22 h. After curing, a positive photoresist (AZ 701 MiR) was spin coated on top of the PFCB film stack and exposed with the trench array pattern by optical contact lithography. A 120 nm thick aluminum film was thermally evaporated on the samples, followed by lift-off to create an aluminum etch mask. We varied different etch parameters such as the ratio of the CO to O_2 gas flow rate (which included the extreme cases of etching with only CO and only O_2 , chamber pressure, coil power,



FIG. 4. Aspect ratio dependent etch rate for PFCB with CO/O2 etch.

platen power, and platen temperature to obtain an anisotropic, high aspect ratio etch. The best etch result was achieved with 500 W coil power, 200 W platen power, 5 mTorr chamber pressure, and 25 SCCM CO and 5 SCCM O₂ flow rates (SCCM denotes cubic centimeter per minute at



Aluminum



FIG. 5. Fabrication process schematic.



FIG. 6. Small feature in aluminum film after the aluminum etch. The rough layer on top is the remaining ZEP e-beam resist.

STP). The platen was kept at 10 °C by helium backside cooling. Figure 3 shows the final etch results after a 54 min PFCB etch. Figure 3(a) is a cross sectional SEM image of the trenches, showing sidewall verticality on small features as well as the desired 14 μ m etch depth. Figure 3(b) shows a top view of an 838 nm wide trench achieved on one of the optically patterned trenches after being etched by more than 14 μ m deep. Figure 4 shows the etch rate as a function of trench width, indicating that the etch rate is aspect ratio dependent (as expected).

While the optical lithography results show that it is possible to etch trenches of the desired size and aspect ratio, the alignment accuracy that can be achieved in a contact mask aligner is typically at best $\sim 1 \ \mu$ m. However, to achieve maximum optical efficiency a splitter needs to be placed to within 100 nm of its ideal position.⁴ Moreover, it is very difficult to pattern 800 nm features with reasonable uniformity and yield over a 3 in. wafer. We therefore opted to use our other in-house lithography capability, which overcomes both of these difficulties. This required a variety of process adjustments, which are detailed in the next section.

III. ETCH PROCESS FOR ELECTRON BEAM PATTERNED FEATURES

We have found that we can generally achieve alignment accuracies significantly better than 40 nm using the autoalignment feature of the nanometer pattern generating system (NPGS, from JC Nabity Lithography Systems) when we do electron beam lithography in a LEO 1550 field emission SEM. However, the lift-off process described in Sec. II to pattern the aluminum etch mask cannot be used with e-beam lithography due to charge that accumulates in the absence of a conductive layer during patterning. Charging causes poor visibility of the EBL alignment marks and consequently poor trench-to-waveguide alignment. To avoid this problem we deposit a 120 nm thick uniform aluminum film directly on the top PFCB layer, and then spin coat a positive e-beam resist, ZEP 520A (ZEON Corp.). (See Fig. 5 for fabrication process schematic.) The conductive aluminum film dissipates



FIG. 7. SEM images of fully etched trenches: (a) top view (b) tilted cross sectional view.

electrons during EBL, thereby eliminating the charging problem but necessitating that the Al be etched rather than lifted off.

After exposure and development of the e-beam resist, we physically sputter the aluminum film in the ICP RIE using an argon plasma with ZEP 520A as the etch mask. To survive through the entire aluminum etch process, we found that the ZEP e-beam resist needed to be 550 nm thick and soft baked at the upper end of its temperature range (180 °C) in order to improve its etch resistance. The final optimized aluminum etch process is 10 SCCM argon flow rate, 5 mTorr chamber pressure, 600 W coil power, 300 W platen power, and 20 °C platen temperature. The etch time is 6 min. Figure 6 shows a small trench after the aluminum etch.

Immediately after the aluminum etch, we use the same CO/O_2 etch process discussed in Sec. II to etch comb shape patterns in the PFCB, with each group of comb teeth having a different width ranging from 200 to 800 nm. We find that a 250 nm wide pattern in aluminum results in an 800 nm wide trench etched in PFCB. Thus there is 550 nm of lateral wid-

ening during the etch. Figures 7(a) and 7(b) show top view and tilted cross sectional SEM images of trenches after the PFCB etch. Note in Fig. 7(a) that the trench edges are reasonably smooth, while in Fig. 7(b) the sidewall verticality appears excellent despite some plastic deformation due to cleaving. Also note that the etch depth is over 14 μ m.

