Freestanding HfO$_2$ grating fabricated by fast atom beam etching

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Abstract

We report here the fabrication of freestanding HfO$_2$ grating by combining fast atom beam etching (FAB) of HfO$_2$ film with dry etching of silicon substrate. HfO$_2$ film is deposited onto silicon substrate by electron beam evaporator. The grating patterns are then defined by electron beam lithography and transferred to HfO$_2$ film by FAB etching. The silicon substrate beneath the HfO$_2$ grating region is removed to make the HfO$_2$ grating suspend in space. Period- and polarization-dependent optical responses of fabricated HfO$_2$ gratings are experimentally characterized in the reflectance measurements. The simple process is feasible for fabricating freestanding HfO$_2$ grating that is a potential candidate for single layer dielectric reflector.

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Keywords: HfO$_2$ film grating, fast atom beam etching

I. Introduction

As an excellent optical material, hafnium oxide (HfO$_2$) film presents high laser damage threshold, thermal and chemical stability [1-3]. Since HfO$_2$ film is transparent from visible to infrared range, it often serves as the high refractive index material for fabricating multilayer reflection mirror [4,5], or acts as the waveguiding layer for the realization of guide mode resonant optical filter [6]. These optical devices are originated from the film deposition techniques of HfO$_2$ material. On the other hand, freestanding structures are greatly developed as the promising candidates for producing resonant filter [7,8] or in place of a traditional top distributed Bragg reflector to reflect light within a cavity [9-12]. As a single layer dielectric mirror, freestanding structures are often sandwiched with air on top and bottom. Compared with multilayer reflection mirror, freestanding structure is more compact and reflects light more efficiently [13]. The high refractive index contrast between HfO$_2$/air also endows the freestanding HfO$_2$ micro/nano structures with the capacity to function as single layer dielectric reflector or guide mode resonant filter. HfO$_2$ film is a hard material, and usually serves as etch stop layer [14,15]. Recently, focused ion beam (FIB) milling was developed to fabricate sub-micron HfO$_2$ gratings [16]. In FIB milling, micro/nano structures could be achieved on various material systems by physically removing the sample material with a metal ion beam. However, FIB milling is a single process and difficult to be compatible with other fabrication processes for mass production. Moreover, this etching technology is expensive and time-consuming.

We demonstrate here a simple way to fabricate freestanding HfO$_2$ grating by a combination of fast atom beam (FAB) etching and dry etching of silicon. FAB etching, which is capable of high anisotropy etching because it uses neutral particles or atoms for dry etching, is used as a well-controlled, low-damage etching technique to manufacture HfO$_2$ film [17,18]. To make grating structures freely suspend, the silicon substrate beneath the HfO$_2$ grating region is removed in association of anisotropic and isotropic dry etching of silicon. Period- and polarization-dependent optical responses are experimentally characterized in reflectance measurements.

II. Fabrication

Figure 1 schematically illustrates the fabrication process of freestanding HfO$_2$ gratings, which are implemented on a silicon substrate. The process starts from the blank
deposition of HfO2 film on the silicon substrate with an electron beam (EB) evaporator (step a). A positive EB ZEP520A resist is then spin-coated onto the HfO2 layer, and grating patterns are patterned in ZEP520A resist using EB lithography (step b). Subsequently, the patterns are transferred to HfO2 layer by FAB etching (step c). FAB etching, which is generated by the neutralization of ions extracted from direct-current SF6 plasma (Ebara, FAB-60 ml), is performed with a SF6 gas of 5.6 sccm at the high voltage of 2.0 KV and accelerated current of 20 mA. The HfO2 gratings are then released by a combination of anisotropic and isotropic dry etching of silicon, which makes the HfO2 grating freely suspend (step d). The anisotropic etching of silicon is carried out to produce vertical silicon trenches and the isotropic etching is used to release the HfO2 gratings laterally, where the remained EB resist and HfO2 film act as the etching mask. The freestanding HfO2 gratings are finally generated by removing the residual resist (step e).

III. Experimental results and discussion

Figure 2(a) shows one scanning electron microscope (SEM) image of the cross-section of the HfO2/Si platform. The thickness of HfO2 film is about 180 nm. The FAB is made up of the energetic neutral beam flux with high directionality and thus, the manufacturing method is capable of high anisotropic etching of HfO2 film. There is no special requirement of etching mask, and EB resist can serve as an etching mask. Fabricated freestanding HfO2 grating illustrated in Figure 2(b) consists of 60-period grating with the grating length of 60 μm, and air is the low refractive index materials on the bottom and top. The grating period and the grating width are expressed by \( P \) and \( W \). The duty ratio \( D = W/P \) is defined as the ratio of the grating width to the grating period. Figures 2(c) and 2(d) illustrate the zoom-in SEM images of the fabricated freestanding HfO2 gratings, where the grating period is 1040 nm and the grating height is about 180 nm, the same as the HfO2 film thickness. Since the thickness of EB resist varies due to the proximity effect in EB lithography, the HfO2 gratings generated in reality are trapezoidal profiles and deviate from the designed rectangular elements. The corresponding bottom grating widths \( W_b \) are measured ~780 nm and ~670 nm, and the top grating widths \( W_t \) are about 500 nm and 440 nm, respectively.

