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Surface plasmon resonance sensors – novel architecture and improvements

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The paper deals with an optical sensor based on the phenomenon of surface plasmon resonance. It proposes a new geometry of the measurement head, which allows for measurements with both the change in the incident light angle and the change in the wavelength. The proposed sensors can also be used in a parallel and a series configuration, which increases the functionality of the setup, allows for a greater precision of measurements, and eliminates such distracting factors as temperature change. The article presents the results of a computer analysis of the sensor, as well as the analysis of its capabilities.

Keywords: surface plasmon resonance (SPR), optical sensor, chemical sensor, temperature compensation, multisensing SPR.

1. Introduction

Optical sensors have recently been increasingly used in chemistry and biology; hence the need for constant improvement of their functionality. Optical sensors are based on various phenomena connected with the interaction of light and matter. One of such phenomena is surface plasmon resonance (SPR) [1]. It occurs when conditions for resonance of an electromagnetic wave propagating along the metal–dielectric boundary are fulfilled. Due to the fact that resonance occurs only for a narrow range of parameters, sensors based on this phenomenon are characterized by high sensitivity. In the case of SPR, the main parameter influencing the conditions of resonance is the refraction index of the examined substance. That is why SPR sensors are often used to measure this parameter. In order to measure a different parameter, such as concentration or biospecific interactions, the sensor will be designed in a way which combines the change of the parameter with the change of the refraction index.

So far, the refraction index (RI) has been established by means of measuring the intensity of the electromagnetic wave near the resonance [2], measuring its intensity depending on the angle of the incident light [3], or its intensity depending on the wavelength [4]. There are also setups where the phase change [5] and the polarization of light are examined [6]. Theoretically, the highest precision of measurement is achieved for angular interrogation-based sensors [7, 8]. This precision will be defined as the derivative of the monitored SPR parameter (angle or wavelength) with respect to the parameter to be determined (refraction index). In order to increase the measurement precision and partly eliminate the factors distorting the measurement, such as temperature, one can either simultaneously measure the change of the angle and the wavelength [9] or use multi-canal setups [10]. The typical resolution of the SPR sensors is 10^{-6} RIU (refraction index unit) [7].

This paper presents a new architecture of the SPR sensor based on a prism in an attenuated total reflection (ATR) configuration. This architecture allows for miniaturizing the SPR sensor and using angular and wavelength modulation simultaneously. It also allows for measurements in a wide RI range and a wide range of angles without any rotating elements such as a high-cost goniometer. With the new architecture, it is possible to build multi-channel setups in both the parallel and series configurations. Additionally, for less exact measurements, the low-cost and commonly available elements, such as a laser pointer or an internet camera, can be used.

2. Sensor design

Before designing the setup, several general assumptions were made. The main assumption was that the RI measurement should be possible for both the angular modulation and wavelength modulation without intervening with the head of the SPR setup. This determined the choice of the setup configuration, which was based on the Kretschmann geometry, where the excitation of surface plasmons is performed by the attenuated total reflection (ATR) [11]. As for the type of an input and output beam, the plane wave was chosen. Such a configuration makes the setup user-friendly, and gives a possibility of using non-standard light source elements, as well as non-standard setups to register the output signal. Moreover, it also allows for joining the sensors in parallel and series architectures.

The second assumption concerning the design dealt with signal detection. It was assumed that the modulation of the incident light angle on the dielectric–metal boundary would not be continuous like in the prism coupler setup types, but it would take a finite number of discrete values. Thanks to such an approach, for the wave lighting at a given angle, the surface of the detector registering the signal would be larger than in a classical setup [3]. It would allow for averaging the results and therefore for eliminating part of the errors. That is why the requirements concerning the quality of the fabrication or multiplication of the SPR head would not be critical for the functioning of the setup. As shown further, such an approach does not result in decreasing the sensitivity of the SPR sensor.

