Silicon-on-quartz bonding based SPR chip

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1 Introduction

Surface plasmon resonance (SPR) is the resonant oscillation of conduction electrons at the interface between a negative and a positive permittivity material, stimulated by incident light. The SPR effect has been used as a basic tool for measuring material adsorption on metal surfaces, thin metal films and metal gratings (Otto 1968; Kretschmann 1981; Wood 1902), and it has been used in a number of applications such as gas detection, analysis of biomolecular interactions, detection of chemical analytes and lab-on-a-chip sensors (Liedberg et al. 1983; Flanagan and Pantell 1984; Homola et al. 1999). After the original concept of the traditional and popular Kretschmann coupling configuration based SPR sensor was described by Liedberg et al. in 1983, most commercial SPR sensors are generally based on the Kretschmann coupling configuration because of its simple fabrication and robust measurement of interactions at the metal-environment interface (Hoa et al. 2007).

In SPR based biosensing, the biological and chemical analytes in solution flow through a thin channel above the surface of the thin metal film (Sjolander and Urbaniczky 1991; Liedberg et al. 1993). Generally, gold and silver are most commonly used as the thin metal film (Homola et al. 1999). Figure 1 shows the schematic design of a SPR chip based on the Kretschmann coupling configuration for the observation of the SPR effect. In this configuration, two design constraints have to be considered. The first constraint is the poor adhesion of thin metal films to glass. In order to obtain a good performance of the SPR chip, the thin metal films must adhere strongly to the surface of a glass or quartz substrate. In case of using gold or silver as a thin metal film, they do not adhere well to oxidic surfaces such as glass and quartz (Hooqviet and Van Bennekom 2001). To improve the adhesion between the metal film and...
glass or quartz substrate, Cr or Ti is generally used as an adhesion layer with a maximum thickness of 5 nm (Fontana et al. 1990). However, the quality factor of the SPR effect is decreased by using these adhesion layers. Figure 2 shows the simulation result of the adhesion layer influence on the quality factor of the SPR effect using COMSOL Multiphysics simulator (Altsoft Co.). In this study, all simulations were conducted with near-infrared incident light which has a wavelength of 975.1 nm. The second design constraint is the influence of the air-gap distance on the SPR effect. Miniaturization of the channel height (i.e. air-gap distance) in microfluidic systems is an important trend, motivated by the need to use expensive reagents and precious samples sparingly. Also a number of researchers have proposed microfluidic systems with miniaturized channel height (Xiang et al. 2006; Cesaro-Tadic et al. 2004). In the Kretschmann coupling configuration, the channel height cannot be miniaturized because it has to be much larger than the wavelength of the incident light, to avoid interference with the upper wall or substrate (Hoa et al. 2007). Figure 3 shows the simulation result of the air-gap distance, d, influence on the SPR effect in the Kretschmann coupling configuration. As a result of this simulation, in order to minimize the degradation of the quality factor of the SPR effect, the air-gap distance has to be about 4 times larger than the wavelength of the incident light.

In case of the Otto coupling configuration which has not been studied thoroughly for optical sensing, the surface of the metal film is separated from the glass substrate as shown in Fig. 4 (Otto 1968). To prevent the influence of the substrate on the SPR effect in the Otto coupling configuration, the thickness of the thin metal film has to be larger than the penetration-depth of the incident light. Thus, the adhesion layer between the metal and substrate does not affect the quality factor of the SPR effect. Additionally the channel height can be thinner compared to SPR chips based on the Kretschmann coupling configuration (Fontana et al. 2015).

Fig. 1 SPR chip based on the Kretschmann coupling configuration

Fig. 2 Adhesion layer influence on the SPR effect in the Kretschmann coupling configuration

Fig. 3 Air-gap distance influence on the SPR effect in the Kretschmann configuration

Fig. 4 SPR chip based on the Otto coupling configuration
Furthermore, opaque materials such as silicon, which is being widely used in electronics for the fabrication of integrated circuits, can be used as a substrate. In this paper, an Otto coupling configuration based SPR chip is designed, simulated and fabricated using a silicon-on-quartz (SoQ) bonding process.

2 Experimental methods

2.1 Design and simulation

A schematic view of the Otto coupling configuration based SPR chip is shown in Fig. 5. The proposed SPR chip consists of a quartz substrate and a silicon substrate with an initial cavity of 2.5 μm-depth. An air-gap distance for the critical SPR effect with optical constants of the materials, which are interpolated from (Lide 2005) and (Rakic et al. 1998), was calculated to be about 2.16 μm using Fresnel reflection formulation (Kretschmann 1981; Chen and Chen 1981). In this design, a 300 nm-thick gold layer is used as a thin metal film which is placed on the silicon cavity. Figure 6 shows the simulation result of the SPR effect based on the design parameters using COMSOL. Because of the thick metal film of the proposed SPR chip, the substrate layer is not taken into account on the simulation of the SPR effect. The simulated resonance angle and reflectance of the critical point are 42.21° and 0.363°, respectively.

2.2 Fabrication

Generally, glass and silicon wafers are bonded together using the anodic bonding process. The anodic bonding process is an effective method to achieve a perfect bonding including a precise gap distance between the glass and silicon layers (Henmi et al. 1994). In this process, a voltage of 500–1000 V is applied between the silicon and glass at a temperature of 350–400 °C. In case of our proposed SPR chip, a very high electric field, formed due to the very large area of the gold surface in addition to having a very small gap of 2.2 μm between the glass and the silicon substrate, can damage the surface of the gold film (Wallashi and Levit 2003).

