

MSc in Optoelectronic and Laser Devices
Heriot-Watt/St. Andrews Universities 1995/96

GIFT هدية

Project Dissertation



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**Project Title: Analysis of Diamond Thin Films Using Quesant
Atomic Force Microscope**

Abstract:

The Atomic Force Microscope AFM is an excellent tool in generating three-dimensional images of surfaces. Description of the principle and structure of the AFM is given. Details concerning the alignment of the AFM, positioning, approaching, engaging, and scanning the sample have been stated. The extreme properties of diamond that make it technologically attractive are stated. Images from the AFM, optical microscope, and SEM showed that diamond particles have different shapes, sizes, and clustering density when different areas on a film were scanned. It has been found that the roughness value of the polished areas could be lower than 30 nm. In fact, the convolution between the tip and surface features may distort the image. Finally, the effect of etching method on the morphology of semiconductor surfaces has been investigated.

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1: The Atomic Force Microscope AFM

The atomic force microscope (**AFM**) is an excellent tool designed to generate a three-dimensional image of a surface so it reveals information about the surface properties (Ref. 1 and 2). AFM was introduced by Binnig, Quate and Gerber in 1985 (Ref. 3). Both the **AFM** and the scanning tunneling microscope (**STM**), which is the original version of the **AFM**, work more or less in the same principle to generate three-dimensional images of a surface. Indeed the **AFM**, unlike the **STM**, is not restricted to conducting or semiconducting surfaces (Ref. 1). These two microscopes opened the realm to direct mapping of atomic and molecular shapes at higher resolution than ever before (Ref. 4 and 5).

More than 100 years ago (Ref. 5) the German Physicist and lens maker Ernst Abbe described a fundamental limitation of any microscope that relies on lenses to focus light or other radiation *diffraction obscures details smaller than about one half the wavelength of the radiation.*

The **STM**, for which G. Binnig and H. Rohrer received a Nobel prize in 1986, over come this Abbe barrier with ease. The principle by which they do so was first described in 1956. In that year J.A. O'Keefe proposed a microscope in which light would shine a tiny hole in an opaque screen, illuminates an object directly in front of the screen. Light transmitted through the specimen or reflected back through the hole would be recorded as the sample was scanned back and forth. O'Keefe pointed out that the resolution of such a "scanning near-field microscope" would be limited only by the size of the hole and not by the wavelength of the light. In principle the device could make superresolving image- images showing details smaller than half a wavelength.

Eric Ash of University college, London, adopted the O'Keefe strategy in 1972 to circumvent the Abbe barrier. He passed microwave radiation at a wavelength of three centimeters through a pinhole-size aperture and scanned an object in front of it to record an image with a resolution of 150 μm . In the same year as Ash's demonstration, *Russel D. Young* succeeded in manipulating objects in three dimensions with a precision of about a nanometer. He relied on piezoelectrics. Piezoelectric controls opened the way to the development, in 1981, of the supreme example of a scanning near-field microscope, the **STM** that led to the development, in 1985, of the **AFM**.

To understand how the **AFM** works, it is worth to study first the **STM** principle. A detailed description about the **STM** is given by G. Binnig and H. Rohrer (Ref. 4), H. Wickramasinghe (Ref. 5) and S. Tang (Ref. 6). To scan a surface one push the tip toward the sample until the electron clouds of each gently touch as shown in Fig. 1. The application of a voltage between

the tip and the sample causes electrons to flow through a narrow channel in the electron clouds. This is called the **tunneling current**. Since the density of an electron cloud falls exponentially with distance, the tunneling current is extremely sensitive to the distance between the tip and the surface. A change in the distance by an amount equal to the diameter of a single atom causes the tunneling current to change by a fraction as much as 1000.

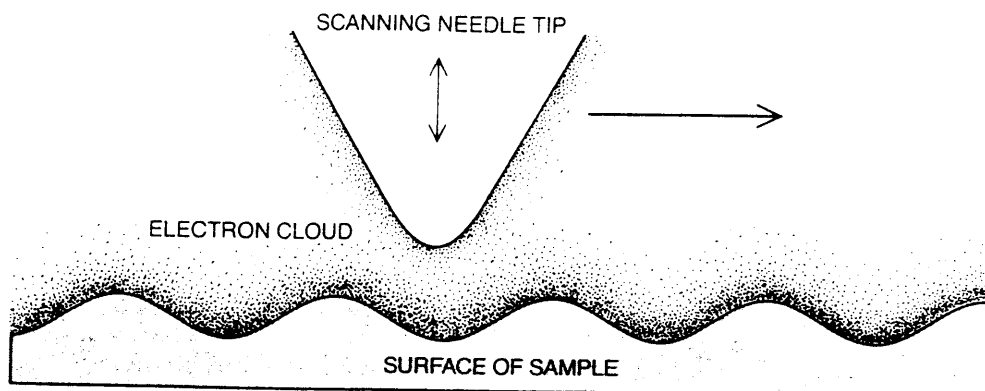


Fig. 1: **Electron tunneling** is the phenomenon that underlies the operation of the STM. An electron cloud occupies the space between the surface of the sample and the needle tip. A voltage-induced flow of electrons through the cloud is therefore extremely sensitive to the distance between the surface and the tip (From ref. 4).

Fig. 2, shows how the tip height is controlled by the feedback mechanism. The motion of the tip is read and processed by a computer and displayed on a screen or a plotter. By sweeping the tip through a pattern of parallel lines a three-dimensional image of the surface is obtained. A distance of 10 centimeter on the image represents a distance of 10 angstroms on the surface: a magnification of 100 million!

The X and Y piezoelectric controls move the probe back and forth across the specimen surface in a raster pattern, its parallel tracks separated by a fraction of nanometer. A feedback mechanism senses the variation in tunneling current and varies the voltage applied to a third, Z, control. The Z piezoelectric moves the probe vertically to stabilize the current and maintain a constant gap between the microscope's tip and the surface.

The invention of the **AFM** was a result of the success of the **STM** (Ref. 3 and 7). In fact, it is a rather surprising that the **AFM** operates much like an old-fashioned phonograph! (Ref. 8). The **AFM** works with a sharp tip mounted on a soft cantilever that laterally scans within a few angstroms of a sample surface.

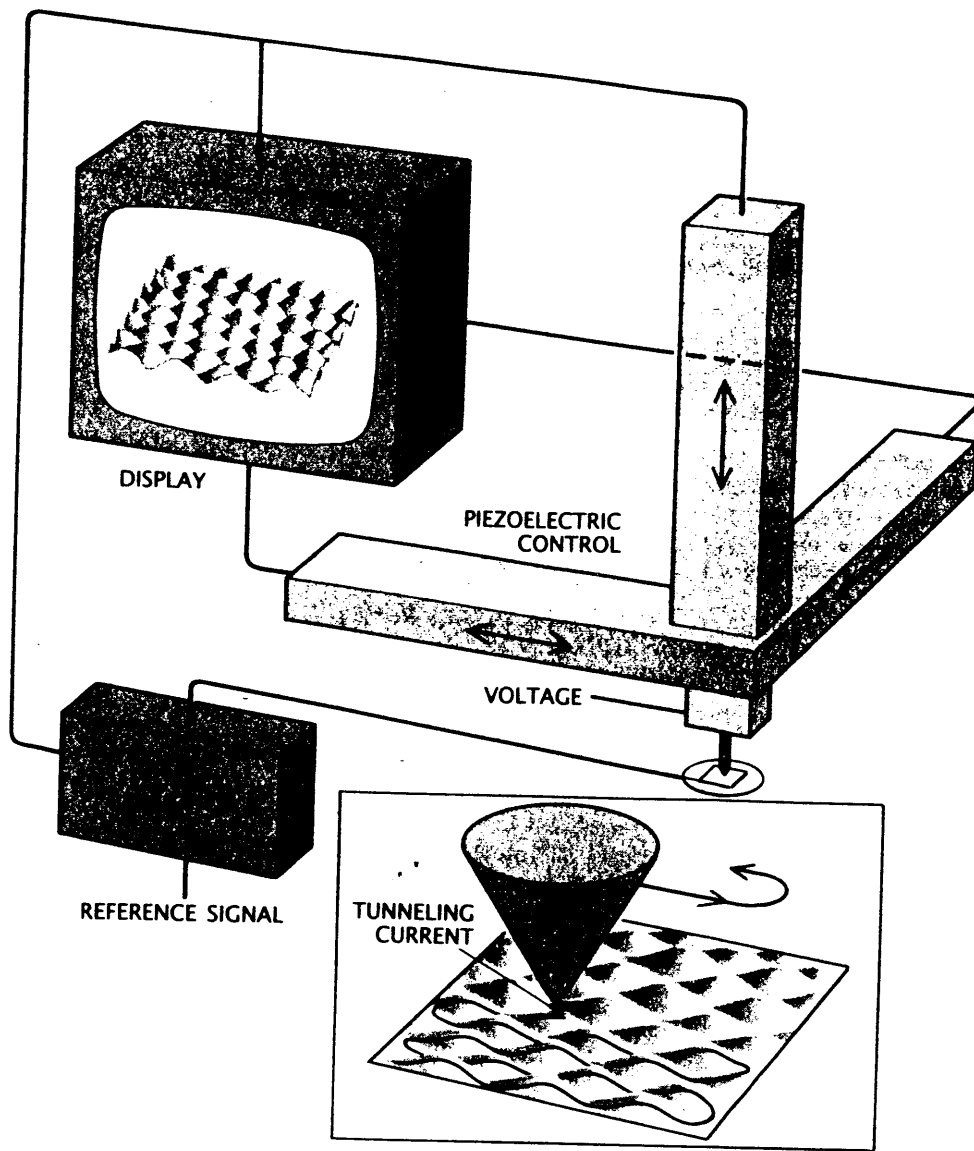


Fig. 2: **Scanning Tunneling Microscope** senses atomic-scale topography by means of electrons that “tunnel” across the gap between probe and surface (From ref. 5).

AFM images are obtained by measurement of the force on a sharp tip created by the proximity to the surface of the sample (Ref. 7). This force is kept small and at constant level with a feedback mechanism. When the tip is moved sideways, it will follow the surface contours such as the trace shown in Fig. 3. The cantilever has a small force constant of about 1 N/m or less (Ref. 8) which ensures that the force detection limit can be smaller than a nanonewton. Indeed this reduces the mechanical damage on fragile surface (e.g., soft biological materials) caused by the scanning motion. In addition the cantilever has dimensions in the micrometer range as shown in Fig. 4, consequently, it has high resonance frequency and does not vibrate in response to any environmental noises (Ref. 3, 7 and 8).

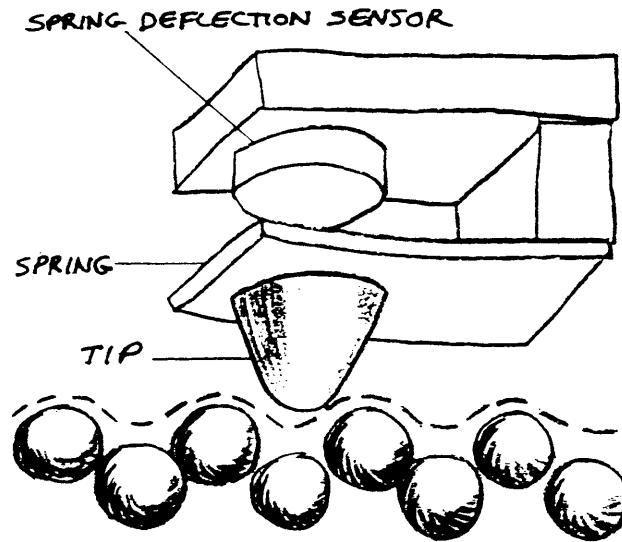


Fig. 3: **Scheme for the AFM.** The tip fastened to a cantilever spring that has a lower spring constant than the effective spring between two atoms. With sufficient sensitivity in the spring deflection sensor, the tip can reveal the surface profile with atomic resolution (From ref. 3).

The effect of external vibration is to cause unwanted motion of the tip with respect to the sample and the deflection sensor. The immunity of the **AFM** to external vibration depends on the frequency of the vibration relative to the lowest resonant frequency of the mechanical system. The mechanical system includes both the cantilever and the rest of the **AFM**. A typical 20 Hz building vibration of amplitude 1 μm results in relative motion of less than 0.01 $\text{\AA}$ (Ref. 3), a nearly harmless level. Because cantilevers can be readily made with high resonant frequency the limiting factor is usually the rest of the **AFM**.

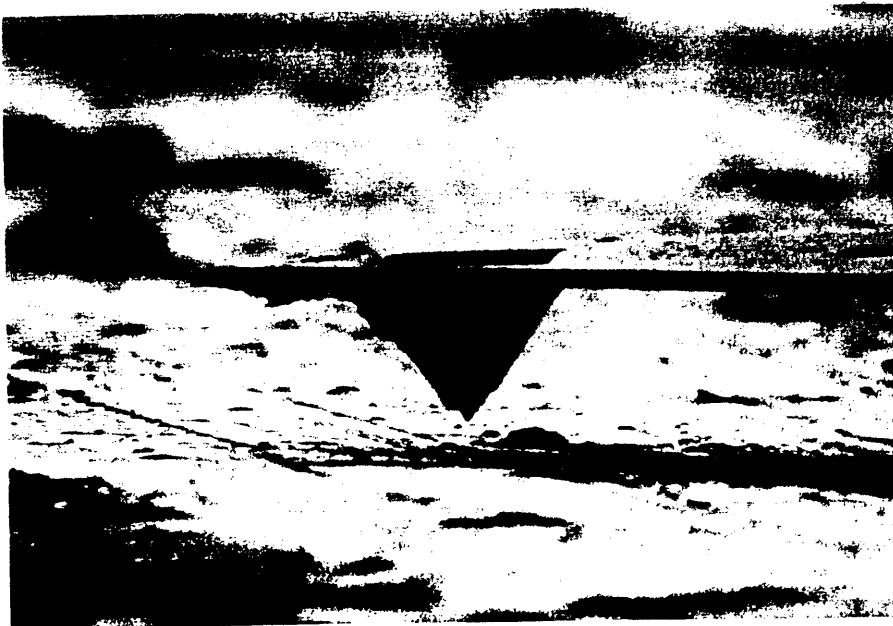
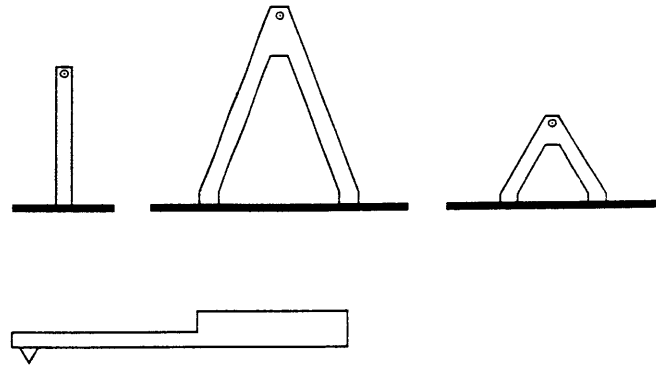


Fig. 4: A micro-cantilever with microfabricated tip (From ref. 3).

As shown in Fig. 4, the tip-cantilever assembly is microfabricated from silicon nitride, it is also possible to make use of silicon and silicon oxide. The microfabrication is done by using photolithographic techniques (Ref. 3 and 8). The commercially available cantilever has an integrated tip, the radius of which is as small as 10 nm at one end. Moreover; the cantilever shape can be optimized for conventional vertical force sensing as well as for lateral and frictional force sensing. Variety of cantilever and tip shapes available commercially. Three examples are shown in Fig. 5.

Fig. 5: **A variety of microfabricated cantilevers.** Tip shapes are optimized for lateral force sensing (left), conventional AFM imaging for soft material (middle) and scanning mode, which vibrates the cantilever at resonance frequency (right). Side view of cantilever with the tip is shown also (bottom) (From ref. 8).



After preparing a sample and placing it on piezoelectric control as shown in Fig. 6, as the **AFM** tip detects the forces arising from the tip and surface interactions the cantilever arm deflects up or down (Ref. 5, 7, 8 and 9). Given that the displacement of the deflection can be as small as a fraction of an angstrom, a very sensitive detection scheme is necessary so microfabricated cantilevers that are piezoelectric in nature have been developed. With these cantilevers; the **AFM** works almost exactly like a phonograph, i.e.; the deflection of the cantilever is converted to the relative height information of the surface.

A feedback mechanism similar to that used in operating the **STM** is used to maintain a constant force. Therefore, a constant separation between the tip and the sample, similarly, the **AFM** also can be operated in the constant average height mode, so that when the tip glides above the surface at fixed height and comes across a protrusion on the surface, it will sense great force and thus deflect the cantilever accordingly. In fact, optical deflection is the more widespread detection method in the most commercial **AFM**'s as shown in Fig. 6. In this type of detection (Ref. 5 & 8) a laser beam is focused onto the back of the cantilever. As the cantilever is deflected upward and downward, the laser on the bent cantilever is deflected to a photodiode.

The difference in the light intensities sensed by the photodiode at any X-Y location can be converted to a height measurement. Both the piezoelectric and the optical detection methods are capable of routinely measuring tip-sample interaction force in the nanonewton range (Ref. 8).

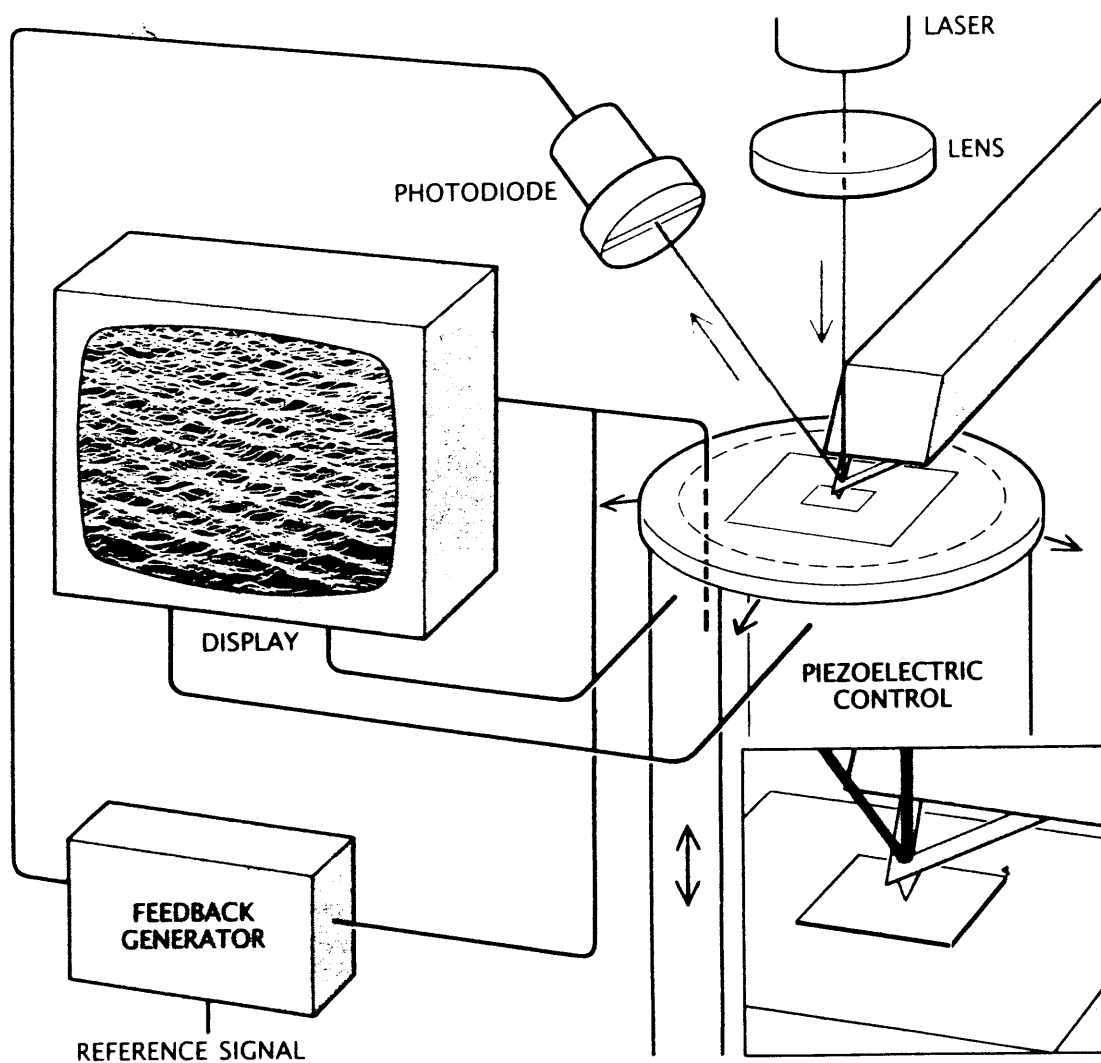


Fig. 6: AFM scans a sample with a shard of diamond mounted on a thin metal arm (From ref. 5).

2 : The Quesant Atomic Force Microscope

The used **AFM** was supplied by the *Quesant Instrument Corporation*. The supplied model is called the **Resolver** that offers many of the features present in higher priced instruments. The computer features a 60 MHz Pentium based system with Quesant's standard scanning and analysis software operating under Microsoft Windows. The software supports a resolution of 1024x768, non-interlaced, with 65,000 colors. The electronic interface unit (EIU) has three 16 bits and one 10 bit D/A converters and two 16 bit A/D converters. The stage can accommodate samples up to 4"x 4"x 1" and has an optical microscope mounted on the scanning head for aid in laser alignment and sample positioning.

2-1: Isolation

Since the **AFM** is a delicate instrument and is sensitive to vibration and acoustical noise as well as fluctuations in temperature; a successful result will depend in part on the extent to which it is isolated from these effects. The microscope must be located on the ground floor or in a basement to minimize building vibrations. If sound waves or air currents are affecting the image, a cylindrical metal or plastic shroud should be built to enclose the stage/scanner assembly, which should be placed on a work bench that has been leveled. The shroud should be positioned around the stage/scanner after the system has been aligned and just before the sample is scanned.

2-2: Probes

The supplied probes by Quesant are named as *Qlevers*, these are made of silicon nitride and have standard pyramidal tips. The Qlevers are gold colored rectangles, approximately 1.5x3.5mm, mounted on a stainless steel mounting plates, referred to as "tabs". These are affixed to a box by a strip magnet. The gold rectangle is a die to which the probe is attached.

Viewing the ends of one of the dies in the box with the eye loop, one can see a single triangle on one end and, on the other end, four triangles of different sizes and one elongated rectangle. the probe is removed from the box by grasping the tab with a forceps.

Both large and small cantilevers may be used to scan a sample. the smaller cantilevers are stiffer but more difficult to align. In general, larger cantilever is better suited for softer samples, such as polymers or plastics whereas the smaller cantilevers may be advantageous in some applications with harder materials such as glass, ceramics, metals or diamond.

There is no hard and fast rule as to how many scans can be made or how many different samples can be studied with a single probe tip. In fact, the nature of the sample studied and the

resolution required are key factors in determining how long a probe tip will last before it has to be changed. Some sample, e.g., hard surfaces with edge shaped features, causes a tip to deteriorate more rapidly. Streaks on an image are often an indication that the tip has picked up small contaminants so changing the probe will sometimes alleviate this problem. In short, the probe should be changed when there is a noticeable deterioration in image quality.

2-3: Alignment

It is necessary to check the alignments of the probe, laser and sensor before using the AFM. The scanner incorporates a class III laser so it is safe to view the probe through the microscope because a greatly filtered and scattered laser light could be seen, which is not hazardous. A summary of how the alignment could be done is as follows:

With the system turned on, Stage under Position should be clicked (see Fig. 10) to bring up the “Scan Head Control” window. The illumination will be off when the programme is just opened so the yellow button should be hold down until there is enough illumination to see the cantilever through the 80x optical microscope. If the cantilever is not in focus there is two lenses' adjustments available. The focus of the ocular lens is controlled by a white plastic screw in the barrel of the microscope. If necessary, a clear image of the cantilevers obtained by loosening the screw and adjusting the lens position and then re-tighten the screw. The position of the objective (lower) lens is controlled by a threaded hold-down knob. This is loosened by turning it counterclockwise. The lens can be focused by turning in either direction and can also be moved laterally to center the image. Once the best position is found, it is fixed by tightening the hold-down knob to the scanning head.

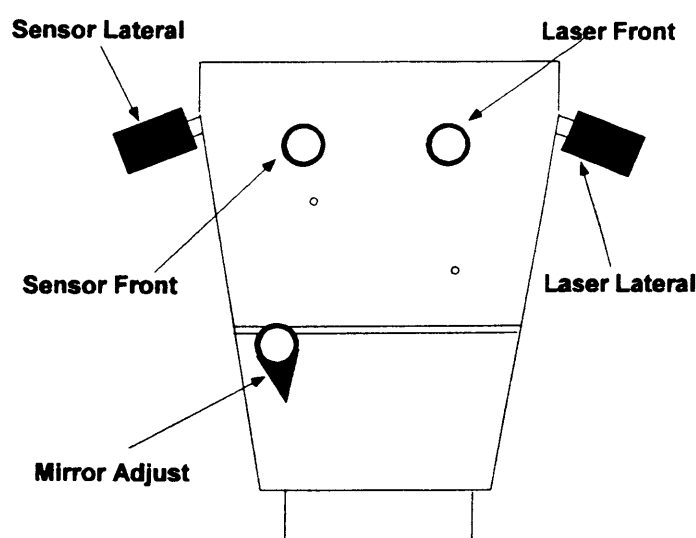


Fig. 8: The laser positioning knobs, the knob labeled laser Lateral moves the laser beam from left to right while the knob labeled Laser Front moves the laser beam from front to back (From ref. 10).

2-3.a: Locating the laser spot

Locating the position of the laser spot makes the alignment easy. The size and location of the spot, as seen through the optical microscope, will depend on whether the spot is being reflected from the die or metal tab. Clicking Align Laser under Acquire turns the laser on and brings up the “Beam Align” window. The Gain in the lower left corner of the “Beam Align” window must be set to X1 as shown in Fig. 10. The red laser spot can be seen above the cantilever by moving the Laser Front knob (see Fig. 8). The spot will have one of the two sizes, depending on whether it is reflecting from the die or metal tab. When the laser is reflecting from the die the spot will be much smaller than the average sized cantilever. If the laser is being reflected from the tab, the spot will be much larger. Fig. 9 shows the relative spot sizes for these two situations. The spot could be moved laterally with the laser lateral knob until it is in the position shown in Fig. 9 (a) or (b).

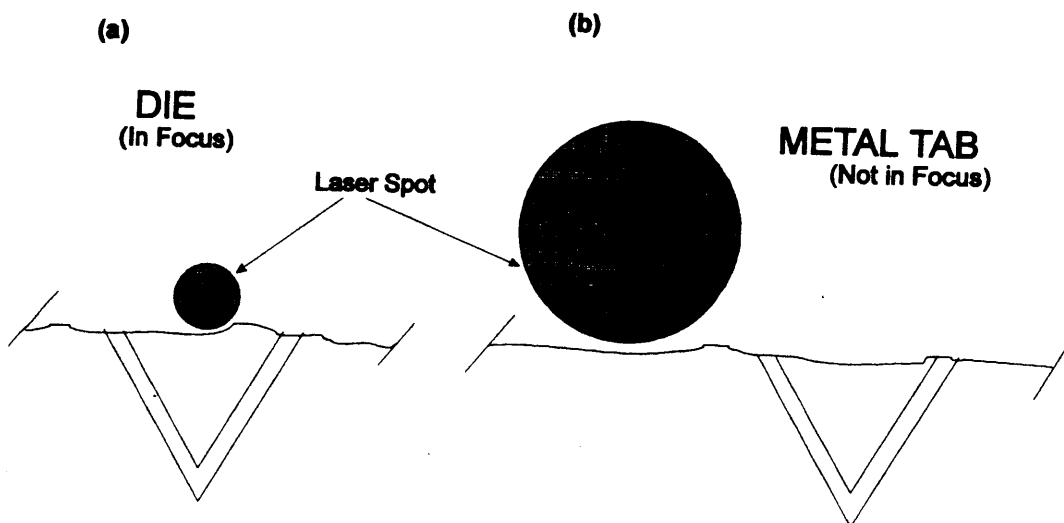


Fig. 9: The two sizes of the spot and how they should be positioned with the laser lateral knob (From ref. 10) .

As shown in Fig. 10, the position of the red circle on the “Beam Align” window indicates where the reflected laser light hits the photodiode sensor. The arrow on the vertical red-yellow-green scale indicates the intensity of the reflected beam. Using the Laser Front knob, the spot can be drawn towards the apex of the cantilever while, at the same time, observing the position of the beam intensity arrow on the “Beam Align” window. The arrow should rise as the spot is drawn towards the apex. Both the Laser Lateral and Front knobs should be adjusted until the arrow on the intensity scale is at or near a maximum.

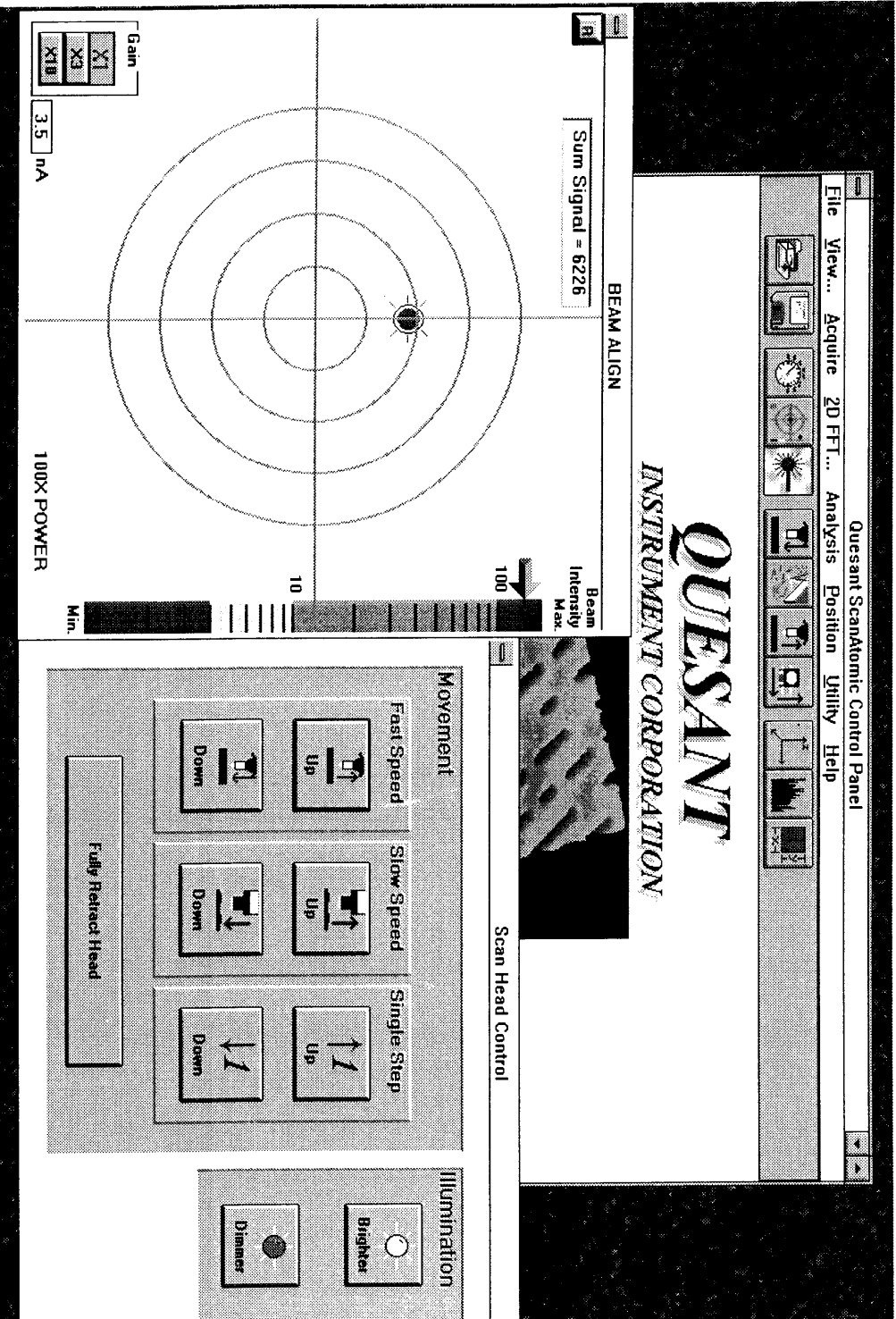


Fig. 10: The “Beam Align” window. The position of the circle indicates where the laser beam hits the sensor. The arrow should be in the green region, indicating adequate intensity of the laser beam.

2-3.b: Optimizing the position of the photodetector (sensor)

Since moving the laser affects where the reflected beam will hit the sensor, it is necessary to adjust both laser and sensor positions sequentially until the optimum locations are found. The “correct” locations of the sensor controls must be found. The correct location of the Sensor Front knob (see Fig. 8) is found when the circle can be translated several cm. above and below the central horizontal green line with little or no drop in beam intensity. The correct location of Sensor Lateral knob is found when the circle can be translated several cm. on either side of the central vertical green line with little or no drop in beam intensity.

