SU-8 protective layer in photo-resist patterning on As₂S₃ film

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Amorphous chalcogenide thin films, especially arsenic tri-sulphide (As₂S₃) are emerging as an excellent platform for integrated non-linear optic devices due to their high non-linearity as well as low linear and nonlinear optical loss. Fabricating planar As₂S₃ waveguides, however, is not straightforward because As₂S₃ is dissolved in alkalis such as photo-resist developer. In this study we present the application of thin SU-8 film as a protective layer to prevent the attack of the developer on the As₂S₃ during photolithographic process. Despite excellent coating feature and simple process of SU-8 as a protective layer, the delamination between SU-8 and polymer cladding during chip cleaving hindered its application. We deposited thin Al₂O₃ on SU-8 by atomic layer deposition

and this intermediate layer could solve the problem. We measured the insertion losses of fabricated waveguides as a function of device lengths and widths. The propagation loss becomes significant in a narrow guide due to the enhanced surface scattering. It was clear that extra losses were engaged in long guides containing bending structures. SU-8 layer and thin Al₂O₃ combination allowed reliable and repeatable fabrication process in that it did not induce any extra light scattering or absorption and prevented attack of the As₂S₃ film by the alkaline developer. Moreover, the device characterisation revealed that SU-8 layer is superior to PMMA/BARC in terms of propagation loss of waveguide.

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1 Introduction Chalcogenide glasses (ChGs) are based on chalcogen elements S, Se and Te and the addition of other elements such as Ge, As and Sb leads to the formation of stable glasses [1]. Owing to the wide transmission range from visible to mid-infrared (IR), their bulk forms have been extensively applied in IR optic components for several decades. Moreover, optical data storage media such as compact disc and digital versatile disc, are made from ChGs films utilizing photo-induced structural change in the materials. Phase change between crystalline and amorphous in Ge-Sb-Te, and resulting high contrast electric resistivities, is the basic principle behind non-volatile random access memory. Because of very high nonlinearity and its ultra-fast response time, low linear and nonlinear losses in the telecommunication wavelengths, our research group (CUDOS) have worked on the materials, the fabrication of optical waveguides, and the demonstration of all-optical signal processors [2,3]. Thermally-evaporated arsenic tri-sulphide (As₂S₃) film and subse-

quent semiconductor processing - photolithography and plasma etching - is a general procedure to producing rib-type planar waveguides [4,5].

There are, however, two material/process-related problems in fabricating As₂S₃ guides. Firstly, room light illumination induces photo-oxidation of the film surface in ambient atmosphere, producing micron-sized As₂O₃ crystallites [6]. These surface irregularities incur optical scattering loss in the waveguide as well as make it impractical to apply contact lithography. Secondly, As₂S₃ is dissolved with a quite fast rate of several tens nanometre per second in alkaline photo-resist developer solution, which normally contains several weight percent of tetra-methyl-ammonium hydroxide (TMAH) [6]. To address these difficulties, we have coated thin protective layer prior to photo-resist patterning. By covering the film surface the layer prevents not only the oxidation of As₂S₃ but the penetration of alkaline developer into the film. So far, several types of layers have been utilized for this purpose. A thin photo-resist inten-

tionally left on the film by controlling development time was the early version of protective layer; nonetheless, this approach compromised the process reproducibility and simplicity [6]. Bottom anti-reflective coat (BARC), which is a standard one in semiconductor processing, was employed in the waveguide production, especially in thick film [4]. To obtain higher nonlinear effect in device the guide dimension needs to be scaled down for strong light confinement in the medium. In sub-micron thick film BARC was not so effective because BARC stripping process introduced roughening of the film surface [7]. Instead, the combination of poly(methyl methacrylate) (PMMA) and BARC was successfully applied in sub-micron thick waveguide processing [7].

