Scanning Microscopy

Volume 2 | Number 1 Article 1

11-5-1987

# Tip-Techniques for Microcharacterization of Materials

Y. Martin
*IBM T.J. Watson Research Center*

C. C. Williams
*IBM T.J. Watson Research Center*

H. K. Wickramasinghe
*IBM T.J. Watson Research Center*

Follow this and additional works at: https://digitalcommons.usu.edu/microscopy

Part of the Biology Commons

## Recommended Citation

Martin, Y.; Williams, C. C.; and Wickramasinghe, H. K. (1987) "Tip-Techniques for Microcharacterization of Materials," *Scanning Microscopy*: Vol. 2 : No. 1 , Article 1.
Available at: https://digitalcommons.usu.edu/microscopy/vol2/iss1/1

This Article is brought to you for free and open access by the Western Dairy Center at DigitalCommons@USU. It has been accepted for inclusion in Scanning Microscopy by an authorized administrator of DigitalCommons@USU. For more information, please contact digitalcommons@usu.edu.

Scanning Microscopy, Vol. 2, No. 1, 1988 (Pages 3-8)
Scanning Microscopy International, Chicago (AMF O'Hare), IL 60666 USA
0891-7035/88$3.00+.00

# TIP-TECHNIQUES FOR MICROCHARACTERIZATION OF MATERIALS

*Y.Martin, C.C.Williams and *H.K.Wickramasinghe*

IBM T.J. Watson Research Center
P.O.Box 218
Yorktown Heights, NY 10598

(Received for publication May 24, 1987, and in revised form November 05, 1987)

## Abstract

The invention of the Scanning Tunneling Microscope has stimulated the development of new techniques for microcharacterization of materials, which are based on the use of a very fine tip. Two of these techniques have emerged about one year ago, the Thermal Profiler and the Atomic Force Microscope. Both techniques have recently demonstrated the capability to profile and image conductors and insulators. The resolution attained varies from approximately 50 nm by the Thermal Profiler to a few nanometers with the Atomic Force Microscope, therefore competing with the resolution obtained with electron beam microscopy. We shall describe the principle of these techniques, and present recent results obtained for surface profiling, as well as for temperature mapping, force measurement and mapping of magnetic field on a nanometer scale.

Keywords: atomic force, thermal microscopy, surface profiling, magnetic imaging, near field microscopy.

*Address for correspondence:

K. Wickramasinghe
IBM T.J. Watson Research Center
P.O. Box 218
Yorktown Heights, NY 10598
Phone No. (914) 945-3794

## Introduction

As electronic device sizes get smaller and smaller, there is an increasing need for measurement tools to characterize them with high spatial resolution. In particular, new microscopes capable of imaging features below one micrometer are essential to the microelectronic industry, since conventional optical microscopes fail to work beyond this critical limit. The electron microscope is the predominant instrument used to image smaller details, although it also brings about some drawbacks and limitations (need of a vacuum, electron induced damages, charging effects and loss of spatial resolution on insulating materials, difficult calibration when measuring linewidth or profile of structures).

Among alternative techniques to the electron microscope are some recent near field techniques that followed the invention of the Scanning Tunneling Microscope [4,5]. They have in common the measurement of some interaction between an object and a fine tip. A non-exhaustive list of these techniques (and of the type of interaction that they measure) would include: Scanning Tunneling Microscopy (current), Scanning Tunneling Potentiometry (voltage) [18], Atomic Force Microscope (force) [6,15,17], Scanning Thermal Profiler (temperature) [20,21], Near Field Scanning Optical Microscope (optical radiation) [2,9], Scanning Capacitance Microscopy (charge) [16], Scanning Micropipette Molecule Microscope (vapor elements) [12].

The high spatial resolution of the STM technique has made it an important tool in surface science. Surface states and surface reconstructions are observable as well as the atomic structure of small clusters, such as a few layers of metals on a semiconductor [1,10]. A major drawback of the STM is the need of a conducting sample surface, together with very clean sample and tip surfaces. These restrictions are overcome with the Scanning Thermal Profiler and the Atomic Force Microscope. We shall focus on these two particular techniques for which the spatial resolution can vary between the best of optical resolution (somewhat less than one micrometer) and the atomic resolution that was pioneered by the STM. We shall also identify some specific capabilities of these techniques that could not readily be achieved by other methods.

