Strong near-field light-matter interaction in plasmon-resonant tip-enhanced Raman scattering in indium nitride.

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Abstract: We report a detailed study of the strong near-field Raman scattering enhancement which takes place in tip-enhanced Raman scattering (TERS) in indium nitride. In addition to the well-known first-order optical phonons of indium nitride, highly localized Raman modes, not detectable in the far-field, appear when approaching the plasmonic probe. The frequencies of the localized modes coincide with calculated energies of second order combinational modes consisting of optical zone center phonons and acoustic phonons at the edge of the Brillouin zone. The appearance of these combinational modes suggests that TERS in indium nitride represents a special case of Raman scattering in which a resonant condition on the nanometer scale is achieved between the localized surface plasmons (LSPs) and surface plasmon polaritons (SPPs) of the probe with the charge oscillations of the material. This resonance results in a strong enhancement of the highly localized Raman modes. We suggest that the surface charge accumulation in InN¹,²,³ is the dominating reason for the unusually large enhancement of the TERS signal as compared to other inorganic semiconductors. Thus, the plasmon-resonant TERS process in InN makes this technique an excellent tool for defect characterization of indium-rich semiconductor heterostructures and nanostructures.

During the last two decades, group-III-nitride semiconductor hetero- and nanostructures have established a dominant role as versatile materials for solid-state light-emitting devices. The large tunability of the direct band gap in the ternary alloys InGaN and AlGaN makes them the best choice for optoelectronic applications ranging from the ultraviolet to the green spectral region⁴. The bottleneck, which prevents nitride-based devices to be available as longer wavelength emitters, is the efficiency droop⁵ for high indium concentration due to the drastic increase of non-radiative recombination centers such as defects, dislocations, interfaces and related non-radiative recombination processes like Auger recombination⁶. Raman spectroscopy is recognized as an essential tool for the characterization of strain, compositional fluctuations and defects in III-nitrides, however it remains a diffraction-limited technique, which cannot access subwavelength information, i.e. it is not suitable for characterization on the nanometer scale. Optical near-field techniques are powerful solutions that circumvent these limitations and enable the characterization of III-nitride nanostructures with few nanometers of spatial resolution. Tip-enhanced Raman spectroscopy (TERS) exploits the plasmonic and polaritonic properties of a metallic tip in order to enhance and localize the electromagnetic field which interacts with the sample⁷. While the majority of TERS studies focus on the vibrational properties of molecules and clusters⁸,⁹,¹⁰, only few works exist that address near-field Raman scattering in inorganic semiconductor and their nanostructures. These include Si¹¹,¹², Ge and SiGe¹³,¹⁴, GaN¹⁵,⁷, BaTiO₃¹⁶, GaAs¹⁷, as well as nanowires of InP¹⁸, GaN¹⁹, Ge²⁰. In the case of the III-nitride material system, we have previously reported the occurrence of InN clusters in InGaN nanostructures using TERS and derived detailed information on the strain distribution, chemical composition, polymorphism, and charge accumulation with a spatial resolution below 35 nm²¹.
In this work, we demonstrate the potential of TERS for nanoscale characterization of III-nitrides by studying the plasmonic and polaritonic interaction between InN and a gold tip which leads to a pronounced enhancement of the localized Raman scattering. We investigate epitaxial InN samples grown by two different techniques on different substrates. The MEPA sample is composed of a 90 nm thick InN heteroepitaxial film grown by migration-enhanced plasma-assisted metal organic chemical vapor deposition (MEPA-MOCVD) on a c-plane sapphire substrate\textsuperscript{22}. The MBE sample consists of a 2 µm thick InN film grown by plasma-assisted molecular beam epitaxy (PA-MBE) on a 100 nm buffer layer of C-doped GaN. The buffer layer was deposited on a 3.5 µm thick Fe-doped GaN template commercially available from Lumilog. Residual indium was etched using HCl after unloading\textsuperscript{23}.

