Demonstrating Applications of Non-optically Regulated Tapping-Mode Near-Field Scanning Optical Microscopy to Nano-optical Metrology and Optical Characterization of Semiconductors

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We demonstrate the applications of a near-field scanning optical microscopy (NSOM) system based on a short-probe tapping-mode tuning fork (TMTF) configuration to nano-optical metrology and the optical characterization of semiconductors. The short-probe TMTF–NSOM system is constructed to operate in both collection and excitation modes, in which a cleaved short fiber probe attached to one tine of the tuning fork is used as a light collector/emitter as well as a force-sensing element. Interference fringes due to standing evanescent waves generated by total internal reflection are imaged in the collection mode. Excitation-mode short-probe TMTF–NSOM is applied to near-field surface photovoltage measurement on distributed-Bragg-reflector-enhanced absorbing substrate AlGaInP light-emitting diode structures. [DOI: 10.1143/JJAP.45.2187]

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1. Introduction

Near-field scanning optical microscopy (NSOM) is one type of the scanning probe microscopic techniques that enables local optical and optoelectronic measurements beyond the diffraction limit. Being imposed by the wave nature of light, the spatial resolution of a conventional standard optical microscope is determined by the Rayleigh criterion $0.61\lambda/NA$, where λ represents the wavelength of light and NA is the numerical aperture of the microscope. For this reason, the spatial resolution is about one-half wavelength due to the diffraction limit. For example, the wavelength of visible light is between 400 and 700 nm; therefore, the spatial resolution is restricted to a few hundreds of nanometers in the visible range. However, bringing a subwavelength-sized test object close to the sample surface modifies the field distribution on a shorter scale and an excellent spatial resolution overcoming the diffraction limit is obtained. This concept can be realized by implementing NSOM, in which a variety of near-field probes can be used for the generation and detection of evanescent (near-) field photons and the probe scans over the sample surface within a distance of near-field regime.¹⁾

In NSOM experiments, the commonly used probes are tapered optical fibers coated with an opaque metal film leaving a subwavelength aperture at the apex.^{2,3)} In principle, apertured NSOM has two types, depending on the functionality of the optical fiber probe: collection-mode NSOM, in which the sample is excited homogeneously and the probe is used to locally detect the radiation; and excitation-mode NSOM, in which instead of acting as a local detector, the fiber probe serves as a nano-illuminator to locally excite the sample in combination with detection in the far field.

To measure a near-field optical signal, the separation

between the tip and the sample surface must be kept in the near-field zone, practically, at 1–20 nm, during scanning. This can be achieved through optical shear-force feedback control, in which a special bimorph or piezotube holds the optical fiber, and the fiber is dithered laterally with an amplitude of approximately 5 nm at a low-order resonant frequency. As the fiber tip approaches the sample, this resonance is damped due to the interaction with the sample surface. Dithering amplitude decreases to zero as tip–sample separation approaches null. Tip–sample separation can thus be regulated by means of a feedback loop in which dithering amplitude is detected by monitoring the lateral variations of the fringe pattern on the differential photodiode projected by an auxiliary laser beam impinging on the fiber while dithering.⁴⁾ Although this optical method was widely used, its major disadvantage is that the spurious wandering light arising from the feedback laser can result in a large background optical noise and reduce signal-to-noise ratio for the near-field optical contrast.

A non-optical shear-force method using a crystal quartz tuning fork as the force sensor was initially proposed by Karrai and Grober.⁵⁾ This technique becomes the most commonly used NSOM feedback control method today.^{6,7)} In contrast to the conventional optical-shear-force-mode NSOM, this type of regulation method eliminates problems resulting from elaborate optical beam alignment and the interference between the near-field optical signal and the force sensing optics, and thus reduces instrumentation complexity. An alternative method of distance regulation for NSOM by utilizing a mechanically driven tuning fork operating in the inverted tapping mode instead of the shear force mode has been demonstrated by Tsai and co-workers.^{8,9)} Tapping-mode tuning fork near-field scanning optical microscopy (TMTF–NSOM) has all the advantages afforded by tapping-mode atomic force microscopy, including a high spatial resolution, a high sensitivity, and an excellent stability. Recently, we have also modified TMTF–NSOM

