Experimental Optical Characterization and Polymeric Layouts of Gold PDMS Nanocomposite Sensor for Liquid Detection

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Abstract—We present a new concept of liquid sensing based on gold nanocomposite material showing enhancement of optical radiation and coupling. In particular, we experimentally characterize the optical transmittivity response of a millimeter pillar-type sensor made of gold micro/nanoparticles in PDMS material. The choice of the millimeter pillar-type layout is studied in order to collect better the light in a multimode receiver optical fiber. After a first three-dimensional (3D) finite-element method (FEM) modeling which analyzes the physical aspects of the proposed sensor, we experimentally characterize the PDMS/PDMS-Au pillar-type layout, and we measure the variation of the transmittivity response comparing different liquids, such as ethanol and oil, placed on the PDMS-Au sensor. Concerning the reusable process of the sensor, we study the dynamics of the sensing by measuring the transmittivity response during the time by applying ethanol as target liquid. Finally, we propose an implementation of the proposed sensor in robotics finger.

Index Terms—Nanocomposite materials, optical sensors, permittivity sensing.

I. INTRODUCTION

SENSORY information for feeling materials and determining their physical properties is provided by different kinds of sensors. Presently, many researchers are attempting to apply the detection ability to intelligent robot systems, and, many kinds of sensors have been introduced [1]–[19]. These sensors, which are capable of detecting contact force, vibration, texture, and temperature, can be recognized as the next generation of information collection systems. We introduce in this paper a new class of material oriented on these applications able to increase the sensing effect: this class, named nanocomposite materials (NMs), is generated by

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Fig. 1. Pillar type sensor excited by a broadband lamp source. Inset: image of the fabricated PDMS-Au prototype: the height of a single pillar is 1.5 mm, the diameter is 1 mm and the side of the square layout is 2.5 mm.

metallic/organic nanostructures added in polymer matrices. In particular, NMs strongly absorb the light becoming technologically interesting, since the light absorbed by the nanostructure can be locally released as heat. Therefore, researchers have utilized strongly absorbing metal nanostructures for a variety of applications, such as localized photothermal ablation treatment of cancer [1], [2]. Conversely, the ability of the metallic nanocomposite to strongly scatter light is critical for optical sensing, tagging and imaging applications [3], [4]. Applications involving light scattering are numerous, and include the use of metal nanostructures as contrast agents in cancer cell imaging [5], [6], the use of composite metal nanostructures for optical labeling and tagging applications [7]–[9], and the use of metal nanostructures as optically active strain sensors [10]. The use of this class of NMs are suitable for dielectric permittivity sensors [11]–[15] regarding the detection of materials. In this class is collocated the proposed sensor illustrated in Fig. 1 which is oriented for the detection of liquid materials. The new sensor is based on the detection of the radiated optical field enhanced by gold micro/nanoparticles [16]–[19] embedded in Polydimethylsiloxane (PDMS) polymer film.

Being the PDMS is able to generate by chemical reduction gold nanoparticles starting from gold precursors [16]–[19], the PDMS-Au device is characterized by a good electromagnetic (EM) field confinement due to the high refractive index consequence of the micro/nano metallic inclusions. Moreover, the pillar layout allows to direct the light as a T optical junction [see Fig. 2(a)]. This allows to collect the light source in the region above the pillar as radiated electromagnetic field. Due to the light scattering of gold micro/nano nanoparticles in PDMS [see schematic principle in Fig. 2(b)], the intensity of the radiated light will increase by supporting the detection. According to the optical collecting properties and with the proper dimensions of the pillar (the pillar diameter is similar to the optical

Fig. 2. (a) Schematic sketch of PDMS pillar and light propagation as in a T optical junction. (b) Light propagation and scattering in PDMS-Au pillar. (c) Radiation of a single pillar and experimental setup. (d) Radiation of multiple pillar system with liquid and experimental set up; inset: difference of colour between PDMS and PDMS-Au pillars.

fiber probe receiving the radiated optical signal) it is possible to characterize the single pillar layout using the system reported in Fig. 2(c) simply by placing on the pillar an optical fiber behaving as probe and connected to an optical multichannel analyzer (OMA). A proper layout useful for the detection of liquids is the pillar matrix layout. In particular, the 2×2 square layout configuration is the basic layout used in this work in order to trap and to detect liquid materials following the scheme indicated in Fig. 2(d): part of the EM field will be coupled with the liquid which behaves as a waveguide cladding by changing the intensity of the radiated field and, consecutively, by detecting the variation of the permittivity. The intensity of the optical transmittivity will decrease by increasing the permittivity value of the cladding (liquids to detect). The cylindrical geometry is chosen in order to facilitate the measurements. We resume the topic developed in the proposed work by the following points. In particular, we

- i) explain the physical basic principle of the sensor by means of a properly designed 3D finite-element method (FEM) tool;
- ii) provide the technological aspects;
- iii) experimentally characterize the single PDMS and PDMS-Au pillar in order to highlight the difference of the radiation properties between PDMS and PDMS-Au materials;
- iv) experimentally characterize the sensing of the 2×2 PDMS-Au pillar layout by considering ethanol and oil as liquids;
- v) we experimentally characterize the dynamics of the sensing during the time and by considering ethanol as liquid;
- vi) and, finally, we provide a configuration for robotic implementation including packaging aspects.

