

Near-field scanning optical microscopy and spectroscopy for semiconductor characterization.

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Abstract

The applicability of near-field scanning optical microscopy (NSOM) for optical characterization of semiconductors is discussed. The NSOM technique and some of its properties relevant to real-time in-situ measurements are reviewed. Several optical characterization methods widely used in the far-field, including reflectance, reflectance-difference spectroscopy, Raman spectroscopy, ellipsometry, and carrier lifetime, are evaluated for their use with NSOM. Experimental data are included for some of these methods. We conclude that several, but not all, of the standard optical characterization methods can be coupled with NSOM to provide higher spatial resolution. The applicability of NSOM as a real-time in-situ probe shares some of the problems of other proximal probe methods, but offers enough new capabilities to warrant its application.

Introduction

Powerful methods exist for the optical characterization of semiconductor materials and devices. But the data obtained can sometimes be difficult to interpret, especially when the sample is inhomogeneous on a mesoscopic scale. In such cases one must search for an alternative technique with higher spatial resolution. Near-field scanning optical microscopy (NSOM) offers the possibility of performing several of these optical techniques with only slight modification, but substantially higher spatial resolution. The application of NSOM does entail some subtleties, however. The purpose of this paper is to review NSOM as it relates to semiconductor characterization, and to point out some features which differ from the far-field counterparts.

The NSOM Technique

The NSOM technique increases spatial resolution of optical measurements by using geometry to confine the optical field. An aperture is placed in close proximity to the sample. This aperture can be used to limit the extent of the input light, i.e., providing a small light bulb, or as a spatial filter for detection. We will consider the former case in this paper. In this section, we will first consider the aperture, then describe the method used to keep it properly positioned near the sample as it is scanned by a piezoelectric transducer (usually a segmented tube). Data are acquired for an array of points during the scanning, and are used to construct an image in a computer. We will not discuss methods of coarsely positioning the probe or of other features of the head design, as these are covered in many books on scanning probe microscopes.

The aperture is the heart of the NSOM instrument. The aperture is made by tapering the end of an optical fiber, and coating the sides with metal a few penetration depths thick. If the taper is produced by heating the fiber and pulling[1] until the fiber breaks, a flat cleave is produced at the tip[2]. Such a tip is shown in figure 1. The sharp corner shown here is clearly evident in the figure. This helps in the definition of the aperture, which can be produced by tilting the flat tip away from the evaporation source so that it is left uncoated while rotation is

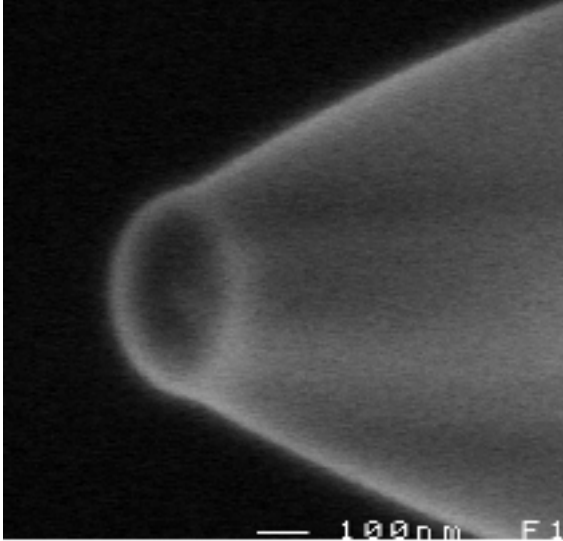


Figure 1. A scanning electron micrograph of an uncoated tapered optical fiber is shown. The fairly large (~ 300 nm) cleave at the end was chosen to illustrate the sharp edges. These help define the aperture at the tip when the sides are coated with metal.

