Scattering attributes of one-dimensional semiconducting oxide nanomaterials individually probed for varying light-matter interaction angles

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We report the characteristic optical responses of one-dimensional semiconducting oxide nanomaterials by examining the individual nanorods (NRs) of ZnO, SnO₂, indium tin oxide, and zinc tin oxide under precisely controlled, light-matter interaction geometry. Scattering signals from a large set of NRs of the different types are evaluated spatially along the NR length while varying the NR tilt angle, incident light polarization, and analyzer rotation. Subsequently, we identify material-indiscriminate, NR tilt angle- and incident polarization-dependent scattering behaviors exhibiting continuous, intermittent, and discrete responses. The insight gained from this study can advance our fundamental understanding of the optical behaviors of the technologically useful nanomaterials and, at the same time, promote the development of highly miniaturized, photonic and bio-optical devices utilizing the spatially controllable, optical responses of the individual semiconducting oxide NRs. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4933400>]

One-dimensional (1D) nanomaterials based on semiconducting oxides (SOs) have demonstrated their useful properties in numerous applications of photonics,^{1–4} electronics,^{5–8} optoelectronics,^{9,10} photovoltaics,^{11–15} and chemical/biological sensing.^{16–24} In many of these technologically important applications, the fundamental optical properties of 1D SO nanomaterials govern their functional outcomes. Light can produce various optical and optoelectronic responses from the materials and, therefore, light-matter interactions can be engineered to produce desirable optical properties such as spontaneous and stimulated emission,^{1,25–29} waveguiding,^{1–3} and evanescent field enhancement.^{4,30–32} Such examples can be seen in the research efforts previously reported for the development of SO nanomaterials for applications in nanoscale lasers,^{25,26,29} subwavelength waveguides,^{1–3,33,34} and biodetection platforms.^{17,18,20,23,24,33,35}

Specifically, nanomaterials of zinc oxide (ZnO), tin oxide (SnO₂), indium tin oxide (ITO), and zinc tin oxide (ZTO) have been widely utilized as signal transduction elements in optical detection devices.^{5,8,36,37} These past applications have primarily exploited the optical properties of bulk materials or ensembles of nanomaterials. With the ever-growing demand for device and sensor miniaturization, novel constructs with highly reduced dimensions have also been explored recently. Therefore, elucidating the exact light interaction profiles with individual nanostructures can provide much needed insight and further benefit the burgeoning efforts in single nanorod (NR) optical, optoelectronic, and biosensing devices.

In this study, we carry out dark-field (DF) experiments in a forward scattering geometry and investigate the fundamental optical responses of individual nanomaterials by systematically controlling the interaction angle between the direction of an incident light oscillation and the main crystal axis of a nanomaterial. 1D nanomaterials, specifically NR forms of ZnO, SnO₂, ITO, and ZTO are examined for their

interaction angle-dependent, elastic scattering profiles. We determine characteristic scattering responses from each position along the main axis of the SO NRs while controlling the NR orientation, incident light polarization, and analyzer rotation. Our endeavors signify the first systematic investigation of spatially resolved, forward light scattering of single NRs, examining various individual SO NRs in order to determine the precise influence of the effective angle of the light-matter interaction. Our findings demonstrate strikingly different scattering responses depending on the NR tilt angle and the polarization direction of the incident light. The knowledge gained can be used to better understand the fundamental optical responses of the technologically useful nanomaterials at the single NR level. Insight from our research efforts may also benefit the analysis of scattering and emission signals acquired from highly miniaturized, photonic and bio-optical devices utilizing the spatially controlled, optical responses of the 1D SO nanomaterials.

Our experimental approach employs a home-built series of optical components in addition to an Olympus BX51F optical microscope, whose setup is configured in a DF mode. The DF setup involves two scattering geometries, one with a forward scattering configuration and the other with a reflected scattering configuration. The forward DF (FDF) configuration shown in Figure 1(a) ensures that the measured signal comes only from the NR under investigation by eliminating the source light from the collection optics via total internal reflection (TIR). In addition to the FDF channel, the setup is also equipped with a reflected DF (RDF) scattering pathway. This detection mode involves a wide-field, unpolarized illumination above the sample plane and subsequent collection of the scattering signal in the backward direction towards a DF objective lens.

