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Design and Calibration of a Vacuum Compatible Scanning Tunneling Microscope

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SUMMARY

A vacuum compatible scanning tunneling microscope has been designed and built, capable of imaging solid surfaces with atomic resolution. The single piezoelectric tube design is compact, and makes use of sample mounting stubs standard to a commercially available surface analysis system. Image collection and display is computer controlled, allowing storage of images for further analysis. Calibration results from atomic scale images are presented.

INTRODUCTION

Solid surface interactions between materials in tribological contact remain of interest in the effort to understand and control friction and wear in mechanical systems. The prospect of probing surfaces on an atomic or molecular scale holds great promise for understanding these complex interactions. In particular, exploring the forces acting at these small length scales should allow a much more detailed understanding of the wear mechanisms operating in tribocontacts.

Development of the Scanning Tunneling Microscope (STM) was first reported in 1982 by Gerd Binnig and Heinrich Rohrer (ref. 1). Already by 1986 the recognized potential of this new technique and its extensions had earned Binnig and Rohrer a share of the Physics Nobel Prize (ref. 2). The technique takes advantage of the quantum mechanical "tunneling" of electrons between a sharp conductive tip and a conductive surface a few atomic diameters away. The quantum mechanical probability of finding an electron "outside" a material decays exponentially away from the surface, providing exceptional sensitivity to changes in the tip to sample separation. Coupling that sensitivity with sub-angstrom tip positioning accuracy allows the STM to map electron density topographies with atomic resolution.

STM design has advanced rapidly over the few years since the technique was first reported. Initial tunneling experiments were performed by Binnig and Rohrer with a platform magnetically suspended in a superconducting lead bowl for vibration isolation (ref. 3). Various generations of advancing STM design have lead to the "pocket-sized" instrument (refs. 4 to 8), which is small enough to be incorporated into vacuum chambers with traditional surface analysis instruments. One of the noteworthy design innovations was the use of a single piezoceramic tube for tunneling tip positioning in all three dimensions (ref. 9). Use of a single piezotube rather than three orthogonal elements simplified the mechanical design as well as allowing for more compact configurations.

Even more recently, the high spatial resolution of the STM has been used to measure the minute deflections of miniature cantilever beams in order to measure force (refs. 10 to 15). The level of force sensitivity achievable is in the range of interatomic forces. The Atomic Force Microscope (AFM) not only provides a probe of forces at the atomic to molecular level, it also allows imaging of insulating surfaces on the nanometer scale. Since the cantilever interaction with the sample surface does not depend on conduction of electrons, the AFM is not restricted to conducting or semiconducting samples as is the STM. Additionally, by monitoring the AFM cantilever deflection parallel to the sample surface, a local measure of friction can be made. Much of the development effort for a STM is directly applicable to an AFM. The development of the working STM described in this report is a step toward the goal of investigating surface interaction forces at the smallest scales.

DESIGN GOALS AND CONSTRAINTS

The design goals, in addition to creating a working Scanning Tunneling Microscope (STM), were vacuum compatibility, use of standard sample mounting stubs from our commercial surface analysis system (ref. 16), in-vacuum sample exchange capability, and small size.

To allow control of the ambient of the surfaces under study, the STM should be ultrahigh vacuum compatible. Vacuum compatibility primarily requires that the materials used in construction not evolve gases (outgas) when placed under vacuum. Many metals and ceramics meet this requirement, though very few plastics, adhesives, or lubricants do. Steel, stainless steel, and ceramics, with minor exceptions, were used exclusively in the construction. In no case was a material used which would provide a major source of outgassing when exposed to vacuum. The specimen holder was designed to accept standard sample-support stubs compatible with an existing surface analysis facility (ref. 16). The specimens were to be removable remotely, since the STM would be inaccessible to the researcher when under vacuum.