used to apply metal to all sides. In practice, surface diffusion reduces the aperture size from the size of the flattened tip. The minimum aperture size is limited by the optical penetration depth of light into the metal to $>\sim 10$ nm. Aluminum has the best figure of merit in this respect[3], and is almost universally used. One important point is that the tip should not be smaller than that which the required resolution necessitates. The reason is that the optical throughput decreases strongly with aperture size "a". If the aperture were an infinite perfectly conducting plane with a round hole in it, this dependence would scale as $(a/\lambda)^6$ for wavelength λ and aperture size a [4]. A more applicable geometry in an experiment in the microwave regime suggest a dependence of $(a/\lambda)^4$ [5],[6]. We will not discuss it in detail here, but note that the throughput depends on a number of factors such as tip shape, size, and the modal occupation within the fiber (i.e. twisting the fiber can change it). It is not a well-controlled parameter.

To yield high resolution images, the probe must be held in the near-field of the sample. Light quickly diffracts after leaving the aperture. The

width of the optical field can be roughly estimated as $a+\pi z$ for aperture size a and axial distance z. The distance regulation method must be independent of the optical method for maximum utility, and be capable of feedback ~ 10 nm or less from the sample surface. Lateral shear force microscopy has been found to be an excellent choice, as it can be used on a very wide range of samples in many environments, from ambient to superfluid helium, from vacuum to within solution. The technique utilizes a resonant bending vibration of the probe[7, 8]. Figure 2(a) shows an amplitude-frequency curve from which the resonant frequency and quality (Q) factor is found. Typically, resonant frequencies of ~ 15 -80 kHz are used, with Q's up to several hundred. For a specific example, one obtains a ~ 50 kHz resonant frequency for ~ 2 mm of fiber and tip extending beyond the fiber mount, which corresponds well to simple non-uniform bending beam calculations [9]. The vibration amplitude at the free-space resonant frequency decreases as the sample is approached. This is shown in figure 2(b) and provides the feedback signal. Note the distance scale over which the signal decreases is a few 10's of nm, as is required. The feedback loop request is set to some position on this curve, and a voltage applied to a piezoelectric material to close the loop. The voltage required is recorded at each scan point within a rectangular array, and is used to create a topographic image simultaneously with the optical data.

The specific types of optical measurements which can be performed include most of those available in the far-field. It has been operated in several contrast modes including polarization in both transmission [10] and reflection [11], magnetic [12], spectroscopic [13], and fluorescence [14]. Specific examples will be discussed in more detail below.

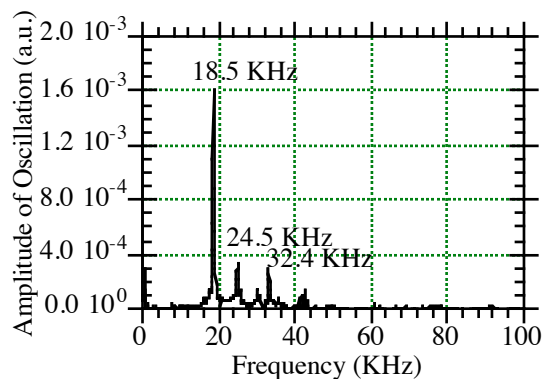
Scanning Proximal Probe Microscope Considerations

Scanning proximal probes share many features in common which are relevant to their applicability for real-time in-situ studies. Since NSOM is a member of the scanning probe family, we present this section as an orientation so that the reader will understand the motivations for particular choices of probes and design parameters. Note that we wish to give as accurate assessment as is possible, so that although some of the comments below may seem

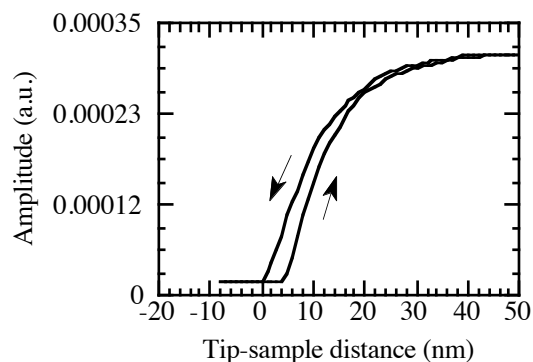
harsh, proximal probes can contribute unique information about growth processes, so that the surmounting of these hurdles is a worthwhile venture.