Single crystalline NRs of ZnO, SnO₂, ITO, and ZTO were synthesized in a home-built chemical vapor deposition reactor as reported earlier.^{15,17,18,20,38,39} The size, morphology, and crystal structure of the nanomaterials were reported previously along with their scanning electron microscopy

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FIG. 1. (a) The overall schematics of the FDF measurement setup are shown. (b) A detailed view of the refractive index-matching NR sample assembly is displayed. (c) The schematic representations illustrate the key light-matter interaction parameters including NR_θ and $\mathbf{E}_\theta$. (d) Two example configurations of the light-matter interactions are shown to depict different NR_θ scenarios under $\mathbf{E}_\theta = 0^\circ$.

and X-ray diffraction data.^{15,17,18,20} The typical diameters and lengths of the NRs employed in our measurements range from 150 to 300 nm and from 5 to 30 μm , respectively. As-grown NR samples were first dispersed in ethanol via sonication from their growth substrate and deposited on a clean glass slide by drop casting. Then, a small drop of glycerol (refractive index of 1.4729) was placed on the NRs on the glass slide before lowering a cover slip onto the assembly, as depicted in Figure 1(b). This refractive index-matching sample assembly results in TIR only at the topmost interface of the air/coverslip beneath which the layers can be treated as an optically uniform medium with no interface-mediated interferences.⁴⁰

In the FDF measurements, a linearly polarized 642 nm laser (Spectra Physics Excelsior-PS-DD-CDRH) entered the NR plane via an oil-immersion DF condenser (Numerical Aperture, $\text{NA} = 1.2\text{--}1.4$) from below the sample stage after passing through a half-lambda (HL) wave plate. The HL plate controlled the incident direction of the linearly polarized laser, $\mathbf{E}_\theta$ in Figure 1(c), to be between 0° and 90° . For the polarization case referred to as $\mathbf{E}_\parallel$ (and $\mathbf{E}_\perp$), $\mathbf{E}_\theta$ is 0° (and 90°) and the polarized light direction is within (and perpendicular to) the plane of incidence. For an illumination case with an arbitrary angle of $\mathbf{E}_\theta$, the incident polarization lies in a plane angled between $\mathbf{E}_\parallel$ and $\mathbf{E}_\perp$. The intersecting axis between the plane of incidence (the y-z plane) and the sample plane (the x-y plane) is further defined as y. The angle formed between the NR main axis and the y-axis is then referred to as the NR tilt angle of NR_θ . Two cases of the light-matter interaction configurations involving NR_θ of

0° and of an arbitrary value are displayed in Figure 1(d) along with the projected components ($\mathbf{E}_\parallel^*$ and $\mathbf{E}_\perp^*$) of the incoming polarization field. NR scattering signals were collected with a $40\times$ plan apochromatic objective lens (Olympus PlanSapo, $\text{NA} = 0.90$). An analyzer was placed between the microscope tube lens and the charge-coupled-device detector (QImaging Exi Blue CCD camera, Surrey, Canada).

Unpolarized white light (a 100 W, 12 V halogen lamp) was also used to examine the scattering behavior of the NRs in a RDF mode. The wide field illumination and collection of the NR signals were carried out with $20\times$ (Olympus MPlanFL N, $\text{NA} = 0.45$) and $50\times$ (Olympus MPlanFL N, $\text{NA} = 0.80$) DF objective lenses. After systematically examining the FDF and RDF scattering profiles of at least 30 NRs for each type of nanomaterials, we consistently observed characteristic scattering behaviors that are NR_θ - and $\mathbf{E}_\theta$ -dependent. Herein, we focus on reporting the NR_θ - and $\mathbf{E}_\theta$ -contingent scattering responses from these NRs that are material-indiscernable.

Figures 2 and 3 present the typical NR_θ -dependent scattering profiles evaluated spatially along single NRs as a function of the analyzer rotation (ϕ). The representative data are displayed in the increasing order of NR_θ in Figures 2 and 3. Figures 2(a) and (b) and Figures 2(c) and 2(d) summarize the characteristic scattering behaviors determined from the NRs of $\text{NR}_\theta = 0^\circ$ and 25° , respectively. Similarly, Figures 3(a) and 3(b) and Figures 3(c) and 3(d) are acquired from those NR cases of $\text{NR}_\theta = 55^\circ$ and 80° , respectively. The particular data sets shown in Figures 2 and 3 are from an ITO NR

FIG. 2. Typical scattering profiles of the NRs with $\text{NR}_\theta = 0^\circ$ and 25° are presented. (a) and (b) The scattering data are obtained from a $\text{NR}_\theta = 0^\circ$ ITO NR via the (a) RDF and (b) FDF mode. (c) and (d) The scattering signals collected in the (c) RDF and (d) FDF mode are from a $\text{NR}_\theta = 25^\circ$ ZTO NR.

FIG. 3. Typical scattering profiles of the NRs with $\text{NR}_\theta = 55^\circ$ and 80° are displayed. (a) and (b) The scattering data are obtained from a $\text{NR}_\theta = 55^\circ$ SnO_2 NR via the (a) RDF and (b) FDF mode. (c) and (d) The RDF and FDF scattering shown in (c) and (d), respectively, are taken from a $\text{NR}_\theta = 80^\circ$ ZnO NR.