Taking into consideration the general assumptions discussed above, three versions of the SPR "head" were proposed and analyzed. Their simplified schemes are presented in Fig. 1. Each scheme shows only two measurement channels, but the architecture allows for using a number of them. In each of the cases discussed, both the incident and the output beam presented take the form of a plane wave. The measurement

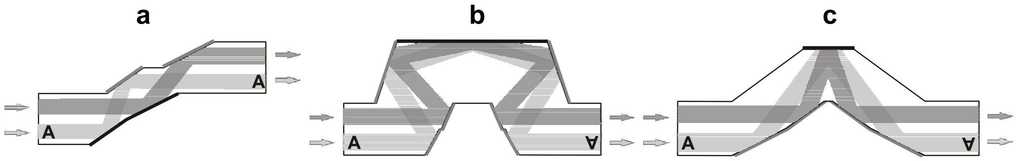

Fig. 1. Three schemes of the simplified version of the SPR sensor head. The bold black line marks the part of the sensor where SPR takes place. The bold gray line marks the reflecting surfaces whose aim is to form the light beam.

element where SPR takes place is marked with a bold black line. The surfaces where the conditions of wave reflection will be fulfilled are marked in bold gray lines.

In the case of the sensor in Fig. 1**a** the light wave gets to the tilted planes (the bold black line) where SPR takes place. The tilt of the surface, different for each of the channels, allows for the interaction of the electromagnetic wave with the dielectric–metal–dielectric boundary at various angles. The task of the reflecting elements (a bold dark gray line) is to reshape the beams from the separate channels into one output plane wave. An advantage of such an approach is that all the light reflections within the head take place in the conditions of total internal reflection, and the beam leaving the sensor has parameters similar to the incident beam. The main disadvantage of this head is the fact that SPR takes place on the surface which is not flat. Technologically, it may result in problems with the exact spattering of the metal layers of the required thickness. It also eliminates the possibility of using exchangeable working elements such as the dielectric–metal plate with biomolecular coating, and makes it difficult to use the sensor in flow systems. Another disadvantage of the setup may be the adjustment because the output plane wave will be shifted in relation to the incident wave.

Figure 1**b** presents the second proposal for the measurement head of the SPR sensor. In this case, the incident plane wave is formed by two sets of mirrors (bold dark gray lines) in the area where SPR takes place (a bold black line). The aim of the mirrors is to divide the input wave into smaller beams and to change their lighting angle. In order to obtain the plane wave at the output of the sensor, the setup is symmetrical and the beam achieves the desired form thanks to another two sets of mirrors. The main advantage of this setup is the flat metal–dielectric boundary where SPR takes place. A drawback is the necessity of using four sets of mirrors forming the beam and the fact that the setup works outside the total internal reflection region, which requires an additional metallization of the side surfaces of the SPR head. Due to the limitations in the precision of fabricating the mirrors, such a number of reflections in the setup may considerably influence the quality of the results obtained. However, the use of such a configuration gives a chance of reducing the size of the SPR head. With this configuration it is also possible to continuously change the lighting angle of the incident beam.

Figure 1**c** shows the third proposal for the SPR measurement head of the sensor. In this case, the first system of mirrors (bold gray lines) forms the incident wave into several beams, each of them lighting at a different angle on the plane where SPR takes place (a bold black line). The second symmetrical system of mirrors finally forms the beam into the plane output wave. The advantage of this setup is once again the flat metal–dielectric boundary where SPR takes place, which allows for the use of exchangeable working elements. Additionally, in this setup all the light reflections within the head take place in the conditions of total internal reflection, which does not require a special sputtering of the mirrors and considerably simplifies the fabrication process of the element. A disadvantage is the lack of possibility of obtaining the continuous change of the lighting angle of the incident beam, when considering the real values of the refraction indexes of the materials of the head and the substances examined. Also, the image registered at the output of the setup is not continuous due to the fact that the surface of the mirrors forming the beam takes the form of steps.

In all the versions of the SPR head described above the input plane wave is divided into narrower beams and each of the beams is directed towards the SPR surface at a different angle. The concrete values of the lighting angles of the beams depend on the materials of which the sensor is fabricated, the range of the refraction index to be measured by the sensor, as well as the length of the electromagnetic wave used. A characteristic of the setups presented in Figs. 1**b** and 1**c** is the fact that the beam is reversed in each of the channels, which is symbolically presented with a letter *A* at the input and the output of the setup. This feature does not influence the quality of the work of the setup. The advantage of the two setups is the fact that the output plane wave is not shifted, as in Fig. 1**a**, which simplifies the adjustment of the setup.

