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Optical polarization response at gold nanosheet edges probed by scanning near-field optical microscopy*

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Optical properties of metallic edge-like structures known as knife-edges are a topic of interest and possess potential applications in enhanced Raman scattering, optical trapping, etc. In this work, we investigate the near-field optical polarization response at the edge of a triangular gold nanosheet, which is synthesized by a wet chemical method. A homemade scanning near-field optical microscope (SNOM) in collection mode is adopted, which is able to accurately locate its probe at the edge during experiments. An uncoated straight fiber probe is used in the SNOM, because it still preserves the property of light polarization though it has the depolarization to some extent. By comparing near-field intensities at the edge and glass substrate, detected in different polarization directions of incident light, the edge-induced depolarization is found, which is supported by the finite differential time domain (FDTD) simulated results. The depolarized phenomenon in the near-field is similar to that in the far-field.

Keywords: nanosheet, scanning near-field optical microscopy, edge effect, depolarization

PACS: 78.67.–n, 68.37.Uv, 42.25.Gy, 42.25.Ja **DOI:** 10.1088/1674-1056/27/8/087801

1. Introduction

Optical properties of metallic edge-like structures known as knife-edges have been a topic of interest studied for a long time.[1–4] However, most of the experimental and theoretical researches have been limited to a far-field region, which means that it is much larger than one wavelength of incident light away from the edge. The near-field optical properties of the edge have rarely been investigated due to the limitations of experimental methods and conditions.

With the rapid development of nanotechnology, various nanocharacterization methods and shape-controlled nanomaterials are emerging constantly, allowing us to investigate the near-field optical properties of the materials.[5–7] A scanning near-field optical microscope (SNOM), able to directly probe the optical intensity distribution with high resolution,[8] can be used to study the near-field optical properties of the edge. In parallel, the advanced synthesis methods provide various metal nanomaterials with shape-controlled structures, such as nanoparticles, nanowires, nanosheets, etc. Metallic nanosheets, as a special kind of two-dimensional nanostructure with a lateral size of several microns and a thickness of less than 100 nm, provides new platforms for both fundamental research and technological applications.[9] For instance, the sharp edge of a metallic nanosheet has functioned as a substrate to enhance Raman scattering,[10] and the metal thin-film edge has been applied to optical trapping.[11] Therefore, it is of great significance to study near-field optical properties at the edge, which has rarely been studied to date.

In this work, we focus on probing the near-field polarization response at the edge of a triangular gold nanosheet, which is synthesized by a wet chemical method and then deposited on a glass substrate. All experiments are performed using the combined setup of an inverted optical microscope and homemade SNOM.[12–14] In order to remove the effect of an SNOM probe on the optical property of the edge, the polarization characteristic of the probe is measured. The near-field intensities at the edge and glass substrate each as a function of the polarization direction of incident light are respectively detected for evaluating the polarization response of the edge. Moreover, near-field distributions for the polarizations parallel and perpendicular to the edge are theoretically simulated based on the finite differential time domain (FDTD) method.[15,16] The near-field depolarization phenomenon induced by the edge is found and compared with far-field results.

2. Material synthesis and experimental setup

The sample was synthesized by the chloroauric acid reduction method.[17] In a typical process, a 6-ml EG solution was preheated at 150 °C for 20 min, then 1-ml $HAuCl_4$ solution (0.2 M) was added to the EG solution and stirred slowly at 150 °C. Meanwhile, 600-mg poly(vinylpyrrolidone) (PVP, $Mw = 4 \times 10^4$) was dissolved into the 3-ml EG solution. Next, the PVP-EG solution was added to the prepared $HAuCl_4$-EG solution drop by drop at a rate of less than 0.5 ml/min. After

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the dropping process was finished, the mixture was stirred continuously at 150 °C for 60 min. When the color of the reaction solution started to shine, the gold nanosheets were produced. Finally, the reaction products were added into acetone and centrifuged at 2000 rpm for 15 min. The subsidence was rinsed by ethanol several times to remove the capping agent and other contaminants. Final products were obtained and dispersed in ethanol for use in the following experiments.

