Surface enhanced infrared absorption (SEIRA) spectroscopy using gold nanoparticles on As$_2$S$_3$ glass

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Abstract:

The development of infrared sensing platform requires adapted materials. In this work, we used sputtered gold nanoparticles on a chalcogenide glass to demonstrate that these infrared materials can be used as substrate for surface enhanced infrared absorption (SEIRA) spectroscopy. The sputtering parameters were optimized to get the highest possible enhancement. To assess it, a self-assembled monolayer of 4-nitrothiophenol was used. These preliminary results are promising and pave the way for various configurations of waveguide for integrated optical components.

Keywords: Chalcogenide glass ; Sputtering Gold nanoparticles ; 4-Nitrothiopheno l; SEIRA ; Surface enhanced infrared absorption
1 Introduction

Surface enhanced infrared absorption (SEIRA) was observed for the first time in 1980 by Hartstein et al. [1]. It was shown that the infrared absorption of organic compounds can be enhanced thanks to metallic nanoparticles by many orders of magnitude. Since this discovery, several studies have been published on the SEIRA effect to elucidate this phenomenon with various metals (Ag, Au, Cu, Pt) and molecules [2-7]. Surface plasmon resonance was found to be the origin of SEIRA even if it is not fully understood. Plasmon resonance frequency of metallic nanoparticles can be tuned in the infrared domain depending on their morphologies [8]. Thus, SEIRA have been demonstrated with various morphologies such as thin films [4, 7, 9-12], nanorods [13, 14] and nanoshells [15, 16]. Since most of the infrared signatures of organic species and biomolecules are included in the mid-infrared domain, SEIRA can be used to improve the infrared detection limits of molecules. Most reports on SEIRA [16-18] have been implemented using crystals (CaF$_2$, Ge, Si) which cannot be easily shaped into integrated optical devices devoted to mid-infrared. In this paper, chalcogenide glass is used as substrate due to its transparency in the mid-infrared. These materials can be transparent up to 20 µm, depending on their composition and, moreover, they can be shaped into integrated optical waveguide such as optical fibers, planar or ridge waveguides in order to fabricate some advanced optical sensing devices [19-24]. As far as we know, all the SEIRA substrates are made of crystalline material. For this first study about chalcogenide glass SEIRA substrate, the well documented fabrication method of DC sputtering was chosen as a starting point.

The monitoring of the environment requires reliable and highly sensitive devices. Many international research teams have then worked on the development of in-situ sensors for reducing the analysis time and cost. Among the different techniques, infrared spectroscopy is
robust, versatile and reliable [25-27]. However, in coastal and estuarine natural waters, concentrations of pollutants such as hydrocarbons vary between ppb to ppm [28]. The aim of this preliminary study is to demonstrate that chalcogenide glass could be a new material for SEIRA substrate and then pave the way to the development of integrated optical waveguide for SEIRA spectroscopy. To our knowledge, this is the first observation of SEIRA on gold nanoparticles carried out in the infrared range thanks to a chalcogenide glass substrate.

2 Materials and experimental methods

2.1 Synthesis of infrared glass substrate

The infrared glass substrate used in this work was As$_2$S$_3$ glass. It was synthesized in an amorphous silica tube with high purity raw As (99.999%) and S (99.999%) elements. Sulfur was pre-purified at 130 °C for 19 h to eliminate water and sulfur oxide (SOx). Arsenic and sulfur were placed in two different tubes and turbomolecular vacuum ($10^{-6}$ Torr) was used to exclude water and oxygen. During vacuum, arsenic was heated at 290 °C for 2 h to remove, by volatilization, the As$_2$O$_3$ surface layer. After this step, the elements were placed under vacuum in the same silica tube which was then sealed. To exclude organic impurities, the silica tube was inserted in a distillation furnace and the temperature was increased to 750 °C and held at this temperature for 2 h before decreasing to room temperature. The glass was then melted in a rocking furnace at 850 °C overnight to obtain a homogeneous mixture and then next gradually cooled down to 500 °C and held for 1 h at this temperature in order to reduce the gas pressure in the tube. The melted glass was quickly quenched by immersing the silica tube in water (20 °C) and immediately placed in a furnace for annealing. The temperature of the furnace was a few degrees under the glass transition temperature Tg (192 °C) and the glass was annealed for a few hours before a gradual cooling to room temperature. Annealing is necessary to reduce
the residual stresses resulting from the cooling. The resulting glass rod was 1 cm in diameter and 10 cm in length. It was sliced into 3 mm thick disks using a diamond wire saw. The surface of the disks was mirror polished using alumina powder (grain size: 0.5 µm). The resulting glass disks were 2 mm thick.