3. Computer simulation

The aim of the computer simulation was to check the influence of various parameters of the SPR head on the accuracy of the results obtained. The parameters used were: a different number of the measurement channels, the change of the wavelength and the use of a parallel architecture and series architecture of the sensor. In the simulations it was assumed that the SPR head was made of SF11 glass of refraction index $n = 1.77862$ ($\lambda = 632.8$ nm) [12] and the active area was covered with a 50 nm layer of silver. The range of the refraction index measured was set between 1.3 and 1.4.

For parameters defined in such a way, the range of the incident angles varies from 50 to 58 degrees because for such angles it is possible to obtain the minimum intensity of the reflected light. Figure 2 presents the dependence of the reflection on the incident angle for two wavelengths. The gray rectangle shows an arbitrary choice of angles from 48 to 58 degrees, for which measurements are to be taken. Figure 3 shows an example of the measurement results obtained for the proposed SPR head with 11 measurement channels. The figure also shows a scheme of the measured RI value of the examined substance. The measurement consists in interpolating the SPR curve to the results obtained with the change of one parameter only, which is RI. The main parameter influencing the accuracy of the results obtained is the number of the mea-

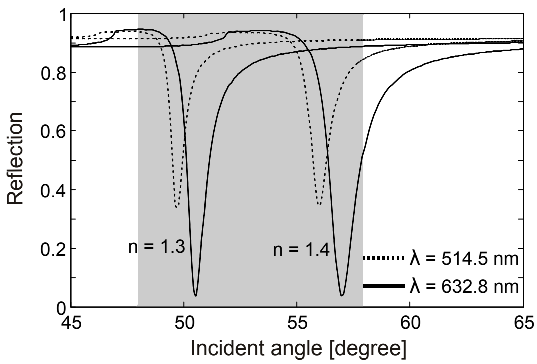

Fig. 2. The dependence of the reflection on the incident angle for two wavelengths. The gray rectangle shows an arbitrary choice of angles from 48 to 58 degrees, for which measurements are to be taken.

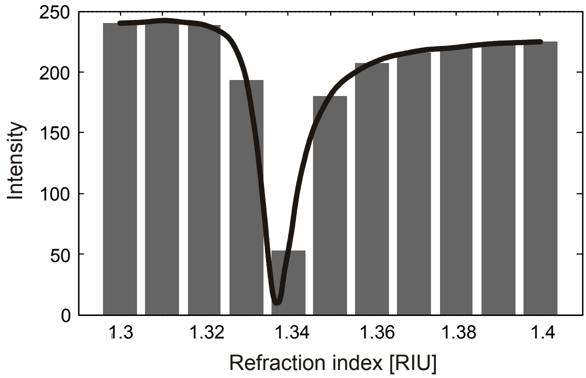

Fig. 3. Sample measurement results obtained for the proposed SPR head with 11 measurement channels, for RI $n = 1.33$. The figure also shows a scheme of the measured RI value of the examined substance.

surement channels of the SPR head. In this case, the number of channels is understood as the number of various angles for which a single measurement can be taken.

In order to estimate the sensitivity of the setup, it was checked how the minimal change of the refraction index (Δn) of the examined substance changes the results on the detector. The examination was carried out for the full range of RI from 1.3 to 1.4 with the step of $\Delta n = 0.0001$ [RIU]. Due to the non-continuous characteristic of the measurement, its accuracy depends on the RI of the examined substance. Figure 4 presents exemplary results obtained for a head with 11 channels and an 8-bit dynamic range of the detector.

Figure 5 shows how the number of channels and the dynamic range of the detector influence the accuracy of the measurements. From the data in Fig. 5 it seems that the theoretical sensitivity of the setup increases together with the growing number of channels. Theoretically, the number of channels should be as large as possible; however, the geometry of each of the proposed heads limits the number of the discrete

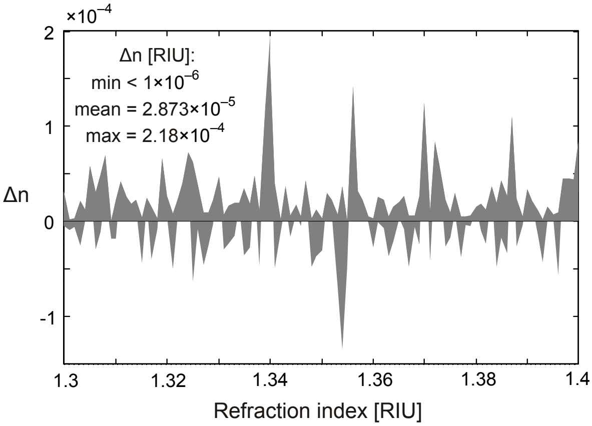

Fig. 4. The dependence of the sensitivity of the SPR on the RI of the examined substance. Exemplary results obtained for a head with 11 channels and an 8-bit dynamic range of the detector.