The experimental setup was built based on the combination of an inverted optical microscope and homemade SNOM operating in the transmission-collection mode as shown in Fig. 1(a). The sample, dripped onto a clean glass substrate and left to dry in air, was placed on a three-dimensional (3D) piezoelectric scanning stage with a resolution of 1 nm in the x/y direction and 0.5 nm in the z direction (P-517.3CL, PI). The incident light from a xenon lamp passed through a polarizer and then was focused onto the nanosheet via an objective lens (50×, NA 0.45, Olympus). The near-field optical signal was collected by a tapered optical fiber probe with a tip diameter of ∼ 300 nm, which was fabricated by etching a single mode fiber in hydrofluoric acid covered with a protection layer but without coating as shown in Fig. 1(c). The uncoated probes have the advantage of high collection efficiency compared with metallic coated aperture probes. The probe was attached to a piezoelectric bimorph, which was used as a shear force sensor to control the probe-sample distance. In order to position the probe at the edge of the nanosheet during near-field detection, a laser light was coupled into the distal end of the fiber and the light spot emitted from the probe was observed by the CCD camera. The light spot was used to indicate the position of the probe. When the probe was moved over the edge of the nanosheet and half of the light spot was blocked, the probe was believed to be located at the edge as shown in Fig. 1(b).

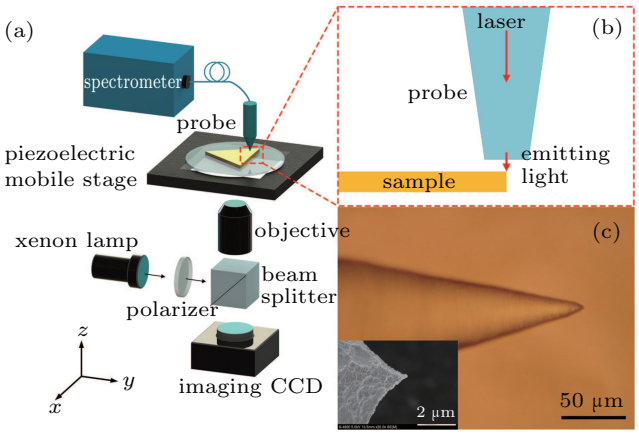


Fig. 1. (color online) (a) Schematic of experimental setup for near-field optical detection, in which the gold triangle nanosheet is not to scale. (b) Enlarged configuration showing probe position during detection. (c) Optical image of a representative etched tapered fiber probe without coating (the scale bar is 50 μm), and inset shows SEM image.

The near-field optical signal collected by the probe was detected by a spectrometer equipped with a linear array CCD. During the measurement, the initial orientation of the polarizer is set to be almost parallel to the bottom edge of the nanosheet. The optical signals S_{edge} and S_{glass} from the edge of the nanosheet and glass substrate were respectively collected in the same polarization of the incident light, in order to show the optical effect of the edge. The background signal S_{back} in the dark case was also measured. Thus, the light intensity $I(\theta)$ collected by the probe in different polarization directions can be calculated from the following equation:

$$I(\theta) = \frac{1}{700-600}\int_{600}^{700}(S_{\text{i}} - S_{\text{back}})\cdot \mathrm{d}\lambda, \tag{1}$$

where S_{i} refers to S_{edge} and S_{glass}. The bandwidth from 600 to 700 nm was chosen because gold material has the least damping for this band in the visible region.[18] In order to suppress the random noise, each signal was acquired 3 times at each position and the exposure time was set to be 5 s.

3. Results and discussion

The topography of the triangle nanosheets is measured by scanning electron microscopy (SEM). Typical images are given in Fig. 2, showing that the edges are very straight with a length of 10 μm. Atomic force microscopy (AFM) image in Fig. 3 shows that the average height of the edge is 90 nm. These results reveal that the sharp edge of the gold nanosheet is suitable for serving as a knife-edge in this study.

