Far-field single nanoparticle detection and sizing

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Whispering gallery mode based optical microcavities are important for highly sensitive optical sensing. However, the current experimental realizations are strongly dependent on high-resolution tunable lasers and evanescent coupling, which are too cumbersome and too expensive for portable devices. Herein we experimentally demonstrate a cost-effective and robust approach to detect and size a single nanoparticle with far-field laser emissions. By placing a limacon microdisk close to a spiral microdisk, chiral resonances have been successfully generated. In contrast to previous research, here the internal chirality is strongly correlated with the far-field patterns (FFPs) and thus can be transduced to far-field emissions. Once a nanoparticle is attached to the limacon microdisk, the asymmetrical backscattering at the notch of the spiral can be averaged by the symmetrical scattering of the nanoparticle. Consequently, the internal chirality and the corresponding FFPs are changed. By measuring a far-field directional laser emission, nanoparticles with a radius of ~50 nm have been successfully detected and sized without employing any spectral information. As a narrow-linewidth tunable laser is not used in our experiment and microdisks lasers may be electrically driven, this research will provide a new path to cost-effective, portable, highly sensitive optical sensors.

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1. INTRODUCTION

Label-free and highly sensitive optical detection plays an essential role in important applications ranging from clinical diagnostics and environmental monitoring to single-virus and single-nanoparticle detection [1–4]. Whispering gallery mode (WGM) microcavity-based optical sensors are a prominent example [5]. The light is trapped by total internal reflection along the cavity boundary for a long time and thus ensures ultrahigh quality (Q) factors. The record Q-factors of WGM microcavities such as microspheres, microtoroids, and silicon microdisks are on the order of $10^9$, $10^8$, and $10^6$, respectively, making them suitable for optical detection with ultrahigh sensitivity [6–9]. In the past decade, several sensing mechanisms have been successfully developed to achieve single-nanoparticle detection. In 2002, Vollmer et al. pioneered sensing experiments by measuring the shift of resonant wavelength in a silica microsphere [10]. Soon after, this technique was proposed to detect a single molecule [11], single virus [12], and single-molecule nucleic acid [13]. In 2010, Zhu et al. utilized mode splitting in a silica microtoroid to detect the size information of a nanoparticle [14,15]. Recently, the linewidth broadening [16] and optomechanical effects [17] have also been successfully applied to detect a single nanoparticle. For all of these techniques, ultra-narrow-linewidth ($\Delta \nu \sim 100$ kHz) tunable lasers are required to achieve accurate spectral changes. However, this kind of tunable laser is too cumbersome, too expensive, and too complex to realize portable sensing devices for point-of-care applications and thus strongly hinders practical applications of WGMs sensors.

With the rapid developments in avoided resonance crossing [18,19] and parity time symmetry breaking [20–23], the exceptional points (EPs) in non-Hermitian systems have also been studied for applications in optical sensing. In 2014, Wiersig pointed out that nanoparticle-induced mode splitting is proportional to the square root of the perturbation strength [24]. While this finding can significantly enhance the detecting limit by several times, it still requires high spectral resolution and is hard to apply practically. Recently, another important phenomenon around EPs, optical chirality, has begun to attract considerable research attention [25–30]. In optical microcavities, asymmetrical backscattering at the boundary breaks the chiral symmetry and thus induces optical chirality [25,26]. In addition to the conventional spectral information, optical chirality also corresponds to a quasi-degenerate pair of copropagating wave modes, which can provide additional measures for detecting the internal chirality [31,32]. To date,
optical chirality has been intensively studied in a number of microcavities such as a spiral cavity [25], circular cavity with multi-scatters [27], and deformed limacon cavity [28]. In 2015, Sarma et al. even theoretically proposed detecting the rotation with optical chirality [31]. However, most of the previous research has been restricted within theoretical studies. Experimental realizations, especially practical applications of optical chirality, are still challenging and rare [33,34]. In this research, we experimentally demonstrate a simple and robust mechanism to form chiral resonance in microdisk lasers. We have confirmed the close relationship between the internal chirality and the external far-field patterns (FFPs), and utilized far-field laser emissions to detect and size a single nanoparticle.