Real-time or Interrupted Growth?

One ideally would like to observe a growth process as it occurs. This requires several abilities not always found in probe microscopes. The first consideration is imaging speed. An image must be acquired in a time short compared to the time it takes to grow a pre-defined fraction of one monolayer. This can be accomplished if the growth rate is not too rapid, but limits the number of probes to those with sufficient signal to noise using very short averaging times. Another, more serious, problem is that the probe may affect the growth, either by shadowing the region adjacent to it on the surface, modifying surface diffusion, etc. This may or may not be easy to detect. The probe may be retracted for a short interval of time and the resulting growth compared to that while the probe was near the surface. More likely, comparisons to interrupted growth will be required. Interrupted growth has problems of its own. The surface may relax before the probe images it, or the interrupted surface may differ from the surface during growth because the vapor above the surface has changed. To minimize these possible artifacts, we believe the probe microscope is best operated in the environment present during growth. These methods may require the possibility of a slowed growth while imaging (it may be slowed by the presence of the probe anyway) to minimize signal constraints.



(a)



(b)

Probes Image a Small Fraction of the Sample

Probe microscopes have very high magnification, hence they image small areas. These regions range from submicron dimensions up to hundreds of microns. Some mechanism must be found to identify that this area is typical of the sample, especially given the considerations in the last paragraph. We suggest the use of an instrument with two different methods to test this. The first is large scale motion of the tip. Images can be acquired at several widely spaced locations. If they agree, one can assume they are typical of the sample. The second is to use the probe to measure a property which can also be measured by another probe which averages over large regions of the sample. One then obtains the large picture and a magnified view to compare. Since far-field optical techniques are easily implemented in a real-time system, NSOM is particularly easy to implement in this way.

Figure 2. (a) The amplitude of lateral tip vibration is shown as a function of frequency. The tip resonant frequency is shown at 18.5 kHz as are some of the driving piezo resonances, which only vary slightly with disparate tips. (b) An approach curve, or tip vibration amplitude as a function of the tip-sample separation is shown. This sets the scale of the axial positioning.

In-Situ Head Designs

Special considerations must be accounted for if a probe microscope is to operate while in a growth environment. A coarse mechanism to provide the opportunity to scan disparate regions of the surface must be devised. Little other modification is usually required for operation in an ultra-high vacuum environment, but significant modification is required for operating in a gas-deposition system such as CVD, MOCVD, etc. It is important to solve these problems, however, since most semiconductor material growth in industry is or will be by the use of vapor deposition systems. Probes can be modified for this environment, whereas it severely restricts some types of electron beam measurements. The major problems for the probe microscope are deposition on the instrument and problems related to the high voltage needed to drive the piezoelectric scanner. Shielding the instrument reduces the former. The latter stem from arcs or plasma discharges which are likely for pressures from $\sim 10^{-4}$ torr to several hundred torr. The piezo should either be near atmospheric pressure or in a high vacuum. Unfortunately, growth in a vapor deposition chamber is usually in neither of these limits. One solution is to place the scanning mechanism and certain other parts of the microscope in a sealed container, which is pressurized to near atmospheric pressure. The other alternative -- to differentially pump the region to a low enough pressure, is also possible with an identical head design.

Probe Artifacts

Any proximal probe is susceptible to problems due to interactions of the probe with the sample or artifacts from a damaged probe. The former must be considered for each type of probe and sample situation. The latter are eliminated by using a variety of contrast modes which are related enough that their correlation can be used as an indication of probe reliability.