The TERS equipment consists of a commercially Raman spectrometer (Horiba-Jobin Yvon LabRam HR-800) coupled with an atomic force microscope (Park XE-100 AFM). An 80x long-distance microscope objective, tilted by 60° degrees with respect to the z-axis, illuminates the bulk gold tip produced by electrochemical etching of a 50 µm gold wire with the light from a 633 nm He-Ne laser. The near-field Raman signal generated in the tip-sample junction is collected by the same objective. A detailed description of the far-field scattering geometry and the near-field signal acquisition is reported in Ref. 7.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Backscattering $z(\cdot,z)$ (black curve), far-field (blue curve) and near-field (red curve) Raman spectra of (a) 90 nm thick InN epitaxial layer grown by MEPA-MOCVD on sapphire (MEPA sample) and (b) 2 µm thick InN grown by PA-MBE on C-doped GaN (MBE sample). The near-field (red) curves represent the TERS spectra acquired with the gold tip approached.}
\end{figure}
Fig. 1a and Fig. 1b display the Raman spectra of the MEPA sample and the MBE sample, respectively. The black curves represent the far-field spectra acquired in the z(-)-z backscattering geometry. Both samples show the $E_2$(high) and $A_1$(LO) Raman modes of InN$^{23}$ in the displayed spectral range between 400 and 750 cm$^{-1}$. A direct comparison between the samples reveals a smaller FWHM of the $E_2$(high) mode in the MBE sample, indicating a superior “bulk” crystalline quality as compared to the MEPA sample. The blue curves show the far-field spectra acquired with the 60° tilted TERS objective and retracted gold tip, which are displayed as reference for the near-field spectra. By approaching the tip to the surface of the sample in this geometry, the TERS spectra are obtained (red curves in Fig. 1). Both samples exhibit a drastic change of their Raman spectra expressed by variations of mode intensities and the appearance of new peaks in the near field. This observation is in agreement with previously reported TERS spectra of InN clusters embedded in InGaN quantum wells.$^{21}$ The reason for the pronounced change of the Raman spectra lies within the increased surface sensitivity (probing-depth approximately 5-10 nm) and greatly reduced scattering volume (nanometer scale)$^{24}$ as well as the breakdown of classical Raman selection rules in in near-field Raman scattering$^7$.

In the following paragraphs, we discuss the near field Raman spectra of the different InN samples in detail. The $A_1$(TO) phonon is strongly enhanced in both near-field spectra, disregarding the far-field selection rules. Based on the smaller FWHM of the $A_1$(TO) mode we can conclude that the crystal quality in the surface region that gives rise to the TERS signal is higher in the MEPA sample than in the MBE sample. This is in alignment with the surface roughness measured by AFM. The MEPA sample exhibits a smooth surface (mean surface roughness $R_{\text{rms}} = 5.7$ nm), whereas the MBE sample shows domains of different heights possibly related to the post growth chemical etching ($R_{\text{rms}}= 11.8$ nm). A mode close to the frequency of the $E_2$(TO) phonon is observed in the MEPA sample, but absent in the MBE sample. On the contrary, the $E_2$(high) phonon is largely enhanced in the MBE sample but not enhanced in the MEPA sample. Furthermore, a broad Raman mode between 500 cm$^{-1}$ and 600 cm$^{-1}$ is visible in both spectra but at different frequencies. Thakur et al. observed a similar feature in far-field Raman spectra of InN and assigned it to the $B_1$(high) phonon$^{25}$. The authors reported experiments over a set of samples with different free carrier concentrations from $3.8 \cdot 10^{20}$ to $1.6 \cdot 10^{21}$ and observed a red shift of the phonon frequency with increasing free carrier concentration. In our case, the observed peaks in this spectral region could be related to plasmon coupled Raman modes, which depend on the surface carrier concentration; however, the available data is insufficient for an unambiguous attribution of the phononic origin of these bands.

Apart from the first order Raman modes, two previously unreported peaks, labeled $C_1$ and $C_{1u}$, appear in the near-field Raman spectra of both InN samples at the same spectral positions between 646 cm$^{-1}$ and 661 cm$^{-1}$ (see Fig. 1 and Tab. 1). These modes deserve particular attention as no Raman modes of InN are expected at these frequencies, neither based on calculation of the phonon dispersion, nor based on other far-field experimental works$^{26,23,27}$. In order to understand the origin of these two Raman modes, we performed a TERS surface line scan on the MEPA sample with a point-to-point distance of 43 nm, as shown in Fig. 2. Over most of the distance of the line scan, the $A_1$(TO) phonon energy scatters equally around its nominal value and towards its end shifts to higher wavenumbers. These fluctuations of the $A_1$(TO) phonon energy are likely caused by local variations of strain and charge accumulation at the surface$^{28,23}$. Interestingly, the Raman modes $C_1$ and $C_{1u}$ seem to follow the same behavior. In order to explain this correlation, we compare the position and energy spacing of these modes.
The frequencies of the phonons $A_1$(TO), $C_L$, and $C_H$ as a function of the spatial coordinates are shown in Fig. 2b as obtained by peak fitting. Subtraction of the experimentally obtained values of the $A_1$(TO) mode from those of the $C_L$ and $C_H$ modes gives average values of 195 cm$^{-1}$ and 211 cm$^{-1}$, respectively. In this frequency range, a Raman mode around 200 cm$^{-1}$ was reported in the literature which was attributed either to the $B_1$(low) silent mode$^{29}$ or to overtones of transverse acoustic (TA) phonons near the symmetry points K or M of the Brillouin zone$^{30}$. Comparing our experimental data with the phonon dispersion calculations reported in Ref. 27, Ref. 30, and Ref. 31, we suggest that the modes $C_L$ and $C_H$ are combinational modes composed of the $A_1$(TO) phonon with M point phonons originating from the longitudinal acoustic (LA) branch. We can therefore calculate the expected values for the combinational modes as the experimental value of the $A_1$(TO) phonon plus the calculated values for the acoustic phonons:

