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基于 AZ5214 光致保护层的 As_2S_3 硫系脊型波导制备

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摘要: 实验研究发现 AZ5214 光刻胶在一定曝光剂量下显影后会留存一定厚度的底膜, 该底膜可以在干法刻蚀过程中避免 As-S 薄膜与碱性显影液直接接触, 减轻薄膜表面损伤, 起到保护作用。基于此, 采用该底膜作为保护层制备 As_2S_3 脊型波导, 研究表明, 在 AZ5214 光刻胶匀胶厚度为 $2.1\ \mu\text{m}$ 、紫外曝光剂量为 $200\ \text{mJ}/\text{cm}^2$ 、显影时间为 45 s 的条件下会留存约为 220 nm 厚的光致保护层, 该条件下保护层均匀性较好, 且在刻蚀阶段可以完全去除。实验表明利用此保护层制备的 As_2S_3 脊型波导具有良好的形貌特征, 波导脊宽约为 $3\ \mu\text{m}$ 、脊高约为 800 nm 的 As_2S_3 脊型波导的传输损耗约为 $0.74\ \text{dB}/\text{cm}@1\ 550\ \text{nm}$ 。

关键词: 集成光学; 硫系玻璃; 脊型波导; AZ5214 光刻胶; 光致保护层

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0 引言

硫系玻璃是以硫族元素如 S、Se、Te 为基, 结合 As、Sb、Ge、Ga 等元素而形成的无机玻璃, 具有极高的非线性折射率(石英材料的 $10^2\sim 10^3$ 倍)^[1]、超快的非线性响应(响应时间小于 200 fs)^[2]、在 $0.4\sim 22\ \mu\text{m}$ 存在宽广的红外透射窗口等优良光学特性^[3], 是一种新型的光子器件基质材料^[4-6], 是近中外波段最佳非线性材料之一^[7-9]。其中 As-S 玻璃体系因具有较为稳定的化学性能而被广泛研究。随着硫系光子器件的发展, 对高质量硫系光波导的需求日益增多, 可用于红外传感器^[10-11]、全光信号处理^[12-14]等领域。在硫系光波导制备方面, 常用的制备方法有离子注入^[15]、湿法刻蚀^[16]、干法刻蚀^[17]、剥离法^[18]等。其中, 利用干法刻蚀技术制备硫系波导取得一定的进展: 2004 年 RUAN Y 等^[19]制备出了损耗约为 $0.5\ \text{dB}/\text{cm}$ 的 $3\ \mu\text{m}$ 宽 As_2S_3 脊型波导; 2007 年 MADDEN S J 等^[20]制备出了 $4\ \mu\text{m}\times 2.6\ \mu\text{m}$ 的 $22.5\ \text{cm}$ 长的蛇形 As_2S_3 脊型波导, 其传输损耗约为 $0.05\ \text{dB}/\text{cm}$; 2020 年 SHEN W 等^[21]制备出的宽约 $2\ \mu\text{m}$ 、厚约为 600 nm 的 As_2S_3 脊型波导损耗约为 $1.44\ \text{dB}/\text{cm}$ 。由于在干法刻蚀过程中显影阶段会使用到碱性显影液, 而硫系玻璃材料尤其是 As_2S_3 玻璃易受到碱性化学物质的腐蚀, 使得制备过程需要非常精密地设计, 同时制备出波导表面粗糙, 而导致波导传输损耗较大。

为了改善这一状况, 研究人员提出增加保护层减轻碱性化学物质的腐蚀, 如 CHOI D Y 等^[22-23]首先尝试引入了底部抗反射涂层与聚丙烯酸甲酯作为保护层, 而后又尝试使用 SU-8 用作保护层, 均制备出损耗有一定改善的 As_2S_3 脊型波导。但此类保护层的增加会使得制备波导流程繁琐, 而且保护层在剥离时存在一定的难度, 同时也会一定程度上损伤波导表面。本文实验发现 AZ5214 光刻胶在一定的曝光剂量、合适的显影时间下会残存一定厚度的底膜附着在 As_2S_3 薄膜上, 基于此提出利用该底膜充当保护层进行干法刻蚀制备 As_2S_3 脊型波导, 该保护层在显影阶段能避免硫系薄膜与碱性化学物质的接触, 减轻显影液腐蚀, 制备

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得到的波导传输损耗较低,约为0.74 dB/cm@1 550 nm。