2-3.c: Final adjustments

Fig. 11 shows the correct position of the laser when it is being reflected from the apex of the cantilever. It is important to ensure that the laser is being reflected from the apex of the cantilever and not from the arm. This requires visual observation through the microscope. With the gain button in the lower left corner of the screen set at X1, the Sensor Lateral knob should be used to bring the red circle to the center of the screen so that it is bisected by the central green line which is parallel to the Y. Then the Sensor Front knob should be used to rise the circle slightly above the center until the number in the small box in the lower left of the screen (next to the gain control button) is 3.5 nA as shown in Fig. 10. This corresponds to a set point force of 3.5 nN. Finally, closing the “Beam Align” window makes the system ready to set the scan parameters and to scan the sample.

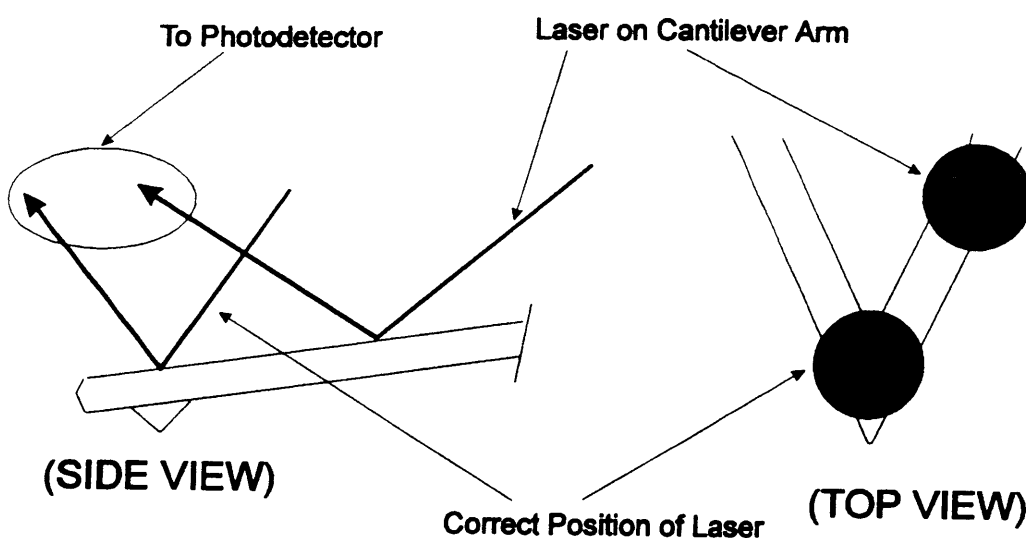


Fig. 11: The correct position of the laser when it is being reflected from the cantilever (From ref. 10).

2-4: Scan Parameters

Scan parameters are instrumental variables that could be changed through the “AFM configuration menu”, which accessed through Configure under Acquire. The scan parameters are listed in table 1 and described in appendix A.

Table 1: Scan Parameters menu.

Parameter	Range	Default
Scan Size*	Max: 30 μm *	see note *
Scan Rate	0.5 to 20 Hz	2 Hz
Setpoint Offset	-2.0 to 2.0	0
Scan Resolution	100 to 600	200
Z Dynamic Range*	Max: 9 μm	see note *
Z Input Gain	X1, X3 and X10	X1
Scan Direction	0 to 355	90
Integral Gain	0 to 4095	500
Proportional Gain	0 to 4095	500
Derivative Gain	0 to 4095	100
Image Mode	Z-height <u>or</u> Lateral Only <u>or</u> Semper Fidelity	Z-Height
X/Y Disable	Yes or No	No
Scan Profile	Hills <u>or</u> Valleys <u>or</u> Hills & valleys	Hills & Valleys

* The scan size and Z Dynamic Range depend on the type of scanning head being used.

2-5: Positioning and Approaching the Sample

The sample is placed on the stage and moved by hand. The desired region of the sample is located through an 80X optical microscope. The scanner should be several centimeters away from the stage. If it is not, the “Scan Head Control” is brought up by clicking Stage under Position on the main menu (see Fig. 10) and the scanner could be raised by holding down the mouse on the “up” button.

With the sample on the stage so that it is roughly under the probe tip, the “down” button is used to bring the probe tip to within about 0.5 mm or less from the sample. Then the sample is moved such that the scanned region is below the tip of the cantilever. After that, the probe is brought very close to the surface by carefully lowering it to the point where it actually touches the surface.

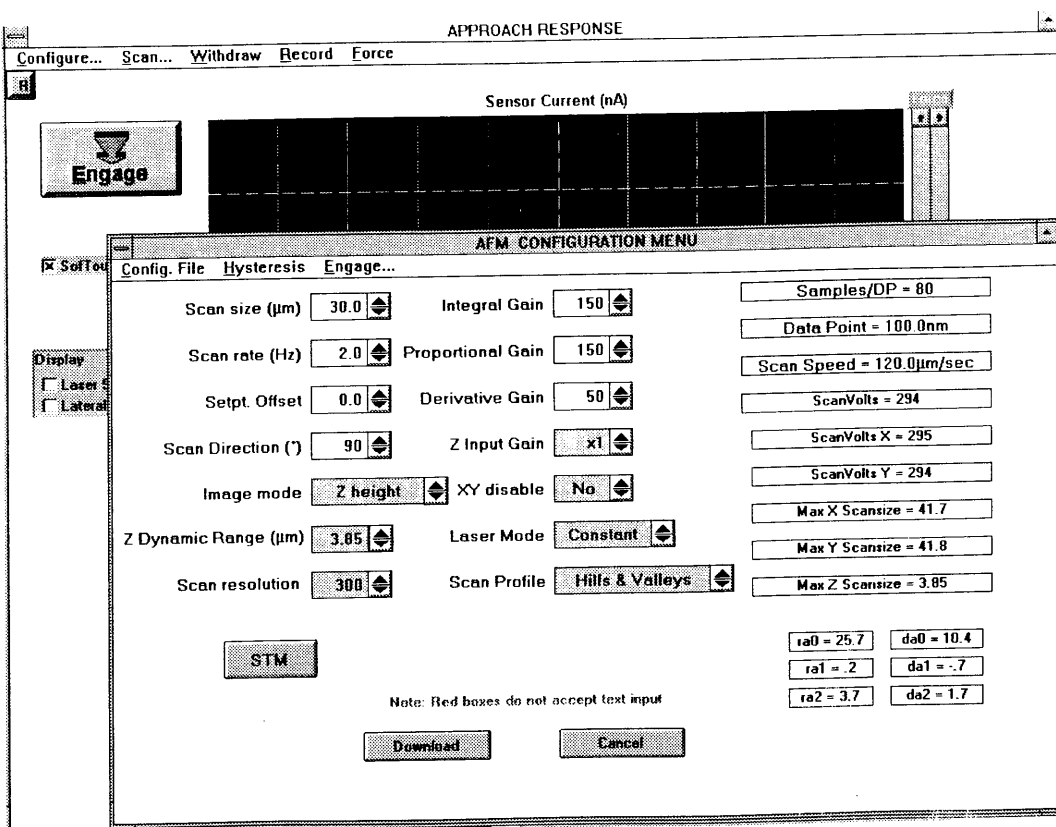


Fig. 12: Clicking Configuration in the “Approach Response” window; brings up the “AFM Configuration” menu. From this menu the appropriate values of Scan Parameters could be entered.

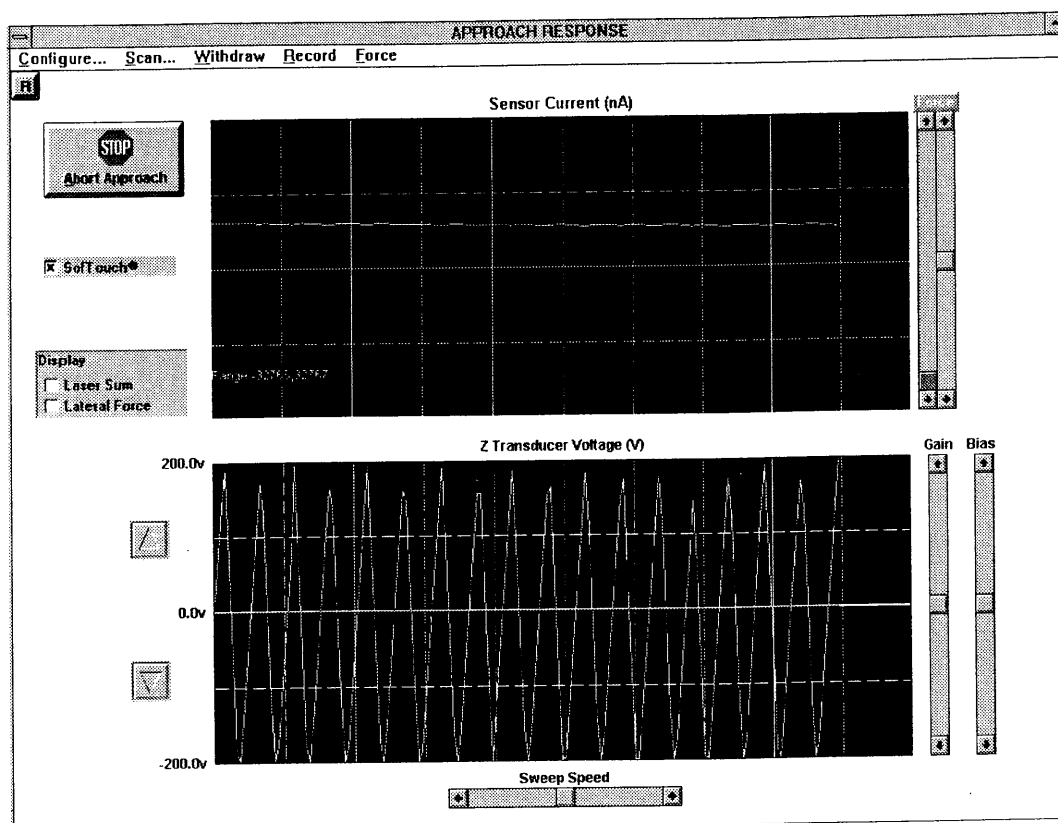


Fig. 13: Engaging the probe and the sample.

When contact with the surface is made, the cantilever will bend and suddenly appear very bright and the slow speed button is used to withdraw slightly, breaking the contact with the surface and removing the bend from the cantilever. Then the “Scan Head Control” is closed and the Align Laser under Acquire clicked. If necessary, adjustments with the Sensor Control knob are made to center the spot along the Y axis and to restore the setpoint force to 3.5 nN then closing the “Beam Align” window.

2-6: Engaging and Scanning the Sample

After the sample and the probe are brought close to each other, the probe and the sample are engaged and the system brought into a condition referred to as “**feedback**”. This is a mechanism that maintains a constant force between the sample and cantilever by sensing the position of the cantilever and adjusting the voltage to the piezoceramic accordingly. Once feedback has been achieved the sample can be scanned. The system should remain in feedback until the probe has been withdrawn.

Engaging the probe and sample can be started by clicking Engage under Acquire of the main menu as shown in Fig. 7. The “Approach Response” screen appears and configure is clicked to bring up the “AFM Configuration” menu as shown in Fig. 12. Once the appropriate values of Scan Parameters have been entered the menu, the Download button at the bottom should be clicked to load the scan configuration parameters into the DSP board controlling the EIU and to close the “AFM Configuration”. Clicking the green Engage arrow results in drawing the probe slowly and automatically to the surface. As shown in Fig. 13, the bottom sawtooth trace under “Z Transducer Voltage” reflects the expansion and contraction of the piezoceramic as the probe moves toward the surface. The top trace reflects the changing position of the laser beam with respect to the photodiode as the cantilever approaches the surface.

As shown in Fig. 14, the appearance of a flashing sign of “**Good Feedback**” means the system is truly in feedback. The top trace should abruptly snap to zero and the bottom trace level off and it should be positioned near the center if the Hills & Valleys (the default setting) was selected. In case of getting the flashing signs of “**Lost Feedback**” as shown in Fig. 15, this means that the probe tip is either too close or too far away from the surface for the feedback loop to be maintained so Withdraw should be clicked and the approach is repeated. In good feedback case, where the probe is in contact with the surface. As shown in Fig. 16 (a) and (b), the “Real Time Image” window appears after clicking Scan under the menu of the “Approach Response” window. Then for the image to be generated and shown on the screen, the SCAN button in the upper left corner of the window should be clicked and each scan line is shown as a

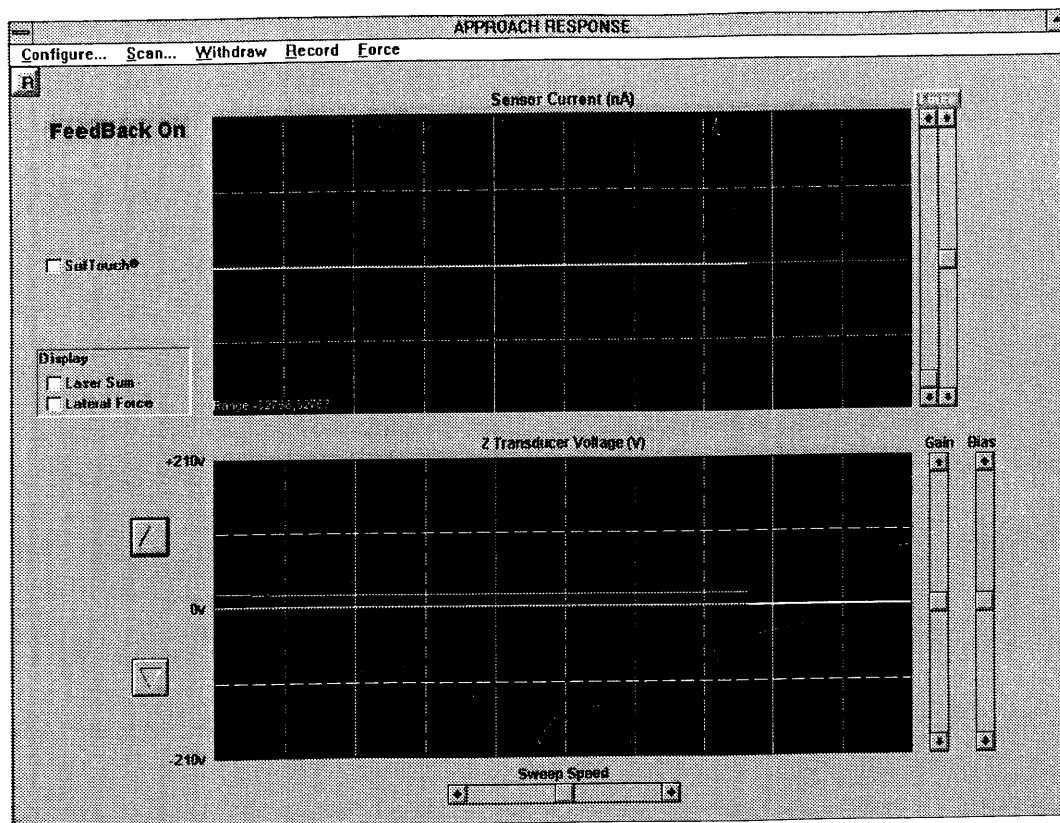


Fig. 14: The system is truly in a good feedback state so it is ready to scan the sample.

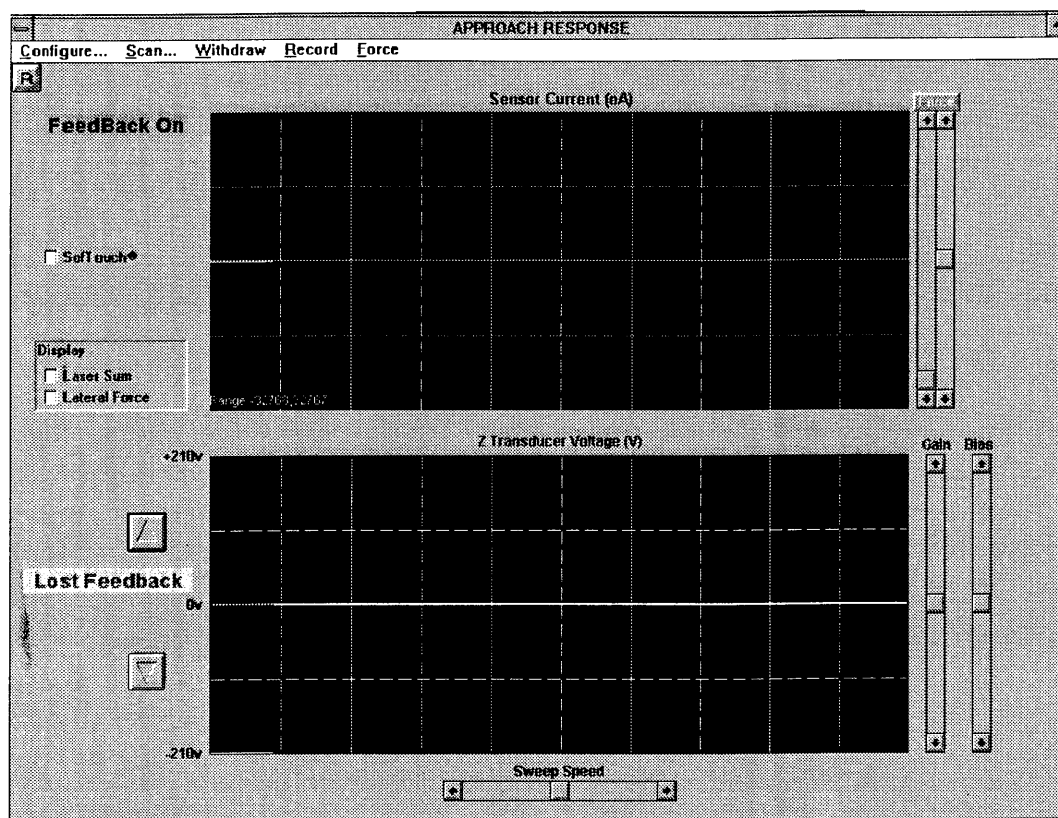


Fig. 15: Engaging the sample should be restarted in the case of Lost Feedback.

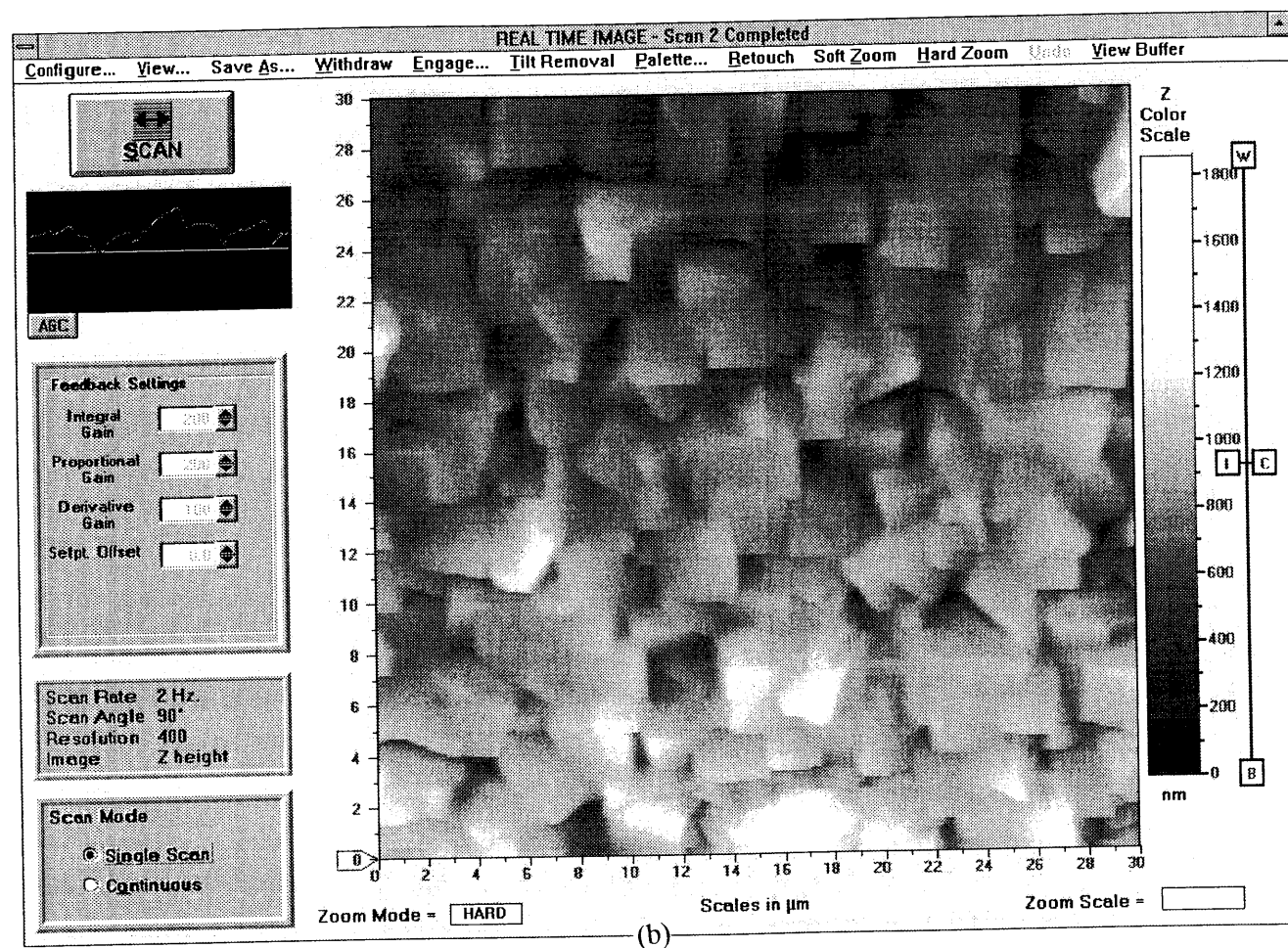
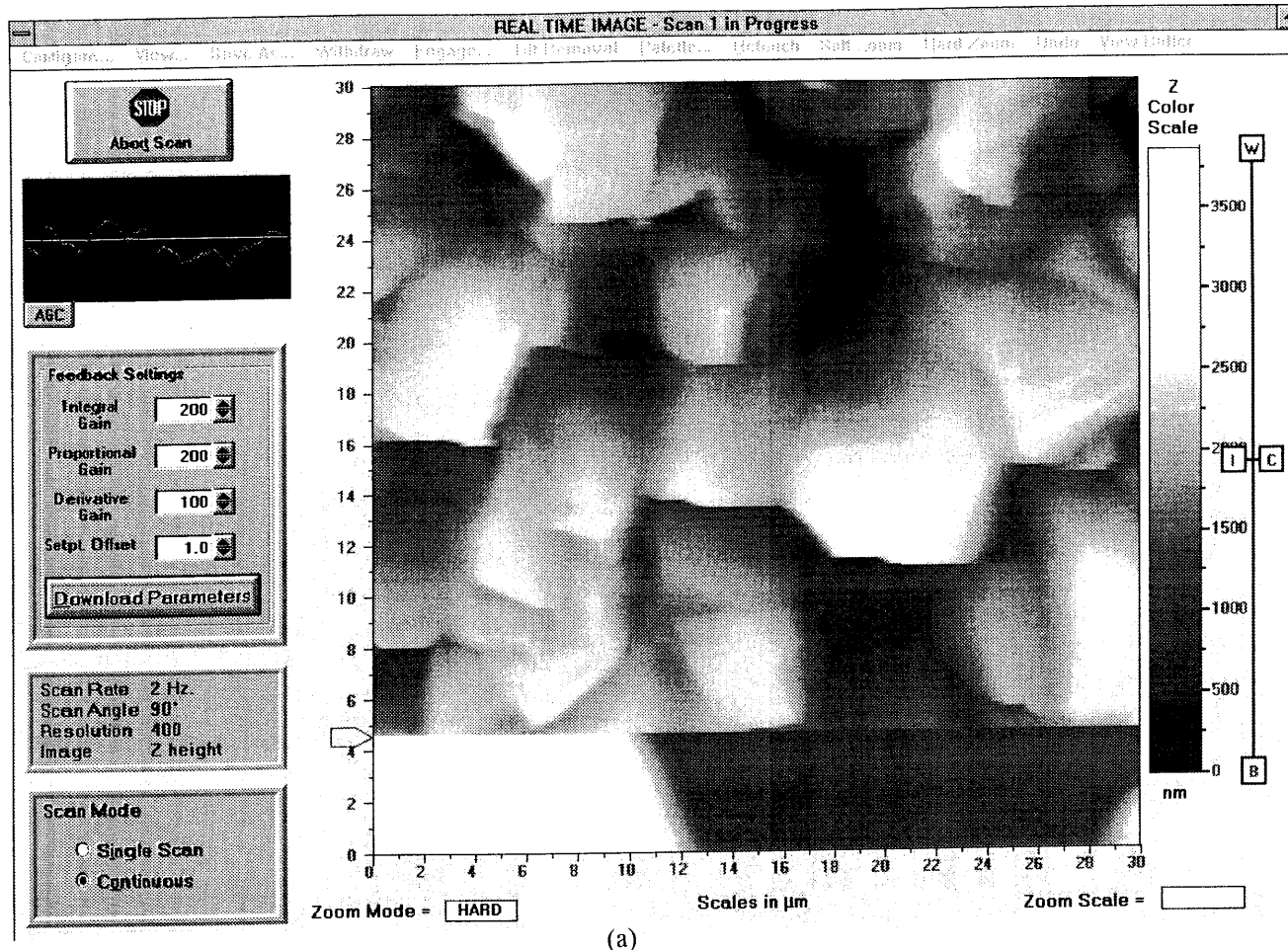


Fig. 16: (a) The shown image while the sample is under scanning. (b) After the scan is finished; the probe should be withdrawn from the sample before doing any analysis or saving to the image.

green trace in the box below the SCAN button {Clicking the AGC button (Automatic Gain Control) enlarges the features of the scan line.}. To store the image in the computer's memory; Single Scan button should be clicked before scanning. The generated image will be in an image buffer referred to as the "Z-Buffer" and remains there unless the system is shut down or new image placed in the buffer through the open command.

2-7: Refining the Image

As shown in Fig 17, the result of any deviation of the sample plane from the scanning plane is a tilted image. If the tilt not removed, it will cause an artifactual image. Clicking Tilt Removal on the menu at the top of the screen; shows several ways to remove a tilt from the image. In fact, it is important to note that if tilt is first removed and /or the image is modified in some other way and these actions are then followed by saving the image, the original image cannot be recovered. It is therefore recommended that the raw image first to be saved without any modifications. Then, after the image has been corrected, the newly modified image can be saved under another different name. The raw image is, of course, still available if, for example, one wants to compare different methods of tilt removal.

If, after using a command from Tilt Removal menu one is not satisfied with the result, clicking **Undo** restores the original image. After completing a scan, it is helpful to assess the nature of the tilt in the image by clicking the View command to generate a small or large screen 3D view as shown in Fig. 17. This may help in deciding which of the Tilt Removal commands best suits the image. Undo cannot be used to reverse a Tilt Removal command if any other Tilt Removal (or Retouch) command follows it (or even if these menus are pulled down) but clicking View command does not disable Undo. More details about Tilt Removal ways are given on the Quesant user's manual, a summary of them is given below.

Edge Fit, automatically removes tilt from the image. it is useful when the image consist of isolated features extending above or below the plane surface.

Parabolic, like Edge Fit, also automatically removes image tilt. This method works best where the features are many small deviations on a surface that is distorted like a dish or cylinder. In fact, Parabolic option will do a plane fit to any image but an irregular image will be forced to fit a planer surface.

Line by Line/V and **Line by Line/H**, these two command by themselves can be used to level very smooth surfaces. However, they may introduce distortions if the surface contains many prominent features.

Parabolic line by line, combines line by Line commands and Parabolic tilt removal.

Looking now at the **Retouch** options which are:

Streak Removal, It is very common for foreign matter to get under the probe and produce a streak line in the image. When the Streak Removal clicked and the cursor positioned above and to the left of the streak, then holding down the cursor button and dragging horizontally produces two parallel lines. When the cursor released, the streak between the two lines will be removed. The smaller the separation of the lines vertically, the better will be the removal.

Spot Removal, although it works in similar manner to Streak Removal, it intended to work with imperfections that are smaller in area.

Rescale, rescale the Z-axis of the image if either of the above two commands is used and the feature removed contains the highest or lowest points of the image. In fact, the first two commands can be reversed by Undo, but Rescale cannot and should only be used after one is satisfied with the way the image has been touched.

2-8: Analyzing the Image Data

The Analysis menu could be used to perform statistical and dimensional analysis of the image. The statistical distribution of the Z height of all points in the image and certain calculated measures of surface roughness could be seen by selecting Histogram under the Analysis menu item, then clicking Height Histogram, an example of such analysis is shown in Fig. 18. Not the display of the cursor position in X and Y. This allows the user to make rough statistical measurements by eye by moving the cursor. Average height (Thickness), the standard deviation of the surface distribution (RMS) and the mean deviation (Ra) which is a measure of the surface roughness are displayed under “Surface Characteristics”.

Executing the Dimension command opens the window titled “Image Analysis” as shown in Fig. 19, it shows a top view of the current Z buffer image with a line that slices through the image. The graph below the image is a profile curve and shows a cross sectional slice through the image corresponding to the line. The direction of the line can be selected to be either horizontal or vertical by selecting option buttons in the profile panel. The line position can be changed by using the scroll bars on the right and bottom of the image. note the profile changes as the line is moved.

A third option in the profile panel is Diagonal section as shown in Fig. 20. Clicking on it and positioning the cursor some where on the image. Then clicking and dragging a line to any other point of the image and when the mouse button is released, arrow heads are drawn on the line indicating the direction of the profile. Read and blue lines can be moved on the section graph by placing the cursor arrow on the line and then dragging. This display the coordinates of the points where each line intersects the graph in the box in the upper right under “Red cursor” and “Blue cursor” the distance between these points is calculated and displayed in the second box.

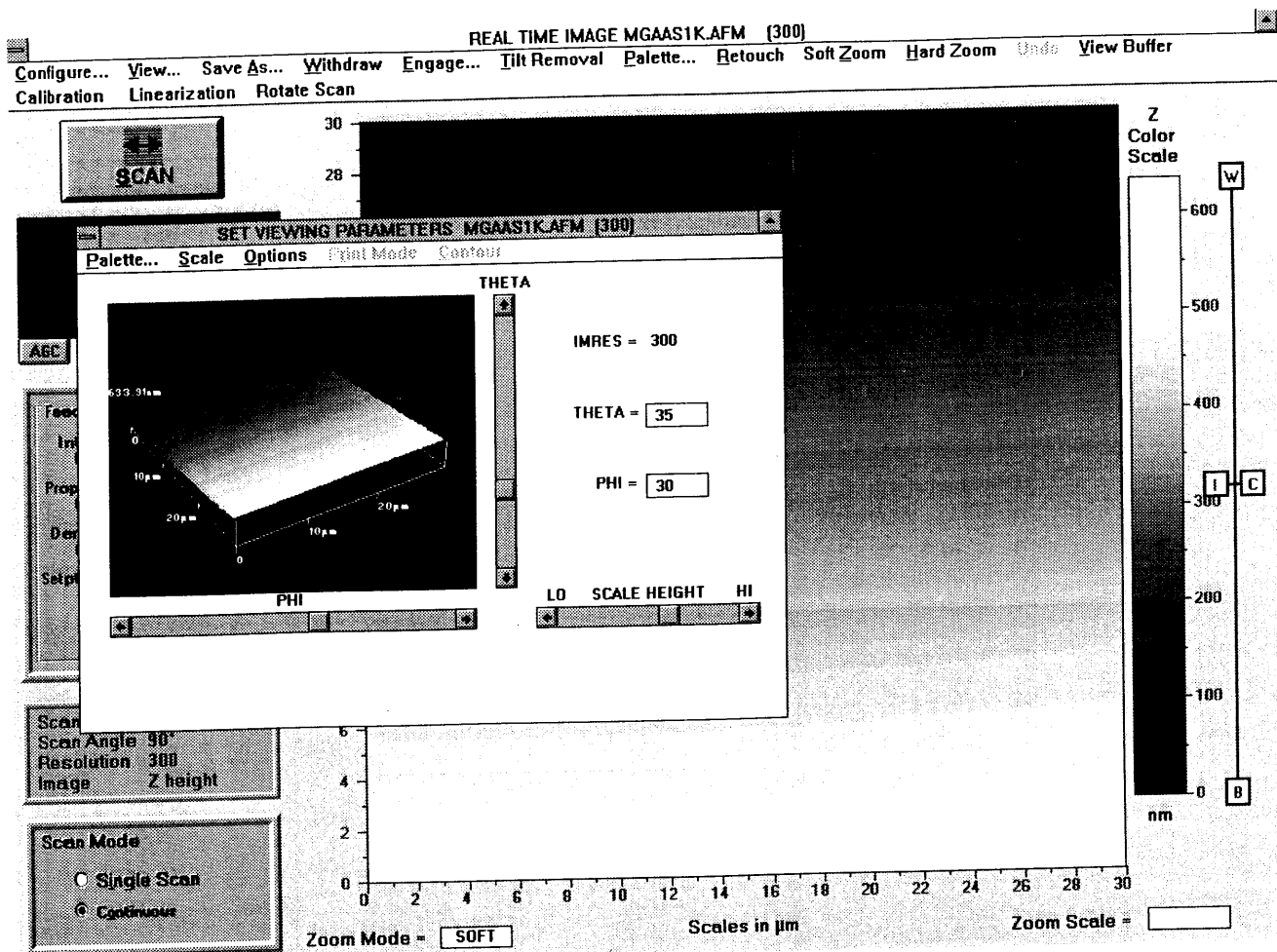


Fig. 17: The result of any deviation of the sample plane from the scanning plane is a tilted image. Clicking the View command generate a small or large screen 3D view.