$$C_H^{expected} = A_1^{(TO)}_{\text{exp}} + M_H^{\text{calc}} \quad \text{and} \quad C_L^{expected} = A_1^{(TO)}_{\text{exp}} + M_L^{\text{calc}}.$$  \hspace{1cm} (eq. 1)

We use the strain free values of 194 cm$^{-1}$ for the $M_L^{\text{calc}}$ phonon and 213 cm$^{-1}$ for the $M_H^{\text{calc}}$ phonon from Ref. 27. The impact of local strain variations can be estimated by consideration of the reported pressure coefficients of phonon modes of InN in the literature. Calculations for the phonon dispersion of the InN lattice under hydrostatic pressure predict a pressure coefficient of around 1.1 cm$^{-1}$/GPa and 1.6 cm$^{-1}$/GPa for $M_L$ and $M_H$ respectively$^{32}$, which is much lower than the measured hydrostatic pressure coefficient of...
the \( A_\text{I}(\text{TO}) \) phonon of \( 4.3\text{cm}^{-1}/\text{GPa} \). Consequently, no significant deviation from the strain profile of the \( A_\text{I}(\text{TO}) \) mode is expected for the combinational modes \( C_\text{L} \) and \( C_\text{II} \). The expected values are indicated in Fig. 2b by the dashed lines, which reveal a good agreement with the experimental values. Furthermore, we note that the two-phonon density of states calculated in Ref. 23 exhibits a pronounced double peak around 660 cm\(^{-1}\) which provides additional support for our interpretation of the \( C_\text{L} \) and \( C_\text{II} \) peaks as two phonon combinational modes. The plasmon and polariton-mediated localized excitation in TERS relaxes the far-field Raman selection rules and momentum conservation rule, which enables the observation of these modes exclusively in the near-field spectra of InN. The explanation for the selectivity of the strong enhancement of the \( A_\text{I}(\text{TO}) \) and zone edge phonons \( M_\text{L} \) and \( M_\text{H} \) can be found in the nature of the scattering mechanism of TERS as discussed in Ref. 7 where the TERS process is described as a photon tunneling event. However, it should be noted, that the complex momentum carried by localized surface plasmons and surface plasmon polaritons and its conservation in the photon tunneling process is still a matter of scientific debate.  

| Near-field (this work) | Far-field |
|-----------------------|-----------|
| Phonon                | MEPA sample | MBE sample | Literature (Ref. 23) | Predicted (Ref. 23+Ref. 27) |
| \( A_\text{I}(\text{TO}) \) | 451.8 cm\(^{-1}\) | 451.5 cm\(^{-1}\) | 450.5 cm\(^{-1}\) |  |
| \( E_\text{I}(\text{TO}) \) | 480.1 cm\(^{-1}\) |  | 477.8 cm\(^{-1}\) |  |
| \( E_\text{II}(\text{high}) \) |  | 491.2 cm\(^{-1}\) | 492.7 cm\(^{-1}\) |  |
| \( C_\text{L} = A_\text{I}(\text{TO})+M_\text{L} \) | 648.9 cm\(^{-1}\) | 646.4 cm\(^{-1}\) |  | 644 cm\(^{-1}\) |
| \( C_\text{II} = A_\text{I}(\text{TO})+M_\text{H} \) | 661.4 cm\(^{-1}\) | 660.1 cm\(^{-1}\) |  | 663 cm\(^{-1}\) |

Tab. 1: Phonon frequencies of InN. Near-field values are measured by TERS in the MEPA and MBE grown samples. Far-field and expected phonon frequencies based on literature values and phonon dispersion relation calculation as described in Eq. 1 and in the text.  