Tunneling tips were electrochemically etched to a sharp point in a one molar sodium hydroxide solution. A 6 to 8 V ac potential was applied between the 0.016 in. tungsten wire to be etched and a concentric cylindrical stainless steel electrode fashioned from thin sheet stock. The current was not monitored. Scanning Auger electron spectroscopy analysis of a number of tips indicated the existence of a carbonaceous contamination layer on every tip examined. Argon ion sputtering removed most of the contamination layer relatively quickly. Both sputter-cleaned and unsputtered tips were used in the STM.

Two major concerns in the mechanical design of a STM scanning head are for thermal stability and for insensitivity to vibration (ref. 17). Small STM size can help with both design goals. As well, small size simplifies mounting of the STM head in a vacuum chamber such as the sample preparation chamber of our commercial surface analysis system.

Thermal stability becomes important when two objects are to be held a few angstroms (10^{-8} cm) apart with the support points for each from millimeters to centimeters away. With the difference in thermal expansion coefficient between piezoceramics and most metals of the order of 10^{-5} to 10^{-6} C^{-1}, a change by a

single degree Celsius for a centimeter of support distance will cause a gap distance change of the order of 100 to 1000 Å. If the gap is to be stabile to better than an angstrom, then the temperature regulation must be to better than 1/100th to 1/1000th of a degree Celsius, which is difficult to achieve. Minimizing the separation between points of support and matching coefficients of thermal expansion to the extent possible help reduce the temperature control requirements. High data collection rates also help minimize temperature control requirements by reducing the thermal drift that occurs during a data collection scan.

Acoustic vibration of the STM head can defeat attempts to control the position of a STM tip to within a fraction of an Angstrom. Complimentary approaches to avoiding this problem exist. Isolation of the STM head from sources of acoustic excitation can be pursued, and is employed in nearly all STM heads to some degree. The methods used range from the simplicity of a stack of damper-separated support plates to multistage, magnetically damped hanging platforms. Insensitivity to vibration by design can also be employed to reduce the vibration isolation needed. This is a complimentary approach to reducing the effects of vibration. By designing relatively stiff STM head components, low acoustic frequencies can be made to only oscillate the entire assembly as a unit, leaving the tip to sample separation unaffected. For vibration insensitivity, smaller, stiffer components can be advantageous. Since the STM generally cannot be driven faster than the frequency of its lowest resonance vibration mode, the lighter, stiffer, smaller designs will allow higher frequency scan rates as well.

The STM design described in this report utilizes a single piezoelectric tube for fine mechanical positioning of the tunneling tip, with a fine-screw-thread miniature translation stage for coarse and initial fine sample positioning (fig. 1). Data collection occurs synchronously during computer-controlled piezocrystal-generated raster motion of the tip. Fine positioning of the tip in three orthogonal directions with a single piezoelectric tube is achieved by sectioning the outer electrode of the radially-poled, horizontally mounted tube into four equal quadrants (ref. 9). Applying a voltage between all of the outer sections and the inner cylindrical electrode produces an overall elongation or contraction of the tube (Z direction). Applying a differential voltage between opposing outer electrode quadrants causes bending of the tube (X and Y directions). The sectioning was accomplished by masking all but narrow axial strips of the outer electrode, and using a very fine sand blasting to remove the nickel electrode material.

Coarse sample positioning is accomplished with a miniature translation stage having a 6-mm travel. As the sample surface is brought up to the tunneling tip, the sample support stub encounters pivot points close to the tip. The entire specimen holder then pivots around these points with further advance of the translation stage. Flex pivots (ref. 18) allow pivoting of the entire specimen support (fig. 2). Because the distance between the tunneling tip and pivot points is much smaller than the distance from tip to flex pivots, the stage advance is mechanically deamplified for the sample approaching the tip. The ratio of the distance from tip to pivot point versus tip to flexure joint determines the deamplification ratio. A typical distance ratio would be from 20 to 100, depending on exact tip mounting position and chosen height for the pivot points. For a deamplification ratio of 50, the sample advance per fine division on the translation stage micrometer is reduced from 0.02 mm to 400 nm,

allowing careful manual advance of samples to within range of the tunneling tip.