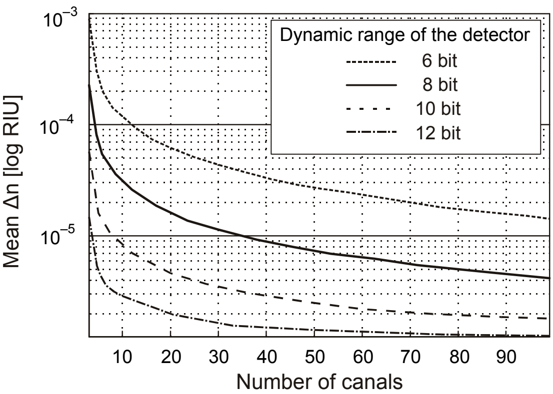

Fig. 5. The dependence of Δn on the number of channels for the various dynamic ranges of the detector.

angle values. Taking into consideration the technological possibilities of fabricating the SPR heads, the single channel should not be too narrow, since then the precision of the element fabrication may considerably influence the quality of the results obtained. So, the ultimate number and size of the channels depend on the size of the SPR head and the technology of its fabrication. For the purpose of the simulation it was assumed that the SPR head was ideal and its size unimportant.

Yet another parameter influencing the quality of the work of the setup is the dynamic range of the detector. The data in Fig. 5 show that increasing the sensitivity of the detector considerably influences the accuracy of the measurements. However, it is possible to measure the refraction index at the level of 3×10^{-5} RIU already with the 256 levels of the light intensity registered on the output of the SPR head.

The advantage of the presented SPR sensor is the possibility of increasing its sensitivity through the use of the same head to take measurements for several different

T a b l e. The accuracy of the refraction index measurements.

Wavelength [nm]	Mean Δn [RIU]
632.8	2.85×10^{-5}
514.5 + 632.8	1.95×10^{-5}
488 + 514.5 + 632.8 + 694.3	1.09×10^{-5}
450−850 (step 1 nm)	1.05×10^{-8}

wavelengths. The Table presents the possible accuracy of the RI measurements for different numbers of electromagnetic wavelengths used. The data obtained show that for a head with 11 channels and an 8-bit dynamic range of the detector, each doubling of the number of the wavelengths used increases the sensitivity of the setup 1.5 times on the average.

4. Influence of the fabrication flaws

The actual realization of the proposed SPR head will differ from the ideal situation assumed in the simulations. That is why this part of the paper deals with the influence of the fabrication inaccuracy on limiting the work of the sensor.

The first important factor to consider is the level of precision in the fabrication of the SPR head. The inaccuracy, however, does not influence the work of the setup in a negative way because the surface where measurements are taken in each of the channels is quite large, and the results are averaged. On the other hand, errors resulting from the imprecise angles of the mirrors can be included in the analysis of the results and recalculated, either during reference measurements, or on the basis of the measured geometrical parameters of the element.

Another important factor influencing the work of the real setup may be the incident angle of the plane wave. However, obtaining the proper angle does not pose problems due to the character of the proposed head, since it does not change the direction of the plane wave. The accuracy of the setting may be estimated at 6×10^{-4} degrees, assuming that the pitch size of the camera used as detector is 10 μm.

5. Parallel and series configurations

The number of the channels available can be increased in a simple way without changing the size of the SPR head and the size of a single channel. It can be done through parallel joining of several SPR heads, each of them containing channels tilted at a different angle. The problem of penetration of the light wave between the heads can be eliminated thanks to a reflective layer placed between every two heads. An example of such a setup containing three SPR heads is presented in Fig. 6**a**. Yet another possibility of increasing the functionality of the setup is the use of series configuration, which is shown in Fig. 6**b**. In both configurations, the parallel and the series one, it is possible to use a different type of metal or biomolecular coating in

