Reactive Ion Etching of Y_2O_3 films applying F-, Cl- and Cl/Br-based Inductively Coupled Plasmas

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Reactive ion etching of yttrium oxide thin films was investigated using CF_4/O_2 , BCl_3 , HBr and Cl_2 inductively coupled plasmas. For all gas combinations, with the exception of Cl_2 , the etch rate was found to be similar, indicating a primarily physical etch process, enhanced in Cl_2 by an additional chemical-etching component. The highest observed etch rate was 53 nm/min in Cl_2 , suitable for controlled etching of optical ridge waveguide structures several hundred nm in height. Overall, the surface quality of the etched Y_2O_3 films was the highest after etching in CF_4/O_2 plasmas, indicating a trade-off in etch rate and film quality between the various process gases. Preliminary selectivity measurements show that Ni is a suitable material for etch-masking of high resolution structures in Y_2O_3 .

Introduction

Y_2O_3 thin films become increasingly attractive for application in integrated optical waveguide devices operating in the third telecommunications window (~ 1550 nm). This is mainly due to the high refractive index ($n \sim 1.9$), which enables the realization of highly compact integrated optical devices and to the excellent transparency, being the prerequisite for the fabrication of low-loss optical waveguides. The high thermal conductivity and high mechanical stability of Y_2O_3 , combined with its ability to serve as a host material for rare-earth ions such as Er^{3+} and Yb^{3+} , makes Y_2O_3 well-suited for laser and amplifier applications (1). Such Y_2O_3 thin films incorporating rare-earth dopants have been deposited on Si or other substrates by several methods, including spray-pyrolysis (2), reactive sputtering (3) and pulsed laser deposition (PLD) (4,5). Recently, rare-earth doped Y_2O_3 films with low optical losses on the order of 1 dB/cm at both 800nm (6) and in near-infrared wavelengths (7) have been demonstrated.

Compared to bulk materials, integrated thin-film so-called ridge or channel waveguide-based lasers and amplifiers offer the advantage of strong confinement, thus low threshold pump powers and good overlap of pump and signal (8). In order to successfully realize such integrated active waveguide devices, a reliable patterning technique is required. Due to a high chemical stability, structuring of Y_2O_3 has been primarily limited to purely physical etching techniques (9). For high-resolution and low-cost structuring of optical waveguides, however, reactive ion etching (RIE) is generally the preferred method. RIE is based on a combination of physical and chemical etch processes, which allow for the optimization of a suitably anisotropic sidewall etch profile while limiting the damage induced by a purely physical etching process.

Recently the etching characteristics of sesquioxide films has been of some significant interest due to their possible application as high dielectric constant insulating layers (10). In particular, the etch characteristics of Y_2O_3 has been investigated in primarily Cl-based plasmas (11,12). In this paper we present preliminary results on the etching behavior of reactively co-sputtered, polycrystalline Y_2O_3 thin films. The impact of various etch chemistries (fluoride, chloride and chloride / bromide mixture) on the etch rate and film quality is compared.

Experimental Details

Y_2O_3 films of approximately 900 nm thickness were reactively sputtered on Si substrates. The refractive index of the Y_2O_3 layers was measured using a spectroscopic ellipsometer, and found to be 1.93 at 633 nm. The etching behaviour the films was studied using an Oxford Plasmalab 100 inductively-coupled plasma (ICP) reactive ion etch system. In an ICP system the plasma ion density is controlled separately from the RF power supplied to the substrate. The ICP source was controlled by a 3 kW, 13.56 MHz generator, while substrate bias was controlled separately by a 600 W, 13.56 MHz RF Generator. ICP is also characterized by low process pressures, which allows highly directional and well-controlled ion flux onto the substrate, thus highly anisotropic etched features and enhanced etch rate. Various standard process gases and combinations of these gases were used, including BCl_3 , $\text{BCl}_3\text{-HBr}$ (50:50), CF_4/O_2 (90:10), Cl_2 and Cl_2/Ar . The total gas flow, held constant at 50 sccm, was measured by mass flow control units, while in general, process pressure was maintained as low as possible, varying between 9-12 mTorr. Unless otherwise stated, ICP power was held constant at 1500 W. The etch rate of the Y_2O_3 films was determined by measuring the film thickness before and after the etch process with a spectroscopic ellipsometer, while the etch rate of Ni was measure using a Dektak surface profilometer. Surface morphology was acquired using a Digital Instruments atomic force microscope (AFM).