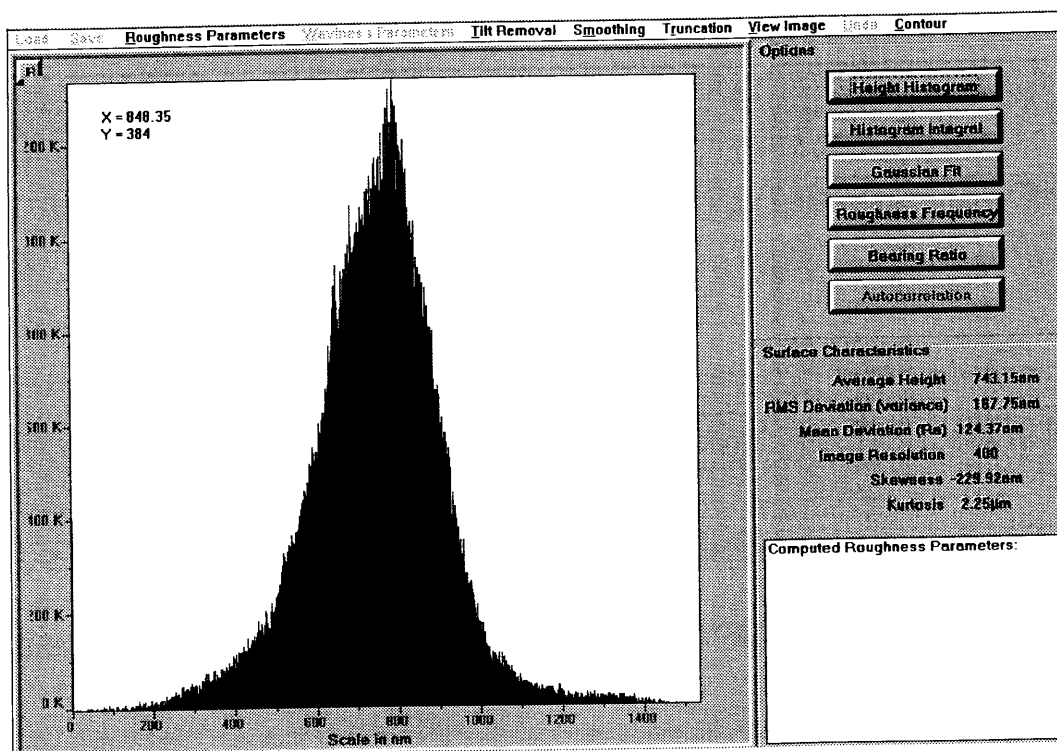


Fig. 18: Statistical analysis of the image shown in Fig. 16(b) after using Fit Edge Tilt Removal.

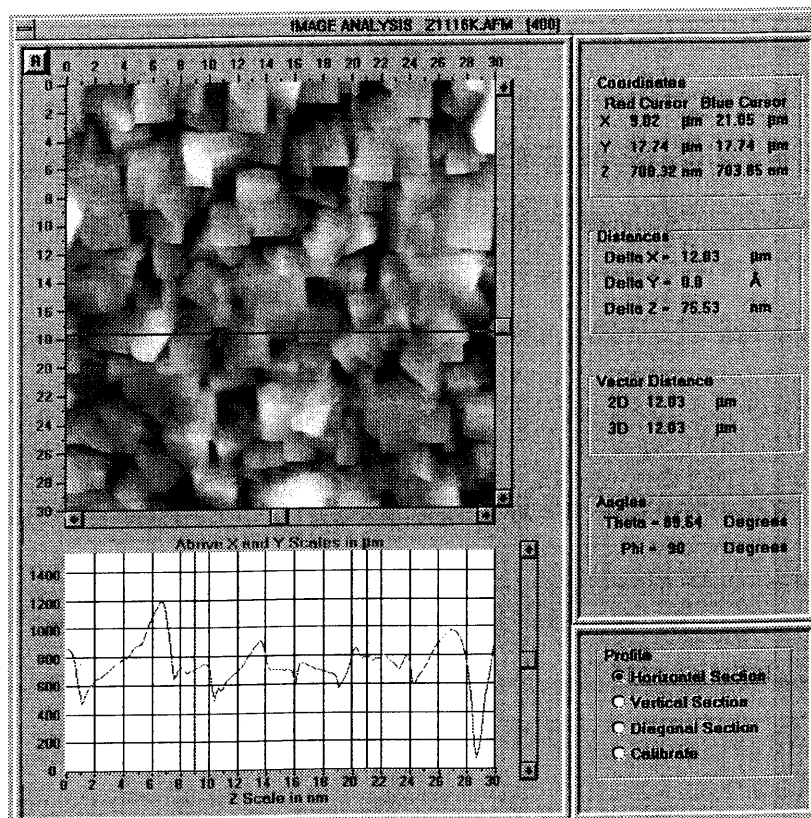


Fig. 19: Executing the Dimension Command helps in measurement of surface features dimensions.

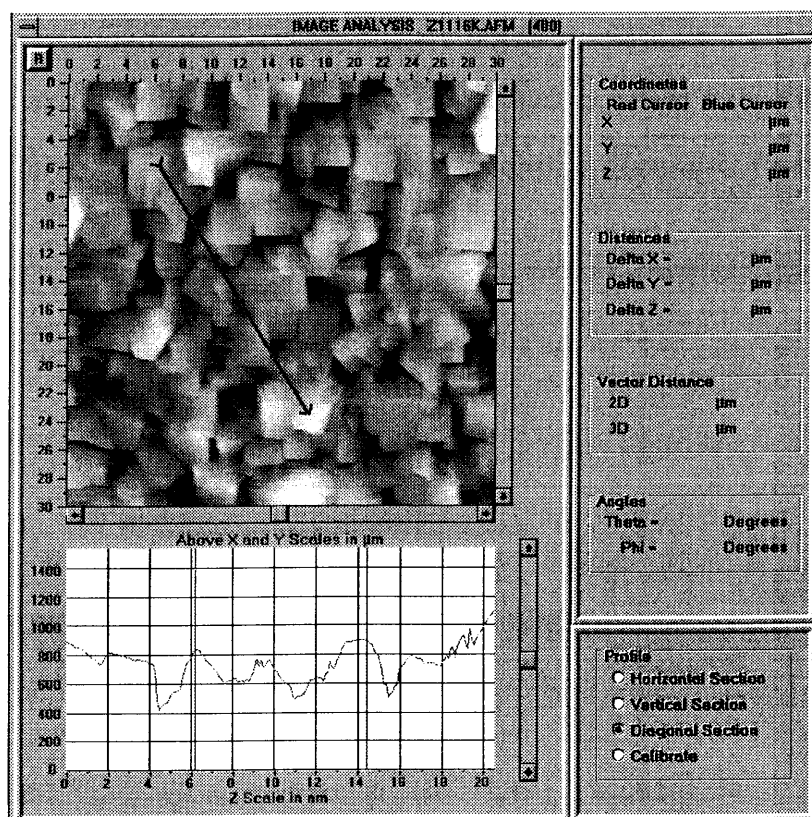


Fig. 20: A diagonal section option could be used for dimensions measurements.

3: Results and Results Analysis

A: Diamond Thin Films

It is well known that diamond has a number of excellent properties (Ref. 11, 12, 13, 14, 15, 16 and 17). Since diamond optical transmission starts from UV to far IR, it is one of the most transparent materials. In fact, diamond is the hardest known substance as well as the stiffest, but its specific density is small. Diamond is the least compressible substance. Although its thermal expansion is small, it has the highest thermal conductivity at room temperature, about five times larger than the value for copper. Electrically, it is a good insulator and the band gap associated with the indirect transition between the covalent and conduction bands is as large as 5.48 eV. Diamond is chemically inert and resistance to X-rays, γ -rays, UV light and nuclear particles. In brief, diamond is a unique and fascinating material.

Diamond has these unique properties as a result of its unusual bonding: the atoms of diamond crystal are more densely packed than those in any other known material. Furthermore, the forces that tie the atoms to one another are stronger than those in most solids.

In addition to their scientific interest, such extreme properties make diamond technologically useful. It has found applications as an abrasive, as surgical cutting tool and as a heat sink for cooling electronic components. Researchers have also shown that the diamonds containing various impurities function as semiconductors since either p- or n-type diamond films can be formed on substrates simply by mixing impurity gases and this is why the gas-phase synthesis has attracted so much attention recently (Ref. 11 and 13).

The unique properties as well as the promising future applications of diamond films attracted many research groups at the Heriot-Watt University and there is an extensive and highly supported research work at its Physics and Chemistry Departments in synthesis, characterization and processing of diamond films.

The major aim of this project was to make use of the Quesant AFM to scan different diamond thin films that has been synthesised at different deposition conditions. In fact, some parts of each film have been polished using a laser beam. The images produced by the AFM will be used to make further development of both the deposition conditions and processing methods.

Nearly more than half the time given for the project has been used to scan different types of diamond films and many different areas of each film. Indeed the results were disappointing; since **non of the resulted images showed a true result!** The work was repeated with different Scan Parameters as well as with new and different cantilevers, but the results were the same, five examples of these result are shown in Figs. 22, 23, 26, 28 & 32. Those results were

compared with results taken with the optical microscope and scanning electron microscope (SEM).

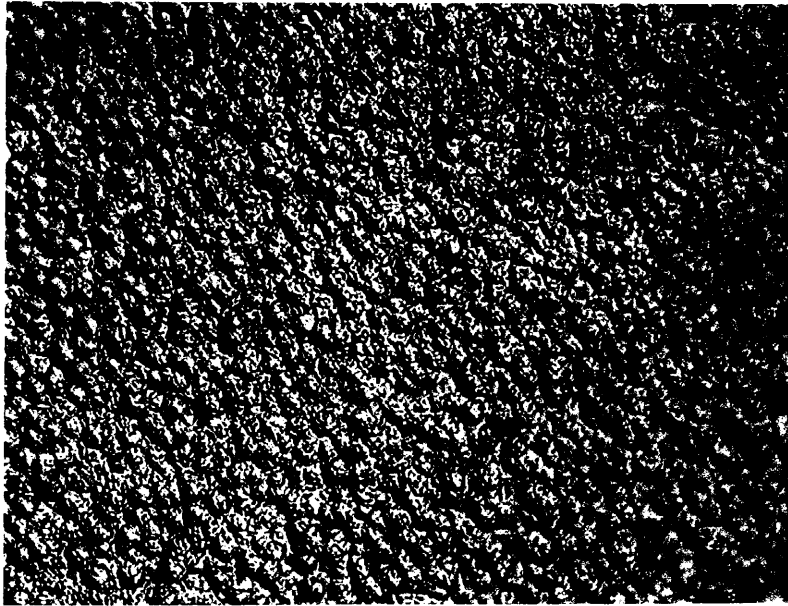


Fig. 21: Optical microscope image of a non polished area of sample no. 78.

Comparing the images shown in Figs. 21 and 22 of a non polished area of sample no. 78, which has been deposited using microwave CVD method at substrate temperature of 805 °C and gas mixture of H₂: 66.5 sccm, CH₄: 3.5 sccm and CO: 70.0 sccm, one can see the huge difference between the two images. Moreover, the AFM head is supposed to measure a maximum Z height of 4 μm since the scanning head has a Z Dynamic range of 4 μm. Thus, one conclude that the AFM gave unreal image.

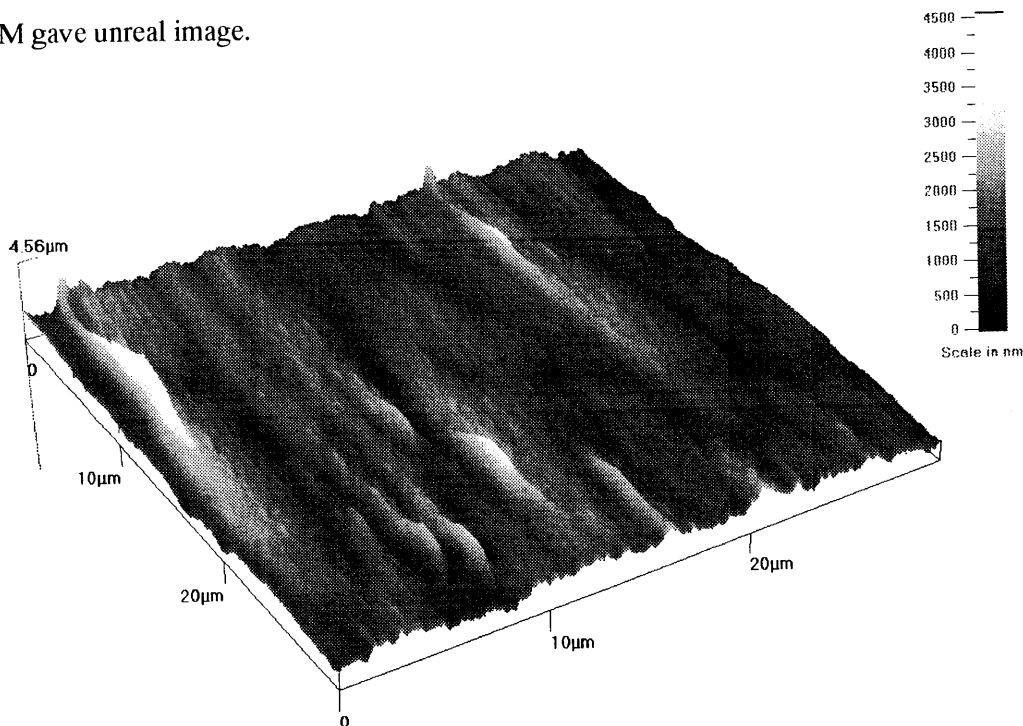


Fig. 22: An AFM image of the scanned area shown in Fig. 21.

Sample no. 106, which was deposited using microwave CVD method at substrate temperature of 863 °C and gas mixture of H₂: 85.0 sccm, CH₄: 2.5 sccm and CO: 70.0 sccm, also has been scanned with the AFM as shown in Fig. 23, the image is very bad when compared with that of Fig. 24.

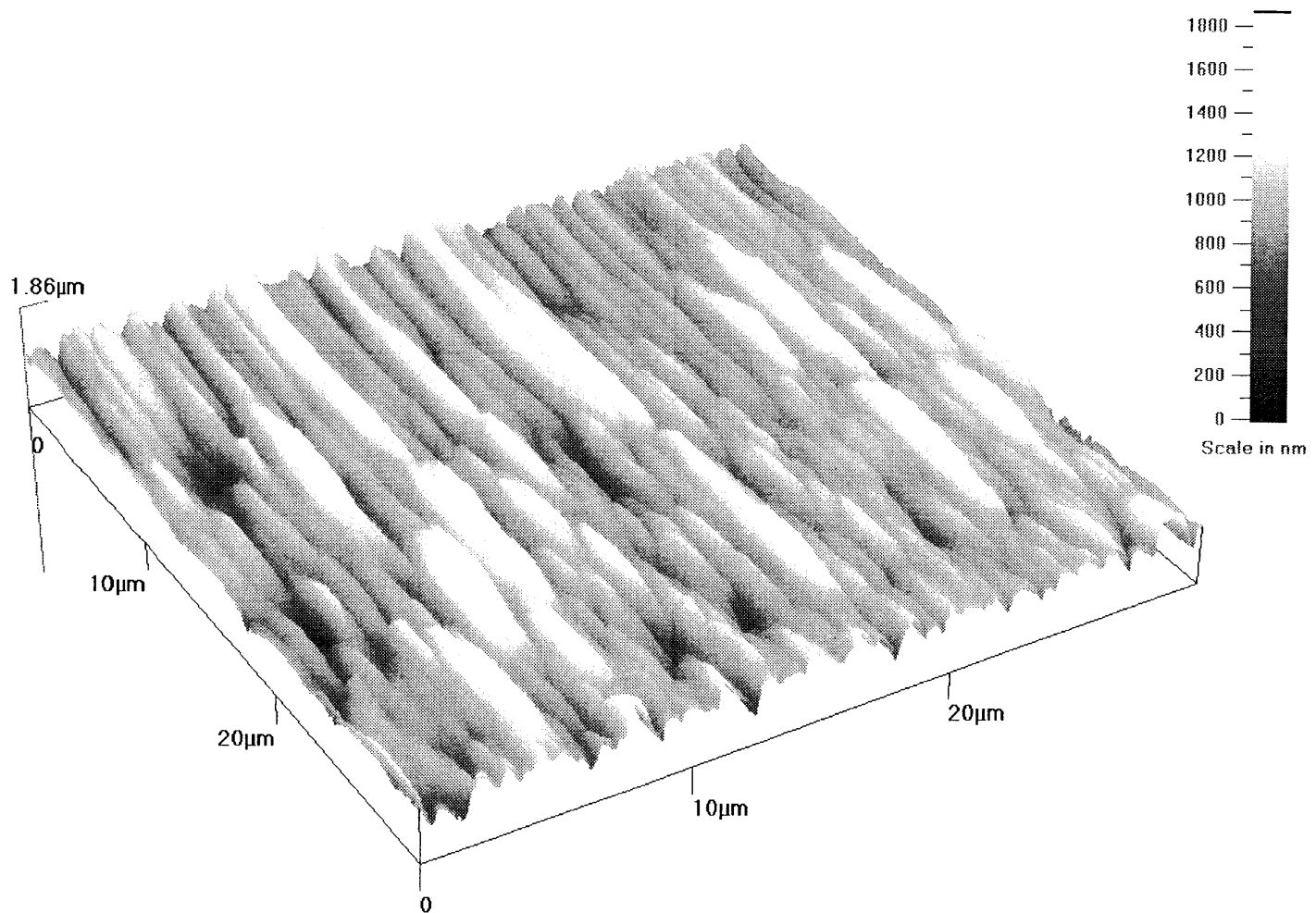


Fig. 23: An AFM image of an unpolished area on sample no. 106.

A scan of a polished area proved that the images given by the AFM were unreal, this could be seen by looking at the images shown in Figs. 25 and 26. In Fig. 25, one can see that the laser beam has removed all diamonds features shown in Fig. 24 and the surface become smoother than before. Moreover, the direction of laser polishing (shadowing) can be recognized. Same result was found with other samples, for example sample no. 78. Although Fig. 26 shows unreal image of diamond film features, laser polishing direction could be seen to certain extent.

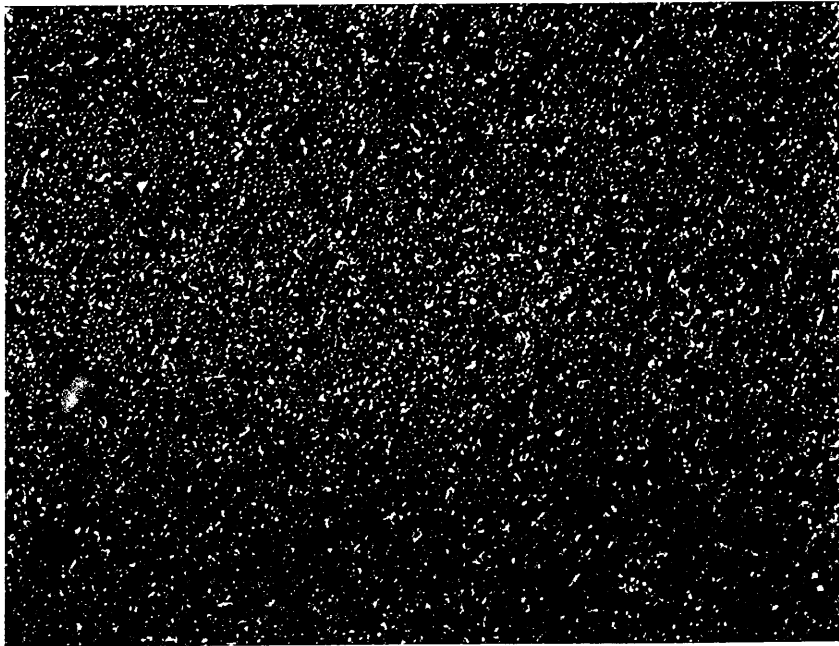


Fig. 24: An optical microscope image of the area shown in Fig. 23.

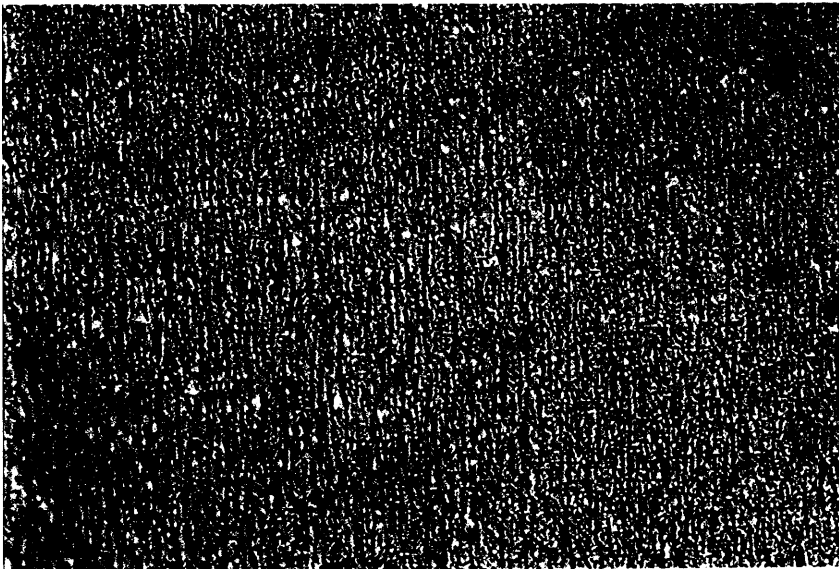


Fig. 25: An optical microscope image of a polished area of sample no. 106.

Further investigation with both the optical and the scanning electron microscope showed that the deposited diamond particles on any diamond film have different shapes, sizes, and clustering density as the film scanned away from its center to the edge of the outer ring. Sample no. 111 is a good example, this sample was deposited using microwave CVD method at substrate temperature of 1241°C and gas mixture of H₂: 300.0 sccm, CH₄: 54.0 sccm and CO: 0.00 sccm. As shown in Fig. 27, the deposited diamond has the forms of twinned cubo-octahedron as well as icosahedron, these structures have been studied in details by S. Matsumoto et al (Ref. 18). The clustering density of these particles increases as one scans toward the central part of the outer ring as shown in Fig. 27 (b). A scan of this area with the AFM gave the image shown in Fig. 28 which is unreal image.

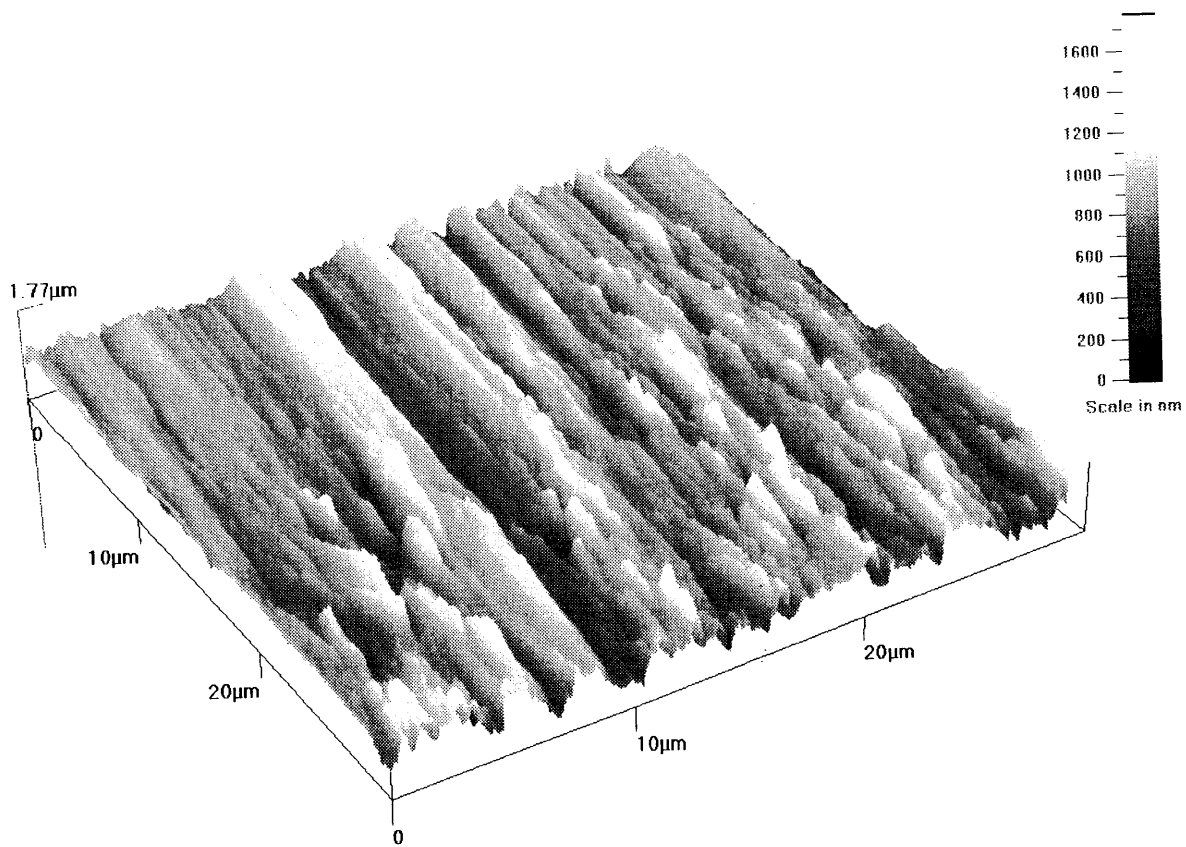


Fig. 26: The AFM image of a polished area on sample no. 106.

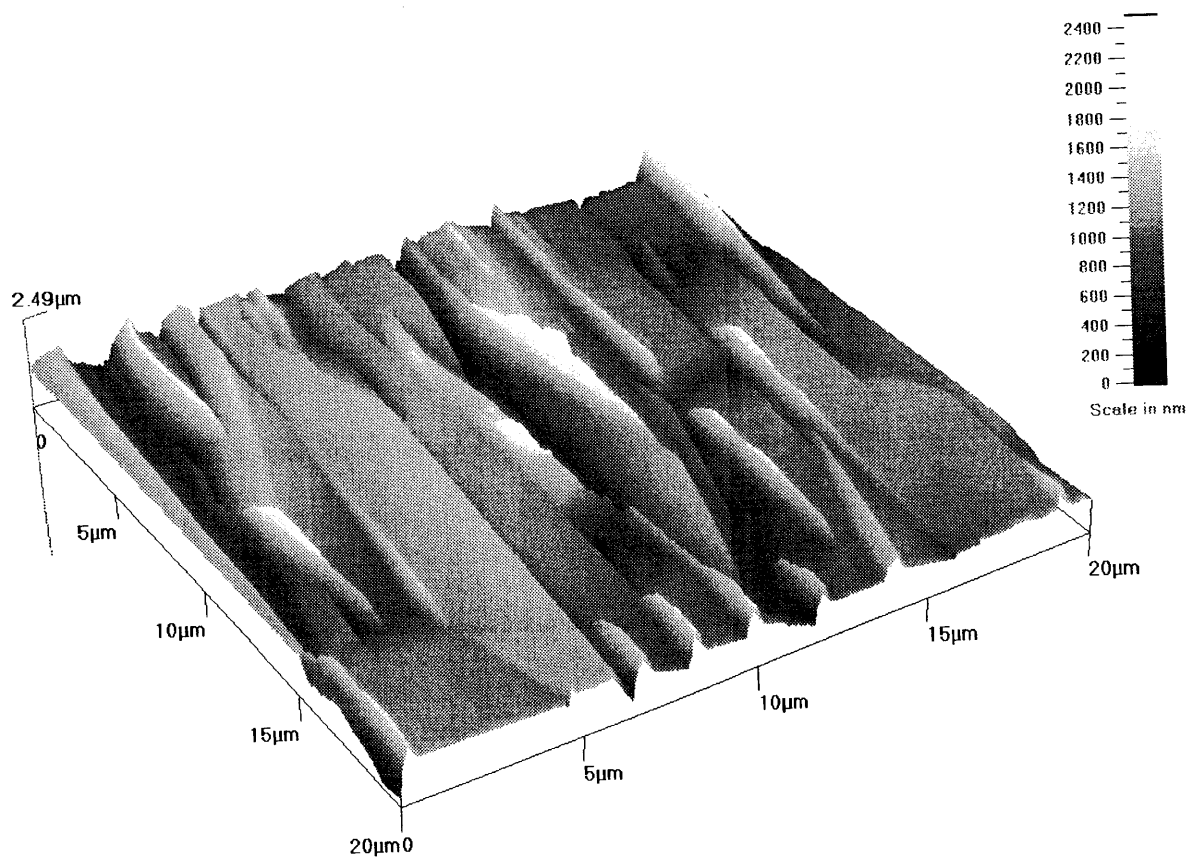
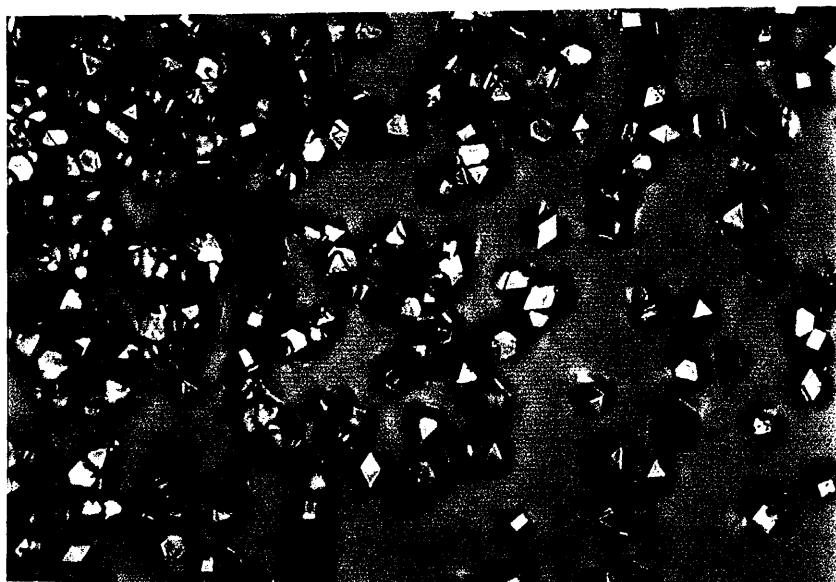


Fig. 28: The AFM image of a scanned area on the edge of the outer ring on sample 111/6.

(a)



(b)

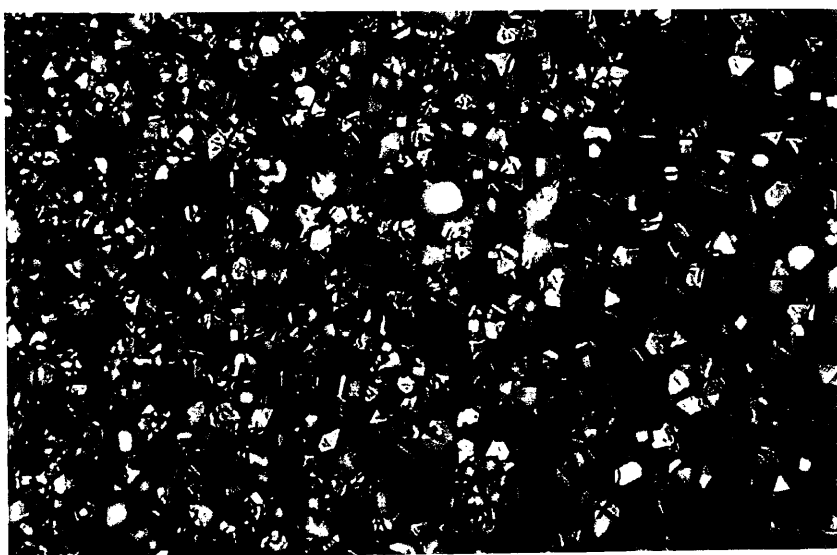
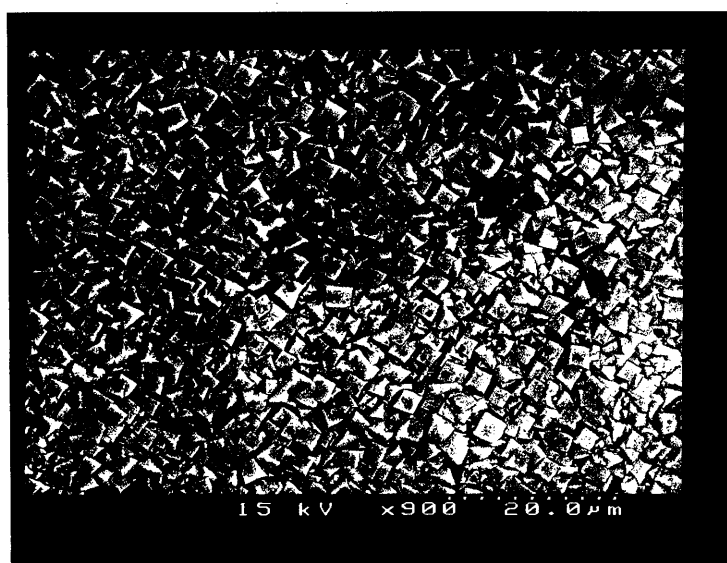


Fig. 27: Optical microscope images of the edge of the outer ring of sample 111/6.

