An Introduction to the
Atomic Force Microscope and its Derivatives

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Introduction

Since its invention in 1986 by G. Binnig and C.F. Quate (Binnig), the atomic force microscope (or AFM) has become an essential tool for the characterization of materials, especially in the field of nanotechnology. This is due to the technique's resolution, surface sensitivity, versatility with respect to sample characteristics, its ability to be used in multiple working environments as well as its low cost. With its lateral resolution of 3 nm and its vertical resolution of less than .1 nm (Binnig) the AFM can image nanostructures previously only able to be imaged with Scanning Tunneling Microscopy (STM) and high resolution Transmission Electron Microscopy (TEM). The surface sensitivity of the technique can both help and hurt depending on the application. This is because, as opposed to scanning electron microscopy (SEM) or TEM which have a finite interaction volume, the AFM is a true surface characterization technique. The AFM is also unique in that it does not require a conductive sample or coating. This is especially relevant to the electronics research community because it allows insulating thin films to be imaged accurately without the charging effects seen in SEM. The AFM is also not hindered by the rigid sample preparation requirements needed for TEM which makes it ideal for nondestructive evaluation. But what makes the AFM truly unique compared to these other techniques is its flexible working environments. Being able to make measurements in atmosphere, high vacuum, and even liquid solution opens whole new areas of study in the field of biology and medicine.

Topographical Data Acquisition

Topographical information has a nearly ubiquitous role in almost all fields of study. As higher resolutions became deisrable new techniques have been developed to accommodate this need. The AFM was first applied in this area. It is, in its essence, a two-dimensional scanning profilometer with incredibly high resolution. The resolution has increased to the point that even atomic resolution is possible as seen in Figure 1 (Morita).

Figure 1: atomic resolution AFM

The AFM, like all scanning probe microscopes, must have a tip which interacts with a sample, a means of measuring that interaction, and a mechanism which allows for motion of the tip in the x and y dimensions in order to create an image. The lateral motion of the tip as well as the means of measuring the tip interaction is generally not dependent on the specific technique being used.

The lateral motion of the tip is generally carried out using piezoelectric crystals which convert potential differences to strains. Very precise voltages can be applied to these crystals to give sub Angstrom displacements. The measurement of the z position of the tip is now done using a laser reflected off of the top surface of the cantilever. In the initial incarnation of the AFM the displacement of the cantilever was measured using an STM on the cantilever (Binnig). This was replaced soon after by the laser method because of the added complexity of the STM. The laser is reflected off the
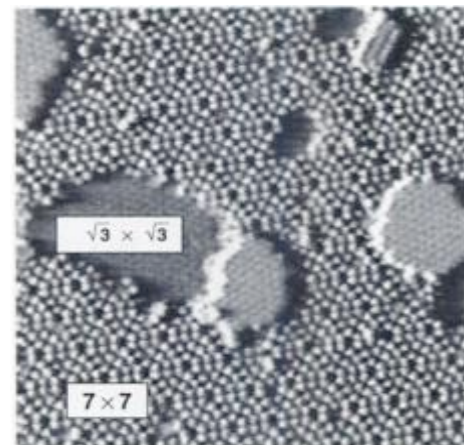
cantilever and onto a photosensitive array. The displacement of the cantilever can be inferred from the displacement of the reflected laser from the center of this array. This allows the force applied by the tip to also be calculated using a Hook's Law relationship assuming the cantilever has an known elastic response. (Bean)

The size and shape of the tip in this application is critical. The tip radius is the limiting factor in the lateral resolution of the microscope. This is because the tip will bridge the gaps between features smaller than the tip. The different shapes of the tips contribute their own problems. The most common pyramidal shaped tip is fabricated using an etching solution to dissolve along crystallographic planes underneath a photoresistive layer (Krause). This can achieve very small tip radius and has become relatively inexpensive, however such tips can generate imaging artifacts. These artifacts are due to the angled edges of the probe because, during any imaging scan, if the topography becomes steeper than the tip sides, then the sides of the tip will make contact with the top part of the feature before the point of the tip will contact with the bottom of the feature. Steep topography then appears as if it has the slant angle of the tip. There are other tips designed for specific imaging applications. One designed to alleviate the above mentioned problem is a carbon nanotube tip. This tip is designed to probe the surface with a single carbon nanotube which gives a very small tip radius and effectively no imaging artifacts due to tip shape (Nguyen). These have their own draw-backs as well, such as the fragility of the slender tip, the high reactivity of the tip, and the high cost of manufacture. As with any other operating parameter, the tip type needs to be tailored to give the correct balance of accuracy and robustness for the experiment being run.

