

High contrast scanning nano-Raman spectroscopy of silicon

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We have demonstrated that scanning nano-Raman spectroscopy (SNRS), generally known as tip-enhanced Raman spectroscopy (TERS), with side illumination optics can be effectively used for analysis of silicon-based structures at the nanoscale. Even though the side illumination optics has disadvantages such as difficulties in optical alignment and shadowing by the tip, it has the critical advantage that it may be used for the analysis of nontransparent samples. A key criterion for making SNRS effective for imaging Si samples is the optimization of the contrast between near-field and far-field (background) Raman signals. This has been achieved by optimizing the beam polarization, resulting in an order of magnitude improvement in the contrast. We estimate the lateral resolution of our Raman images to be ~ 20 nm. Copyright © 2007 John Wiley & Sons, Ltd.

KEYWORDS: tip-enhanced Raman spectroscopy (TERS); scanning nano-Raman spectroscopy (SNRS); apertureless near-field spectroscopy; silicon; depolarization

INTRODUCTION

The field of information technology relies heavily on the performance of silicon-based devices. There is an ever-increasing demand for large-scale integration achieved by a reduction in the size, and an increase in density, of these devices. The thermal effects associated with the device process flow result in local strain in the material,¹ which is critical to the performance of these devices.² Raman spectroscopy has been used extensively to map and understand the crystal orientation^{1,3,4} and stresses³ in silicon structures with micron scale resolution. With the shrinking of structures to smaller than submicron dimensions (currently 65 nm and even 45 nm), there is a need for a Raman spectroscopy technique that can map these stresses with nano-scale spatial resolution. However, the diffraction law limits the lateral resolution of the traditional micro-Raman technique to a few hundreds of nanometers. Raman analysis of sub-100 nm structures with current confocal micro-Raman technique is not feasible.

The strategy to achieve sub-100 nm spatial resolution with optical techniques is to use near-field optics. Near-field optics deals with illumination of an object by light emerging from a subwavelength aperture or scattered by a metallic tip or nano-particle of subwavelength size.⁵ The first concept for near-field optics, discussed by Synge,⁶ is to access the vicinity of a sample's surface with a subwavelength aperture. A few groups used various spectroscopy, including Raman, combined with aperture-limited near-field scanning optical microscope (SNOM).^{7–10} Webster *et al.*^{7,8} applied this approach to stress mapping of a damaged silicon crystal wafer and reported a spatial resolution of ~ 150 nm. Due to the low transmission of light through the aperture, it took 60 s to collect 150 counts of Raman intensity from one spot of a silicon sample. As a result, Raman imaging of a small region (26×21 points) took an unreasonably long time, ~ 9 h. Further, the spatial resolution achieved is still far from that required, and decreasing the aperture (to improve the resolution) will lead to further drastic decrease of the signal. Thus, the low optical transmission of aperture-limited probes makes it almost impossible to measure Raman images with sub-100 nm spatial resolution.

An alternative approach to near-field optics is to use apertureless metallized probes. The local enhancement of the electromagnetic field in the vicinity of the metallized tip's

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apex can result in a large fraction of the enhanced near-field signal being transmitted into the far-field region, making the signal detectable.¹¹ The main enhancement mechanism is provided by surface plasmons of the metallized or metallic tip. This enhancement of the electric field of the light (similar to the surface-enhanced Raman effect) is strongly localized in the vicinity of the metal surface.^{12,13} The positioning of the metallized tip near or on the sample's surface can be realized with the aid of a scanning probe microscope (SPM). The size of the region of the enhancement is close to the size of the contact area between the tip and sample and the radius of this region is usually comparable to, or smaller than, the radius of the tip. Tip-enhanced Raman spectroscopy (TERS) is the name that has most often been used for the recently developed Raman spectroscopy with apertureless near-field optics, because the term TERS emphasizes the enhanced Raman signal at the localized sample area beneath the metallized tip apex. The metallized probe may be scanned over the sample in a manner analogous to that used in SPM, and a Raman image with very high spatial resolution can be constructed. We will use the term 'scanning nano-Raman spectroscopy' (SNRS) to emphasize the scanning capability and use TERS when focusing on the ability to provide a highly enhanced signal from one spot.

Recently, SNRS analysis of single wall carbon nanotubes has demonstrated a lateral resolution of ~ 14 nm with so-called bottom illumination optics.¹⁴ This method uses an inverted optical microscope and metallized probe with SPM control on top. Even though the bottom illumination geometry has the advantages of simplicity in optical alignment and ease of adaptation to off-the-shelf equipment, it faces the serious drawbacks that the excitation radiation and scattered light must pass through the sample substrate and sample. Thus, the bottom illumination geometry is not appropriate for opaque samples and samples on nontransparent substrates. In particular, it is not appropriate for the analysis of silicon structures.