(Figs. 2(a) and 2(b)), a ZTO NR (Figs. 2(c) and 2(d)), a SnO_2 NR (Figs. 3(a) and 3(b)), and a ZnO NR (Figs. 3(c) and 3(d)). Although these selective data sets are chosen to present our results encompassing all four types of SO NRs, the specific scattering characteristics discussed in depth below can be applied to predict and describe the scattering behavior from any of the four nanomaterial types so long as they exhibit a comparable NR_θ .

Panels (a) and (c) of Figures 2 and 3 are the scattering patterns obtained with a wide-field, unpolarized illumination under the RDF configuration, while panels (b) and (d) summarize the FDF NR scattering results examined under the precisely controlled $\mathbf{E}_\theta$. The grey panels in both the RDF and FDF data correspond to the scattering images of each NR. The colored panels of the 3D contour and 2D surface plots included in the FDF data set show the NR scattering intensities analyzed as a function of the position along the NR as well as ϕ . Due to the measurement symmetry, the range of ϕ shown in the plots is from 0° to 90° . The incident polarization was kept the same as $\mathbf{E}_\parallel$ for all FDF results shown in Figures 2 and 3 in order to pinpoint the distinct scattering behaviors solely influenced by NR_θ .

Under the unpolarized light in our RDF setup, the scattering signal is present continuously along all NR positions in all NR_θ cases. In contrast, the same NRs under the

polarization controlled light exhibit highly NR_θ -correlated scattering profiles along the length which we categorize as continuous, intermittent, or discrete patterns. For $\text{NR}_\theta = 0^\circ$, the FDF scattering response is similar to that of the RDF and characterized as continuously present on all NR positions, as exemplified in Figure 2(b). As expected from the cross-polarizer setting, the overall intensity of the signal is observed to be the highest (or lowest) when the analyzer rotation is parallel (or perpendicular) to the incoming polarization of $\mathbf{E}_\parallel$, regardless of the position on the NR. Typical scattering behaviors of $\text{NR}_\theta = 25^\circ$ are shown in Figure 2(d). Overall, the FDF scattering response is continuous along the NR whose scattering intensity is maintained above 75% of the highest signal measured on all areas of the NR. For NRs with even a larger NR_θ value of 55° as displayed in Figure 3(b), the scattering signal reveals an intermittent pattern along the NR length with alternating peaks and valleys. In this case, the intensities on the same NR fluctuate from high to low along the NR length in which the valley signals drop to at least 50% of the peak intensities. This effect is clearly seen from the grey panel of $\phi = 0^\circ$ in Figure 3(b) with alternating bright and dark spots along the NR. For NRs with a much higher NR_θ value of 80° , the FDF scattering responses turn out to be highly localized and exclusively present on the two NR ends, yielding a distinctively discrete scattering pattern in Figure 3(d). All FDF scattering intensities are from the NR end positions only, with no signal present on the NR main body.

These NR_θ -dependent scattering behaviors are further substantiated in the data provided in Figure 4(a). For each type of NR, the above-mentioned scattering patterns examined as a function of NR_θ are categorized as continuous or discontinuous (intermittent/discrete) groups. The solid blue and open red symbols indicate the NR_θ value determined to have a continuous or discontinuous scattering profile, respectively. When collectively evaluating all nanomaterial data in Figure 4(a), the continuous to discontinuous scattering transition is noted at $\text{NR}_\theta = 45^\circ$ and, due to the geometrical symmetry of the NR, additionally at 135° . From these experimental observations, we determine that NRs exhibit spatially continuous scattering for $0^\circ \leq \text{NR}_\theta \leq 45^\circ$ and $135^\circ \leq \text{NR}_\theta \leq 180^\circ$, whereas NRs with $45^\circ < \text{NR}_\theta < 135^\circ$ yield discontinuous scattering patterns.