II. TECHNOLOGICAL ASPECTS: EXPERIMENTAL CHARACTERIZATION OF PDMS-AU NANOCOMPOSITE MATERIAL AND SENSOR PROTOTYPE

The PDMS elastomer is obtained by mixing a curing agent of 1:10 weight ratio to base monomer. PDMS polymer is known to be a good stabilizing/reducing agent of gold precursor leading to the generation of gold nanoparticles immediately by

the chemical reduction process occurring at room temperature [16]–[19]. The effect of the chemical reduction is to generate a violet color sample: this color indicates the chemical reduction process which happens by mixing for about 10 min the liquid PDMS with the gold precursor prepared in water solution. In our prototype, we establish a very high gold nanoparticles concentration in order to increase the effective refractive index of the PDMS and support the EM coupling. In the specific case, we use a chloroauric acid salt as gold precursor in water solution ($M_w(\text{HAuCl}_4) = 339.785 \text{ g/mol}$; $[\text{HAuCl}_4] = 0.01 \text{ M}$) with a concentration of about 10% in weight. A rigid silicon mask with millimeter holes is immersed in PDMS-Au liquid in order to achieve the pillar layout. After two days at room temperature is enough in order to easily remove the mask and to obtain solid PDMS-Au pillars without heating process. The atomic force microscopy (AFM) images of the cross section of the gold nanocomposite material (GNM) shown in Fig. 3 provide information on the dimensions of the nanofillers inside the PDMS-Au nanocomposite material used for the sensor reported in Fig. 1. The presence of gold, deduced by Figs. 3 and 4, is also proved by Fig. 5 representing the experimental UV-Visible spectra of the GNM sample used for the sensor prototype: pure PDMS does not show any absorbance in the visible region whereas PDMS-Au has an absorbance centered at $\lambda \cong 530 \text{ nm}$ (average effect of gold particles) as also proved in [18]. As observed also in the inset of Fig. 2(d) the presence of gold nanoparticles are indicated by the violet color. The full characterization of the formed gold nanoparticles will be addressed elsewhere.

III. BASIC PRINCIPLES: LIGHT ENHANCEMENT, WAVEGUIDE BEHAVIOR, AND EM FIELD RADIATION SYSTEM

The EM analysis of the pillar type sensor is based on the concept of guided and radiated optical modes. By exciting the sensor, as illustrated in Figs. 1 and 2, the light is guided inside the PDMS-Au slab. The light is propagated in the pillar and around the pillar region, and a part of the EM field will be radiated in the surrounding space. The pillar structure represents an optical waveguide discontinuity: the effect of the discontinuity is to radiate the EM field, and this effect is accentuated if the effective refractive index of the waveguide is high. For this purpose, the gold nanoparticles formed in the PDMS material increase the effective refractive index of the PDMS waveguide and support the EM coupling with a liquid.

The effective refractive index of the PDMS-Au waveguide can be approximately estimated assuming spherical gold nanoparticles in PDMS material. In this case, the exact effective dielectric function ε_{eff} is difficult to model due to the different sizes and geometries of gold micro/nanoparticles, as observed in Figs. 3 and 4. In order to analyze the difference between the optical behavior of the PDMS and PDMS-Au structures we assume, as approximation, spherical gold particles having dielectric function ε_m which varies with the optical working wavelengths [20]. The spherical gold particles embedded in a medium ε_s (dielectric permittivity of the PDMS) define the following effective permittivity [21]:

$$\varepsilon_{eff} = \varepsilon_s \frac{\varepsilon_m(1 + 2\phi) + 2\varepsilon_s(1 - \phi)}{\varepsilon_m(1 - \phi) + \varepsilon_s(2 + \phi)} \quad (1)$$

Fig. 3. Atomic force microscopy (AFM) images made on cross section of the PDMS gold nanocomposite material. (a) Error signal. (b) Topography. (c) 3D topography of the AFM image.