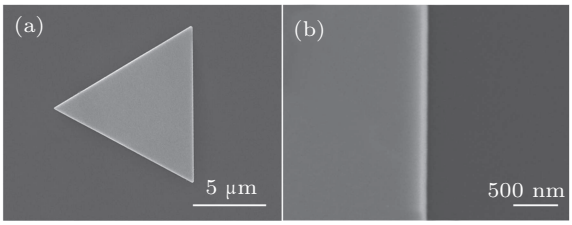


Fig. 2. (a) SEM image of single gold triangle nanosheet. (b) SEM image of edge of gold triangle nanosheet.

Since polarization characteristics of the fiber probe play an important role in the SNOM measurement, a number of experimental and theoretical researches have been performed to study the orientations of electromagnetic field components in the near-field of the probe.[19–24] Unlike the radiating fields in homogeneous media that can be described in terms of transverse wave, the near-fields around sub-wavelength structures are completely vectorial, and the concept of polarization was reported to be unsuitable in the near-field region.[20–24] However, due to the fact that the predominate orientation of transverse field is preserved over the transmission through the straight fiber probe, the polarization contrast has been widely used for SNOM in the collection,[25–27] illumination,[28–30] and reflection modes,[31–33] as well as in a dual-probe SNOM system.[34] The polarization of the probe used in our experiment was measured by the same setup shown in Fig. 1(a) in the following ways: the probe fixed on the bimorph is moved at the optical axis of the microscope and the focal point of the

objective, and then the light signal collected from the probe is measured by the CCD when rotating the direction θ of the linear polarizer in steps of 30°. A representative result is shown in Fig. 4(a), where the light collected by the straight fiber probe shows a two-lobe pattern and the intensity of a distinct polarization direction θ has a maximum value. The collected light can be described by[34]

$$I(\theta) = I_{\max}\cos^2(\theta-\theta_0) + I_{\min}\sin^2(\theta-\theta_0), \qquad (2)$$

where $I_{\max}$ and $I_{\min}$ represent the maximum and minimum intensity of collected light, respectively. The parameters $I_{\max}$, $I_{\min}$, and θ_0 can be obtained by fitting the measured data to expression (2). It should be mentioned that when the probe is mounted to the bimorph prior to the experiment, θ_0 is fixed due to the technical limitation of the setup. In order to quantitatively assess the polarization property, the degree of polarization (DOP) can be defined as[35]

$$\rho = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}}. \qquad (3)$$

The value $\rho = 1$ or 0 means perfect polarization or depolarization, respectively. According to Eq. (3), the DOP of the fiber probe is 0.37, implying that the orientation of the linear polarization is preserved though the depolarization still exists to some extent.

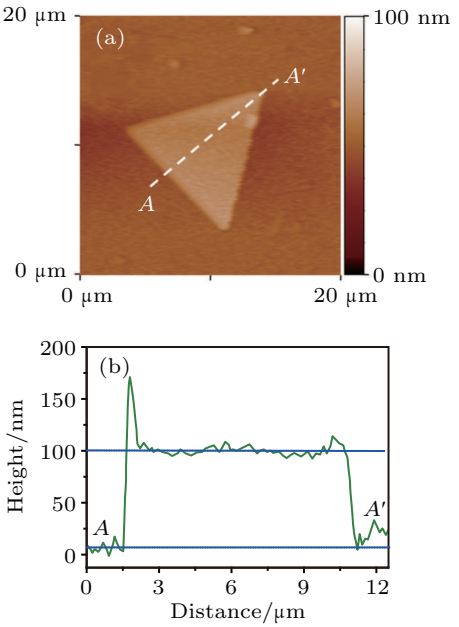


Fig. 3. (color online) (a) AFM topographic image of a triangular gold nanosheet with a size of ∼ 10 μm. (b) Cross-section profile extracted from the line AA' marked in panel (a), showing the height of the nanosheet.