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Fig. 1. Schematic experimental setup for near-field imaging of stationary evanescent fields by collection-mode short-probe TMTF-NSOM. An evanescent field is generated by total internal reflection. The light source is a 632.8 nm He-Ne laser or a diode-pumped Nd:YVO₄ solid-state laser with a wavelength of 532 nm.

using the short-probe scheme, which allows a more versatile design and construction of the TMTF-NSOM system, especially when the inverted tapping mode is not feasible or long optical fiber probes have to be used in the experiments.¹⁰⁾

In this study, we demonstrate the versatile applications of this novel non-optically regulated tapping-mode NSOM to nano-optical metrology and the optical characterization of semiconductors. Collection and excitation modes of short-probe TMTF-NSOM are used to image the interactions of evanescent fields and measure the near-field surface photovoltage (NFSPV) on distributed-Bragg-reflector-enhanced absorbing substrate (DBR-enhanced AS) AlGaInP light-emitting diode (LED) structures, respectively.

2. Experimental

The experimental setup of our homemade short-probe TMTF-NSOM system is based on a commercial atomic force microscope (Stand Alone, Park Scientific Instruments). The force-sensing element consists of a near-field optical fiber probe attached to a crystal quartz tuning fork. The NSOM probe was first fabricated by applying a heating-and-pulling method to a single-mode optical fiber and subsequently immersed in a saturated solution of ammonium bifluoride (NH₄F·HF) for about 5 min. The probe was finally coated on its sides with a thin layer of aluminum to confine light. By this procedure, typical aperture sizes with diameters between 50 and 80 nm were obtained.

In the short-probe TMTF-NSOM scheme, the optical fiber probe is attached perpendicularly to the end of one tine of the tuning fork, while the other tine is glued to a ceramic plate with the fork protruding by about 3.5 mm from the edge of the plate. The ceramic plate is tightly clamped onto and mechanically excited by a piezoelectric bimorph. Both the tuning fork and fiber tip vibrate in a direction normal to the sample surface. The resonance frequency of the tuning fork proper is approximately 32.768 kHz. The tip protrudes by about 1 mm from the fork edge and the short fiber probe

is obtained by cleaving the optical fiber halfway between the two prongs. A flat-ended multimode fiber is then glued on the other prong of the tuning fork with an air gap between the flat end faces of the short probe and the multimode optical fiber being approximately 1 μm. All these procedures are performed with the aid of a precision multi-axis translation stage and a 90× stereo optical microscope. Care and patience must be exercised in aligning the long axis of the short fiber probe with that of the multimode optical fiber for the efficient coupling of light. The typical throughput (i.e., output power from the tip aperture vs input power through the multimode fiber) of this configuration is about 10⁻⁴ or higher.

The asymmetric configuration of the two prongs induces a voltage difference between the two electrodes of the tuning fork under excitation. The frequency dependences of the induced voltage differences show sharp and pronounced resonance peaks at approximately 31.5 kHz with *Q* factors of about 190. The significant feature of the resonance response provides a noticeable change in the feedback signal as the tip approaches the sample surface, by which a stable and sensitive distance regulation scheme is realized.

The schematic experimental setup for the near-field imaging of stationary evanescent fields is presented in Fig. 1. An evanescent field is generated by total internal reflection (TIR). The light source is a 632.8 nm He-Ne laser or a diode-pumped Nd:YVO₄ solid-state laser with a wavelength of 532 nm. A right-angled fused quartz prism (45°–90°–45°) is illuminated at normal incidence on one side face and TIR occurs at the hypotenuse face. The normally incident light beam therefore has an angle of incidence of 45° on the TIR interface. The partially reflected light from the exit face of the prism returns along the original path and then interferes with the incident light in the evanescent regime. The probe tip is brought close to the prism TIR surface and scanned to collect the evanescent wave interference fields.