Our data indicate that the continuous and discontinuous FDF scattering behaviors are governed by the light-matter interaction geometry. In our FDF setup, the light is aligned onto a single spot of the ring-like light pathway of the DF condenser lens, rather than passing through the entire lens. Therefore, when NR_θ is 0° as depicted in the top panel of Figure 1(d), the NR long axis lying in the plane of incidence is under propagating electromagnetic fields of the incoming light, producing a continuous scattering signal from the NR. On the other hand, the case of $\text{NR}_\theta \neq 0^\circ$ in the bottom panel of Figure 1(d) will yield a scenario in which only a portion of the NR directly interacts with the light along the NR width. Considering that the NR has a diameter smaller than λ , this circumstance effectively sets up a condition of isolated illumination to a limited portion of the NR, and NR_θ will determine the number of illumination point sources along the NR long axis. The number of point sources

FIG. 4. (a) The NR_θ -dependent scattering profiles are examined on the four types of NRs for the continuous (solid symbols) to discontinuous (open symbols) response. (b) E_θ -dependent scattering behavior is shown for the three cases of $E_\theta = 0^\circ$, 45° , and 90° by plotting the normalized scattering intensity distributions at different positions along the same ZnO NR with $\text{NR}_\theta = 10^\circ$ while changing ϕ .

that can exist along the NR length will be the largest at $\text{NR}_\theta = 90^\circ$, which will lead to two strong signal peaks analogous to the interference wave patterns expected from a linear array of multiple point sources.⁴¹ In an arbitrary NR_θ case, the interplay between $\mathbf{E}^*_{\parallel}$ and $\mathbf{E}^*_{\perp}$ will determine the dominance of the continuous or discontinuous scattering nature in the resulting signal. For $\text{NR}_\theta > 45^\circ$, the dominant $\mathbf{E}^*_{\perp}$ component turns the NR scattering from continuous to discontinuous, and the effective illumination is similar to $\text{NR}_\theta = 90^\circ$ but with a decreased number of point sources. In this case, additional scattering peaks of lower intensity will exist in between the bright end signals, leading to the intermittent scattering pattern. We can also expect that, depending on the proximity of k with the non-normal incident angle ($\theta_{\text{inc}} = 62^\circ$) to each end of the NR, the scattering intensities measured at the two NR ends or along the NR length will not be identical to each other or perfectly symmetric along the NR. Indeed, this is evidenced from the majority of our data whether they are of a continuous or discontinuous nature. In contrast, our RDF measurement layout with the wide field, unpolarized illumination setting will be equivalent to a combination of all FDF illumination scenarios, which leads to continuous scattering patterns in all NR_θ cases.

Additionally, NR scattering responses are examined with respect to the different polarization direction of the incident light, E_θ , and the results are shown in Figure 4(b). All three plots of E_θ equal to 0° , 45° , and 90° display the

normalized scattering intensity distributions at different positions along the same ZnO NR with $\text{NR}_\theta = 10^\circ$ plotted as a function of ϕ . Despite the change in E_θ , the $\text{NR}_\theta = 10^\circ$ NR exhibits a continuous scattering profile along its length. Therefore, E_θ does not seem to affect the NR_θ -contingent continuous, intermittent, or discrete signal yielded from the NR scattering pattern. However, regardless of the position on the NR, the maximum scattering intensity is observed when ϕ is the same as E_θ for the three cases shown in Figure 4(b). Hence, altering E_θ shifts the scattering intensity to peak at the analyzer rotation parallel to the E_θ of the incoming light.

Our study, while systematically elucidating the NR_θ - and E_θ -governed effects on single NR scattering, uniquely presents the characteristic scattering intensities revealed explicitly at different positions along individual NRs, whose specifics cannot be drawn from the past theoretical and experimental efforts on light scattering. Our study represents an important step forward to exploit the locally resolved NR scattering responses for ultimate use in a single NR device. We demonstrate the possibility of not only optically addressing each NR individually but also triggering position-specific scattering signals on the NR by controlling NR_θ and E_θ . Such knowledge may offer a distinctive advantage in individual NR-based biosensor devices and scattering-based biomedical imaging owing to the new capability of tuning the light coupling locally and spatially to biomolecules by controlling the tilt angle of the biofunctionalized NRs. Another biological application may include the straightforward and simultaneous identification of dynamic directional changes in NR-linked, biological fibers and tubules in a mixture of complex orientations. These benefits pertain to our findings of material-independent scattering phenomena revealed from a single wavelength study. Further work is also underway to elucidate nanomaterial-dependent scattering characteristics and to identify different physicochemical properties of these NRs using a multiwavelength scattering technique, whose efforts will be reported elsewhere.

In summary, we have systematically examined the light-interaction characteristics of individual ZnO, ITO, SnO₂, and ZTO NRs. Subsequently, distinctive and interesting scattering responses are revealed explicitly as a function of the position along individual NRs. We focus on reporting the NR position-specific scattering profiles of these individual SO NRs that are material-independent but NR_θ - and E_θ -dependent. The spatially resolved scattering information from single NRs will be useful for further local manipulation of the NR position-specific scattered light. Our findings will be particularly useful in highly miniaturized biomedical applications where single NRs are exploited as scattering-based detection and imaging elements capable of probing both luminescent and non-luminescent bioconstituents.

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