Fabrication and Characterization of Planar and Channel Polymer Wave Guides. I. Plasma-Polymerized HMDS Films

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SYNOPSIS

Plasma-polymerized hexamethyldisiloxane (HMDS) films have been prepared as both planar and channel wave guides. The optical attenuation results measured in both the planar and channel HMDS wave guides were found to be similar, thus demonstrating that the inherent solvent resistance and chemical inertness of the plasma polymerized films allows the use of common photoresist techniques, including application of the photoresist, photomasking, and subsequent etching. This may be contrasted with wave guides made from conventional polymers, where careful consideration must be given to photoresist/polymer compatibility, because the photoresist solvents may adversely affect the underlying polymer and lead to degradation of the material during processing. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

Over the last 10 years or so it has been shown that organic thin films have great potential for use in photonic applications.[1] Organic molecules showing nonlinear optical NLO behavior have typically been dispersed in polymer matrices, or the NLO groups have been attached to the polymer chains as pendent side groups. A number of polymeric chains have also been reported that have NLO activity resulting from the backbone structure of the polymer molecules. These developments have, thus, led to a renewed interest in preparing polymeric thin films of high optical quality.[2] It is also of particular importance for photonic device applications[3] that methods for fabricating polymeric thin films into complex wave guide configurations become better defined. Conventional solution-coating techniques and Langmuir-Blodgett films have been widely used for preparing polymeric thin films in a variety of configurations. Plasma polymerization methods may also be employed to prepare thin films that are defect free and, hence, of high optical quality.[4-10] Plasma-polymerized films, by nature, are very adherent to a substrate, resistant to solvents, and can be produced with a variety of chemical and optical properties.[9,11] This technique also has the advantage that multilayered structures are easily prepared and the use of solvents may be avoided.

Described below are some investigations that have been carried out in our laboratories on fabricating and characterizing polymer films in both planar and channel wave guide configurations. The films described here were prepared using plasma polymerization and conventional photomasking methods.

EXPERIMENTAL

Materials

The hexamethyldisiloxane (HMDS) (NMR grade) was purchased from Aldrich Chemical Company and used without further purification. The argon and oxygen used in the plasma system were Ultra High

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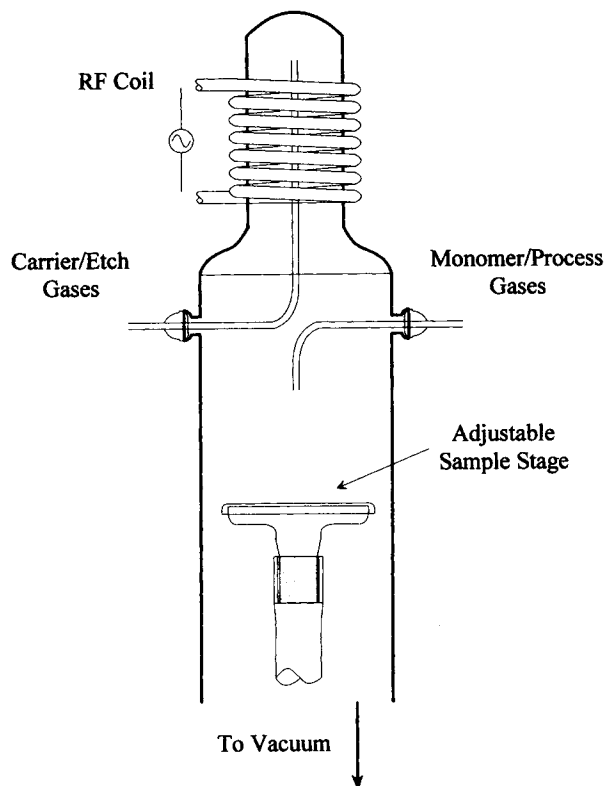‡ To whom correspondence should be addressed.

Figure 1   RF Plasma polymerization reactor.

Purity grade and were supplied by Matheson. The photoresist used was Shipley Microposit 1400 (a positive photoresist) and the developer was Shipley

Microposit 351 solution. The wave guide substrates were <100> orientation 3 inch diameter silicon wafers and were purchased from International Wafer Service. The wafers were base cleaned for 10 min at 70°C on a hot plate in a mixture of 50 cm^3 30% NH_4OH, 50 cm^3 30% H_2O_2, and 250 cm^3 deionized water. The cleaned wafers were then rinsed with deionized water. A 2.25 μm oxide layer was next grown onto the silicon wafers in a tube furnace at 1050°C using steam ambient oxygen atmosphere. A second base clean was performed immediately prior to the plasma-polymer film deposition.