Measuring spatially resolved NFSPV on DBR-enhanced

Fig. 2. Schematic experimental setup for near-field surface photovoltage measurement by excitation-mode short-probe TMTF-NSOM. A laser beam ($\lambda_p = 532$ or 632.8 nm) mechanically chopped at 500 Hz is coupled to the optical fiber and then delivered to the cross section of the sample through the subwavelength tip aperture. Local SPV detection is based on the local induction of photovoltage on the sample edge by near-field optical excitation and the SPV signal is measured across the sample front surface and sample substrate.

AS AlGaInP LED structures is performed by excitation-mode short-probe TMTF-NSOM, the experimental layout of which is shown in Fig. 2. The DBR-enhanced AS AlGaInP LED structures investigated in this experiment were grown by low-pressure metal organic vapor phase epitaxy in a *p*-side-up configuration on a misoriented conductive *n*-type GaAs substrate tilted 2° off the (100) plane toward the [111] direction. The epitaxial structure consists of a 34-period Te-doped $n\text{-Al}_{0.5}\text{Ga}_{0.5}\text{As}/\text{Al}_{0.92}\text{Ga}_{0.08}\text{As}$ distributed Bragg reflector following the initial growth of an *n*-GaAs buffer, a $0.5\text{ }\mu\text{m}$ Te-doped $n\text{-(Al}_{0.7}\text{Ga}_{0.3})_{0.51}\text{In}_{0.49}\text{P}$ cladding layer, an undoped active region comprising three 5 nm compressively strained $\text{Ga}_{0.47}\text{In}_{0.53}\text{P}$ quantum wells embedded within $(\text{Al}_{0.7}\text{Ga}_{0.3})_{0.51}\text{In}_{0.49}\text{P}$ barriers, a $0.5\text{ }\mu\text{m}$ Mg-doped $p\text{-(Al}_{0.7}\text{Ga}_{0.3})_{0.51}\text{In}_{0.49}\text{P}$ cladding layer, and a $\sim 9\text{ }\mu\text{m}$ Mg-doped $p\text{-Ga}_x\text{In}_{1-x}\text{P}$ current spreading layer. An overview of the epitaxial layers is illustrated in Fig. 3. The device structures are designed to emit light with a wavelength of $632 \pm 5\text{ nm}$ operating at 20 mA . For NFSPV measurement, a laser beam ($\lambda_p = 532$ or 632.8 nm) mechanically chopped at 500 Hz is coupled to the optical fiber and then delivered to the cross section of the sample through the subwavelength tip aperture, which serves as a local pumping source. Local SPV detection is based on the local induction of photovoltage on the sample edge by near-field optical excitation and the SPV signal is measured across the sample front surface and sample substrate.¹¹⁾

3. Results and Discussion

The incident laser beam and the partially reflected light from the exit face generate evanescent waves propagating in opposite directions and interfere with each other at the TIR interface. The combination of these two fields produces standing evanescent waves, the intensity of which can be written as^{12,13)}

$$I = |E|^2 = E \cdot E^* \\ = |E_1|^2 + |E_2|^2 + 2E_1 \cdot E_2 \cos(2k \sin \theta x) \quad (1)$$

The period d of interference fringes can then be derived using eq. (1) as

$$d = \lambda / 2n \sin \theta, \quad (2)$$

in which λ is the wavelength of the light source in vacuum, n denotes the refractive index of the fused quartz prism, and θ represents the angle of incidence at the TIR interface. Given that $\lambda = 632.8\text{ nm}$, $n = 1.457$, and $\theta = 45^\circ$, the calculated period of the standing evanescent waves is 307.1 nm . The corresponding experimental results are shown in Fig. 4. Figures 4(a) and 4(b) show the simultaneously measured surface morphology and evanescent wave interference fringes obtained by collection-mode short-probe TMTF-NSOM, in which a 632.8 nm He-Ne laser is used as the light source. The diameter of the tip aperture is about 80 nm , estimated by scanning electron microscopy. The scanned area is $8 \times 8\text{ }\mu\text{m}^2$ and the topographic variation on the scanned prism surface is less than 5 nm . A sectional plot along the line shown in Fig. 4(b) is displayed in Fig. 4(c). The measured period of the interference fringes is 312.5 nm , which agrees well with the theoretical value of 307.1 nm within 1.8% error. Also shown in the upper part of Fig. 5 is the evanescent wave interference pattern obtained when the incident laser beam is replaced by a diode-pumped Nd:YVO₄ solid-state laser with a wavelength of 532 nm . The scanned area is $8 \times 4\text{ }\mu\text{m}^2$ and the cross-sectional view along the central horizontal line in the scan is plotted in the lower part of the figure. The measured average period is approximately 262.6 nm . Substituting 532 nm for λ , 1.461 for n , and 45° for θ in eq. (2), we calculate the interference fringe period to be 257.5 nm . In this measurement, the experimental value is also rather consistent with the