Sun and Shen^{15–17} were the first to report successful SNRS mapping of silicon/silicon oxide nano-structures. They used a modified top-illumination scheme. The Raman intensity when the metallized tip was in contact with the silicon surface (contact signal) was about 50% higher than the Raman intensity measured at the same focal spot on the sample without the tip (withdrawn signal). This enhancement from silicon is rather modest,^{17–20} and might not be enough for nano-Raman imaging due to the low contrast between the 'contact' and 'withdrawn' signals. Poborchii *et al.*²¹ have recently proposed the use of top-illumination geometry with a depolarization configuration to improve the difference between the contact and withdrawn signals for silicon. It is known that the Si Raman mode at 520 cm^{-1} is strongly polarized and that the far-field signal (or withdrawn signal) can be suppressed using an analyzer set for a depolarized geometry. In their case, a silver nano-particle at the apex of a quartz atomic force microscopy (AFM) tip depolarized the optical field in its vicinity, resulting in a partially depolarized near-field signal. Using this idea, the authors were able to achieve a contact signal more than two times higher than the far-field signal (withdrawn signal).²¹

Our work is focused on improving the nano-Raman image quality and the contrast between the contact and withdrawn signals in SNRS with side illumination optics by using the polarization scheme first put forth by Poborchii *et al.*²¹. By suppressing the withdrawn signal more than the contact signal using depolarized optics, the nano-Raman image quality and contrast between contact and withdrawn signals can be markedly improved.

EXPERIMENTAL

The nano-Raman system consists of a Horiba Jobin Yvon Labram HR-800 Raman spectrometer coupled with a Quesant (QScope 250) SPM operated in AFM contact mode. A long working distance Mitutoyo (APO SL50) objective ($\times 50$, 0.42

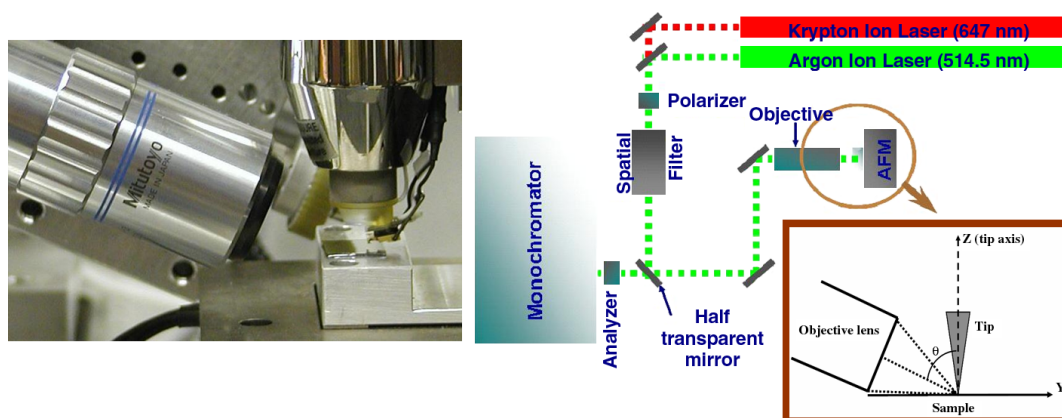


Figure 1. A schematic of the entire SNRS instrument with a photo (left) and schematic (lower right) of the side illumination optics showing the long working distance objective focused on the AFM tip. This figure is available in colour online at www.interscience.wiley.com/journal/jrs.

NA) is tilted 65° from the sample surface normal, as shown in Fig. 1. The objective is fixed on a XY stage controlled by step motors with an accuracy of ~ 40 nm to position the objective with respect to the tip. The samples are fixed on a XY piezo stage (nPoint XY100A). The depolarization configuration in our setup is attained by placing a polarization rotator in the incident beam path before the objective and an analyzer before the entrance slit of the monochromator as shown in Fig. 1. A mirror placed at an angle directs the laser beam through the objective.

Silicon nitride AFM tips (Park Scientific Instruments, Sharp Microlevers), each coated with a gold or silver layer using thermal vacuum deposition, were used for TERS and SNRS. The details of tip preparation are described in a previous publication.²² Silver-coated tips were used for scanning and imaging measurements and gold-coated tips were used for the other measurements. The enhanced signal was measured in contact mode in AFM with a normal force below 2 nN. Measurement of the Raman spectra was performed using the 514.5 nm line of an argon ion laser, with an incident power of <10 mW on a circular spot of the sample of about $2\ \mu\text{m}$ diameter. For the nano-Raman scanning, the contact position between tip and sample is rastered by moving the XY sample stage, rather than moving the tip, to attain minimal change in the relative position between the tip and the illumination spot. The Z piezo, on which the AFM tip is mounted, is used to maintain the normal force between tip and sample and to measure the sample topography.

