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Non-Contact Atomic-Level Interfacial Force Microscopy

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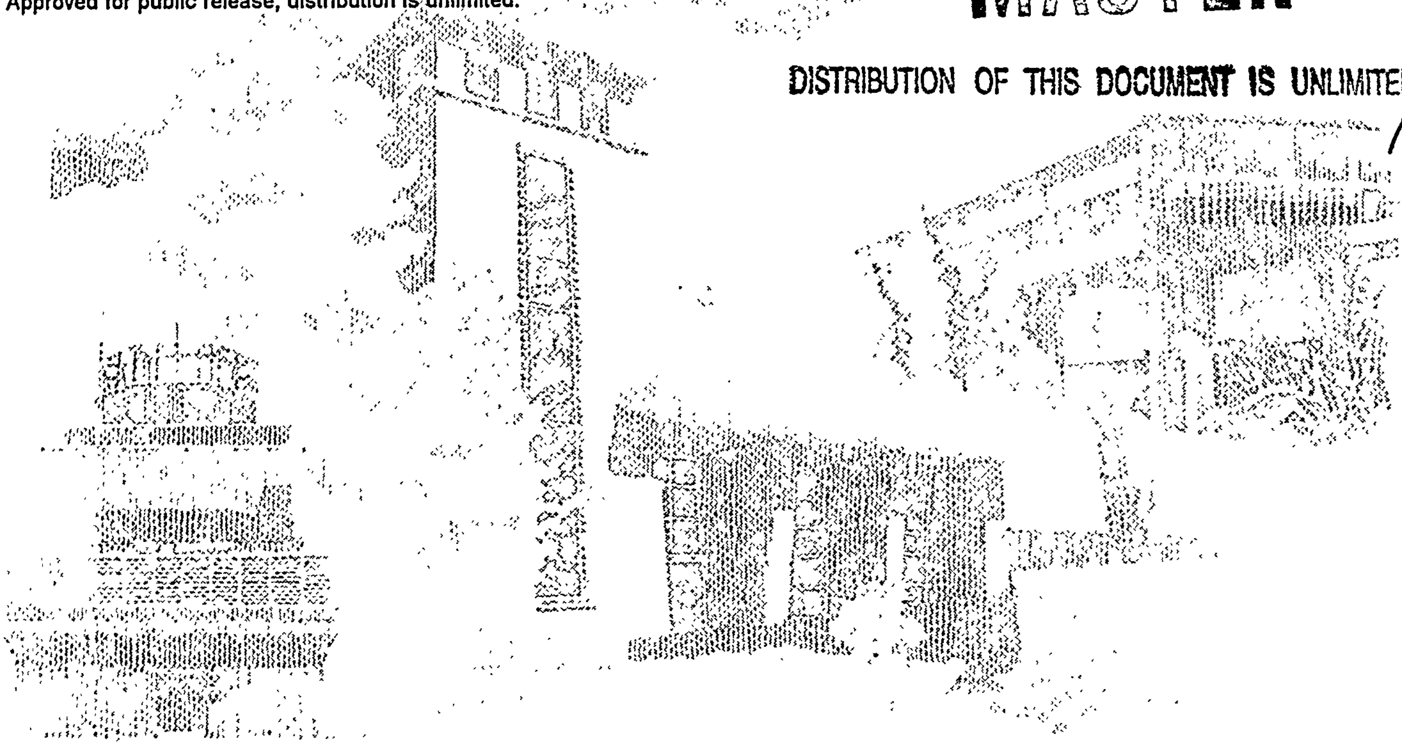

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Non-Contact Atomic-Level Interfacial Force Microscopy

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Abstract

Since the introduction of the Scanning Tunneling Microscope in the early 1980's, scanning-probe microscopies have developed very rapidly and are presently widely used in remarkably diverse applications ranging from research into fundamental material-science problems, through nanoscale fabrication and characterization to advanced metrology at the nanometer scale. The scanning force microscopies (notably the Atomic Force Microscope--AFM), because of their applicability to nearly all materials, are presently the most widely used of the scanning-probe techniques. However, the AFM uses a deflection sensor to measure sample/probe forces which suffers from an inherent mechanical instability that occurs when the rate of change of the force with respect to the interfacial separation becomes equal to the spring constant of the deflecting member. This instability dramatically limits the breadth of applicability of AFM-type techniques to materials problems. In the course of implementing a DOE sponsored basic research program in interfacial adhesion, a self-balancing force sensor concept has been developed and incorporated into an Interfacial Force Microscopy (IFM) system by Sandia scientists. This sensor eliminates the instability problem and greatly enhances the applicability of the scanning force-probe technique to a broader range of materials and materials parameters. The impact of this Sandia development was recognized in 1993 by a Department of Energy award for potential impact on DOE programs and by an R&D100 award for one of the most important new products of 1994. However in its present stage of development, the IFM is strictly a research-level tool and a CRADA was initiated in order to bring this sensor technology into wide-spread availability by making it accessible in the form of a commercial instrument. The partners in the CRADA were selected to complement Sandia's strengths and included: Digital Instruments, Inc. (DI)--presently the largest manufacturer of Atomic Force Microscopes; AT&T Bell Laboratories--who had taken our concept and incorporated it, with considerable success, into a general metrology setting; and the University of New Mexico (UNM), which had achieved wide recognition for the development of "super tips"--probes for scanning-force microscopies which are extremely sharp (only tens of atoms wide at their tip). The present report describes the goals, approach and results of this CRADA effort.

Summary

The ultimate goal of the IFM CRADA involved the development of an advanced IFM sensor incorporating state-of-the-art fabrication techniques, which was fully tested in a metrology environment, and to transfer this technology to the manufacturing partner for commercialization. Sandia was to handle the sensor development, AT&T was to test the sensors in a realistic metrology setting, DI was responsible for testing the sensors in their commercial instrument, in order to determine compatibility and to ascertain the ultimate feasibility of commercialization, and UNM was responsible for advanced probe development. Two critical criteria were implicit in the commercialization tech-transfer process for it to be fully successful: the first was that the sensor be manufacturable at low cost and the second was that a viable probe be included as an integral part of the manufacturing process. Thus, the program was immediately faced with the design of a new sensor configuration, the process for its fabrication as well as the design and processing steps for the inclusion of an integral probe. Because of Sandia's experience and unique facilities with surface micromachining techniques, it was decided to accomplish the design and establish the process at the Microelectronic Development Lab (MDL--a class one prototyping facility). In a parallel effort, a second generation IFM sensor similar to the original prototype was to be designed and fabricated in Sandia's Compound Semiconductor Research Laboratory (better suited for quick turn around, prototyping activities) in order to permit initial testing both in the metrology instrumentation and the commercial DI equipment. To facilitate the latter, and to eliminate the delays involved in the multiple handling of the sensors, DI provided Sandia with one of their commercial AFM instruments for retrofitting with the IFM-type sensors. This permitted the immediate testing of newly fabricated sensors and the determination of any problems involved with the lack of compatibility with the commercial instrument. In addition, a more sophisticated set of IFM control electronics was to be developed which operated at a higher frequency in order to compensate for the smaller capacitance values anticipated in the new sensors.

In the early stages of the CRADA effort, DI expressed an intense interest in having Sandia develop the process for producing integral probes mounted on cantilevers as a possible replacement for their present "tapping-mode"® AFM sensors. They explained that their business (and that of the entire scanning probe industry) was at present virtually held captive by their single foreign supplier of these sensors. The development of the process for including probes on test-bed sensors was a success. The process permits the batch

fabrication of probe tips which are sharper and considerably more consistent in shape and structural integrity than were the commercially available batch fabricated parts at the time. In addition, several US foundries were identified which could handle processing. The retrofitting of prototype and second generation IFM sensors to the commercial DI AFM instrument was accomplished during the beginning of the second year of the program with very satisfying results. In addition, several prototype surface-micromachined sensor structures were produced. However, the CRADA time period expired before the process for a viable combination of advanced sensor and incorporated probe could be established.

Non-Contact Atomic-Level Interfacial Force Microscopy

Introduction

The scanning force microscopies (notably the Atomic Force Microscope--AFM), because of their applicability to nearly all materials, are presently the most widely used of an important class of scanning probe techniques. And, while these techniques are making a significant impact on advanced-materials characterization, they are limited by the use of an inherently unstable deflection-based force sensor. Sandia's recent development of a stable force sensor, and its subsequent incorporation into an Interfacial Force Microscopy (IFM) system, has demonstrated a dramatic increase in the range of applicability of scanning force-probe techniques. In the interest of increasing the use of this important new technique, Sandia entered into a CRADA agreement in order to bring this sensor technology into wide-spread availability by making it accessible in the form of a commercial instrument. The partners in the CRADA were selected to complement Sandia's strengths and included: Digital Instruments, Inc. (DI)--presently the largest manufacturer of Atomic Force Microscopes; AT&T Bell Laboratories--who had taken our concept and incorporated it, with considerable success, into a general metrology setting; and the University of New Mexico (UNM), which had achieved wide recognition for the development of "super tips"--probes for scanning-force microscopies which are extremely sharp (only tens of atoms wide at their tip). The present report describes the goals, approach and results of this CRADA effort.