Fig. 3. Schematic illustration of DBR-enhanced AS AlGaInP LED structure. Included are the *n*-type GaAs buffer, *n*-Al_{0.5}Ga_{0.5}As/Al_{0.92}Ga_{0.08}As distributed Bragg reflector, undoped (Al_{0.7}Ga_{0.3})_{0.51}In_{0.49}P/Ga_{0.47}In_{0.53}P multi-quantum wells sandwiched between *n*- and *p*-(Al_{0.7}Ga_{0.3})_{0.51}In_{0.49}P cladding layers, and top *p*-Ga_xIn_{1-x}P current spreading layer.

Fig. 4. Simultaneously measured (a) surface morphology and (b) evanescent wave interference fringes obtained by collection-mode short-probe TMTF-NSOM, in which a 632.8 nm He-Ne laser is used as the light source. The scanned area is $8 \times 8 \mu\text{m}^2$. A sectional plot along the line perpendicular to the interference fringes shown in (b) is displayed in (c). The measured period of interference fringes is 312.5 nm.

theoretical one with an error of 2.0%. The near-field imaging of the interactions of evanescent fields clearly provides a novel method for the metrology calibration of length and surface on the nanometer scale. For example, the nonlinear response of the piezoelectric scanner of the NSOM system can be immediately calibrated using the stationary evanes-

cent wave interference fringes shown in Figs. 4(b) and 5.

We have considered index dispersion in calculating the periods of interference fringes from different coherent sources. Normal procurement will produce glass with an index of refraction within ± 0.001 of the nominal indexes of refraction from the supplier's catalog. However, a variation

Fig. 5. Shown in the upper part are evanescent wave interference fringes produced by the incident laser beam of a diode-pumped Nd:YVO₄ solid-state laser with a wavelength of 532 nm. The scanned area is $8 \times 4 \mu\text{m}^2$ and the cross-sectional view along the central horizontal line in the scan is plotted in the lower part. The measured average period is approximately 262.6 nm.

of ± 0.001 in the index results in a change in interference fringe periods by only $\pm 0.2 \text{ nm}$, which cannot account for the experimental observations. On the other hand, the optical path has been elaborately aligned to ensure a precise normal angle of incidence on the entrance face of the right-angled prism and the prism was also carefully processed to keep the TIR surface free of contaminants such as water or fingerprints that would interfere with TIR.¹⁴⁾ Therefore, the differences between the measured results and the calculated interference fringe periods may come from the nonlinearity of the scanning using our NSOM system. Because the measured near-field signals are the convolution of the collection function of the optical fiber probe and the real interference function, the resolution of the interference patterns obtained is restricted by probe size. In principle, the resolution of using this technique as a calibration method is determined by the size effect of the near-field probe and the background noise of the detection system. On the basis of the deconvolution results of the interference patterns, we estimate that the probe size is about 60 nm.¹⁵⁾

When the semiconductor surface is illuminated by light with a photon energy larger than the fundamental band gap, a photoresponse signal is generated due to the non-equilibrium accumulation of photoexcited minority carriers at the surface and the surface depletion of photoexcited majority carriers driven by a built-in surface electric field. The photoresponse signal can be detected in the form of a short circuit current (photocurrent) or an open circuit voltage (photovoltage) in the external circuit.¹⁶⁾ In this work, we apply excitation-mode short-probe TMTF-NSOM to spatially resolved NFSPV measurement on DBR-enhanced AS AlGaInP LED structures.