Results and Discussion

The etch rate of the Y_2O_3 films was investigated as a function of applied RF power for various plasma compositions. Figure 1 shows the resulting etch rate as a function of RF power for CF_4/O_2 (90:10%), BCl_3 (100%), BCl_3/HBr (50:50%) and Cl_2 (100%) gases. From these results, it can be seen that with the exception of Cl_2 , the various etch chemistries do not significantly impact the etch rate. This indicates that primarily a physical etch process is involved for these process gases. The possible etch products of Y_2O_3 (such as YCl_3 , YBr_3 , YF_3) are known to be non-volatile (indicated by high melting points). We therefore expect low etch rates, with primarily physical mechanisms dominating. Cl_2 clearly exhibits the highest etch rates, with a maximum value of 53 nm/min observed at 400 W RF power. This indicates that perhaps a stronger chemical component is involved in the purely chlorine-based etch process.

Figure 1 - Comparison of etch rate and RF bias as a function of RF power for 100% BCl₃, BCl₃:HBr (50:50), CF₄:O₂ (90:10) and Cl₂ at a total flow rate of 50 sccm.

When measuring the thickness of the etched films, the quality of the ellipsometric fit varied significantly. The mean square error, an indication of the fit quality, was found to increase significantly for films after etching in BCl_3 , BCl_3/HBr and Cl_2 , while it remained relatively low after etching in CF_4/O_2 . This indicates a possible deterioration of films etched in chemistries other than CF_4/O_2 . In order to account for possible roughness at the surface, an effective medium approximation was used. The EMA method can be used to measure films with a rough surface by approximating the rough portion of the film with an additional layer composed of a mixture the film material itself and a certain percentage of void-space in the film. Table 1 shows the relative fit error for samples etched in the different plasma chemistries, and the fitted “rough” surface layer using the EMA method. Including this EMA layer for Cl_2 , BCl_3 and BCl_3/HBr greatly improved the fit, while it affected the fit of CF_4 -etched films very little. This data suggests that the film surface quality is not degraded significantly by etching in CF_4/O_2 plasma.

In order to verify these results, surface measurements were obtained using AFM for an un-etched sample and a sample etched in Cl_2 . After etching at an applied power of 400W, the EMA fit gave a surface layer thickness on the order of 45 nm. Surface morphology and particle (or grain size) analysis using AFM are shown in Fig. 2. In (a), the unetched film shows good uniformity and relatively small particle size, with an average particle height on the order of 10 nm. In (b), the surface uniformity is clearly poorer, and average particle height is 47 nm, which agrees with the EMA measurements.

TABLE I. Comparison of film quality after application of different plasmas at 100W RF power (etch time = 2 min.).

Plasma	Relative RF Bias	Relative Error of Fit	Fitted Surface Layer Thickness using EMA
BCl_3	338	0.75	13.7
BCl_3/HBr (50:50)	292	1	18.0
CF_4/O_2 (90:10)	290	0.29	4.2
Cl_2	271	0.80	14.8

Figure 2 – AFM 3-D surface plots and 2-D images showing average particle height for a Y_2O_3 film (a) before and (b) after etching in Cl_2 plasma with 400W RF power applied.

Figure 3 – Etch rate and fitted surface roughness using an effective medium approximation after an applied Cl_2 -Ar plasma. The data is plotted as a function of Cl_2 to Ar ratio, with a constant total flow rate of 50 sccm and constant applied RF power of 300 W.

