

Application of Whispering-Gallery-Mode Optical Microcavities for Detection of Silver Nanoparticles in an Aqueous Medium

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Abstract—The results of an experimental investigation of a sensor intended for detection and measurement of concentration of nanoparticles in an aqueous medium, which is based on optical-dielectric whispering-gallery-mode microcavities, are presented. Variation of the frequency and Q-factor of the eigenmodes of the microcavity upon its interaction with silver nanoparticles is studied. It is demonstrated that this type of sensor can be used for measurement of infinitesimally low concentrations of nanoparticles.

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INTRODUCTION

Optical whispering-gallery-mode dielectric microcavities combine unique characteristics, such as the high Q-factor and frequency stability of these modes, high field concentration in a small volume inside the cavity and near its surface, and the possibility of their implementation in a wide range of wavelengths, along with the possibility of using environmentally stable dielectrics. This allows using such microcavities as effective converters of small changes in the environmental parameters into frequency changes that can be measured with high precision. Such microcavities can play an important role in sensors intended for detection of nanoscale objects, such as nanoparticles and biological substances. Many of existing methods are based on measurement of a particular parameter averaged over a considerable volume containing the objects under investigation. Such methods include, e.g., the method of dynamic light scattering [1]. There are also methods based on optical spectroscopy, including a high-resolution one [2].

Application of optical-dielectric microcavities allows designing a continuously operating sensor that provides real-time information about attachment of individual nanoparticles and allows (under certain circumstances) estimating their size [3]. The high sensitivity of such a sensor is caused by the Q-factor of the cavity, which can exceed 10^8 , and the fact that the optical field is concentrated in a small volume near the microcavity equator. In so doing, part of this field is located outside of the microcavity material. It is through this “falling-out” field that interaction of the microcavity with the studied objects takes place.

Immersing the microcavity into a liquid medium changes its resonance frequencies. However, for a wide class of liquids, including distilled water, alcohols, and acetone, the Q-factor of the cavity decreases only slightly. This allows detecting the objects under investigation (nanoparticles, in our case) when they enter the region of the falling-out field by a shift of the resonance frequency and a change in the Q-factor. Combination of these two parameters contains information on the size of the nanoparticles and complex dielectric permittivity of their material.

EXPERIMENTAL

In the present work, we used spherical fused silica microcavities with a diameter between 300 and 600 μm . The microcavities were fabricated by melting in the flame of an oxygen–hydrogen torch. Such microcavities exhibit a high Q-factor and can be easily fabricated in any quantity over a short period of time.

The experimental layout is presented in Fig. 1. We used tunable external cavity semiconductor laser 1 (Vita Wave ECDL-6710R) delivering 10 mW at 670 nm [4]. The laser radiation was focused by lens 4 on the inner surface of prism 5 that was used as a coupling element. Microcavity 8 was placed into cell 6 filled with a solvent containing nanoparticles. The cell was constructed so that the face of the prism element served as one of the cell walls. Thus, optical radiation could not interact with the nanoparticle solution other than through the microcavity.

Rough positioning of the microcavity was accomplished with the help of a three-axis translation stage

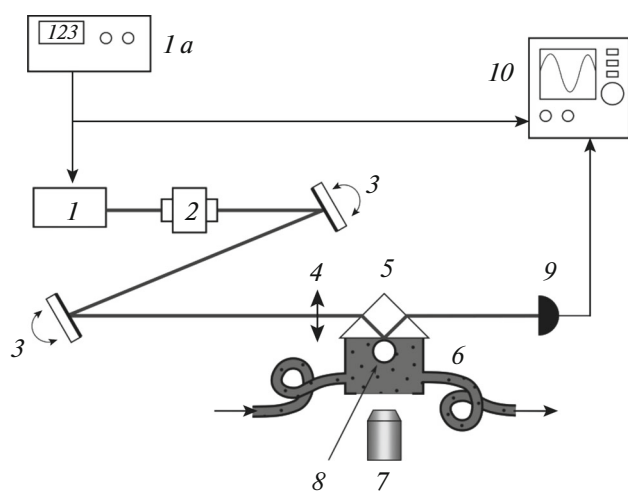


Fig. 1. The layout of the setup for studying sensors based on optical-disk whispering-gallery-mode microcavities: (1) laser, (1a) laser controller, (2) Faraday isolator, (3) adjustable mirrors, (4) focusing lens, (5) prism coupler, (6) cell containing colloidal solution of nanoparticles, (7) microscope, (8) microcavity, (9) photodetector, and (10) digital-storage oscilloscope.