RESULTS AND DISCUSSION

A major advantage of choosing side illumination optics is the flexibility of analyzing both transparent and nontransparent

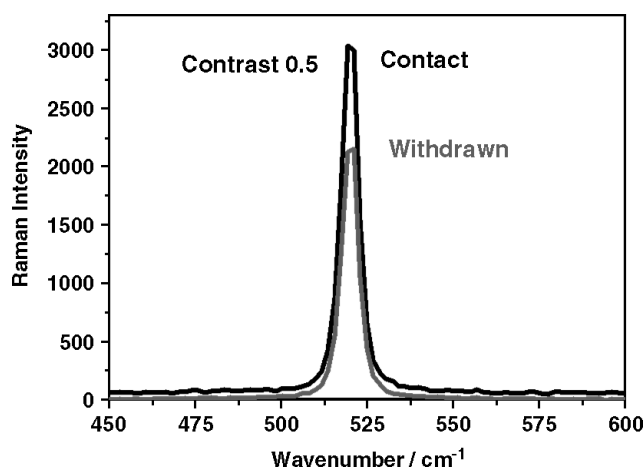


Figure 2. A 50% increase (contrast 0.5) in measured intensity is observed for a silicon sample when the Raman signal is enhanced by the presence of a gold-coated tip in contact with the surface. The incident polarization is -20° , measured relative to the axis of the tip.

samples. Another important advantage is the ability to orient the polarization of the incident light along the tip axis. Such orientation is known to be important for attaining maximum enhancement under the tip²³ (analogous to the optimization of enhancement for an elliptical particle^{24,25}). Figure 2 shows the Raman signal intensities for the $520\ \text{cm}^{-1}$ mode of Si (100) measured with a modified tip in contact with the silicon wafer surface and withdrawn $10\ \mu\text{m}$ from the surface. The signal measured with the tip withdrawn (withdrawn signal) consists of only the far-field Raman intensity. The contact signal is measured with a gold- or silver-coated silicon nitride tip positioned in contact with the sample and with the objective focused so as to maximize the Raman intensity. In order to understand the significance of the signal comparison, one must clearly delineate the different contributions to the contact signal. The contact signal contains contributions of far-field intensity not shadowed by the tip, near-field enhanced signal from the vicinity of the tip, and any additional signal induced by scattering and reflections from the tip. When we refer to the withdrawn signal in the remainder of the paper it is understood that this is the conventional far-field Raman signal and the contact signal refers to the signal measured in the presence of the tip, which contains multiple contributions.

Comparison of the signals and quantification of the various contributing phenomena is central to defining operational limits for imaging, and requires careful definition of the terms 'enhancement factor' (EF) and 'contrast'. A generally accepted definition of EF is the actual signal enhancement per scattering volume^{17,19,20,26} expressed as:

$$EF = \frac{I_{\text{near}}}{I_{\text{far}}} \frac{V_{\text{far}}}{V_{\text{near}}} = \left(\frac{I_{\text{total}}}{I_{\text{far}}} - 1 \right) \frac{V_{\text{far}}}{V_{\text{near}}} \quad (1)$$

where V_{near} and V_{far} are the sampling volumes from the near- and far-fields, respectively. For practical purposes, we introduce another parameter, contrast, that is defined as the ratio of the near-field signal intensity I_{near} to the far-field signal intensity, I_{far} ,

$$\text{Contrast} = \frac{I_{\text{near}}}{I_{\text{far}}} = \frac{I_{\text{total}}}{I_{\text{far}}} - 1 \quad (2)$$

The sum of I_{near} and I_{far} , or I_{total} , is what is measured in the contact state, based on the assumption that there is no additional signal contribution to I_{total} . Contrast is more important for characterizing the ability to do Raman imaging than is signal enhancement *per se*. The contact signal in Fig. 2 is about 50% larger than the withdrawn signal, and so the contrast in this case is 0.5. For a silicon sample, V_{far} is defined by the surface area of the laser spot multiplied by the smallest of three parameter values: sample thickness, light penetration depth or the depth of focus of the confocal optics ($\sim 1\ \mu\text{m}$). The penetration depth of the 514.5 nm laser light in silicon is²⁷ approximately $0.68\ \mu\text{m}$, and using this depth V_{far} is estimated as $\sim 2\ \mu\text{m}^3$. The localization of the tip-enhanced signal in depth has been estimated in our previous

publication²² using CdS films with different thickness. It appears²² to be ~ 20 nm. So, V_{near} can be approximated²² as the hemispherical volume of radius 20 nm under the tip, $2\pi R^3/3 \sim 2 \times 10^{-5} \mu\text{m}^3$. Thus, the contrast 0.5 corresponds to an EF of $0.5 \times \left(\frac{2}{2 \times 10^{-5}}\right) = 5 \times 10^4$. Surface enhanced Raman spectroscopy (SERS) reports²⁸ have claimed EFs of the order of 10^{14} . However, the values of EF seen for TERS^{15–17,29,30} have only reached about 10^2 – 10^4 . The EF we have achieved is comparable to that reported by Sun and Shen.^{15–17}

