Fabrication of low loss $Ge_{33}As_{12}Se_{55}$ (AMTIR-1) planar waveguides

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The authors describe the fabrication of low loss $Ge_{33}As_{12}Se_{55}$ rib waveguides. Pulsed laser deposition was used to obtain high quality, dense films with the same stoichiometry as the bulk glass, while standard semiconductor processing was used to pattern the waveguides. They obtained rib waveguides of 3, 4, and 5 μm wide with propagation losses (0.3 dB/cm at 1550 nm) more than ten times lower than previously reported for this material. © *2007 American Institute of Physics*.
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Chalcogenide glasses (ChGs), which contain the chalcogen elements S, Se, and Te covalently bonded to network forming elements such as Ge, As, Sb, and Si, have attractive properties for a wide range of applications. Although being widely used as phase change materials for optical disks and nonvolatile random access memories, ChG films are also good candidates for planar integrated nonlinear optical devices due to their high nonlinearities and low linear and nonlinear loss.[1]

Many binary or ternary ChG glasses (As–Se; As–S–Se), most interesting from a nonlinear optics perspective, have, however, low glass transition temperatures ($\approx$200 °C). They also display mediocre glass forming properties because of the dominantly two-fold bond coordination of the constituent elements, which compromises their stability and processability.

One commercially available glass with the composition $Ge_{33}As_{12}Se_{55}$ and the trade name AMTIR-1,[2] however, has a significantly higher glass transition temperature (362 °C) and is expected to have superior glass properties because of the presence of fourfold coordinated germanium atoms which results in better network forming properties. Additionally the material has high refractive index (2.55 at 1550 nm), intrinsic losses around 0.1 dB/cm at 1550 nm,[3] and $\sim$200 times larger Kerr-nonlinear coefficient (n_2) than that of silica.[4] Therefore this material seems an attractive candidate for the production of nonlinear planar waveguides. Previous work on Ge–As–Se glasses is, however, rather limited and unpromising. Films with propagation losses of 10 dB/cm at 1064 nm were made in 1974,[5] a glass with 5% Ge was used to make photodarkened waveguides with minimum loss of 3.5 dB/cm at 1300 nm,[6] and most recently, silver photodoped waveguides were fabricated with losses around 4 dB/cm at 1550 nm.[7]

In this letter we describe the fabrication of AMTIR-1 rib waveguides and determine their optical losses by the cutback method. In particular, we have developed a fabrication process (deposition, photolithography, and plasma etching) which is compatible with standard semiconductor processing. The fabricated waveguides showed propagation loss more than an order of magnitude lower than previously reported for this material, opening up the possibility that AMTIR-1 can be used to fabricate nonlinear waveguide devices for all-optical signal processing.

AMTIR-1 films were deposited on thermally oxidized silicon wafers from commercial glass targets [Amorphous Materials of Garland, Texas, USA (Ref. 3)] using ultrafast pulsed laser deposition (UFPLD).[8,9] This technique employs a frequency doubled mode-locked $Nd:YVO_4$ laser producing 25–28 W average power (900–1000 nJ/pulse) at 532 nm and a repetition rate of 28 MHz. The laser beam was directed into a vacuum chamber pumped down to $(\sim 3-5)\times 10^{-7}$ Torr and focused on a target with a telecentric scanning lens. The vapor was deposited on the wafers placed on a rotating carousel located $\sim$400 mm from the target. The film thickness was 1.75 μm with uniformity better than $\pm$0.01 μm.

We proved that the composition of the deposited films was the same (within$\pm$1%) to that of target AMTIR-1 glass using energy dispersive x-ray spectroscopy. Precise stoichiometry transfer of material is one of the major advantages of using UFPLD, as illustrated by the fact that thermally evaporated films of AMTIR-1 are known to have incorrect stoichiometry.[10] The refractive index of as-deposited films was 2.67 at 1550 nm, which is higher by 0.12 than that of the target due to differences in the bond structure in the as-deposited films compared with the bulk glass.[11] Scanning electron microscope observations of the microstructure of the deposited films indicated that, unlike rf-sputtered films,[6,12] no porosity or microcracks existed in our films.