For vibration isolation a stack of plates separated by Viton spacers was employed. Viton is a high vacuum compatible elastomer which helps attenuate high frequency acoustic vibration. To prevent high frequency acoustic coupling through the electrical connections, the wires used were fine gauge (38 AWG), and were mechanically coupled to the stack plates through split Viton pieces. When the STM was used in air, an acoustic cover lined with sound deadening foam isolated the STM head from room noise. For low frequency isolation, the entire STM head assembly was initially swung from elastic cords close to the controlling computer. This proved sufficient to allow atomic resolution images to be collected.

ELECTRONICS

Raster voltage generation, data collection, and a tip-height control feedback loop are needed for constant-current imaging of sample surfaces. An existing two-channel, digital-to-analog computer interface card (ref. 19) was programmed to generate the required raster pattern voltages to drive the X and Y quadrants of the STM piezocrystal. An unused analog-to-digital conversion channel on the same interface card was programmed to digitally capture the STM output without disabling the computer-controlled instrument with which the computer originally was purchased (ref. 20). The digital-to-analog and analog-to-digital channels of the computer interface card all had 12-bit resolution with a maximum range of 10 to -10 V. The digital-to-analog channels were in fact configured for 0 to 10 V full scale output for increased programming resolution. Over a 10 V span, 12-bit resolution yields control of one part in 4096, or approximately 2.4 mV minimum programmable step size. The analog-to-digital input channels also had programmable gain factors of 2x, 4x, and 8x. These gain factors yield resolutions of one part in 4096 over full scale ranges of ±5.0, ±2.5, and ±1.25 V, respectively, in addition to the ±10 V range with unity gain.

Tunneling tip to sample distance was controlled using an analog feedback loop. As the tip position over the sample surface changes, constant adjustment of the tip position perpendicular to the surface (Z direction) is needed to prevent physical contact between the tip and sample. For this purpose, the output signal from the STM head was amplified and used to control a Kepco BOP 1000-M high voltage power supply driving the Z electrode (inner cylinder) of the piezoelectric tube scanner.

As with any large-gain feedback-controlled system, instability can develop for some combinations of loop gain and phase delay. A somewhat simplified analysis of the requirements for STM stability has already been published (ref. 21). Fundamentally, the response speed is limited by the mechanical response of the scanner head to driving voltages. If the feedback loop gain is too large for a given response time constant, the system will be unstable and will oscillate. However, the lower the gain the larger will be the variation in tip to sample distance during a scan, unless some form of integrating amplifier is used. Without an integrating amplifier, the feedback control output is essentially an amplified error signal, which will then be larger for lower gain loops. A constraint in using the STM then, particularly for large area scans, is the speed and magnitude of the tip response.

To minimize stray electromagnetic signal pick-up, the first stage of electronic buffering was brought as close to the tunneling junction as possible. A high input impedance operational amplifier integrated circuit was mounted directly behind the tunneling tip, aboard the piezoelectric tube support. This acts as a voltage follower, buffering the signal to outside circuitry. When operating properly with feedback control of tip position, the tip potential is kept very close to 0 V (fig. 3). The circuit shown allows independent control of tip to sample potential and tunnel current because of the high input impedance of the operational amplifier (ref. 22). The next stage of amplification was an EG&G Princeton Applied Research Model 162 dual channel boxcar averager with two Model 164 Processor Modules. Using the integrating mode this instrument eliminates steady state error of the tip voltage, and provides control over time constants as well. The boxcar averager output was used to drive a Kepco model BOP 1000-M power supply, modified to reduce 60 Hz ripple (ref. 23). A passive voltage divider was used between the boxcar averager and high voltage power supply as needed to control overall loop gain.

