Surface Plasmon Resonance in Thin Gold Films

Martin Magnuson

Laboratory of Applied Physics, Linköping Institute of Technology,
S-581 83 Linköping,
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Abstract

Surface plasmon resonance (SPR) is an optical technique that can be used for biosensing. The present work accounts for calculations and experiments with different glass prisms in both air and water. The possibility of making useful sensing probes combined with optical fibres is discussed.
1. Introduction

A surface plasmon is a charge-density oscillation in the surface of a thin metal film. P-polarized light can excite such an oscillation, and a resonance phenomenon can be observed (SPR) by measuring the reflected light intensity (fig. 1). At a certain angle of incidence the wave vector for the incident light coincides with the wave vector for the surface plasmon. Resonance will occur with a dramatic drop in intensity. By measuring the change in the angle of minimum reflectance for a given system it is possible to use SPR as a sensor for surface adsorption processes.

![Fig. 1: A surface plasmon in a free-electron metal film.](image)

The present work aims at investigating the possibility of making small SPR probes combined with fibre optics. These probes can possibly be used for in situ measurements, eg. in blood vessels.

2. Experimental setup

A xenon light source was used together with a monochromator with a bandwidth of 24 Å. The wavelength was kept at 6328 Å (He-Ne laser wavelength) and the light was focused with a lens having a focal length of 10 cm.

In the experiments all metal films were composed of 500 Å gold on top of a 2 Å chromium layer. The substrates used were either prisms made of BK7 glass with an index of refraction of 1.5151 or pieces of microscope slide glass. The index of refraction of the slide glass was measured with an ellipsometer to be about 1.506. The thickness of the gold film was optimized by computer calculations. The films were fabricated by thermal
evaporation with the substrate at room temperature and a pressure of $5\cdot10^{-7}$ Torr in the evaporation chamber.
The morphology of one gold film was studied by SFM (scanning force microscopy) which showed variations in thickness of ±100 Å.

Two different modes of observation of the surface plasmon resonances can be used;

1) Measuring the reflectance as a function of wavelength.
2) Measuring the reflectance as a function of angle of incidence.

In these experiments both collimated and focused light were used and the reflectance was measured as a function of the angle of incidence. In the case of focused light, dark field detection can be used.

![Diagram of experimental setup](image)

**Fig. 2:** Experimental set-up were the lens is used when focused light is desired.

### 3. Experimental results in air

Calculations were first made for a metal film on a glass plate in air, using the parameters for $n$, $k$, and $d$ as listed in table 1. A specially designed software package (SPANA) was used for the calculations. The minimum reflectance was found to be at an angle of incidence of 43.5°, comprising a sharp dip according to fig. 3.
Table 1: The parameters and the resulting thickness of the gold film used for calculating the angle of minimum reflectance.

<table>
<thead>
<tr>
<th>Layer</th>
<th>n</th>
<th>k</th>
<th>d (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>glass</td>
<td>1.5151</td>
<td>0</td>
<td>∞</td>
</tr>
<tr>
<td>gold</td>
<td>0.16</td>
<td>3.63</td>
<td>500</td>
</tr>
<tr>
<td>air</td>
<td>1</td>
<td>0</td>
<td>∞</td>
</tr>
</tbody>
</table>

Fig. 3: The calculated reflectance as a function of the angle of incidence with the metal surface in air.

3.1. Experiments with 60° glass prisms

Fig. 4: The light beam configuration in a 60° prism.
Fig. 5: Measured reflectance with a 60° prism in air.

As seen in fig. 5, the minimum reflectance was obtained at an incident angle to the SPR surface of 44.7°; corresponding to the theoretical value 43.5° (fig. 3). As seen in fig. 5 the dip does not go down to zero. This shows that the thickness of the film probably diverges from the calculated. The deviation of the angle of the dip is probably due to the fact that the refraction indices (n, k) of the gold film are not known exactly. External angles were measured with the instruments but here only the internal angles between the incident light and the glass-metal interface are given, taking the refraction at the air-glass interfaces into consideration. Because of repeated total reflections inside a 60 degree prism, this configuration can not be used with one side as a mirror to get the reflected light back in the incident direction.
3.2. Experiment with a 90-45° glass prism, one of the walls serving as a mirror

![Diagram of a 90-45° prism with a mirror](Image)

**Fig. 6:** The light beam configuration in a 90-45° prism.

The SPR dip was found at 44.5°, and the light was reflected back in the incident direction with a small lateral displacement. The divergence from the theoretical value (43.5°), is probably due to a slightly different index of refraction in the prism and the gold film.

3.3. Experiment with only one reflection in glass

In this experiment an attempt of miniaturization of the prism was made with an home polished microscope slide glass piece. The surfaces were polished such that the incident and refracted light should hit the surfaces at right angles according to the calculated SPR value.

![Diagram of a small slide glass piece](Image)

**Fig. 7:** The light beam configuration in a small slide glass piece.
Fig. 8: Measured reflectance with a small microscope slide glass piece.

With this small glass piece the SPR dip was found to be at 42° according to fig. 8. The SPR-angle was reasonably close to the expected value 43.5°. The divergence can be explained by varying index of refraction within the glass piece, and that the home polished surfaces were not perfectly planar.