Although this section on probe considerations applies to all types of proximal probe microscopes, we specialize for the rest of this subsection to the NSOM case, which is of more interest here. NSOM data consists of a topographic image and at least one optical image. Several optical images utilizing different contrast mechanisms can help identify artifacts. In this sense, NSOM provides more diagnostic abilities than most other probe microscopies. It is also interesting to note that damaged probes can produce reproducible images, but with limited usefulness. Probe crashes can produce damage, so must be avoided. Bad NSOM tip crashes will increase the aperture size and so the user will note an increase in the optical signal. This increase signals trouble. Less drastic crashes can damage the metal coating which can hurt resolution, modify the polarization properties of the light emitted by the tip, and cause the force feedback system to exhibit strange behavior.

Resolution and Signal Levels

It is useful at this point to consider what kinds of simultaneous temporal, spatial, and spectroscopic resolution that can be achieved. The energy-time uncertainty principle limits the combined temporal and spectral resolution, and can be reached in some ultrafast studies. Spatial resolution does not present such a hard or straightforward barrier. Rather the signal intensity decreases rapidly as a function of the resolution (aperture size) divided by the wavelength, as we have discussed before. One must then take into account statistical and electronic noise, for the signal and background, to determine if a signal can be identified within some time duration, i.e., we must define an averaging time Δt_{ave} . The maximum Δt_{ave} may be limited by either the desirable time sampling interval (e.g., in video-rate imaging) or by the instrument stability,

whichever is more restrictive. Recall that for imaging applications, the measurement must be repeated at each point in the image (usually a minimum of several thousand) while the microscope drifts less than the resolution. The choice of Δt_{ave} depends upon the probe in terms of the number of photons/second it delivers (depends upon spatial resolution), the experimental cross section for the process under study, the collection efficiency, the time interval (time resolution), and the wavelength interval (spectral resolution). These considerations can be used to estimate the signal levels required and/or measurement modes possible for a given growth process.

Possible Peculiarities of Proximally-Performed Optical Semiconductor Characterization

We have seen several optical techniques applicable to the characterization of semiconductors within this volume. Many of these techniques can be adapted for use with NSOM. They will be discussed qualitatively below, with examples from our laboratory inserted where applicable.

Reflectance Measurements

Reflectance can be measured in several schemes depending upon whether the light is incident, collected, or both with passage through the probe tip. The first thing to note is that the intensity of light emanating from the aperture is not a well-controlled parameter, which can complicate quantitative analysis. The throughput fraction depends upon the particular probe in use, the coupling of light into the fiber (the mode density produced), and the way the fiber is twisted and bent. One might think that these problems can be overcome by measuring the light output from the fiber without a sample in place, then comparing to those with a sample in the near-field. This method is not exact, since evanescent light can couple from the tip to the sample, thus changing the effective input intensity.

The choice of measurement geometry also involves several considerations. If the light is coupled through the probe in both directions, the reflected light will have a very large background comprised of light reflected from the inside of the tip. This can be avoided by collecting the light reflected outside the probe in the far-field or by oscillating the tip vertically and detecting in phase [11]. The latter complicates the measurement and is not often used. The former introduces a new complication -- the light must make its way around the probe. Although only crucially important for metallic samples for which the close proximity of the metal coating on the tip and the sample produce a narrow wave guide, it nevertheless can effect imaging on other samples. Shadowing effects can occur if reflected light is collected to one side of the sample/probe by inserting a lens from the side. We minimize these effects in our lab by mounting the probe in a hole drilled through a few-mm-focal-length, high-numerical-aperture lens. It allows symmetric collection of a large solid angle of light.

