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Analytical photon scanning tunneling microscopy

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The photon scanning tunneling microscope (PSTM) has been operated in spectroscopic mode to both image the topography and analyze stress features on a microindented, chromium-implanted sapphire surface. Light originating primarily from the evanescent field generated by an internally reflected beam, with some contribution from scattering by roughness features and radiation from fluorescence and luminescence, was coupled into the probe tip. The topographic resolution observed (~50 nm) is appreciably finer than the diffraction limit. Stresses of 3 kbar around microindents were measured by monitoring shifts in the photoluminescence peaks. A discussion of the spectroscopic resolution obtainable with this "analytical PSTM" and applications are presented.

The scanning tunneling microscope senses the exponentially decaying electron tunneling probability above the surface of a conductor. A similar type of microscope called a photon scanning tunneling microscope (PSTM) has recently been developed.[1,2] With the PSTM, one senses the exponentially decaying evanescent field above a dielectric in which total internal reflection is made to occur. The probe tip of this optical scanning microscope is a sharpened optical fiber.[1,2] The resolution of the PSTM has been shown to be as small as $\lambda/20$ laterally and $\lambda/100$ vertically, where λ is the wavelength of the radiation which is suffering total internal reflection within the dielectric. In addition to topographical surface data, spectroscopic information can also be obtained from the internally reflected beam.[1]

In the region above the surface of the sample where the evanescent field is sensed in a PSTM, there may also be radiation which contains information about this surface. For example, if a Raman or a photoluminescence event takes place, then this signal can also couple into and propagate through the fiber whose end constitutes the PSTM probe tip.[3] A microscope which utilizes this radiation adds an analysis function to a PSTM. In an analytical PSTM, provision must be made to measure two signals: (i) the signal resulting from detection of the evanescent field, the intensity dependence of which gives the topographic image of the surface, and (ii) the signal resulting from the Raman or photoluminescence event, the spectral dependence of which yields information about the nature of the surface. In the following, results demonstrating the successful construction and operation of an analytical PSTM are presented. The spatial resolution of the spectroscopically analyzed signal in this PSTM is briefly discussed, and a number of possible applications of this novel microscope are mentioned.

To demonstrate the operation of the analytical PSTM, a cylindrical rod of sapphire was cut with a diamond saw in a plane along its axis so as to have two parallel flat faces. The end of the cylinder was cut along a plane with the normal tilted 45° with respect to the cylinder's axis. The schematic drawing of the experimental setup in Fig. 1 shows a cross-sectional view of the sample. All flat faces of the sapphire were polished with a series of diamond grits, the final grit size being 250 nm. The longest face of the sample was then implanted with Cr^{3+} ions so as to achieve a profile of ruby (Cr^{3+}-doped sapphire) with peak dopant concentration approximately 44 nm beneath the sample surface and a profile width on the order of 25 nm. The sample was annealed to relieve any implantation-induced damage. To create surface features that might be used to determine the lateral resolution of the analytical PSTM, an array of microindents was then placed in the implanted surface of the sample. Each indent creates a stress field that decreases with radial distance from the indent. In ruby, a photoluminescence doublet occurs at 692.7 and 694.2 nm and shifts linearly to lower energy with increased hydrostatic pressure—i.e., compressive stress—by 0.0365 nm/kbar. This luminescence doublet provides the spectroscopic signal measured.

The sapphire was put on the sample stage of an existing PSTM (Ref. 1) which uses as a sensing probe an optical fiber chemically etched to a tip radius on the order of 100 nm. Using the 442-nm line of a He-Cd laser, light was made to suffer total internal reflection at the polished and implanted surface in the region of the indent array. The relative orientation of the sample surface, the probe tip,

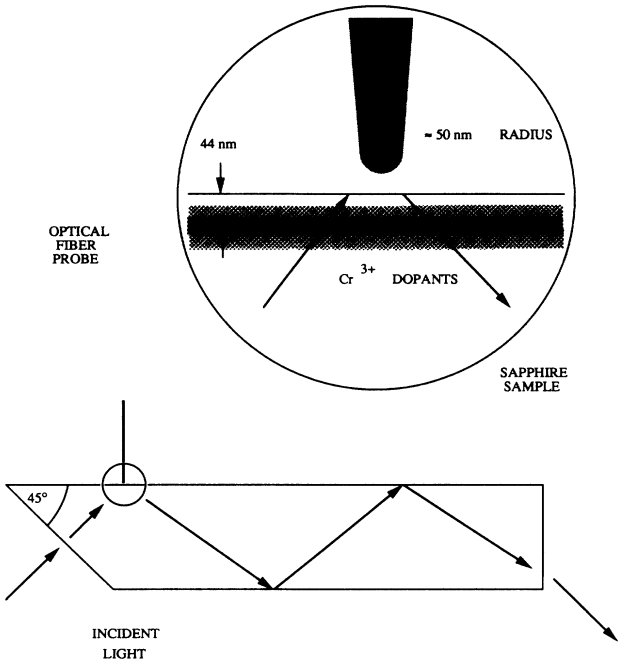

FIG. 1. Schematic diagram of the sapphire sample and microscope probe. The chemically etched optical fiber which serves as the microscope probe tip has a radius of curvature on the order of 100 nm. A layer of Cr^{3+} ions implanted into the sample surface result in a thin layer of ruby beneath the sapphire surface. The peak in concentration of the Cr^{3+} ions occurs at approximately 44 nm beneath the surface.

