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Citation for published version (APA):

DOI:
10.1063/1.1333045

Document status and date:
Published: 01/01/2001

Document Version:
Publisher’s PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:
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Download date: 19. Apr. 2021
Low-temperature scanning-tunneling microscope for luminescence measurements in high magnetic fields

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(Received 25 July 2000; accepted for publication 24 October 2000)

We have designed and built a low-temperature (1.3–4.2 K) scanning-tunneling microscope which is capable of collecting light that is generated in the tunneling region. Light collection is done by means of two fibers whose cleaved front is in close proximity (\textasciitilde1 mm) to the tunneling region. The whole system can be operated in high magnetic fields (11 T) without loss of optical signal strength. As a demonstration, we measured the electroluminescence spectra of an InGaAs quantum well at various temperatures. At 4.2 K, we found an electron-to-photon conversion factor that is three orders of magnitude higher than at room temperature. © 2001 American Institute of Physics.

[I DOI: 10.1063/1.1333045]

I. INTRODUCTION

Briefly after the invention of the scanning-tunneling microscope (STM), it was realized that its extreme spatial resolution can be extended into the optical domain.\textsuperscript{1} By injection of minority carriers\textsuperscript{2} or impact ionization\textsuperscript{3} in semiconductive samples, or by exciting local plasmon states on metallic samples,\textsuperscript{4} the optical properties of various materials can be studied with extreme spatial resolution. Therefore, STM-induced luminescence (STL) is very promising as a tool for the study of nanostructures like self-assembled quantum dots, metallic clusters, or single molecules.\textsuperscript{5,6} However, especially for semiconductor nanostructures, the optical signal that has to be detected in such a STL experiment can be extremely weak due to the low tunneling current and a low overall electron-to-detected-photon efficiency. Moreover, the thermal energy at room temperature (RT) is often of the same order of magnitude as, or larger than, the relevant energy scales in the nanostructure. Working at liquid-helium temperatures can, therefore, be extremely advantageous since it not only reduces the thermal smearing, but also enhances the quantum efficiency. For semiconductor heterostructures this enhancement of the peak height is typically three orders of magnitude.

In this article, we will describe a compact low-temperature STL setup, in which magnetic fields up to 11 T can be applied parallel to the direction of the tip. The STM-induced luminescence signal is collected by two large-diameter fibers and analyzed by means of a liquid-nitrogen-cooled charge-coupled-device (CCD)-based spectrometer.

II. INSTRUMENT DESIGN

A. STM and cryostat

The main constriction in the design of the STL setup we describe here is the demand that the STM can be inserted in the 53 mm bore of a superconducting magnet. Consequently, it is extremely hard to create a direct line of sight from the outside to the tip–sample region. Light collection has, therefore, to be done by means of optical fibers, which are brought close to the tunneling region. In Sec. II B, the optical part of the setup will be discussed in detail. The STM head that was used has been described in detail in an earlier publication,\textsuperscript{7} and is depicted in Fig. 1. Due to its concentric and compact design, it is extremely stiff and insensitive to vibrations. A differential screw is used for course approach in the \textit{z} direction. In principle, course \textit{X}–\textit{Y} movement by means of the inertia mover principle is possible by applying appropriate voltages to the outer piezo (\textsuperscript{5} in Fig. 1). This can only be done if the sample plate (2) is friction coupled to

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the sample holder (1) and can move in the X–Y plane. In praxis, a feeling knock against the setup has the same effect. The single modification that had to be made to allow for the insertion of two fibers are the slits (5) through which the fibers (3) are led. These slits should not influence the performance of the STM with respect to topography and (electrical) spectroscopy, as was described in Ref. 7, since the outer piezo is only used to compensate the thermal contraction of the inner (scanning) piezo. The head is screwed on a stainless-steel tube (not shown) through which the wiring and the driver for the differential screw that is used for coarse approach, are fed. About 30 cm above the STM head the fibers are also guided into this tube for protection. The STM is contained in a stainless steel tube with an outer diameter of 38.1 mm (Ref. 8). The outer tube can be evacuated and is filled with high-purity He contact gas prior to measuring. This procedure prevents dirt from freezing to the sample surface and the tip during cooldown. At room temperature, the absence of a water film on the sample surface avoids local anodic oxidation of semiconducting samples at positive sample bias,9 which otherwise rapidly damages the optically active layers. Moreover, it was found that the removal of the ambient water film by flushing with He and evacuating the system to $3 \times 10^{-4}$ mbar caused an increase in STL signal at room temperature by almost a factor 3. The outer tube, containing the STM, is mounted in an insert that is separated from the main bath by a vacuum shield. By separately pumping the insert, the STM can be cooled below 4.2 K without large losses of cryogenics. The used cryostat is a superinsulated helium-only system, equipped with a superconducting coil that can generate magnetic fields up to 11 T. Decoupling from external vibrations is done by four air legs, carrying a heavy metal frame containing the cryostat. Vacuum pumping and helium handling are done through long flexible bellows. In order to change the tip or the sample, the outer tube has to be taken out of the insert. Depending on the amount of helium left in the insert the total turn-around time is between 2 h and 1 day.