In this study, to avoid the effect of the electric field, formed by the high voltage, the proposed Otto configuration based SPR chip was fabricated using a SoQ bonding process as shown in Fig. 7. First, a 2.5 μm-deep cavity is machined on a 500 μm-thick Silicon substrate using a deep reactive ion etching (DRIE) process using a positive photoresist mask. Then a 300 nm-thick Au layer is deposited with an adhesion layer (Cr, 10 nm-thick) on the bottom of the silicon cavity, using the lift-off process. After RCA1 cleaning of the quartz and the silicon wafers, the surface of the silicon wafer is activated using oxygen RIE plasma. Then the prepared silicon wafer and the 500 μm-thick quartz wafer are manually bonded (Suga et al. 2004). Finally, the bonded wafer is annealed on a hotplate under the proper condition (temperature of 200 °C) for 2 h. Figure 8 shows the fabricated SPR chip.
2.3 Measurement

The SPR effect of the fabricated Otto chip is measured using an automated reflectometer with the measurement setup shown in Fig. 9. In order to observe the SPR effect, the fabricated SPR chip is placed on the top surface of a right angle coupling prism (BK7). To avoid unwanted reflection at the interface of the SPR chip quartz and prism, the SPR chip is kept in optical contact with the prism by using an index matching gel. The incident laser beam, which has a wavelength of 975.1 nm, is polarized parallel to the incidence plane. The part of the laser beam, which is reflected by the splitter, is detected by a reference photodetector, $D_r$. After the laser beam is reflected at the surface of the gold layer, the output laser beam is detected by a photodetector, $D_o$. The detected signals from $D_r$ and $D_o$ are
sent to a computer through the channels of a data receiving device (DAS-16) for data processing.

In order to measure the SPR effect which has a narrow angular range of the incident laser beam at resonance, angle and position of the sample (i.e. a SPR chip) have to be controlled with high-precision. In this measurement setup, the angle of the incident laser is controlled across 12° with a resolution of 0.005° by using computer-controlled step motors. A translation compensation algorithm is also used to ensure that the footprint of the laser beam on the surface of the gold layer is maintained at the same position during an angular scan (Fontana and Cavalcanti 2013). Figure 10 shows the measured SPR curve according to the angle of incidence. The measured resonance angle and reflectance at the critical point are 42.19° and 0.411°, respectively.

3 Discussion

The resonance angle and reflectance of the measured critical point does not agree perfectly with the simulation result. One possible source of discrepancy is the surface roughness of the gold layer. In practice, the surface roughness and average gold grains per area, in gold films deposited using evaporation and sputtering processes, were investigated as a function of the temperature and length of substrate prebake time by a number of researchers (Levin et al. 1997; Semaltianos and Wilson 2000; Elbel et al. 1995). When the wavelength of the incident beam is comparable to the dimensions of the surface roughness, the diffraction of electromagnetic waves can be changed according to the surface roughness of the material (Toigo et al. 1977). The surface roughness of the deposited gold film is measured using an atomic force microscope before the SoQ bonding process. The root-mean-square value of the surface roughness is calculated to be 2.3 nm. This measured value is too small to affect the SPR effect, in the infrared spectral region (Fontana and Pantell 1988). Another important consideration is a wide range of measured optical constants (Boreman et al. 2011). In the Otto coupling configuration, the air-gap distance for maximum resonance depends on the gold optical constants. Figure 11 shows the simulation result of the influence of the optical constants, which are reported in (Olmon et al. 2012; Babar and Weaver 2015; Gao et al. 2011) as shown in Table 1, based on the design parameters. A large variation of SPR couplings can be observed by considering gold optical constants available in the literature. Thus, when developing an SPR chip based on the Otto coupling configuration, the air-gap distance must be designed according to the used optical constant.

Table 1 Gold optical constants reported in references

| Reference         | Refractive index, $n$ | Extinction coefficient, $k$ |
|-------------------|-----------------------|-----------------------------|
| Hamola et al.     | 0.26796               | 6.0045                      |
| (1999)            |                       |                             |
| Suga et al.       | 0.15037               | 6.4188                      |
| (2004)            |                       |                             |
| Toigo et al.      | 0.096397              | 6.1986                      |
| (1977)            |                       |                             |
| Wallashi and Levit| 0.68698               | 6.1700                      |
| (2003)            |                       |                             |

In order to obtain the optical constant of the gold film fabricated in this study, we used the “FindFit” function available on the Mathematica software with a nonlinear regression analysis algorithm. This function gives the optical constant of the fabricated gold film for a given measured SPR curve and design parameters. Figure 12 shows the result of the “FindFit” function and the obtained optical constant of the gold film is shown in Table 2, this value is used as a design parameter in a post simulation. A
simulation result of SPR effect based on the fabricated SPR chip with the newly obtained optical constant is shown in Fig. 13. The simulated resonance angle and reflectance of the critical point are 42.15° and 0.402°, respectively. Substantial agreement is found between the simulation result and the experimental data.

4 Conclusions

In this study, the Otto configuration based SPR chip is designed and fabricated. The SPR effect was measured. Discrepancy between the measurement result and simulation result is discussed by optical constant of the gold layer used as a thin metal film. The measured result agrees well with the simulation result based on the fabricated SPR chip and the SoQ bonding process is a feasible approach for implementation of Otto configuration based SPR chips. The optimization of the air-gap distance will be conducted as a further study based on the optical constant of the fabricated thin gold film.

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