Background

Since the introduction of the Scanning Tunneling Microscope in the early 1980's [1], scanning-probe microscopies have developed very rapidly and are presently widely used in remarkably diverse applications ranging from research into fundamental material-science problems, through nanoscale fabrication and characterization to advanced metrology at the nanometer scale.

In addition, the spectrum of materials under study by these techniques is equally broad covering the range from biological systems to nano-level precipitation hardening in metal alloys. The scanning force microscopies (notably the Atomic Force Microscope--AFM [2]), because of their applicability to nearly all materials, are presently the most widely used of the scanning probe techniques. In conventional instruments in this class, the interaction force between the probe and the surface of the material under study is detected by a deflection force sensor. For example, in the normal AFM, the sensor consists of a micromachined cantilever which deflects under the influence of the probe-surface force. The deflection is usually detected by the movement of a laser beam reflected off the cantilever and the amount of deflection determines the interfacial force [3]. However, all force sensors of this type suffer from an inherent mechanical instability which occurs when the rate of change of the force with respect to the interfacial separation becomes equal to the spring constant of the cantilever. Under these conditions, the probe violently "snaps" into contact with the surface, similar to what happens when kitchen magnets are held in each hand and slowly brought together; at some point the magnets cannot be held apart and will suddenly snap into contact. This inherent sensor instability has dictated that AFM-type instruments are limited to imaging surfaces only under contact (surface profilometry) and that the interfacial force can only be mapped with respect to separation over a limited range.

In the course of implementing a DOE sponsored basic research program in interfacial adhesion, a self-balancing force sensor concept was developed and incorporated into an Interfacial Force Microscopy (IFM) system by Sandia scientists [4]. This sensor eliminates the instability problem and greatly enhances the applicability of the scanning force-probe technique to a broader range of materials and materials parameters. The impact of this Sandia development was recognized 1993 by a Department of Energy award for potential impact on DOE programs and an R&D100 award for a most important new product of 1994. However in its present stage of development, the IFM is strictly a research-level tool and the present CRADA was initiated in order to bring this sensor technology into wide-spread availability by making it accessible in the form of a commercial instrument. Sandia's unique position in implementing such a program included the initial invention, development and application of the sensor as well as a broad expertise in the fabrication of advanced sensors, especially small-scale devices in a micromachining, fabrication-lab setting. The partners in the CRADA were selected to complement Sandia's strengths and included: Digital Instruments, Inc. (DI)--presently the largest manufacturer of Atomic Force Microscopes [5]; AT&T Bell Laboratories--who had taken our concept and incorporated it, with considerable success, into a general metrology setting [6]; and the University of New Mexico (UNM), which had achieved wide recognition for the development of "super tips"--probes for scanning-force microscopies which are extremely sharp (only tens of atoms wide at their tip) [7].

Results and Conclusions

Surface Micromachining Prototypes

The ultimate goal of the present CRADA involved the development of an advanced IFM sensor incorporating state-of-the-art fabrication techniques, which was fully tested in a metrology environment and the transfer of this technology to the manufacturing partner for commercialization. Sandia was to handle the sensor development, UNM was responsible for advanced probe development, AT&T was to test the sensors in a realistic metrology setting and DI was responsible for testing the sensors in their commercial instrument to determine compatibility and to ascertain the ultimate feasibility of commercialization. Two critical criteria were implicit in the commercialization tech-transfer process for it to be fully successful: the first was that the sensor be manufacturable at low cost and the second was that a viable probe be included as an integral part of the manufacturing process. The overall program was to draw heavily in its initial stages on Sandia's accumulated technical experience with the IFM sensor which was in use at the time. However, these prototype sensors were hand made, not readily manufacturable and did not include an integral probe (the probes were hand attached subsequent to sensor assembly). Thus, the program was immediately faced with the design of a new sensor configuration, the process for its fabrication as well as the design and processing steps for the inclusion of an integral probe. Because of Sandia's experience and unique facilities with surface micromachining techniques, it was decided to accomplish the design and establish the process at the Microelectronic Development Lab (MDL--a class one prototyping facility). In this approach, surface micromachining was to be used to fabricate capacitive drive and sense elements as well as the integrated tip. Since the topography is not extreme, integration with on-chip CMOS processing also appeared to be possible.

In the course of the first 18 months of the CRADA work, several interesting and potentially useable test structures were fabricated using micromachining techniques. The general design was based on a spring-mounted shuttle. The design is schematically illustrated in Fig. 1. The shuttle is suspended above the substrate by long, thin leaf springs and a model tip is placed on one end of the shuttle and is part of the fabrication process. Two sets of interlaced comb-drive assemblies are located on either side of the shuttle and are designed to act as both the electrostatic drive mechanisms for the force-feedback control and as the differential-capacitance displacement sensors. Since electrostatic forces are only attractive, the combs are interlaced slightly off center--to the right on one end and to the left on the other--in order to be able to produce both positive and negative electrostatic forces on the shuttle. Several different modifications of the design were produced having the same overall configuration but differing in the details of the fabrication. This design had several appealing features as a force-feedback sensor. First and foremost was the fact that it was ultimately manufacturable at low cost. In addition, the shuttle-displacement spring constant was very low, which implies that the sensor would be sensitive to very small forces applied along the axis of the shuttle, i.e., on the tip, while having a large in-plane spring constant normal to the shuttle axis.

However, there were several problems encountered with this approach. The most serious was the fact that the stray capacitance between the signal connects and the substrate dominated the small capacitance values of the comb-drive elements. This meant that the signals resulting from a small displacement of the shuttle/ comb-drive structure, which was driven by high-frequency ac voltages, would be buried in a sea of unwanted components at the same frequencies. This problem is inconsistent with high displacement sensitivity. A second problem involved the fact that the fabrication steps for ultimately fabricating viable tips in the plane of the shuttle was not obvious. Other fabrication problems included: (1) the comb-drive arms, for a high-capacitance configuration, were flimsy along the shuttle axis and would sometime snap into contact with the stationary elements under only a small bias voltage and (2) applying a voltage on the comb drives causes a out-of-plane forces on the shuttle.

As a result of these problems, it became clear that the design philosophy of the present generation of sensors (which consist of a differential-capacitor design requiring radio frequencies for the detecting of small displacements) did not fit well with the surface micromachining of parts that were perhaps only a few hundred μm in size (the prototype units for the adhesion program are some 25 times larger). Although some of the problems appeared to have possible fabrication solutions, it was clear that the use of high-frequency displacement detection was not compatible with silicon-based surface micromachining. The use of this very appealing type of fabrication will have to wait on a more advanced displacement-detection technique. Examples of such techniques include: incorporated laser-diode cavity interferometric schemes or the use of integral field-emission probes. Both of these lend themselves to the scaling necessary to operate on the dimensional level required for micromachined sensors and do not suffer from the need to use high-frequency deflection-detection schemes.

The second part of the work undertaken at the MDL was the fabrication of separate components for an IFM sensor, specifically the plate suspended by springs onto which is fabricated an integral tip. Such parts were successfully fabricated using "mold" micromachining techniques. Mold micromachining is an emerging technology that enables the batch fabrication of relatively thick plates with the deposition of relatively little structural material. The basis of the technique is shown in Fig. 2. First a mold is formed by trench etching into single-crystalline silicon. This forms the array of closely spaced pillars which forms the mold. The thickness and therefore stiffness of the plate is determined by the depth of the etch. The mold is then filled with low stress silicon nitride. The silicon nitride is then patterned to open holes to allow removal of the single crystalline Si mold material by KOH. A integral tip is added to the plate using a process similar to that discussed below with respect to the fabrication of "Tapping Mode" sensors. Examples of finished parts are given in Fig. 3. More details on this process are suppled in reference [8].

Second-Generation IFM Sensors

In a parallel effort, a second-generation IFM sensor similar to the original prototype was to be designed and fabricated in Sandia's Compound Semiconductor Research Laboratory (better suited for quick turn around, prototyping activities) in order to permit initial testing both in the metrology instrumentation and the commercial DI equipment. In addition, a more sophisticated set of IFM control electronics was to be developed which operated at a higher frequency in order to compensate for the smaller capacitance values anticipated in the new sensors. To facilitate the overall testing of these developments, and to eliminate the delays involved in the multiple handling of the sensors, DI provided Sandia with one of their commercial AFM instruments for retrofitting with the IFM-type sensors. This permitted the immediate testing of newly fabricated sensors and the determination of any problems involved with the lack of compatibility with the commercial instrument.