3.4. Experiments in glass with multiple reflections at the gold film

Fig. 9: The light beam configuration in a glass with 6 reflections at the gold film.
Fig. 10: The calculated reflectance as a function of the incident angle for 6 reflections.

Multiple reflections give rise to widening of the dip. In this case calculations were made by multiplying the Rp spectrum 6 times (see fig. 10). It was found that the dip becomes much broader and less well defined than for a single reflection.

Fig. 11: Measured reflectance with multiple reflections in a microscope slide glass piece.
After the repeated reflections the minimum of the dip should be at 43.5° but, because of the widening caused by repeated reflections, the SPR dip was found at 42 to 43 degrees. If multiple reflections against the gold film is desired one should use a film thickness where the resonance dip does not go down to zero for a single reflection.

4. Experimental results with water as medium

Calculations were made using the parameters in table 2 for n, k, and d. The minimum reflectance should be at 71.0° with a wider dip than in air according to fig. 12. The experiments were done with the SPR surface of the glass pressed against a hole in a cuvette containing water.

Table 2: The parameters used for the calculation of the angle of minimum reflectance in water.

<table>
<thead>
<tr>
<th>Layer</th>
<th>n</th>
<th>k</th>
<th>d (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>glass</td>
<td>1.5151</td>
<td>0</td>
<td>∞</td>
</tr>
<tr>
<td>gold</td>
<td>0.16</td>
<td>3.63</td>
<td>500</td>
</tr>
<tr>
<td>water</td>
<td>1.334</td>
<td>0</td>
<td>∞</td>
</tr>
</tbody>
</table>

Fig. 12: The calculated reflectance as a function of the incident angle in water.
4.1 The 60° prism.
The incident angle to the prism should be 71.0 degrees by calculations. Experimentally, the dip was found at about 73-76 degrees according to fig. 14. It is also seen that the dip is wider and more shallow than in air. Measurements in water are more difficult than in air and the divergence from the calculated curve is larger. Measurements above about 78 degrees were nearly impossible since the incident beam to the prism became parallel with the SPR surface.

![Fig. 13: The light beam configuration in a 60° prism in water.](image)

4.2 A piece of glass with one reflection.
Experimentally, the SPR dip was found at 68.5 to 70 degrees according to fig. 16, compared to the 71.0° calculated. However, at low angles the measurements were not good, probably due to that part of the light beam undergoes a second reflection.

![Fig. 14: Measured reflectance in a 60 degree prism in water.](image)
Fig. 15: The light beam configuration in a microscope slide glass in water.

![Figure 15](image)

Fig. 16: Measured reflectance in the microscope slide glass shown in fig. 15.

![Figure 16](image)

5. Suggestions for further work

The configuration in fig. 6 is very interesting for measurements in air. It should be possible to construct a dark field probe with light detection on the same side as the light source by using eg. a semi-transparent mirror to separate the incident and reflected light beams. It should also be possible to combine this prism with a bunch of optical fibres with a diode array for detection according to fig. 17a. However, the prism would have to look quite different if it should be used for detection in water. In this case one could perhaps use a prism configuration as shown in fig. 17b or a parabolic prism as shown in fig 17c.
Fig. 17: A possible prism-fiber combination; a) in air, b) and c) in water.

To show that the light within the fibre can be coupled out and a spatial resolution of the different light modes be obtained, both step index fibres and gradient index fibres (diameter 62.5 µm) have been etched with hydrogen-flouride. The etching was controlled with a laser and a detector at each end to measure the damping and to know when the cladding was gone. The fibres were first etched for 30 minutes to get rid of the cladding and a few extra minutes on the core to make it thinner at the end. Glycerol was used as an index of refraction-match to couple out the different light modes. However, it appeared to be difficult to couple out all the light and, as the core was made thinner and thinner, it became very delicate.

There is still much work to be done in order to make a complete sensor. One possible sensor configuration is shown in fig. 18 where eg. laser light is focused into the left part of the fibre and the different light modes are coupled out at the right hand side with spatial resolution, after interacting with the SPR film. The light can eg. be detected with a CCD array along the etched part of the fibre.

Fig. 18: A possible probe using a simple glass fibre without a prism.
6. Conclusions

It has been shown that it is possible to generate surface plasmons in gold films on thin slide glass pieces. There are no principal obstacles to miniaturize this further into a useful probe. It has also been shown that such a probe would work in a liquid.

When several reflections against the gold film is desired, one should use a film thickness where the reflectance minimum does not decrease to zero for a single reflection. The geometry with a 90-45° prism should be useful in a prism-fiber combination in air. For water the prism must have a different geometry according to fig. 17b, or a parabolic prism might be used (fig. 17c).
7. Acknowledgements
This work was done at the Institution for Applied Physics, University of Linköping, as part of a project for development of new biosensors. The following persons contributed substantially to this work: Ingemar Lundström, Hans Arwin, and Bo Liedberg in the Applied Physics group in Linköping and, concerning optical fibres, Gunnar Edwall and Adel Asseh, Royal Institute of Technology, Stockholm.

8. References

1. Jorgenson Ralph C., Yee Sinclair S., Johnson Kyle S., Compton Bruce J.; *A Novel Surface Plasmon Resonance Based Fiber Optic Sensor Applied to Biochemical Sensing* (to be published)