2.2 Gold film deposition

Chalcogenide glass used in this study contains sulfur which ensures a good adhesion of gold on the surface via Au-S bonding and no surface treatment is required. Chalcogenide disks were sonically cleaned using acetone and isopropopanol (analytical grade, Prolabo and Carlo Erba). Gold films were then directly deposited on disks by direct current sputtering. Deposition was performed in a home-built small-sized DC sputtering device using a water-cooled coaxial cathode allowing target-substrate distance control. Deposition was performed under a pure Argon atmosphere, pumped-off with a LN2 sorption device (Riber) especially efficient against oil and water contaminations. The stainless steel substrate holder had a Thermocoax and a thermocouple built-in, and gold was sputtered at room temperature and at 180 °C taking into account the glass transition temperature Tg (192 °C) of the chalcogenide substrate. Temperature was adjusted with the use of a KS-40 industrial temperature controller. Before deposition, the base pressure was reduced below $10^{-2}$ mbar and then increased between $5 \times 10^{-2}$ and $6 \times 10^{-2}$ mbar after adding of argon via a needle microvalve. A number of sputtered gold films were deposited at room temperature varying the sputtering time from 30 seconds to 110 seconds with a current intensity of 0.62 mA and at 180 °C with a lower current intensity of 0.14 mA for 30 minutes. A 2.54 cm in diameter target was used and was constituted of a pure gold foil (99 % purity, 0.5 mm thick, Goodfellow), glued with silver charged conductive epoxy glue on a copper holder screwed on the negative lead of the coaxial electrode. A small part of
the substrate was masked with felt-tip ink to locally prevent gold deposition and then cleaned with ethanol to measure of film thickness by Atomic Force Microscopy (AFM).

2.3 Self-assembled monolayers preparation

The self-assembled monolayer of 4-nitrothiophenol (technical grade, Sigma-Aldrich) was prepared by soaking the gold-coated substrate in 20 mL of a $6.5 \times 10^{-5}$ M aqueous solution of 4-nitrothiophenol for 5 h at room temperature, and then rinsed with ethanol and dried under a flow of dry nitrogen.

2.4 Infrared measurements

2.4.1 Optical properties

The infrared transmission spectra of the chalcogenide glass As2S3 and the optical extinction spectra of gold films were acquired with two spectrometers. The extinction spectra were recorded by the measurement of absorbance which represent Log(1/T). For the visible to near-infrared region (0.6-2 µm), measurements were made by a UV-visible-NIR Perkin-Elmer lambda 1050 spectrometer. And for the near to mid-infrared range (2-14 µm), measurements were performed by a Fast Fourier Transformed Spectrometer (FFTS, Bruker VECTOR 22, Bruker optics Inc, Germany). All the spectra were recorded using a co-addition of 30 scans at 4 cm$^{-1}$ in transmission mode. The background spectrum of the atmosphere was measured as a single beam and was used as reference.

2.4.2 SEIRA spectra
The SEIRA spectra of 4-nitrothiophenol on gold films were recorded on FTIR Thermo Nicolet spectrometer in transmission mode, using a DTGS detector, a resolution of 4 cm\(^{-1}\) and a co-addition of 256 scans. The background spectrum of atmosphere was measured as a single beam and was used as reference. A baseline correction was performed to eliminate the absorption of the substrate covered by gold nanoparticles in order to show the absorption of 4-nitrothiophenol. The SEIRA spectra are represented according to the thickness as previously reported by others authors [3, 5, 7].

2.5 Atomic Force Microscopy

Gold film morphology and thickness were investigated by Atomic Force Microscopy (Dimension 3100, Nanoscope V, Digital Instruments, Santa Barbara CA, USA), operated in tapping mode using a doped silicon tip (BS-TAP300Al) with an average radius of less than 10 nm and a resonant frequency of 300 kHz. The AFM was calibrated using a 21.5±1 nm step height Z axis calibration grating (TGZ1 from NT-MDT). The mean thickness was measured thanks to a mark left by a permanent marker on the substrate surface prior to gold deposition. After which it was dissolved away with ethanol.