1 高品质 As_2S_3 薄膜制备

制备硫系薄膜的方法有溶液凝胶法^[24]、脉冲激光沉积法^[25]、磁控溅射法^[26]和热蒸发法^[27-28]等,其中真空热蒸发法具有成膜速度快、均匀性好、薄膜致密性高等优点,现阶段常采用它来制备 As-S 体系硫系薄膜。本文采用真空热电阻蒸发法制备 As_2S_3 薄膜,选用成都光明有限公司的 HWS27 型 As_2S_3 玻璃作为蒸发源,真空度为 3×10^{-3} Pa,保持约为 50 nm/min 的沉积速率在 SiO_2 基片上沉积 As_2S_3 薄膜。图 1(a) 是真空热蒸发法得到的沉积态 As_2S_3 薄膜,由台阶仪测得其表面均方根粗糙度 R_q 约为 0.853 nm。为了进一步降低 As_2S_3 薄膜表面粗糙度,对沉积态 As_2S_3 薄膜在真空氛围内进行退火处理,退火温度为 180 °C,约在 As_2S_3 玻璃转化温度附近,退火时间为 1 h。图 1(b) 是经过退火处理后得到的退火态 As_2S_3 薄膜,其表面均方根粗糙度 R_q 约为 0.501 nm。从图 1 中比较发现,沉积态 As_2S_3 薄膜在玻璃转化温度附近退火处理后,可以减小薄膜表面粗糙度,获得高品质硫系薄膜。

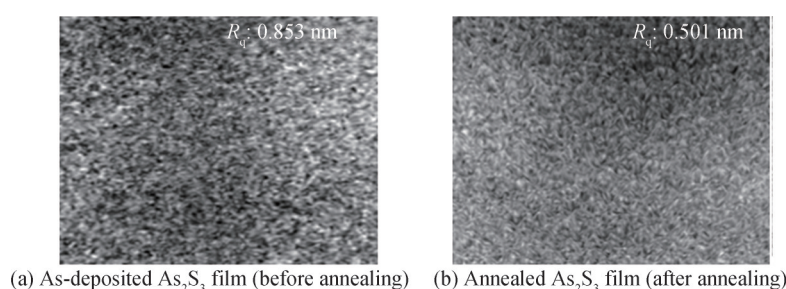


图 1 As_2S_3 薄膜退火前后的表面粗糙图

Fig. 1 Surface roughness diagram before and after annealing of As_2S_3 film

2 As_2S_3 脊型波导制备

使用上述方法制备膜厚约为 1 μm 的高品质 As_2S_3 薄膜样品后,采用干法刻蚀制备 As_2S_3 脊型波导,具体制备流程如图 2。先在样品上旋涂一定厚度的 AZ5214 正性光刻胶,并对其前烘处理(烘烤温度约 90 °C,烘烤时间约 90 s);然后使用 MLA100 无掩膜光刻机(激光直写仪)进行曝光,并使用 ZX-238 显影液进行显影处理;之后采用反应离子刻蚀(Reactive Ion Etching, RIE)方式刻蚀硫系薄膜,最后使用去胶液(N-甲基吡咯烷酮:NMP)进行去胶处理,获得 As_2S_3 脊型波导。由图 2 中局部放大图可知,显影后在曝光区域形成一定厚度的光刻胶底膜,即光致保护层,该光致保护层在显影阶段起着对 As_2S_3 薄膜的保护作用,避免了硫系玻璃薄膜与碱性化学物质的接触。