In this paper we present the process development of SU-8 single protective layer for thin As_2S_3 waveguide fabrication. Despite of pinholes-free coating and simple process, the delamination between SU-8 and inorganic polymer cladding (IPGTM) was observed. By depositing 2–5 nm thick Al_2O_3 on SU-8, we could achieve strong adhesion between the layers. Using the conventional cut-back method the propagation losses of fabricated waveguides could be attained. We also discussed how the guide width and length impact on the device losses as well as the effectiveness of SU-8.

2 Device fabrications As_2S_3 films with 0.85 μm thick were deposited by thermal evaporation onto oxidized silicon wafers in a vacuum chamber. Crushed bulk glass in a tungsten boat was used as a source and it was evaporated at around 300 °C. Deposition occurred with the source to substrate distance of ~ 40 cm at a rate of ~ 0.2 – 0.3 nm/s. A complete description of our procedure for thermal evaporation of As_2S_3 films can be found elsewhere [8]. As described in our previous publications [9], as-deposited As_2S_3 film contains significant amount of molecular phases, wrong bonds (e.g., As-As and S-S) and voids dispersed in As_2S_3 -based glass network. To reduce these light attenuation sources in the virgin material, thermal annealing was done at 130 °C for 24 hours in a vacuum oven. Prior to heat treatment, 125 nm thick SU-8 (SU-8 2 from Micro-Chem Corp.) layer was spin-coated and UV-cured on the film to prevent As_2O_3 crystallites formation on the surface during subsequent processing steps [6]. Furthermore, this SU-8 was exploited as a protective layer in the following photolithographic process. SU-8 protective layer is superior to previous ones in several aspects. Similarly to PMMA [7], SU-8 is almost transparent at telecommunication window so that it is not necessary to remove it from the guide surface before cladding formation; this makes the fabrication process simpler. Moreover, its excellent coating feature without any pinholes makes it attractive for a protective layer. When PMMA was coated and thermally-cured, a large number of pinholes appeared in the PMMA due to evaporation of the solvent [7] and the alkaline developer could penetrate through these holes and dissolved the As_2S_3 , roughening its surface. As a result the BARC

had to be used to cover these holes. On the contrary we could not observe any defect on UV-cured SU-8 layer.

Photo-resist waveguide patterns were produced on the SU-8/ As_2S_3 film using a Karl Suss MA6 i-line ($\lambda = 365$ nm) mask aligner followed by wet development. The SU-8 and As_2S_3 layers were etched in an inductively coupled plasma reactive ion etcher (Plasmalab System 100, Oxford Instruments) utilizing oxygen-based and CHF_3 plasma, respectively. A 15 μm thick layer of inorganic polymer glass (RPO Pty Ltd, IPGTM) was spin coated on top of the As_2S_3 waveguides and cured with UV light to create a top cladding. Waveguide end facets were obtained by hand cleaving the wafer along the {110} silicon cleavage planes.

Figure 1(a) show the scanning electron micrograph of cross sectional view of fabricated waveguide. Whilst SU-8 was clearly intact to the guide top surface, the IPG cladding was peeled from the surface when the wafer was cleaved. Considering the delamination was worse near the guides, the weak adhesion between SU-8 and the cladding seems to cause this anomaly. We deposited thin (~ 2 nm) Al_2O_3 on the SU-8 by atomic layer deposition (ALD) in order to promote the adhesion. Interestingly, this intermediate layer performed well; we could not see any peeling-off of the cladding as shown in Fig. 1(b). However, Al_2O_3 thickness affected the cleaving behavior of the chip so that optimal thickness had to be sought. Figure 2(a) illustrated a non-flat cleaved facet when Al_2O_3 thickness was 30 nm, presumably due to very strong adhesion of Al_2O_3 to the polymer and SU-8. Very thin and smooth ($R_{\text{rms}} \sim 0.3$ nm) Al_2O_3 is not expected to incur extra optical loss.