To investigate further the etch rate and quality of Cl-etched films, which showed high etch rate but poor surface quality, a mixture of Cl_2 and Ar was used. The etch rate and surface layer roughness, approximated by an EMA fit, is plotted as a function of Cl_2/Ar mixing ratio in Fig. 3. The physical component of etching can be controlled to some extent by increasing or decreasing the ratio of Ar in the gas mixture. Ar is a non-reactive noble gas, which means Ar ions reaching the substrate will only contribute to the etch process through physical breaking of the highly stable Y-O bonds. It has been suggested that adjusting the amount of physical etching could affect the overall rate by both breaking bonds at the surface and removing non-volatile etch products produced by reaction of Cl ions with Y and O atoms from the surface (13). Non-volatile etch products remaining on the surface could be the cause of high surface roughness observed in the Cl-etched films. However, the addition of Ar is in fact shown to further degrade the quality of the etched films and also decrease the etch rate.

Figure 4 – Etch rate as a function of ICP RF power in CF_4/O_2 (90:10) plasma at a total flow rate of 50 sccm, for varying RF chuck power and chamber pressure.

The highest surface quality was found using a CF_4/O_2 gas mixture. The reason for this is as yet unknown. In order to determine the maximum possible etch rate with this plasma, several parameters were varied and the etch rate measured. The important results are plotted in Fig. 4. As expected, etch rate was found to vary little with flow rate (not shown), and is primarily dependant on chamber pressure, ICP RF power and applied RF substrate power. The combination of these parameters determines the intensity and directionality of ion bombardment at the substrate, and the ion density of the plasma. The etch rate increased with increasing ICP power and RF power, and decreasing chamber pressure. The highest etch rate measured was 45 nm/min at 400 W, 2500 W

ICP and 10 mTorr. This value is still lower than the highest etch rate measured in Cl chemistry, which was not yet optimized, a further indication that a stronger chemical component is involved in the Cl-based etch process. Therefore, it is suggested that for high etch rate and good film surface quality, a combination of $\text{Cl}_2/\text{CF}_4/\text{O}_2$ be used.

An additional consideration is the selection of possible mask materials for etching of Y_2O_3 . Due to the high required RF powers required for a suitable etch rate, the selection of mask materials is limited. Based on initial studies, photoresist layers of several μm in thickness would be required for etching through several 100 nm of Y_2O_3 . For higher resolution patterning, metal layers are of interest because of their stronger resistance to physical etching. In particular, nickel is a promising candidate because it can be easily patterned by wet chemical etching or lift-off, resulting in uniform layers and smooth features. Figure 5 shows the results of etching both Ni and Y_2O_3 in a BCl_3/HBr chemistry. At approximately 400 V and below, good selectivity of Y_2O_3 to Ni is demonstrated. However, at an applied bias above 500 V (300 W) the Ni etch rate is shown to increase significantly above that of Y_2O_3 . This may mean that a threshold bias level has been reached for physical sputtering of the metal. Therefore, for further studies of the etch behaviour of Y_2O_3 using Ni as a mask material, the applied RF bias should be sufficiently limited.

Figure 5 – Etch rate and selectivity of Y_2O_3 and Ni in BCl_3/HBr (5:2) as a function of DC Bias, at a total flow rate of 35 sccm, ICP power of 1750 W and chamber pressure of 7 mTorr.

Conclusion

Reactive ion etching of Y_2O_3 thin films was studied using various plasma chemistries and parameters. Overall, the measured etch rates in all gases were sufficiently high for the controlled etching of optical waveguide structures to an etch depth of several hundreds of nanometers. A maximum etch rate of greater than 50 nm/min was measured using a Cl_2 plasma. However, films etched in Cl_2 showed significant deterioration of surface quality, while those etched in CF_4/O_2 demonstrated a mostly insignificant change in surface roughness. This suggests using a combination of CF_4/O_2 and Cl_2 for further investigation of RIE of high quality Y_2O_3 layers at a maximum etch rate. Further process investigation will focus on the optimization of optical waveguide channel fabrication. The effect of various process conditions, particularly various combinations of process gases, on the etch rate, anisotropy, waveguide sidewall roughness, surface roughness and selectivity to mask materials will be studied. For such an investigation, Ni was shown to be a promising mask material with a selectivity of greater than 1 already demonstrated in BCl_3/HBr plasma.

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