For practical nano-Raman analysis of Si samples, a contrast of 0.5 may not be enough. In order to increase the contrast, we have used the approach proposed by Poborchii *et al.*²¹ to minimize the contribution of far-field Raman intensity to the contact signal by suppressing the far-field signal more than near-field signal. When the polarization of the incident beam is varied from -20° to 160° with respect to the axis of the metallized tip, the Raman intensities for the Si mode in both the contact and withdrawn states are affected (Fig. 3). Though the contact and withdrawn signals are both substantially suppressed at

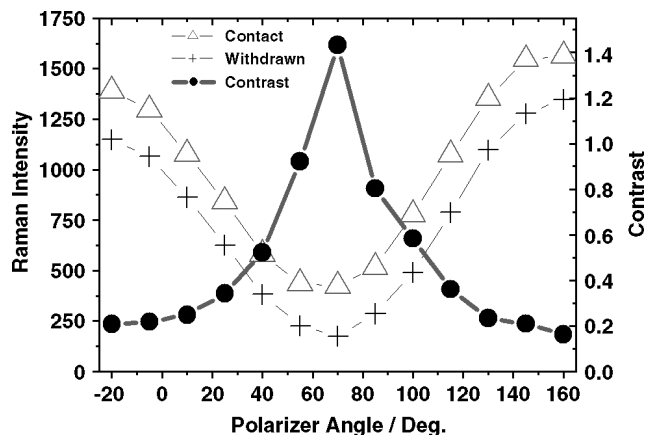


Figure 3. Intensities for the contact and withdrawn measurements vary with incident laser beam polarization angle with respect to the axis of the metallized (gold-coated in this case) tip such that the contrast is maximized for a polarization of 70° .

70° incident polarization, their difference remains constant during the variation of the polarization angle. This means