## The Scanning Thermal Profiler (STP)

The Scanning Thermal Profiler is a non-contacting high resolution surface characterization technique which has the

potential for mapping surface topography with lateral resolution below 100 nanometers. The profiling is achieved by scanning a very small temperature sensor on the end of a heated tip above the surface of a solid. When the heated tip is in close proximity to the solid, the tip temperature is reduced by the thermal coupling between tip and solid. Since the thermal loading of the tip temperature varies rapidly as the gap between the tip and surface approaches zero, it provides a highly sensitive means for measuring and controlling this gap. In a servo system similar to that of the STM, the detected temperature of the tip is fed into a servo control loop which adjusts the average vertical height of the tip, via a piezoelectric element, to maintain constant the thermal coupling as the tip is scanned laterally over the surface. Because the conduction in any solid is so large relative to conduction through air, the solid surface temperature remains essentially unchanged during a scan and the thermal loading felt by the tip is dependent only on the tip to sample spacing. This permits the replication of the true surface topography while traversing structures which have substantially different thermal properties.

The key element of the STP is the ultra small thermal probe which provides the sensitivity and the spatial resolution necessary to achieve high resolution profiling. The probe consists of a conical tip with a thermocouple sensor at its end. See figure 1. As shown schematically in the figure, a thermocouple sensor is produced at the tip by the junction of the dissimilar inner and outer conductors. An insulator separates these conductors in all areas remote from the tip. The thermocouple junction produces a temperature dependent voltage which can be sensed at the other end of the probe across the two conductors. This voltage provides the means for remotely sensing the thermal coupling between tip and solid as the probe is scanned laterally across the solid surface. The thermal probe tips can be made to have dimensions on the order of 100 nanometers. The minimum detectible change in tip temperature is less than 0.1 millidegree. The thermal time constant of the tip is less than 1 microsecond.

The thermal energy transfer between tip and sample is driven by a DC temperature difference between the thermal probe and sample. This temperature difference can be achieved by running a DC current through the thermocouple sensor. To facilitate imaging, the thermal probe is mounted on a piezoelectric structure which provides up to 100 micrometers of travel in any of the three dimensions. To avoid the problems of DC drift in the thermal signal, the tip to sample spacing is modulated at a frequency near 1 kilohertz, and the resultant AC thermal signal is detected and rectified before being sent to the servo loop. The proximity control is therefore provided by the gradient of the thermal loading versus tip to sample spacing, rather than the loading itself. Generally, the AC modulation of the spacing is small relative to the average spacing.

Several structures were profiled to demonstrate the profiling capabilities of the Scanning Thermal Profilometer. Figure 2 contains the profiling results on a seven micron photoresist film on a silicon substrate. Two interesting features are contained in these line scans. The first is that as the fly height of the probe over the resist edge is reduced, a considerable improvement in lateral resolution is achieved. Secondly, the signal to noise ratio is also improved as the tip approaches the surface. The slight differences in the line scans can be attributed to a lateral drift in the scanning system. The profile of the edge of a 100 nm thick aluminium film on silicon is shown in fig.3. The imaged surface structure on the aluminium line

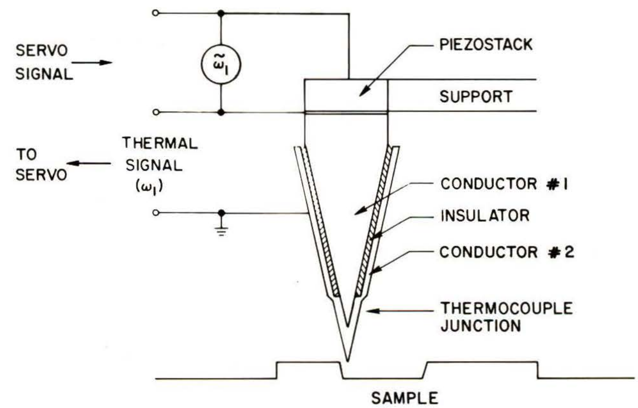


*Fig. 1:* Schematic diagram of the thermocouple probe for the Scanning Thermal Profiler.