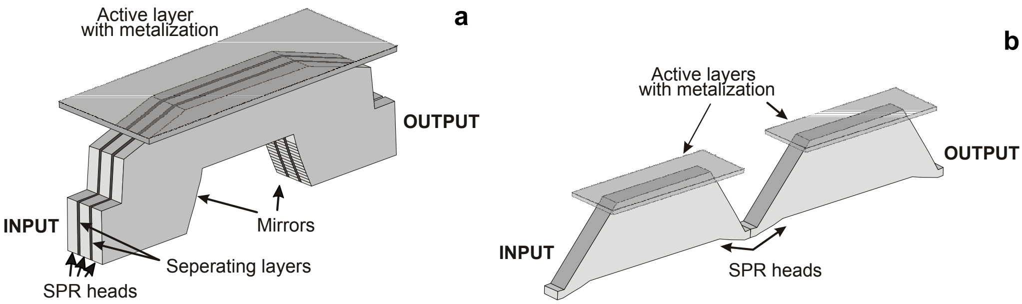

Fig. 6. A complex SPR head with the separated active area where SPR takes place. Parallel configuration (**a**), series configuration (**b**).

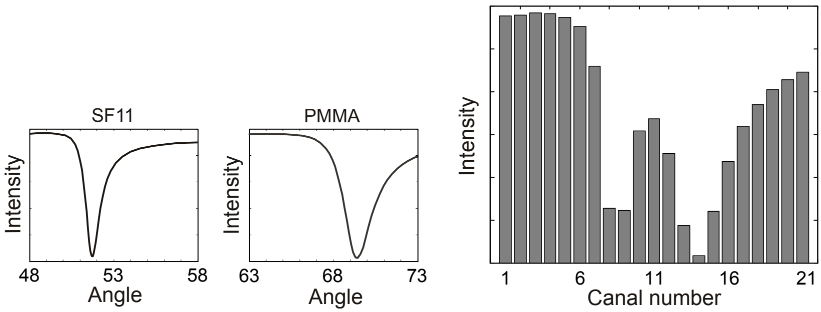

Fig. 7. The characteristics of the SPR for the SF11 glass + Ag and PMMA + Ag setups. The results for the series configuration with 21 channels in each head.

each of the heads. The material of each head can also be different, which further increases the capabilities of the setups discussed. It is also possible to separate the active part of the head where SPR takes place from the part with channels, as presented in Fig. 6. The separation allows for reusing the heads.

Simulations carried out show that the minimal thickness of a single head will depend on both the quality of the plane wave and other size parameters of the head. The maximum thickness, on the other hand, will depend on technological limitations and problems with fabricating thick elements. Taking into consideration these two parameters, the thickness of the head can be set at 250 μm, because such elements can be easily obtained, for instance with the use of the LIGA method [13]. For practical reasons, when designing the parallel configuration of the SPR heads, it is necessary to place the input and the output areas of the heads in one plane next to each other.

Figure 7 presents exemplary results of a computer simulation for a series configuration of two heads made of SF11 glass and PMMA and coated with silver.

6. Compensating for the influence of temperature

One of the main parameters distorting the accuracy of RI measurement is temperature [14]. With measurement resolution of about 10^{-6} RIU it is important that the RI of all the elements of the setup is temperature dependent. Temperature changes not only the RI of the examined substance, but also the RI of the dielectric and the metal of which the head is fabricated. Optimally, the temperature of the setup and the examined substance should be stable; however, it is not always possible. That is why various possibilities of limiting the influence of the temperature are sought [15].

In the case of the heads presented here, compensating for the influence of the temperature is achieved through the use of parallel or series configuration of the heads, and can be done in two ways. Firstly, one of the heads takes the measurement proper and the second control head is kept in stable conditions to give a reference measure. A disadvantage of this solution is the difficulty of using the configuration in flow setups, where the parameters of the examined substance change dynamically. Such a difficulty is avoided in the second case where each of the SPR heads is fabricated from a different dielectric. Here, however, the temperature dependence of the refractive index of each of the materials of the heads must be different. The bigger the difference, the more precise is the compensation of the influence of temperature on the work of the setup.

In order to explain the measurement with the compensation for the influence of the temperature, let us assume that the two heads are made of SF6 glass (n = 1.77862, t = 20 °C) and BK7 glass (n = 1.51322, t = 20 °C). The temperature dependence of their refractive indexes are 7.5×10^{-6} and 1.7×10^{-6}, respectively (for room temperature) [16]. Let us also assume that the change of the refractive index of the examined liquid (n = 1.333333, t = 20 °C) is 2×10^{-5} for each grade. Additionally, we can assume that both the heads have 21 channels and the detector has a 10-bit dynamic range.