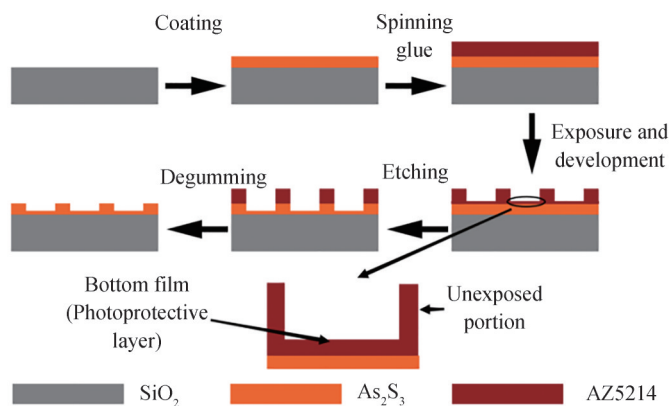


图 2 As_2S_3 脊型波导制备流程

Fig. 2 Preparing process of As_2S_3 ridge waveguide

2.1 保护层

实验发现一定厚度的AZ5214光刻胶经过合适剂量的曝光后,因其底部光刻胶相对上层光刻胶而言,与显影液的反应速率大幅度减慢,造成显影后会留存在一层因光致作用形成的底膜,如图2中局部放大图所示。为了探究该底膜的在显影过程中产生的影响,对比了在存在底膜与无底膜情况下的显影效果,如图3。图3(a)是无底膜情况下的显影效果,显示出As₂S₃薄膜受到碱性显影液腐蚀,这是因为在显影过程中AZ5214光刻胶被显影液溶解掉,导致显影液与As₂S₃薄膜相接触,产生腐蚀,该腐蚀速率约为80~100 nm/s,同时,因整体光刻胶厚度有差异而呈现腐蚀不均匀性。图3(b)是存在底膜情况下显影效果,将底膜用去胶机去除后,可以发现显影液并未对As₂S₃薄膜产生腐蚀作用。由此可得,该底膜可以有效地防止碱性显影液在显影过程中对As₂S₃薄膜的腐蚀,保持As₂S₃薄膜的完整性。

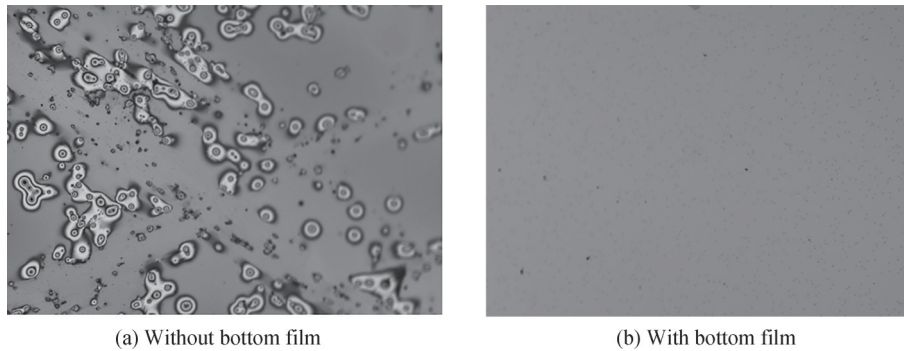


图3 有、无底膜条件下显影后的As₂S₃薄膜表面

Fig. 3 Surface of As₂S₃ film developed with and without bottom film

为了进一步了解该底膜的形成条件,选定一定厚度AZ5214光刻胶以满足刻蚀时的需求(即提高分辨率和保护As₂S₃薄膜),实验研究了底膜厚度与曝光剂量、显影时间之间的关系,结果如表1。从表1分析可知:在匀胶厚度2.1 μm时,曝光剂量200~240 mJ/cm²、显影时间45~50 s的条件下得到的底膜厚度约在210~220 nm之间。实验还发现,如曝光剂量过低或显影时间过短,该底膜的厚度会较大幅度增加,不利于后面刻蚀系薄膜的刻蚀。

表1 底膜(光致保护层)厚度与曝光剂量、显影时间的关系

Photoresist thickness/μm	Exposure dose/(mJ·cm ⁻²)	Development time/s	Bottom film thickness/nm
2.1	150	45	~400
2.1	200	45	~220
2.1	240	45	~210
2.1	200	50	~210
2.1	200	40	~300

基于上述结果,提出采用该底膜作为保护层制备As₂S₃脊型波导,选定参数如下:涂旋光刻胶厚度为2.1 μm,曝光剂量为200 mJ/cm²,显影时间为45 s。图4(a)、(b)分别是该参数下As₂S₃硫系薄膜经过曝光显影后得到的保护层的厚度和均匀度。图4(a)是使用去胶液NMP清洗掉部分保护层形成台阶而测量得到的保护层厚度,显示该保护层厚度约为220 nm;图4(b)是采用台阶仪测量的保护层表面均匀度,该保护层表面均匀度较好,保护层表面均方根粗糙度R_q约为17 nm;由图4(b)还可得到显影后未曝光部分光刻胶厚度约为1.7 μm,对于脊高约为1 μm的波导制备能起到很好的保护作用。