Contact Mode:

The original and most intuitive method that was employed to measure the topography is called contact mode. This set up uses a feedback loop to maintain a constant force (and thus bending) on the cantilever as the tip scans across the sample surface. However, this type of data acquisition has numerous problems. The most restrictive of these being sample damage and tip damage. In contact mode, when the tip encounters an obstacle, it applies a shear stress to the tip. When the angle of the tip is not steep enough to convert the lateral component of the force to a vertical component, thereby allowing the tip to slide over the obstacle, one of two things can occur. First if the force applied is greater than the strength of the tip then the tip will break off, making subsequent imaging either inaccurate or incomprehensible. On the other hand if the material causing the obstruction is weaker than the tip then the tip can damage the sample. This is fairly common in thin film materials where the tip can peel up the applied film from the substrate when trying to image. In addition, the operation of contact mode will always be associated with some level of surface damage. This is because the force applied to the tip is always more than the loading limit of a single atom which ranges from .1-1 nN (Morita).

Non-Contact Mode:

The development of non-contact AFM allowed for a vast improvement over the traditional contact AFM. One of these improvements included an increase in resolution which allowed for true atomic scale imaging. With this development the AFM became the only instrument which could image insulating material to that level of resolution. (Morita)

The reason that this improvement stems from the details of inter-atomic forces. The inter atomic potential between any two atoms can be described with a repulsive term, which is due to the overlap of electron orbitals, and an attractive term, which is due to the van der Waals interaction between the atoms. These two terms give the well known interatomic potential well diagram. In contact mode the force that is acting on the tip and bending the cantilever is caused by this repulsion

term. However in non-contact mode the tip is brought in just close enough for the attractive force to act on it which bends the cantilever down. Then, using a feedback loop, the cantilever is kept at a constant negative force by measuring the deflection and is scanned across a surface in the same fashion as in the contact mode. (Morita)

Dynamic AFM:

The most versatile and commonly used AFM operational mode is called dynamic or tapping mode AFM. This differs from the previously examined acquisition modes in that the cantilever is not held at a fixed point. Instead the cantilever is oscillated at its resonant frequency, which is dictated by its mass and stiffness. Changes in this resonant frequency caused by external forces can then be used to determine the topographical displacement.

This method is applied to both the contact and non-contact modes of measurement. In dynamic contact mode, the tip is brought down until the tip just starts to contact the sample at the bottom of the oscillation leading to a higher resonant frequency. This is opposed to dynamic non-contact AFM, where when the oscillating cantilever becomes attracted to the sample the resonant frequency decreases because both the spring potential energy and the potential energy due to the van der Waals interaction will contribute to the systems internal energy (Morita). Using these differences the feedback system is able to maintain a constant distance away from the sample by maintaining a resonant frequency slightly higher than the isolated cantilever frequency for dynamic contact mode or a slightly lower resonant frequency for dynamic non-contact mode.

Each of these techniques have their own limitations and advantages over their static counterparts. The major advantage of both of these techniques is that they have no lateral force acting on the cantilever. This greatly increases the robustness of the system. Even though there may be some tip and sample damage from impact with the dynamic contact mode, it is generally the lateral force which causes failure of the system. This lack of lateral force also has the distinct advantage of not moving things on the surface around the sample. One major disadvantage of the technique is that the scan rate must be slower. This is because every oscillation gives one data point, therefore the sampling rate is determined by the frequency. With all things considered, dynamic topographical measurements have shown themselves to be the most widely applicable to research.