Plasma-Polymerized Films

Plasma-polymerized films were produced in a glass/quartz tubular reactor using a 13.56 MHz generator (RF Plasma Products), the power being inductively coupled through external electrodes (see Fig. 1). The electric field, when applied to the gaseous monomers at low pressures (0.1 to 1.0 Torr), produces active species that may react to form highly crosslinked polymer films. In these experiments, hexamethyldisiloxane HMDS (a liquid monomer at STP) was carried into the chamber from an external manifold. Argon was used as the primary plasma, and the monomer vapor was injected downstream of the primary argon glow discharge, as shown in Figure 1. The conditions used for this study were 40 Watts RF power, 1.0 Torr system pressure, 150 sccm Ar,
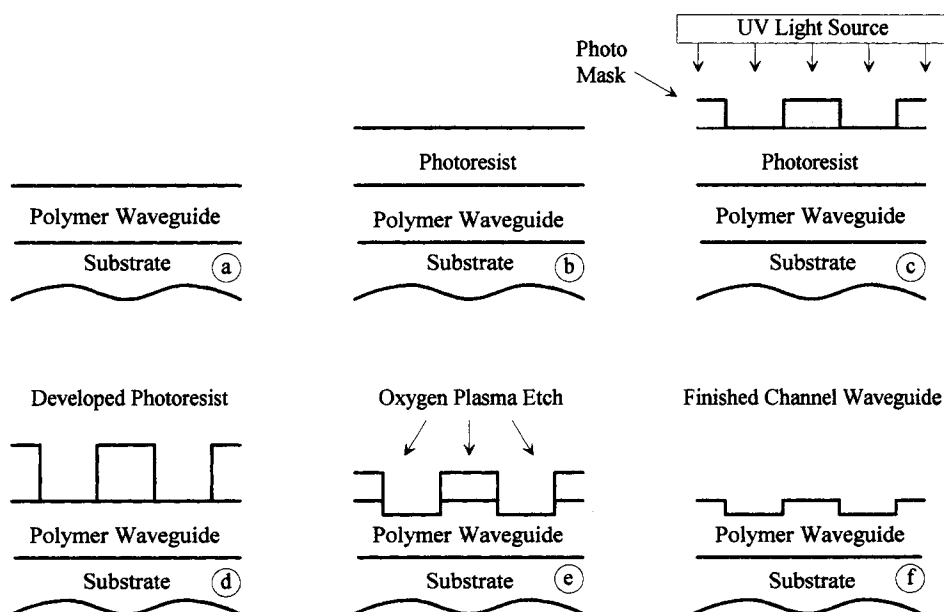

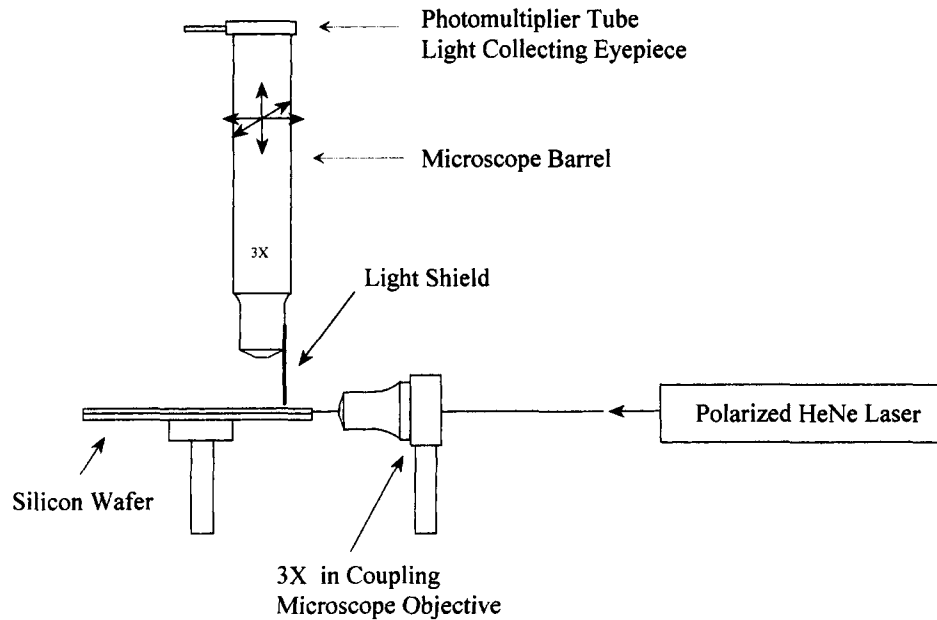**Figure 2**   Formation of channel wave guides using photolithography.

Figure 3 Optical loss measurement of an end-fire coupled thin film polymer wave guide.

and 0.5 sccm HMDS flow rates. The films were found to be uniformly deposited across the wafer and were produced at a rate of 421 Å/min.