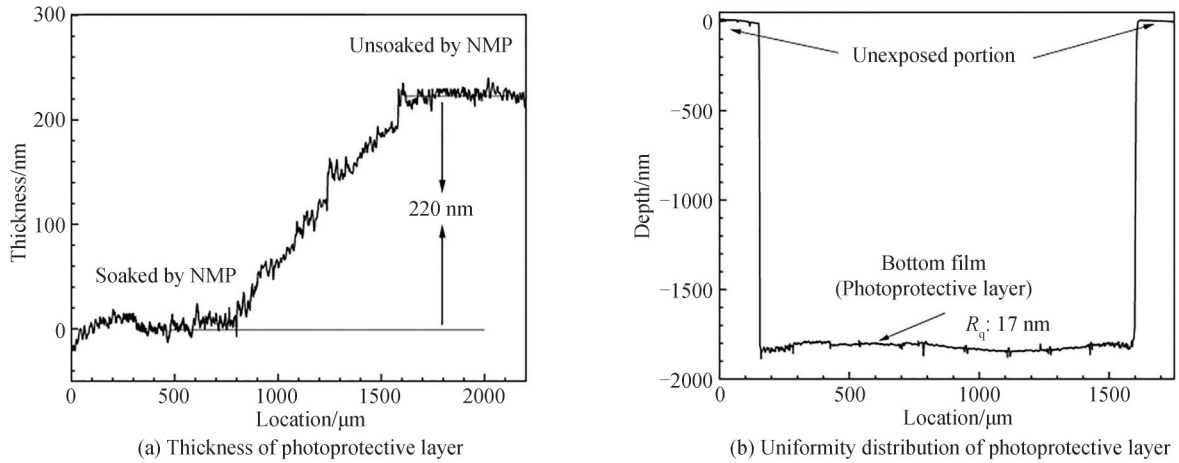


图4 光致保护层参数

Fig. 4 Photoprotective layer parameters

2.2 刻蚀参数确定

在显影阶段,上述形成的光致保护层完成了对硫系薄膜的保护,即避免硫系薄膜被碱性显影液腐蚀,接下来使用JSD-300R反应刻蚀机刻蚀硫系薄膜形成脊型波导结构。在刻蚀之前应先对该光致保护层进行去除,采用氟气刻蚀去除保护层,并同时完成对硫系薄膜的刻蚀,但是由于AZ5214光刻胶与 As_2S_3 薄膜之间的刻蚀选择比较低,该方法在一定程度上会影响波导的侧壁质量。为了改善波导侧壁质量,本文尝试优化刻蚀参数,图5为通