Sample Tip Interaction Measurements

The next branch of measurements that can be made with the AFM are those dealing with sample tip interactions that do not have to do with topography. Of course the effects of these can be, and often are, superimposed with topographical data. This can make interpretation of the data difficult. Many of these techniques require specialized tips which restricts application to specific fields of research.   These techniques generally work on the same principle as the topographical types of characterization however the attractive or repulsive forces are dictated by some other longer range force.

One of the problems with these types of probes is that they usually require cannot achieve the same resolution capabilities as the standard topographical AFM. This arises from two main causes. The first of these is that the tips are usually made with another material or geometry which limits the tip's ultimate radius. The second limiting factor of these techniques is that they do not have to be true surface techniques. This is because for many of the probes measure long range effects such as magnetism or capacitance. These can affect the probe from distances further than one atomic spacing, so in effect the probed sample has a finite interaction volume.

Magnetic Force Microscopy(MFM):

Magnetic Force Microscopy is a technique which measures the strength of the magnetic field on a surface. It does this in the same manner as the topographical set up with the exception that instead of using a plain silicon tip, the tip is either made of, or coated with, a magnetic material so that it can interact. However, this coating or material has yet to be fabricated with a tip radius as precise as topographical AFM tips.

The largest application for these types of instruments are in the research of data storage. Data is generally stored using alternating domain fields. These can be imaged using MFM but are not detectable using topographical scanning (Nanosensors). Digital data storage is becoming more and more dense due to advances in material as well as read and write mechanisms. With its spatial resolution better than 25 nm, the MFM is left as one of the only characterization techniques available to test the effectiveness of these data storage mechanisms.

This type of characterization could have other applications as well. The decomposition of superconductivity in materials could also be characterized. MFM could determine whether it was a type one or type two semiconductor by scanning the surface and determining if it the superconductivity collapses by the nucleation and growth of vortices or if pairing breaks down all at once. It could also differentiate between hard and soft superconductors by scanning over a surface multiple times to see if the vortices move.
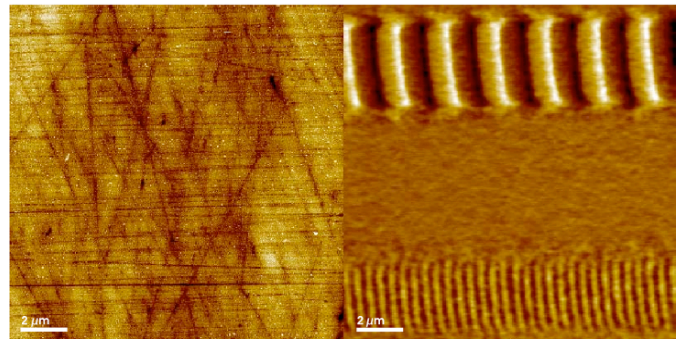
Figure 2: left) AFM, right) MFM

Lateral Force Microscopy (LFM):

The lateral force microscope, or sometimes called the frictional force microscope, is one of the few operational modes which uses the same types of tips used for topography measurements. This is advantageous because the additional information gained does not detract from the topographical information gained. Unfortunately the sensing apparatus must be slightly more complicated. In LFM the probe is run in the same fashion as in
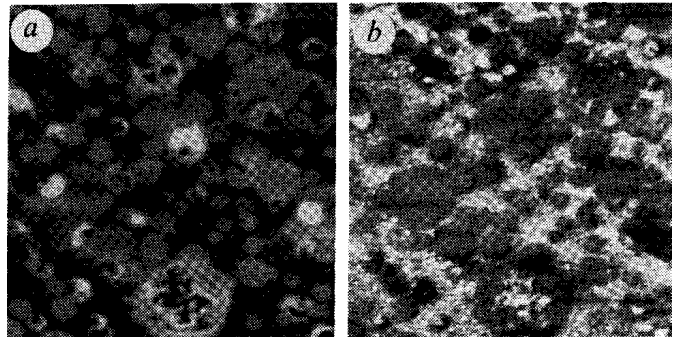
Figure 3: a)AFM, b)LFM

contact mode, which is described above. However, when scanning perpendicular to the cantilever, the friction between the tip and sample cause a torsional strain in the cantilever. This directs the reflected laser to the left or right. Depending on the torsional displacement of the cantilever, contrast is given to the various levels of friction which varies based on material.