2.6 Conductivity of gold film

The conductivity of the gold films was checked with an ohmmeter. The two tips were placed in contact with the gold surface on different areas and were separated by few millimeters. Gold film was considered to be discontinuous when resistance higher than 1000 W was measured and the ohmmeter was made a sound.
3 Results and discussion

3.1 Optical transmission of As$_2$S$_3$ glass

The transmission spectrum of a representative As$_2$S$_3$ glass disk is shown in Fig. 1.

In terms of bulk glass, the optical transparency of the As$_2$S$_3$ glass extends from 0.7 µm to 10 µm, in Fig. 1. The lower limit of the transmission is due to the electronic band gap of the glass where the electronic lone-pair p states forming the top part of the valence band play an important role. In the infrared region, the transparency is limited by low energy phonons. It’s worth noting that most of the infrared signatures of organic species or biomolecules are included in this spectral domain. This chalcogenide glass is suitable for infrared detection. Moreover, it can be shaped into sophisticated optical devices such as optical fibers, planar or ridge waveguides. First of all, one has to demonstrate the feasibility of the SEIRA effect on polished vitreous plates of As$_2$S$_3$.

3.2 Atomic Force Microscopy characterization of As$_2$S$_3$ glass disk surface and gold films

The roughness of the polished As$_2$S$_3$ glass disk surface and the topography of sputtered gold films were characterized by Atomic Force Microscopy (AFM). Fig. 2 shows AFM pictures of the polished substrate surface before (a) and after the sputtering of gold (b, c, d) and the cross-section profile of a gold film (e, f).

As seen from Fig. 2a, the surface of the substrate remains rough even after polishing. The surface roughness computed over 1×1 µm$^2$ area is about 3.5 nm. The scratches are about 1 nm
in depth. Note that a smooth surface is essential to ensure the homogeneity of the metal film growth [29]. As shown in Fig. 2b, 2c and 2d, nanoparticles cover the whole surface of the vitreous substrate after gold sputtering. The film is made up of densely packed gold nanoparticles. At room temperature, the diameter of these gold islands is around $17\pm4$ nm (Fig. 2c) but because of the AFM tip curvature radius the real diameter might be slightly less than 17 nm. Conductivity tests show that gold films are non-conducting until 80 seconds of sputtering at room temperature and also for 30 minutes of sputtering at lower current intensity at room temperature and at 180 °C. For non-conducting gold films, gold nanoparticles are supposed to be separated. The distance between each nanoparticle, called air gap, could not be directly measured because of the AFM tip size of about 10 nm and the density of coverage. The mean gap between the particles is supposed to be less than 10 nm. Furthermore, Scanning Electron Microscope (SEM) images did not show the nanoparticles. They are then below the resolution of the microscope. The nanoparticles are supposed to be separated by only few nanometers. For the sputtered gold film deposited at 180 °C (Fig. 2d), the islands seem to be larger with a diameter of about $34\pm8$ nm.

Fig. 2e and 2f show the AFM picture and section profile of a gold film sputtered at room temperature for 70 seconds. The step between the uncovered As$_2$S$_3$ substrate surface (right) and the gold surface (left) is clearly visible. Using such a profile, the average height of each step has been evaluated versus the deposition time and reported in Table 1.

The results show an increase in gold thickness with the sputtering time. The percolation threshold is reached between 80 seconds and 110 seconds of sputtering. Gold islands touch each other and the gold film becomes conductive.
3.3 Extinction of discontinuous gold island films

The optical extinction of a metallic nanoparticle represents the scattering of light and the absorption due to surface plasmons collective oscillations [30, 31]. Fig. 3a and 3b show the optical extinction spectra of a series of gold island films sputtered at room temperature with various thicknesses on As$_2$S$_3$ glass and for 30 minutes at room temperature and at 180 °C, respectively.

Fig. 3a shows that the extinction becomes broader and more intense as the thickness of the films increases. Such a kind of SEIRA substrate does not present a well-defined plasmon band and the same dependence of extinction on the thickness of silver film on CaF$_2$ substrate [32, 33] and gold films on silanized glass [33] has already been reported. For isolated gold spherical nanoparticles with diameter ranges from 10 nm to 30 nm, the extinction is located in the visible range at around 520 nm and is quite sharp [8, 30, 34, 35]. When several spherical nanoparticles are very close to each other, the optical extinction becomes broader and shifts to longer wavelengths (red-shift) because of an inter-particle coupling [31], as also explained by Gans theory. The plasmon resonance in the infrared is supposed to happen because of the coupling between nanoparticles that requires an air gap of few nanometers.