In this second-generation sensor design, an attempt was made to correct several of the defects encountered in the prototype. To illustrate some of these difficulties, we show in Fig. 4 schematic diagrams of the original prototype sensor. The common plate of the differential capacitor (Fig. 4a), which is the heart of the force-feedback IFM sensor, was chemically milled into a 0.005 " thick BeCu sheet. This "teeter totter" element was suspended by narrow torsion bars which were dimensioned, as much as possible, to give a weak torsional mode while maintaining as stiff an up/down deflection mode as practical. The teeter totter element was then glued (by hand, using strain-gauge cement [9]) to a substrate consisting of a 0.040" glass plate. The substrate (Fig. 4b) was chemically etched such that the two subsequently deposited capacitor pads and their leads resided in a pit which, after assembly, defined the capacitor gaps. The capacitor-gap was usually of the order of 5 μm, resulting in capacitor values of a few pF. Electrical leads were then either indium soldered or silver epoxied to the individual bonding pads. After assembly (Fig. 4c), the probe tip, which was normally a 0.004" W wire electrochemically etched to a tip radius of from 50-250 nm, was silver epoxied onto one end of the teeter totter. The weaknesses in this design included: (1) the fact that the individual-unit assembly was tedious, time consuming and unreliable (the M-Bond often delaminated after only limited use); (2) mounting the probes, again, was a tedious job and replacement was very difficult; and (3) the torsional "spring" constant was larger than desired and the ratio of this value to the up/down constant was not large enough (these difficulties resulted from inherent limitations in the chemical-milling process).

In the second-generation design, common-plate configurations were laid out at the wafer level in 0.003" Alloy 42 (Kovar). Substrates were designed similar to the prototype unit except, again, at the wafer level. A schematic illustration of this design is shown in Fig. 5. The problem of the large torsional spring constant was corrected by using a serpentine spring design, which decreased the chemical milling tolerances and achieved a small torsional constant by effectively increasing the spring length. The resulting weakening of the up-down spring constant was corrected by including a fulcrum in the etching mask for the substrate capacitor-gap pit. The problem with the assembly was approached by using a thin-film indium soldering technique. The overall result was a design which could be reliably assembled 24 units at a time. Unfortunately, the development of this second-generation design took two years and it is just now in the initial stages of testing. The initial tests indicate that these units are robust and have the highest sensor-gain values that we have yet encountered. These sensors appear to be capable of satisfying the low-cost criterion of the IFM CRADA but still have the remaining problem that the probes have to be hand attached to the common-plate elements after sensor assembly.

Tapping Mode Sensor/Probe Process Development

After the CRADA had started, and DI became more familiar with the full range of Sandia capabilities, other areas of possible collaboration were identified. One such area of vital interest to DI involved the production of integral probes mounted on cantilevers as a possible replacement for their present "tapping-mode" ® AFM sensors. They explained that their business (and that of the entire scanning probe industry) was at present virtually held captive by their single foreign supplier of these sensors. In addition, the sensors were not of consistently high quality and DI was interested in exploring the development of a process that could be handled by a reasonably well equipped US facility. (The difficulty here is that the extraordinary fabrication facilities available for advanced electronic-component manufacturing (e.g., Intel, Motorola, etc.) are not available for low-volume electromechanical-device fabrication.) Sandia agreed to honor DI's request, since a similar process would have to be developed in the course of the CRADA-sensor work. Since Sandia cannot commercially manufacture the sensors, it was agreed that the Lab's role would be limited to the process development and the identification of US foundries that would be able to handle the manufacturing. A good overview of current tip fabrication processes is contained in reference [10].

There are four major components to a tapping mode interfacial-force sensor "tip" (Fig. 6). The first is the tip itself. It should be narrow and relatively long, for example, ~100 nm in diameter by ~500 nm long. The tip should narrow down to a sharp point on the order of 10 nm radius of curvature. The tip in turn should be on the end of a more rigid shaft on the order of 5 μm high. The purpose of the shaft is to ensure that it is the tip, and not a corner of the cantilever that is interacting with the sample. The tip and shaft sit on a cantilever. In the Tapping Mode, the cantilever is driven to oscillate at its resonant frequency (usually hundreds of KHz) with amplitudes of several tens of nm. An interaction between the sample and the tip damps oscillatory motion and lowers its amplitude. Feedback electronics are arranged to keep the oscillation amplitude and the morphology is determined by the amount of z-axis piezo motion required to maintain this constant amplitude. In order to achieve the proper compliance and resonance frequency, the cantilever must be roughly 1 μm thick, 40 μm wide and 300 μm long. In addition, it is important that the cantilever material have stable mechanical properties and not be distorted by internal stresses. The materials typically used in this application are poly silicon and silicon-rich silicon nitride. Finally, there is a supporting handle which enables the sensor to be easily mounted on the piezo xyz manipulator. The handle is on the order of 0.5 mm thick, 1 mm wide and 3 mm long. In order to avoid interaction between the handle and the sample it is important that the handle be situated on the opposite side of the cantilever relative to the tip (Fig. 6).

There are two general approaches to the fabrication of these structures. The first involves the bulk removal of silicon, typically combined with a heavily doped boron KOH etch stop. In the second approach, a mold is used to form the tip. The cantilever material is deposited into the mold and then patterned (Fig. 7). The final steps of this process involve the bonding of the handle to the cantilever material followed by removal of the silicon mold. The mold process is widely understood and the bonding and removal processes are commonly employed in industry. In this work we have elected to concentrate on mold processing since it appears to be the approach most applicable to volume production.

The major problem with the technique is that it does not produce a sharp, high aspect-ratio tip. The mold is typically formed as a KOH pit which dictates that the tip and shaft angle is a constant 71°. Since the bonding and mold removal parts of the process have been amply proven to be manufacturable, we have concentrated on novel ways to modify the mold process to create a more optimal tip.

The first modification we developed involves a long oxidation process to enhance tip sharpness of KOH pits. This process takes advantage of several effects. The first is a slight difference in the oxidation rate between the {111} and {100} planes. The second is a reduction in the oxidation rate due to enhanced stresses generated at the intersection of two oxidizing planes. The third and probably most important is the effective lengthening of the diffusion path through the oxide in the vicinity of the tip, Fig. 8. The thermal oxidation process employed was 16 hours in steam at 1050°C. Following formation of the mold, either 0.8 μm of silicon nitride, or 2 μm of low stress poly silicon were deposited and patterned to form the cantilever. The parts were then protected with SiN and the mold was partially removed. The bulk micomachined handles were then added and the reminder of the mold was removed. Micrographs of various views of the parts generated are given in Fig. 9. Note that the long cantilever has virtually no curvature indicating that the fabrication has resulted in a negligible level of residual stress. An example of a test sample scanned with one of these tips is given in Fig. 10. The major problem encountered in the fabrication of complete parts was the bonding of the handle to the cantilever. As was mentioned above, this is a well understood problem in industry and our problems stemmed from a lack of the necessary anodic bonding equipment. In the final phase of this part of the project we identified two small foundries which appeared to be capable and willing to fabricate this type of part.

With the limitations on Sandia's efforts with respect to manufacturing mentioned above, the development of the process for including probes on test-bed sensors was successful. The performance of some of the completed tips was investigated by DI using gold on graphite test structures (Fig. 10) which were found to be superior to the presently used units. Sandia then identified two small foundries, Seaway Engineering and IntelliSense Corporation. The process flow was explained in detail to each company in separate facility visits. Both companies were judged to be capable of producing parts after a reasonable period of technology transfer and process development.

As a side part of this project we investigated the possibility of using a recently developed deep silicon trench etch process as a potential substitute for the KOH pit. The advantage of such an approach is that it would enable the fabrication of a much steeper "shaft" portion. A range of different molds were formed and fabricated. Examples of the tip shapes achievable are given in Fig. 11. The major problem with this approach is that the reactive ion etch process necessary is not widely available, especially in the small-foundry environment.

In addition, UNM provided Sandia with "super tips" for test on the DI instrument and these were shown to operate in satisfactory manner in the normal contact mode of surface imaging.

Commercial Instrument/IFM Sensor Retrofit Results

The retrofitting of prototype IFM sensors to the commercial DI AFM instrument was accomplished during the beginning of the second year of the program with very satisfying results. Not only does the inclusion of this sensor clear the way for nondestructive, noncontact surface imaging on the near-atomic scale, but it also opens up completely new areas of applicability dealing with the study of interfacial adhesion and the mechanical properties of materials both at the nanometer scale (over areas of only tens of atoms in diameter). In addition, these increased capabilities were obtained with only minor programming alterations of the operating software on DI's part. An example of one of these new capabilities is shown in Fig. 12a. In fact, DI is using an example similar to this in upcoming promotional material concerning unique user applications.

In Fig. 12a we show a repulsive-force image of the surface of a 500 nm Au film grown on a sputtered Si(001) surface. The xyz motion of the sensor is controlled by piezo tube manipulator. The x and y axis are rastored in a TV-like format while the z displacement is controlled in order to maintain a small, constant repulsive force between the probe tip and sample surface. The z motion is then translated into a color contrast on the image. In the figure, the xy scan is over a range of 500 nm square, representing a total of about 25,000 Au atoms, and the total z-axis range covers about 30 nm. The image clearly shows the grains of the polycrystalline Au film, which have an average diameter of approximately 90 nm. The surface roughness is measured to be about 10 nm, or about 20 Au atom diameters.