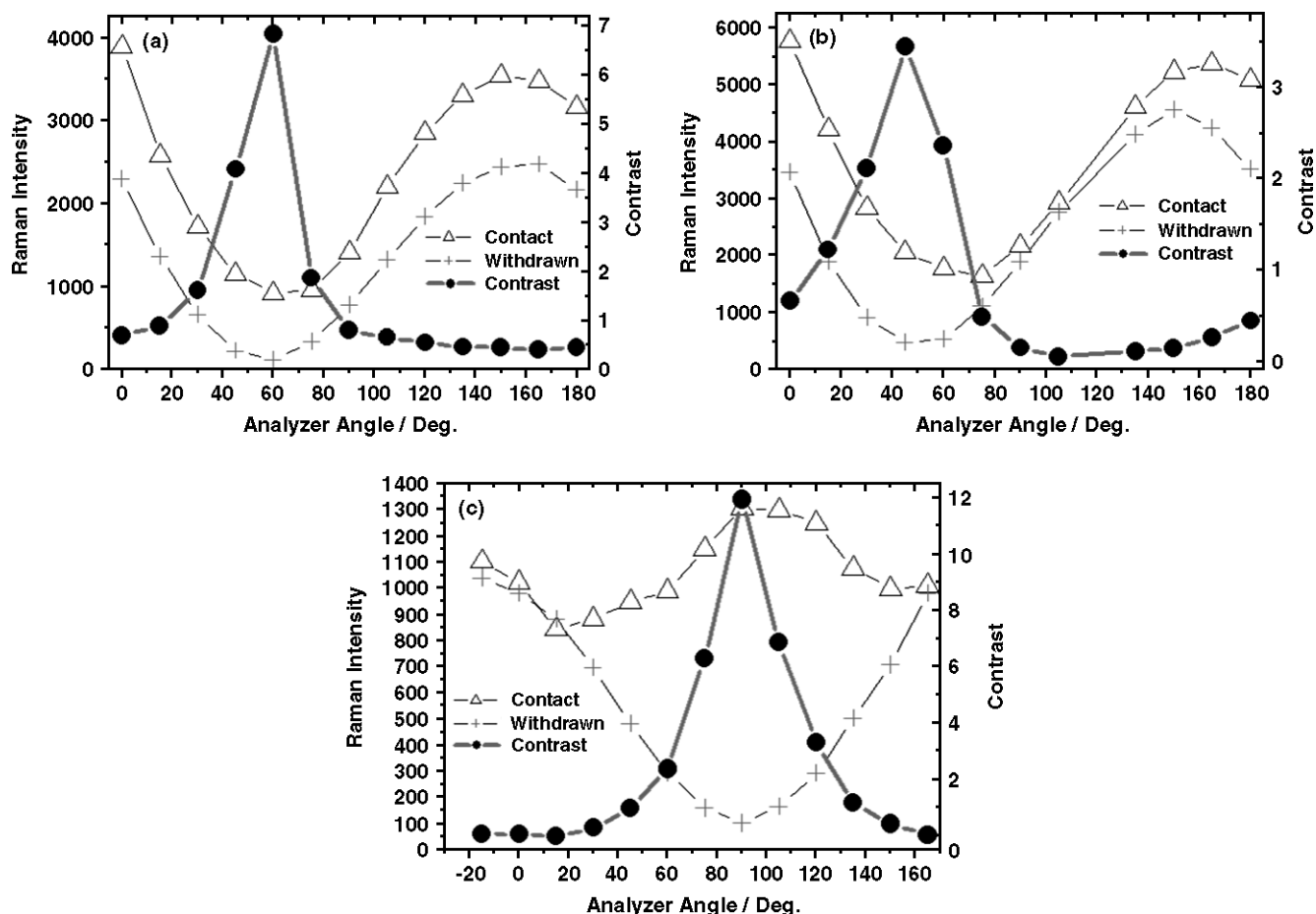


Figure 4. Contrast dependence on the analyzer angle for three different incident beam polarizations with respect to the metallized tip axis: (a) -20° (b) 25° (c) 70° .

that I_{near} remains essentially constant and the contrast is maximized at 70° polarization angle. In fact, the contrast for incident polarization 70° is seven times higher than that for -20° polarization. It is generally accepted that better field enhancement may be obtained by placing the polarization of the incident beam along the tip axis (p -polarization) than by placing the polarization perpendicular to the tip axis (s -polarization).^{31–33} Rotating the polarization (70°) relative to the tip axis does incur a loss in enhancement, but this loss of enhancement is more than outweighed by the gain in contrast due to effective suppression of the far-field signal. The morphology and geometry of the tip end may also change the optimum polarization conditions, and details of the relationship between tip structure and polarization behavior are being investigated with well-defined tips. Even greater contrast can be achieved by positioning a polarization analyzer before the spectrometer. The graphs in Fig. 4 show how the contrast varies with the analyzer angle for three different incident polarization angles. Selecting the optimum polarizer angle, 70° , denoted as 'P 70' hereafter, and analyzer angle, 90° , denoted as 'A 90' hereafter, increased the contrast more than 20 times, to a value of 12 (Fig. 5). One possible reason for the polarization dependent suppression of the contact and withdrawn signals is that the tip can depolarize the incident and scattered light. If so, the near-field contribution included in the contact signal may be depolarized more than is the far-field signal.

If this enormously improved contrast (from 0.5 to 12) is indeed due to the near-field signal, nano-Raman analysis of Si may be a reality. However, the contact signal may include a component that is unrelated to the plasmon resonance enhancement due to reflection and scattering from the tip. The tips used for TERS measurement were commercially available silicon nitride tips coated with silver or gold. These tips have a four-sided pyramidal shape. If the incident laser beam is normal to the front surface of the pyramidal tip, the reflection will be greater than if the beam is incident on a

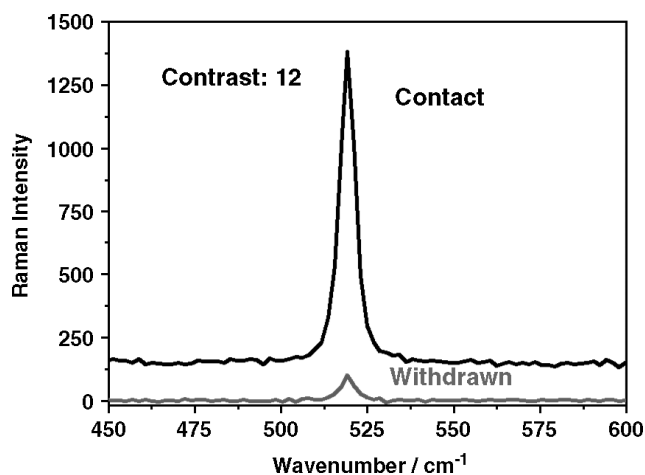


Figure 5. Si Raman contact and withdraw signals at P70 and A90 and a gold-coated tip.

pyramidal edge. Figure 6 shows TERS intensities collected with the incident beam oriented in two different directions with respect to a pyramidal face of the AFM tip. The contact intensities are sharply different, indicating that the contact signal includes some additional Raman signal that is not related to the enhanced near-field signal, but rather to the illumination direction. We believe that the front face of the pyramidal tip acts as a mirror, increasing the illumination intensity, and leading to additional unlocalized Raman signal. To minimize the contribution of unlocalized signal in the contact signal, further TERS and SNRS measurements were performed with the incident beam 35° off-normal to the front surface of the pyramidal tip.

