NANOMETROLOGY

Nanometrology involves the measurement of geometrical features of size, shape and roughness at the nanoscale. Though not an innate aspect of the specimen under study, these geometrical features are often measured against an arbitrarily fixed co-ordinate system, more so in an engineering application. With advent of new age metrological instruments (e.g. SPM), the parameters of measurement also include a physical quantity (e.g. force). Another important aspect that has to be borne in mind when the scale of objects reduces is the relative importance or relevance of the physical features namely size, shape and roughness. Below are three plots that illustrate the relative importance of the physical features as the scale reduces.





Macro scale measurements are usually encountered in conventional engineering applications; micro scale measurements are encountered in micro mechanics while nanoscale measurements are encountered in quantum engineering. Also, as scale reduces,

additional physical constraints such as tunneling and other such related phenomenon are also to be considered. The above plots, in a crude way, explain how the very definition of roughness changes as we reduce the scale.

AT THE NANOSCALE:

In conventional, macro scale engineering, the size, shape and roughness are purely functions of manufacture process mechanisms like time of machining, depth of cut and the path, which the cutting tool adopts. Loss of co-ordination between any of these parameters leads to large errors. But in nanoscale or at molecular level, the scenario is rather strange. The size and shape are themselves so small that, roughness as a separate physical quantity is not definable.

For the sake of demarcating various engineering materials, the whole spectrum has been split into four levels.

- a) Bulk (µm): This covers all systems right from micro electro mechanical systems (MEMS) to large structures.
- b) Particles (100nm): It includes powders and composites, for example aluminium.
- c) Micro clusters (10nm): It includes alloys, catalysts, nanotubes, and fullerenes.
- d) Molecular scale (1nm): It encompasses thin films such as Langmuir-Blogett and self-assembly proteins.

Geometric features and the scale of size:

The total geometry of an object can be mathematically represented as

$$G = S_i \cap S_{sh} \cap R$$

The sanctity in representing the geometry in such a manner is that it represents the nominal independence of the physical features. For e.g. in conventional engineering, the size (S_i) is much greater than shape (S_{sh}) and roughness (R) and is hence nominally independent of it. In other words, the values of shape and roughness do not alter the size value. In the same way, we can observe that as the size factor decreases there is growing influence of shape and roughness. As the scale reduces, it becomes progressively difficult to describe size, shape and roughness independently. This apparent arises due to difficulty in scaling down roughness.

Reduction in size is pretty simple for there is only a general reduction in all dimensions and there is not a large variation to make the reduction process so tedious. In a similar fashion, the shape is more dependent on, as already mentioned on the path, which the cutting tool takes up and is dependent on parameters that need not be rigorously balanced. To put it more perspicuously, while it does not require much approximation to measure a rectangular block, but it does requires assumption of a mean length, when the block is only a few microns in length and has rather rugged ends.

FORCE BALANCE VARIATION WITH SCALE:

As the scale of size is reduced, there is profound change in the relative importance of the components of the force equations. At the mm level and above, the inertial component dominates. However, as the scale is reduced to the nanometre level, the inertial component falls off faster in magnitude than either the damping or elastic terms. The most important component becomes the elastic force. However, elastic properties are controllable since the material properties are known. The inertial term is negligible leaving the damping term which is areal and is consequently dependent on the surface finish. Adhesion and friction depend a great deal on the relatively uncontrolled surface. The change in the relative importance of the forces is the reason why insects have such different movement capabilities than mammals. Insects can change direction instantly indicating little inertia. They can walk upside down on ceilings indicating that adhesion forces are much greater than force on mass.

DEPENDENCE OF ROUGHNESS ON SCALE:

Roughness at nanoscale can occur in three broad cases,

- 1) On large objects such as mirrors
- 2) On small objects such as in micro dynamics
- 3) At the molecular and atomic scale.

On large objects, roughness has been regarded as irrelevant and the only probable effect it can have is on the reactivity at the surface due to increased surface area. This might probably the only limit that can be set for roughness in macro scale.

At cluster level, the priorities change. The clusters themselves are made up of few hundreds of atoms and more so, the whole functionality of the cluster lies at the surface than at the bulk. Thus what we might call roughness in macro level actually turns out to be shape at cluster level. To resolve this anomaly, the definition of roughness has been changed. While shape is defined as the configuration of molecules (that is automatically generated), the roughness is defined as the defects that arise during shape generation.