Reflectance Comparison Methods

In these spectroscopies, the reflectance R of two surfaces are compared. The surfaces could have different surface preparations as in differential reflection spectroscopy, be from different areas of the surface (especially with NSOM), or be the same sample area with the comparison between two polarizations of the input light as in reflectance-difference spectroscopy (RDS). The latter probably is most suitable for NSOM. The far-field measurement technique gathers $\Delta R/R$ as a function of photon energy. NSOM is an imaging technique which could enable one to gather the same $\Delta R/R$ as a function of photon energy at every point in the two

dimensional spatial image. The 3-d data set could be analyzed to yield, for example, the magnitude or energy of a dimer absorption as a function of position. Alternatively, it may be desired to collect less data at each point to speed imaging. In that case one may choose to image $\Delta R/R$ at only one or a few energies for each point, and sweep energy only at a few points.

Polarization is maintained through the NSOM probe tip if it is symmetric. One can control the polarization by rotating the polarization of the light before coupling to the fiber. If short lengths ($< \sim 0.5\text{m}$) of optical fiber are used for the probes, then it appears not to matter what type of fiber is used. One does not need to use polarization preserving fiber, although it appears to cause no harm and probably will be necessary if long lengths of fiber are used. The quality of the metallic coating at the end of the tip is much more important. A damaged or improperly manufactured probe tip may pass elliptically polarized light and/or it may not be possible to access all directions of linearly polarized light at the output of the probe. Problems in probe manufacture can be related to metal grain sizes which are too large. This can create a bouldered or crown like appearance when observed with a scanning electron microscope. The lack of symmetry changes the output at different linear polarizations. A crashed tip can also lose symmetry. One must be extra careful, as the tip can be damaged for polarization purposes before the light intensity increases enough to give the user the usual indication that it is time to change tips. There is one other detail of using polarization with NSOM. It is that, due to the metal at the tip, a single linear polarized state is only an approximation. The polarization in the lateral x and y directions can be quite independent, but there is usually a small amount of light polarized in the axial (z) direction! This is observed experimentally when imaging the fluorescence of single molecules [15] -- which really corresponds to using the molecular dipole to act as a point and image the field from the probe tip. The axially polarized light drops rapidly in intensity as the tip is retracted from the surface, a fact which can be used to gain insights into the polarization properties of a surface in all 3 directions. The polarization, including that in the axial direction, is modeled well by the early theory of Bethe [4, 15].

Since the wavelength is usually varied in this experiment, we must consider the wavelength dependence of the probe. As mentioned above, the resolution is independent of the wavelength of the light, being determined by the aperture size if good distance regulation is used. The intensity of the light will depend upon the wavelength. The simplest dependence comes from the transmission characteristics of the fiber, which can be quite good over the entire optical range for the \sim meter lengths of fiber used. There are other sources of wavelength dependence. One of these stems from the coupling of light through the small aperture. As mentioned above, the amount of light which passes through the aperture is a strong function of the wavelength and aperture size: $(a/\lambda)^4$ or 6 . Another, less obvious, dependence results from the modal properties of light traveling down an optical fiber. These modes travel in a dielectric waveguide until they are most of the way down the taper at the probe end of the fiber. By then, the waveguide is so small that the mode has become spatially large, and the metal coating becomes important in guiding the light. Cylindrical metal waveguides do not support any modes when the diameter of the waveguide is below a certain critical size called the cut-off diameter. From that point to the end of the probe, the light is evanescent, so the coupling efficiency falls exponentially. The cut-off diameter depends upon the wavelength of the light and the mode of the light. The lowest order mode is cut-off at a diameter of $0.293\lambda/n$, for n the index of the glass. The mode distribution will depend upon the distribution input to the fiber and any redistribution which may occur. Since these quantities are difficult to calculate, it will probably be most convenient to measure the wavelength dependence in the far-field with no sample in place. This may not give an exact

calibration due to evanescent coupling to the sample once it is brought into the near-field of the probe, but one must await theoretical efforts to determine if these effects have a strong wavelength dependence.