Fig. 4. Atomic Force Microscopy (AFM) images made on a zoomed area of the PDMS gold nanocomposite material illustrating some gold nanoparticles. (a) Topography, (b) 3D error signal, (c) topography and (d) 3D topography of the AFM image.

where ϕ indicates the gold concentration. We calculate in our simulations a real part of the effective dielectric permittivity of $\epsilon_{\text{eff}} = 10$ (with a gold nanoparticle concentration of $\phi = 0.5$ and $\epsilon_s = 2.25$). Fig. 6 shows how the PDMS-Au pillar is suitable for EM interactions with liquid: the used 3D FEM tool [22] highlights that at the working wavelength of $\lambda_0 = 1.3 \mu\text{m}$ the electric field around the PDMS-Au pillar is more

Fig. 5. Experimental UV visible spectra of the GNM sample used for the pressure sensor.

Fig. 6. (a) Top view and (b) cross section view of the electric field distribution around a PDMS pillar. (c) Top view and (d) cross section view of the electric field distribution around a PDMS-Au pillar. For the PDMS-Au pillar ϕ is assumed equals to 0.5.

Fig. 7. PDMS-Au pillar type structures. Electric field distribution in ethanol liquid surrounding: (a) a single pillar and (b) a 2×2 pillar matrix.

intense compared to the case of PDMS. The electric field distributions in ethanol at the same working wavelength for the single PDMS-Au pillar and for the 2×2 matrix PDMS-Au are illustrated in Fig. 7(a) and (b), respectively. The ethanol behaves as a cladding material and, consecutively, a part of the EM energy will be localized inside it.

Being the pillar is similar to a waveguide/probe system, the remaining part of the EM energy will be transferred and propagated, as illustrated in Fig. 2(a).

Fig. 8. 3D FEM electric field radiation pattern of PDMS-Au waveguides. (a) Single pillar layout and corresponding radiation pattern (b) calculated above the waveguide. (c) 2×2 matrix layout configuration and corresponding radiation pattern (d) calculated above the waveguide.

In particular, in the air space above the pillars (in the region closest to the pillars), the radiated electric field $\mathbf{E}(x, y, z)$ (radiation pattern) will be defined as [22]

$$\mathbf{E}(x, y, z) = \int_S ((j\omega\mu_0 \mathbf{H}_{\text{tan}})G + (\mathbf{E}_{\text{tan}} \times \nabla G) + (\mathbf{E}_{\text{normal}} \nabla G)) dS \quad (2)$$

where S represents the radiation boundary spherical surface, j is the imaginary unit, $\mathbf{H}_{\text{tan}}$ is the component of the magnetic field that is tangential to the surface, $\mathbf{E}_{\text{normal}}$ is the component of the electric field that is normal to the surface, and G is the free space Green's function. Fig. 8 illustrates the FEM electric field radiation pattern of a single PDMS-Au pillar and of a 2×2 pillar PDMS-Au matrix. According to these simulation results, we choose the experimental setup configuration of Fig. 2(c) and (d).

IV. SINGLE PILLAR EXPERIMENTAL CHARACTERIZATION AND DISCUSSIONS

In order to highlight the importance of the PDMS-Au material in the detecting process, we first experimentally characterize the single pillar in the optical frequency range under investigation. We use the experimental configuration of Fig. 2(c) where a broad lamp source is connected to a multimode optical fiber exciting the slab waveguide, and an optical multichannel analyzer (OMA) is connected with another multimode fiber placed in contact with the terminal part of the pillar. The experimental spectrum of the broad lamp source is illustrated in Fig. 9. In order to have a true comparison, we fabricate two pillars (PDMS and PDMS-Au pillar) with the same dimensions (1.3 mm of height, 1 mm of diameter, and 0.4 mm of the slab's thickness t indicated in Fig. 6). By using the same working conditions of the experimental setup (aperture of the OMA grating, positions of the input and of the output fiber, etc.), we measure the output of both the pillars

Fig. 9. Optical spectrum of the used broad lamp source. The spectrum is measured by an in-plane experimental set up.

Fig. 10. Comparison between the transmitted light intensity of a single PDMS/PDMS-Au pillar. Inset: Configuration of the experimental radiation setup used during the measurements.

and we observed that the transmitted optical intensity through the PDMS-Au pillar is enhanced due to presence of gold (see Fig. 10). Moreover, in both cases, a deep of the transmitted intensity around 1186 nm is observed. In order to explain this optical loss, we characterize the PDMS and the PDMS-Au materials by means of the FTIR in absorbance modality. As illustrated in Fig. 11, we found two peaks corresponding to the deep of Fig. 10: as also proved in [23] the PDMS material is characterized by an absorption around 1190–1200 nm and PDMS-Au material presents a higher absorbance.