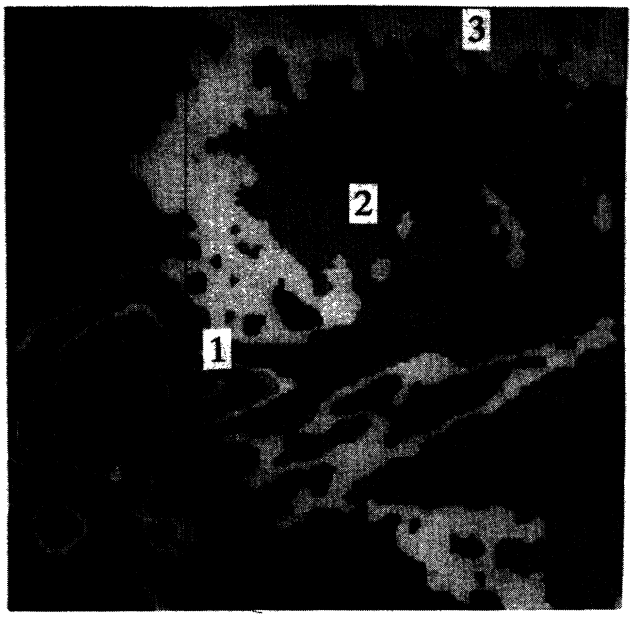

FIG. 2. Grey-scale contour map of the surface of the indented sapphire sample taken with the photon scanning tunneling microscope (PSTM). This micrograph represents a region on the sample surface 10×10 μm^2. Each grey-scale cycle represents a 50-nm height change. The indent extends to a depth of 250 nm below the sapphire surface plane. The wavelength of the radiation which was used to make this micrograph was 442 nm, and resolution of surface features is $\sim$50 nm.

and the ruby implantation are shown in the inset of Fig. 1.

A beam-splitting Y was put in the sensing fiber of the PSTM so as to allow the light sensed by the tip to proceed down two identical channels. One of these channels was returned to the z axis (vertical) control of the PSTM while light emerging from the fiber comprising the second channel of the Y was dispersed in a monochromator (dispersion of 2.5 nm/mm and resolution of 0.5 nm) and detected with a cooled photomultiplier. The output of the photomultiplier was plotted versus the wavelength setting on the monochromator to obtain photoluminescence spectra.

The PSTM image of a region of the sample surface near a microindent is shown in Fig. 2. This grey-scale contour map represents a region 10×10 μm^2, and features a microindent (in the lower left-hand corner) which extends to a depth of 250 nm below the average plane of the sample surface. Features on the polished sample surface (towards the upper right-hand corner) appear to be several tens of nanometers across, thus indicating the topographical imaging resolution to be less than 100 nm.

Three photoluminescence spectra were taken at positions marked by the numbers 1, 2, and 3 in Fig. 2. Spectrum 3 represents a ruby photoluminescence doublet which is indicative of a relatively low-stress region. The distance from the center of the indent is approximately 8.6 μm. As the probe tip approaches the microindent in taking spectra 2 and 1, the rather large compressive stresses known to exist in the region around the microindent manifest themselves in a shifted and broadened photoluminescence spectrum. The luminescence peaks taken at positions 1 and 3 are shown in Fig. 3. The shift of the peaks to lower energy indicates the region of higher compressive stress, as expected closer to the indent. Perhaps more important than the shift in the peak position is the significant broadening toward compressive stress that occurs as the probe is moved closer to the indent (i.e., for spectrum 1).