CALIBRATION RESULTS

In order to calibrate the transverse motion of the piezoelectric crystal tube scanner, a highly oriented pyrolytic graphite (HOPG) sample was attached with drops of silver paint to the sample mounting stub from our commercial x-ray photoelectron spectroscopy system (ref. 16). Before insertion of the sample stub into the STM, fresh graphite basal planes were exposed by cleaving the HOPG crystal using adhesive tape. Figure 4 is a gray scale "top-view" of the HOPG basal plane data. The image is 31 Å on a side. Only half of the HOPG surface atoms are typically visible via STM (refs. 24 to 25). Every other atom in the HOPG surface net does not have an atom directly below it in the next atomic plane down, causing the asymmetry between adjacent surface atoms.

The piezoelectric drive calibration constants can be calculated from the known spacing of the visible atoms on the HOPG surface, 2.46 Å at room temperature. The presence of thermal drift during imaging introduces additional uncertainty into the slow scan direction calibration. In order to minimize this uncertainty, the fast and slow scan input leads were reversed for a number of images, then switched back. The calibration constant for the Y-direction motion is calculated from images in which the Y-direction quadrants were the fast-scan electrodes.

An assumption made for the calibration calculation is that the surface net itself is not greatly distorted, which is reasonable for the relatively stiff in-plane carbon-carbon bonds of the graphite sample used. The observed image distortion is then assumed entirely due to thermal drift, initially ignoring any possible nonlinear piezocrystal motion or quadrant crosstalk. Again, this is a reasonable assumption due to the relatively low drive voltages used. The data taking rate is known, as is the sequence in which the points were collected. The following iterative procedure was used to minimize the effect of thermal drift on the calculated calibration constants. Lowest order approximation values are derived for both the X- and Y-direction quadrants from atomic resolution images, assuming negligible drift during a single fast-direction line scan. From distance and angle distortions of the atom positions in the slow-scan direction of the images, a first approximation of the thermal drift

rate and direction can be calculated. The effect of thermal drift during a fast-direction scan can then be determined. If warranted, the corrected fast-direction piezoelectric motion constants are then used to recalculate the rate and direction of the thermal drift.

For the calculation, two atoms centered on the same fast-scan line are chosen, and the center pixel of each determined. The center-to-center separation of the atoms in pixels, bits changed per pixel, millivolts per bit, and known graphite atom spacing and geometry are then used to calculate the angstroms of motion generated per volt across the piezoelectric crystal electrodes. The X- and Y-motion constants are 48 and 52 Å/V, respectively, from images in which the low measured drift rates indicated no need for iterative corrections. Motion constants of this magnitude allow relatively low voltages to be used to image useful areas, without requiring microvolt control in order to achieve atomic resolution.

SUMMARY

The design, construction, and calibration of a scanning tunneling microscope has been successfully completed. Using a single piezoelectric crystal for fine-scale motion, atomic resolution images of the graphite basal plane have been collected. The design is vacuum compatible, requiring only mounting modifications in order to be supported in a vacuum chamber. The instrument can be used immediately for relevant research at atmospheric pressure. The expertise already gained during the development process also can be applied in atomic force microscopy. There are currently no atomic force microscopes commercially available, while the potential applications are numerous. The scanning tunneling microscope currently provides very fine scale measurements over a limited scan range. Ongoing development work is underway to extend the accessible scan area by more than an order of magnitude, as well as provide simple spectroscopy capabilities. Developments in the STM/AFM field should continue to make important contributions in a number of research areas for many years to come.

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23. Per a conversation with Dr. Michael D. Pashley of Philips Laboratories, North American Philips Corporation, Briarcliff Manor, NY, the leads running from the front panel voltage and current programming controls of the BOP 1000-M were disconnected internally at the programming circuit board, significantly reducing output ripple. Programming voltages connect to the control circuit directly via the back panel.