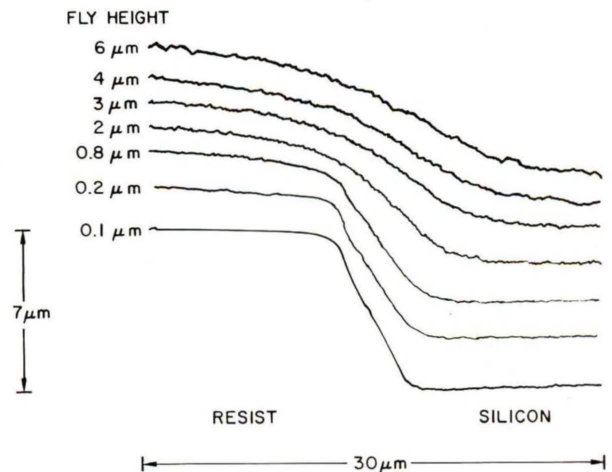


*Fig. 2:* Series of line scans across the edge of a 7μm resist film on silicon as the average fly height is reduced.

demonstrates a lateral resolution on the order of 30 nm. The resolution seen here is consistent with the dimensions of the tip used to profile the structure.

The STP also allows the independent measurement of surface temperature, while profiling. Since high resolution temperature mapping can only be achieved if the tip is in close proximity to the surface, the combination of profiler and thermal microscope provides an ideal means for the measurement of high resolution surface temperature in a non-destructive and non-contacting way. For this purpose, the profiler feedback loop is locked to the gap modulation frequency $f_1$, and the sample temperature is modulated at a frequency $f_2$ so that a constant gap between tip and sample can be achieved without interference from the sample temperature variations if $f_2$ is sufficiently outside the bandwidth of the profiler servo loop. In fig.4a, the profile of a 1 micrometer square area of an aluminium film indicates that a sub micrometer particle with a thickness of 30 nm is attached to the aluminium surface. The heating of the aluminium film is generated by a 4kHz current driven through the aluminium. The resultant temperature variation is detected around 8kHz and displayed in fig 4b. The particle induces here a temperature change of about 1 millidegree centigrade. The lateral resolution in the temperature map

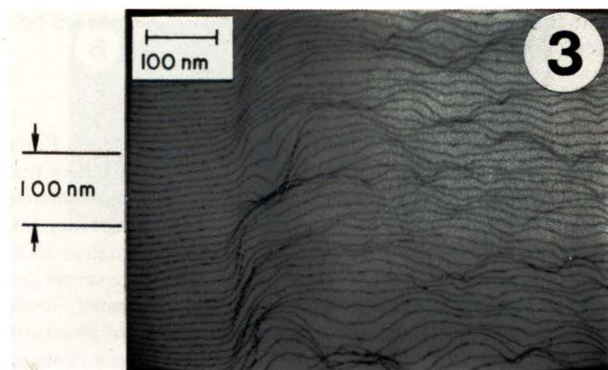


*Fig. 3:* Scans over an edge of a 100 nm thick aluminium film on silicon obtained by STP.