There is an obstacle to the application of standard photolithographic process with ChGs; namely, they dissolve in alkaline solutions (e.g., photoresist developer) at a relatively rapid rate. For example, rf-sputtered AMTIR-1 films were reported to have an etch rate of 53 nm/s in NaOH solution (molarity=0.07).[12] This explains why there has been few reports of PR patterning of ChG films. However, our UFPLD films showed a very slow etch rate ($\sim$4 nm/min) in the PR developer solution (AZ MIF300, containing 0.26*N* of tetramethyl ammonium hydroxide); hence a standard photolithographic process could be applied. We assume that our AMTIR-1 films were relatively inert because of their dense microstructure. The PR was patterned using contact photolithography at 365 nm using a high-resolution resist (Clariant AZ MiR 701). The pattern was then transferred into the glass using inductively coupled plasma (ICP) reactive ion etching (RIE) (Oxford, Plasmalab 100) system.

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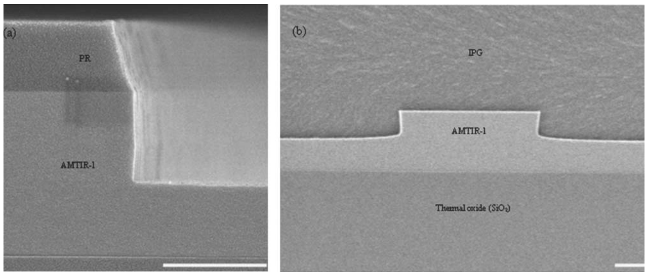

FIG. 1. Cross-sectional SEM micrographs of AMTIR-1 film etched in CHF_3 plasma (a) and finished rib-type waveguide (b). The scale bars in each image denote 1 μm.

In previous work, laser writing has often been used to define ChG waveguides utilizing the inherent photosensitivity of these materials to light near their band edge.[13,14] Photoinduced refractive index changes are, however, likely to be unstable due to relaxation of the glass structure even at room temperature and are unsuitable for practical devices. Thus it is preferable that the core is created by etching the film to create ridge or rib-type waveguides.

Plasma etching of chalcogenides has many advantages over alkaline[12] or amine-based[1] wet etching methods, providing tight dimensional control, vertical sidewall profiles, and clean processing. It is, therefore, preferable for the production of planar waveguides with a small cross section which is needed for strong confinement of the optical beam to create a large nonlinear response. Previously we have reported the etching of As_2S_3 using with CF_4 and oxygen gases.[2,15]

Since all the elements (Ge, As, Se) within AMTIR-1 can form volatile compounds with fluorine, a CF_4-O_2 gas mixture was tried first as an etch plasma in ICP-RIE. Using pure CF_4 plasma, the etched profile was isotropic. When using a fluorocarbon plasma, a Teflon-like polymer can be deposited on the etched sidewall which helps us to make the etched profile vertical.[16] However, it seemed that there is insufficient precursors for the polymer in CF_4 plasma. Therefore, chemical etchants, such as fluorine radicals, can attack the inadequately protected sidewall resulting in isotropic etching. However, when we added 70% oxygen to the CF_4 (the same gas mix used in As_2S_3 etching[2,15]), a quasivertical sidewall could be obtained. The problem of this recipe is, however, the lower etch selectivity to PR and this results in large etch bias upon patterning.

To obtain a more vertical profile and high etch selectivity, we used CHF_3 plasma that has a higher tendency for polymerization.[16] By optimizing the plasma process conditions, bias power, induction power, gas pressure, and gas flow rate, we obtained a suitable etch recipe for AMTIR-1 waveguides. Figure 1(a) shows the typical etched sidewall profile. The etch rate was around 0.5 μm/min and the etch selectivity to photoresist was greater than 10, which is sufficient to produce a tolerable etch bias (below 0.1 μm for 1 μm etch depth).

The waveguides were clad with a 10 μm thick inorganic polymer glass (IPG™, RPO Pty Ltd.[17]), which has a refractive index of 1.53 at 1550 nm. This was applied by spin coating followed by UV curing with a 300–400 nm filtered 500 W Xe–Hg lamp. The use of a liquid IPG cladding deposited by spin coating allowed the waveguide patterns to be covered without voids remaining on the corners. End facets were prepared by manually cleaving the silicon substrate with a diamond scriber. A finished AMTIR-1 rib waveguide with IPG cladding is shown in Fig. 1(b).