The choice of optical fiber from which to fabricate the probe is also important. Fiber can be broadly divided into two types: multi-mode and single mode (at a particular wavelength). Obviously, if a single mode fiber is chosen, then the wavelengths used should be shorter than that for which the fiber is single mode. Otherwise the light will be strongly attenuated. But the fiber will be few-mode for a range of wavelengths shorter than that for which it was designed as single mode. The number of modes present can be estimated from the modal volume as can be found in standard optics texts [16]. The problem with using few-mode fibers is that the mode distribution is notoriously environmentally dependent. This is in fact the basis for several fiber optics sensors (e.g. temperature, flexing). But for NSOM it is not good since the intensity of light output from the probe depends upon the modal distribution in the fiber. Environmentally sensitive fibers can make a very noisy NSOM signal even before a sample is inserted. Multi-mode fibers offer such a high density of modes that the distribution is not changed in any important fashion by environmental effects. One may be slightly concerned that the overall throughput may be lower for these fibers in comparison with a similarly-shaped single mode fiber [17].

Raman Spectroscopy

The signal level from spontaneous Raman spectroscopy is small. Therefore real-time measurements with Raman spectroscopy are difficult. This problem is compounded with NSOM as the light intensity is small and decreases strongly as the aperture size is reduced to improve spatial resolution. The small Raman cross section and the dispersion of the spectrometer reduce the intensity at a given wavelength to very small levels. Although Raman spectroscopy is possible with NSOM, as is shown in the spectra of figure 3 taken in our laboratory as well as in Raman images not shown, the averaging times of several hours required prohibit its use in real time. It may be possible during interrupted growth if the surfaces are stable.

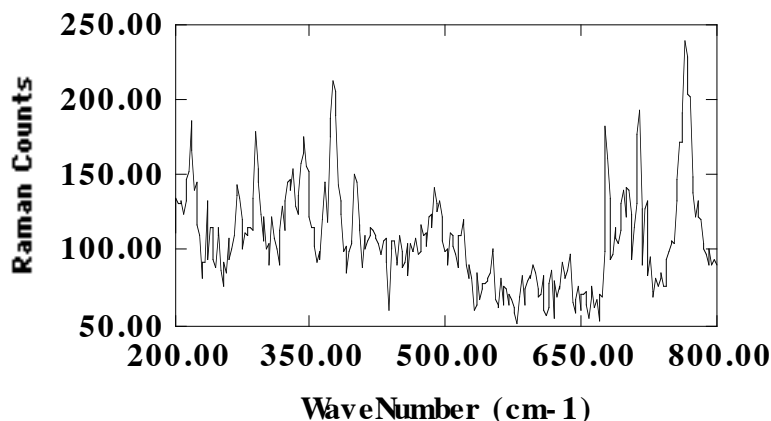


Figure 3. A nano-Raman spectra taken from part of a small Rubidium doped region of a KTP (KTiO_2PO_4) sample. The acquisition time for the spectra was approximately 2 hours. Few-hundred nanometer resolution was verified by comparing to the thermal drift of topographic scans before and after and by other Raman images.