This is especially useful for characterizing composite materials such as the polymer thin films on a silicon substrate seen above (Overney). The coefficient of friction in polymeric materials is very sensitive to the end groups that make up the surface of the material due to the bonding of the surface groups to the tip. The advantage of the LFM in this situation is that it can give chemical composition information and topographical information simultaneously. In certain situations this allows for strong contrast between materials. (Overney)

Phase Imaging:

The other technique which can be used without a specialized tip is a method called phase imaging. However the technique is limited to the tapping mode of operation. This is because the cantilever must be oscillating, but the phase differences between materials when operating in non-contact mode are too small to be detected. (Magonov)

This imaging mode measures the phase angle difference between the measured oscillation read by the laser and the signal used to drive the oscillation. (Magonov) This difference can be caused by multiple effects which is both advantageous and detrimental, depending on the application. This is because the multiple effects allow for more types of materials to be distinguished based on the phase contrast, however, it also makes the interpretation of the data more difficult because there are more independent variables.

Two of the most prevalent of these effects are stiffness variations and adhesion variations. A lower stiffness will cause the difference in phase angles to increase while a lower adhesion will cause a decrease in the difference between phase angles. The usefulness of this technique was shown by the comparison of the AFM and phase imaging micrographs of a micro-layered polyethylene sample to the right. (Magonov)
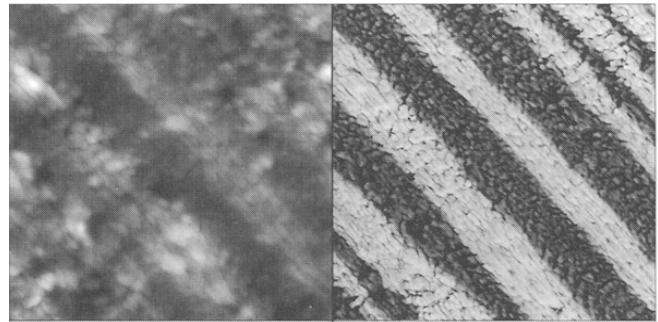
Scanning Kelvin Probe Microscopy (SKPM):

The scanning Kelvin probe mode of measurement uses a conductive probe to measure the work function of a surface as it scans across a surface (i.e. it maps the minimum energy necessary to strip away electrons).

This type of measurement is different than those discussed previously because it uses a completely different method of measuring the effect. Previously all the forces were still measured using the laser reflection method associated with the topographical measurements. In contrast the SKPM measures a capacitance between the sample and the tip to control the feedback loop. It does this by performing a nano scale version of the experiment Lord Kelvin used to measure the work function. The basic idea of this experiment is that when two conductors are coupled electrically their Fermi levels equilibrate. This flow of charge has to be facilitated by the movement of electrons, therefore there will be a positive charge on the material with a higher Fermi level and a negative charge on the material with a lower Fermi level. These two materials are then brought into close proximity of one another and a capacitance between the two is formed. The voltage across the capacitor is then measured to give the contact potential. (Baikie)
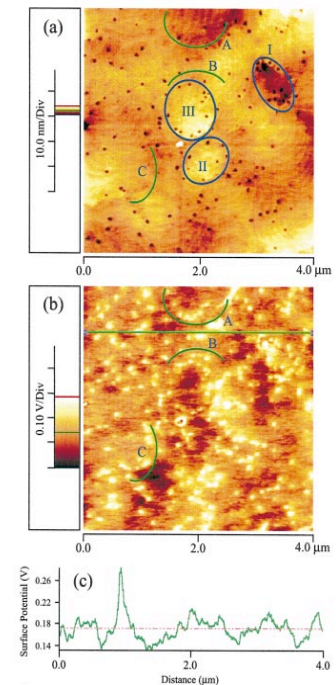

Figure 5: a) AFM b)SKPM

Among other things this technique has shown itself to be useful in characterizing the dislocation structures found on thin films. The ability to do this characterization non-destructively could prove itself invaluable to the semiconductor industry. As devices become smaller these dislocation structures, which were once neglected due to their small effect in bulk materials, become more and more critical to the operation of the device. Some of the dislocations can be seen using purely