The experiment consists of positioning the probe tip at the top of the grain in the center of the image. A force profile (or loading curve) is then acquired by first retracting the probe 5 nm and then ramping toward the grain at a speed of 0.5 nm/sec while recording the interfacial force. This force profile is shown in Fig. 12b. The initial rise in force follows the classic Hertzian (or elastic) behavior up to the point marked by the vertical arrow. At this point, the loading deviates from elastic and signals the onset of some plastic motion (i.e., displacement that will not recover upon probe withdrawal). This plasticity continues up to a load of about 10 μN--one μN is approximately the force required to separate the two atoms in a NaCl (table salt) molecule--after which the probe is withdrawn at the same rate. The result of the plastic motion is reflected in the profile by the opening of a hysteresis loop in the plot--the z-width of the loop is measures the amount of the plastic motion, or about 0.5 nm in this case. The image in Fig. 13c show the physical result of the loading. Clearly, the center grain has been pushed into the surface. Repeated loadings to slightly higher peak loads give similar and progressive results shown in Figs. 12d through 12g and result in the center grain virtually disappearing from view. In addition, one can see that neighboring grains also move as a result of pushing the center grain into the surface. Note also that the hysteresis loop gradually narrows for the later profiles indicating that grain motion requires larger forces as the grain goes deeper into the surface. These results are evidence of a "super-plastic" behavior for this particular film and are a crucial element in understanding the fascinating properties of a new class of "nanophase" materials.

Other applications of the retrofitted instrument include: studies, in conjunction with the Beckman Laser Institute at the University of California at Irvine, of the surface of tooth enamel to determine the affect of laser ablation in achieving a considerable increase in the fluoridation passivation of ablated surfaces; a study of the nanomechanical properties of turkey-leg tendon in conjunction with work by the Dreyfus Dental Institute in Boston of the affect of osteoporosis on the structure and mechanical properties of bone (turkey-leg tendon is often used as a bone model); and an ongoing investigation of the potential of nanoindentation techniques to measure residual stress at the nanometer level--this study has the potential for major impact on the microelectronics industry where residual stress can play a devastating role in structural failure in electronic components (especially those required to have extended operating lifetimes). The response to this kind of work from the materials research community has been very strong and points to potential strength for marketing a system with an advanced, IFM-type sensor.

Summary and Conclusions

In summary, while all of the original goals of the CRADA were not met during the coarse of the agreement, progress in defining processing parameters and limitations have been significant, especially with respect to the processing of high-quality probe tips for sensor inclusion. All the parts were essentially in hand in the fabrication process described in conjunction with Fig. 3. Effectively, the parts shown in Fig. 3a take the place of the chemically milled common-plate structures in the original IFM sensor (discussed with respect to Fig. 4). If these common plate and probe-tip assemblies were subsequently wafer bonded to an <u>insulating</u> substrate, e.g., glass or quartz, the problems with the stray capacitances encountered in the surface micromachined method could be eliminated and a sensor with ac displacement detection and an integral tip could be mass produced. Unfortunately, time ran out on the CRADA program before such a wafer-bonding process could be developed. All things considered, this may very well represent the most viable initial solution to the manufacturable prototype problem. Despite the difficulties, however, our CRADA partners have indicated that they are pleased with the progress and are confident that viable testing units will be available shortly. DI indicates that there is a significant user interest in the additional capabilities afforded by the advanced IFM-type sensors and that they would definitely be interesting in pushing ahead with plans for commercialization if a low-cost, easily manufacturable design becomes available.

A second-generation IFM sensor was defined and fabricated which was designed to correct many of the problems with the original prototype. In addition, the concept included a wafer-level hybrid layout consisting of a metal sheet, into which the common plates of the capacitors were chemically milled, bonded to a glass substrate into which the capacitor gaps were etched and which included the metal deposition for differential-capacitor pads. The two components were assembled by a new thin-film soldering technique yielding 24 sensors per hybrid wafer assembly. The soldering scheme required more development time than anticipated and these units are just now coming into initial testing and application. The early testing results, however, are very encouraging. The sensors have 100 times higher gain values than the original prototype, are reliable and robust and are extremely stable against thermal drift, etc. We hope to be able to supply DI and AT&T with units for their testing in the very near future.

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Figure Captions

Figure 1. A scanning electron micrograph of one of the prototype surface micromachining force sensors. The long plank down the center is the shuttle which, along with two sets of finger-like combs on each side, is suspended above the substrate by the long, narrow springs on both ends of the shuttle. Two other sets of combs are interlaced with the shuttle sets on both sides and these are mounted on the substrate. The interlaced combs are offset slightly toward the right on one end and toward the left on the other. The two sets of substrate combs along the shuttle axis and the entire set of shuttle combs are electrically isolated. This allows the shuttle to be moved in one direction by placing a voltage between the shuttle and one substrate set and in the other by placing the voltage between the shuttle and the other set of substrate combs. A model probe tip is located on the right-hand end of the shuttle.

Figure 2. Schematic illustrating the basis of the mold fabrication approach. The mold is generated through the etching of high aspect ratio, closely spaced pillars. The mold is then filled with the structural material of interest, in this case silicon nitride. Since this material deposits on all surfaces, the amount of material needed to be deposited to fill the gaps between the pillars is relatively small. The thickness of the material is therefore dependent not on the thickness of the deposited material, but on the depth of the pillars.

Figure 3. Micrographs showing various view and magnifications of parts fabricated in the course of the work using the mold-fabrication technique. The common plate of the differential capacitor is suspended on torsional springs from a support structure. The probe tip is fabricated as an integral part of the common-plate fabrication so that the common plate, probe tip and torsion springs all exist as a single unit. To make a sensor, this unit would have to be bonded to a substrate which contained the individual capacitor pads, their electrical leads and bonding pads.

Figure 4. Schematic illustrations of the original prototype IFM sensor: (a) shows the capacitor common plate which, along with its torsion bars, were chemically milled into 0.005" BeCu sheet; (b) illustrates the substrate which consisted of two capacitor electrodes, their electrical leads and bonding pads deposited into a pit etched into the glass or quartz substrate. A third electrode was deposited on the substrate surface to make electrical contact with the common plate after assembly; (c) shows the final assembly with the common-plate structure glued to the substrate.

Figure 5. Schematic illustrations of the wafer-level, 24 unit, second-generation IFM sensor. The top drawings show the capacitor common-plate configuration which is chemically milled into a 0.003" Kovar sheet, as shown in the right-hand figure. The torsion springs have a serpentine structure to relieve milling tolerances and reduce the torsional spring constant. The bottom portion of the illustration shows the substrate layout, which at the individual unit level is essentially identical to that shown in Fig. Fig. 4. The exception is the inclusion of a "fulcrum" between the capacitor pads to compensate for the weak up-down compliance of the serpentine springs. The common plate rotates about the fulcrum in the manner of a teeter totter.

Figure 6. An schematic showing the component parts of a Tapping Mode sensor.

Figure 7. An illustration of the etch-pit mold process for fabrication integral probe tips for the Tapping Mode sensors. A mold is formed as a substrate etch pit which is then partially filled with the structural material used to form the tip, shaft and cantilever.

Figure 8. An illustration of the lengthening of the probe tip by increasing the mold stress in the vicinity of the apex. The slightly higher oxidation rate of {111}-type planes may also play a role.

Figure 9. Scanning electron micrographs of various views of the parts generated during work on the Tapping Mode sensors.

Figure 10. A Tapping Mode image of a Au on graphite test sample performed by Digital Instruments on their commercial Atomic Force Microscope using a Tapping Mode sensor fabricated during the CRADA work.

Figure 11. Example of a tip formed using the deep-silicon trench etch process. The tip has a much higher aspect ratio. However, the process requires equipment which is not readily available in the average foundry.

Figure 12. Repulsive-force images taken with the retrofitted IFM sensor on a Digital Instruments Nanoscope III commercial AFM microscope: (a) shows the image of a 500 nm Au film grown on a Si(001) surface; (b) illustrates the results of obtaining a loading curve by placing the probe tip above the center a the grain shown in the very middle of image (a) and ramping the tip into the grain at a constant rate to a force load of 1.6 μN and then backing it out at the same rate--the hysteresis loop indicates that a plastic motion of about 8 nm has resulted; (c) illustrates the physical result of this grain loading--the grain is seen to move into the surface by about 8 nm; (d) a second loading to a peak force of 2.3 μN and its associated post-loading image (e); (f) a third loading to a peak force of 2.8 μN and its associated post-loading image (g).