**B. Optics**

Figure 2 schematically shows the system for guiding the two fibers towards the tunneling region. The fiber positioning system is clamped to the STM head by three screws (not shown) in the mounting ring (5). All metallic parts are made of nonmagnetic stainless steel, apart from the guiding tubes (3), which are made of “new silver.”10 The central part of the positioning system are the guidance tubes, directing the fiber towards the tunneling contact. The fibers are held in the tubes by friction. Fiber alignment is done by sliding the guidance tubes back and forth in the slits in the mounting ring, by adjusting screw (1) and by manually bending the guidance tubes or the tip itself. In general, mounting a new tip takes 5–10 min. Finally, it should be pointed out that no effects of fiber dealignment, which could result from stress relaxation in the guidance tubes, are observed after cooldown, nor do we observe any dealignment due to the application of a magnetic field.

Given the fixed diameter of the outer tube which contains the STM, the choice of the optimal fiber is a tradeoff between the solid angle of detection and the allowable bending radius $R$. Usually, thicker fibers have a higher numerical aperture (NA) and can collect more light simply because of their larger diameter, but have a larger minimal bending ra-
anium. We used a fiber with a core diameter of 600 $\mu$m, a NA of 0.37, and a specified minimal bending radius of 58 mm.\textsuperscript{11} The additional transmission losses at 77 K as compared to RT were found to be $0.06 \pm 0.01$ dB per meter. The typical distance between the fiber end and the tunneling region is about 1.0–1.2 mm, which gives a solid angle of detection of 0.2–0.3 Sr per fiber. Note that in this geometry the detection is limited by the fiber’s area and not by its NA, which makes the system relatively insensitive to slight misalignments.

The collected light is coupled into a 300 mm monochromator by an aperture adjusted fiber coupler. The dispersed light is focused on a cooled-silicon CCD camera with an enhanced sensitivity in the near infrared. The CCD camera has the advantage over a photomultiplier that a whole spectrum can be taken at once, and that for longer integration times the signal-to-noise ratio is better due to the lower dark count rate, although the sensitivity itself is lower.

Due to the relatively large angle between the sample surface and the tip, it is imperative to use slender tips with a narrow top angle, to prevent blocking of the generated light.\textsuperscript{12} Consequently, cut tips cannot be used. Moreover, since the tip mounting has to take place under ambient conditions, the tip material has to be inert to oxidization. For these reasons, we used electrochemically etched tips, made from 0.15 mm platinum wire.\textsuperscript{13,14} Generally, these tips do not give atomic resolution, which is due to their smoothness and the relatively large radius of curvature of about 50 Å.\textsuperscript{13} For optical spectroscopy, however, this is not a problem since then the resolution is limited by either the macroscopic size of the apex or the carrier diffusion length, depending on the material system studied.