Photomasking of Wave Guides

Channels were formed in the planar wave guide films by utilizing electronic photolithographic techniques, which involve photomasking and subsequent reactive ion etching, as shown schematically in Figure 2. The inherent chemical inertness and resistance to solvent swelling of the plasma polymerized films
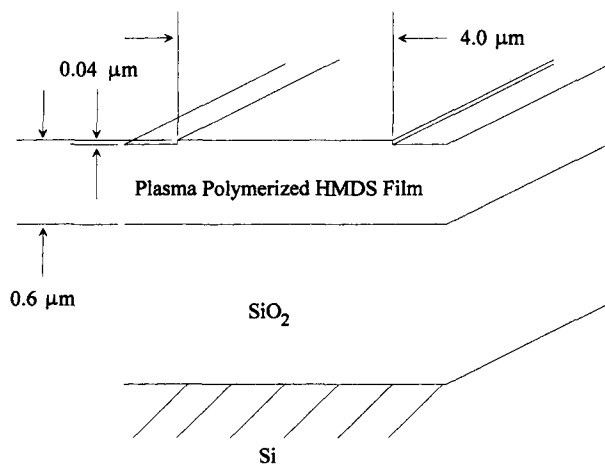

Figure 4 Plasma polymerized HMDS channel wave guide showing dimensions (not to scale).

allows the use of common photoresist systems. The Shipley Microposit 1400 photoresist was applied by spin coating using a programmable Solitec 4 head vacuum chuck photoresist spinner to produce an imageable layer approximately 0.5 μm thick. This was followed by mask exposure using a mercury arc lamp Karlsuss MGB3 mask aligner. The developer solution was then applied as five parts deionized water to one part Shipley Microposit 351. Once a suitable pattern had been developed in the photoresist, an oxygen reactive ion etch in a RF plasma was used to produce the channels. This etching step used an oxygen plasma in the RF reactor described above. The process conditions were 100 Watts applied RF power, 0.5 Torr system pressure, and 20 sccm oxygen flow rate. This yielded an etch rate of 155 Å/min in the photoresist and 40 Å/min in the plasma HMDS film. Because the etch rate in the resist was four times that of the plasma HMDS, the resist thickness had to be at least four times the desired channel depth. The resulting wave guide was then rinsed with acetone to remove the remaining photoresisit, as shown in Figure 2. The wafer was cleaved to permit end-fire coupling.

Determination of Channel Dimensions

The film thickness measurements and film etch rates were performed for films on unoxidized silicon wafers using a Gaertner L116A Ellipsometer with a He : Ne (6328 Å) source. The refractive indices of
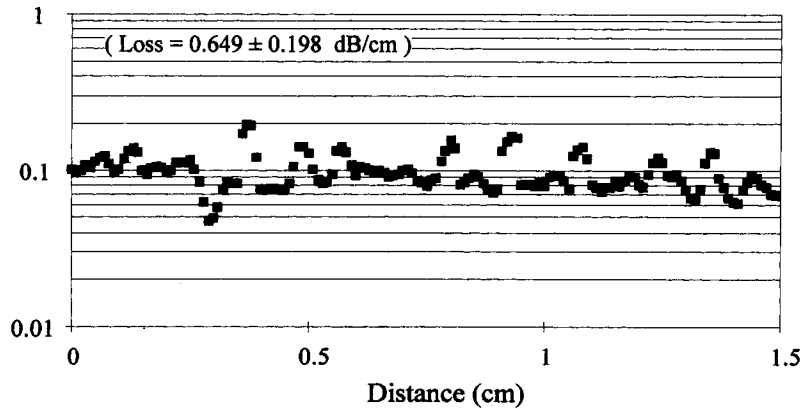
Figure 5   Propagation loss of a 6500 Å plasma polymerized HMDS planar wave guide.

the HMDS films were also determined by ellipso-metry. The film thicknesses and channel step-height were determined using a Technor Alpha Step 100 Stylus Profileometer. In order to use the stylus to measure film thicknesses, a groove was first cut into the film using a 0.5 mm stainless steel point. Special care was taken not to scratch the oxide during this procedure. The etched rib step was then measured conventionally with the stylus.

Optical Loss Measurements

Optical loss measurements were made by measuring the relative outscattering intensity as a function of distance along the light streak. The incident light from a 2 mW polarized He : Ne laser (λ = 6328 Å) was endfire coupled into the HMDS plasma films through a 3X microscope objective lens. The outs-cattering intensity perpendicular to the wafer along the streak was measured using a fiber optic cable

and a photomultiplier tube, as shown in Figure 3. The measured out-scattering intensity, $I(z)$, as a function of distance, z, is related to the intensity, $I(0)$, at $z = 0$ by

$$I(z) = I(0) \, e^{-\alpha z}$$

When $I(z)$ is plotted on a log scale as a function of z, the slope is $-\alpha$. The loss is then calculated in dB/cm. From the slopes of the logarithmic intensity vs. distance plots, the propagation losses for the various wave guides were determined.