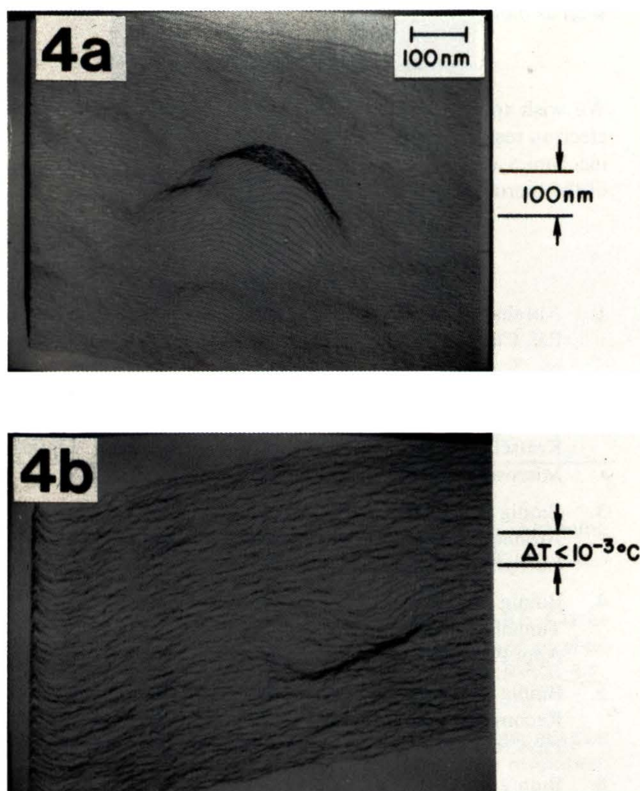


*Fig. 4:* a)Surface profile (by STP) of a one micrometer square area of an aluminium film upon which a sub-micron particle is found, with a 30 nm height. b)Temperature map of the same area. The presence of the particle is seen in the temperature map. The measured variation in thermal signal due to the particle was found to be below 1 millidegree.

appears to be less than 100 nm. The ultimate resolution achievable may be well below this value. The acquisition time for the image is 2 minutes.

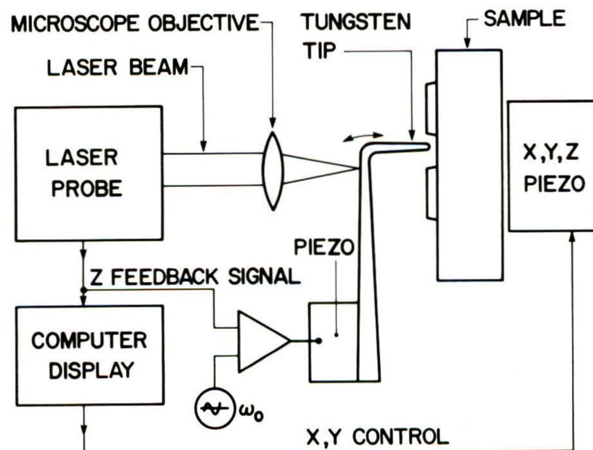


*Fig. 5:* Block diagram of the Atomic Force Microscope.

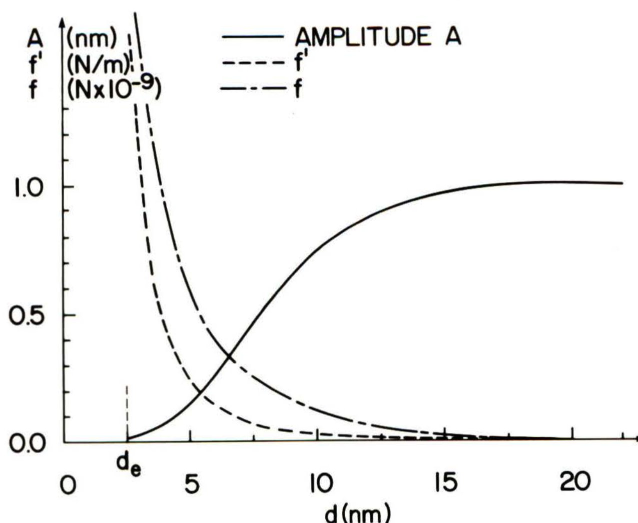


*Fig. 6:* Magnitude of the force derivative f' and force f between a silicon sample and the tungsten tip, calculated from the measured vibration amplitude A as a function of the tip-sample spacing d ; the force is attractive.

## The Atomic Force Microscope (AFM)

The atomic force microscope is a combination of the principles of the Scanning Tunneling Microscope (STM) and the stylus profilometer. In the initial design of the instrument [6], a diamond tip at the end of a cantilever spring follows the surface of an object. The force between the object and the tip deflects the cantilever, and this deflection is accurately measured by a second tip which is part of an STM. A lateral spatial resolution of 3 nm has initially been achieved [6], and more recently, atomic resolution has been obtained on graphite [3].