Also, the optical extinction of gold films depends on sputtering temperature, as observed in Fig. 3b. With increasing temperature of the substrate during sputtering, the intensity of extinction becomes weaker and a shift to shorter wavelengths is observed (blue shift). In fact, higher temperature of the substrate enables the Au adsorbed atoms to move more freely over the surface of the substrate during the deposition to interact among themselves forming bigger islands by agglomeration. Consequently, the air gap between the nanoparticles can increase leading to the disappearance of inter-particle coupling, as already mentioned in many reports [9,
The correlation between extinction spectra and SEIRA spectra will be detailed later in this article.

### 3.4 SEIRA spectra of 4-nitrothiophenol

As in previous studies [3, 5], a monolayer of 4-nitrothiophenol was used to assess the SEIRA phenomenon. The infrared spectrum of 4-nitrothiophenol monolayers and randomly oriented are well known. Fig. 4 shows the infrared transmission spectrum of randomly oriented 4-nitrothiophenol on the chalcogenide substrate.

The band assignment is made according to the work of Zhang and Imae [3]. The strongest bands are located at 1336 cm\(^{-1}\) and 1512 cm\(^{-1}\) and correspond to NO\(_2\) symmetric and antisymmetric stretching modes, respectively. Regarding SEIRA, we focused our attention on the 1300/1530 cm\(^{-1}\) region.

The infrared spectra of 4-nitrothiophenol on sputtered gold films on As\(_2\)S\(_3\) substrates at room temperature and at 180 °C are shown in Fig. 5a and 5b, respectively.

First of all, a strong band is observed at 1500 cm\(^{-1}\) which is attributed to normal CS\(_2\) residual impurities in the As\(_2\)S\(_3\) bulk glass (Fig. 5a and 5b). This band is observed in transmission mode without gold nanoparticles on the substrate. Its intensity depends on the impurity concentration in the substrate. Moreover, the SEIRA effect is mainly located between the gold nanoparticles. Thus, no enhancement of the CS2 impurities absorption is expected.

On the other hand, the intensity of the NO\(_2\) symmetric stretching band at 1336 cm\(^{-1}\) is clearly dependent on the thickness of the gold island films (Fig. 5a). Discontinuous gold films enhance this infrared absorption band compared to a continuous gold film with a thickness of 31±4 nm.
Enhancement is observed only with discontinuous gold films because of the gap between nanoparticles whereas they are connected each other in a continuous film. As previously observed by Osawa [7], there is an optimal film thickness for enhancement which is not associated with the stronger extinction. In his paper, the optimal thickness of a silver island film is 10 nm because they become nearly continuous with the increase of thickness. In this paper, the optimal thickness is 16±1 nm and shows the best enhancement, about 10 fold higher. The gold film with a 30±3 nm is nearly continuous and the enhancement is lower about 6 fold higher.

A slight shift, about 5 cm$^{-1}$, in the frequency of the NO$_2$ symmetric band between gold films is also observed and is not explained for the moment. Also, the SEIRA effect leads to band asymmetry as previously observed [4, 5, 38].

Whatever the gold films, the band associated with the antisymmetric stretching mode of the nitro group is not observed as previously reported by Bjerke and Griffiths [5] and was explained by surface selection rules. Indeed, on a gold surface, thiol compounds are known to be linked to Au by the SH group via Au-S bonding. A monolayer of 4-nitriothiophenol is formed by molecules oriented perpendicularly on a gold surface with the nitro group on top. The dipole moment of the NO$_2$ symmetric and antisymmetric stretching are perpendicular and parallel to the gold surface, respectively. Surface plasmons of gold islands are excited by the electromagnetic field of incident light. A local electric field is generated around gold nanoparticles and is perpendicular to the gold surface at every point. In surface selection rules, only groups with their dipole moments parallel to the electric field lines are enhanced and are SEIRA active. For the NO$_2$ group of 4-nitriothiophenol, only the symmetric stretching mode is parallel to the electric field lines and is the only one enhanced and observed. In comparison, the SEIRA spectra of randomly oriented 4-nitriothiophenol show both symmetric and antisymmetric stretching modes.