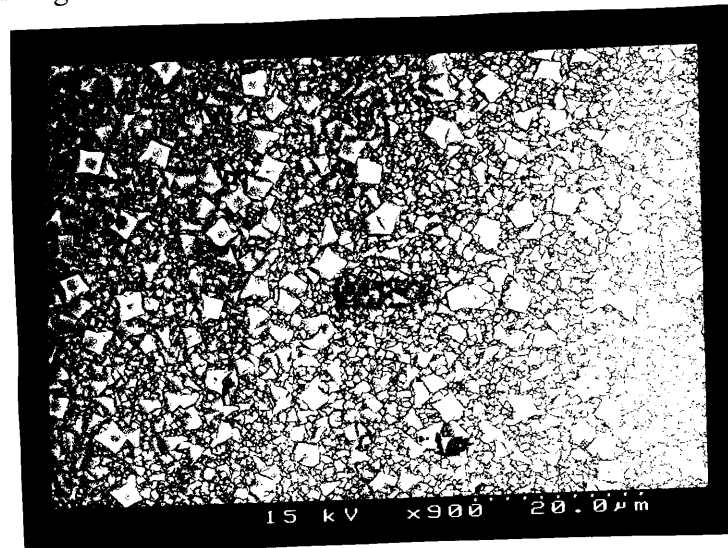
As the film has been scanned toward the center of the outer ring, the structure of diamond particles changes to the cubic (100) structure of small sizes and high clustering density as shown in Fig 29.

Fig. 29: SEM image of the central part of the outer ring on sample 111/6.



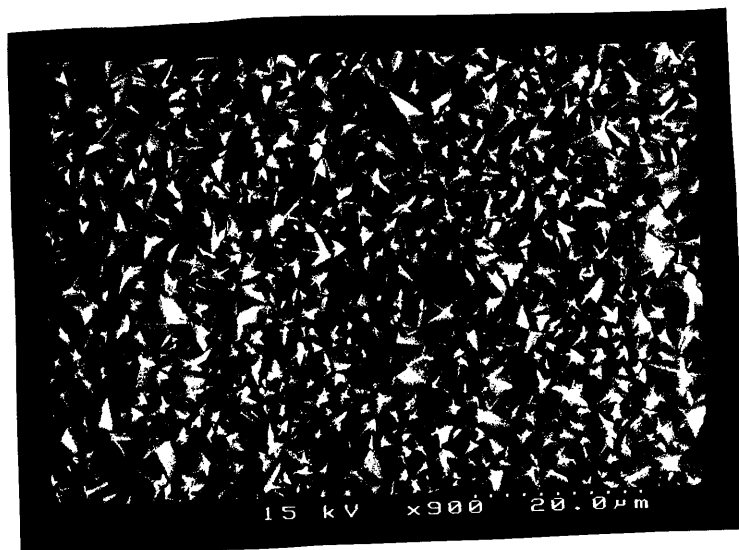
Scan of areas between the center of the film and the outer ring showed that the shape, clustering density and the size of diamond particles are different a good example is the image shown in Fig. 30. In fact, a scan of areas at the center of the film showed a totally different features for the diamond particles (see Fig. 31). Diamond particles structures became a mixture of cubic (100) and octahedron (111) structures; moreover, their size and clustering density are larger than those on the center of the outer ring. The AFM scans gave unreal images of smeared off features as the one shown in Fig. 23.

Fig 30: SEM image of area between the center of the film and the outer ring on sample 111/6.



Diamond films could be made smooth to certain extent using a laser beam of appropriate power. Polished diamond films have many applications. An AFM scan of a polished area of sample 111/6 is shown in Fig. 32. Features of this image are far away from those shown in Fig. 33, this image shows that the polished area became smoother than before i.e., flat, as a result of ablation of the top part of diamond particles. When a large polished area is scanned; laser polishing direction (shadowing) could be recognize, images of such case will be shown.

Fig. 31: SEM image of an area at the center of the film of sample 111/6.



From the previous AFM results; one conclude that **the AFM gave unreal images of the scanned samples**. Fortunately, it has been found that the AFM's head was damaged so it should be replaced with another one. Indeed images from both the optical microscope and scanning electron microscope show that diamond films have different diamond particles shape, size and clustering density as a result of deposition conditions. The effects of deposition conditions on the deposited diamond particles have been investigated in many research works (Ref. 13, 16, and 19).

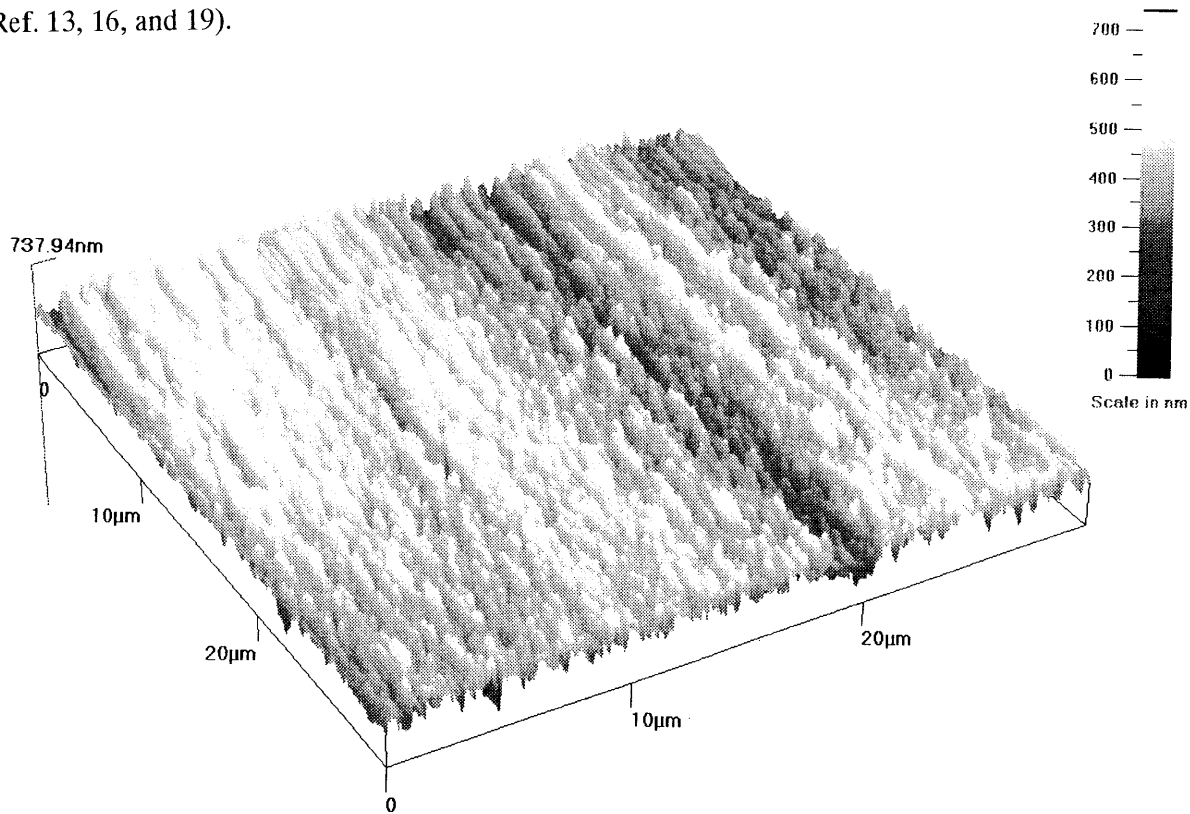


Fig. 32: AFM image of a polished area on sample 111/6.

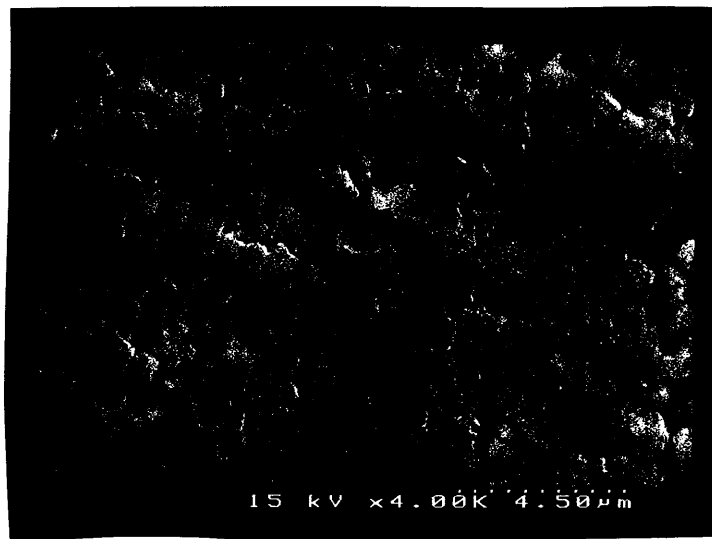
With the new head, the previous work have been repeated. Unfortunately, the results were not much better than those from the old head . The images showed a smeared off features. For example, see Fig. 34. It has been thought that the new head is similar to the old one and has the same Head Parameters. In fact, it has been found that the new head has two different Head Parameters from those of the old one as shown in table 2.

Table 2: The AFM Head Parameters.

Head Parameters	The old head	The new head
Ceramic X Expansion	41.9047617263534	41.91
Ceramic Y Expansion	244.76977131362459	45.00
Ceramic Z Expansion	4.00	4.00

After editing the new Head Parameters, the resulted images are much better than before. some of the good results are shown in Figs. 35, 36, 37, and 38. In fact, these and other results have been produced after many trials in keeping the feedback stable and optimized. The AFM feedback loop was so sensitive to the nature of diamond films as a result of the interaction between the tip and features of diamond particles . Approaching and engaging the sample was repeated many times to get a scan of the chosen area; moreover, many trials were done to get the appropriate values of the Feedback Setting parameters. This parameters optimization of the feedback loop could be done during the scan of the sample.

Fig. 33: SEM image of a polished area on sample 111/6.



Another way to get the sample engaged with the probe is to scan another adjacent area or scan area of different features. Even some times the feedback was lost when while the sample is being scanning. More details about the problems and error messages that have been encountered when approaching and engaging the sample are stated in appendix B. Although each of the two dimensions images shown in Figs. 35, 36, and 37 shows a nice profile -with some fuzzy features- of the scanned surface. Those in three dimensions showed how distractive is the effect of these fuzzy features. They destroyed the true features of the image and in many cases they produced a Z height greater than the Z Dynamic Range of the used head. Fore example, see Fig. 35 (b). It has been found that reducing the values of both the integral (I) and proportional (P) gains reduce these fuzzy features, but not eliminating them. Reducing the Setpoint Offset helped in some cases such that the net force be no less than 1.5 nN.

Whenever a change is introduced to any of the Feedback Setting parameters, the result will be a change on the feedback of the system as well as on the generated images. Thus, as a rule of thumb: when one of the Feedback Setting parameters is changed; the continuous scan mode should be used first to see the changes on the image and when with the resulted image is acceptable, the single scan mode is clicked to store it.

Trials have been done to get rid of the fuzzy features on the images by using different set of : Scan Direction, Scan Profile, Scan Resolution, Scan Rate and Image Mode as well as Feedback Setting. It has been found that the only one gave good results is to change the Image Mode from

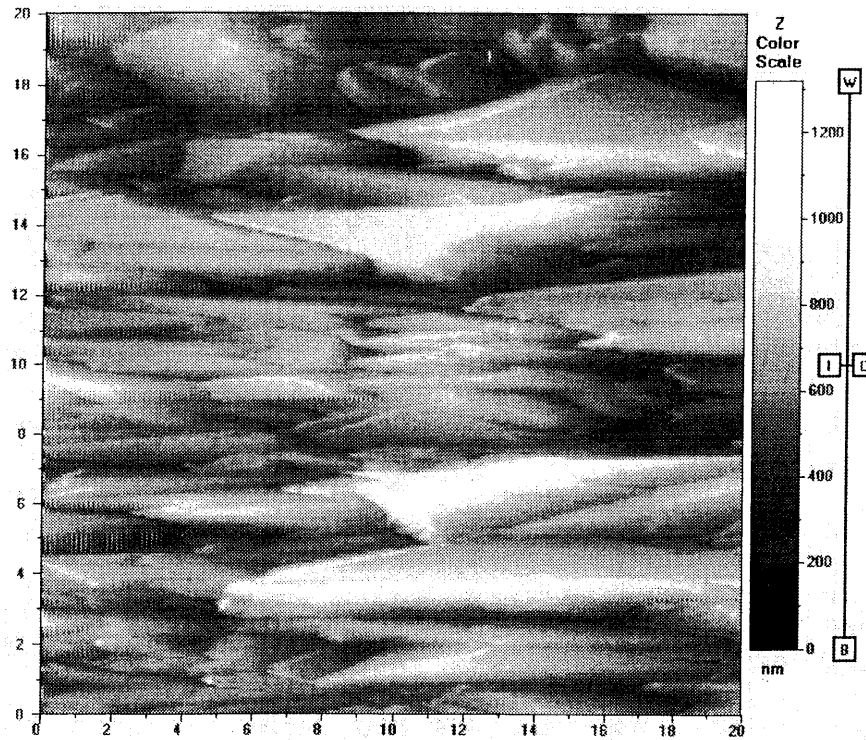


Fig. 34: AFM scan of unpolished area on sample 111/5 using the new head.

Semper Mode to Z height Mode under appropriate Feedback Setting. This fact is illustrated with Fig. 38, in which a non polished area of sample no. 111/6 was scanned first with Semper Image mode and then with Z Height Image Mode under same Feedback Setting parameters.

It has been found that, when scanning diamond films the best values of both the Integer gain and Proportional between 150V and 250V, While that for the Derivative gain are between 50V and 100V. The setpoint Offset should not be lower than 1.5 nN. In fact, an applied force between the tip and the surface less than 1.5nN results in unsensible interaction between the tip and the surface features so the image will be flat featureless plane. Forces greater than 3.5nN may cause the cantilever to generate a high pitched screeching noise as well as clear fuzzy features on the image. In addition, This high value of force may deteriorate the tip, because diamond particles are very hard.

In the following paragraphs, results and result analysis of two samples of diamond thin films are shown such that a general conclusion about the scanned diamond thin films and the Quesant AFM could be stated.

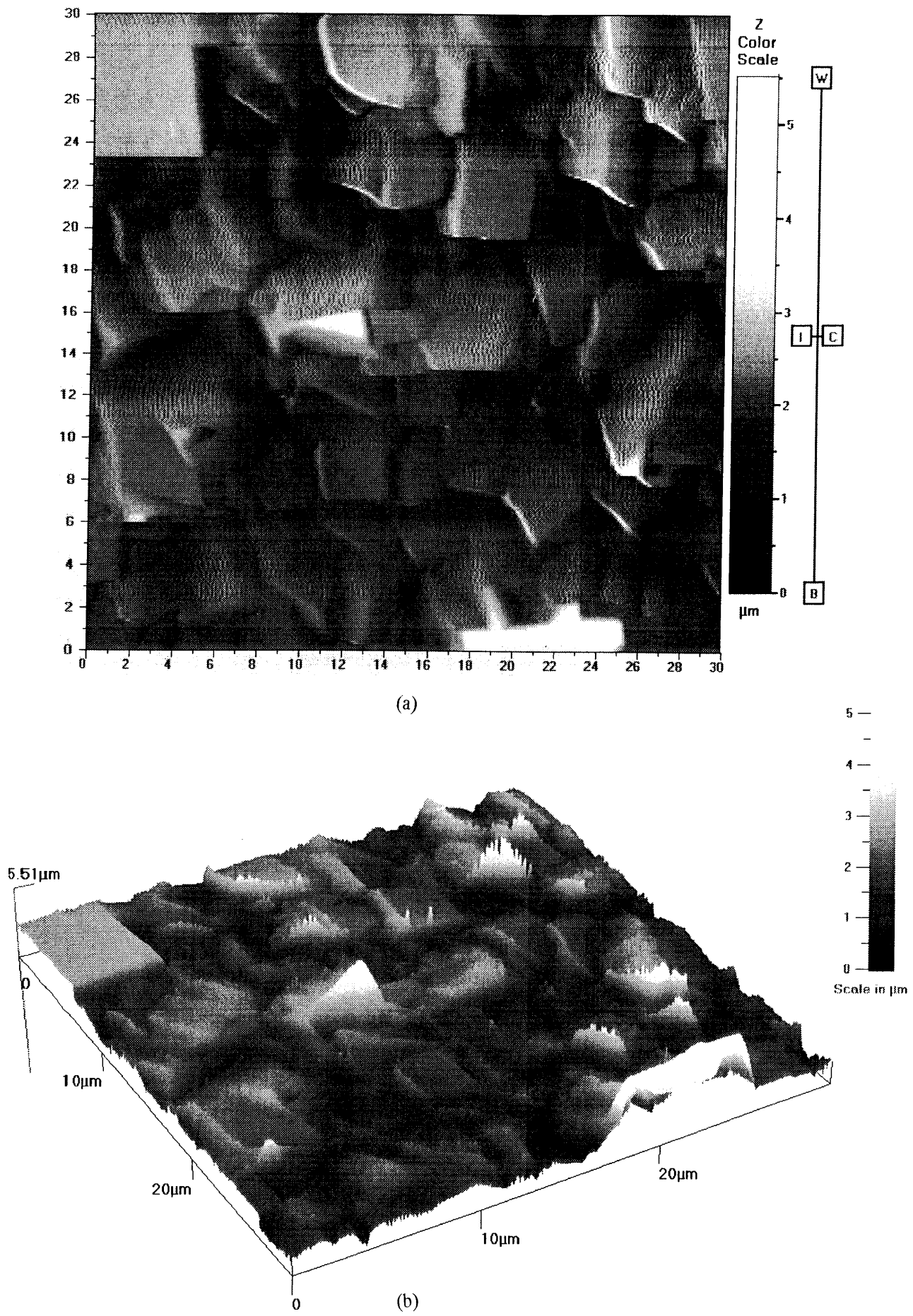


Fig. 35: AFM image of unpolished area at the center of the outer ring on sample 111/6 in 2D (a) and 3D (b).

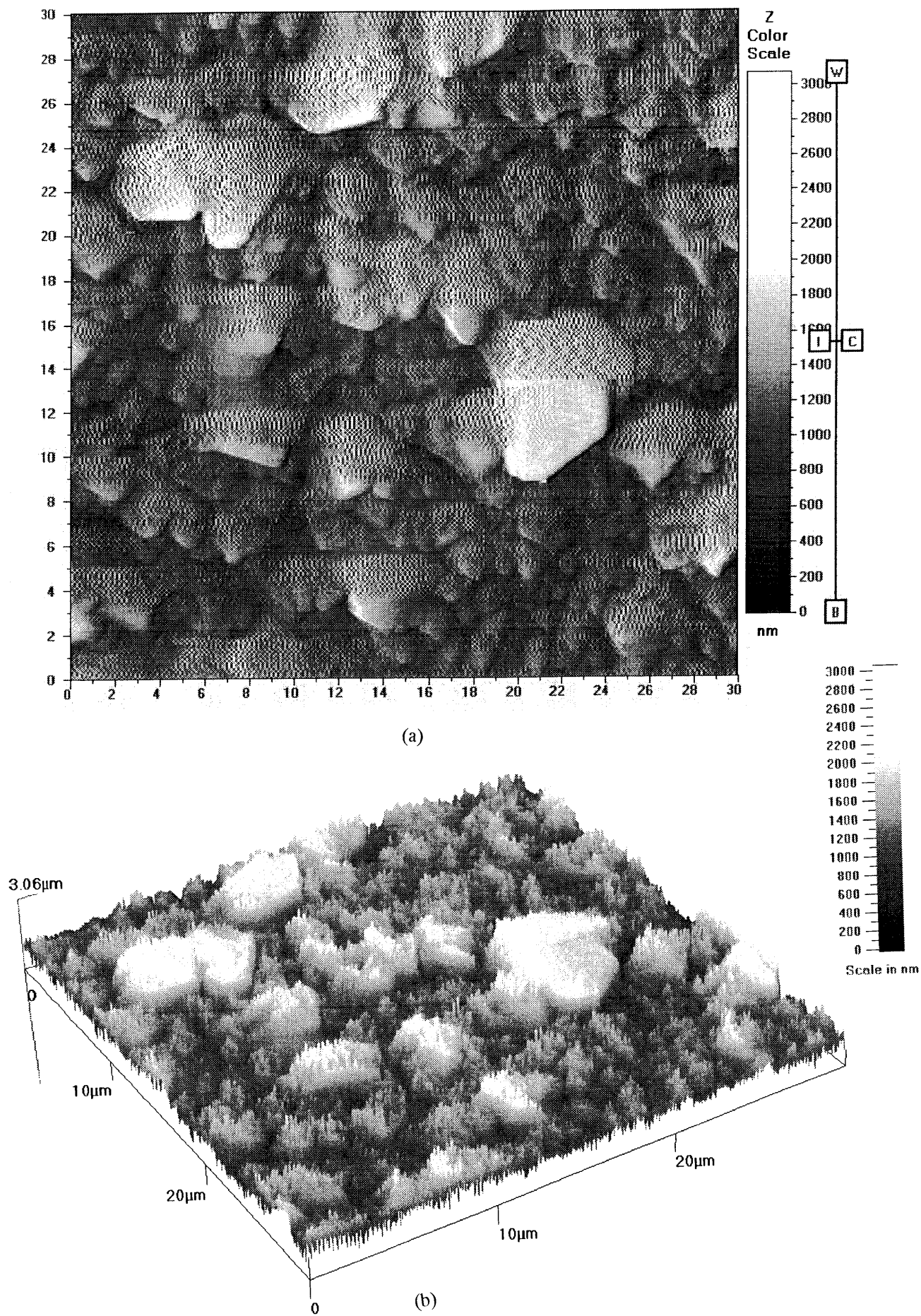
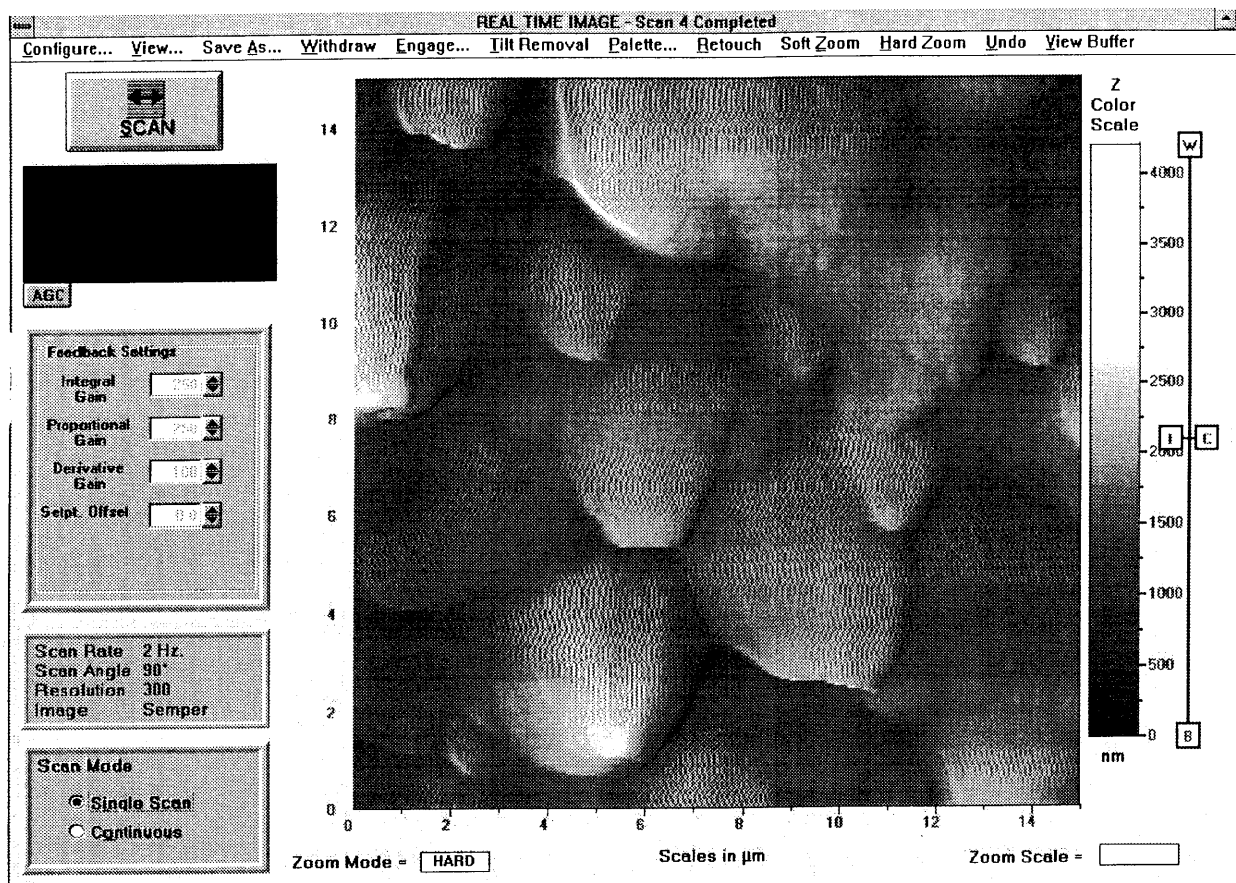


Fig. 36: AFM image of unpolished area away from the center of the film of sample 111/6 in 2D (a) and 3D (b).



(a)

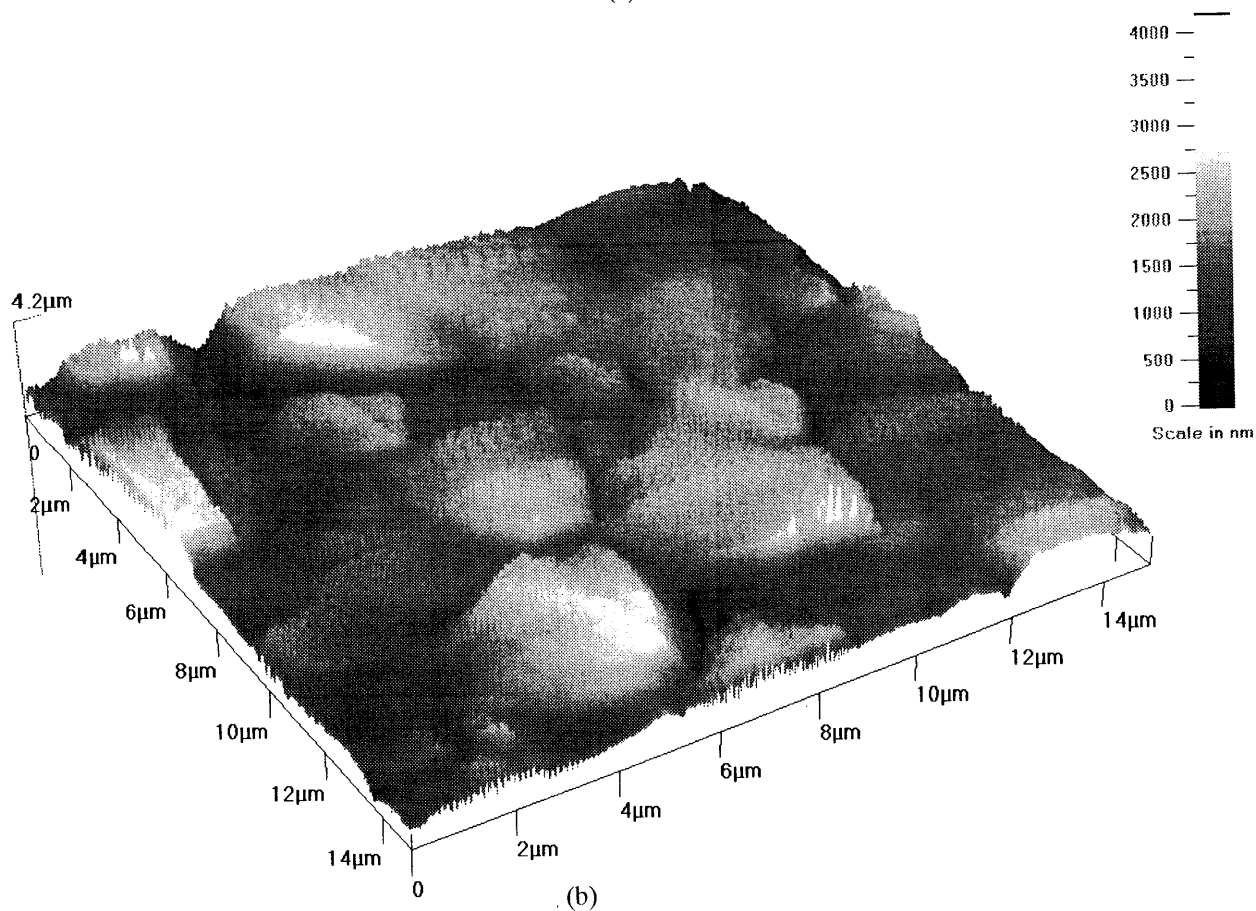


Fig. 37: AFM image of unpolished area on sample 78 in 2D (a) and 3D (b).

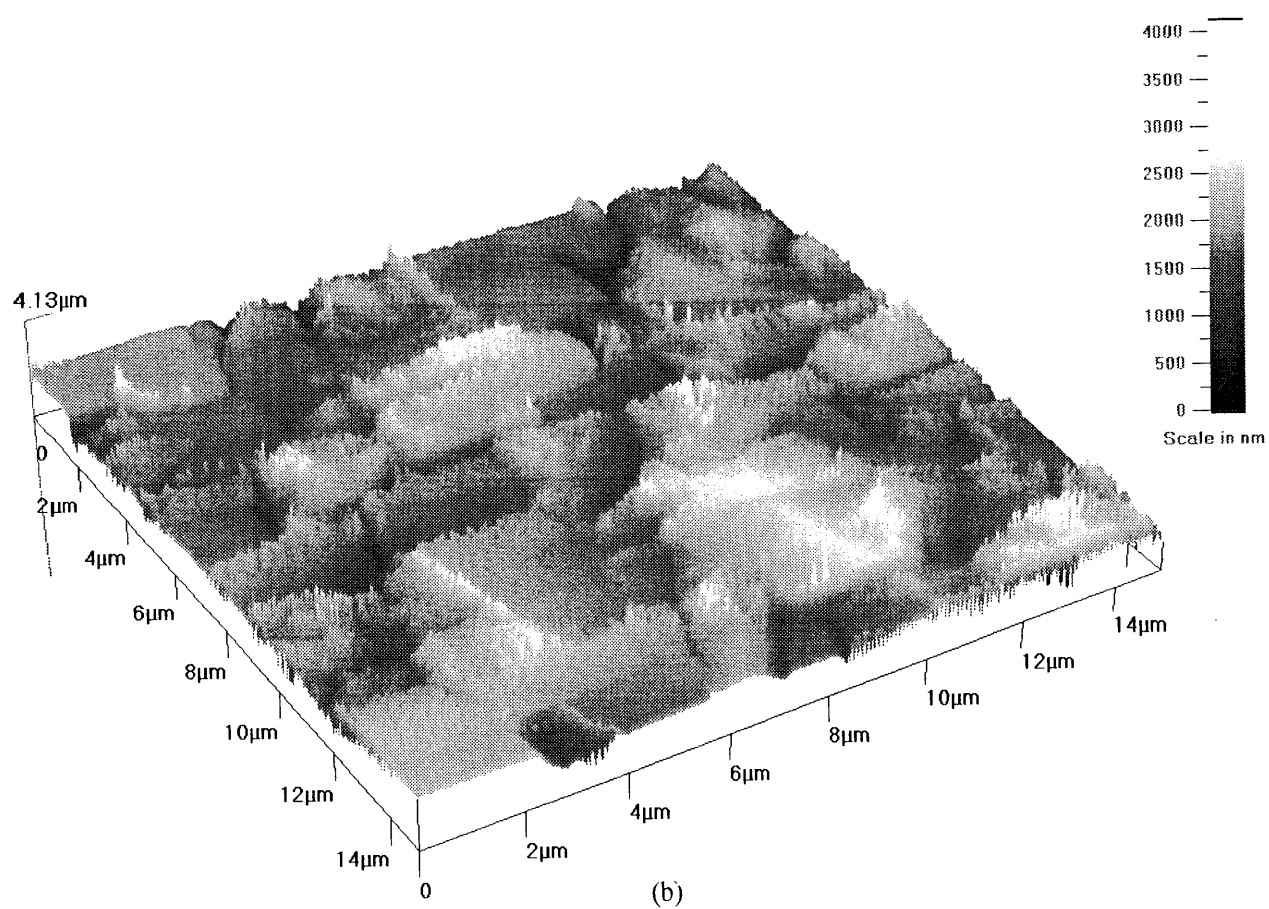
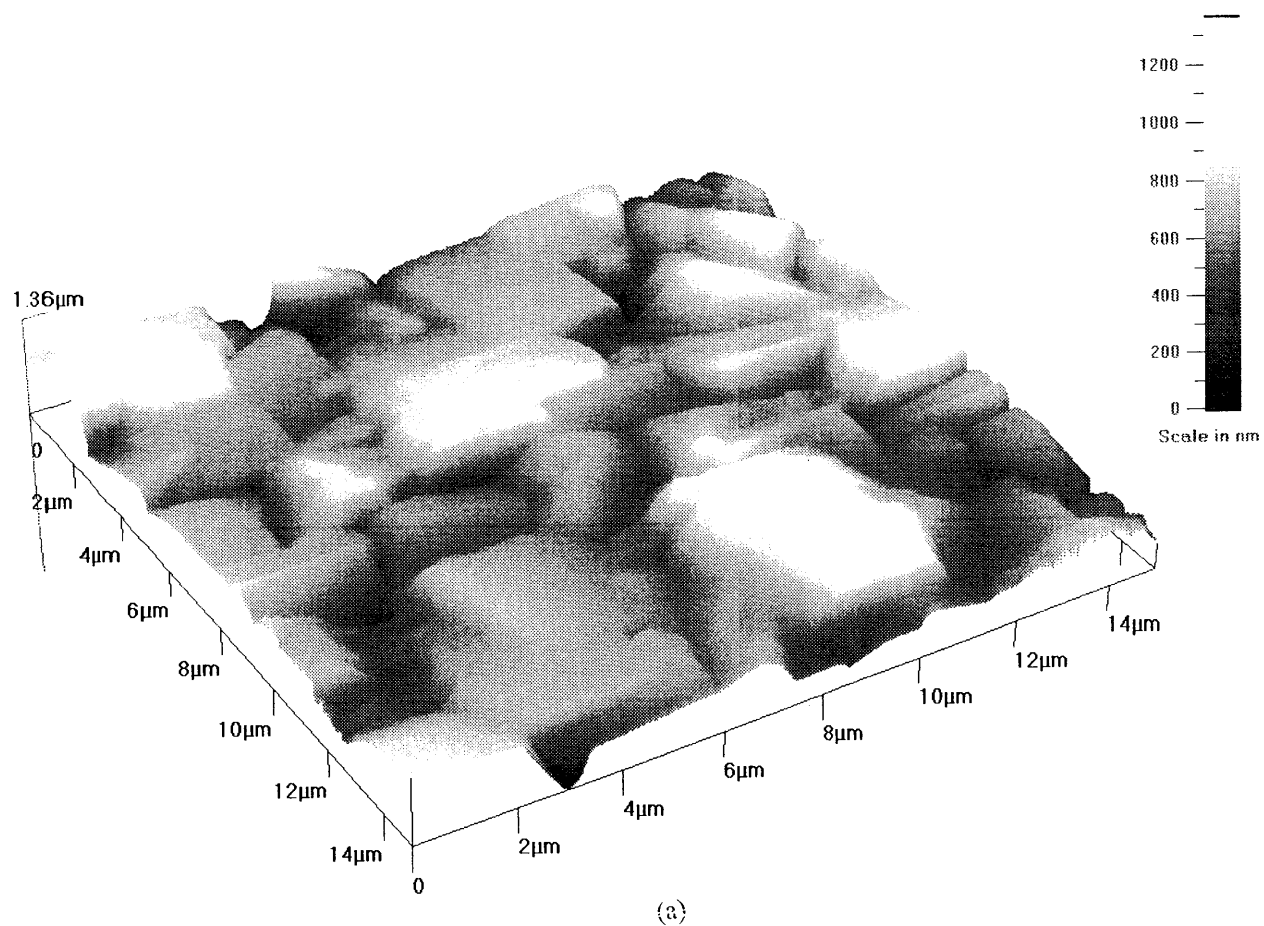


Fig. 38: AFM image of unpolished area at the center of the outer ring of sample 111/6, using the Z height Image Mode (a) and using the Semper Image Mode (b).