V. MEASUREMENTS OF THE 2×2 PDMS-AU SENSOR AND DISCUSSIONS

According to the FEM simulations, the 2×2 PDMS-Au matrix layout is suitable for the EM confinement near the pillars. For this purpose, we measure the variation of the transmittivity intensity by putting on the sensor prototype two liquids, such as ethanol and oil, which have different refractive indexes. The variation of the transmitted intensity is function of the refractive

Fig. 11. FTIR measurements: comparison between the PDMS and the PDMS-Au absorbance.

Fig. 12. 2×2 PDMS-Au matrix: variation of the transmitted intensity by considering ethanol and oil as liquids to detect. Inset: Experimental setup configuration for the evaluation of the noise (the 2×2 matrix is absent during this measurement).

index permittivity and, as expected, we found in Fig. 12 a lower optical intensity when we used the oil: oil has a higher refractive index compared to ethanol ($n_{\text{oil}}(\lambda = 590 \text{ nm}) = 1.468$ [24], and $n_{\text{ethanol}}(\lambda = 590 \text{ nm}) = 1.3571$ [25]) and more energy will be confined inside the oil cladding.

The detection principle is in the variation of the transmittivity intensity: this variation can be associated to the variation of the dielectric contrast $dn = n_{\text{liquid}} - n_{\text{air}}$ and an intelligent system can correlate the different variations dn to well defined liquid materials. An initial acquisition of the transmittivity intensity reduction due to the use of different liquids is necessary in order to distinguish the relative permittivity. Due to the impermeable property, the proposed sensor prototype of PDMS elastomer can be reused for different times (useful aspect for robotics applications). A characterization of this property is performed in

Fig. 13. Detection of ethanol and transmittivity response after different time steps by 2×2 PDMS-Au sensor. (a) Transmittivity response immediately after the deposition of ethanol. (b) Transmittivity response after 1 min, (c) 5 min, and (d) 10 min.

Fig. 14. FTIR absorbance measurement of the ethanol material.

Fig. 13 by the analysis of transmittivity response during the time by applying ethanol as a reference liquid to detect. Fig. 13 shows the dynamic after different times of the transmittivity response proving the reversibility of the sensing properties and the reusability of the sensor device. As it appears in the same figure, the variations in the optical response are sensitive in the region between 1200 and 1350 nm (sensitivity region).

This result proves that upon ethanol evaporation the signal recovers. The recovering effect in different spectrum regions is explained by means of the absorbance measurement (FTIR spectrum) reported in Fig. 14 representing the absorbance of the ethanol material: the sensitivity region of the transmittivity response corresponds to the region of the absorbance variation. In order to not move the experimental setup, we put the liquid droplets directly on the sensor. We observe that we have measured the oil and the ethanol transmittivity in separate time steps

Fig. 15. (a) Schematic diagram of the sensor implementation in robotic finger, (b) design of the packaging, cross section, (c) top view and (d) perspective of the packaging design, the dark violet indicates the PDMS border.

(no mix up between ethanol and oil are considered). All the measurements were repeatable.

VI. APPLICATION: LIQUID SENSING FOR ROBOT SYSTEMS AND PACKAGING DESIGN

The proposed sensor can be easily implemented in robotic finger systems. By using small laser diode sources and small photo diode detectors (useful for the conversion of optical signals in electric signals) the liquid sensor requires small spaces and assures very fast times of detection response. A possible schematic representation is illustrated in Fig. 15(a), where the sensor is coupled by optical fibers and the variation of the signal can be processed by an intelligent system which provide commands about the movement of the robotic finger by following defined procedures. According to the simulations of Fig. 6(d), an optical fiber can detect the variation of intensity and also if it is enclosed by the PDMS-Au material. For this purpose, it is preferable to consider all the components in an embedded system as represented in details in the packaging design of Fig. 15(b). Fig. 15(c) and (d) show the cross section and top view of the packaging design of Fig. 15(b), respectively. As shown in the design, the liquids will be collected by means of PDMS borders arranged along the boundaries of the sensor (see Fig. 15(d)). A fiber bundle will be coupled to an optical coupler connected to a photodiode. The electric signal outgoing to the photodiode will be processed by an electronic chip which will command the mechanical movements of the finger by following the procedure related to a defined application.

VII. CONCLUSION

In this work, we have presented a new concept and an accurate study of the optical liquid sensing based on PDMS-gold nanocomposite materials. The sensor is designed and then characterized by means of experimental results. The proposed optical sensor can be easily integrated in a robotic systems including tactile and shape sensing. By implementing a database of dielectric contrast variations in an intelligent system, it is

possible to detect different liquid materials. Being the proposed sensor is responsive to low variation of the optical permittivity, it can be also used for verifying the quality of ethanol of automotive fuels. The complete mechanical control system and the signal processing is actually under investigation.

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