Turning our attention to the molecular level, roughness usually means the disorder in the layers of whiskers of carbon nanotubes and fibre, which could easily be avoided by careful processing and preparation techniques. Except in cases where the roughness is present at the substrate interface and the effect it causes is very much similar to what roughness might cause at macro level¹. Another common observation though not strictly classified, as roughness is the forming of oxides, sulphides that depends on the electron structure at the surface and hence comes under the classification of shape rather than roughness². Other such related observations are that of formation of a separate symmetry at surface of semiconductors, which produces a new unit cell at the surface called as

'surface reconstruction' and is a consequence of the structure seeking low potential energy. This surface reconstruction affects deposition of metal on the surface³. Other phenomena include formation of surface colloids⁴, coverage of surface with biological molecules like proteins and enzymes to impart activity⁵ etc. All these interesting applications just go on to show that roughness which is more of than not pejorative at macro scales turns out to be rather useful at nanoscale.

SURFACE AND BULK PROPERTIES IN NANOSCALE:

Roughness in the conventional sense as we know makes little sense at nano levels. More specifically, priority should be given to how well controlled is the production process to attain the desired reactance and stability rather than on attaining smooth polished surface. The surface starts playing so important roles that, cleavage have to be done in vacuum environments to maintain reactance and stability. Exposure to atmosphere can result in adsorption of chemicals onto the surface, diffusion of these chemicals into the bulk, alteration of electric and magnetic properties and so on. This is plainly the reason why roughness was defined as the defects present on the surface.

Defects rather than roughness take over as the important surface characteristics in the nanometer and sub-nanometer domains.

Defects are usually characterized by their physical dimensions like zero-dimensional defects (point defects), one-dimensional defects (dislocations), two-dimensional defects (slip and twin) and three-dimensional defects (voids and cracks). However, the maximum dimension of any defect is purely dependent on that of the parent sample. What we call a 3-D defect at nanoscale becomes a point defect at macro scale and hence the break in definition occurs at the nanoscale.

ENGINEERING SHAPE:

There are a number of shapes in engineering which are especially relevant. Of these, roundness is probably the most important. Practically every dynamic application involves rotation. The measurement and control of roundness is well understood at the macro level. However, as the size reduces it becomes more difficult, at the millimetre level and below, to include MEMS. The measurement is tricky. This is mainly due to centering. In addition, the signal as seen by the instrument is not the same as that obtained at the macro level.

For machined objects, shape is usually determined by the movement of the tool. This involves movement in two or three dimensions. The curve produced in space is usually simple, conic forms such as parabolas and spheres are often required.

MOLECULAR SHAPE AND SCALE OF SIZE:

The aspect of shape at the nanoscale is that it can have a meaning i.e. it is predetermined by energy or other considerations. Whereas in engineering the shape usually determines whether the work-piece can move (i.e. roundness can determine how well a shaft revolves in a bearing). At the nanoscale, the shape of an object can be a result of the internal energy balance between, say, chemical and elastic energies. In clusters or even liquid droplets, the energy balance is between Coulomb attraction and surface energy. From these considerations, it is clear that in nanotechnology shape is often an output from energy considerations rather than an output to an engineering function. Shape can therefore have completely different implications in macro- as compared with nanoregimes.

In molecular domain, shape is determined by the chemical composition and the structures have a fixed shape depending on the particular molecular configuration. In other words, the shape of a structure is preordained. The question is whether it is inhibited by defects or not. Shapes are discrete, not continuous as in macro engineering. The measurement of shape therefore is usually not the issue: failure to achieve the expected shape is.

In quality control terms in the nano- and molecular regimes shape is often an attribute and not a variable, e.g. is it the correct shape rather than the 'value' of the shape. The shape of interfaces can also influence the quantum behaviour of materials. The solution of Schrodinger's electron wave equation can be influenced by the boundary conditions; an ideal parabolic shape for the potential barrier in a quantum well depends on the shape of the boundary, which could be an interface or a surface.

CARBON MOLECULAR SHAPES:

Due to their extremely precise shapes and sizes, carbon nanotubes and similar structures such as fullerenes have such remarkable electronic and mechanical properties. In principle, they can be used in gears, rack and pinion. These structures relate to the parent planar graphite.