### III. EXPERIMENTAL RESULTS

As a first test of the system, we performed STL measurements on a $p$-type InGaAs quantum well (QW) in planar geometry, i.e., with the layer plane perpendicular to the tip axis. The structure was grown on a GaAs substrate and consists of the following layers. An undoped GaAs substrate, a short-period GaAs/AlAs superlattice, a 450 Å Al$_{0.25}$Ga$_{0.75}$As barrier, and finally, a 170 Å GaAs capping layer. The only doping present in the structure are two beryllium delta-doping layers in the center of each AlGaAs barrier, with a nominal Be concentration of $1 \times 10^{12}$ cm$^{-2}$. The measured doping concentration in the well is $6 \times 10^{11}$ cm$^{-2}$. Prior to measurement the samples were briefly etched in diluted HCl and passivated by a 2 min dip in a sulfur containing solution (Na$_2$S in isopropanol) and a subsequent 10 min. anneal at 400 °C. This results in a surface that is inert to oxidation\textsuperscript{14} and that has no surface states inside the GaAs band gap.\textsuperscript{15} By applying a positive sample bias (the tip is virtually grounded in our circuitry), electrons can be injected into the structure, and above a certain threshold luminescence can be observed. Figure 3 displays the measured STL spectrum of the QW at room temperature, 77 K, and 4.2 K. Clearly visible is the well-known blueshift with decreasing temperature. More important for the applicability of STL to other, less luminescent, semiconductor structures is the increasing peak intensity with decreasing temperature. From RT to 77 a factor 50 in peak intensity is gained, and from 77 to 4.2 K another factor 20 is gained. In temperature dependent photoluminescence (PL) experiments on such structures one generally finds this kind of intensity increase. The blue-shift of the 10 T spectrum with respect to the 0 T curve is identical to what is found in magneto-PL measurements on the same sample.\textsuperscript{16} The additional spike in the 10 T spectrum is due to recombination in the capping layer.\textsuperscript{17} From the integrated intensity of the room-temperature spectra we generally find, for a tip-sample bias $U_t$ of 3.5 V, an electron-to-photon conversion efficiency $\epsilon$ of $2.5 \times 10^{-5}$. At 4.2 K we find $\epsilon = 7 \times 10^{-4}$.

Figure 4 shows the measured intensity of the same sample versus bias at a constant current of 10 nA. The power law dependence on bias with an exponent of 2.5 is well known from ballistic electron emission microscopy (BEEM) theory.\textsuperscript{18} In BEEM spectroscopy one generally measures a similar dependence of the ballistically injected current on the tip-sample bias. We will now explain why the current experiment can be regarded as the optical counterpart of BEEM. Self-consistent band-structure calculations show that for $U_t > 1$ V a hole accumulation is formed at the sample surface which effectively screens the electric field. This is exactly what the metallic gate does in BEEM. The injection barrier, which in BEEM is the Schottky barrier between the metallic gate and the semiconductor, is now the first AlGaAs barrier layer. The onset voltage of the luminescence, 2.15 V, is in good agreement with the expected value of 1.9 V for ballistic injection over the Al$_{0.25}$Ga$_{0.75}$As barrier. Finally, the ballistically injected current is not detected electrically, like in BEEM, but optically, via capture by the quantum well and subsequent radiative recombination. A strong advantage of the optical detection scheme is its enhanced sensitivity as compared to electrical detection. Including refraction at the semiconductor–gas interface, our setup has a total photon collection efficiency of $8 \times 10^{-4}$. Since the quantum effi-
Efficiency $\eta$ of our optically active layers is almost unity at 4.2 K, and the detection limit of our cooled CCD camera is a few photons per second, we can optically detect an injected current of the order of 1 fA. Electrically, the detection limit is about 20 fA. This sensitivity makes “optically detected BEEM” a very promising technique for studying subsurface resonant states, e.g., in superlattices, or self-assembled quantum dots, which in normal BEEM are at or below the detection limit. Finally, it should be noted that in “optically detected BEEM” the injected carriers necessarily are minority carriers, whereas in normal BEEM majority carriers are injected, which makes the technique, in this respect, complementary to normal BEEM.

**ACKNOWLEDGMENTS**

The research of one of the authors (M.K.) has been made possible by a fellowship of the Royal Netherlands Academy of Arts and Sciences. Furthermore, the authors would like to acknowledge H. H. J. M. van Helvoort for skillfully modifying the STM, K. Sauthoff for performing part of the shown measurements, and P. A. M. Nouwens for contacting our samples.

8. Oxford Instruments RST 0032, outer diameter 38.1 mm, wall thickness 0.38 mm.
10. New silver is a nonmagnetic alloy of Cu (62%), Ni (18%), and Zn (20%), and is easier to bend than stainless steel.
11. Newport F-MSC, operating wavelength 500–1100 nm, NA = 0.37, core diameter 600 μm, coating diameter 1040 μm, min bend radius 58 mm (for 60 min).