In our contact mode SNRS system, three experiments were performed to determine the scale of localization of the contact signal that includes the localized near-field signal. The first experiment with various CdS films demonstrates²² a detection depth of ~ 20 nm, a length scale

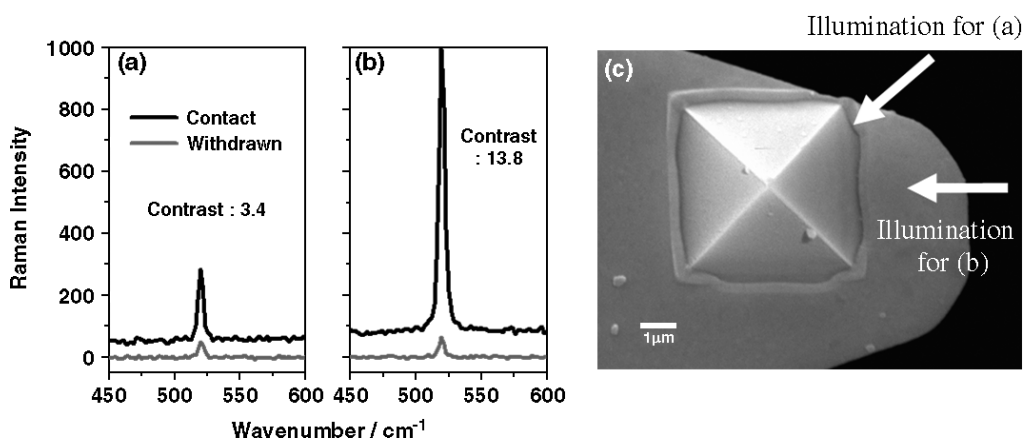


Figure 6. Comparison of TERS intensities achieved with the incident beam (a) off-normal ($\sim 35^\circ$) and (b) normal to the front pyramidal surface of a silver-coated tip under optimized conditions of polarization (P70A90). (c) SEM image of a 50 nm silver-coated silicon nitride tip and the illumination directions for (a) and (b).

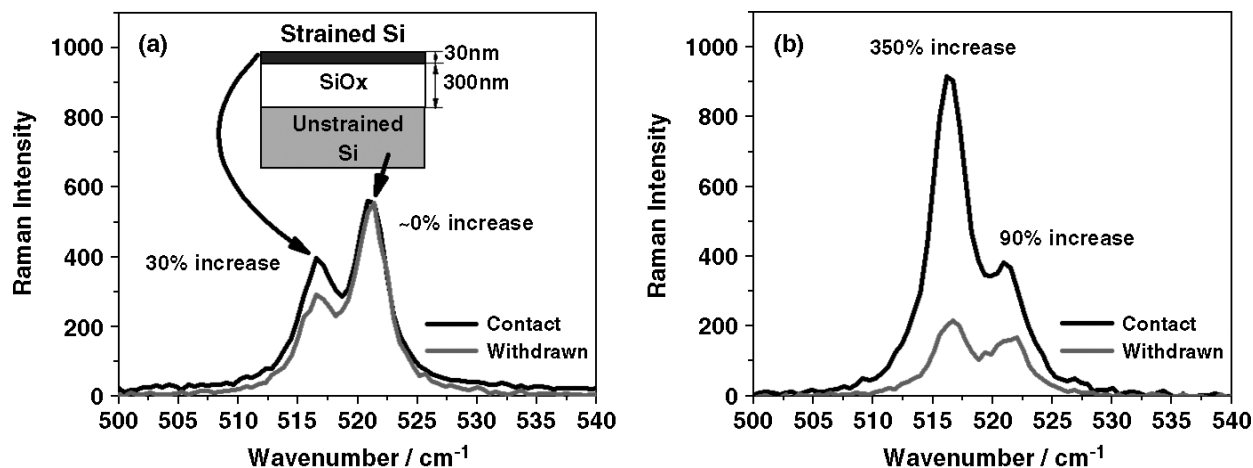


Figure 7. TERS spectra for Si from a strained Si film structure using a gold-coated tip. (a) Contact and withdrawn spectra at polarization and schematic of the structure in the inset. (b) Contact and withdrawn spectra at P70.

similar to that found from measurements of contrast vs tip-sample distance.³⁴ The second experiment to investigate the localization of the depolarized signal involved 30 nm-thick strained Si structures on a 300 nm SiO_x layer on Si substrate (Fig. 7). First of all, the measurements with regular polarization showed enhancement only for the strained Si layer. No enhancement was observed for the unstrained Si (Fig. 7(a)). Using the optimized polarization, we improved the contrast for the Raman signal from the strained Si: the contrast increased to 350% for strained Si while the signal from unstrained Si increased by only 90% (Fig. 7(b)). This result clearly indicates that an increase in Raman signal from optimized polarization is mostly localized at the surface. However, it also suggests that some extra signal exists from the silicon substrate positioned below the 300 nm silicon oxide layer that cannot be related to the surface plasmon resonance. Even when off-normal illumination to the front surface of the tip was used, the contact signal still included

the contribution of the unlocalized signal. The mechanism responsible for the increase of the signal from the underlying Si layer is not known. We speculate that polarization-dependent reflection or interference of illumination at the two interfaces of silicon oxide, and, in particular, at the tip-surface interface, may contribute to the additional Si signal.