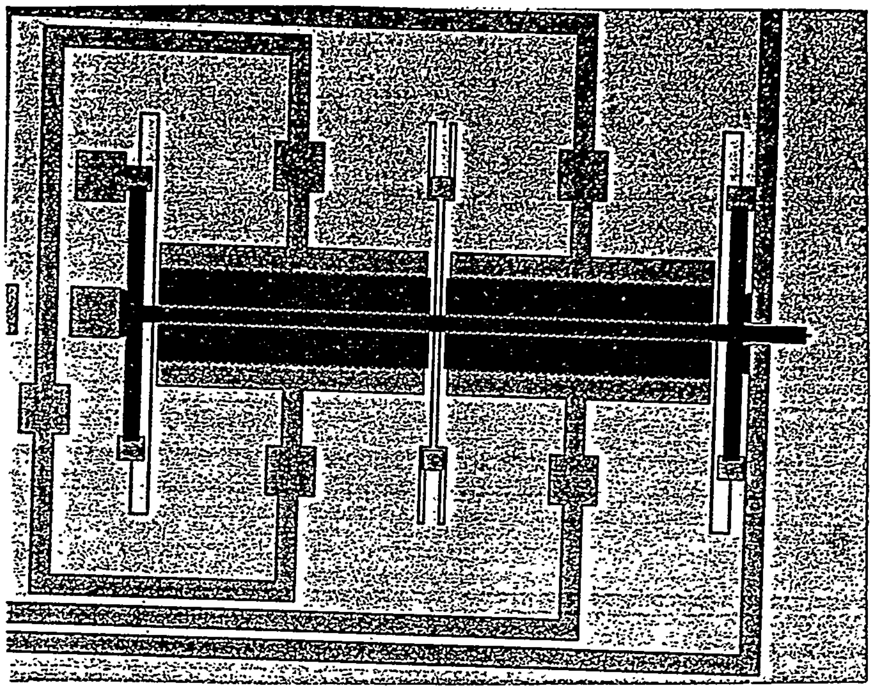
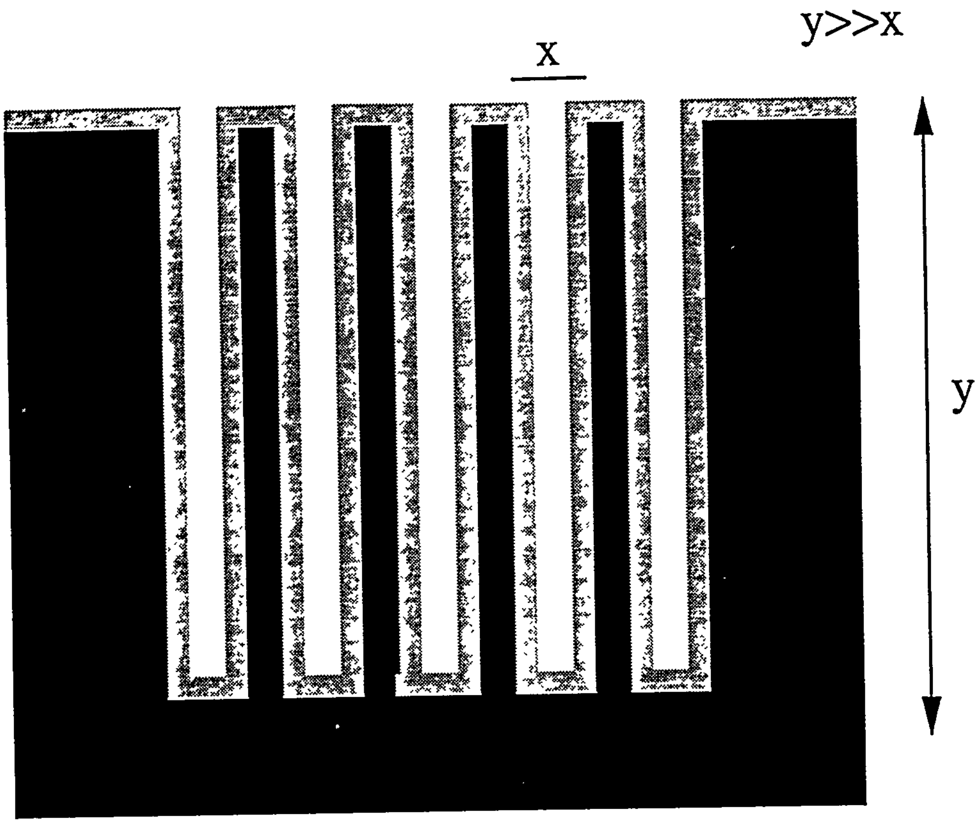
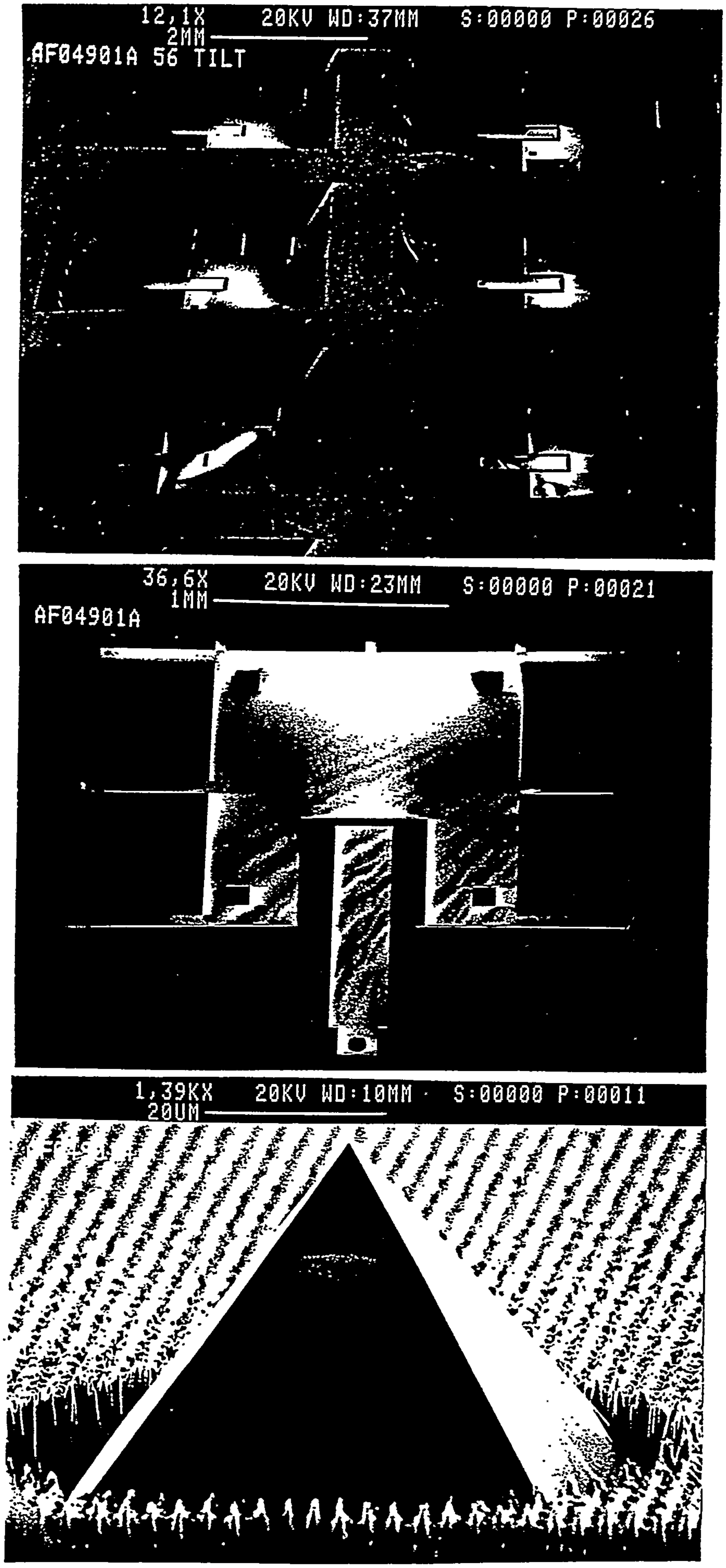