Some enhanced mechanical properties include better stability, strength, stiffness and elastic modulus. The helicity, which is the arrangement of the carbon hexagons on their surface layer honeycomb lattice, is determined by the symmetry of the structure, the helix angle and the diameter of the tube (i.e. the shape and size).

It should be noted that it is not only carbon which has the potential for forming tubes, but also Molybdenum disulphide (MoS_2) the well known lubricant also has a similar structure.

Thus to sum it up - metrological factors change in some way or the other as the size reduces below the nanometre level. The balance of forces also changes with size, with each component changing disproportionately. However, the metrological components change form rather than value. For the purely mechanical applications in microdyanmics, roughness, shape (form) and size become scrambled. Forces are small enough so that we need not consider the sub-surface stress and waviness.

Fig 1. shows the diversity of the geometric relationships. However there is inherent difficulty in establishing a common framework of standards to back the different types of measurements. Also, as the size decreases the nature of the signal changes. What appears as a straightforward deterministic measurement in engineering gets replaced by spatial statistical averaging and, finally, temporal statistics brought about by quantum mechanics.

METROLOGY AT NANOSCALE

Even after monitoring carefully how various geometrical features and force balance vary in their relative meaning significance, we face yet another problem and that arises from the instrument we use⁶.

STABILITY OF SIGNALS:

LENGTH:

The system shown is conventional engineering and is stable to the signal S. Uncertainty is so small that noise is negligible. The signal is acceptable. In (b) the actual signal at any point changes with position so, to get a meaningful signal S, the geometry has to be smoothed by integration giving m_1 and m_2 . The signal is now $S = m_1 - m_2$ where m_1 and m_2 are mean planes. The distance S can be accurate to much less than a nanometre because of the spatial integration.



Fig.2. Signal form in (a) Engineering Metrology (b) Nanometrology

In (c) at molecular and atomic levels the available signals passing between the two points is limited by the distance between points p_1 and p_2 which are molecular or atomic un size. In this regime tunneling across the gap or through a barrier is subject to quantum laws. The only way to increase the probability of an electron or photon moving between p_1 and p_2 and so closing the measurement loop is to increase the observation time (i.e. to integrate temporally). This automatically makes the instrument sensitive to the environment. This temporal problem is not the same as the bandwidth of electrical noise.



Fig. 2(c) Quantum Mechanics Metrology

The increase in resolution and accuracy of modern instruments forced by application pressure has brought their specification into the nano regime-in effect almost the same scale of size spatially as that of the surface roughness. This is why the subjects of surface metrology and nanometrology are blending and why position and spacing are being lumped into the new discipline of surface nanometrology.

NANO POSITION SENSING:

This is defined as the technology of moving and measuring with sub-nanometre precision and has values, which are comparable to very fine surface finish⁷. It is also obviously related to length. Optical methods can be used to measure position using white light fringes. However, a very common method is the use of piezoelectric crystals suitably energised by a known voltage to produce the movement, and some gauge, usually capacitative to control the movement. The control is necessary because, although piezoelectric crystals are very stiff, hey suffer greatly from non-linearity and hysteresis. It needs a very precise and accurate gauge to find out where the end of the crystal is in space.

As there are three components involved in the geometry of a work piece each having its idiosyncrasy it is wise to review the calibration procedure in order to see if three can be brought together in a traceable way at the nanoscale.

CALIBRATION

GENERAL

The real problem lies in the way engineering surface metrology and microscopy have evolved. In many engineering applications such as those which occur in tribology have more emphasis on the height variations than on spacing or lateral dimensions. They are mainly observed for aerial structure and position in the plane of the object. There are three types of length calibration artifacts:

1) LINE SPACING : In this type of calibration the effect of the shape of the probe and its interaction with the surface are small because any aberration introduced due to the interaction is common to both lines if measured from the same direction.

2) LINE WIDTH

3) SIZE OR EXTENSION



Fig.3.Length standards showing the effect of interaction between the probe and the artifact.