RESULTS AND DISCUSSION

The plasma polymer film thicknesses and dimensional profiles were determined using ellipsometry, profileometry, and scanning electron microscopy,
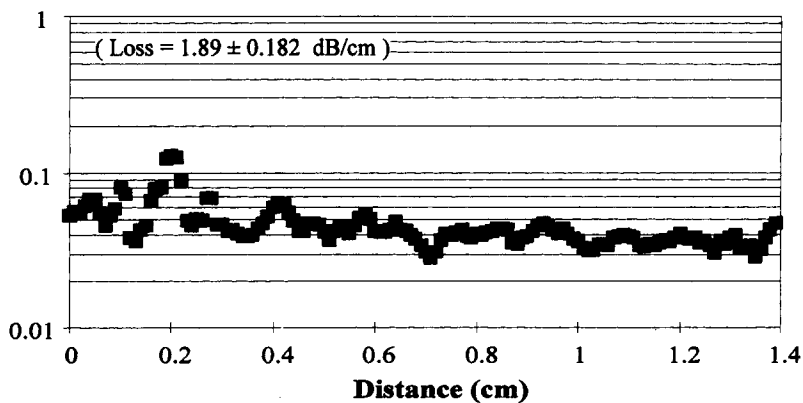

Figure 6   Propagation loss of a plasma polymerized HMDS channel wave guide. The dimensions of the channel are depicted in Figure 4.

and gave a typical channel configuration, as depicted in Figure 4. Refractive index of the HMDS film was found to be 1.596 at the wavelength 6328 Å. Tien and co-workers[4] have previously reported plasma HMDS to have a refractive index of 1.4880 at 6328 Å and plasma vinyltrimethylsilane (VTMS) 1.5279–1.5356 at this wavelength. They, thus, suggest plasma copolymerization of the HMDS/VTMS as a means of controlling the refractive index of plasma polymerized siloxane films. We have shown elsewhere that the refractive index of our plasma HMDS may be controlled by varying the RF power during the plasma deposition.[12] In Figures 5 and 6 are shown the propagation losses for plasma-polymerized hexamethyldisiloxane films in planar and channel wave guide configurations, respectively. The loss for the 6500 Å planar guide was found to be 0.649 ± 0.198 dB/cm, and for the 4.0 mm channel etched to a depth of 0.04 μm, the loss was found to increase to 1.890 $\pm$ 0.182 dB/cm. Losses of less then 1 dB/cm are required for device applications,[1–3,13] and the high optical quality of the planar and channel plasma-polymerized HMDS films is demonstrated here by the losses being of the same order of magnitude for the two cases. This fabrication methodology, thus, allows us to change the geometry of the wave guide[14] with only a small effect upon the optical propagation characteristics. It is, therefore, clear that the inherent solvent resistance and chemical inertness of the plasma-polymerized films allows the use of common photoresist techniques, including application of the photoresist, photomasking, and subsequent etching. This observation is of particular importance in device fabrication, as the plasma polymerized films are (i) stable to the photoresist solvent and chemistry; (ii) stable during the UV exposure of the photoresist; (iii) stable to the oxygen plasma reactive ion etch used to produce the channel contour; and (iv) stable to the subsequent removal of the photoresist after photomasking. This may be contrasted with wave guides made from conventional polymers, where careful consideration must be given to photoresist/polymer compatibility, because the photoresist solvents may adversely affect the underlying polymer and lead to degradation of the material during processing. It should also be noted that the plasma-polymerized HMDS films have excellent thermal stabilities,[12] which also make them attractive for optical device processing and applications.

Some previous studies of plasma-polymerized vinyltrimethylsilane and hexamethyldisiloxane planar wave guides have been reported in the literature, and losses in the range 0.04 to 10 dB/cm were ob-

tained.[4] In such studies, it was reported that the formation of fine powder during the plasma polymerization detrimentally effect the optical performance of the resulting films by the powder inclusions forming scattering centers that lead to light attention in the propagating medium.[4,5] Powder-forming regimes are subtly governed by the parameters used for the plasma film deposition[9,11] and also depend on the particular reactor system used. It is clear that the formation of powders must, therefore, be carefully avoided during plasma polymerization, as in the present study, when high optical quality is required. In considering this methodology for the fabrication of polymer wave guides, it should be noted that a variety of conventional organosilicon polymeric materials also find widespread use in multilayer plasma resist processing,[15,16] and this continues to be a very active area of research.[17,18]

Investigations of polymeric thin films, which include materials having NLO activity, are currently being carried out in wave guide and device configurations and will be reported later.

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