Figures 6(a) and 6(b) exhibit the simultaneously obtained surface morphology and NFSPV images on the sample edge at excitation wavelengths λ_p of 532 and 632.8 nm, respectively. The scanned area is $10 \times 10 \mu\text{m}^2$. The two NFSPV images are measured in the same region by comparing their

Fig. 6. $10 \times 10 \mu\text{m}^2$ surface morphology and NFSPV images on the DBR-enhanced AS AlGaInP LED sample edge at excitation wavelengths of (a) $\lambda_p = 532 \text{ nm}$ and (b) $\lambda_p = 632.8 \text{ nm}$, respectively. The photoresponse is rather uniform in the lateral direction.

Fig. 7. Cross-sectional NFSPV profiles along the central lines perpendicular to the active regions at excitation wavelengths of (a) $\lambda_p = 532 \text{ nm}$ and (b) $\lambda_p = 632.8 \text{ nm}$, respectively. The transverse photoresponses on the sample edge are quite similar except that the peak SPV intensity obtained at $\lambda_p = 632.8 \text{ nm}$ is about three times larger than that obtained at $\lambda_p = 532 \text{ nm}$.

corresponding topographic images. As can be seen, the spatial variations in NFSPV show a bright stripe in regions corresponding to the active region, cladding layers, distributed Bragg reflector and current spreading layer of the investigated AlGaInP LED structure. NFSPV is rather uniform in the lateral direction. Plotted in Figs. 7(a) and 7(b) are the cross-sectional NFSPV profiles along the central lines perpendicular to the active regions corresponding to Figs. 6(a) and 6(b), respectively. As is shown, both profiles show similar broad structures.

There is a close resemblance between the near-field photoresponse images observed on a p - n single homojunction and heterostructure lasers. By measuring the near-field photocurrent (NFPC) of the p - n homojunction on a Si substrate, Fukuda and Ohtsu can estimate the dopant concentration of the p - n junction based on the simple relationship between the full-width at half maximum of the cross-sectional profiles of the NFPC signal and the applied reverse-biased voltage.¹⁷⁾ On the other hand, by performing NFPC measurement, in which different excitation lasers with suitable wavelengths were used, we were able to identify the energy range of the defect level as well as locate the positions of the defects in a multi-quantum-well semiconductor laser.¹⁸⁾

As in NFPC measurement, NFSPV signals comprise the absorption of active layers and defect- or impurity-related absorption induced by defects or impurities distributed within the LED structures. The broad features appearing in the cross-sectional NFSPV profiles are similar to the NFPC results observed in laser diode structures, in which the broad backgrounds are due predominantly to unguided propagating waves that traverse the active region only once.¹⁹⁾ Furthermore, the peak NFSPV intensity obtained at $\lambda_p = 632.8$ nm is about three times larger than that obtained at $\lambda_p = 532$ nm. This phenomenon is due to the fact that the excitation wavelength of 632.8 nm is closer to the emission wavelength of the DBR-enhanced AS AlGaInP LED structure.

4. Conclusions

In summary, we have described the optimum design of the NSOM system based on a short-probe TMTF configuration and demonstrated its versatile applications to nano-optical metrology and the optoelectronic characterization of semiconductors. Interference fringes induced by standing evanescent waves generated by total internal reflection are imaged in the collection mode. Both the 632.8 nm He-Ne laser and 532 nm diode-pumped Nd:YVO₄ solid-state laser are used as light sources. The measured periods of the interference fringes are in good agreement with the theoretically calculated results. Our experiments show that the method can be applied to nano-optical metrology with a high precision. On the other hand, excitation-mode short-probe TMTF-NSOM is applied to the NFSPV measurement

on DBR-enhanced AS AlGaInP LED structures. The NFSPV results obtained using excitation sources with different pump wavelengths show that NFSPV signals can be significantly enhanced at excitation wavelengths close to the emission wavelength of such LED structures.

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