Using the same probe, the near-field intensity is measured at the positions of the gold edge and glass substrate marked by the circle and the star in Fig. 4(b), respectively. The distance between two positions is larger than 5 μm to avoid the edge influencing the optical signal collected on the glass substrate. Typical results in Figs. 4(c) and 4(d) show significant polarization difference between the edge and glass substrate. When the polarization directions of the incident light are at 150° and 330°, the near-field intensity reaches a maximum value, while the directions are at 60° and 240°, the intensity reaches a minimum. The intensity varies between the maximum and minimum in other polarization directions. According to Eq. (3), the DOP of the glass substrate is 0.37, which is similar to that of the probe. This result implies that the glass substrate is unable to change in the polarization of the near-field light. In contrast with the glass substrate, depolarization occurs at the edge because the DOP of the edge decreases to 0.25. The phenomenon could be explained based on the near-field superposition of the transmitted light near the edge with the scattered light by the edge.[36] It is believed that the transmitted light keeps the original polarization state of the incident light, while the scattered light may change the polarization direction of the incident light, consequently leading to the occurrence of the depolarization. To further compare the differences between the edge and glass, the light intensities at the edge and glass are plotted in the Cartesian coordinate as shown in Fig. 4(e), respectively. It is clearly seen that the average intensity of light collected at the edge is much smaller than that at the glass due to reflection and absorption of light by the metallic edge.

In order to further understand the phenomenon, the near-field distribution of light intensity near the edge is simulated. This work is done by the full-field FDTD calculation method. As shown in Fig. 5(a), the model used here is a gold triangular nanosheet with an edge length of 5 μm and thickness of 90 nm. Relative material parameters come from the data of Johnson and Christy.[37] The incident light was set to be a linearly polarized plane wave with the wavelength in a range from 600 nm to 700 nm. A near-field monitor of 300 nm×300 nm is placed at the edge position that is indicated by a yellow rectangle in Fig. 5(a). The distance between the near-field monitor and the upper surface of the gold triangle is set to be 5 nm. These parameters for simulation are similar to those for the experimental investigation.

Figure 5(b) shows the simulation result of the normalized light intensity near the edge, as a function of the polarization direction in polar coordination. Figures 5(c)–5(f) exhibit the simulation results of the light intensity distributions in the yz-plane for polarized angle θ at 0°, 30°, 60°, and 90°, respectively. It is quite clear that the near-field distributions near the edge are significantly different for different polarization directions. Especially for the polarization perpendicular to the edge ($\theta = 90°$), the near-field is strongly localized at the edge of the nanosheet, and the intensity at the edge is higher than that for the polarization parallel to the edge ($\theta = 0°$). These phenomena could involve the excitation of surface plasmon polaritons (SPPs).