It is worthwhile to highlight two important experimental observations of this work: 1) the significant signal enhancement of TERS in InN under specific excitation conditions, i.e. bulk gold tip with 633nm laser excitation, and 2) the exclusive appearance of strong combinational modes in the near-field spectra. In order to understand these observations, we consider the interaction between all constituents of the near-field scattering process. The complex scattering mechanism comprises of one particle and three quasiparticles: photons, phonons, localized surface plasmons (LSPs), and surface plasmon polaritons (SPPs). All of these particles populate the tip-sample junction, making the scattering process particular sensitive to the individual surface properties of the samples. Depending on the surface free carriers of the studied material, the plasmon population can interact weakly or strongly with the LSPs and SPPs of the tip apex, thus influencing the electron-phonon coupling and the amplification of the Raman signal. The mechanism of near-field light-matter interaction, described in Ref. 7, considers a complex resonant condition composed by three energy levels: the exciting photon energy and both the plasmon and polariton energies of tip and sample. A resonant matching can occur when the plasma frequency of the sample is close to the frequency of the surface plasmon polaritons which populate the tip apex. The simplest plasmon-resonant TERS configuration is the one composed by a gold substrate and
a gold tip, which is known in TERS literature as “gap mode” configuration and it is usually adopted for studying 2D materials and single molecules positioned inside the tip-substrate gap\textsuperscript{36,37,38}. The particular case of TERS in InN discussed in this work likely presents a similar plasmon-resonant configuration due to the strong charge accumulation at the surfaces of InN\textsuperscript{2}. It is important to note that the observed enhancement cannot be explained either by near-field Raman scattering or by resonant Raman scattering alone. In order to underline this point, we have varied the excitation wavelength in both near-field and far-field configurations. When the gold tip is retracted from the InN surface, the signal enhancement disappears; the same applies when the laser excitation is changed from 633 nm to 532 nm while the tip is engaged. This demonstrates that the strong near-field Raman scattering in InN is a resonance effect with plasmonic and polaritonic mediation that strongly benefits from the high surface charge carrier concentration in InN. Consequently, the enhancement factor of TERS strongly depends on the surface properties of the sample which also explains the pronounced differences between the studied MEPA and MBE samples.

Following this discussion, we can classify the tip-sample interactions in TERS experiments in three different categories: strong interaction regime, weak interaction regime, and non-interaction regime. An example of strong interaction regime is TERS in InN as studied in this work and also investigated in Ref.\textsuperscript{21}, where Raman peaks, not present in the far-field spectrum, appear in the TERS spectrum. A detailed study of an example of the weak interaction regime has been reported in Ref.\textsuperscript{7} for TERS on graphene and carbon nanotubes, where the far-field Raman peaks are enhanced around twice of the intensity by approaching the tip. TERS on ZnO samples can be chosen as a non-interaction regime example, where the absence of literature and the lack of enhancement in our own experiments (not shown) underline the missing near-field interaction. The breakdown of the far-field selection rules in the weak interaction regime has been studied in details in Ref.\textsuperscript{7}. In this work, the breakdown of selection rules is also confirmed for the strong interaction regime; however, following a different pattern than the one for the weak interaction regime. The $A_2$(TO) phonon is selectively enhanced, disregarding the crystal orientation or the hexagonal or cubic modification in agreement with previous observations\textsuperscript{21}. However, two additional modes $C_1$ and $C_{1u}$ appear along the $A_2$(TO) phonon which experience a pronounced resonance effect resulting in the strongest enhancement of all near-field Raman modes. The presence of these two-phonon Raman scattering processes confirms that in the case of TERS in InN, a plasmon-resonant condition is fulfilled that leads to the observed strong interaction.

In conclusion, we have shown that a strong near-field light-matter interaction occurs for tip-enhanced Raman scattering (TERS) of InN, which is reproducible and independent of the growth technique of the samples. In the spectral range around 650 cm\textsuperscript{-1}, two distinct modes were observed in the near-field spectra, which exhibit particularly strong enhancement for plasmon-resonant excitation. The frequency of these modes matches a maximum in the two-phonon density of states of InN. We have identified these modes as two-phonon combinational modes, $C_1$ and $C_{1u}$, consisting of the optical phonon $A_2$(TO) and the acoustic phonons $M_{1}$ and $M_{1u}$ near the M point of the Brillouin zone. The unusually strong enhancement of these modes was attributed to a resonance between the exciting photons with the plasmon and polariton energies of the tip and the InN surface. Based on these observations we conclude that near-field Raman characterization of semiconductors with large surface charge accumulations is particularly powerful and has tremendous potential for the characterization of In clusters and InN inclusions in technologically relevant InGaN heterostructures and nanostructures with high In content.
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