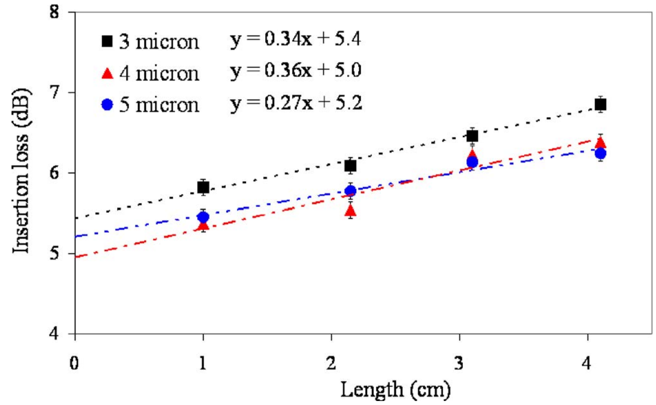

FIG. 2. Raw cutback insertion loss data with linear regression fit lines for the each width waveguide. All data points have the error range of ±0.1 dB, which is the nominal error in repeated measurement. The slopes of three lines also have uncertainty of ±0.05 dB/cm.

Measurements of the insertion loss for the waveguides were made using a 1550 nm Fabry-Pérot laser diode, a polarization controller/scrambler, and an InGaAs power meter. Coupling to the waveguides was accomplished using Nufern UHNA-3 fiber with a mode field diameter of 4.1±0.3 μm at 1550 nm. Figure 2 presents the raw insertion loss data gathered during the cutback measurements for the TE mode. The average propagation loss was 0.3 dB/cm for the waveguides, almost independent of their widths. This indicates that the sidewall roughness of the waveguides from the photolithography and etching processes was not the dominant loss source.[18] The measured losses are at least an order of magnitude smaller than previously reported. We attribute the low loss to the high quality film deposition of UFPLD and appropriate waveguide fabrication process. The y-axis intercepts for the three trend lines, which determine the coupling loss between the waveguides and the input fiber, were around ~5 dB. This value agrees with that calculated from mode overlap calculated using C2VOLYMPIOS software.[19] Figure 3 is the dark field optical microscope image of 5 μm wide waveguide. Given the presence of the particulate contaminants (white dots in the image) in the film, there would appear to be scope for further improvement of the propagation loss by optimizing the deposition conditions.

During waveguide testing it was observed that the waveguides showed a substantial increase in the insertion loss upon illumination with the ring light used on the alignment microscope. For a 5 cm long waveguide, the insertion loss increased by up to 6 dB. Even room lighting increased the insertion loss by 0.6 dB. Turning off the illumination restored the low loss state. Using an IR pass filter with the microscope (RG780) almost completely eliminated this effect (0.05 dB change for light on/off), and all further mea-

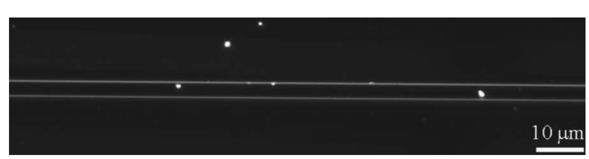

FIG. 3. Dark field optical microscope image of 5 μm wide waveguide. Two parallel lines indicate the edges of the waveguide and the white dots are particulate contaminants in the film.

surements were carried out with this configuration and with the room lights off. The extra loss induced by visible light irradiation is thought to result from free carriers generated in the material, most likely due from the presence of Se clusters which have been identified in the as-deposited films.[11] The elimination of this phenomenon is under investigation.

In conclusion we produced AMTIR-1 waveguides using high quality film deposited by UFPLD patterned using standard semiconductor processing. Low waveguide propagation losses down to 0.3 dB/cm at 1550 nm were obtained. Measurements on bulk glasses indicate that the intrinsic loss of AMTIR-1 at 1550 nm is around 0.1 dB/cm. Eliminating the remaining particulates and relaxing the bond structure of the glass into the bulklike state should, therefore, reduce waveguide losses, even further making AMTIR-1 a viable glass for nonlinear optical devices based on planar waveguides.

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