The simple process is scalable for fabricating suspended HfO2 nanostructures, and facilitates monolithic integration of optoelectronic devices on various material systems. Figure 3(a) shows freestanding circular HfO2 grating, and the inset is the zoom-in SEM image of circular grating with the grating period of 500 nm, where cross arms are connected to the freestanding circular gratings. From the fabrication point of view, the undercut of silicon beneath the HfO2 grating region tends to be difficult when the duty ratio \( D \) increases. On the other hand, the long HfO2 grating beams are in the tendency of being fragile, and the deflection and fracture of HfO2 grating beams take place when the duty ratio \( D \) decreases. According to our experimental results, the duty ratio \( D \) is feasible in the range of 0.3~0.7 to successfully achieve freestanding HfO2 gratings. Moreover, anisotropic and isotropic dry etching of silicon will result in rough silicon surface and large variation in air-gap between HfO2 grating and silicon beneath HfO2 grating region, which will degrade the optical performance. In association of deposition and etching techniques, this fabrication issue can be solved and such freestanding HfO2 nanostructures are possible to be incorporated into other material system for serving as
the top mirror. Freestanding HfO$_2$ photonic crystals illustrated in Figure 3(b) are realized on a GaN-on-silicon platform, and the inset is the zoom-in SEM image of freestanding photonic crystal structures with the period of 600 nm. Between HfO$_2$ film and GaN layer, one sacrificial film is inserted. After removing the sacrificial layer, HfO$_2$ photonic crystals are freely suspended and the airgap is controlled by the sacrificial layer thickness. These results indicate that the proposed process is feasible to fabricate freestanding HfO$_2$ nanostructures.

It should be noted that the HfO$_2$ gratings are designed by using rigorous coupled wave analysis (RCWA) method with a commercial code. The generated HfO$_2$ gratings deviate much from the ideal elements used for RCWA simulations (not shown here). The trapezoidal grating profiles, roughness of the grating sidewalls, and
variations in silicon surface beneath the grating region degrade the optical performance and result in the spectral shift. Moreover, the available spectral range is from 1460 nm to 1580 nm in our measurement system. Hence, a variety of HfO$_2$ gratings with different grating parameters are fabricated for optical characterization. Figure 4(a) illustrates one optical micrograph of fabricated HfO$_2$ gratings, where the upper two gratings are with the grating widths $W_t$ of 440 nm. The color varies as the grating width changes. The grating widths $W_t$ are about 500 nm for the bottom gratings, and the grating periods are 1020 nm and 1040 nm, respectively. The inset is the magnified view of fabricated HfO$_2$ grating, where the grating period $P$ is 1020 nm and the grating width $W_t$ is about 440 nm. A tunable laser (Agilent 81682A) is used as the light source to characterize the optical response of the fabricated freestanding HfO$_2$ gratings in the telecommunication range. The polarized light beam is incident onto the HfO$_2$ gratings by an infrared objective lens with a numerical aperture of 0.25, and an infrared CCD camera is installed on the setup to acquire sample images. The reflected light is collected and sent to an infrared spectrometer. The experimental spectra are normalized to those of a commercial gold mirror. Figure 4(b) illustrates the reflectance spectra of freestanding HfO$_2$ gratings, where the grating widths $W_t$ are about 440 nm. Taken 1040 nm period HfO$_2$ grating as an example, a broad reflection band that is determined by the refractive index contrast is observed under transverse electric (TE) polarization (TE is polarized in the plane of the grating and parallel to the grating lines) [19]. Two sharp reflection dips are found at 1486 nm and 1562.7 nm with measured reflectance of 10.7% and 4.6%, respectively. Measured reflectances are over 70% in the range of 1499.2 m~1539.5 nm. Since fabricated HfO$_2$ gratings are configured with one-dimensional symmetry, their optical responses are polarization dependent, which are measured by rotating the sample
with an angle of 90° with respect to initial measurement. The reflection band shifts and the shape changes under transverse magnetic (TM) polarization (TM is polarized in the plane of the grating and perpendicular to the grating lines). The linear grating reflector is useful for controlling the polarization on a vertical cavity surface emitting device. A blue-shift is observed in reflectance spectra with decreasing the grating period. As the grating period decreases from 1040 nm to 1020 nm, the broad reflection band shifts to shorter wavelength. These results indicate that freestanding HfO2 grating is a promising candidate for single layer dielectric reflector.

IV. Conclusions
In summary, freestanding HfO2 gratings are realized by a combination of FAB etching of HfO2 film and dry etching of silicon substrate. Period- and polarization-dependent optical responses of fabricated HfO2 gratings are experimentally characterized in the reflectance measurements. The simple process is feasible for fabricating freestanding HfO2 grating that is a potential candidate for single layer dielectric reflector.

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Authors' contributions
YW carried out the device design and fabrication, performed the optical measurements, and drafted the manuscript. TW carried out HfO2 film evaporation. YK participated in its design and optical characterization. KH conceived of the study, and participated in its design and coordination. All authors read and approved the final manuscript.

Competing interests
The authors declare that they have no competing interests.

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