Figure 1



Amount of SiN needed to fill the gaps is x/2, independent of y

Figure 2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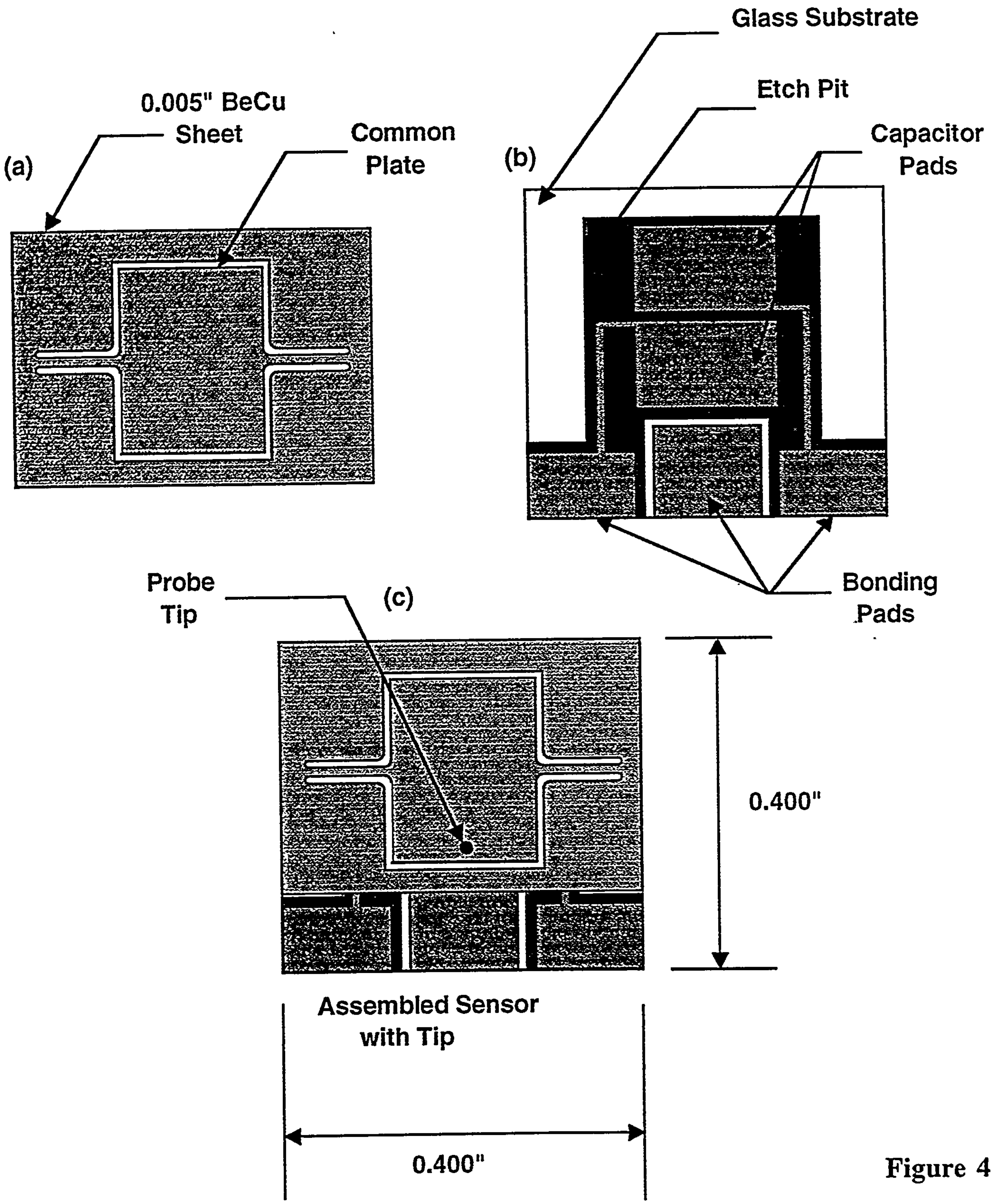
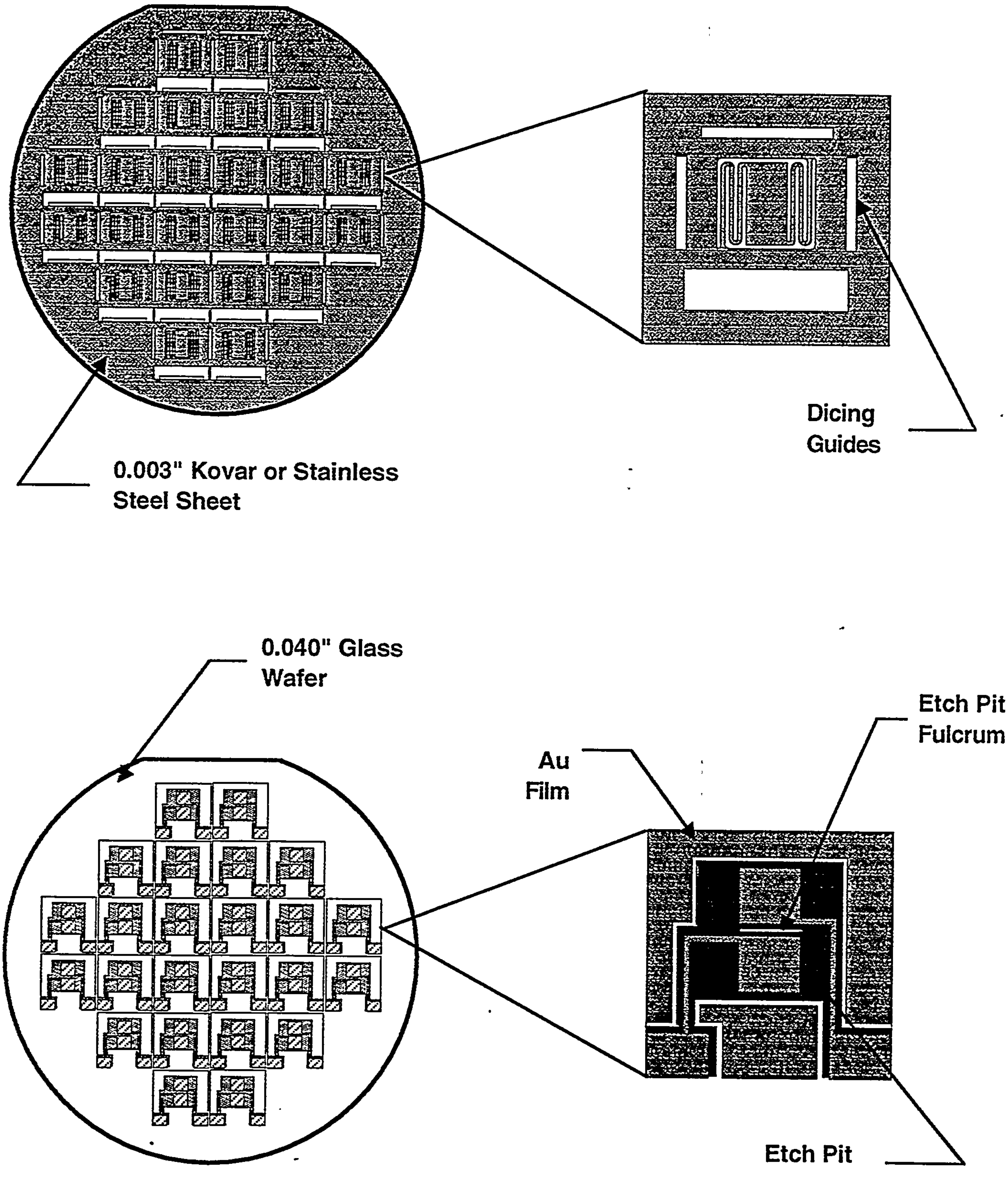
2MM
AF04901A 56 TILT
36,6X 20KV WD:23MM S:00000 P:00021
1MM
AF04901A
1,39KX 20KV WD:10MM S:00000 P:00011
20UM

Figure 3



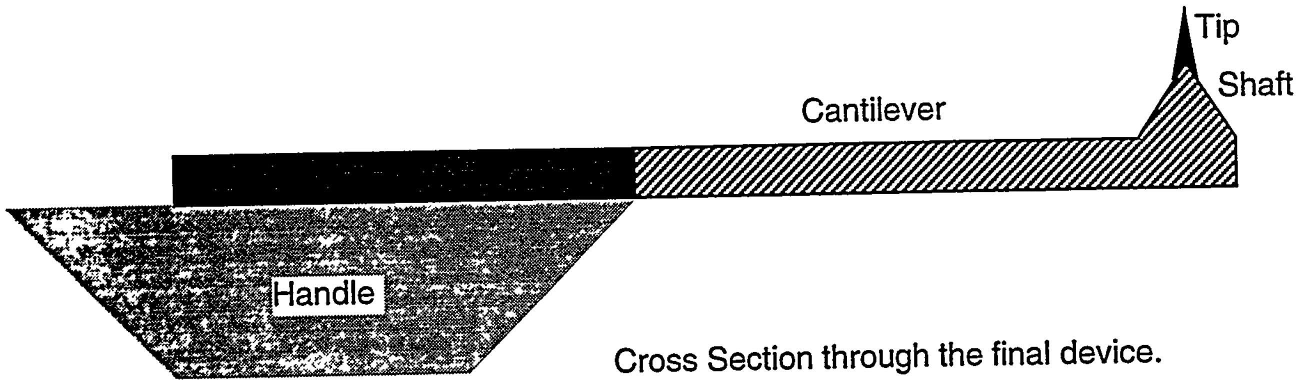
Glass Substrate
Etch Pit
0.005" BeCu
Sheet
Common
Plate
Capacitor
Pads
(a)
(b)
Probe
Tip
(c)
Bonding
Pads
0.400"
Assembled Sensor
with Tip
0.400"

Figure 4



Dicing
Guides
0.003" Kovar or Stainless
Steel Sheet
0.040" Glass
Wafer
Etch Pit
Fulcrum
Au
Film
Etch Pit

Figure 5



Cross Section through the final device.