with micrometer drives and was controlled by means of microscope 7. A PZT transducer was used to fine tune the gap between the prism and the microcavity. The position of the focal spot on the prism face was corrected by means of three adjustable mirrors 3. The radiation emerging from the microcavity was detected by photodetector 9. The obtained signal was recorded by a storage oscilloscope 10 and was subsequently processed on a computer with special software. Laser controller 1a tuned the laser frequency during operation. The maximum tuning range was 600 MHz, and the frequency was tuned at a repetition rate of 25 Hz. The control signal of the frequency tuning was also recorded by the oscilloscope. The maximum Q-factor of the microcavities before introduction of nanoparticles into the solution was 3×10^8 . The relative frequency shift could be determined with an accuracy of better than 0.2 MHz, while changes in the width of the resonance curve could be detected with an accuracy of $\cong 5\%$.

RESULTS OF MEASUREMENTS

In the present study, we used a colloidal solution containing silver nanoparticles with a diameter of 61 ± 4 nm. The size of the nanoparticles was determined by the Malvern Zetasizer Nano ZS system based on the dynamic light scattering. The solution was added into the cell containing deionized water until required concentration of nanoparticles in the fluid was obtained. The fluid temperature was maintained constant to within 0.5 K. It was also necessary to prevent formation of air bubbles and ensure homogeneity of the obtained suspension. Since the intrinsic frequency

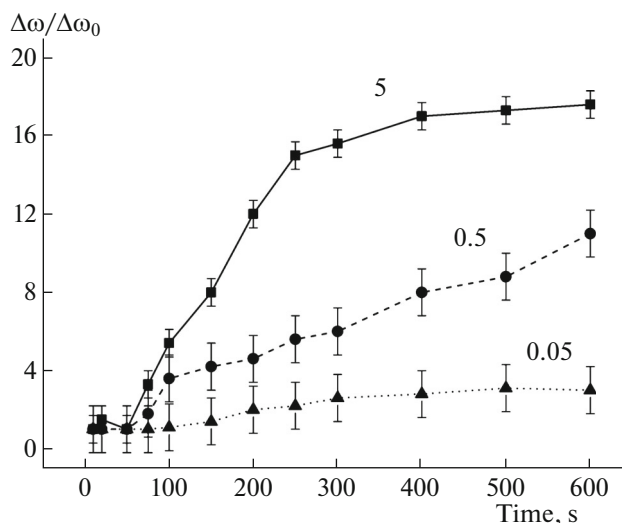


Fig. 2. Variation of width of the resonance peaks for silver nanoparticle concentrations of 0.05, 0.5, and 5 ppm as a function of time elapsed after introduction of nanoparticles into the cell.

instability of the used laser was on the same order of magnitude as the change caused by the deposition of the nanoparticles at low concentrations, we used the change in the microcavity Q-factor caused by particle precipitation as the measurable quantity.

The examples of the obtained dependences of the relative change in the width of the selected resonance peak on time elapsed after introduction of the solution into the cell for three concentrations of the obtained suspension—namely, 5, 0.5, and 0.05 ppm—are presented in Fig. 2. The initial value of the Q-factor was $(1.0 \pm 0.5) \times 10^8$ in all cases. It was found that the quality factor did not experience any degradation in the first 30–50 s. Observation by means of a high resolution camera through a microscope revealed that the nanoparticles in the falling-out-field region (they could be discovered by weak flashes of the scattered radiation) did not stick to the microcavity surface during this time. After that, active formation of a “layer” of nanoparticles on the surface took place, causing degradation of the quality factor. It can be seen from the diagrams in Fig. 2 that the rate of broadening of the resonance peaks depended on concentration of particles in the solution. In so doing, saturation for the studied type of the nanoparticles was observed at a concentration of 5 ppm in about 4–5 min, whereas no saturation could be detected at lower concentrations.

DISCUSSION

Interaction of the “falling-out” optical field with a nanoparticle continuum is affected by several factors, including the field geometry, the shape of the particles, their polarizability, as well as the properties of

materials of the nanoparticles, fluid, and the microcavity. The possibility of formation of clusters of nanoparticles of much larger size should be taken into consideration. Based on these circumstances, for unique interpretation of the obtained dependences, the latter should be calibrated taking into account different sizes and concentrations of the nanoparticles to be detected. We underscore that it is not necessary to clean the microcavity after the measurements. It is much easier and cheaper to use a new microcavity for each cycle of measurements.

Thus, we have demonstrated that the discussed type of the sensor can be used for detecting silver nanoparticles with a concentration of 0.05 ppm and higher in an aqueous medium. After proper calibration, other nanoparticles and media can also be used.

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