In addition, the dependence of the SEIRA spectra on the increasing temperature of the substrate during sputtering has also been investigated (Fig. 5b). The results show that the
intensity of the 1336 cm$^{-1}$ band for a gold film sputtered at room temperature is four fold higher than a gold film sputtered at 180 °C. As previously, the NO$_2$ antisymmetric absorption band is not observed. A reduced coupling between the island particles is involved in the case of our gold deposition on As$_2$S$_3$ glass substrates at 180 °C. In fact, as the strength of a local electric field generated by the interaction of metal nanoparticles depends strongly on the distance between them [31, 39], the SEIRA performance is also sharply affected and the enhancement of absorption decreases with inter-particle gap increase.

4 Conclusion

Surface enhanced infrared absorption (SEIRA) spectroscopy was investigated using gold nanoparticles deposited by DC sputtering on As$_2$S$_3$ glass. For this first study on chalcogenide glass based SEIRA substrate, this well documented fabrication method was chosen as a starting point. Many other fabrication processes were reported and some demonstrate much higher enhancement factor. The highest enhancement factor ever reported is 300 000 [14]. It was achieved using nanoantennas synthesized by electron beam lithography. The high chemical and physical stability of chalcogenide glass would enable one to adapt some of these fabrication processes to these materials. It would then be possible to develop integrated optical waveguide for very sensitive SEIRA spectroscopy.

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References

Fig. 1. Transmission spectrum of As₂S₃ bulk glass with thickness of about 2 mm.

Fig. 2. AFM pictures of polished As₂S₃ surface (a), covered by gold nanoparticles at room temperature (b, c) and at 180 °C (d), cross-section profile of a gold film (e, f). All pictures are in phase contrast excepted Fig. 2f.

Fig. 3. Extinction spectra of sputtered gold films with various thicknesses on As₂S₃ glass at room temperature (a) and for 30 minutes of sputtering at room temperature and at 180 °C (b).

Fig. 4. Infrared spectrum of randomly oriented 4-nitrothiophenol on As₂S₃ substrate.

Fig. 5. Infrared transmission spectra of 4-nitrothiophenol monolayers on sputtered gold island films of various thicknesses at room temperature (a) and for 30 minutes of sputtering at room temperature and 180 °C (b).

Table 1. Change of gold film thickness with the sputtering time and conductivity. The pressure of Argon was 6.10⁻² mbar with a current intensity of 0.62 mA.
Figure 1
Figure 2f
Figure 3a

- Extinction values for different wavelengths:
  - 31 ± 4 nm
  - 30 ± 3 nm
  - 16 ± 1 nm
  - 11 ± 1 nm
Figure 3b
Figure 5a
Figure 5b
<table>
<thead>
<tr>
<th>Sputtering time (sec.)</th>
<th>Gold film thickness (nm)</th>
<th>Standard deviation (nm)</th>
<th>Conductivity</th>
</tr>
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<tr>
<td>30</td>
<td>11</td>
<td>1</td>
<td>Non-conductive</td>
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<tr>
<td>60</td>
<td>16</td>
<td>1</td>
<td>Non-conductive</td>
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<tr>
<td>80</td>
<td>30</td>
<td>3</td>
<td>Non-conductive</td>
</tr>
<tr>
<td>110</td>
<td>31</td>
<td>4</td>
<td>Conductive</td>
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Andre Perrin got the PhD degree in chemistry in 1968 and in physics in 1977 from the University of Rennes (France). He was appointed by the CNRS as researcher from 1968 to 1990 and then as Research Director, at the University of Rennes. He was involved in solid state chemistry, especially the synthesis and structure of octahedral Re₆ cluster chalcogalides and the growth and structural characterisation of thin films of various materials, including Chevrel phases, HTc superconductors and more recently ferroelectric oxides. He is the co-author of about 200 papers in these fields.

Jean-Pierre Guin received the PhD degree from the University of Rennes (France) in 2001. From 2002 to 2005, he was appointed as a guest researcher at the National Institute of Standards and Technology (NIST, USA). Currently, he is a CNRS researcher at LARMAUR laboratory from the University of Rennes. He is in charge of the glass surface damage, aging and mechanical durability research activity. Specialist in scanning probe microscopy, his research focuses on the study of fracture, permanent deformation and durability of glasses at the nanometer scale. He is the author and co-author of 30 papers, 2 patents and 50 conferences.