RefRACTIVE INDEX SENSING USING THE METAL LAYER IN DVD-R DISCS

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In recent years, grating based bio-sensors have received much attention due to their promising applications in integrated sensing devices. However, production of high quality, large scale and low cost metal gratings is still challenging. Here, we introduce an extremely simple and low cost method to fabricate metal gratings by peeling off the metal layer from a DVD-R disc. An atomic force microscope image shows that the metal layer is a high quality grating, the period and depth of which are 740 nm and 86 nm, respectively. Based on the fabricated metal grating, refractive index sensing is experimentally achieved using two configurations, where either the resonant wavelength or the modulated laser power is measured. The sensitivity of the sensor by wavelength modulation reaches as high as 637 nmRIU⁻¹, which is comparable with or even higher than that of the existing grating coupled sensors. Our method largely reduces the cost to fabricate high quality metal gratings and will promote the development of grating based SPR sensors.

Characterization of the metal grating

CD, DVD and Blu-ray are three types of Commercial optical discs. They generally consist of polycarbonate layers, data layers and metal layers. The surface microstructures of these layers are spiral gratings with typical periods of 1600 nm, 740 nm and...
320 nm for CD, DVD and Blu-ray, respectively. In this paper, we choose DVD-R to prepare metal gratings for its appropriate grating period to excite SPPs in the visible. Compared with the infrared and the ultraviolet, visible signals are much easier to be detected by silicon based detectors, which are low cost and high-performance.

In our experiments, a SONY brand DVD-R disc was used to prepare the metal grating. The structure of the disc is schematically drawn in Fig. 1. It consists of several layers, including the label or printable surface, polycarbonate substrate, metal reflective layer, organic dye, polycarbonate substrate and readout surface. The surfaces of the polycarbonate substrate and also the metal layer are spiral gratings that are fabricated by the manufacturer. To isolate the metal layer, we first cut the DVD-R disc into slices by scissors along the red dashed lines in Fig. 1. And then we split the disc into two sides at the interface between the metal layer and the organic dye layer, as shown in Fig. 1. These two layers are glued together and it is easy to separate them. At last, the side with metal layer was washed with isopropanol to remove the residual dye. After the above steps, we successfully prepared the metal grating, the image of which is shown in Fig. 2(a).

An atomic force microscope (AFM) was used to characterize the surface topography of the metal grating. The AFM image and the 2D profile are shown in Fig. 2(b) and (c), which show a grating with a period of 740 nm and a peak-to-valley modulation depth of 86 nm. The fabricated metal grating has a smooth sinusoidal profile. This profile guarantees the generation of sharp and deep plasmon resonant peaks.

We measured the Energy-dispersive X-ray (EDX) spectrum of our sample to analyze its composition. In our experiments, the metal layer is adhere to the polycarbonate substrate very tightly. It is hard to separate them. So we measured the EDX spectrum of the sample that contained both the metal layer and the polycarbonate layer. The EDX spectrum are shown in Fig. 2(d), which have three major peaks corresponding to carbon (C), oxygen (O) and aluminum (Al). Therefore, the material of the metal layer here is Al. We also measured DVD-R discs purchased from other companies, namely UNIS and RITEK. The periods of the gratings are 737 nm and 735 nm, respectively. The modulation depths are 106 nm and 91 nm, respectively. As can be seen, the structural parameters of DVD-R discs produced by different manufacturers are very close.

**RIS by wavelength modulation**

The metal grating prepared by our method can be used to construct various plasmonic devices. Here we experimentally demonstrate RIS by measuring the resonant wavelength shift resulted from the refractive index change. Fig. 3 shows the schematic of the setup, where the reflection spectra of the metal grating irradiated with oblique incident white light were recorded. The white light source was a deuterium halogen combo light source. Firstly, the white light was collimated by an achromat lens. Then it transmitted through a Glan–Taylor polarizer to select the TM polarization (electric vector being
perpendicular to the grating direction). An aperture diaphragm was added after the polarizer to adjust the spot size illuminated on the metal grating. The metal grating was fixed on the inner surface of a cuvette, where the test liquids were added. The angle between the incident light beam and the cuvette surface normal direction was set to 30 degrees. Finally, the reflected light were directed into a fiber optic spectrometer to record the reflection spectra.

Theoretically, for surface plasmons propagating at a flat interface between two semi-infinite media, the SPP wave vector can be expressed by

$$k_{\text{spp}} = \frac{\omega}{c} \sqrt{\frac{\varepsilon_m \varepsilon_d}{\varepsilon_m + \varepsilon_d}}$$

where $\omega$ is the angular frequency, $c$ is the speed of light in vacuum, $\varepsilon_m$ and $\varepsilon_d$ are the dielectric constants of the metal and the dielectric, respectively. The dispersion relation of surface plasmons on grating surfaces here will diverge a little with this wave vector. But it is still a good approximation for us to calculate the resonance wavelength using this wave vector. In the grating coupling scheme, the additional wave vector should be introduced to match $k_{\text{spp}}$. In the SPP coupling scheme, the additional wave vector is provided by diffraction of light on the grating. For the optical geometry as shown in Fig. 3, surface plasmons are excited when the following relation is satisfied:

$$\frac{2\pi}{\lambda} n_d \sin \theta + m \frac{2\pi}{A} = \text{Re}\{k_{\text{spp}}\}$$

where $n_d$ is the refractive index of the dielectric, $\theta$ is the incident angle, $m = 0, \pm 1, \pm 2, \ldots$ is the diffraction order, and $A$ is the grating period. This relation implies that one can obtain the value of $n_d$ by measuring the resonant wavelength $\lambda$.