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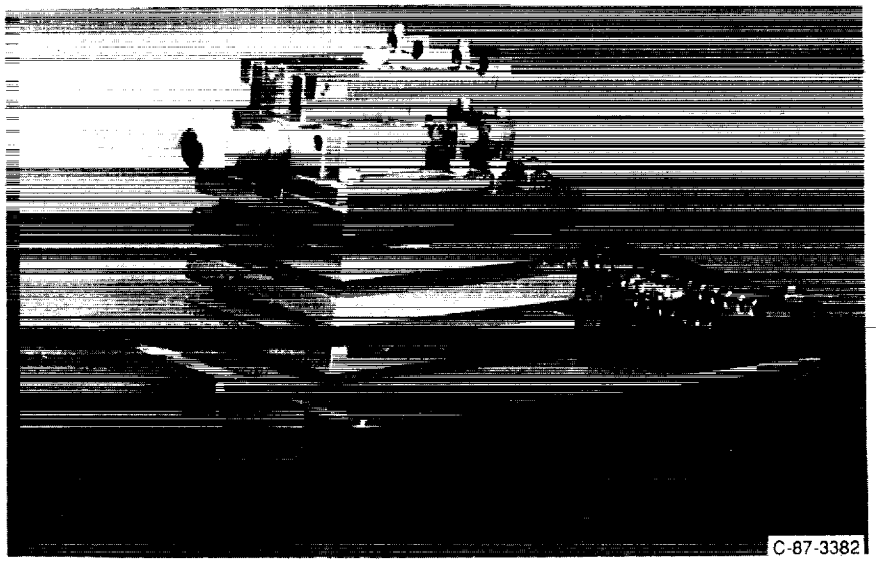
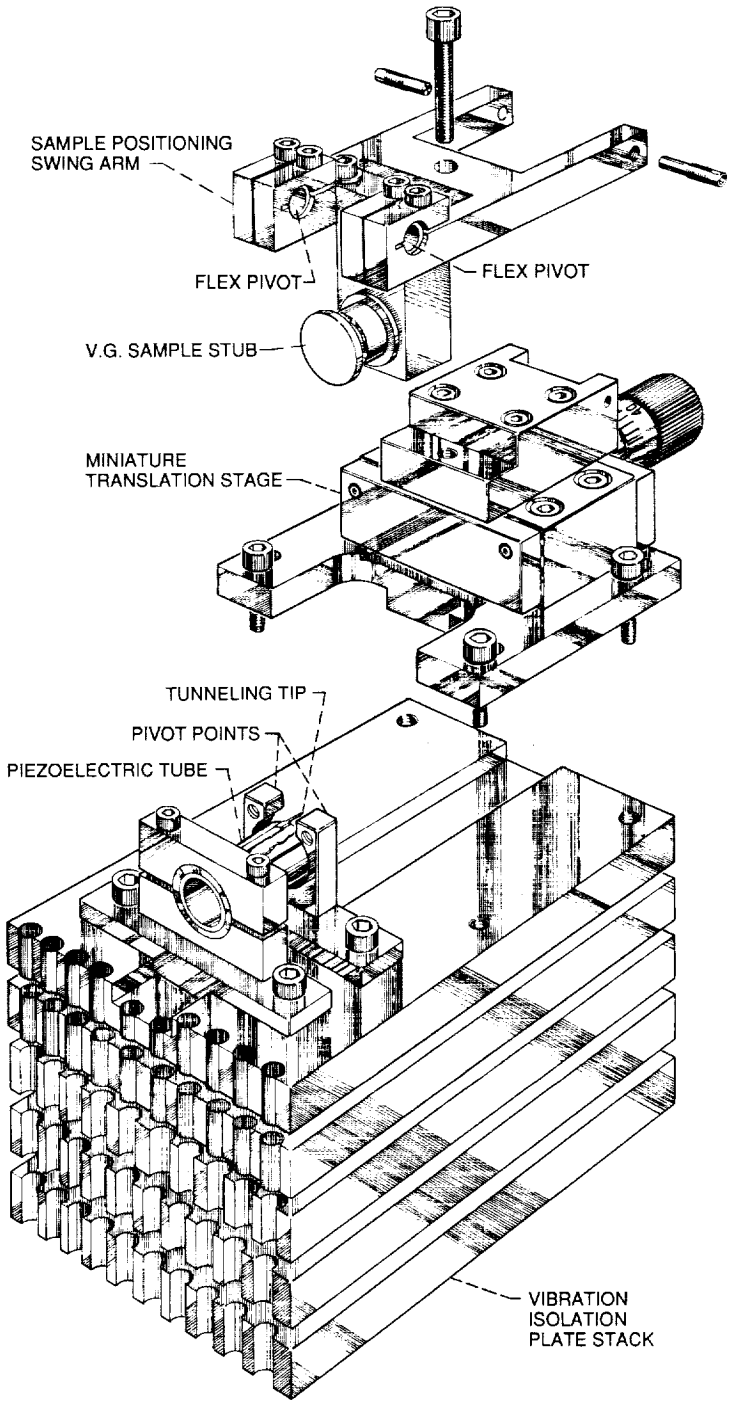
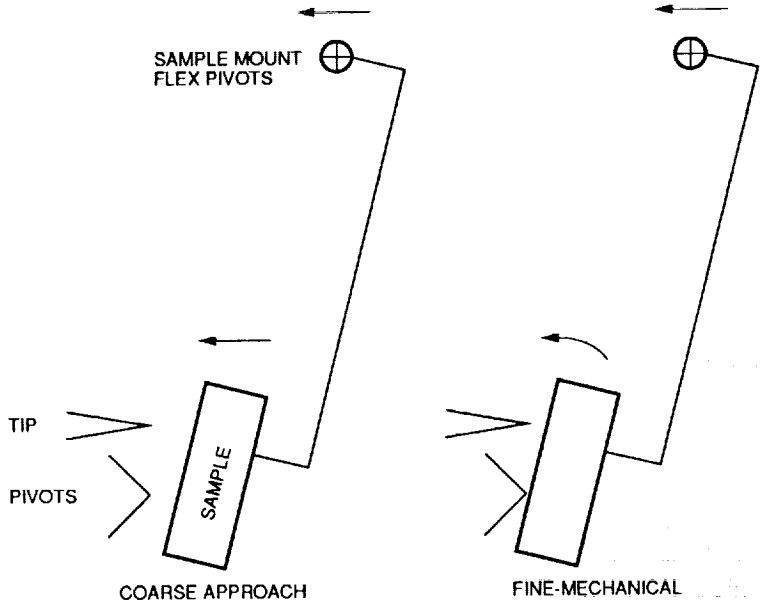