For these microindented sapphire samples the lateral resolution of the PSTM is clearly finer than the diffraction limit, but it is difficult to accurately determine the resolution of the analytical PSTM. It is straightforward, however, to show that the lateral resolution of the analytical PSTM is significantly finer than that obtainable in conventional microscopic spectrometers. Using the 40×, 0.6 numerical aperture microscope objective on one such instrument,[4] a spot size on the order of 1 μm is obtainable. Conventional photoluminescence spectra taken with this instrument at positions 1, 2, and 3 on the sample shown in Fig. 2 result in linewidths considerably larger than those obtained in the analytical PSTM. Care was taken to duplicate the instrument resolution of the spectrometer used in the analytical PSTM experiment so that the measured linewidth was determined solely by the collection optics. A plot of the linewidth of the two lines of the ruby photoluminescence doublet for the analytical PSTM and for the conventional micro-Raman spectrometer appears as Fig. 4. These data must be viewed in light of the fact that the stress is known to be monotonically increasing as one ap-

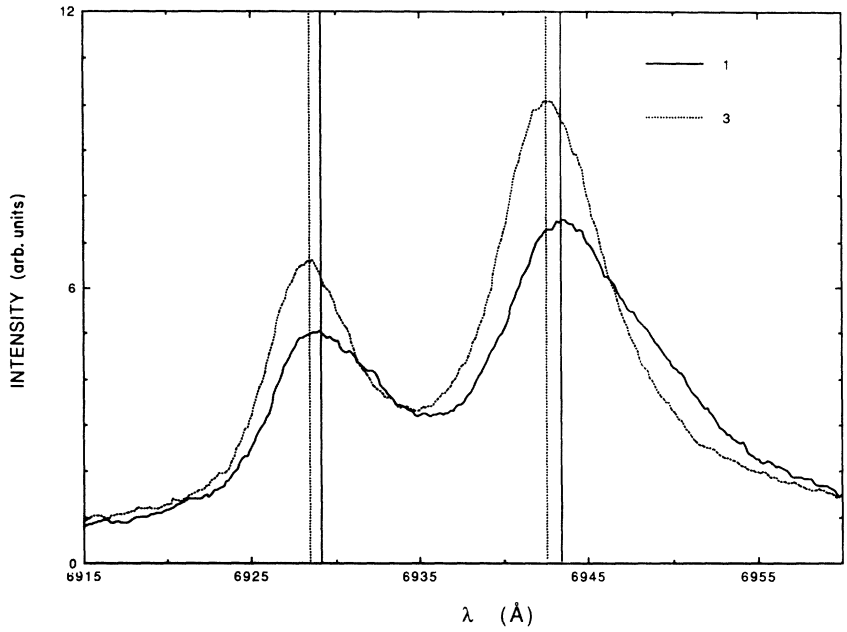

FIG. 3. Ruby photoluminescence spectra taken with the analytical PSTM at positions 1 and 3, as indicated in Fig. 2, are shown. The spectrum taken on the surface plane (spectrum 3) indicates a region of low stress with relatively unshifted and unbroadened luminescence lines. As the probe tip approaches the indent, the spectra shift slightly and broaden significantly, thus indicating increased compressive stress in the region of the indent.

proaches the microindent. As seen in Fig. 4, the widths of the spectra at any given position are measurably greater for the conventional microscope, thus the analytical PSTM samples an appreciably narrower region than the microscope in the micro-Raman spectrometer. Although these data do not confirm that the resolution of the analytical PSTM is finer than the diffraction limit, they clearly demonstrate that resolution is considerably better than that provided by the 1-μm spot obtainable in the micro-Raman spectrometer.

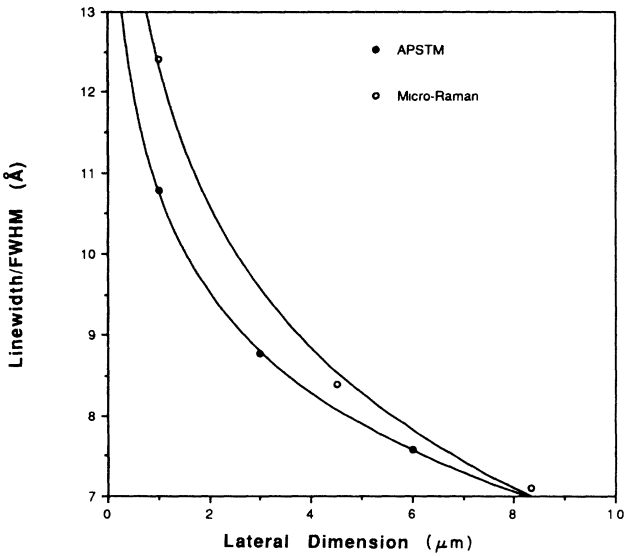

FIG. 4. The linewidth of the photoluminescence spectra taken at positions 1, 2, and 3 shown in Fig. 2 for the analytical PSTM (solid circles) and for a conventional micro-Raman spectrometer (open circles). The conventional microscope on the micro-Raman spectrometer was focused to a 1-μm probe spot. The decreased linewidth of the analytical PSTM spectra taken at the same positions on the sample indicates considerable improvement in spatial resolution for the analytical PSTM over the conventional microscope.

