Scanning Probe Microscopy: A Rapidly Emerging Instrumental Method

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Scanning Probe Microscopy (SPM) was introduced to the scientific community twelve years ago, and its inventors (Binning and Rohrer) were awarded the Nobel Prize in Physics in 1986. In the last decade it expanded from a single technique limited to a single medium of imaging, namely ultrahigh vacuum (UHV) scanning tunneling microscopy (STM), to an array of techniques operating in essentially any medium (vacuum, air or solution with and without electrochemical control). In all of these techniques, a probe, positioned in close proximity to the sample, scans its surface and monitors some property that is related to the surface topography or to any other surface property. Not only can surface structures and properties be investigated at ultrahigh resolution with SPM, but surfaces can also be modified by design. The latter capability is ushering in a new era of nanotechnology.

The first member of the SPM family is the STM. In STM, the measured property is a tunneling current flowing between the probe and the sample. The second member of the probe family is atomic force microscopy (AFM). In AFM, the probe responds to surface forces. The nature of the measurements (tunneling currents in STM and surface forces in AFM) limits STM imaging to conducting surfaces, but enables any substrate, conducting or insulating, to be imaged by AFM.

Because of their versatility, SPMs can be applied to a wide variety of industries, including semiconductor, magnetic recording, composite materials, sensor technology, pharmaceutical, environmental monitoring, biotechnology, and polymer. Recent instrumental developments in SPM include various types of non-contact modes of AFM for soft samples, magnetic force microscopes, new types of AFM cantilevers, large automated SPM stages that can hold an entire silicon wafer, combined UHV STM/AFT microscopes, new SPMs dedicated to monitoring biological materials, a near-field optical microscope combined with a SPM to allow nano-optical sensing, and a line of instructional SPMs.

Arizona State University (ASU) has one of the largest SPM efforts in the U.S., with over 15 faculty members in science and engineering departments who are using SPM in their research. ASU also has one of the best equipped SPM laboratories in the nation, with vacuum, ambient and solution capabilities for both STM and AFM. This laboratory serves industrial personnel as well as internal research programs.

The SPM facility at ASU is dedicated to making significant contributions to SPM research and education. One area of emphasis is to help transfer scanning probe techniques from the academia to the industrial world. These techniques appear to be ready to enter the industrial workplace for any applications. For example, the capabilities of the SPM both complement and extend those of the electron microscope. In some applications, SPM instruments can perform a non-destructive analysis that is more accurate, faster and less expensive than electron microscopes. Also, SPM measurements do not require a vacuum environment and usually do not require any extensive sample preparation prior to imaging.

In 1990, the SPM Industrial Associates Program (IAP) was established to accelerate industrial use of SPM by providing a mechanism for collaborative research and development, assistance in problem solving and training in SPM technology. The IAP services members from different industries, including semiconductor manufactures, pharmaceutical companies, aerospace industries, chemical companies and instrument vendors. Sample analysis and method development are performed either as part of a membership agreement or on an hourly basis.

The ASU effort in SPM education now encompasses undergraduate training and SPM workshops. Until last year, the SPM techniques were limited primarily to faculty members and graduate students, at which time it was decided to introduce these techniques to undergraduate students. The program involving SPM undergraduate research was so successful that a multidisciplinary SPM laboratory course is being developed to introduce these revolutionary techniques to juniors and seniors. A flexible laboratory manual is also being developed that contains units appropriate for the introduction of SPM experiments into existing analytical or surface characterization courses. The SPM course will also have a SPM laboratory dedicated to undergraduate use.

The most recent addition to the repertoire of the SPM-IAP is to conduct Specialty Conferences/Workshops in specific industries that can benefit significantly from the use of SPM techniques in the near term. These Conferences/Workshops consist of a technical program featuring leading scientists and engineers in the SPM field, exhibitions of the state-of-the-art in SPM technology, and a "hands-on" workshop to demonstrate instrumental capabilities on samples provided by participants. The first Specialty Workshop, "SPM Applications to Semiconductor Materials and Devices," will be held at ASU on October 31 - November 2, 1994. For more information on the SPM/IAP or the upcoming Specialty Workshop, please contact the authors at telephone: (602)965-7155 or Fax: (602)965-1979. ■

A Simple Method To Make High Resolution Projection Slides of Electron Micrographs

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The traditional method of preparing high quality projection slides of one's best electron micrographs was to "print" negatives onto Kodak 2" x 2" emulsified glass slides using a standard enlarger fitted with a reduction lens and then to tray develop the slide to the desired contrast. As A. Kent Christensen pointed out in the June/July 1994 issue of this publication, the cost of 2" x 2" slides has become prohibitive. In any event, I always found adjusting the reduction lens on the enlarger to be a major hassle and searching for the correct exposure and developing times to be a frustratingly inexact experience with much plate wastage.

Several years ago while making "positive inter-negatives" of freeze fracture negatives on Kodak 4489 film from which images with (the more natural) black shadows of replicas could be printed, if occurred to me that one could cut out a 2" x 2" square or 35 mm rectangle from the positive inter-negative and mount it in a large format 2" x 2" or standard 35 mm glass slide binder for projection. It worked like a dream! The technique is simple and fast, yields high resolution projection slides of good contrast and is remarkably inexpensive.

Making positive projection slides by contact printing onto EM film 1) takes advantage of the high resolution inherent in the small grain size of Kodak 4489 EM film, 2) avoids focusing problems because the negative and film are in contact, 3) allows several seconds for dodging if necessary, and 4) in principal doesn't even require an enlarger as any light source will do. Once the film is contact exposed, it is tray developed in D19:water (1:2) under safelights in a darkroom. It is then rinsed, fixed and washed as usual. You should watch the image form in the developer and pull it when the contrast looks right (usually between 60-120 seconds). You really can't tell if the contrast is perfect until the film has been in fix for a few minutes and the emulsion backing dissolved. After washing briefly in water, hold the film over a light box to check on contrast and detail. Finish washing or make adjustments in illumination intensity and repeat.

If you want the entire field of an EM negative in the projection slide, you must still reduce it before exposing the EM film. I prefer to make my projection slides from lower magnification EMs of fields of interest from which I can select the best areas. This method is also excellent for making montage slides of original EMs without requiring the preparation of a master plate of positive EM prints. Simply cut out the desired sizes of images from the contacted film and mount with thin strips of tape to keep the film from moving around in the multiple image slide binders sold by manufacturers such as Gepe. ■