Our version of the Atomic Force Microscope includes an optical detection instead of an STM detection. An overall diagram of the technique is shown in figure 5. A tungsten tip at the

end of a wire is mounted on a piezoelectric transducer. The transducer vibrates the tip at the resonance frequency of the wire, which acts as a cantilever. A laser heterodyne interferometer [19,7] accurately measures the amplitude of the a.c.vibration. The gradient of the force between the tip and sample modifies the compliance of the lever, hence inducing a change in vibration amplitude due to the shift of the lever resonance. Knowing the lever characteristics, one can measure the vibration amplitude as a function of the tip-sample spacing in order to deduce the gradient of the force, and thus, the force itself [6,8]. Figure 6 shows the vibration amplitude as well as the deduced force derivative and force, as a function of the tip-sample spacing. The accuracy of the measurement can be one percent or less. In the active field of intermolecular and surface forces [11], this technique appears therefore as a new tool for precise measurement of forces on a sub-micron -and perhaps atomic- scale.

The vibration amplitude of the lever also provides a feedback signal for profiling applications. The feedback signal applied to the piezoelectric transducer of the lever moves the tip in the direction normal to the surface in order to follow the profile of the surface. It is recorded as a function of the tip position, and displayed either in the form of line scans or of grey scale images.

Figure 7 shows the topography obtained over an anisotropically etched silicon wafer. A high contrast is obtained over small step-like structures, having dimensions of several tens of nanometers, resulting from the etching process, and that would be barely visible under an SEM. Figure 8 shows an enlargement of one of these small steps, which demonstrates that the spatial resolution achieved is somewhat below 5 nm. Figure 9 shows the capability of the force microscope to inspect nonconducting surfaces; in this example, an electron resist grating deposited on silicon. The periodicity of the grating was 200 nm and the thickness 90 nm. Small structures at the top of the lines are observable. However, the grooves themselves show no detail and appear narrower since the tip end has a diameter comparable to the width of the grooves. A tip thinner than 50 nm over a length of 100 nm or more at its end would enable one to resolve most of the structure at the bottom of the grooves.

We believe that the heterodyne interferometric detection has several advantages over the tunneling detection. The tunneling tip induces a significant force and force gradient on the cantilever when operated in air [8,13]. The force caused by the laser beam is negligible and the force gradient is null. The interferometric detection is also somewhat more reliable, easier to implement, insensitive to the roughness of the lever, and has a smaller sensitivity to thermal drifts. The laser probe used here is described in references 19 and 7. It can measure the amplitude and phase of a.c. displacements as small as $10^{-5}$ nm, in a 1 Hz bandwidth. In addition, it is totally insensitive to thermal drifts in the optics, an attribute of the heterodyne technique. Hence, the distance between the lever and the optical probe need not be accurately controlled. Furthermore, the optical detection scheme enables one to monitor the vibration of the lever even when it is excited into vibrations having amplitudes of several 10 nm, which would prove extremely difficult by tunneling techniques

Replacing the tungsten tip with a magnetized iron tip [14], we have been able to map magnetic fields with a high spatial resolution by relying on the magnetic force interaction between the magnetized tip and the sample. Fig.10 shows the magnetic image over a thin film magnetic recording head of the type used in the high density magnetic storage system. The magnetic field is coming out of the left pole and entering back into the right pole. The enlargement obtained over the left pole demonstrates a spatial resolution of 100 nm.

## Conclusion

The Scanning Thermal Profiler and the Atomic Force Microscope are two tip techniques among several others that have recently proven their capability to visualise or characterize electronic materials with spatial resolutions varying from 100 to a few nanometers. They could become alternative techniques to the SEM when non-destructive criteria are severe and prevent the use of vacuum and electronic beams, or in micrometrology, when the measurement of edges, linewidth and profiles demands cannot be met by SEM. Tip techniques have also some unique capabilities such as the mapping of temperature, force, magnetic field or even charge, with high sensitivity and spatial resolution, which will make them attractive in microelectronics, as well as in other fields of science such as biology.

## Acknowledgements

We wish to thank Steve Rishton for the preparation of the electron resist grating, Bob Jackson for help with the scanning mechanics and Chris Jahnes and Russ Allen for the preparation of the thermocouple tips.