According to the above equation, the resonant wavelength is dependent on the incident angle. By fixing the refractive index to be 1.333, the dependence of resonant wavelength on the incident angle is calculated. The results are shown in Fig. 4. It should be noted here that the value of the angle in Fig. 4 is fixed on the inner square with this wave vector. By extracting the resonant wavelength from the reflection spectra, we can plot the resonant wavelength as a function of the refractive index, which is depicted in Fig. 6. The wavelength shows a linear relationship with the refractive index, and the deviation is very small. For a SPR sensor, the sensitivity (S) and the figure of merit (FOM) are two important parameters that are widely used to quantify its performance. The sensitivity is defined as the ratio of the change of the wavelength to the change of the refractive index ($S = \Delta \lambda/\Delta n$). According to the linear fitting of the experimental data, which is shown as the red line in Fig. 6, the sensitivity of this setup is 637 nmRIU. The figure of merit is defined as FOM = $S$/FWHM, where FWHM is the full width at half maximum of the resonance. In our experiments the FWHM is less than 30 nm, and the corresponding FOM is more than 21.

In previous reports, H. Chen et al. chemically prepared five types of different gold nanoparticles. The index sensitivities of those nanoparticles range from 44 to 703 nmRIU$^{-1}$ and the FOM range from 0.6 to 4.5, with their FWHM ranging from 50 to 140 nm. By controlling the plasmon resonance wavelength to be around 730 nm, they measured the sensitivities of gold nanocrystals of 156 to 326 nmRIU$^{-1}$. Moreover, L. Shao et al. prepared macro-scale colloidal metal nanocrystal arrays with sizes up to $10 \times 10$ cm$^2$. The refractive index sensitivity of the gold nanorod arrays is 304 nmRIU$^{-1}$ and the FOM is up to 3.09.
Recently, T. Iqbal et al. fabricated 1D gold grating using FIB etching, which has a sensitivity of 525 nmRIU⁻¹. Compared with these previous reports, the metal grating prepared in this paper has comparable or even higher refractive index sensitivity. More importantly, due to the smooth surface morphology of our grating, we obtain SPR spectrum with small FWHM, resulting in a much higher FOM than those nanocrystals and nanostructures.

**RIS by power modulation**

RIS by wavelength modulation needs a broadband light source and a spectrometer, which increase the complexity and cost of the system. Alternatively, RIS by power modulation needs a laser and a power meter, which is more promising to design biosensors with high integration densities. Thus we also built a setup to demonstrate RIS by measuring the modulated power. The schematic of the setup is illustrated in Fig. 7(a). The laser beam emitted from a HeNe laser was adjusted to TM polarization. This beam passed through the tested liquid and then irradiated on the metal grating at an oblique angle. The laser beam reflected by the metal grating was diffracted into different orders. Among these diffracted beams, the zeroth order one had the highest power. The power of the first order beam is 19% of that of the zeroth order beam. Other diffraction orders were too weak to be detected. Therefore, detection of the zeroth order beam gave the best signal to noise ratio. In the experiments, the zeroth order beam was detected by a power meter and an aperture diaphragm was installed to block the high order diffracted beams and the environmental light. In order to precisely adjust the incident angle, the cuvette was mounted on a rotating platform.

According to the experimental results in the above section, our sensor has high sensitivity in the wavelength range of 600–800 nm. Therefore, we choose HeNe laser as the probing light, which has a wavelength of 632.8 nm lying in the optimal wavelength range. The incident angle of the laser beam will affect the resonant wavelength and further affect the relationship between the power and the refractive index of the dielectrics. Considering the laser wavelength of 632.8 nm, we shifted all resonant wavelengths in this experiment (corresponding to refractive index of 1.333–1.3635) to be less than 632.8 nm by setting the angle between the laser beam and the cuvette surface normal to 36 degrees. In the experiments, all reflectance measurements are performed with the same incident power of 2.14 mW.

**Fig. 5** Reflection spectra of the metal grating with different liquids on its surface.

**Fig. 6** The dependence of the resonant wavelength on the refractive index. The red line is a linear fitting.
Deionized water and glucose solutions of concentrations of 5%, 10%, 15%, 20% were tested under this configuration. Fig. 7(b) shows the reflected power as a function of the refractive index. As can be seen, the power is linearly dependent on the refractive index, which is a highly desirable property for SPR sensors. The results demonstrate that the metal layer in DVD-R disc is also suitable to design SPR sensors by power modulation. The size of the setup illustrated in Fig. 7(a) can be further reduced by replacing the HeNe laser and the power meter with a laser diode and a photodiode, respectively. This strategy promises the design of lab-on-a-chip SPR sensors.

Conclusions

In conclusion, we presented an extremely simple method to fabricate metal gratings by peeling off the metal layer from a DVD-R disc. The fabricated metal grating has a smooth sinusoidal surface profile, which can support sharp and deep SPR. Based on the metal grating, two refractive index sensors were demonstrated by either measuring the wavelength shift or the power shift. Our sensor has a high sensitivity of 637 nmRIU\(^{-1}\), which is comparable with or even higher than that of the existing sensors based on grating coupling. Our method largely reduces the cost to fabricate high quality metal gratings and will promote the development of grating based SPR sensors.

Fig. 7 (a) Schematic of the setup to demonstrate RIS by power modulation. (b) The reflected power as a function of the refractive index. The red line is a linear fitting.

Conflicts of interest

There are no conflicts to declare.

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