Using the Z Height Image Mode, sample no. 78 has been scanned at different places. Fig. 39 shows a scanned area near the center of the film. The surface features in this image are resemble to those shown in Fig. 22. Scans of many unpolished areas proved that diamond particles on the film have different shapes, sizes and clustering density. For example, see Figs 39 and 40. This result is in good agreement with that found by using both the optical microscope and SEM.

Fig. 43: Optical microscope image of a polished area on sample no. 78.

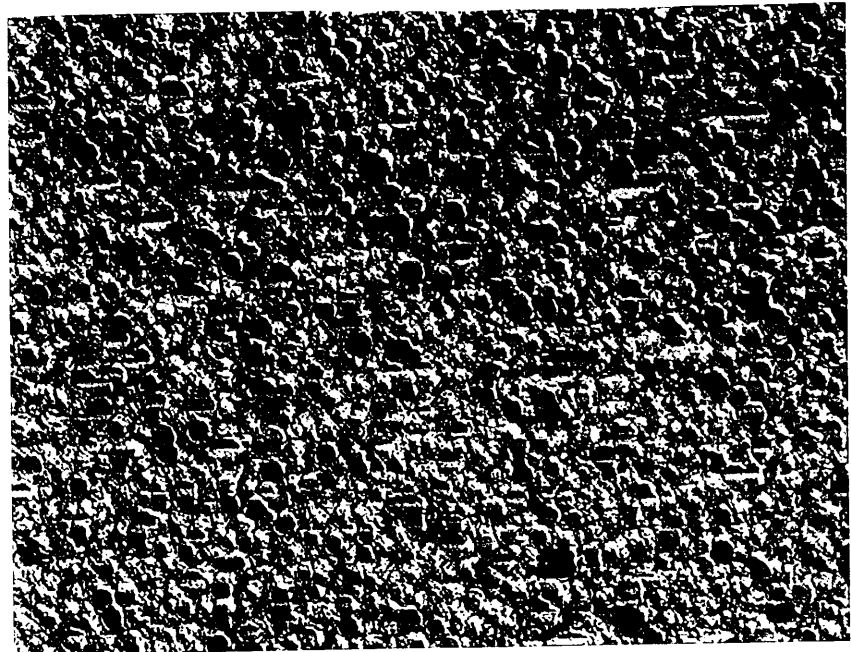


Fig. 40 shows an unpolished area adjacent to a circular polished area. Scan of the outer part of the circular polished area resulted in the image shown in Fig. 41, in this image one can see that some of the upper parts of diamond particles have been removed, statistical analysis shown in table 3 says that the removed amount of diamond is not too large as well as the roughness. Fig. 42 shows the image of the polished area, in which most of diamond particles upper part has been removed so the surface became smoother than that shown in Fig. 40. This result is in very good agreement with that shown in Fig. 43. In brief, diamond film thickness and roughness decreased when it is polished with a laser beam, table 3 shows the quantitative results.

Table 3: Statistical analysis of sample no. 78.

The image	The average height of the film (thickness) (nm)	Roughness of the film (nm)
Fig. 39	831.19	269.70
Fig. 40	812.25	239.49
Fig. 41	806.74	231.72
Fig. 42	653.89	185.27

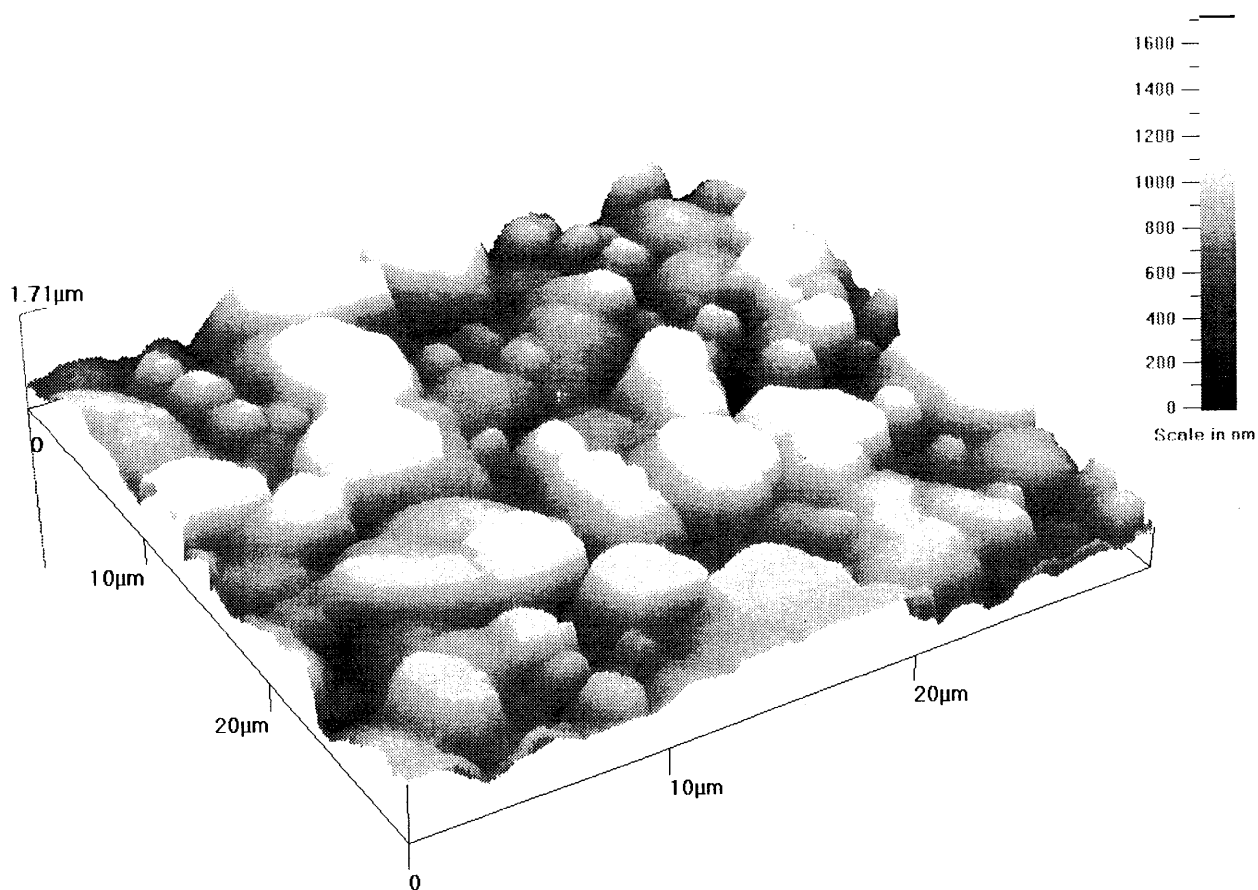


Fig. 39: AFM image of unpolished area near the center of film of sample 78.

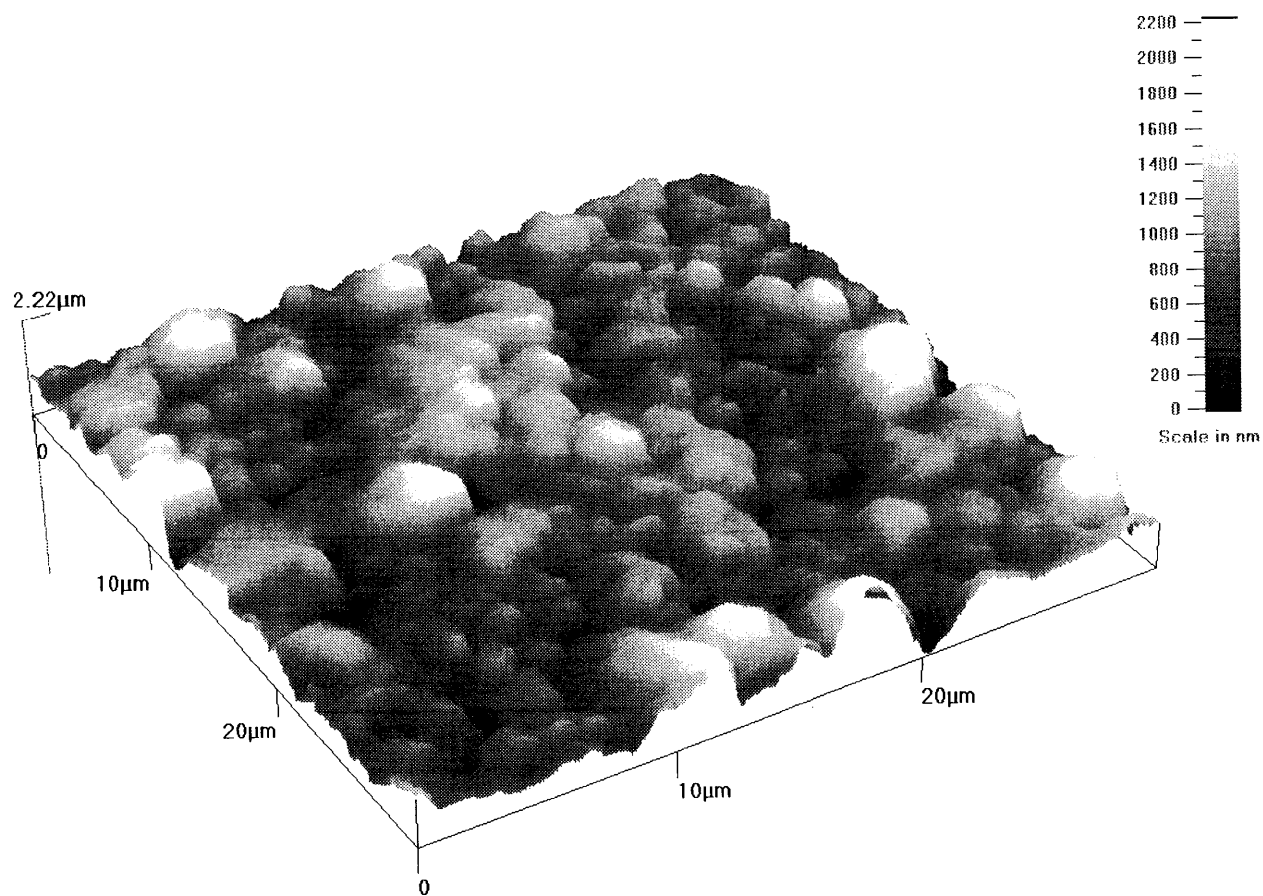


Fig. 40: AFM image of unpolished area which is far from the center of a polished area on sample 78.

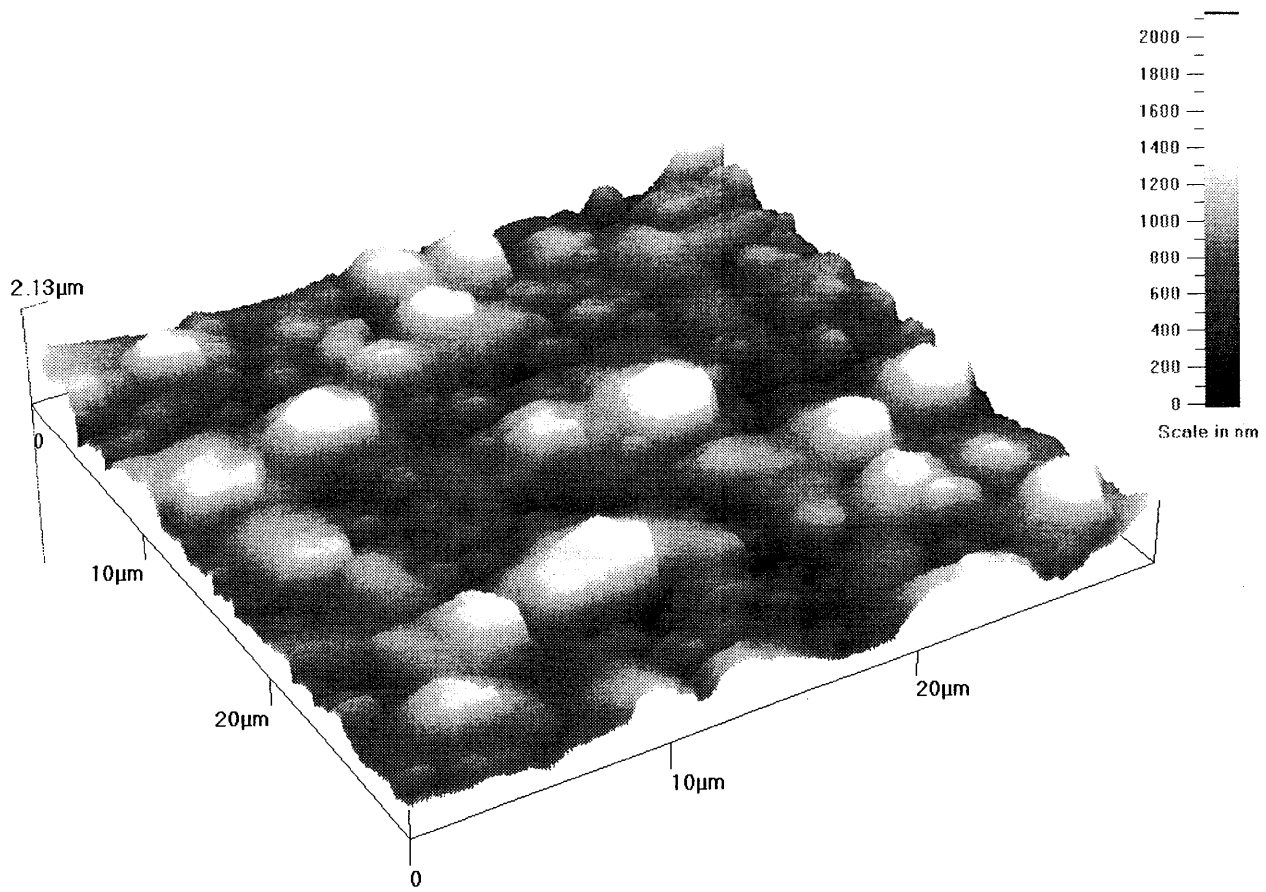


Fig. 41: AFM image of the outer part of a polished area on sample 78.

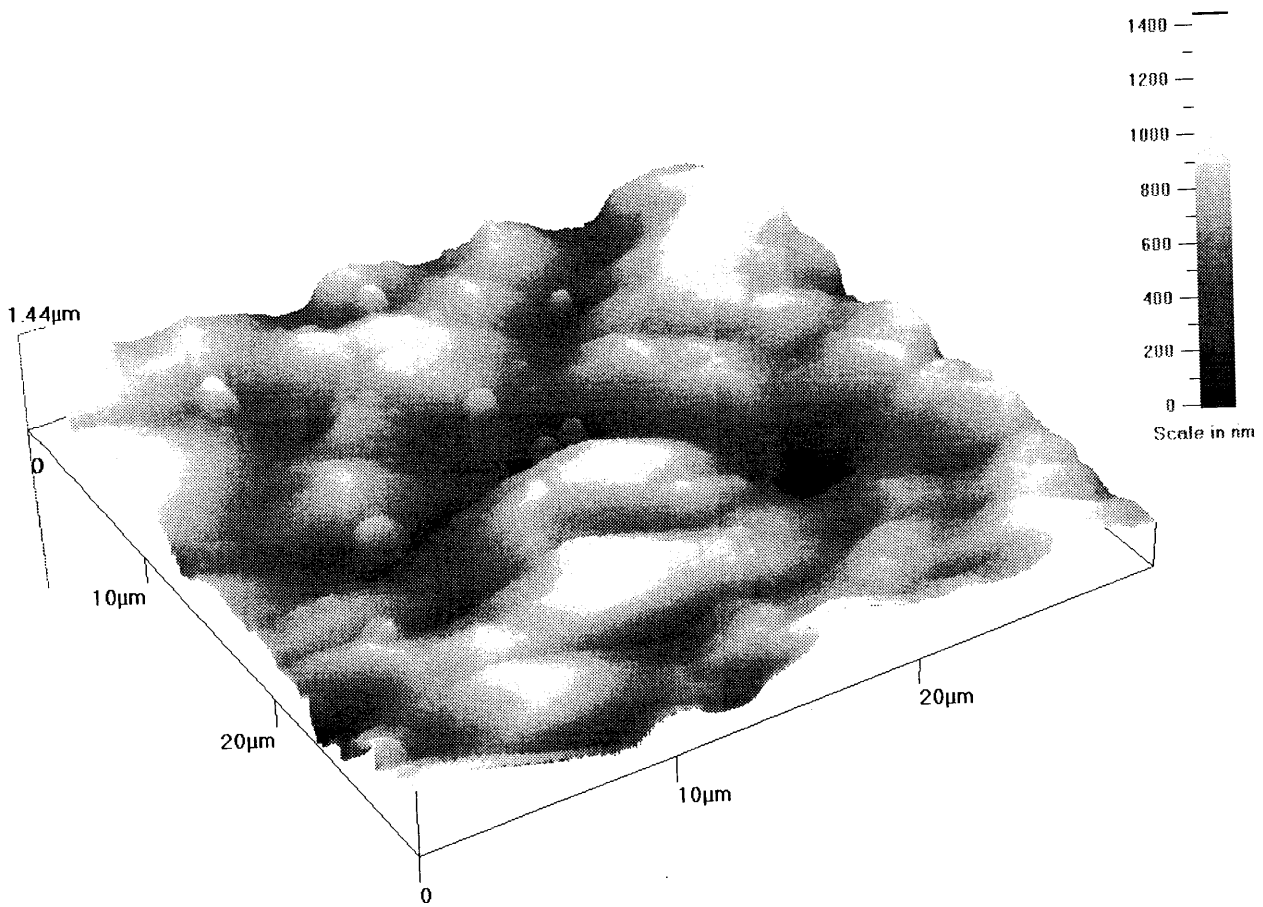


Fig. 42: AFM image of a polished area on sample 78.

The second example is sample no. 111/6. Starting with a scan of the outer ring of the film, Fig. 44 shows two images that are in good agreement with the image shown in Fig. 27. Because the Z Dynamic Range of the used head is smaller than the Z height of diamond particles in that region, it was not possible to get an accurate image of an individual particle. Thus, a head with larger Z Dynamic Range (say 9 μm) is needed. Scanned areas as those shown in Figs. 45 and 46(a) show images of the features found by using the optical microscope and the SEM (see Figs. 29, 30 and 33). The shapes, sizes and clustering density of diamond particles are different at different areas of the film. Thus, those AFM images are real.

In investigating the effect of laser beam on sample 111/6, table 4 as well as the images shown in Fig. 46 and 47 support the conclusion that has been drawn from table 3 and Figs. 40 to 42. Diamond particles have different shapes, sizes and clustering density due to their positions from the center of the film. This may due to existence of different hot zones at the substrate with the hottest one at the center of the substrate where the heating filament is beneath it. Another factor could be the distribution density of the microwave plasma. Since the highest density of the plasma is just above the center of the substrate, where a high and fast rate of deposition is provided so there is no enough time for the deposited diamond particles to form a definite and regular structure like (100). On the other hand, at the outer ring area of the film where the plasma density is low, there is low and slow rate of deposition so diamond particles has the time to grow on the shown structures shown in Figs 27 and 29.

Table 4: Statistical analysis of sample no. 111/6.

The image	The average height of the film (thickness) (nm)	Roughness of the film (nm)
Fig. 44(a)	98.06	66.29
Fig. 44(b)	1850.00	604.98
Fig. 45(a)	1730.00	490.15
Fig. 45(b)	745.07	122.97
Fig. 46(a)	734.41	218.25
Fig. 46(b)	671.42	148.73
Fig. 47(a)	627.29	80.68
Fig. 47(b)	123.87	27.03

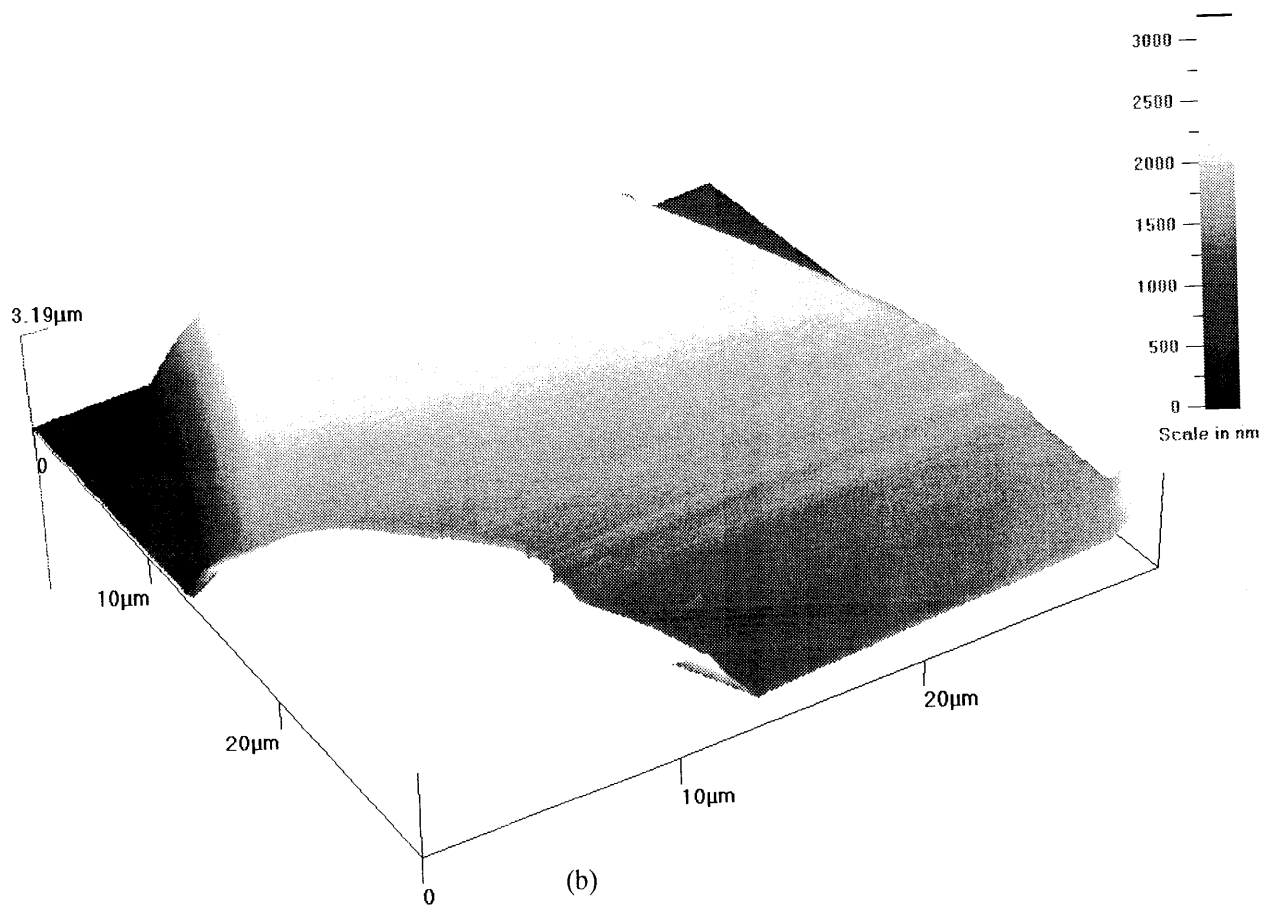
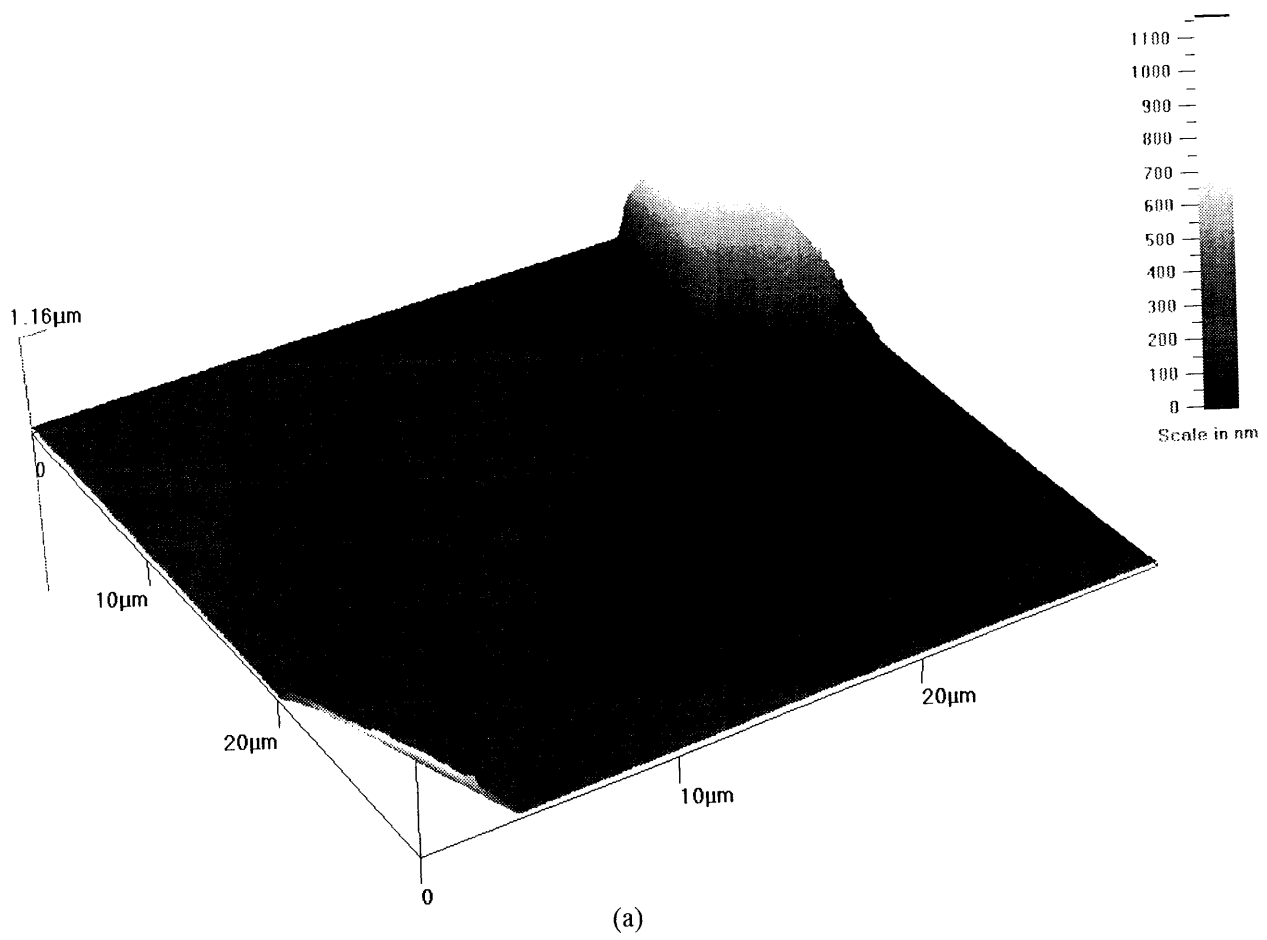


Fig. 44: AFM image of : the farthest part of the outer ring (a), the outer edge of the ring (b) on sample 111/6.