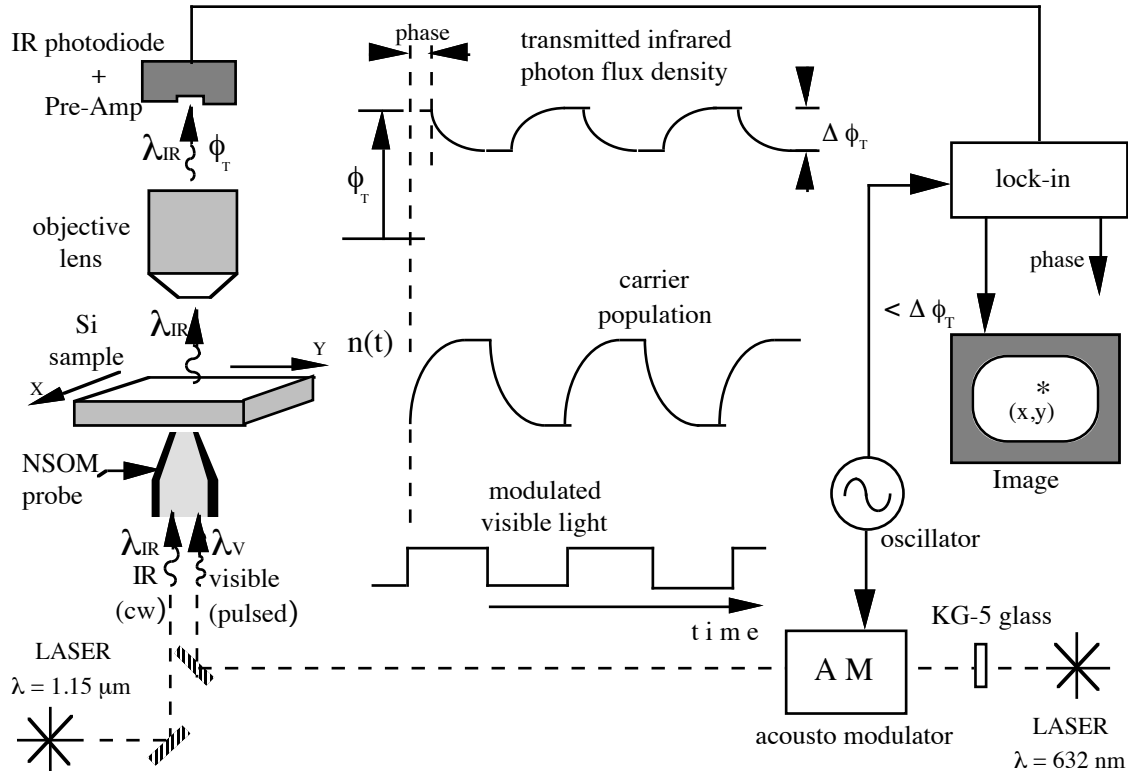


Figure 4. Schematic of the process to extract information of the local dynamics of excess carriers in a semiconductor. Left side : optical path of the IR radiation. Center: A timing diagram of the process. Right : Synchronous detection of the transmitted infrared signal. An image is constructed by repeating this process as the probe is scanned across the sample.

There are some interesting properties of the nano-Raman spectra we have observed to date. The first is that there appears to be an enhancement in the magnitude of the spectra as the tip is brought into the near field. This is probably due to the coupling of evanescent light or the field enhancement which occurs near the metal of the probe coating. Another interesting observation is that the nano-Raman spectra differ slightly from micro-Raman (far-field) spectra taken on the same sample, even though the nano-Raman spectra taken at different parts of this non-uniform sample are consistent with one another, showing only differences relating to the sample non-uniformity. These interesting results will be the subject of a future publication [18].

Ellipsometry or Spectroscopic Ellipsometry

Since polarization can be controlled in the NSOM fiber probe, one might expect that ellipsometry would be possible. However, ellipsometry also requires control of the angles of incidence and reflection. This is not possible with NSOM. The illuminating light diffracts quickly as it leaves the aperture, spreading in all directions [19]. Naively, one might think that this could be remedied by controlling the detection angle and assuming specular reflection from the sample. The small NSOM spot size and the resulting diffraction make this simplistic picture untenable. Ellipsometry will not be possible with NSOM in a form similar to that performed in the far-field.

Carrier Lifetime

We have used NSOM to detect inhomogeneities of the dynamics of excess carriers in oxidized silicon wafers [20, 21]. NSOM was used to improve the spatial resolution of a standard IR-scattering optical technique, which is carried out in a non contact fashion. Continuous wave infrared light is used as a detector of the time dependent carrier population produced by a pulsed visible laser. Specifically, as seen in figure 4, visible laser light is modulated to create a time-varying excess carrier distribution. This distribution is monitored by noting its effect on continuous wave infrared (IR) laser radiation also illuminating the sample. In the experiment reported here, both types of light are input through the same NSOM probe aperture. This configuration insures high lateral resolution, and avoids mismatch in the sample regions illuminated by either laser light.