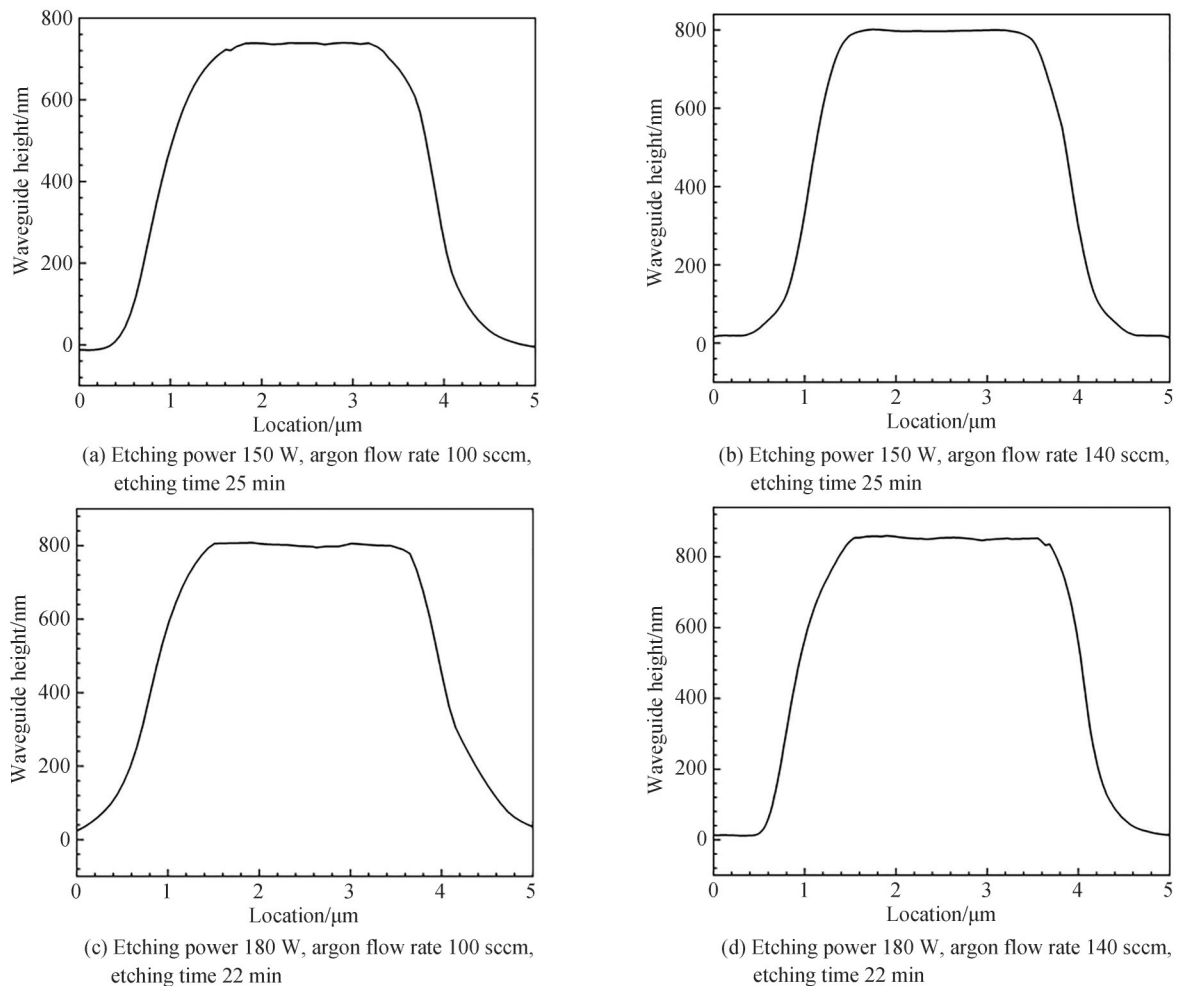


图5 不同参数下的波导轮廓

Fig. 5 Waveguide profile under different parameters

过台阶仪测得数据拟合的波导轮廓图。从图5中分析发现,波导侧壁坡度较缓,曝光时绘制的波导宽度为3 μm,而在脊深为800 nm时实际获得的波导脊宽约为2.3 μm,低于绘图波导宽度。这一现象是由于氩气在As₂S₃玻璃材料与AZ5214光刻胶之间的刻蚀选择比过低造成的。对比图5中腔内压为6 Pa的条件下各参数的刻蚀结果,发现在刻蚀功率为150 W、氩气流量140 sccm、刻蚀时间约25 min的条件下制备出的As₂S₃脊型波导脊型轮廓形貌较好,如图5(b)。

3 波导制备结果

将经过刻蚀后得到的As₂S₃脊型波导放入去胶液NMP进行未曝光部分的光刻胶去除工作。图6(a)、(b)分别是有、无保护层条件下制备出的As₂S₃脊型波导经去胶处理后由电镜SEM观察得到波导俯视图,可以看出有保护层条件下制备出的波导完整性更高,波导表面质量更好。此现象是因为无保护层条件下显影时As₂S₃薄膜表面受到腐蚀(如图3(a)),而在刻蚀过程中,因氩气在As₂S₃薄膜各处刻蚀速率相同,导致显影时产生的损伤被继承到成品波导的表面,从而使得波导完整性受到破坏且表面质量变差。图6(c)是在有保护层条件下制备出的As₂S₃脊型波导截面图,其整体呈现为梯形,这是由于氩气的刻蚀选择比较低,导致制备过程中,存在一定的底切现象,进而导致波导截面形状呈现出梯形。