Figure 1. - Photograph of Scanning Tunneling Microscope head on vibration-isolation plate stack. The sample-mount swing arm and on-board electronics are not mounted so that pivot points, tunneling tip, and piezoelectric crystal are visible.



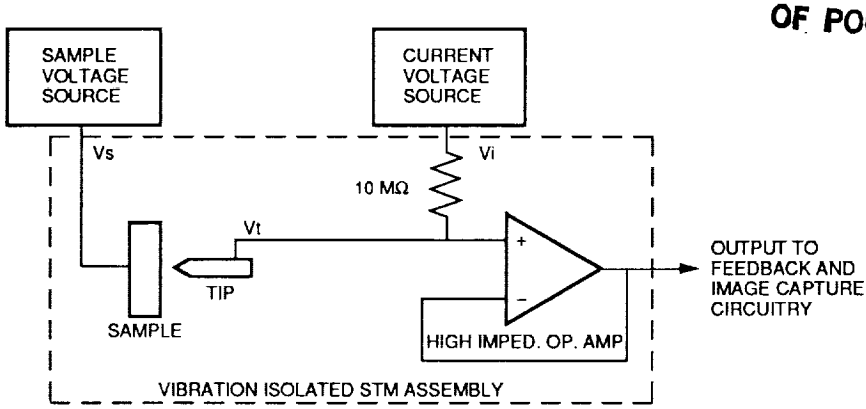
(a) Sketch of main scanning head components; base with piezoelectric tube and pivot points mounted, miniature translation stage with mounting hardware, and sample mount swing arm and holder.