## References

1. Abraham DW, Sattler K, Ganz E, Mamin HJ, Thomson RE, Clarke J (1986) Direct Imaging of Au and Ag clusters by Scanning Tunneling Microscopy, J.Appl.Phys. 49 853-859.
2. Betzig E, Lewis A, Harootunian A, Isaacson N, Kratschmer E, (1986) Near Field Scanning Optical Microscopy (NSOM), Biophys.Journal 49 ,269-279.
3. Binnig G, Gerber Ch, Stoll E, Albrecht T, Quate C (1987) Atomic Resolution with Atomic Force Microscope, Europhys.Lett.(Switzerland) 3 , 1281-1286.
4. Binnig G, Rohrer H, Gerber Ch, Weibel E (1982) Tunneling Through a Controllable Vacuum Gap, Appl.Phys.Lett. 40 , 178-180.
5. Binnig G, Rohrer H, Gerber Ch, Weibel E (1983) 7x7 Reconstruction on Si(111) Resolved in Real Space, Phys.Rev.Lett. 50 , 120-124.
6. Binnig G, Quate CF, Gerber Ch, (1986) Atomic Force Microscope, Phys.Rev.Lett. 56 ,930-933.
7. DeLaRue RM, Humphreys RF Mason IN, Ash EA (1972) Acoustic-surface-wave amplitude and phase measurements using laser probes, Proc.IEE 119 ,117-126.
8. Durig U, Gimzewski JK, Pohl DW, (1986) Experimental observation of forces acting during scanning tunneling microscopy, Phys.Rev.Lett. 57 ,2403-2406.
9. Durig U, Pohl DW, Rohner F, (1986) Near-Field Optical Scanning Microscopy, J.Appl.Phys. 59 , 3318-3327.
10. Hamers RJ, Tromps RN, Demuth JE (1986) Surface Electronic Structure of Si(111) (7*7) Resolved in Real Space, Phys.Rev.Lett. 56, 1972-1975.
11. Israelachvilli JN(1985) Intermolecular and Surface Forces,Acad.Press, London.

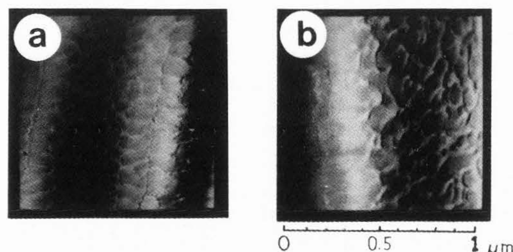


*Fig. 7:* Views of small areas of a silicon sample obtained by AFM. a)unetched and covered with a protective oxide layer b)etched with an anisotropic etchant. Small terraces having sizes of a few 10 nm are seen, which result from the etching process.

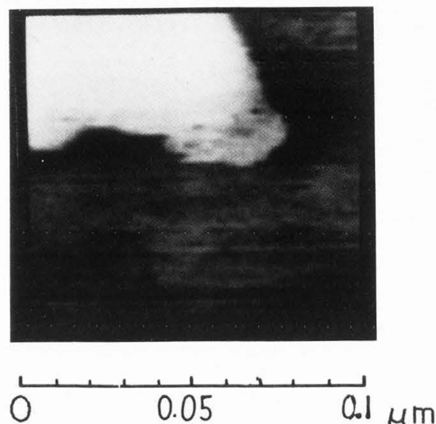


*Fig. 8:* View of a 5 nm high step on silicon by AFM.

*Fig. 9:* Image of a photoresist grating by AFM; line-width = line spacing = 100 nm, photoresist thickness = 90 nm.

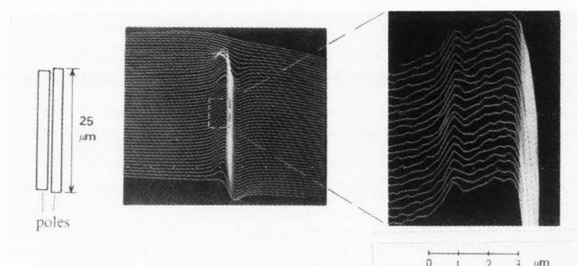


*Fig. 10:* Magnetic image of an IBM 3380 thin film recording head, obtained by AFM and using a magnetized iron tip instead of the tungsten tip. On the center image, one sees the positive and negative field gradients on the left and right poles respectively; on the left is a scaled top view of the head poles; the right image is a high magnification of the left pole.