2. RESULTS AND DISCUSSION

In order to detect a nanoparticle with optical chirality and relieve the strong dependence on extremely narrow linewidth tunable lasers, three key steps will be accomplished: (I) It is necessary to replace the passive microcavities with active microdisks, (II) the active device must have optical chirality that can respond to the attachment of a single nanoparticle, and (III) it is important to build a connection between the internal chirality and the changes in far-field laser emissions beyond the changes in resonant frequencies. In this research, we realize the above goals with the recently developed heteronuclear diatomic photonic molecule laser [29,32]. Basically, a spiral microdisk and a limacon microdisk are placed in proximity. The boundary shapes of the limacon cavity and spiral cavity are defined by the equations $\rho(\theta) = R_1 (1 + \epsilon_1 \cos(\theta + \pi))$ [35] and $\rho(\theta) = R_1 (0.82 + \epsilon_2 \sin\theta)$ [35], respectively, in polar coordinates, where $R_{1,2}$, $\theta$, and $\epsilon_{1,2}$ are the radius, polar angle, and deformation parameters, respectively. In the photonic molecule, the limacon microdisk contributes relatively high Q-factors and directional laser emissions, whereas the spiral microdisk provides asymmetrical scattering at the notch. Consequently, the combination of two microdisks makes it possible to generate relatively long-lived chiral resonances with directional outputs. Compared with conventional studies on chirality in microcavities, the configuration of photonic molecules has two intrinsic advantages: it is much simpler than the sophisticated design of the cavity boundary [25–28,30,31], and the far-field laser emissions from limacon microdisks can transduce the internal properties to the far field and thus relieve the conventional dependence on conventional evanescent coupling between the microdisk and tapered fiber [32].

A. Chiral Resonances in Microdisk Lasers

To verify the above analysis, we have fabricated dye-doped polymer (SU-8, MicroChem; $n \sim 1.56$) microdisks on silicon wafers using electron-beam lithography and two-step inductively coupled plasma etching (see Part I of Supplement 1). Figure 1(a) shows the top-view scanning electron microscope (SEM) image of one fabricated sample. The limacon microdisk is well fitted to the limacon equation with $R_1 = 5 \mu m$ and $\epsilon_1 = 0.4$, whereas the spiral microdisk can be fitted with $R_2 = 4 \mu m$ and $\epsilon_2 = 0.18$. The boundary-to-boundary separation distance between the two microdisks is 300 nm. Then the microdisks are optically excited under an optical microscope with a rotational translation stage (SUSS A1; see Part I of Supplement 1) and the corresponding emission spectra are recorded. When the pumping power is low, the emission spectrum is a broad spontaneous emission peak.

With increasing pumping power, transverse magnetic (TM) (with $E$ perpendicular to the plane; see the experimental results in Fig. S7 of Supplement 1) polarized periodic peaks [see Fig. 1(b)] emerge and quickly dominate the emission spectrum. The dependence of the integrated output intensity on the pumping power has been summarized in Fig. 1(c). A dramatic increase in the power slope at $\sim 0.94 \mu J$ has also been observed, which is related to the transition from spontaneous emission to narrow peaks. Consequently, the lasing actions inside the photonic molecule can be confirmed. Figure 1(d) shows the high-resolution emission spectrum around the threshold. The narrowest FWHM is 0.04 nm, which is limited by the resolution of our spectrometer. Thus the $Q$-factor of our microdisk will be larger than 15,750.

In additional to the laser spectra and threshold, the corresponding FFP of the limacon–spiral photonic molecule has also been experimentally studied by rotating the translation stage. During the experiment, the pumping power is fixed at 3.125 $\mu J$ and the laser spectra are recorded at every 10 deg. (see Fig. S8 of Supplement 1). Figure 2(a) shows the angular distribution of the integrated emission intensity at the far field. We can see that the main emission is predominantly in one direction along $\phi_{FF} = 150^\circ$. The other emissions, especially the laser emissions along the direction $\phi_{FF} = -150^\circ$, are much smaller. Similarly, the far-field angular distributions of individual lasing modes have also been studied. Figures 2(b)–2(d) show the angular distributions of the modes marked as 1, 2, and 3 in Fig. 1(b). We can see that the phenomenon is quite generic in all modes. Each mode has a dominant laser emission along $\phi_{FF} = 150^\circ$, and the emission along $\phi_{FF} = -150^\circ$ is negligibly small. The above experimental results contradict the intuitive picture of a conventional limacon cavity [35]. For an independent limacon cavity, the presence of mirror-reflection symmetry ensures the balance between the clockwise (CW) propagating waves and counterclockwise (CCW) propagating waves. Considering the leakage along the unstable manifolds and the low-refractive-index ($n \sim 1.56$) limacon microdisk, the CW waves generate a directional output beam at $\phi_{FF} = 150^\circ$ and the CCW waves will produce a mirror-reflection symmetric laser beam at...
ϕFF = −150° (see the numerical and experimental verifications in Figs. S2–S5 of Supplement 1).