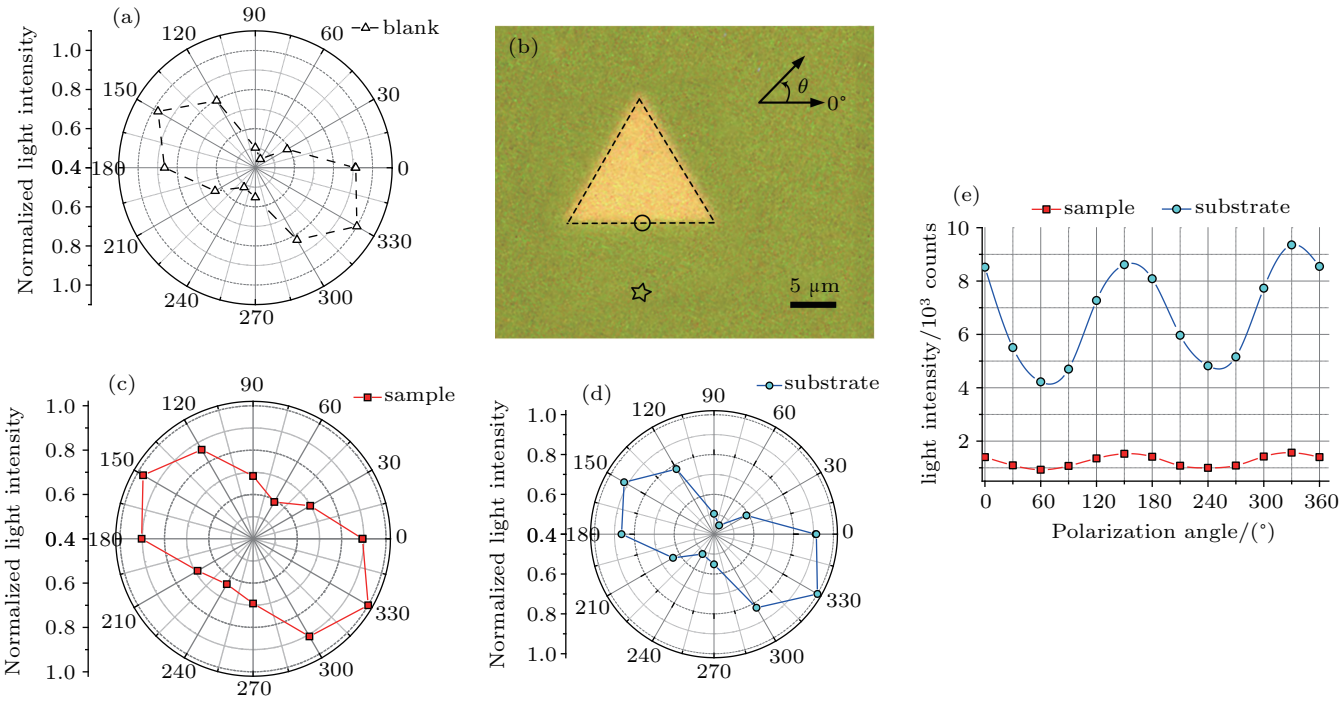


Fig. 4. (color online) (a) Optical intensity of the tapered fiber probe varying with polarization direction of the incident light. (b) Optical image of typical gold triangle nanosheet on glass substrate. Circle and star indicate the detection positions at the edge and the glass substrate, respectively. Angle in the figure represents the polarization direction of incident light. Scale bar is 5 μm long. [(c) and (d)] Normalized light intensities collected at gold edge and glass substrate, each as a function of the polarization direction in polar coordination. (e) Curves of the light intensity at the edge and the glass, each as a function of polarization angle in Cartesian coordination. The polarization angles in panels (a) and (c)–(e) are given in panel (b).