Figure 6

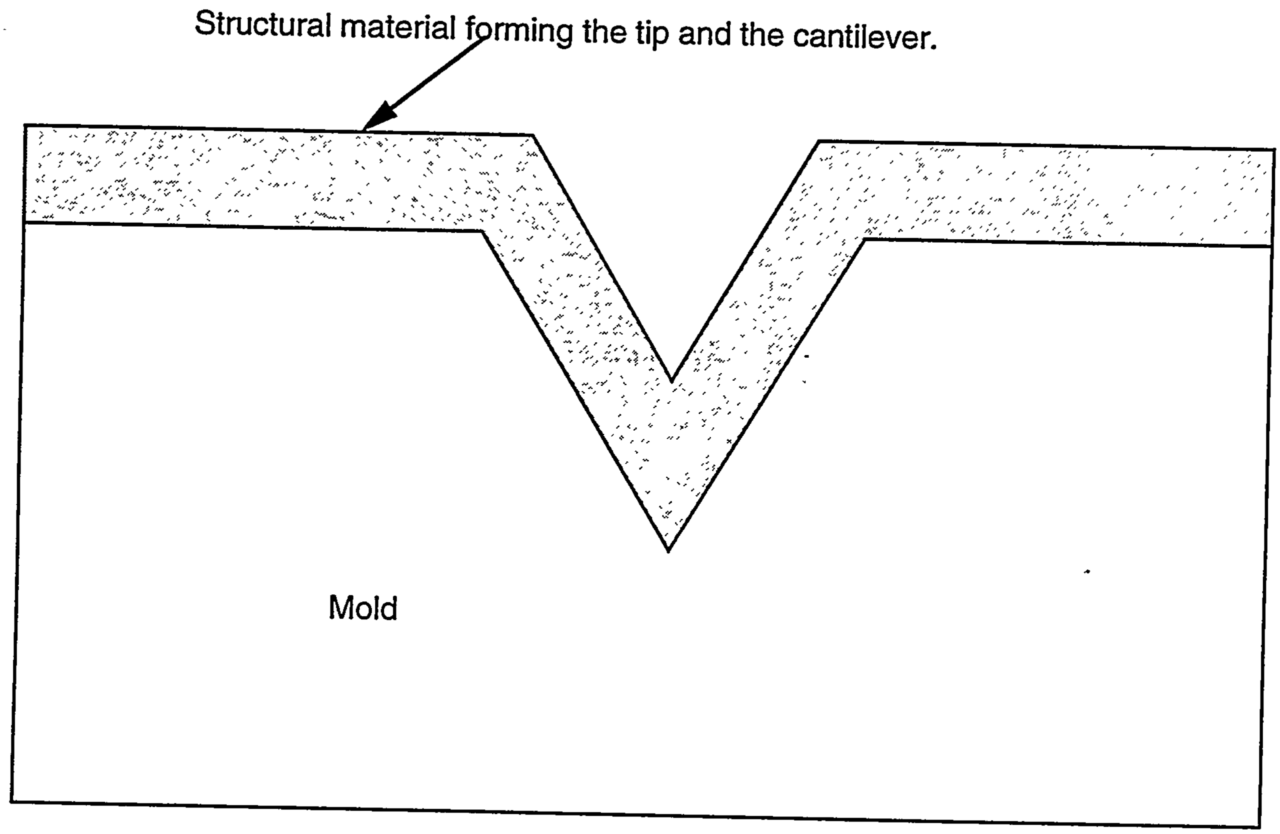

Structural material forming the tip and the cantilever.
Mold

Figure 7

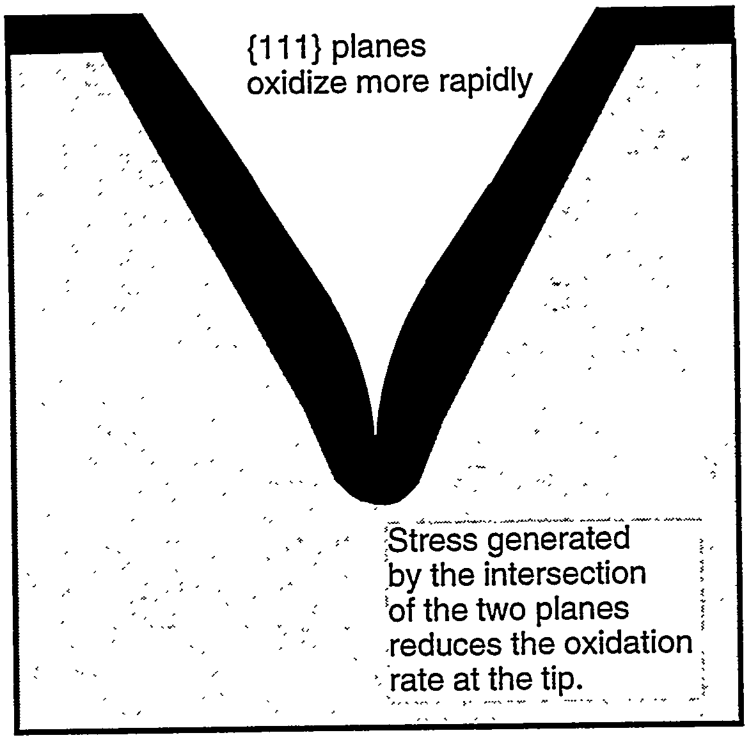
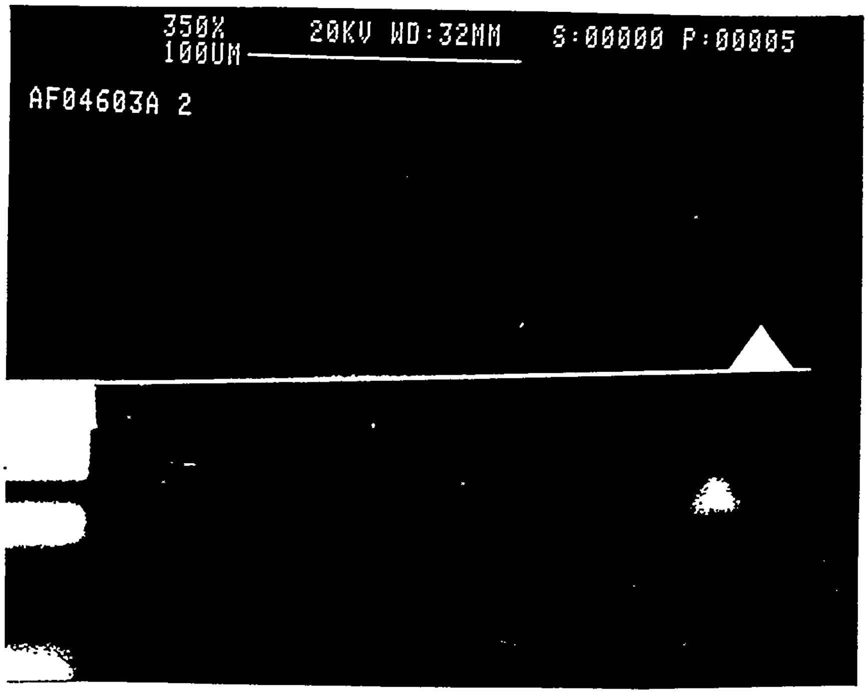

{111} planes
oxidize more rapidly
Stress generated
by the intersection
of the two planes
reduces the oxidation
rate at the tip.

Figure 8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100UM
20KV WD:32MM
S:00000 P:00005
AF04603A 2

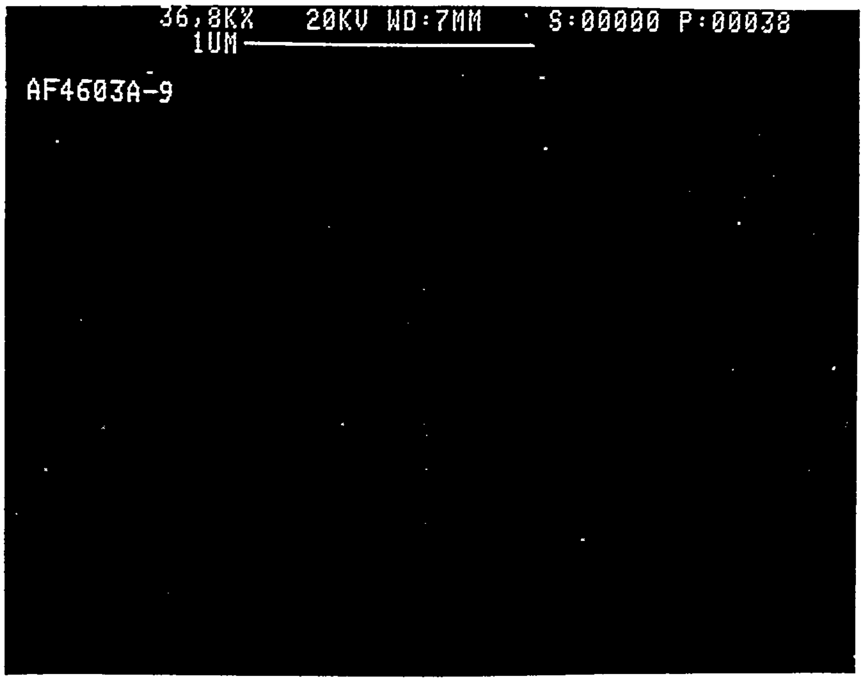
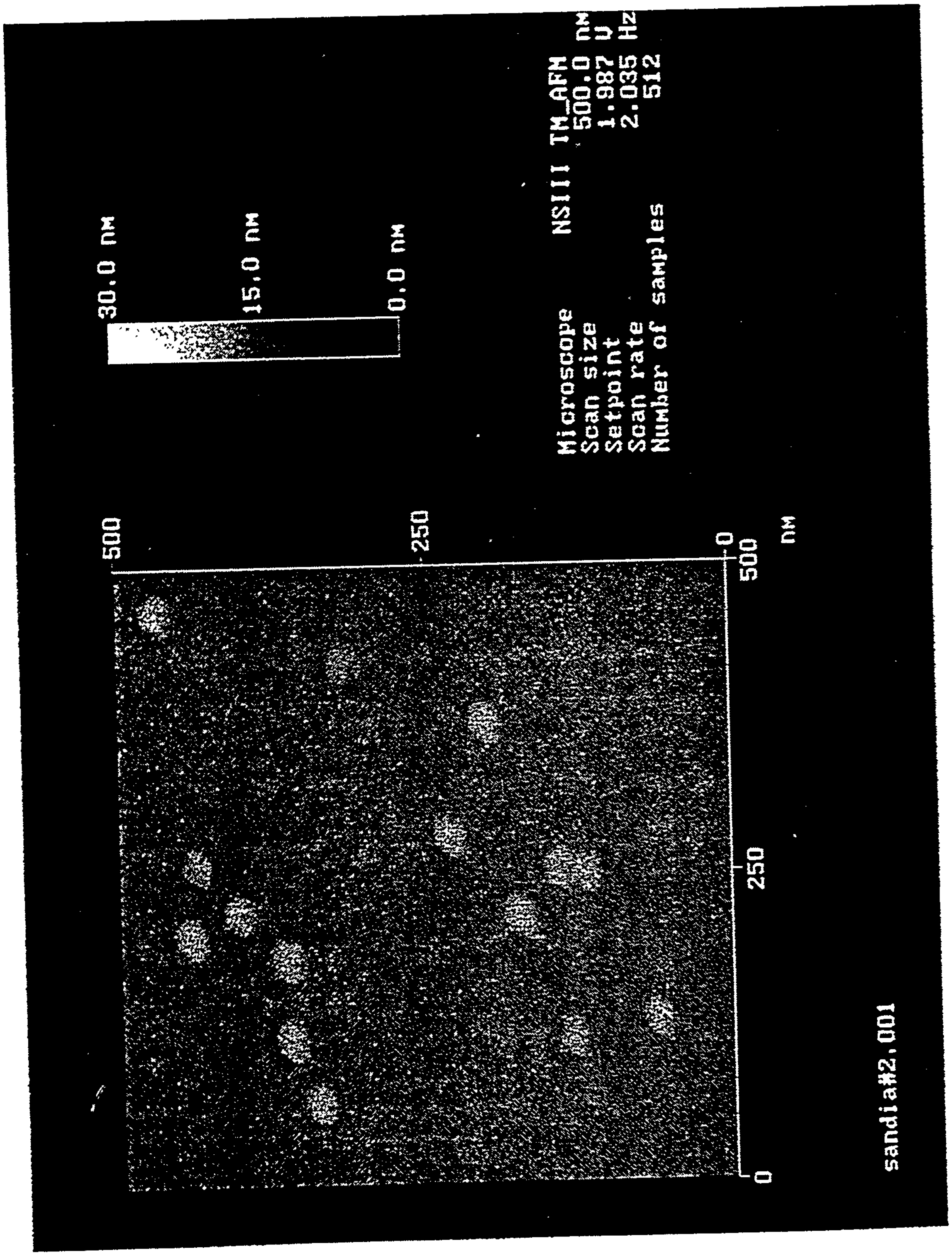