In a third test for localization of the depolarized TERS signal we studied periodic SiO_x lines (~250 nm wide and 30 nm in height) on Si, as depicted schematically in Fig. 8(a). Figure 8(b) exhibits a plot of the topography signal and contact Si Raman signal integrated over the range 510–530 cm⁻¹, collected for a single line scan perpendicular to the direction of the SiO_x features. In Fig. 8(c) the Raman signal is inverted and its scale renormalized so that a direct comparison with the topographic signal can be made. The qualitative agreement is excellent. Figure 9 compares Raman scans obtained with two different

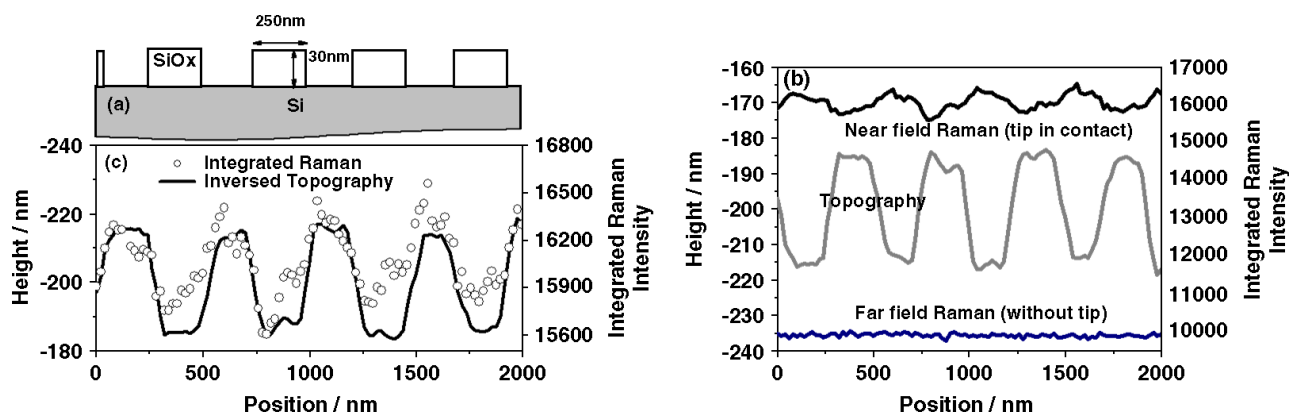


Figure 8. (a) Schematic of the Si/SiO_x structures, (b) line scans for the integrated near-field from a silver-coated tip and the far-field Raman intensities at P70A90 compared with the line scan for the topography from AFM, and (c) correlation between the inverted topography line scan (solid line) and integrated Raman intensity line scan (○). This figure is available in colour online at www.interscience.wiley.com/journal/jrs.