The dominant source of the contrast observed in our images comes from the time-dependent number of carriers available to scatter IR radiation. We found it convenient for imaging purposes to detect the IR signal synchronously with the visible light pulsing, using a lock-in amplifier. This reduces the computational load during acquisition and improves the signal to noise ratio. A set of images is shown in figure 5. The figure shows a topographic image, recorded from the feedback used to maintain the probe in the near field, an infrared transmission image which was taken as a diagnostic, and the image which results from differences in time response of the sample. The excess carrier lifetime is found to be smaller near defects as would be expected. Note that the images use a gray scale range which does not extend all the way to zero. There is signal on all parts of both (b) and (c). Also, the contrast on an absolute scale in the transmission image (b) is actually very small, since the background level is ~ 80 nW. The background on (c) is sub-nW.

There are some interesting aspects of the NSOM measurement of carrier lifetime which have already been discussed in the literature [21]. They stem from probe heating by the visible light, which gives a small background to be considered when choosing an operating frequency,

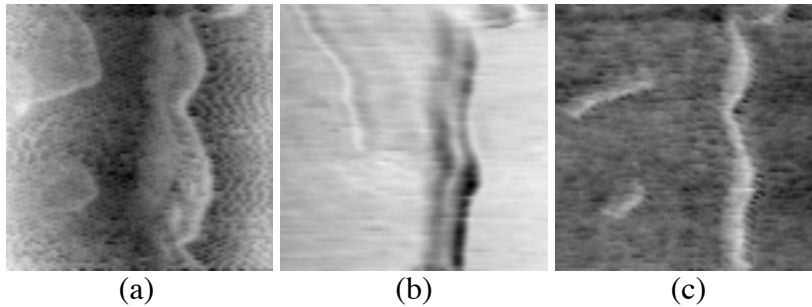


Figure 5: Recent results in our time resolved carrier dynamics work are shown. All three images show the same 20 micron square region of an oxygen terminated silicon surface. (a) the topography is shown with a 100 nm vertical range, with white higher. Note that due to multiple step defects and the plane subtraction, the terraces between seem tilted. (b) The infrared transmission (0.44 nW range) shows regions of contrast, white more intense, near some of the multiple step defects and on some terrace areas. (c) The time-resolved image, of IR amplitude change while the visible light is switched, reveals regions near part of the multiple step defects which have a faster recombination rate (white). The range is 0.038 nW.

and from the effects of diffusion on the interpretation of the data. The latter suggest that the effective resolution will be higher near the fast recombination centers, and that the experimental measurements have surpassed the current state of theoretical models. It is also interesting that different operating (visible switching) frequencies can give different images [22] as is understood by observing the magnitude of IR signal variation as a function of visible light switching frequency. Slower time constant processes dominate at low frequencies,

whereas faster processes become more important at higher frequencies. From a plot of IR variation amplitude as a function of frequency, one can locate the frequency at which the signal sharply drops. This marks where the slower process can no longer keep up with the visible light variation. One can estimate from this frequency the time constant of the process -- here the lifetime of the excess carriers. We have observed samples with lifetime constants measured by other methods to be as different as 10 μ s and 1.6 ms, but have found the same corresponding lifetimes at local regions of the samples with our technique.

Conclusions

Near-field scanning optical microscopy (NSOM) has been found to be applicable for optical characterization of semiconductors is discussed. For optimum use, some properties peculiar to the NSOM technique must be thoroughly understood. Appropriate tests and procedures should be followed. Of the several optical characterization methods widely used in the far-field which were discussed here: reflectance, reflectance-difference spectroscopy, Raman spectroscopy, ellipsometry, and carrier lifetime, most, but not all, can be coupled with NSOM to provide higher spatial resolution. The applicability of NSOM as a real-time in-situ probe shares some of the problems of other proximal probe methods as discussed in detail above, but offers enough new capabilities to warrant its application.

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