X-ray photoelectron spectroscopy (XPS, Escalab 220-IXL) was used to probe the chemical bonding of arsenic atom on the As_2S_3 film surface. A monochromatic Al K α x-ray source ($h\nu = 1486.6$ eV) with power of 250 Watts was used for the analysis. The As 3d spectrum could be decomposed into three sub-peaks as shown in Fig. 2(b), according to the bonding types. Two peaks at middle and lower binding energy (red and purple curves) correspond to arsenic atoms in which each is bonded to three sulphurs (three As-S bonds), and to two sulphurs and one arsenic (two As-S and one As-As bond), respectively [9]. Small peak centred at higher binding energy is inferred to come from arsenic bonded to oxygen.

Figure 1 The scanning electron micrograph of cross sectional view of fabricated guide without (a) and with (b) Al_2O_3 coating.

Figure 2 (a) Cross-section view of cleaved facet when 30 nm thick Al_2O_3 was coated. (b) De-convolution of As $3d$ XPS spectrum into three sub-peaks.

3 Device characterisation The propagation losses of the guides were determined via the standard cutback method. A tunable laser source (JDS Uniphase SWS16101, 1510–1650 nm), a polarization controller, and an InGaAs power meter were used to measure the insertion losses of waveguides. Due to small waveguide cross section lensed-tip fibers which produced a mode-field diameter of $\approx 2.5 \mu\text{m}$ were used to couple laser light into and out of the waveguides.

Figures 3(a), (b), and (c) represent the insertion losses of waveguides as a function of guide length; the width were 4, 2, 1 μm , respectively. Here, the losses of waveguides fabricated using SU-8 and PMMA/BARC are compared to investigate the influence of the protective layers. Since 4 inch wafers were used, waveguides longer than 10 cm comprised of straight lines connected by arcs with 2 mm radius. Every intercept on the loss-axis from straight guides (below 10 cm) agreed well to the coupling loss calculated using FEMSIM software between the guide facet and lensed fiber. We can clearly see that there is an offset between the intercept and the regression line from longer guides, which becomes larger in narrower guide. The cause of these extra losses in longer guides is uncertain; nonetheless rough chromium line edge of curved element in photo-mask seems to be responsible for.

As the guide width gets narrower, the propagation loss becomes significant. This behaviour is trivial in that more light would interact with the guide surface in narrower one,

enhancing surface scattering. It is also noticeable that transverse magnetic (TM) mode showed slightly higher loss than transverse electric (TE) mode, with the difference increasing in the narrower waveguides. Though mode calculation revealed that TM is confined more tightly in the core than TE, its mode shape is more elongated along the film plane; hence more light interacts with the etched, rougher sidewalls, rather than relatively smooth top and bottom interfaces. This could explain the behaviour.

It is obvious from the insertion losses of the guides that SU-8 protective is similar or superior to PMMA/BARC combination. The difference was negligible in 4 μm guide, though. This may come from the feature of mode distribution in wide core: only small fraction of mode experiences surface scattering so that volume scattering or absorption loss is dominant. Our calculation showed that the fraction of light at the sidewalls is very small, 0.04 % in TE and 0.06 % in TM in the 4 μm wide guides. However, the losses reduced significantly in narrower and longer guide, presumably due to smoother bend edge when the guide was produced from SU-8. Another thing to be noted is: even though SU-8 was reported to have $\sim 1.5 \text{ dB/cm}$ absorption loss at around 1550 nm [10], the loss originated from SU-8 protective layer was negligible because only thin layer of it present in the cladding.

4 Conclusion We successfully employed SU-8 protective layer for As_2S_3 waveguide fabrication. The adhesion of SU-8 to polymer cladding was enhanced significantly using very thin Al_2O_3 intermediate layer. SU-8 provides not only excellent coating feature and simple process, but the improvement in the insertion losses of narrow/longer waveguides. These strongly support that SU-8 is an optimal material for As_2S_3 process.

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Figure 3 Insertion losses of waveguides with their lengths when the guide width are (a) 4, (b) 2, and (c) 1 μm .

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