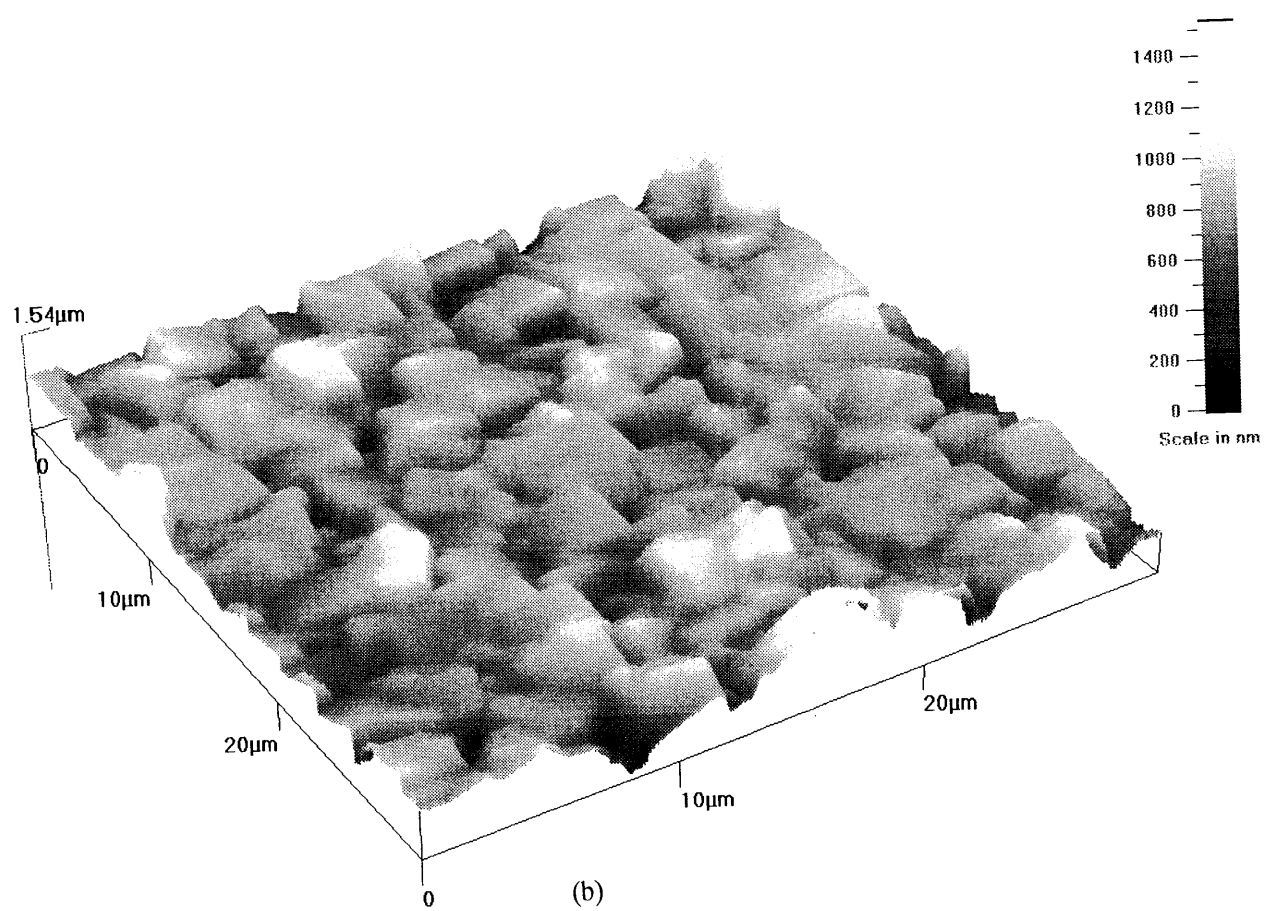
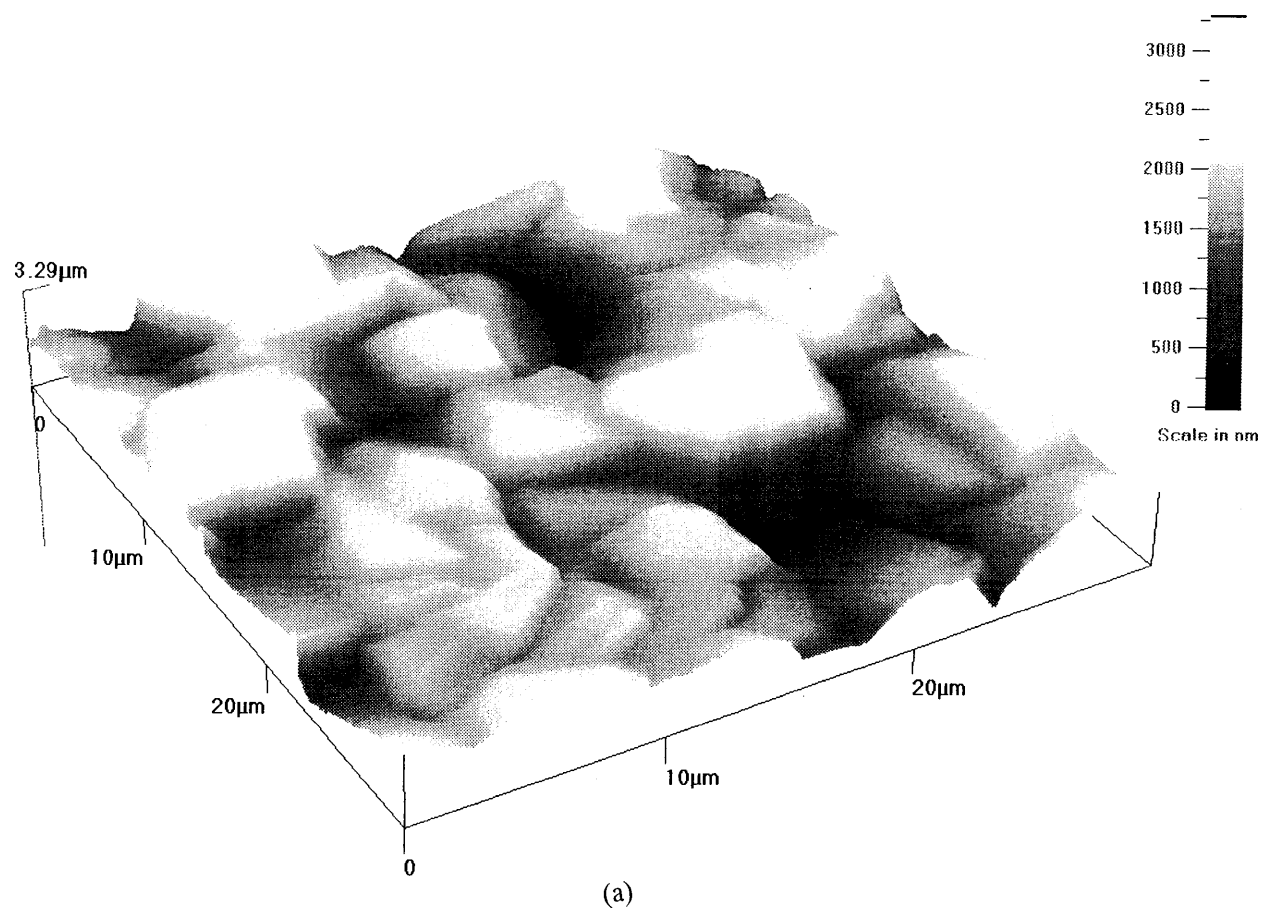


Fig. 45: AFM image of : the central part of the outer ring (a), the inner edge of the ring (b) on sample 111/6.

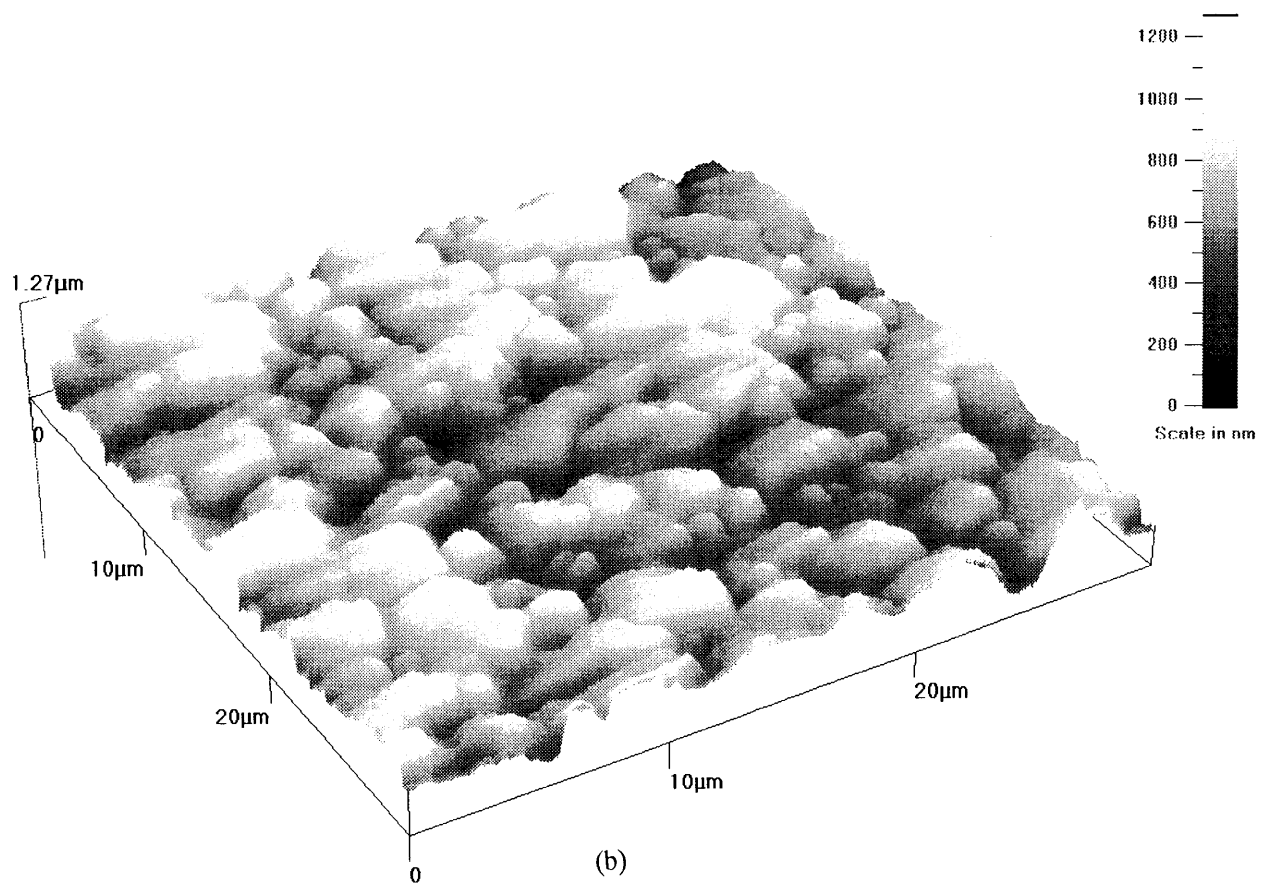
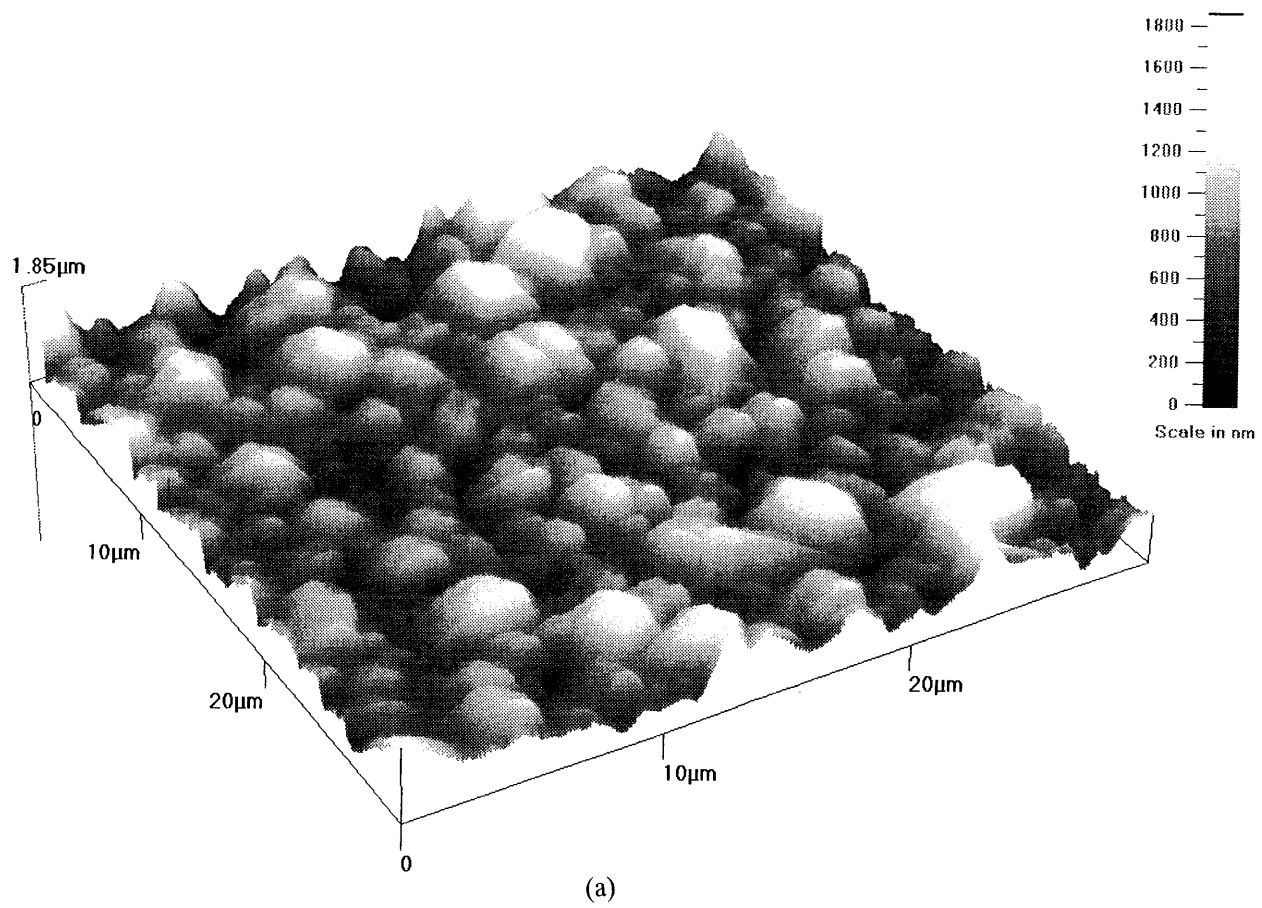


Fig. 46: AFM image of : unpolished area near the center of the film (a), the farthest edge of a polished area on sample 111/6.

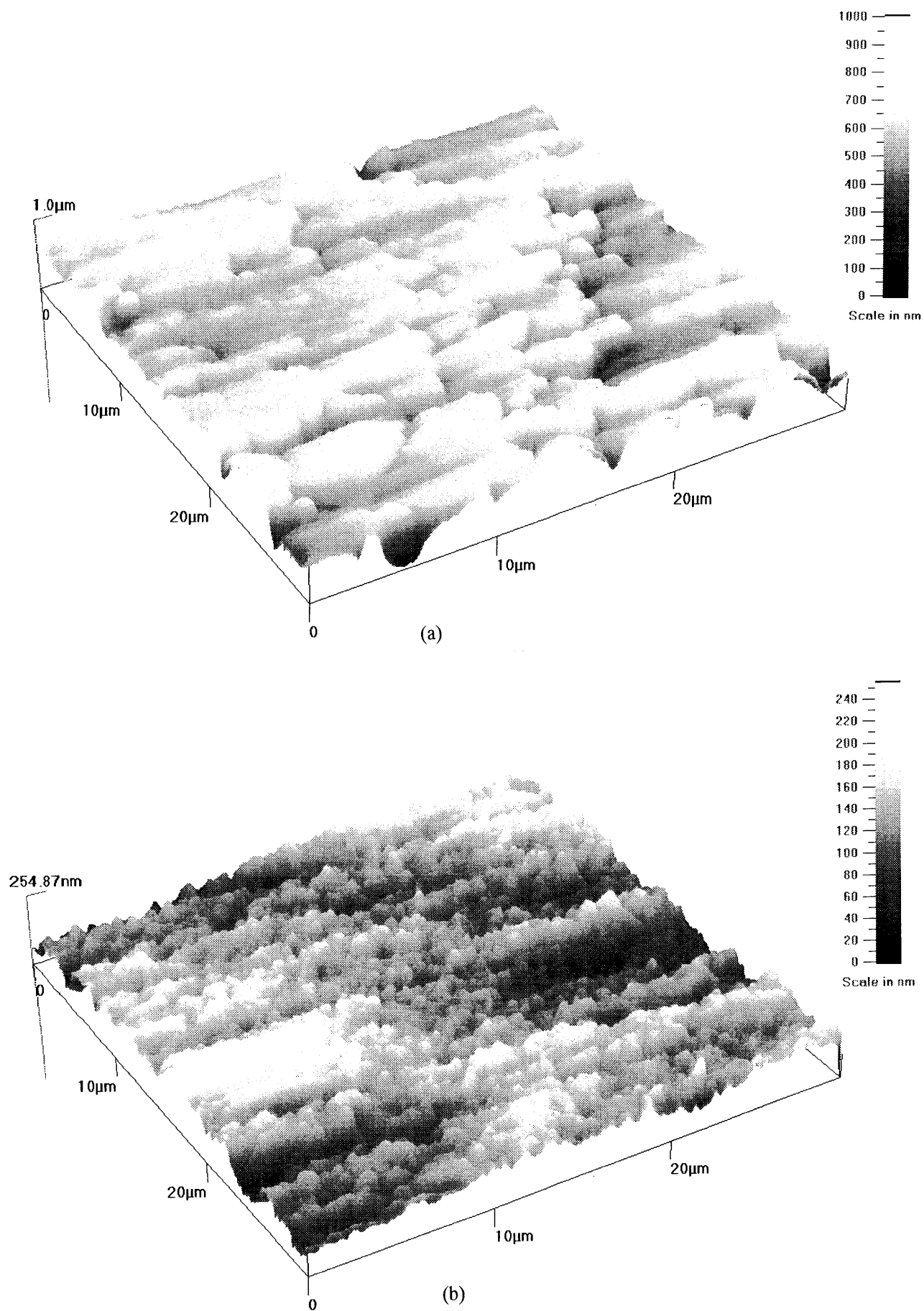


Fig. 47: AFM image of : the outer part of a polished area (a), the center of the polished area (b) on sample 111/6.

It has been found that a laser beam of adequate power has the ability to smooth diamond film, especially the center of the polished area. It is believed that, high quality of polishing could be achieved with a well-collimated laser beam. Since as it shown in Figs. 46 and 47, a non collimated laser beam produced a non uniform polished area.

Analysis of the other diamond thin films lead to the same conclusions.

It is believed that, one of the factors that affects the AFM images of diamond thin films is the geometry of the tip. In the work that have been done by M. George et al. (Ref. 19), they found that the images are affected by the geometry of the cantilever when the imaging of the very steep diamond edges was attempted. The convolution of the AFM tip and a diamond particle is depicted schematically in Fig. 48. The figure shows the interaction geometry of the tip and the cubiclike diamond particle with crystal facets that are sharper than 65° . This result in the displacement of the tip along the faces of the tip itself prior to contact with the real base of the particle. Also considered in Fig. 48 is the 10° tilt of the cantilever which causes the distortion of the crystallinity shape, and in fact more closely resembles to the shape of the cantilever tip as it would be when tilted. The resulted image is depicted in Fig. 48(b), with the diamond particle inside the envelope created by the tip convolution with the particle depicted from the side. In Fig. 48(c), the real feature is shown as it might within the convolution envelope from a top view.

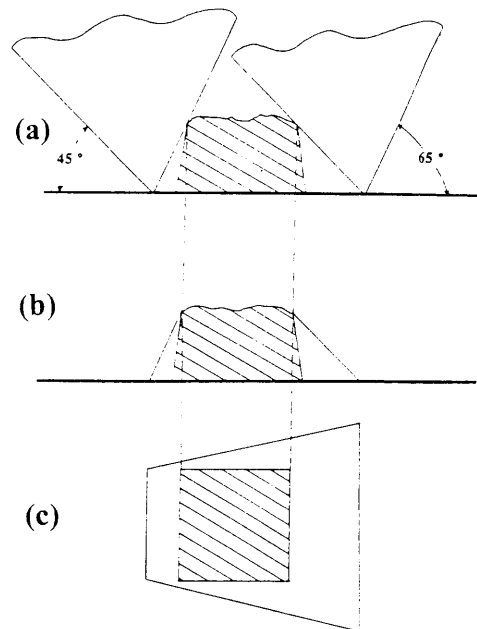


Fig. 48: Schematic drawing of tip-sample geometry. Only the region between the dashed lines would represent the true morphology of the particle. (a) Drawing showing the diamond particle as a sharp protrusion with steep walls, (c) drawing like (b) except as viewed from the top (From ref. 19).

B: Semiconductors Surfaces

An active research work at Physics Department, Heriot-Watt University, is in the growth and characterization of the *Vertical Cavity Surface Emitting Lasers* **VCSELS**. The AFM has been used to scan surfaces of both GaAs substrate and ZnSe before and after etching processes.

Fig. 48, shows a schematic diagram of how a VCSEL is grown. Details of VCSELS growing and characterization are beyond the scope of this project. The aim here is just to reveal the topography of both the GaAs substrate and ZnSe surfaces before and after they have been etched with any of the following etching methods:

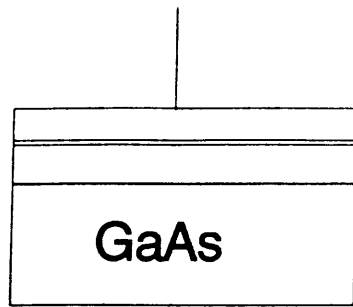
- 1- **Electrochemical etching** using NaOH/H₂O solution (Electrolyte).
- 2- **Dip etching** using NaOH/H₂O₂/H₂O solution continuously stirred.
- 3- **Spray etching** using NaOH/H₂O₂/H₂O solution.

Fig. 49, shows AFM image of GaAs substrate that was ready to grow any epitaxial layer on it. This substrate was supplied from the manufacturer without any pretreatment. The surface is very smooth. After GaAs surface has been thinned from 600 µm to 120 µm and polished with 0.6 µm diamond paste, its surface became rough as shown in Fig. 50. The roughness of GaAs increased when the electrochemical etching method has been used as shown in Fig 51.

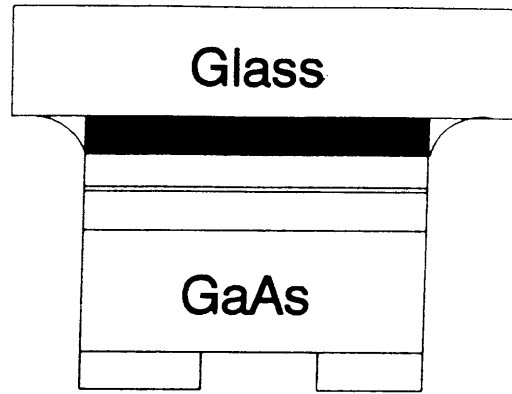
Using the dip etching method makes GaAs surface rough and it could be seen from Fig. 52, that the etching rate is not uniform since it was faster in some places than the others. The features on GaAs surface may be resulted from the way the etching solution was stirred. Random features on GaAs surface resulted when the spray etching method was used, this is shown in Fig. 53. Different surface morphology results when different etching methods have been used. This is the conclusion that can be drawn from Figs. 51, 52, and 53. The etching rate in each method may be not the same.

The above conclusion is true for ZnSe surface. Fig. 54, shows the morphology of unetched ZnSe surface. When the GaAs substrate removed using the spray etching method, ZnSe surface got the morphology shown in Fig. 55. On the other hand, the use of the dip etching method to remove GaAs substrate resulted in a rough surface of ZnSe as shown in Fig 56. To get a smooth surface of ZnSe the spray etching method is recommended. While scanning surfaces of these semiconductor materials there was not any problem with the AFM feedback such as those encountered with diamond thin films. Thus, AFM works much better with semiconductor materials than with diamond thin films.

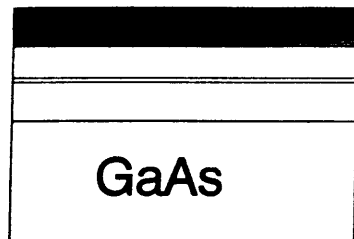
ZnSe/ZnCdSe/ZnSe QW



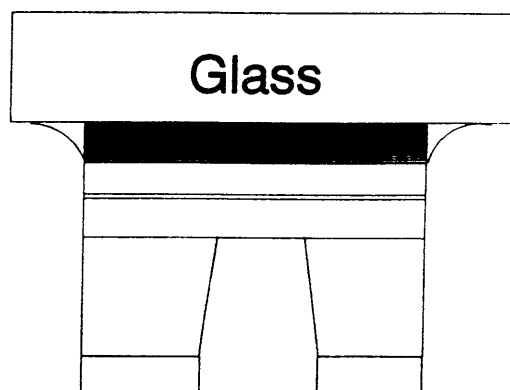
1. Active layer growth by MBE



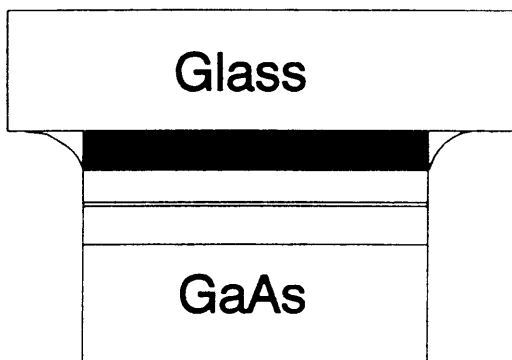
4. Masked for substrate removal



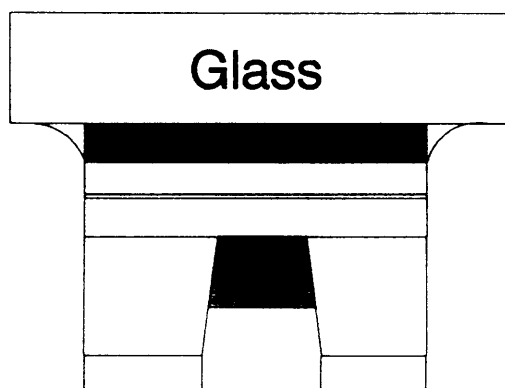
2. Bragg stack growth



5. Substrate chemically etched



3. Mounted on glass



6. Second Bragg stack deposited

Fig. 48: A schematic diagram showing the steps used in growing VCSELs.

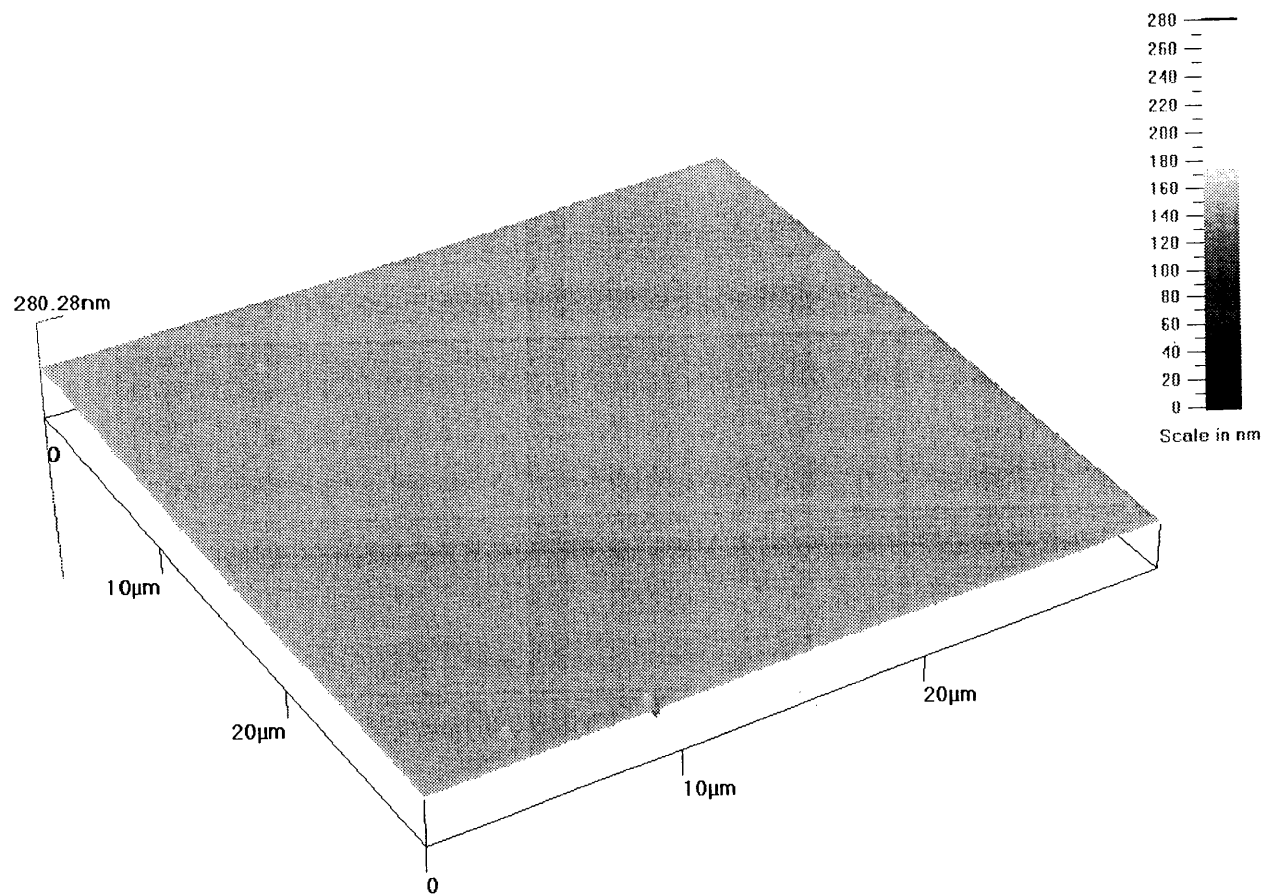


Fig. 49: AFM image of GaAs substrate before polishing or etching it.

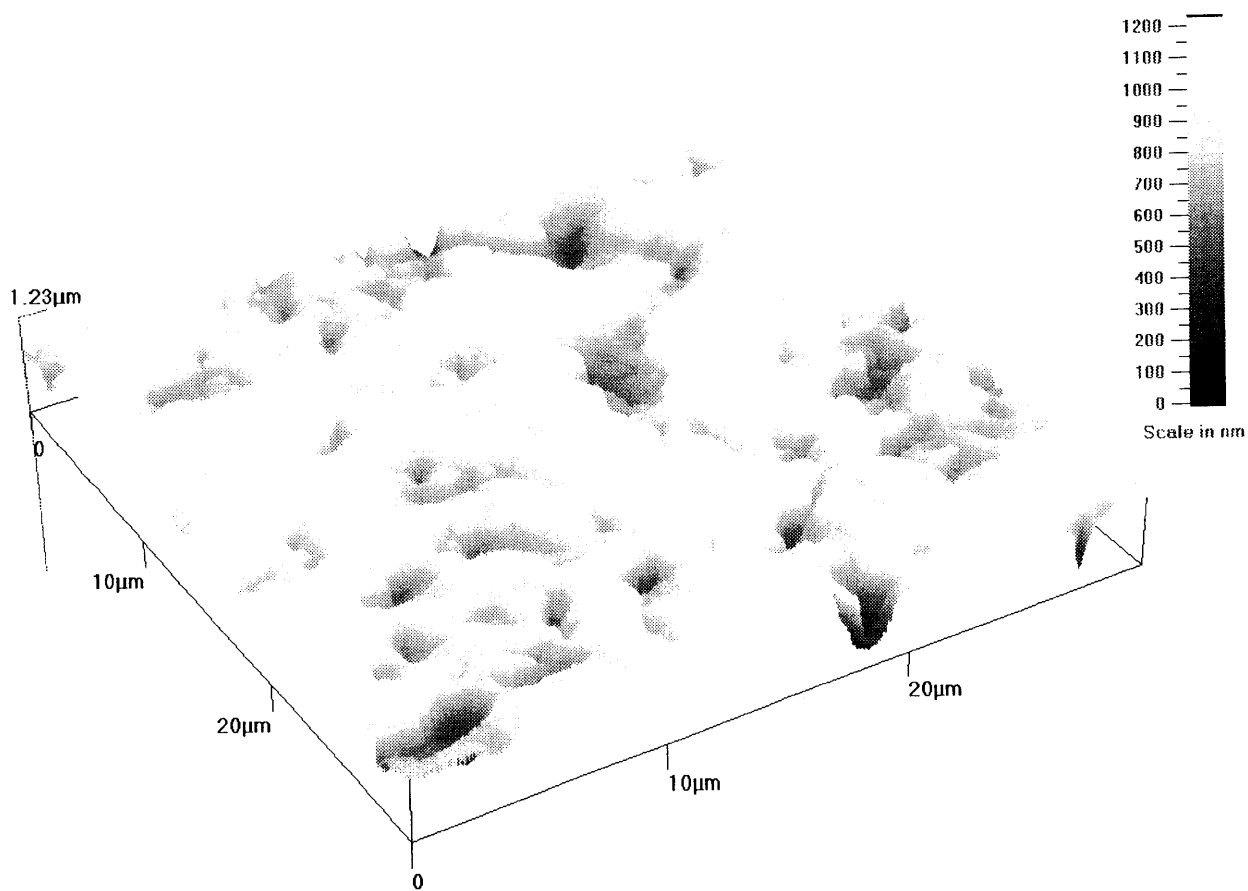


Fig. 50: AFM image of a polished area on GaAs substrate.

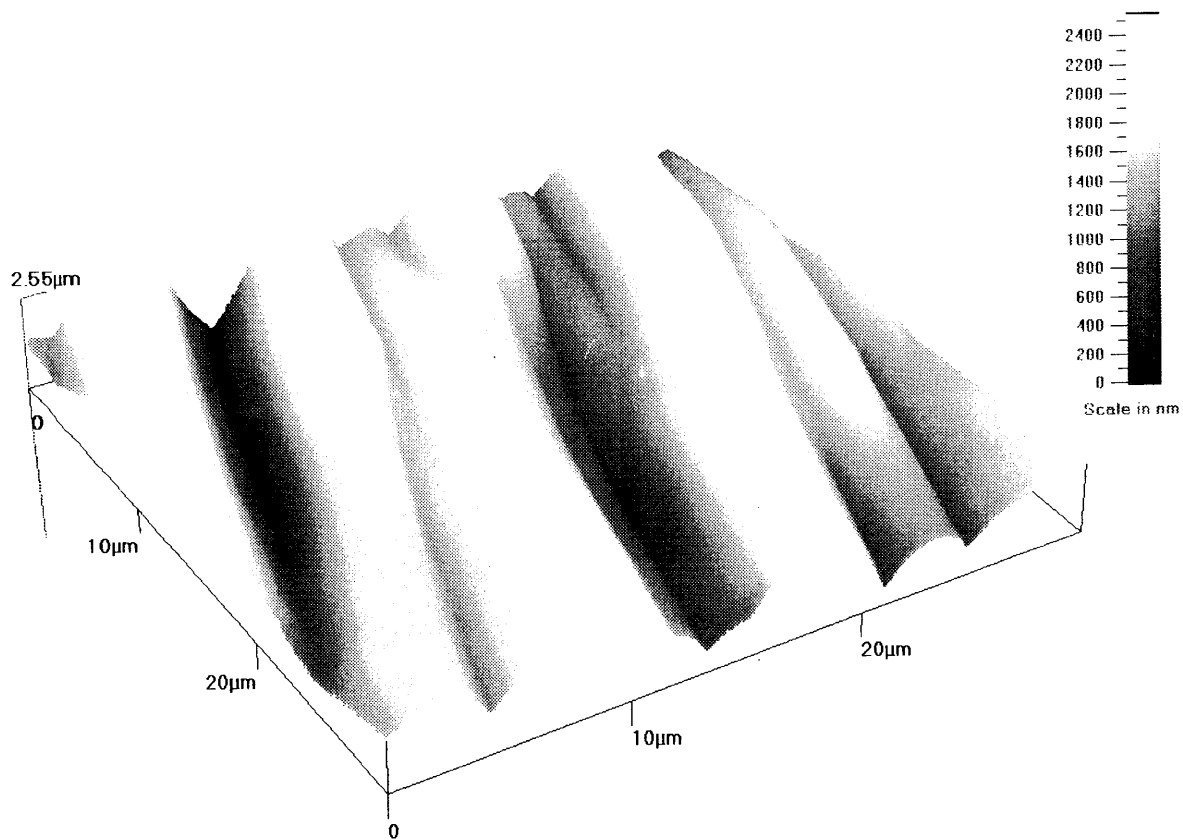


Fig. 51: AFM image of electrochemically etched area on GaAs substrate.