At room temperature t (20 °C) the interpolation of the theoretical SPR curve to the obtained experimental results is the best. That is why, for room temperature t, the measurements taken by each of the two heads of the setup will be identical, taking into consideration the number of channels and the sensitivity of the detector. For the numerical values discussed here, the RI of the examined substance of the SF11 head is n_{SF11} = 1.33333, and of the BK7 glass it is n_{BK7} = 1.33333, which means that the measurement is taking place in optimal conditions. In the next step, let us discuss what will happen if the refraction index of a new examined substance is n = 1.33313. The results obtained from the interpolation of the theoretical SPR curves for both the heads discussed and for the temperature t = 20 °C will also be congruent n_{SF11} = = n_{BK7} = 1.33313.

Let us now examine how the temperature changes by Δt = 5 °C can influence the measurement in the both heads (for the substance of n = 1.33313). It should be remembered that the interpolation of the theoretical SPR curves still happens, on the assumption that the measurement is taking place at room temperature t = 20 °C. It turns out that thanks to the difference in the temperature dependence of the refractive indexes of the two types of glass, the measurements from the two heads will begin to differ (n_{SF11} = 1.33311, n_{BK7} = 1.33313). In this case, in order to establish the real value RI and the temperature of the measurement, it is necessary to try to interpolate the curves in such a way that they give the same RI value at the changed temperature.

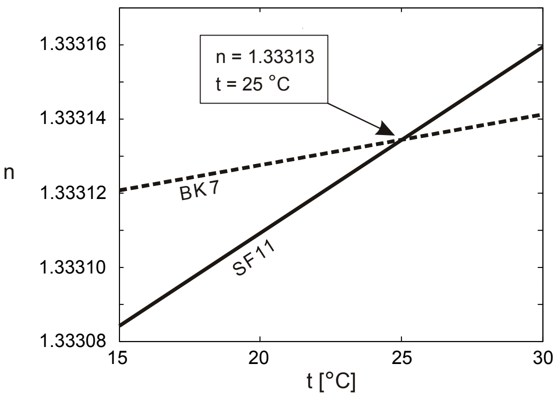

Fig. 8. Compensating for the influence of the temperature. The scheme of the procedure for finding *n* and *t*, for which the interpolation of the theoretical SPR curves is the best.

Figure 8 presents a scheme of such an interpolation. It shows how the minimums of the measurements, for which the interpolation of the theoretical SPR curves is the best, will change for the two heads. The crossing point of the two curves gives the value of the RI ($n_{SF11} = n_{BK7} = 1.33313$) and the temperature ($t = 25$ °C), which we were looking for.

The results presented above clearly show that the presented configuration compensates for the influence of temperature. The RI measure in the setup is independent of temperature without any additional elements such as temperature stabilizing devices. This simplifies the sensor and makes it more flexible and cost--effective.

7. Conclusions

The paper presented a proposal of a new configuration of a sensor for measuring the refraction index with the use of the phenomenon of the surface plasmon resonance. The main advantage of the proposed solution is the possibility of using the same head for the measurements in both the angle function and the wavelength function. This increases the functionality of the setup and allows for higher accuracy of the measurement. An additional advantage of the setup is its scalability consisting in the use of several heads in a parallel or series configuration. This allows for higher precision of the measurements on the one hand, and the compensation of the influence of temperature on the other. The results of the computer simulations carried out show that with a single SPR head with 11 channels, a single wavelength and a detector with an 8-bit dynamic range, the precision of the RI measurement is in the range of 3×10^{-5} RIU. When increasing the number of heads, channels, or wavelengths for which the measurement is taken, this value can be improved. Taking into consideration practical limitations of the parameters mentioned above, the accuracy of measurement of the proposed setup is in the range of 1×10^{-7} RIU. This resolution is comparable with the best methods currently used.

The proposed configuration of sensors opens up a possibility of scaling the setup, depending on the measurement accuracy needed. Taking measurements with the change of the wavelength and/or the change of the incident light angle makes the setup more flexible and adjustable to the character of the measurement taken. The setup is also more cost-effective due to the wider choice of its elements.

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