A number of applications of the analytical PSTM suggest that it may become a versatile optical microscope. Surface physicists will be able to monitor luminescence and Raman signals from a surface while determining surface topography. Thus the electronic as well as the topographic structure of surfaces can be simultaneously measured with high spatial resolution. Biophysical applications can be realized by isolating individual specimens on a transparent substrate (presumably with surface desiccation techniques) and then measuring the structure of the isolated species with the PSTM while obtaining chemical information through luminescence with the analytical PSTM. In another application, by coupling light into a thin transparent insulating film, one could monitor defect luminescence at the interface between the film and a semiconducting substrate.[5] High-resolution structural and spectroscopic signatures of such systems could be of considerable use in microelectronic investigations of interfacial atomic and electronic structure.

Just as the development of the STM (Ref. 6)—and the microscopes[7,8] it spawned—ushered in a new era of electron microscopies, so too does the development of the analytical PSTM represent the breaking of new ground in the development of optical spectroscopies. With the analytical PSTM, physicists studying a broad range of problems have at their disposal a family of high spatial resolution optical spectroscopies with simultaneous topographic imaging of features smaller than the wavelength of the probing radiation.

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[1]R. C. Reddick, R. J. Warmack, and T. L. Ferrell, Phys. Rev. B **39**, 767 (1989).

[2]D. Courjon, K. Sarayeddine, and M. Spajer, Opt. Commun. **71**, 23 (1989).

[3]P. J. Moyer, C. L. Jahncke, M. A. Paesler, R. C. Reddick, and R. J. Warmack, Phys. Lett. A **145**, 343 (1990).

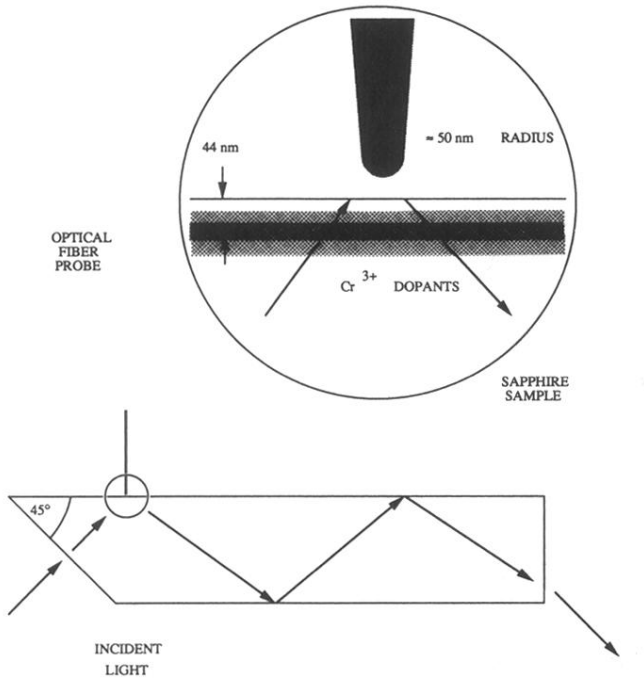
[4]The micro-Raman spectrometer used was the Spex Industries Micramate which is discussed in, for example, R. J. Thibeau, C. W. Goldfarb, and A. Z. Heidersbach, J. Electrochem. Soc. **127**, 37 (1980).

[5]D. P. Tsai, H. E. Jackson, R. C. Reddick, S. H. Sharp, and R. J. Warmack, Appl. Phys. Lett. **56**, 1515 (1990).

[6]G. Binning and H. Rohrer, Physica **127B**, 37 (1984).

[7]H. K. Wickramasinghe, Sci. Am. **261**, 98 (1989).

[8]Robert Pool, Science **247**, 634 (1990).



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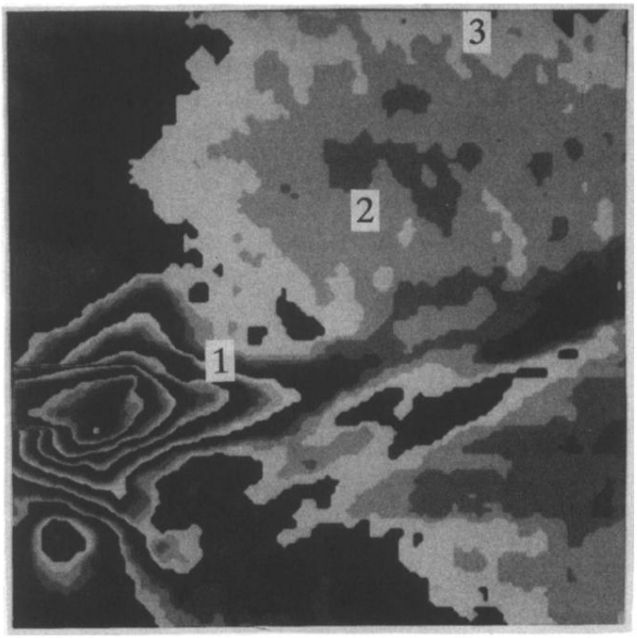

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