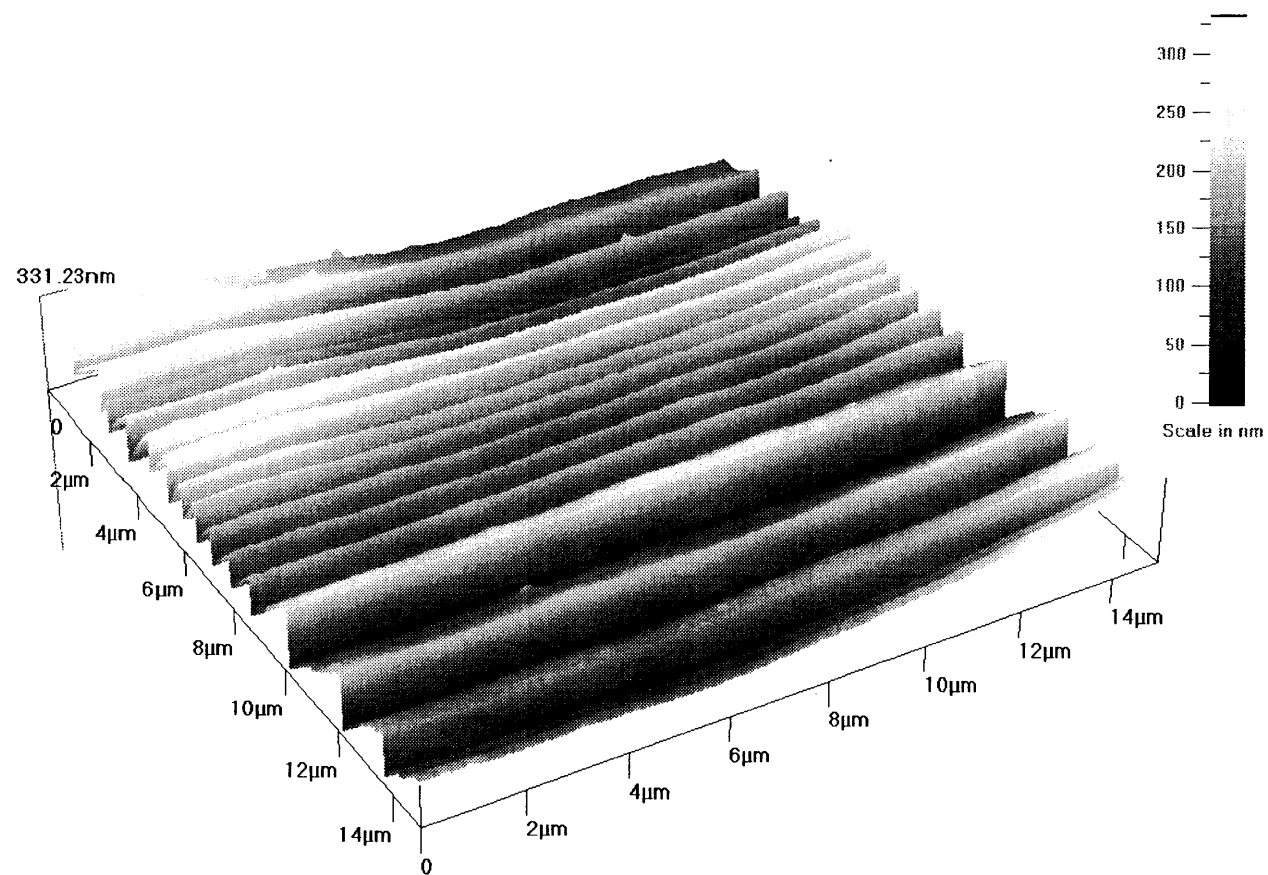


Fig. 52: AFM image of GaAs surface after using the dip etching method.

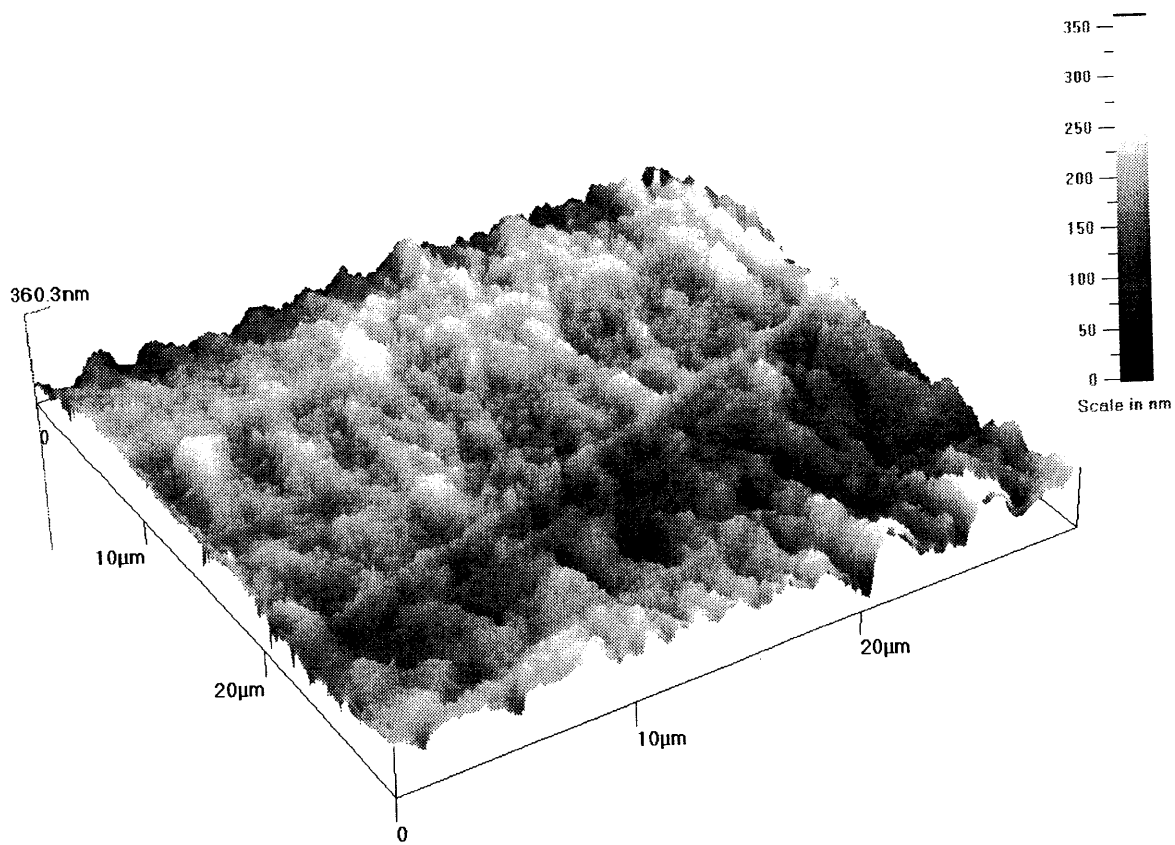


Fig. 53: AFM image of GaAs surface after using the spray etching method.

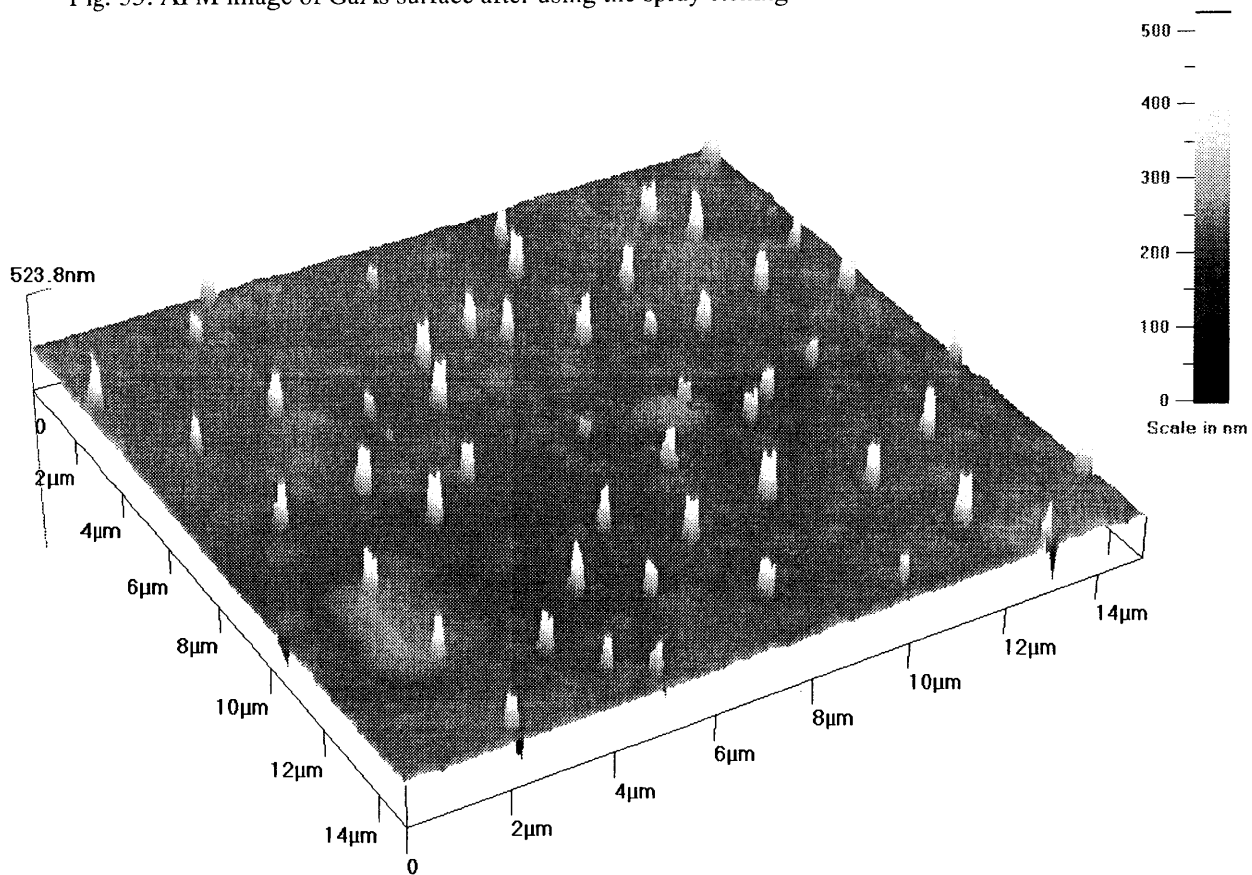


Fig. 54: AFM image of unetched ZnSe surface.

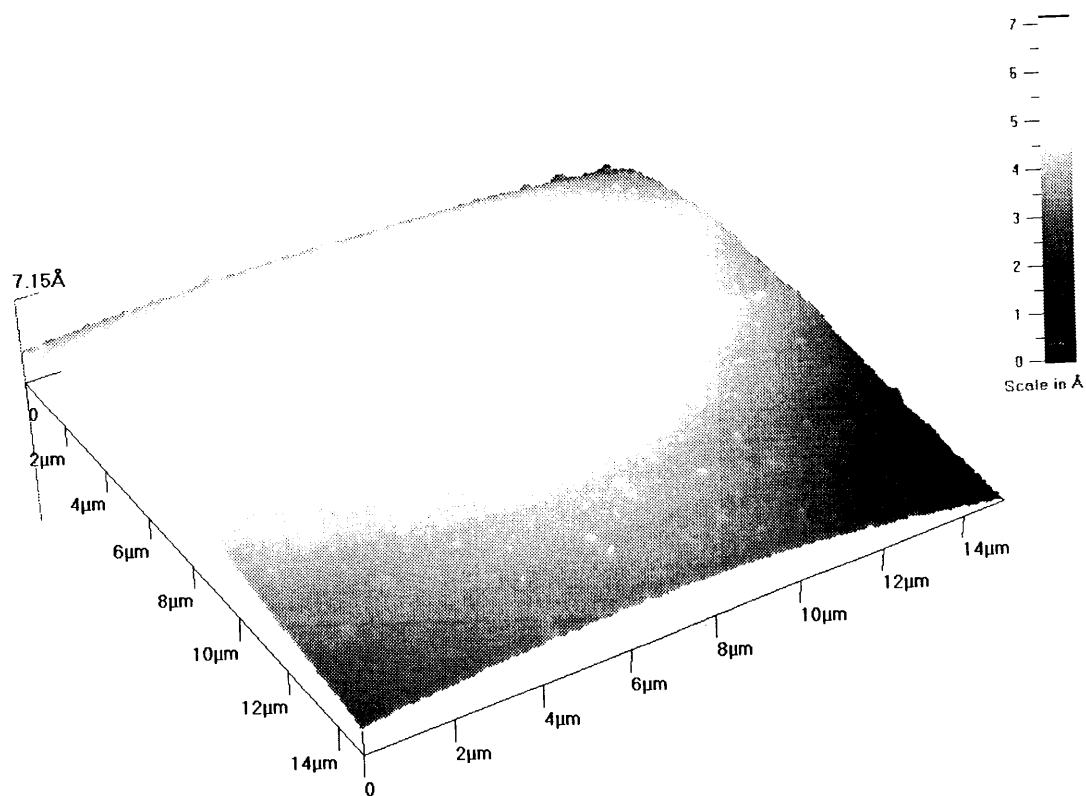


Fig. 55: AFM image of ZnSe Surface with the GaAs substrate removed using spray etching method.

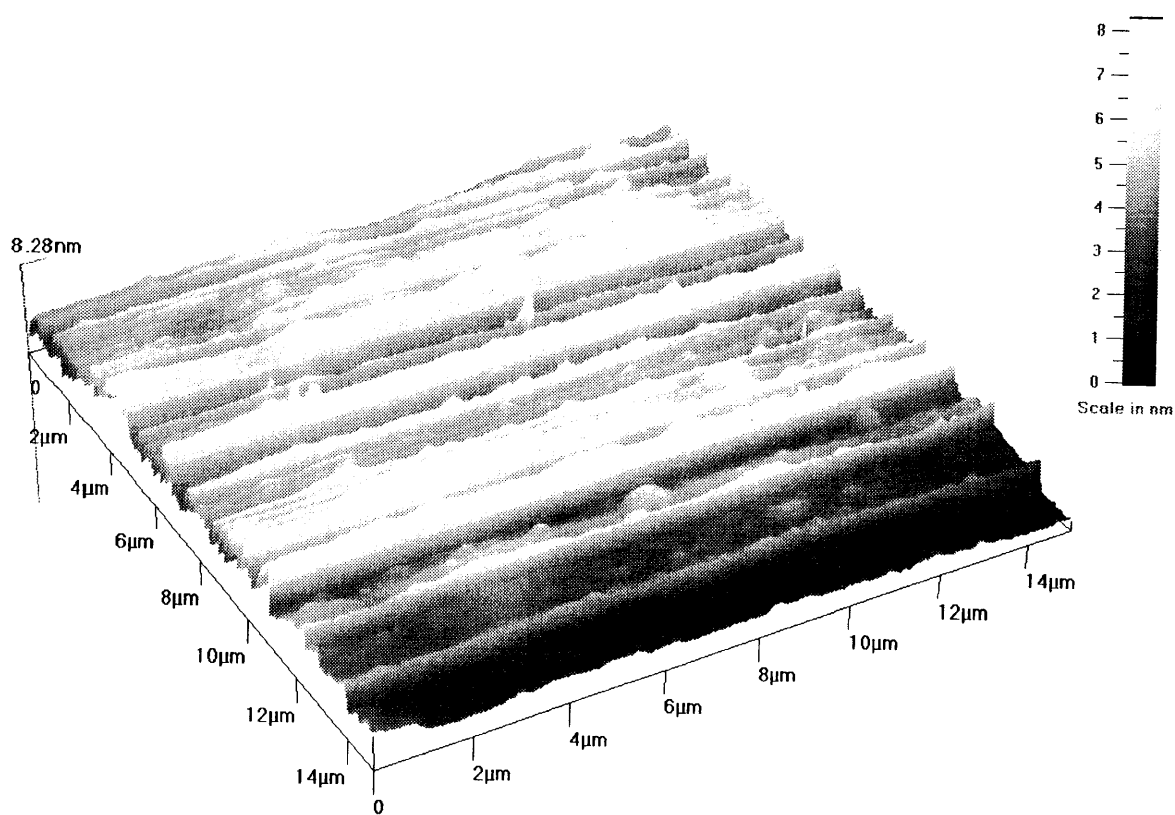


Fig. 56: AFM image of ZnSe surface with the GaAs substrate removed using dip etching method.

Summary

The Atomic Force Microscope **AFM** is a unique and excellent scanning near-field microscope. It has become well-known among scientist and engineers of all disciplines and it finds numerous applications in product development and quality control. **AFM** can generate three-dimensional images of a conductive or nonconductive surfaces. The tip in **AFM** can reveal the surface profiles with atomic resolution. In fact, the **AFM** images are obtained by measurement of the force on a sharp tip created by proximity to the surface of the sample. This force is kept small (3.5 nN) at constant level with feedback mechanisms.

Before engaging and scanning the sample, It is necessary to check the alignments of the probe, laser beam and sensor. Then appropriate Scan Parameters should be downloaded. After the sample and the probe are brought close to each other, the probe and the sample are engaged and the system brought into a condition referred to as “*feedback*”. This is a mechanism that maintains a constant force between the sample and cantilever by sensing the position of the cantilever and adjusting the voltage to the piezoceramics accordingly. Once feedback has been achieved the sample can be scanned. Feedback loop can be optimize during the scan using the “Feedback Settings” box and the Download button.

It is recommended (after scanning the sample) that the raw image first to be saved without any modifications such as tilt removing. The analysis menu could be used to perform statistical and dimensional analysis of the saved image.

Diamond is a fascinating material. It has many unique properties due to its unusual bonding. These extreme properties make diamond technologically useful. The images resulted from the AFM, optical microscope, and scanning electron microscope SEM showed that the deposited diamond particles have different shapes, sizes, and clustering density at different areas of the film. It is believed that, deposition conditions and the spatial distribution of the plasma are the key factors in deposition processes.

It has been found that a laser beam of adequate power has the ability to smooth diamond thin films, especially the center of the polished area. It is believed that, high quality of polishing could be achieved with a well-collimated laser beam. In scanning

diamond thin films, not only the unstabilty of the feedback loop that affects the images but also the geometry of the tip especially when imaging sharp or steep diamond edges. It has been found that Quesant AFM works much better with semiconductor materials than with diamond thin films. From Figs. 51, 52 and 53 one conclude that using different etching methods result in different surface morphology.

Acknowledgments

I wish to express my gratitude to the University of Bahrain for scholarship and the support provided throughout the duration of my MSc study. A special thank to my supervisor Dr. Gerald Buller, Dr. J.I.B. Wilson and Mr. Matthew Trew for thier guidance, encouragement and input. The input of Mr. Wyn Meredith is also acknowledged.

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Appendix A

Scan Parameters

As it was stated in section 2-4, Scan Parameters should be loaded before engaging the probe and sample otherwise an error message will be given as shown in appendix B. A brief description (of that stated in ref. 10) of the Scan Parameters shown in table 1 is given below:

Scan Size indicates the square root of the area scanned. The smaller the value the higher the magnification.

Scan Rate is the number of horizontal scan line per second. The lower value of the scan rate the longer the time required to acquire the image.

Setpoint Offset is used to increase or decrease the setpoint force in nN initially set in the “Beam Align” window. A positive value *decreases* the value of the force initially set.

Scan Resolution is the number of data points per horizontal line, which is also equal to the number of lines within the scan. Higher scan resolutions reveal greater image detail but also take longer.

Z Input Gain is a measure of the sensitivity of the input amplifiers.

Z Dynamic Range affects the maximum Z height and the resolution achievable in Z. Its value depends on the type of scanning head used.

Scan Direction is in degrees and is the direction of scan relative to the stage.

Integral Gain is a feature of Analoop and used to optimize the feedback loop.

Proportional Gain is a feature of Analoop and used to optimize the feedback loop.

Derivative Gain is a feature of Analoop and used to optimize the feedback loop.

Image Mode refers to the way Z height features are measured.

Z-Height: This is a constant force mode. The feedback loop follows the contours of the surface. The true Z height features of the surface are revealed in the height mode.

Lateral Only: This scan mode measures the torsional force, i.e., twisting of the cantilever. Tilt Removal is not needed on lateral force scan because there is no Z axis tilting.

Semper Fidelis is an imaging mode in which the error signal is added to the Z height signal. This image mode allows the system to respond faster and may have advantages in imaging surface with sharp features.

X/Y Disable for normal operation the “No” option must be activated. For diagnostic mode “Yes” option must be activated.

Scan Profile affects the relative partitioning above and below the sample plane of the available Z Dynamic Range which has been selected. The default setting, **Hills and Valleys**, partitions Z equally above and below the sample plane. Selecting **Hills** allows more available Z range above the sample plane whereas **Valleys** does the opposite.

Appendix B

Common Problems and Error Messages

Because AFM is delicate and sensitive instrument, any small perturbation to its software or hardware results in many problems that terminate the head and DIP functions. Some of the common problems and error messages that could be encountered while using the AFM are discussed, their natures, causes and solutions. Many others have been stated in the Quesant AFM User's Manual (Ref. 10).

1- *The beam intensity on the "Beam Align" window has dropped to zero after the probe has been brought close to the sample.* This problem may be resulted from forcing the cantilever into the sample and may lead to cracking the cantilever. Thus, Withdraw the tip from the sample and align the laser. Then repeat the approach and if does not work, change the cantilever.

2- *If a transient electric noise has affected the system, unexplained features may appear in any of the program windows.* Thus, close the main menu and re-boot the computer after exiting from the windows.

3- *The system will not go into feedback as shown in Fig. 1B or goes into extremely unstable feedback as shown in Figs. 2B and 3B.* The surface of the sample may be reflecting laser light into the detector system, the gain parameters may be too high or the setpoint force may be too low. Thus, withdraw the tip from the sample and restart the approach. If it dose not work try the following:

- ◆ Select a greater setpoint force on the "Beam Align" window.
- ◆ Reduce the Proportional and Integral gains.
- ◆ Once you have achieved good feedback and have started a scan, optimize the gain and setpoint force settings during the scan using the "Feedback Settings" box and the Download button.

4- *Feedback control cannot be maintained after the probe and sample are engaged as shown in Figs 4B and 5B.* The probe or probe holder may be defected or dirty. The laser and/or sensor may be out of alignment. The mechanical path from the sample to the stage may be loose. The feedback setting may be wrong. Thus, replace the probe and clean the probe holder. Then re-align the laser and sensor and finally re-optimize the feedback loop.

5- *Control of the feedback loop cannot be maintained during scanning as shown in Fig. 5B.* The cantilever may be or otherwise damaged. The sample features may be too high or deep for the scanner's maximum Z height range. The sample may have too much tilt. Thus, replace the

probe. Level the sample. Then scan a smaller area of the sample and increase the setpoint force in the “Beam Align” window.

6- *Streaks appear in the image* as shown in Fig. 6B. The probe tip may have picked up a particle of dirt or the setpoint force may be too high. Thus, replace the probe and reduce the setpoint offset in the “Feedback Settings” box and clicking Download.

7- *When the sample is engaged a high pitch screeching noise occurs along with dramatic oscillations of both traces on the “Approach Response” window* as shown in Fig. 7B. Engagement has occurred with the probe tip on a steep or otherwise irregular feature of the sample. Thus, Move the probe tip to a flatter area of the sample or until the screeching and oscillations stop.

8- *Approach started without properly setting up the instrument* as shown in Fig. 8B. You have not clicked Download on the “AFM Configuration menu” before starting to approach the sample. Click configure in the menu bar and then click Download at the bottom of the window.

9- *Laser signal dropped to zero. Approach aborted* as shown in Fig. 9B. Something has caused the laser signal to drop to zero. Thus, withdraw from the sample, close the program and re-boot the computer.

10- *The system is currently in feedback. If you proceed then the system will be taken out of feedback. Do you wish to continue?* This error message is shown in Fig. 10B. This message occurs if you try to open the “Beam Align” window with the Align Laser command when the system has already been brought into feedback. You must click the Withdraw command, which will bring the system out of feedback, if you want to access the “Beam Align” window.

11- *Unable to communicate with the DSP card* or the error messages shown in Figs. 11B and 12B. This is either a firmware or hardware problem with the DSP board. Try restarting the program or re-booting the workstation.

13- *Problem trying to start the scan DSP command timed out* as shown in Fig. 13B. This may have resulted from unstable feedback setting. Try to get a stable feedback then optimize it.

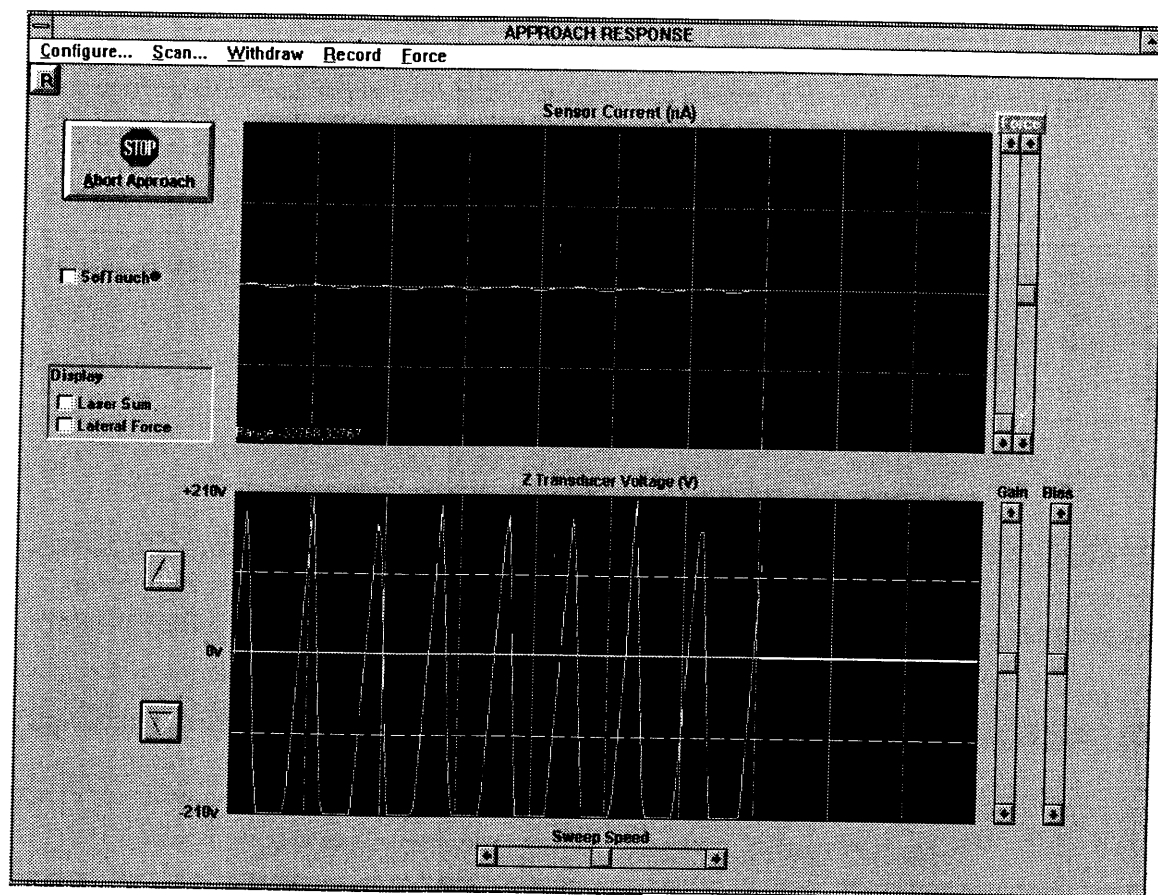


Fig. 1B: The system is not going into feedback state.

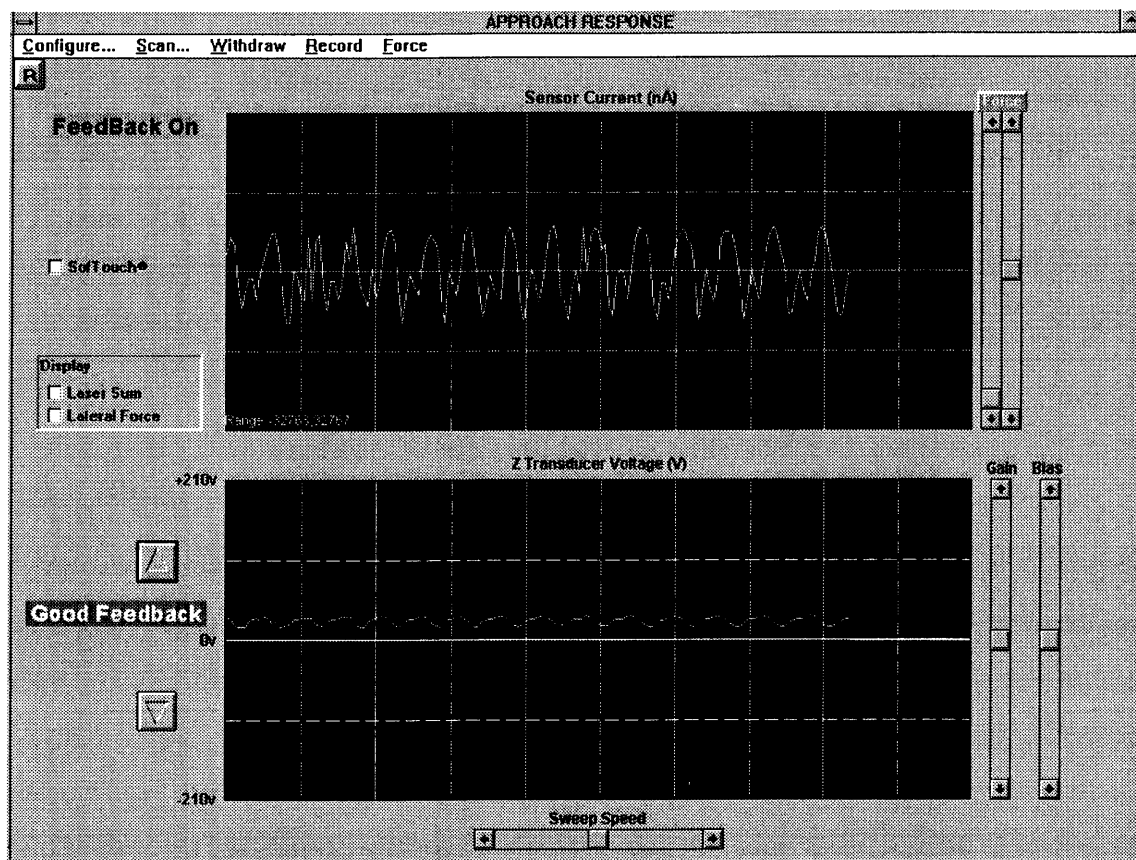


Fig. 2B: The system is going into extremely unstable feedback state.

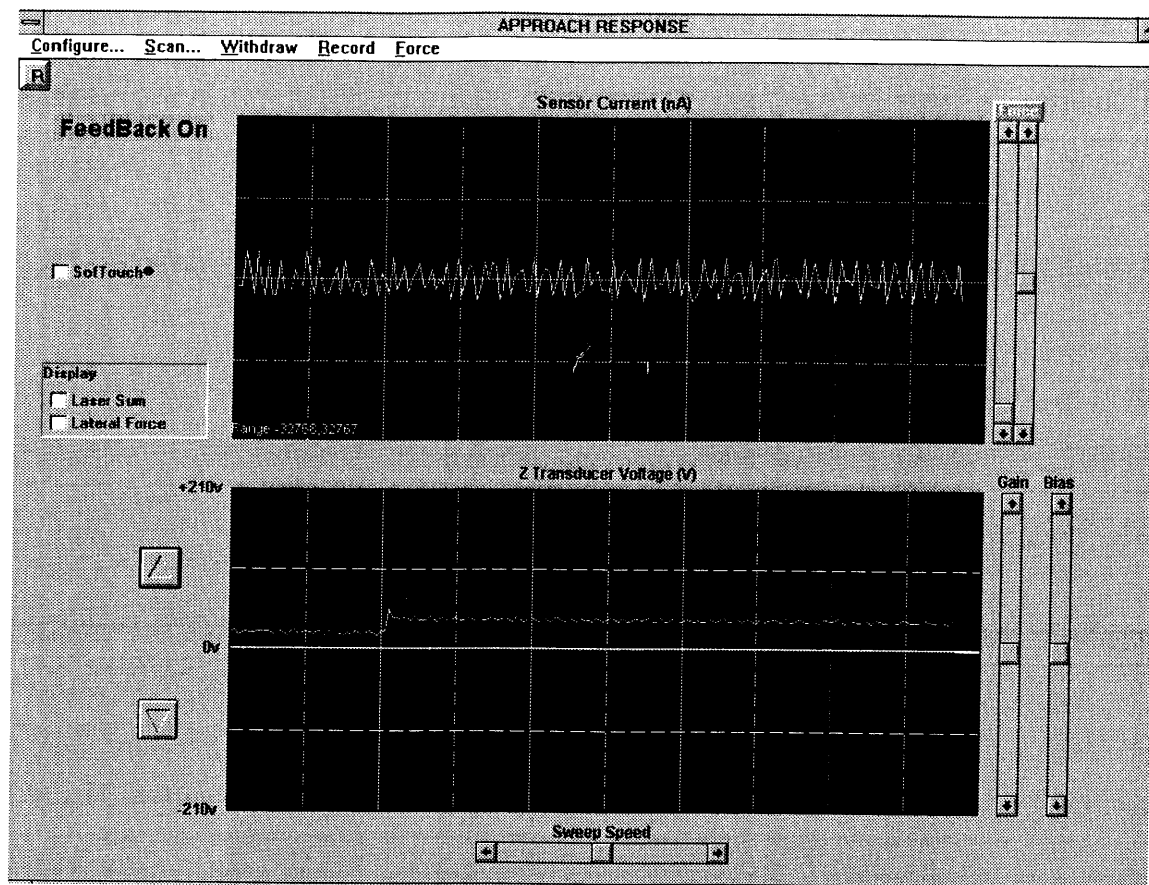


Fig. 3B: The system is going into extremely unstable feedback state.