Figure 2. - Scanning tunneling microscope mechanical design.



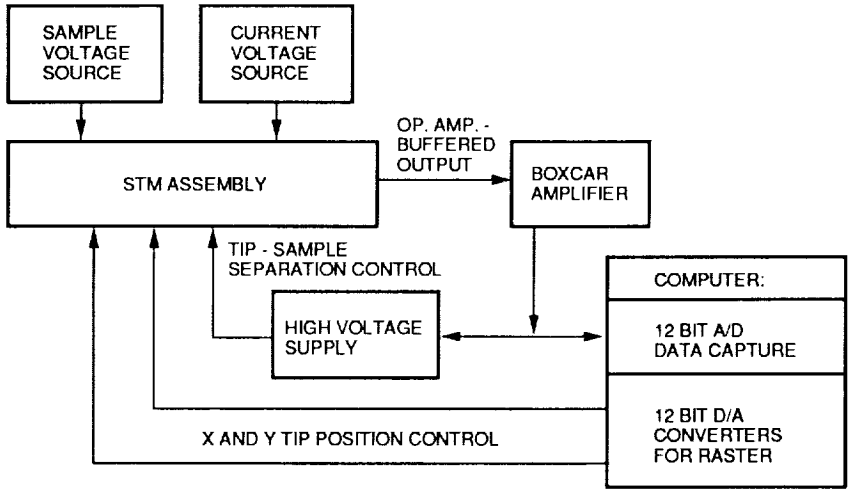
(b) Illustration of mechanical de-amplification of motion achieved by pivoting around points close to tunneling tip.

Figure 2. - Concluded.



WITH FEEDBACK CIRCUITRY OPERATING:
* Vt MAINTAINED AT 0 VOLTS
* TUNNEL VOLTAGE = Vs
* TUNNEL CURRENT, It = Vi/10 MΩ

(a) Onboard voltage-follower circuit, allowing independent control of tunneling current and tunneling potential by external voltage sources.
Suggested by William J. Kaiser, Jet Propulsion Laboratories



(b) Feedback loop, with digitized data capture synchronized to computer control of raster.

Figure 3. - Scanning tunneling microscope electronics design.

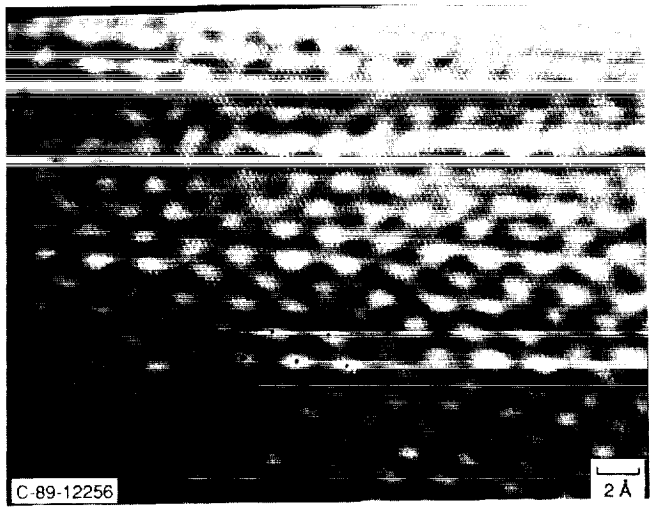


Figure 4. - "Top view" gray-scale image of highly oriented pyrolytic graphite basal plane showing atomic resolution, uncorrected for thermal drift and rectangular screen aspect ratio. The distance between visible atoms is 2.46 Angstroms. Every second surface atom is imaged, giving the "centered-hexagon" pattern as marked with dots.

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16. Abstract

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