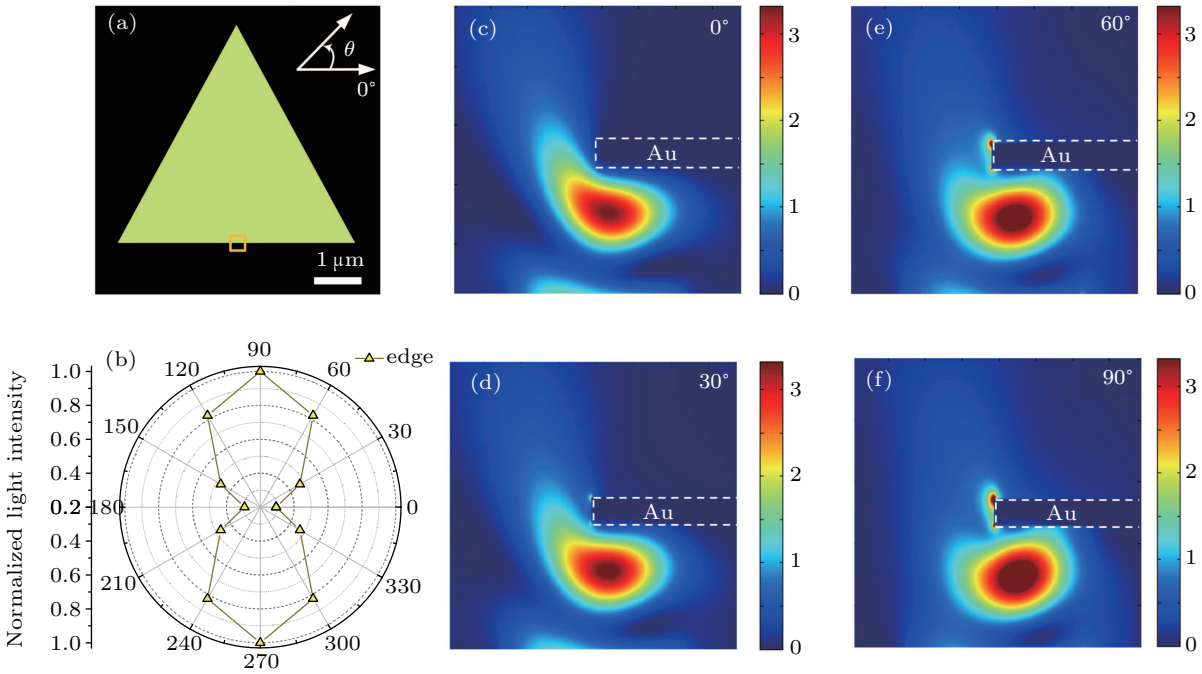


Fig. 5. (color online) FDTD simulated results at the edge of gold triangle nanosheet. (a) Model for FDTD simulation with monitor area 300 nm×300 nm. (b) Normalized light intensity collected at the gold edge, as a function of polarization direction in polar coordination. [(c)–(f)] The *yz*-plane distributions of the light intensity at edge for polarization angle at 0°, 30°, 60°, and 90°, respectively.

As is well known, photons can be coupled to SPPs through the edge of the gold nanosheet. When the polarization direction of the incident light is perpendicular to the edge, the coupling efficiency is highest and more photons will be converted into SPPs that are confined near the edge. As the probe tip approaches to the edge, SPPs should be converted into photons based on the reciprocity theorem. The photons are collected by the probe and detected by the CCD detector to obtain the maximum intensity of light. When the polarization direction of the incident light is parallel to the edge ($\theta = 0°$), no SPPs are excited, resulting in the minimum intensity. At other direction angles, SPPs are only excited by the vertical

component of the polarized incident light, leading to the light intensity between the maximum and minimum. Therefore, the polarization properties of the edge are related to the excitation of SPPs.

There is no doubt that overall polarization characteristics detected by the CCD should depend on the polarization characteristics of both sample and probe. Since the glass substrate is insensitive to polarization, the overall polarization characteristic is similar to that of the probe. The nanosheet edge, however, can greatly affect the overall polarization characteristic due to its high polarization sensitivity. As seen from the experimental results in Fig. 4, the measured near-field intensities reach the maximum values at 150° and 330°, which correspond to weak excitation of SPPs due to the smaller vertical component of the polarized incident light, while the near-field intensities are the minimum values at 60° and 240°, corresponding to strong SPPs excitation induced by the larger vertical component. Consequently, depolarization occurs at the edge of the nanosheet.

It was reported that the far-field intensity distribution of diffraction by a semi-infinite metallic sheet behaves differently for the E-polarization (E-field is parallel to the edge) and H-polarization (E-field is perpendicular to the edge).[3] In other words, the far-field scattering light intensity is closely associated with the incident polarization. It is also pointed out that the metallic diffracting edges produce a very marked elliptical polarization in the far-field. When the polarization is parallel to the metallic edge, more light is absorbed than when the polarization is perpendicular to the edge.[38] The phenomenon is somewhat in agreement with the near-field result obtained in our experiment.

4. Conclusions and perspectives

In conclusion, the combined SNOM setup is able to probe the near-field polarization response of the gold knife-edge, which is obtained by chemically synthesizing the gold nanosheets. An uncoated straight fiber probe is used in the SNOM, because it still preserves the property of light polarization though it has the depolarization to some extent. The edge-induced depolarization is found and explained based on the FDTD simulation results. The polarized similarity between the near- and far-field implies a close relation between them, which needs to be studied further.

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