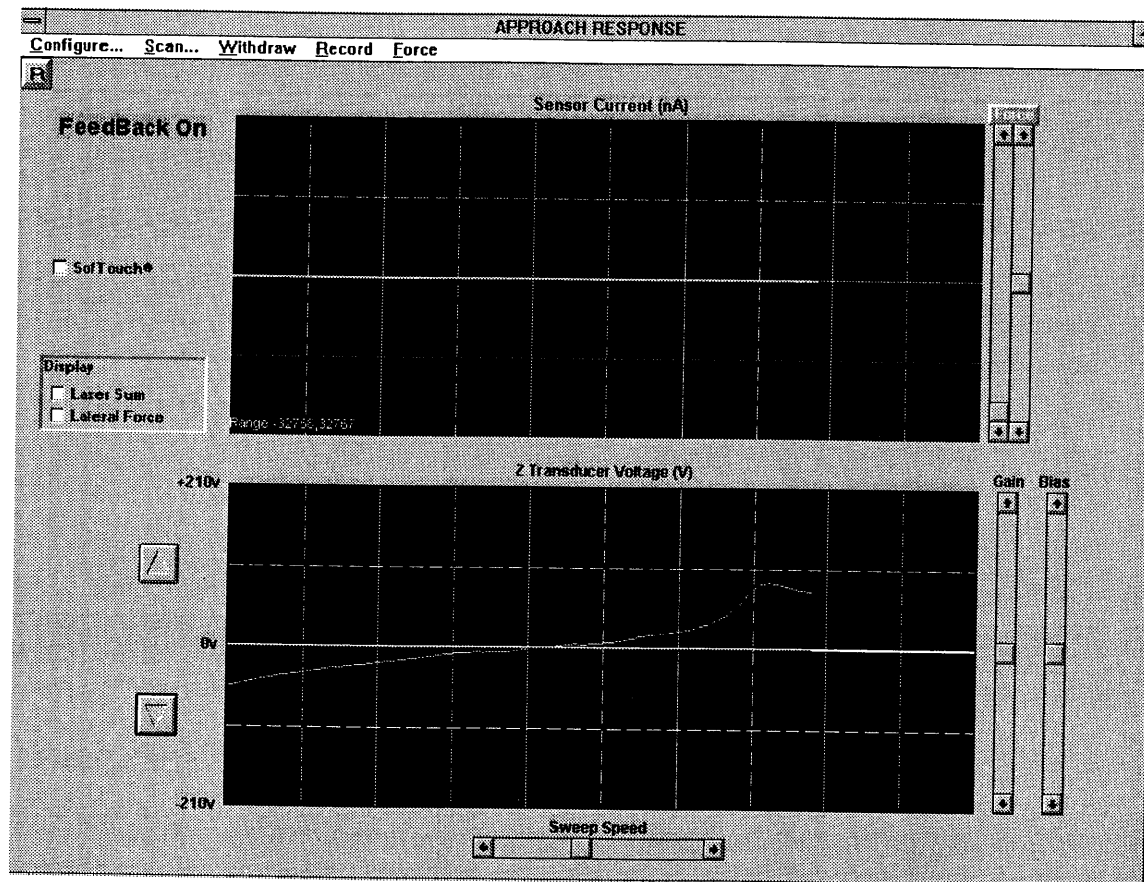


Fig. 4B: Feedback cannot be maintained after the probe and sample are engaged.

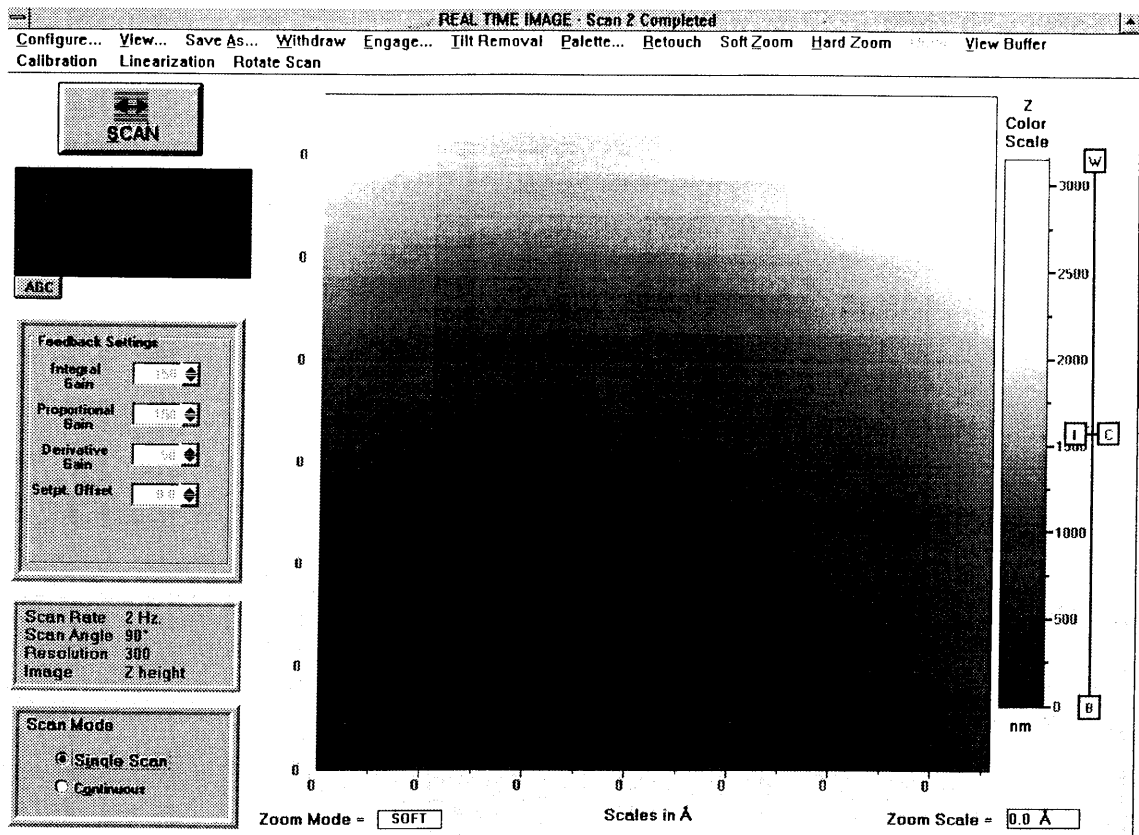


Fig. 5B: The resulted image when the feedback cannot be maintained after the probe and sample are

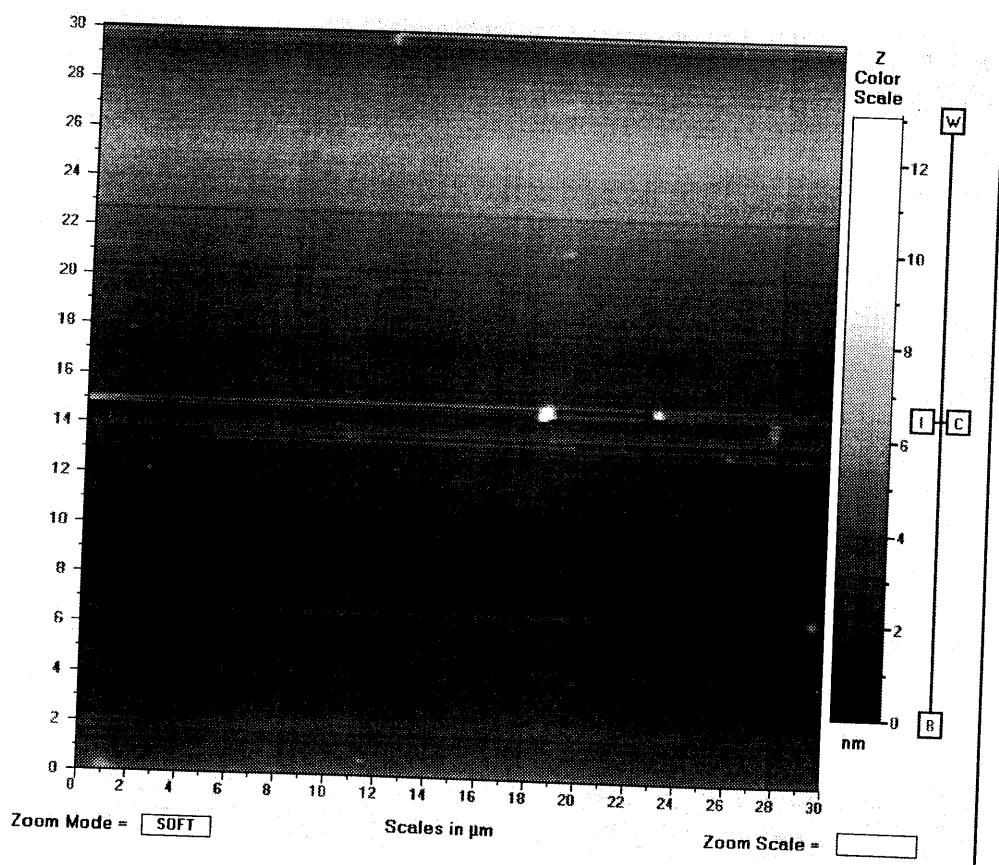


Fig. 6B: Streaks appear in the image because a dirt particle has been picked up by the tip.

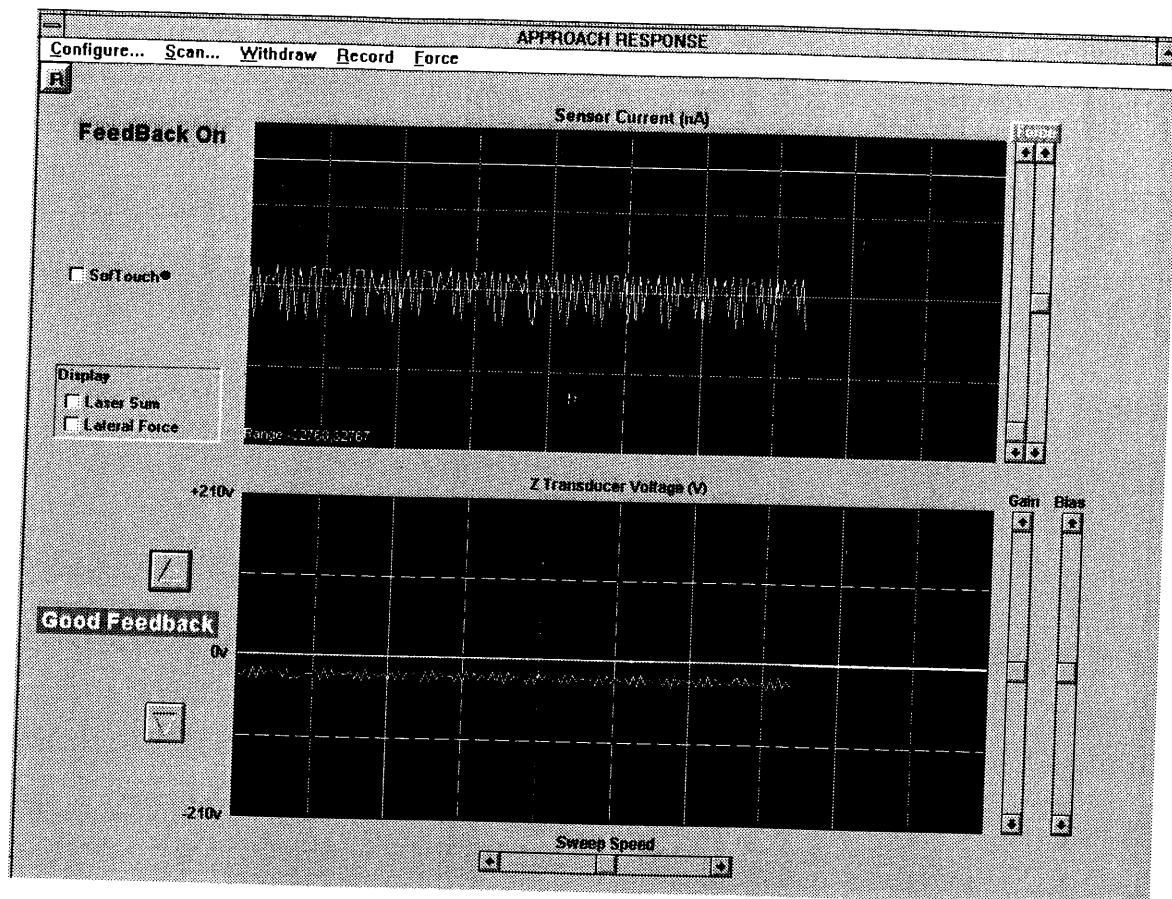


Fig. 7B: Dramatic oscillation of the two traces due to engagement of the tip on a steep or irregular feature of the sample.

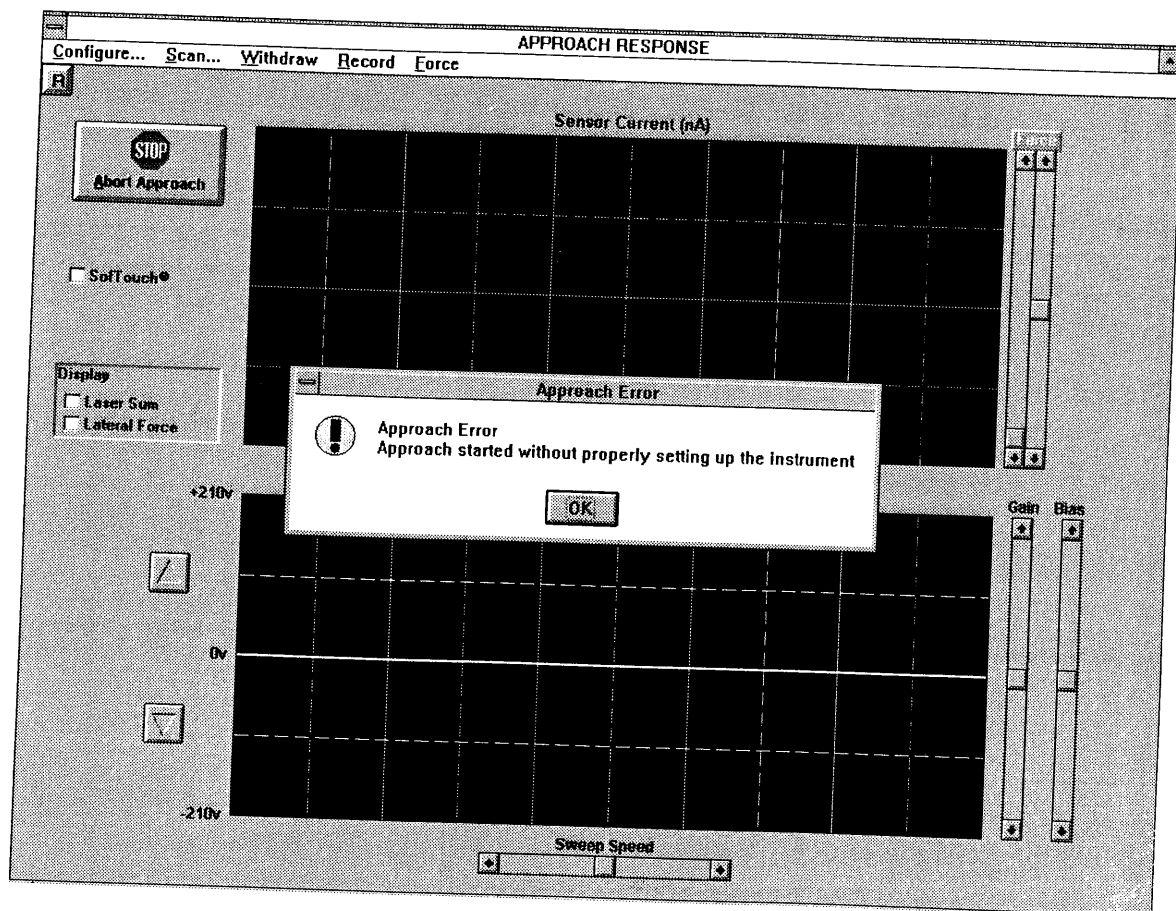


Fig. 8B: Download on the "AFM Configuration menu" has not been clicked before starting to approach the sample.

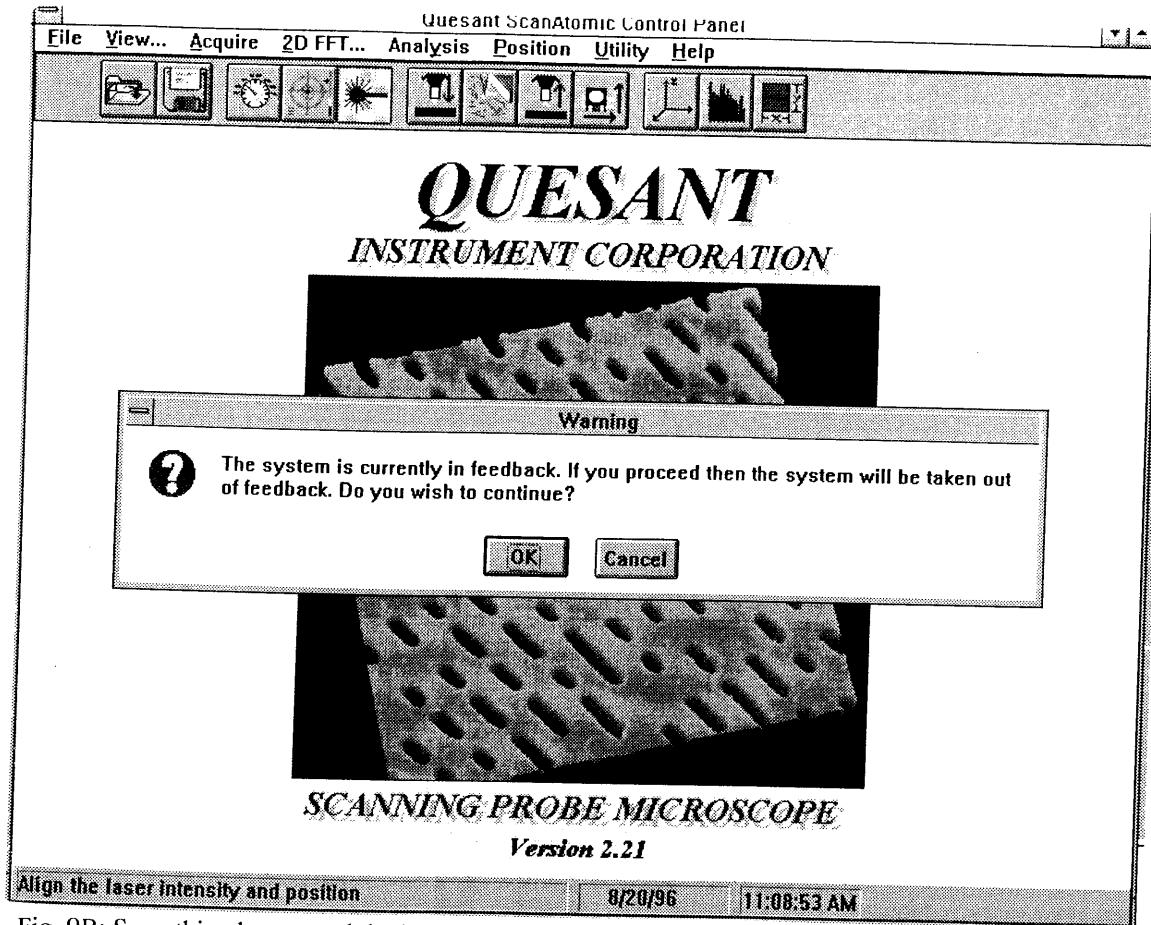


Fig. 9B: Something has caused the laser signal to drop to zero.

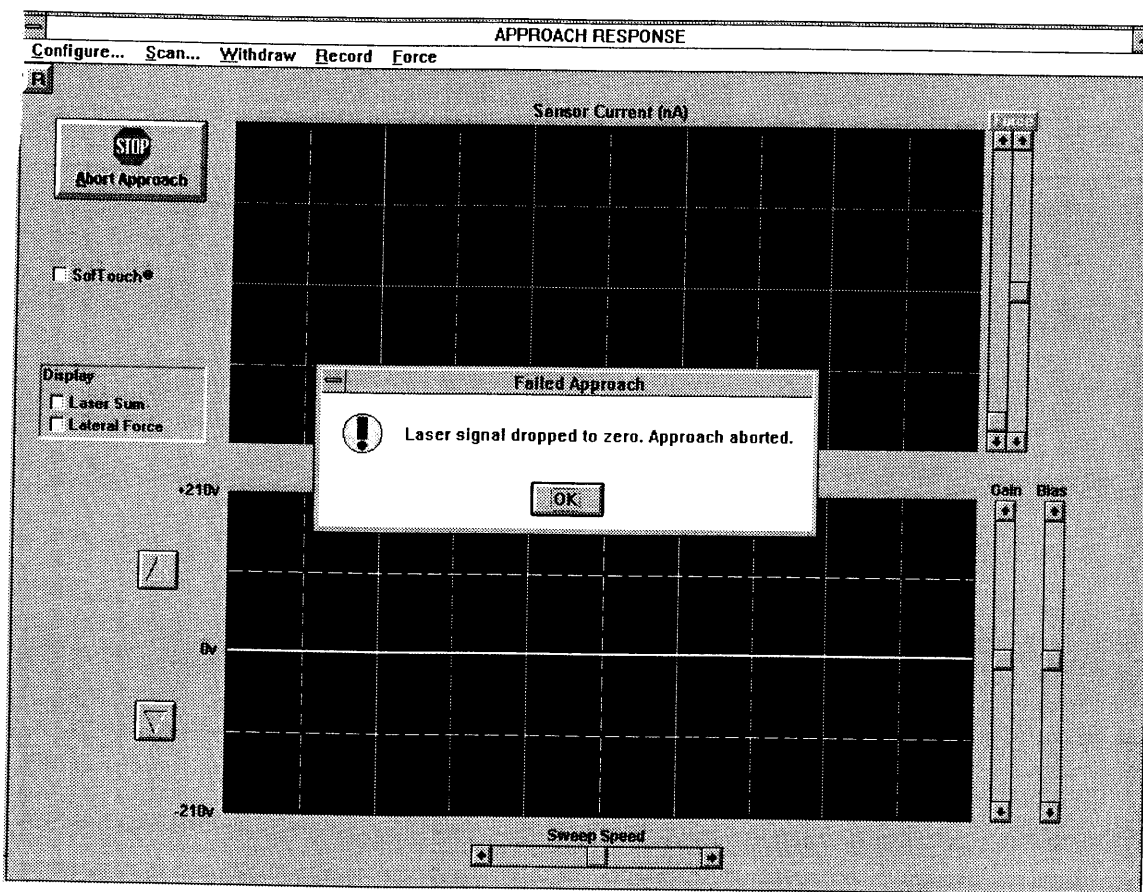


Fig. 10B: Withdraw command should be clicked before trying to open the "Beam Align" window.

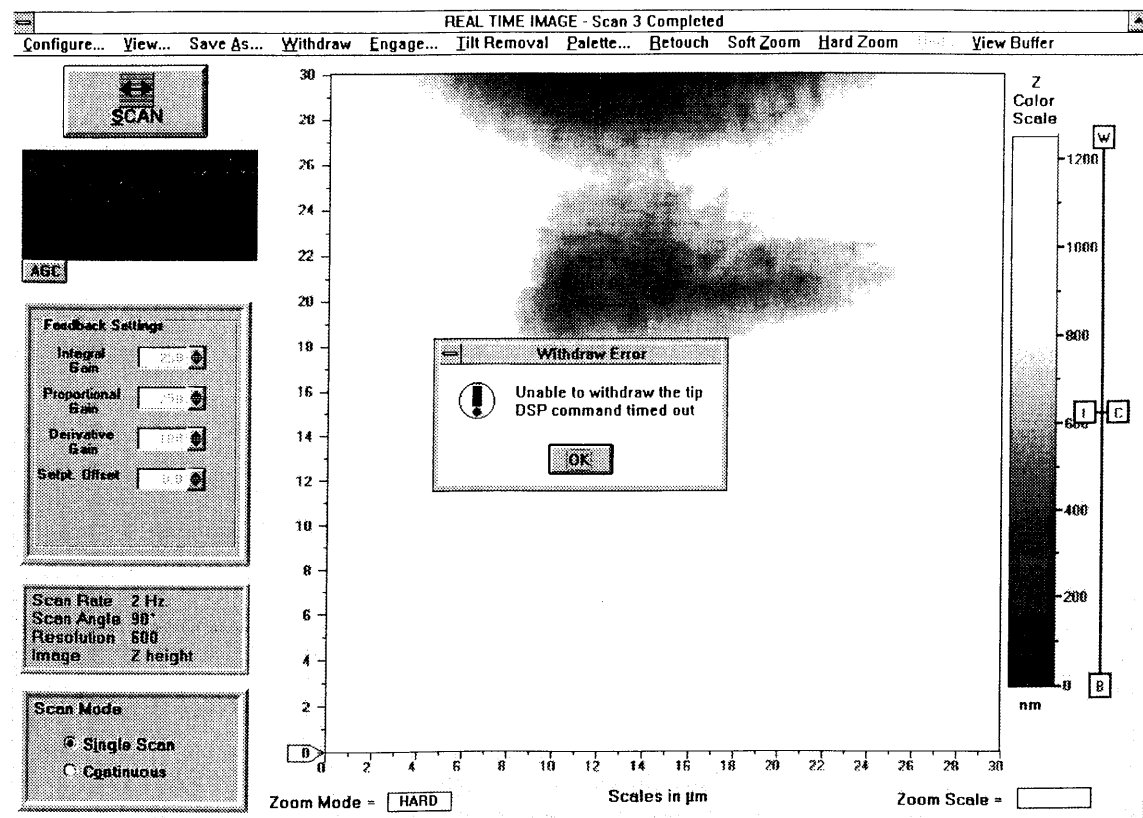


Fig. 11B: The program should be restarted.

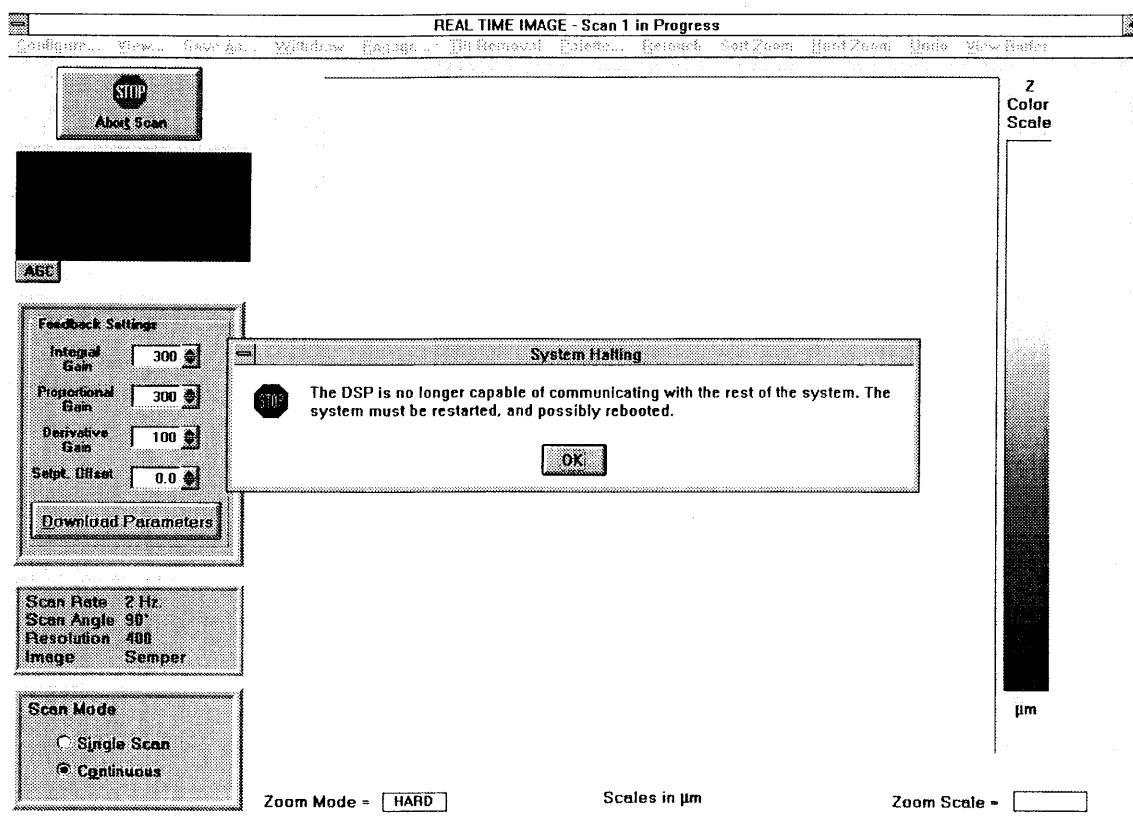


Fig. 12B: The program should be restarted.

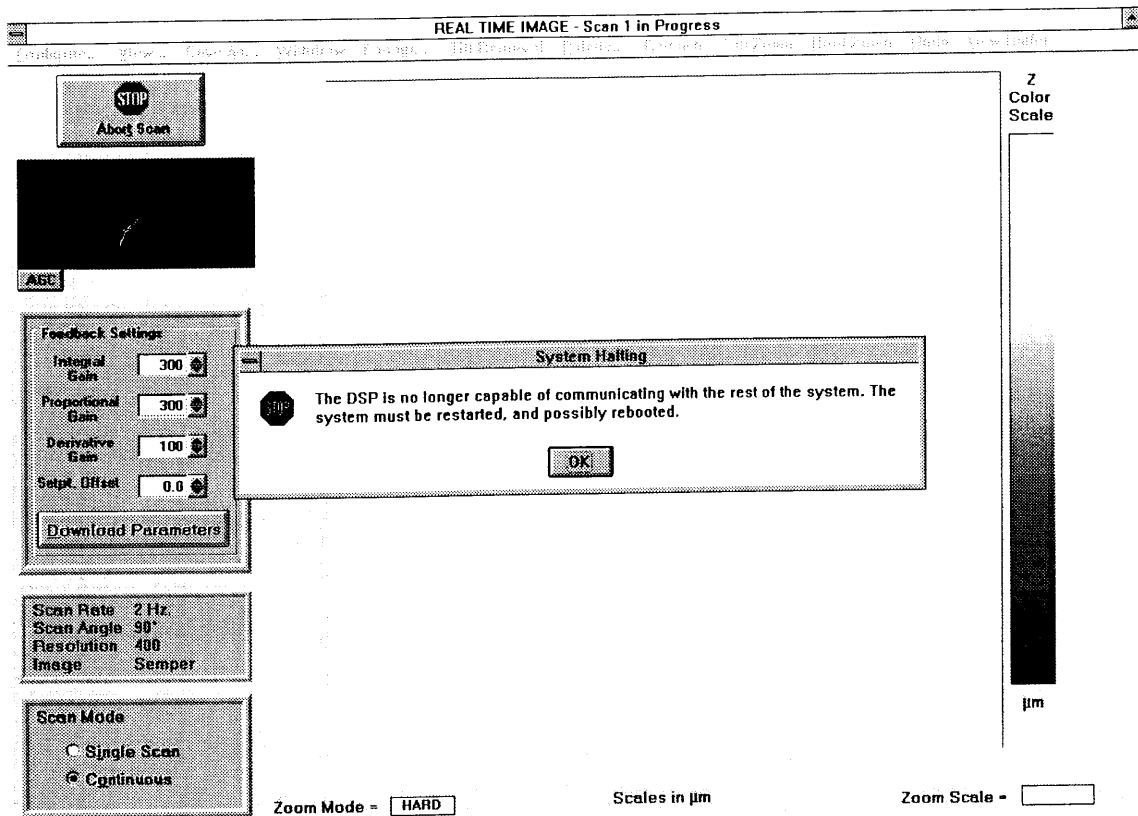


Fig. 13B: The feedback settings are not stable.