To accurately understand the discrepancy between the directional emissions from the limacon–spiral photonic molecule and those from a single limacon, we have imported all the structural information from the SEM image in Fig. 1(a) and numerically calculated the resonances of photonic molecules with finite-element-method-based software (see Part I of Supplement 1). As the pumping density is only a little bit higher than the threshold and the gain material is homogeneous, effects such as "line pulling," self-saturation, mode competition, and parity-time symmetry have not been formed, and thus the lasing modes can be understood with the resonances within passive cavity [36–39]. The calculated results are shown in Fig. 3(a). A series of TM-polarized resonances marked 1–5 have relatively high Q-factors around $10^5 - 2 \times 10^5$. From the corresponding field distribution, these modes are well confined within the limacon cavity [see the example in Fig. 3(b)]. We notice that the Q-factors in limacon–spiral photonic molecules are several times smaller than those in a single limacon microcavity of the same size (see Fig. S2 of Supplement 1), clearly demonstrating the influence of the scattering at the notch of the spiral on these high-Q resonances. As the scattering at the notch is asymmetrical, the balance between CW and CCW waves will be broken, and thus optical chirality is generated.

As the chirality is related to the broken symmetry between CW and CCW waves, it can be straightforwardly confirmed by projecting the wave functions on the boundary of the limacon cavity [40, 41]. The Husimi map of mode 2 in Fig. 3(a) is shown in Fig. 3(c). The waves are localized along a six-bounce unstable periodic orbit far above the critical line, which thus ensures the relatively high Q-factors. In contrast to a conventional limacon cavity [see Fig. S2(c) of Supplement 1], here we can see that the Husimi map is dominated by the CCW waves with $\sin \chi > 0$, and the CW waves with $\sin \chi < 0$ are almost one order of magnitude smaller. Consequently, we can conclude that the chiral symmetry has been broken by placing a spiral cavity close to a high-Q resonator. To quantitatively characterize the chirality, we have defined the chirality as $\alpha_1 = 1 - \frac{\sum_{CCW} \text{Husimi}}{\sum_{CW} \text{Husimi}}$. The chirality of the relatively high-Q modes in Fig. 3(a) have been calculated and shown as green dots in Fig. 4. We can see that all the resonances have high chirality (>0.4) and are dominated by the CCW components.

Compared with the circular cavities, the limacon cavity with $n = 1.56$ has two directional emissions along $\phi_{FF} = \pm 150°$. As mentioned above, these two output beams are caused by CCW waves and CW waves, respectively [see details in Figs. S2 and S5 of Supplement 1]. Therefore, the internal chirality can be transduced to the far field via laser emissions and easily measured without evanescent coupling. Related to the chirality in Husimi map, we have also observed the asymmetrical FFP in Fig. 3(d), where the emissions along $\phi_{FF} = \pm 150°$ are quite different. To confirm the close relationship between the internal chirality and external FFP, we have defined another measure of chirality, $\alpha_2 = 1 - \frac{U_1}{U_2}$, and compared it with the calculations from the Husimi map.

Fig. 2. FFPs of the lasers in the limacon–spiral photonic molecule. (a) The far-field angular distribution of the total emission, (b)–(d) the FFPs of modes 1–3 in Fig. 1(b).

Fig. 3. Resonant modes of the limacon–spiral photonic molecule. (a) The calculated Q-factors in the photonic molecule, (b) the field pattern $|E_z|$ high-Q mode in the photonic molecules. The white line is the cavity boundary. (c), (d) The Husimi map and the corresponding far-field pattern of mode 2 in (a).

Fig. 4. Chirality of high-Q resonances in the limacon–spiral photonic molecule. The dots and open squares show the chirality obtained from the Husimi map and the far-field patterns, respectively. The stars are the experimental results in Fig. 2.
where $U_1$ and $U_2$ are the integrated intensities of emission beams along $\phi_{FF} = -150^\circ$ and $\phi_{FF} = +150^\circ$, respectively. As shown in Fig. 4, we find that $\alpha_1$ and $\alpha_2$ perfectly match one another, indicating that the far-field laser emissions can be an effective way to record the internal chirality.