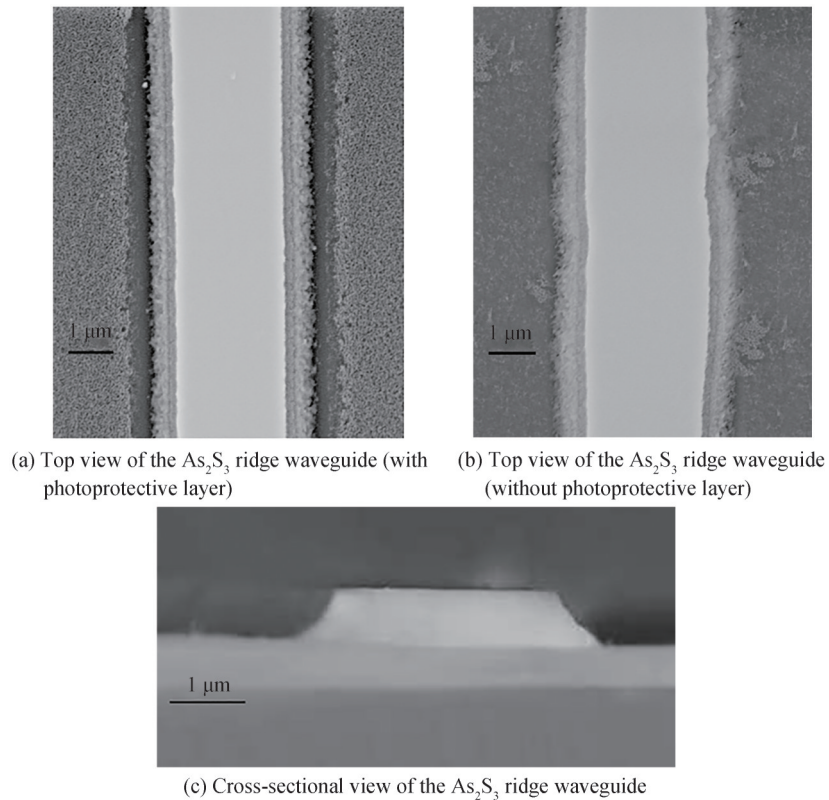


图6 As₂S₃脊型波导形貌图

Fig. 6 As₂S₃ ridge waveguide topography diagram

由于制备得到的As₂S₃脊型波导截面形状是带一定坡度的梯形,而不是标准的脊型,利用COMSOL软件仿真该波导实际参数下的TE模场分布,如图7,显示模场的能量集中于波导内部,这表明不会因存在一定的波导侧壁坡度而导致能量外泄。另外,采用截断法测试了波导的传输损耗,图8是有、无AZ5214保护层条件下制备出的As₂S₃脊型波导插入损耗,线性拟合得到有保护层时波导传输损耗约为0.74 dB/cm,低于无保护层时的1.24 dB/cm,显示该保护层可以有效降低波导的传输损耗。这是因为AZ5214保护层在显影过程中避免了碱性显影液对薄膜完整性破坏以及波导侧壁的损伤,从而降低了波导传输损耗。

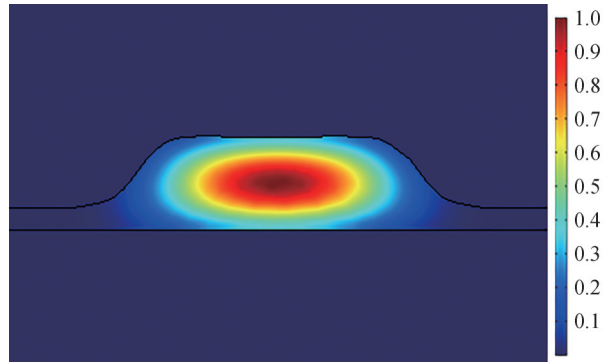
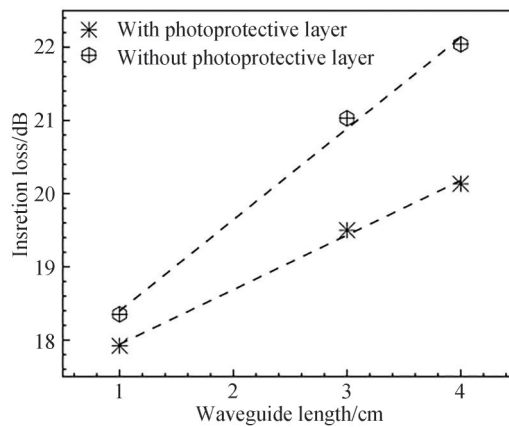
图7 制备得到的As₂S₃脊型波导结构参数下的TE模场分布Fig.7 TE mode field distribution under the structural parameters of As₂S₃ ridge waveguide which was prepared

图8 不同长度的插入损耗(在1550 nm处)

Fig. 8 Insertion loss of different lengths(at 1550 nm guide mode)