Figure 6: left) SThM, right) AFM

topographical AFM however a number of them can are only seen using the SKPM. This is shown in the figure to the right where in the AFM image the dislocations are shown as dark spots while in the SKPM the dislocations are bright spots. (Koley)
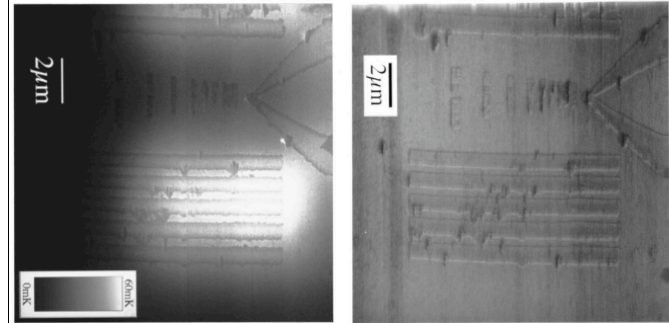
Scanning Thermal Microscopy (SThM):

Scanning Thermal microscopy allows for the relative temperature of a sample to be measured. However this technique is much less precise than the techniques discussed earlier as can be seen in Figure 6 (Mills). This method also needs a specialized tip. This tip consists of a Si_3N_4 cantilever with two dissimilar metals leads, which are applied by lithography, joining at the tip. This creates a thermocoupling effect and the potential between the two which is measured and used as contrast in the image. However this type of tip limits the maximum resolution of the instrument to around 40 nm (Mills). This comes from the fact that it is not able to be made with the radius as small as the traditional AFM tips. This is because components of the thermocouple tip have to be made by lithography, which limits the feature size to larger than the wavelength of the light used. In addition the laser used to heat the sample cannot be focused to that small of a spot size. However there are situations, such as testing running components to see where the power dissipation is occurring, where this last limitation is irrelevant.

Scanning Capacitance Microscopy (SCM):

Scanning capacitance microscopy operates by measuring the capacitance between the sample and the probe tip. It is one of the most relavent AFM operating modes to electrical engineering. This is because it can directly give the two dimensional dopant profiles in semiconductor devices as seen in the figure to the right. (Williams)

The capacitance between the tip and the sample is governed by three parameters. The first of these is the topographic changes because this varies the distance between the tip and the sample (Williams). This distance is usually kept constant using the one of the other AFM operating modes. The next parameter is the dielectric constant of the material between the tip

Figure 7: SCM image of Transistor

and sample (Williams). This is usually constant for a given scan however when comparing tests made in different labs even differences in humidity can affect the dielectric constant. This point is also important to consider when making a quantitative analysis of the dopant profile since these values will
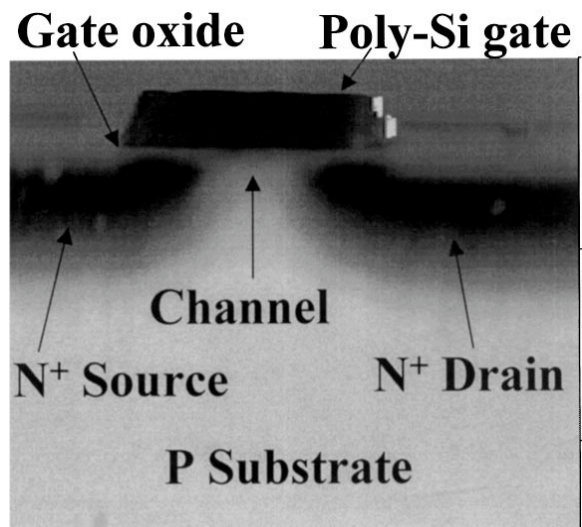
make a contribution in the absolute values of this profile. The final parameter which is relevant to the measured capacitance is the dopant profile. According to the depletion approximation, the measured change in capacitance is proportional to the inverse of the square root of the dopant concentration (Kikuchi). This allows for the different components of the device to be imaged.