12. Jarrell JA, King JG, Mills JW (1981) A Scanning Micropipette Molecule Microscope, Science 211 ,277-279.
13. Mamin HJ, Ganz E, Abraham DW, Thompson RE, Clarke J (1986) Contamination mediated deformation of graphite by the Scanning Tunneling Microscope, Phys.Rev.B 34 , 9015-9018.
14. Martin Y, Wickramasinghe HK (1987) Magnetic imaging by "force Microscopy" with 1000 angstrom resolution, Appl.Phys.Lett 50 , 1455-1457.
15. Martin Y, Williams CC, Wickramasinghe HK (1987) Atomic force microscope- force mapping and profiling on a sub 100 angstrom scale, J.Appl.Phys. 61 ,4723-4729.
16. Matey JR, Blanc J J.(1985) Scanning capacitance microscopy, J.Appl.Phys. 57 ,1437-1444.
17. McClelland GM, Erlandsson R, Chiang S (1987) Atomic Force Microscopy: General Principles and a New Implementation, Review of Progress in Quant. Non-Destructive Evaluation, Vol.6B, Thompson DO, Chimenti DE (eds), Plenum, New York, 1307-1314.
18. Muralt P, Pohl DW (1986) Scanning Tunneling Potentiometry, Appl.Phys.Lett. 48 ,514-516.
19. Royer D, Dieulesaint E, Martin Y (1985) Improved Version of a Polarized Beam Heterodyne Interferometer, IEEE Ultrason.Symp. Proceedings, McAvoy BR (ed), IEEE New-York, 432-435.
20. Williams CC, Wickramasinghe HK, (1986) Scanning Thermal Profiler, Appl.Phys.Lett. 49 ,1587-1589.
21. Williams CC, Wickramasinghe HK, (1986) High Resolution Thermal Microscopy, IEEE 1986 Ultrason. Symp. Proceedings, McAvoy BR (ed), IEEE New York, 393-397.


## Discussion with Reviewers

H. Van Kempen: Could the authors indicate the expected (theoretical or practical) resolution limit of the STP and AFM?
Authors: We believe that atomic resolution may be achievable with the STP. Although the classical notion of heat is not defined on this scale, energy transfer does exist, and may provide the means for atomic resolution. Such resolution has already been achieved by the Atomic Force Microscope (Reference 3).

U. Fischer: At which scan speed were the STP and AFM images recorded?
Authors: At present, a typical scan rate for both STP and AFM is several seconds per line scan.

U. Fischer: What limits are there principally to the speed of STP?
Authors: The ultimate limit to the speed of the STP is the thermal time constant of the thermal probes. At present, the

time constant is on the order of 1 microsecond. However, if speed is desired, smaller tips can be made with response times below 10 nanoseconds.

H. Van Kempen: What is the bottleneck for increasing resolution and sensitivity?
Authors: The limit for increasing resolution is tip size and interaction strength. In the case of the STP, probes with smaller dimensions can be made. The ultimate limit is unknown. The sensitivity is limited by the materials used to make the thermal probe tips. The sensitivity of the AFM is limited by the excitation of the tip lever by thermal noise. Cooling the tip would provide an increased sensitivity.

U. Fischer: Why is a decrease in amplitude associated with an attractive force?
Authors: The decrease in the vibration amplitude is caused by the fact that the tip is being driven above its natural resonance frequency. Since the attractive interaction force increases as the tip approaches the surface, the effective spring constant of the lever is lowered. With a weaker spring constant, the natural resonance frequency of the lever is lowered and the fixed drive frequency becomes further separated from resonance. This produces a lower vibrational amplitude.

P. Muralt: How do you determine the flying height of the STP-probe, as was done for Fig. 2?
Authors: The fly height of the thermal probe can be determined by allowing the tip to actually touch the sample surface, and piezoelectrically measuring the distance as it is brought back to the operating condition.

P. Muralt: How does the heat flow vary in function of the gap distance?
Authors: Initial measurements have shown that the heat flow varies rapidly with distance. These measurements will be published elsewhere, as they do not fit the scope of the present paper.