In addition to the asymmetrical FFPs, the internal chirality within the limacon microdisk has also been experimentally studied by applying evanescent coupling. As the top-view SEM image in Fig. 5(a) shows, we have fabricated a bus waveguide with grating couplers near the limacon microdisk. The width of the waveguide and the separation distance between the waveguide and microdisk are both 300 nm to efficiently couple the laser emissions. When the limacon–spiral photonic molecule is pumped above threshold, the emission at the left grating coupler is much higher than the right one [see the fluorescent microscope image in Fig. 5(b)]. Consequently, it is easy for us to see that the internal resonances are dominated by the CCW waves and the chirality has been formed. As a control experiment, we have also studied the chirality in a single limacon cavity. As shown in Figs. 5(c) and 5(d), the outputs at two ends of the bus waveguide are well balanced. Therefore, we can conclude that the limacon–spiral photonic molecule can simply form internal chirality and the chirality can be easily detected with the FFPs. Interestingly, the formation of chirality is not limited in two coupled microdisks. By inserting one or two circular microdisks between the limacon and spiral microdisks, optical chirality within a limacon disk has also been experimentally observed (see Figs. S11 and S12 of Supplement 1).

**B. Detecting and Sizing a Single Nanoparticle**

Because the internal chirality can be directly observed at the far field, limacon–spiral photonic molecules have potential in applications as portable optical sensors. Here we demonstrate one application in single-nanoparticle detection. The scattering at the notch of the spiral is strong but only affects the evanescent waves of resonances in the limacon cavity. In this sense, if a nanoparticle is attached to the boundary of the limacon cavity, its direct influence on the resonances may be comparable to the influence of a spiral cavity, and thus the asymmetrical scattering will be reduced. Consequently, both the internal chirality and external FFPs will be different. Based on this analysis, we have numerically studied the response of the FFPs of a limacon–spiral photonic molecule to the size of the nanoparticles. Figure 6(a) shows a schematic picture of one nanoparticle on the boundary of limacon microdisk. The nanoparticle has radius $r$ and is attached to the limacon at a polar angle $\theta$. For the resonant mode 2 in Fig. 3(a), the FFP pattern is dominated by the emission along $\phi_{FF} = 150^\circ$ without attaching a nanoparticle (or with $r = 0$ nm). With the increase of the radius $r$, the scattering of the nanoparticle also increases. Thus the asymmetrical backscattering caused by the notch will be more and more averaged by the nanoparticle and the emission beam marked as $U_1$ in the FFP will gradually increase [see Fig. 6(b)]. Beyond simple detection, as the scattering of the nanoparticle is proportional to the particle polarizability $p = 4 \pi r^3 \varepsilon_0 \varepsilon_r \Delta \varepsilon$ [14], where $\varepsilon_{r,\infty}$ are the dielectric permittivities of the nanoparticle and the surrounding medium, the changes in the FFPs can also be used to size the nanoparticles. Figure 6(b) shows parts of the numerically calculated far-field angular distributions with different $r$ values. When the radius of the nanoparticle increases from 0 to 30 nm, 50 nm, and 120 nm, we can see that the emission along $\phi_{FF} = -150^\circ$ is increased from $\sim 20\%$ to $30\%$, $37.5\%$, and $50\%$ of the emission along $\phi_{FF} = 150^\circ$, respectively. Figure 6(c) summarizes the chirality $\alpha_2$ (the changes in FFPs) as a function of radius $r$, where the chirality decreases almost linearly in a wide range of nanoparticles. Consequently, in addition to the conventional changes in resonant frequencies, the changes in chirality and the FFPs can be applied to detect and even size a single nanoparticle. In the experiment, the nanoparticle might not be attached to the same position on the cavity. We thus have examined the chirality with a nanoparticle at different positions. Unlike the circular microdisk, where the resonances have a fixed azimuthal number, the resonances and scattered waves in chaotic cavities have a higher number of angular
momentum (azimuthal number). Since different orbits can give different phase differences between the single nanoparticle and the notch, the interference effect has been averaged out and the dependence of chirality on the position of the nanoparticle is diminished. Figure 6(d) clearly demonstrates this effect, where the optical chirality is almost independent of the position of the nanoparticle.