Unfortunately this technique cannot be used as a non destructive characterization in this context. The test itself is non destructive however since the SCM only gives a two dimensional profile, and the plane of interest when characterizing transistors is normal to the plane of the silicon wafer the devices are built on. This requires that the sample be cut and polished in order to take measurements of the components of the transistor. (Kikuchi)

Non Imaging Uses

The AFM has also proven to be useful in contexts other than imaging. For these types of experiments there is usually no general protocol. Instead the experiments are usually designed to perform a task analogous to one done on a macro scale but on materials on the nano scale. These have generally fallen into two categories, material testing and material manipulation.

Nano Indentation Hardness Testing:

Indentation hardness testing has become one of the most prevalent characterization techniques for macroscopic materials. The reason the method is so popular is that it requires almost no sample preparation, it is very inexpensive, and hardness values provide a general correlation with material strength. In general hardness testing works by forcing a tip with a known geometry and that is made of a harder material into a sample and measuring the amount the tip penetrates into the sample. This gives an idea of how susceptible the sample is to deformation which is the basis for the strength of a material. Unfortunately, hardness is a qualitative measurement because the hardness is not directly related to the monotonic yield strength. Although models to relate the two have been proposed, the complexity of the loading as the sample is deformed, as well as the hardening of the sample during the deformation, have made accurate models difficult to achieve.

The nano indentation hardness is an extrapolation of these same concepts to the nano scale using an AFM. The AFM tip is forced into a sample with a force determined by the deflection of the cantilever and the depth of penetration is measured after the fact using one of the topographical imaging modes. This is of interest to scientists for many reasons. The mechanical properties of thin film materials are difficult to obtain because these properties usually vary from the bulk material. In addition the macroscopic hardness testing is not a local enough technique to probe only the thin film. These types of problems have lead scientists to use the AFM to test the hardness of materials. Usually when this type of analysis is done, specialized tips made of diamond are used so that the damage to the tip is minimized.

This technique has proven itself useful in characterizing the behavior of nano scale grain size materials as well. In a study by Yang and Vehoff, different micro structural features in nano grained nickel were probed using the technique. They showed that the hardness changed drastically when the grain size became approximately the same size as the indentation made onto the sample. Studies similar to these give insights as to how the deformation mechanisms are affected by microstructural features. (Yang)

Nano Scale Mechanical Behavior:

Other tests have been developed in order to test the mechanical properties of nano scale

materials. These tests are used for both practical applications as well as to back up theoretical models of bonding strength and behaviors.

Molecular biology has made great use of the AFM in this type of application. The most common way in which they use this is in a method called single-molecule pulling. This is basically creates a stress strain curve for a single molecule. The way the AFM is used in this is simple. The molecule to be examined is bonded to a substrate at one end and the other end is bonded to the AFM tip(Franco). The tip is then lifted while the vertical displacement and bending of the cantilever is measured. From this data the force applied to the molecule can be calculated using the spring constant of the cantilever and is plotted against the displacement of the cantilever. This information can give researchers a better understanding of the function of certain biologically relevant molecules such as the proteins which control muscle elasticity like titin (Hansma).

However, it is not only biological macro molecules which are of interest to researchers. Studies of the mechanical properties of single layers of graphene have been made (Lee). This study has both practical and theoretical applications. Adjustment of the lattice strain can be used to tune the band gap of a semiconductor. One of the parameters which restricts the use of graphene as a viable device material is its small band gap. Therefore a precise knowledge of how it will react to strains imposed on the lattice could be important to the important to achieving functional graphene devices. The more theoretical application is the test of computer-modeled strength of the material which have been made based off of first principles.

Machining:

One of the more unique applications of the AFM is its use as a machining tool. In their journal article Fang and Chang showed the viability of using an AFM for extreme high precision cutting tool (Fang). For their experimental set up they used a diamond tipped probe to ensure wear would be kept to a minimum. In addition they designed a much stiffer cantilever to allow for the higher loads required to cut the surface. This type of machining could have an extremely wide applicability to many different fields of engineering. (Fang)

This method seems as though it would be very applicable to the development of catalysts. Structural and spacial characteristics play a huge role in the effectiveness on catalysts. Although the actual manufacture of nano scale features could not likely be able to be achieved by this method, the development of new surface features to optimize catalysis could be made on a small scale. This would allow many different designs to be tested. Then the bulk methods could be designed with a goal in mind.