36,8KX
1UM
20KV WD:7MM
S:00000 P:00038
AF4603A-9

Figure 9



500
250
0
nM
0
250
500
30.0 nM
15.0 nM
0.0 nM
Microscope NSIII TM_AFM
Scan size 500.0 nM
Setpoint 1.987 V
Scan rate 2.035 Hz
Number of samples 512
sandia#2.001

Figure 10

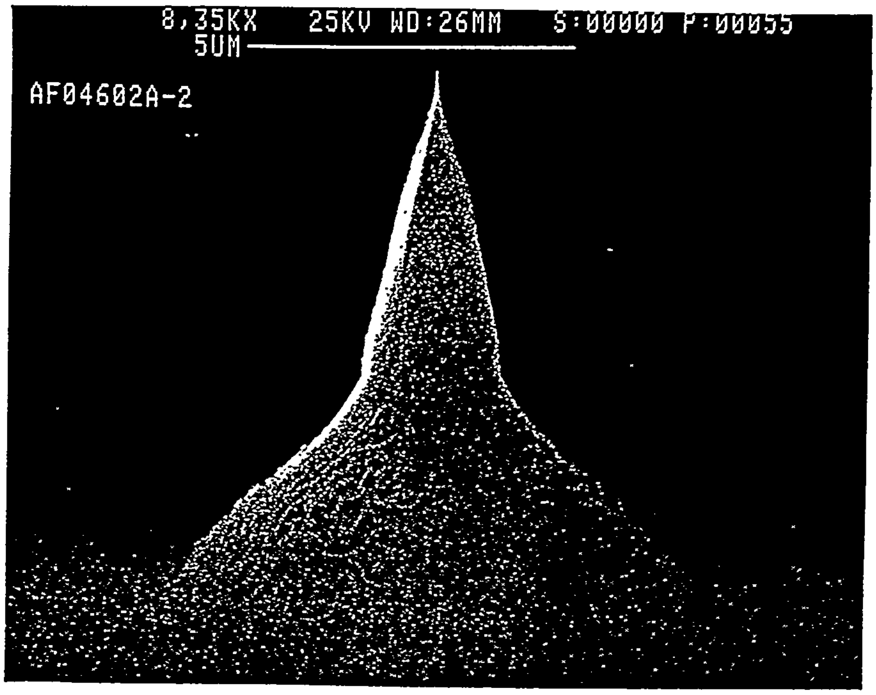


Figure 11

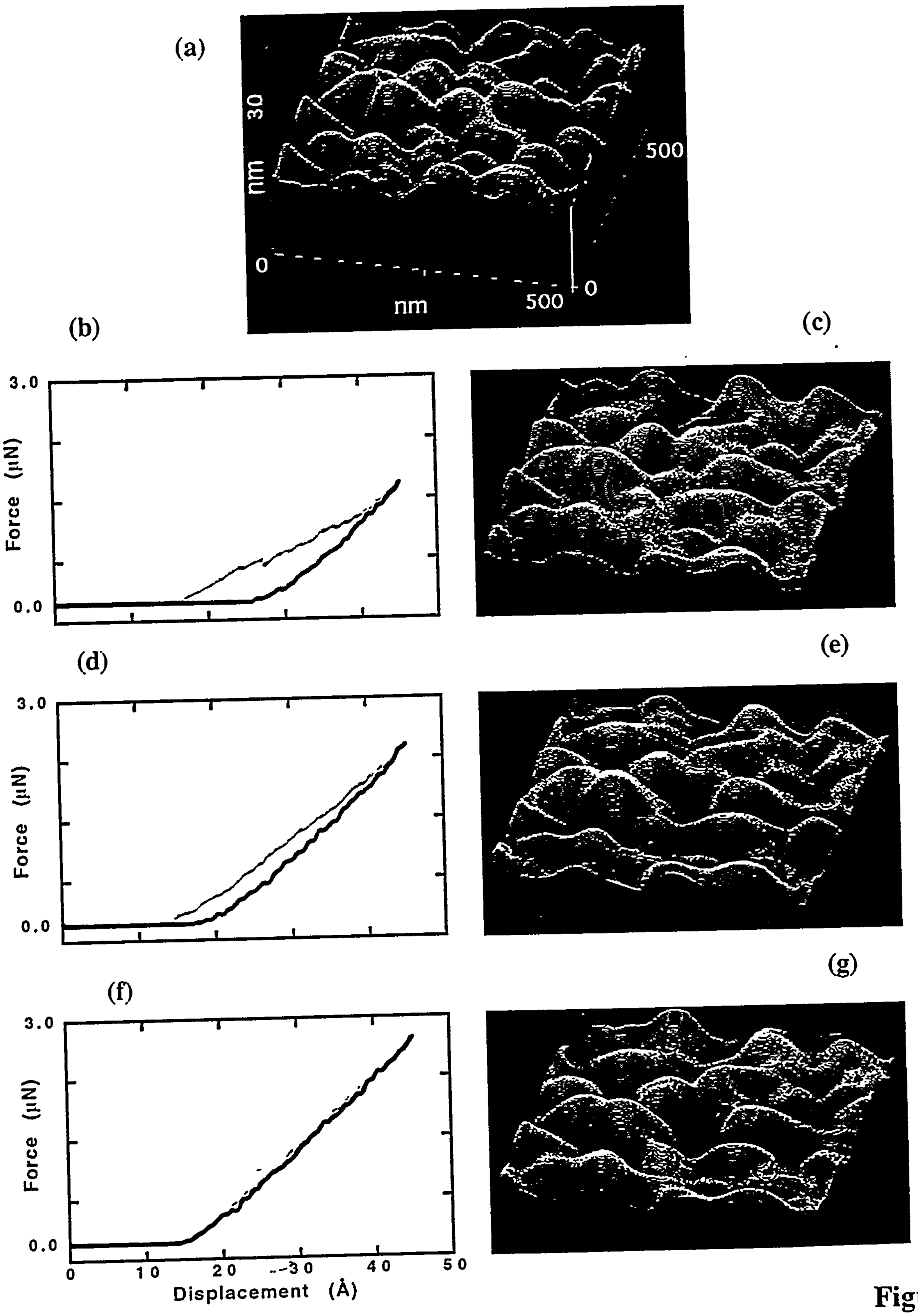

(a)
30
nm
0
nm
500
500
0
(b)
(c)
3.0
Force (μN)
0.0
(d)
(e)
3.0
Force (μN)
0.0
(f)
(g)
3.0
Force (μN)
0.0
0
10
20
30
40
50
Displacement (Å)

Figure 12

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