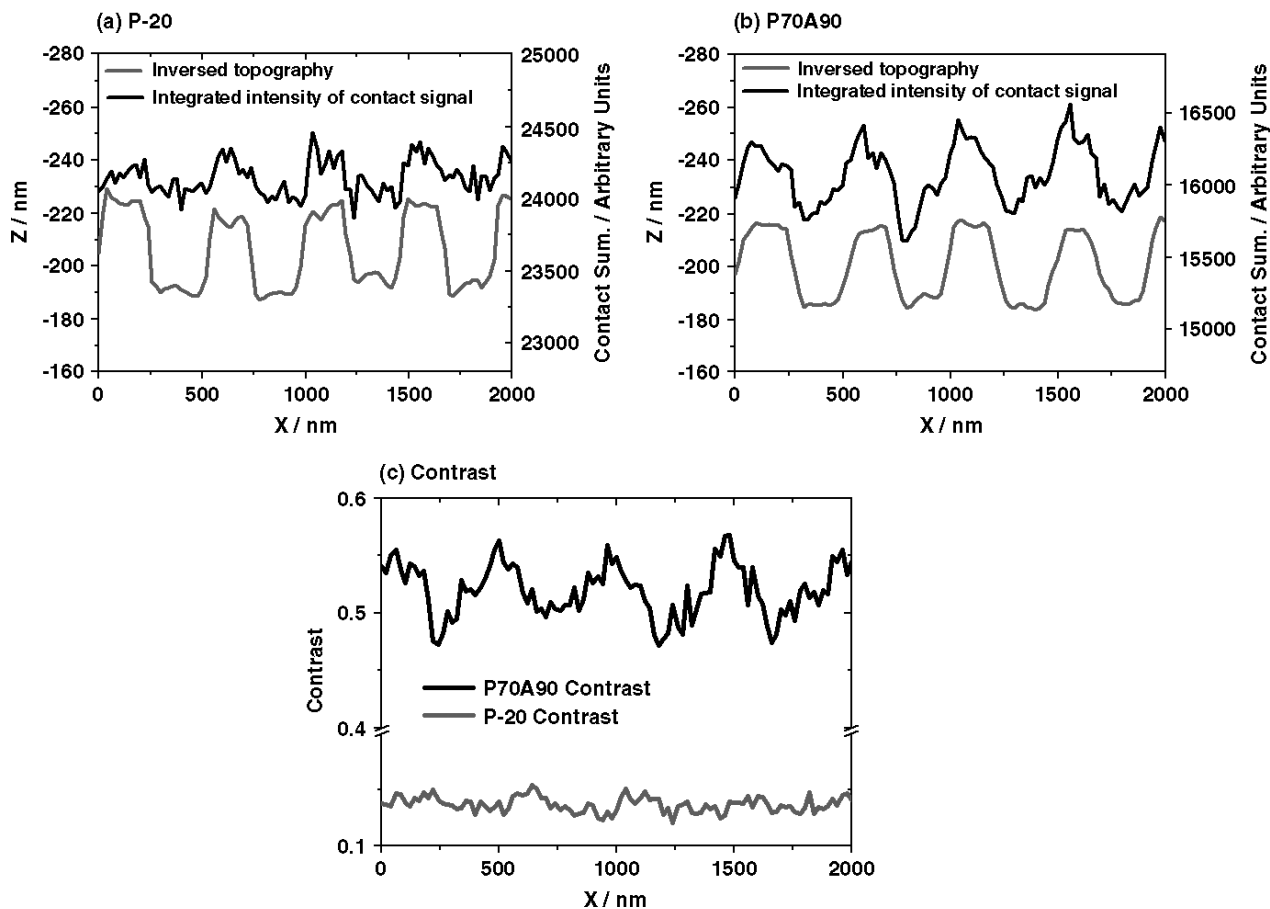


Figure 9. Line scans of Raman signal and topographic signal for the Si/SiO_x structure pictured in Fig. 8, measured for different values of polarization (a) P-20, (b) P70A90. The plot in (c) of contrast for the two polarizations suggests that the lateral localization is much better for P70A90 polarization. A silver-coated silicon nitride tip oriented $\sim 35^\circ$ to the incident beam was used.

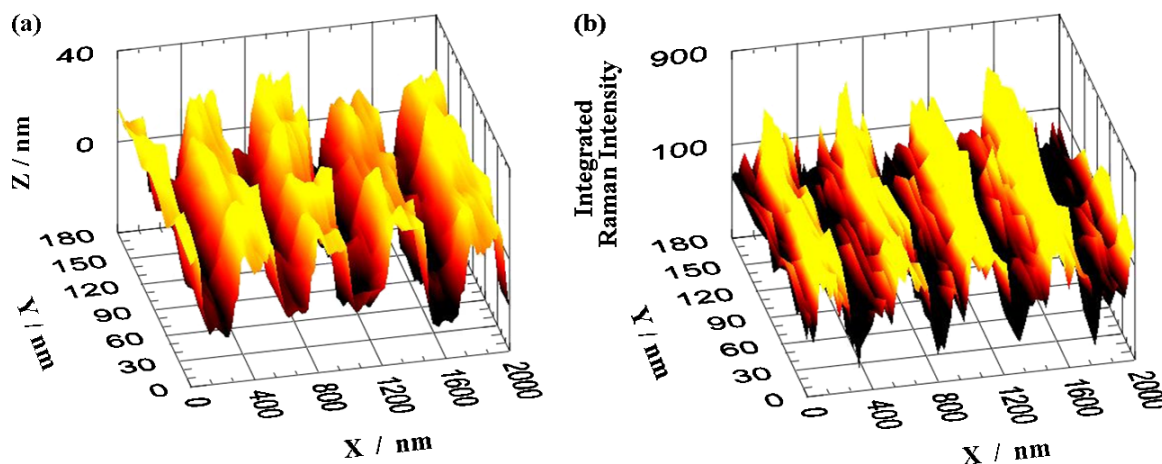


Figure 10. Three-dimensional scanning images (100×10 pixels) of (a) topography and (b) integrated Raman signal (2 s accumulation time per pixel) for the sample described in Fig. 8. This figure is available in colour online at www.interscience.wiley.com/journal/jrs.

polarization conditions. The use of optimized polarization clearly improves the contrast of the Raman image (Fig. 9(c)). Figure 10 shows a nano-Raman-scanned three-dimensional image obtained with optimized polarization. It

demonstrates a good agreement between Raman (chemical) and AFM (topographic) images. The correlation presented in Figs 8(c) and 9(b) illustrates that our tip-enhanced Raman resolution is comparable to our topographical resolution.

We estimated the line widths in both images and they appear to be the same. So, the resolution with optimized polarization has been estimated to be about the step size, ~ 20 nm.

CONCLUSIONS

Optimizing the polarization conditions can improve the contrast for TERS imaging of Si-based structures by about one order of magnitude. This improved contrast should be sufficient for nano-scale Raman analysis if the increase in signal is on account of near-field enhancement caused by plasmon resonance at the apex of the tip. However, our preliminary results suggest that only a portion of the depolarized contact TERS signal is, in fact, localized. There are other (nonlocalized) contributions to the depolarized TERS signal. Nonetheless, this localized component of the contact signal is sufficient for successful imaging of Si-based structures with a lateral resolution of about 20 nm.

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