Another possible application of this would be deposited thin films. Many types of films applied to a surface are not topographically smooth so the thickness and therefore the properties of film may vary across the dimensions of a sample. This type of machining may become necessary as the tolerances on these devices become more stringent and surface preparation becomes necessary.

Although these techniques may never make a full scale production process, they may have applicability to creating a nano lithography technique using x rays. The short wavelength of x rays allows them to create nano scale features using traditional photo resistive methodology, unfortunately they have their own practical problems. One of these is that lenses can not be used to focus x rays, only mirrors can be used. The short wavelength of the light dictates that these mirrors be exceptionally smooth with precise curvatures. This type of machining method could be used for creating the optics which are necessary for x ray lithography to be a reasonable technique.

Lithography:

As the features of devices become smaller than the wavelength of light, new methods will have

to be devised to build these structures. One possible avenue to achieve these structures is the use of AFM. There are three main methods which can be used to do this. These include the direct manipulation of atoms, mechanical cutting, and dip pen lithography.

The first of these, direct manipulation, is performed by physically pushing individual pieces into contact with one another. This could allow components to be made which are as small as physically possible, however this technique would never be practical in a manufacturing sense. The materials would have to be placed on the sample in a nearly random distribution, and then the pieces needed would have to be located and then pushed around one at a time until the necessary was formed. This would most likely have to be done by hand. The type of image processing and automation required to perform this task would be almost inconceivable. These would only really be useful in the context of proof of theory or a promotional kind of experiment, much like those done by IBM with the STM when it was invented.

Mechanical cutting of nano scale features is a much more viable method of obtaining production devices. There are a few ways in which this methodology could be applied. The first of these methods would use many of the same ideas put forward in the nano machining section. The AFM could be used to scratch out features in a structure to get the nano scale features necessary for the device. However there is another approach to this problem using mechanical cutting. This would be to use the same idea of a polymer resist that is used in photolithography. Then this polymer film could be cut away using the AFM tip to make a specific pattern and then etched. One of the main advantages of this technique is that it could be used in conjunction with the traditional photolithography used now. After a mask is applied with the general features needed, the AFM could be brought in to only cut out the features that cannot be done accurately enough. In addition the mechanical wear issue would not be applicable because the polymer would be much softer than the tip. This would be a much more reasonable approach than the manual assembly described above.

Dip pen lithography has a much greater application in the short term than the other two techniques. The process of dip pen lithography simply uses an AFM tip to basically write on the surface of a wafer to create the mask (Bourzac). This technique has its advantages and disadvantages compared with the mechanical cutting method. The main advantage is that polymer AFM tips could be used. These tips are able to be made much cheaper, and they are much more difficult to damage. In addition the mechanical cutting method runs the risk of damaging the substrate when removing the polymer resist. The main disadvantages of the technique is that the tip would have to be reloaded with resist every so often which would add to processing time.   Another factor would be that the precision of the technique is less than the mechanical cutting process because of the nature of the tip. There are two main applications of this technique. The first would be as an interim method of achieving nano structures as the demand rises for the technology. The technique would also be useful for the type of small scale production or prototyping of designs that is often required for military applications or private industry research and development. For any type of consumer full scale production, however, a faster technique such as self assembly or a type of X-ray lithography would still be necessary.

Conclusion

This analysis is by no means a comprehensive one. It is merely an overview of some of the capabilities which the technology can offer. It is meant to discuss a little about techniques available and possibly allow them to be applied to new ideas. The discourse of this field is perpetually growing as new applications and modifications of the technique are added and evolved. The AFM has shown itself to be a versatile and accurate system. Having such few limitations on sample geometry and working environment it has found a place in nearly every field of science and engineering, and with a price which is orders of magnitude less than other measurement systems, it has allowed for countless ideas to be pursued.   The AFM is now and will continue to be one of the most important

characterization techniques available to researchers.

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