Based on this information, we have experimentally fabricated microdisks and studied their potential in nanoparticle detection. Figure 7(a) shows the top-view SEM image of one limacon–spiral photonic molecule. All of the structural information is the same as in Fig. 1(a). When the sample is optically pumped, the corresponding lasing properties are characterized, and they have been summarized in Fig. 7(c). With the increase in pumping power, a transition from a broad photoluminescence peak to narrow peaks and a dramatic increase in power slope have been experimentally observed, confirming the lasing actions very well. Meanwhile, a strong directional emission along $\phi_{FF} = 150^\circ$ has been observed and the emission along $\phi_{FF} = -150^\circ$ is negligible [see the dashed line in Fig. 7(d)]. The calculated chirality of the total FFP is $\alpha_2 \approx 0.8$.

Once a nanoparticle is attached to the boundary of the lima- con microdisk, the increases in the local refractive index and the scattering will change the resonant frequencies, the field distributions, and the far-field laser emissions. In this experiment, the nanoparticle is much smaller than the lasing wavelength. Thus its influence on the frequency shift and mode splitting is relatively small. Without using a narrow-linewidth tunable laser, we haven’t observed any obvious changes in the laser spectra due to a low-resolution spectrometer [see Fig. 7(c); the resolution is $\sim 0.04$ nm]. In contrast to the spectral changes, the changes in chirality should be more drastic. As shown by the solid line in Fig. 7(d), the emission beam along $\phi_{FF} = -150^\circ$ is much stronger than the one without the nanoparticle. The corresponding chirality $\alpha_2$ is only 0.65, which is about 19% smaller than the initial value. In this sense, the far-field laser emission can be more effective in estimating the size of the nanoparticle. By fitting the experimental results to the numerical results in Fig. 6(c), the radius of the nanoparticle should be around 50 nm. This value

is very close to the experimentally measured size in the high-resolution SEM image [see Fig. 7(b)].

We note that the changes in internal chirality and the corresponding FFPs of the laser emission are quite robust and generic in microdisks when a single nanoparticle has been attached. In addition to the results in Fig. 7, we have fabricated another four samples and used the same processes to detect single nanoparticles with different sizes. After attaching a single nanoparticle to the limacon microdisk, we have observed an increase in $U_1$ in the FFPs, and thus the chirality $\alpha_2$ is reduced (the detail experimental results and corresponding numerical simulations are summarized in Part VI of Supplement 1). After measuring the size information of the nanoparticle with high-resolution SEM images, the experimental results have been plotted as crosses in Fig. 6(c) to directly compare with the numerical simulation (the squares). We can see that the experimentally recorded $\alpha_2$ also decreases linearly with the increase in nanoparticle size and all of the experimental results match the numerical simulations very well. This means that the radius of a nanoparticle between 10 nm and 110 nm can be simply detected and sized. We note that a smaller nanoparticle can also be detected if the microcavity has been replaced by a higher Q resonator such as a microtoroid [32]. Therefore, based on systematic studies in both of numerical calculations and experiments, we conclude that the chirality or the FFP of chiral resonances can be an effective way to detect and size a single nanoparticle.

3. CONCLUSION

In summary, we have experimentally studied chirality and its potential applications in optical microdisks. We have successfully generated chiral resonances by utilizing a photonic molecule configuration consisting of a deformed microdisk and a spiral microdisk. Meanwhile, the close relationship between the internal chirality and the external far-field directional laser emission has been experimentally confirmed, meaning the chirality can be simply detected at the far field. Based on these findings, we have developed a simple way to detect and size a single nanoparticle with optical chirality. By measuring the far-field directional laser emission, nanoparticles with a radius of $\sim 50$ nm have been successfully detected and sized without employing any spectral information. Compared with the previous reports on single-nanoparticle detection, this research can directly record the angular distribution of the laser intensity at the far field and doesn’t require an extremely high spectral resolution. This new mechanism can effectively relieve the dependence of single-nanoparticle detection on a narrow-linewidth tunable laser and evanescent coupling. Consequently, the stability and costs of a single-nanoparticle detecting system can be significantly improved. This research will pave a new path to highly sensitive optical sensing and expand the potential applications of optical chirality in microcavities.

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See Supplement 1 for supporting content.

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