IV. STRESS INDUCED LATERAL WIDENING

For actual device fabrication, individual air trench waveguide splitters are patterned and etched at waveguide intersections. Figure 8 shows a top view of one such isolated trench after the PFCB etch, which was initially written as a 250 nm wide line by EBL. Note that the trench is widened to ~2 μ m, and that there is severe cracking at the trench ends. This is caused by tensile stress in the PFCB film, which is a result of the coefficient of thermal expansion (CTE) mismatch between PFCB (~60 ppm/°C) and Si (2.6 ppm/°C at 293 K) and the PFCB curing process. When the wafer is brought to room temperature after being cured at 190 °C, the



FIG. 8. Extreme lateral widening and cracking at the ends on an isolated trench.



FIG. 9. Stress related widening of the outmost trenches in an etched comb pattern. All trenches had the same 250 nm starting width patterned by e-beam lithography.



FIG. 10. Effect of stress relief trenches. (a) Three identically patterned trenches. (b) Half-length stress relief trenches (see text for discussion).

PFCB film attempts to shrink more than the Si substrate, thereby resulting in considerable tensile stress in the PFCB film, which in turn significantly widens isolated trenches.

A careful inspection of the etched comb patterns discussed in Sec. III suggests a method of relieving the stress and obtaining the desired trench widths. As shown in Fig. 9, a comb pattern in which all of the comb fingers are EBL patterned with a width of 250 nm results in all of the inner trenches having the expected width of 800 nm after etching. However, note that the widths of the two outermost trenches are $\sim 1.94 \ \mu$ m. Clearly, the outermost trenches relieve the tensile stress for the region in between them (at least in the direction perpendicular to the trenches), which permit the inner trenches to be broadened only by the reactive ion etch process such that they have the desired final width.

As shown in Fig. 10(a), we can therefore avoid trench widening by placing the desired trench between two stress relief trenches. Each of the trenches shown in Fig. 10(a) began as a 250 nm wide EBL patterned line, with 30 μ m separation between lines. After etching, the middle trench is 777 nm wide, while the stress relief trenches are widened to 1.3 μ m and cracked at the ends. This result confirms the effectiveness of the use of adjacent stress relief trenches to avoid stress induced broadening of the middle trench.

As a further demonstration of this stress-relieving approach, consider Fig. 10(b), which shows stress relief trenches patterned only adjacent to the lower half of the middle trench, which has a post-etch width of 853 nm. The upper half of the middle trench, which is not between stress relief trenches, has a width of 1.32 nm, and this end of the trench is cracked.

The main drawback of the use of stress relief trenches is that some trial and error is required to determine the width of the EBL patterned trenches that result in the desired width of the final etched trench. Nonetheless, our experience indicates that this is relatively straightforward. Also, in the case of fabrication and characterization of waveguide splitters, the stress relief trenches need to be placed far enough from the waveguide such that they do not intersect the waveguide mode. For our waveguide geometry, 99.99% of the modal power is within a circle of 7 μ m radius centered on the waveguide core. Therefore all stress relief trenches have to be placed at least 7 μ m away from the waveguide, which is not onerous for our applications.

To the best of our knowledge, stress induced lateral widening of etched polymer trenches on Si substrates has not been noted by other groups, despite extensive polymer etching reported in the literature. This is likely due to the fact that the effect of this trench widening mechanism is indistinguishable from the lateral widening that is often associated with anisotropic reactive ion etching. For example, we now consider it likely that the lateral widening reported in our previous paper for He/O₂ anisotropic etching of PFCB (Ref. 4) is due to a combination of reactive ion etch effects and tensile stress broadening. Note that in that paper we were, nonetheless, successful in compensating for this widening in our optical patterning in order to accurately place trench edges relative to waveguide intersections. We expect that similar stress induced widening effects are present with other polymer films that are cured on non-CTE matched substrates and in which deep trenches are etched. This will be particularly noticeable for small feature, high aspect ratio trenches that are etched most or all the way through the polymer film.

V. CONCLUSION

We have developed a small feature, high aspect ratio (18:1), anisotropic etch for PFCB polymers that uses a CO/O_2 etch chemistry in an ICP RIE. Anisotropy was achieved by controlled trench sidewall passivation based on the use of CO. Tensile stress in the PFCB film resulted in excessive trench widening for isolated trenches. We have demonstrated how stress relief trenches can be used to avoid widening of the main trench.

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