4 结论

本文实验发现AZ5214光刻胶在一定曝光剂量下显影后会留存一定厚度的底膜,基于此采用该底膜作为保护层制备As₂S₃脊型波导。实验获得AZ5214光刻胶匀胶厚度为2.1 μm时,在曝光剂量为200 mJ/cm²、显影时间为45 s的条件下光致保护层厚度适中,约为220 nm,且保护层表面均匀度较好。基于光致保护层这些参数,实验制备了带保护层的硫系薄膜,发现该保护层能在显影过程中有效避免硫系薄膜与碱性显影液接触,起到良好的保护作用。在此基础上,刻蚀制备出尺寸3 μm×800 nm的As₂S₃脊型波导,该波导形貌良好且具有约为0.74 dB/cm@1550nm的传输损耗,低于无保护层情况下制备出的波导传输损耗。这些工作表明采用AZ5214光刻胶底膜用作光致保护层进行波导制备减少了波导制备流程,取得良好的制备效果,且降低了波导传输损耗,为制备高品质的硫系光子器件带来了新的思路和制备技术,将推进硫系光子学发展。

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Preparation of As₂S₃ Chalcogenide Ridge Waveguide Based on AZ5214 Photoprotective Layer

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Abstract: With the development of chalcogenide photonic devices, there is an increasing demand for high-quality chalcogenide optical waveguides which are widely used in infrared sensors, all-optical signal processing and other fields, therefore, the preparation of high-quality chalcogenide optical waveguides has become one of the hot fields of chalcogenide photonics. In the preparation of chalcogenide optical waveguide, the preparation methods include ion implantation, wet etching, dry etching, stripping and so on. Among these methods, the dry etching for preparing chalcogenide waveguide is used widely. Especially in recent years, researchers have successfully prepared As₂S₃, As₂Se₃, Ge-Ga-Se and other chalcogenide optical waveguides by dry etching. However, due to attack of the chalcogenide films (especially As₂S₃ film) by the alkaline developer which is used in the dry etching, the preparing process of As₂S₃ ridge waveguide needs very precise design and the quality of the chalcogenide waveguide can be affected. To improve this situation, some researchers proposed adding protective layers to prevent the attack of the developer on the As₂S₃ film. Previous researchers have tried to introduce Bottom Anti-reflection Coating, Polypropyl Methyl Acrylate and SU-8 as protective layers. However, the addition of such protective layers will further make the preparation process of waveguides cumbersome. In this paper, it is found that AZ5214 photoresist will retain the residual film with a certain thickness which is attached to As₂S₃ film under a certain exposure dose and appropriate development time. Based on this, this paper proposes to use this residual film as a protective layer to prepare the As₂S₃ ridge waveguide by dry etching. This protective layer can prevent the attack of the developer on the As₂S₃ film, and also be removed by etching, which will simplify the pattern transfer process compared with other protective layers. In this paper, the as-deposited As₂S₃ films with a film thickness of about 1 μm were deposited by vacuum resistance evaporation onto silica wafers, and then were annealed in a vacuum oven. The root mean square roughness R_q of the annealed As₂S₃ films decrease from 0.853 nm to 0.501 nm, which displays the achievement of high-quality film with smooth surface. After that, the AZ5214 positive photoresist was covered on the surface of these As₂S₃ film to produce waveguide patterns in exposure process and prevent the attack of the alkaline developer owing to the residue photoresist bottom film as a photoprotective layer in development process. In the exposure and development process, the preparation parameters of this photoprotective layer were explored experimentally, and show that under the condition of exposure dose of 200 mJ/cm² and development time of 45 s, the AZ5214 photoresist with thickness of about 2.1 μm could form the photoprotective layer with thickness of about 220 nm and the R_q of about 17 nm. On this basis, the As₂S₃ ridge waveguides with width of 3 μm and height of 800 nm were prepared by using reactive ion etching. However, due to the relatively low etching selection of argon gas, there was a certain undercut phenomenon during the etching process, resulting in a trapezoidal cross-section shape of the waveguide. In order to mitigate this influence, the etching parameter was optimized for obtaining the As₂S₃ ridge waveguide with good-morphology in this paper. In addition, the transmission loss of As₂S₃ ridge waveguide is approximately 0.74 dB/cm by adopting the cutting back method. These works show that the preparation of chalcogenide waveguide by using AZ5214 photoresist bottom film as a photoprotective layer simplifies the preparing process, and achieves good results, which will bring new ideas and preparation technologies for the preparation of high-quality chalcogenide photonic devices and promote the development of chalcogenide photonics.

Key words: Integrated optics; Chalcogenide glass; Ridge waveguides; AZ5214 photoresist; Protective layer

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