PROBE

The probe is the most important component of the new generation scanning microscopes. The process of manufacturing a tip of a particular dimension is difficult. The most common example that can be given in this case is electrochemical etching techniques to fabricate tungsten and platinum/ iridium tips for use in STM^8 . These tips have been made with a very high aspect ratio. The main idea of developing such a tip is to obtain a very small angle at the surface. In addition to this by this process it is also possible to keep the radius of curvature very small. The technique of electrochemical etching to make STM is a natural progression from field ion microscopy. We would prefer to use platinum for tips rather than the common tungsten is due to the fact that platinum, although it is softer material, platinum is inert to oxidation. On the other hand tungsten would easily get oxidized to WO_2 . We would also introduce some amount of iridium into the tip surface as it would tend to make the tip much harder and stiffer so that it can withstand much higher temperatures without undergoing any amount of plastic deformation.

SOME OF THE UNCONVENTIONAL METHODS OF STM TIP PREPARATION

ION SPUTTER THINNING METHOD

The ion sputter thinning method is used to get tips which would offer a better resolution and reliability. These tips are capable of achieving atomic resolution spatially can be fabricated. In this process the given material should be made into the dimensions in the micron level. This starting material can be obtained using the ultra-microtome cutting operation. However we should choose the appropriate material for cutting or shearing of the initial sample. Generally diamond coated tungsten carbide blades are used for this purpose. This cut sample which is obtained is then sputtered using an ion bean of Ar (Ar sputtering process). The main disadvantage of this process is that it is very slow and it would take about 18 hrs.

The novel and simple method is to develop a tip fabricated out of pencil lead by coating tungsten tips with colloidal graphite. There is also another method of tip fabrication of tips which involves the shearing of Tt/ Ir wire. By shearing the wire at a very large angle the wire gets a cut tip which is often good enough to use in measuring roughness in micro-areas.

USE OF CARBON NANOTUBE:

One of the most novel methods is to use carbon nanotubes as a probe for STM. These would be very suitable for nanometrology⁹. This is because the carbon nanotubes have a very small diameter, a large aspect ratio (length/ diameter). In addition to this they have the property of high stiffness to lateral movement. This very important because when we apply very large potential through the tip for rastering patterns then the tip would tend to vibrate in the direction parallel to the surface movement. This would lead to misalignment of the probe. The carbon nanotubes have high tensile strength along the axial direction which would contribute to the stiffness. There is also scope for using this carbon nano tube as a nanopipette also along with STM probe usage. Through the hollow section of the tube it is possible to send few molecules or atoms to the particular region which we have just probed. The main difficulty is that of attaching them to the body of the main pick up part.

There are also optical super tips which can be smaller than 10nm. By the use of these optical super tips is possible that only single molecules that can absorb light energy and transcribe to different optical properties. This type of tip is used in Scanning Near-Field Optical Microscope (SNOM) which is a scanning probe microscope that allows optical imaging with spatial resolution beyond the diffraction limit. A nanoscopic light source, usually a fiber tip with an aperture smaller than 100 nm, is scanned much closed to the sample surface in the region called "optical near-field". Particularly, scanning in liquids becomes easily feasible, which is vital for biological applications where the sample is always in a solution medium. For example, its applications reach from routine control of micro contact-printed samples over the determination of the orientation of proteins on surfaces to studies on single fluorescent molecules. This technique makes the sub-100 nm length scale accessible to optical investigations with all the benefits of different contrast mechanisms such as fluorescence and polarization, including the option of chemical

identification. Additionally it has the benefit of recording the sample topography simultaneously with the optical information alike an atomic force microscope.

Special silicon tips capable of being used in the fabrication of semiconductor devices. For example by atomic force microscopy which involves small size movements of atom by atom, resulting in possible locations of 10^{14} GB/m² on silicon wafers. The silicon tips are generally highly acicular in nature having a tip angle of only about 8-10⁰. They are based on silicon beams and quartz tips, which are completely coated with a thin aluminum film. Despite the fact that no physical opening is created at the tip's apex prior to scanning, the energy throughput is sufficiently high and an optical resolving power of around 32 nm has been shown.

HEIGHT MEASUREMENT CALIBRATION AT THE NANOMETER SCALE OF SIZE

One of the big problems associated with the height calibration at the nanometer level is that there is a tendency to try to relate the calibration procedure devised for contact methods with those of optical methods. In particular the optical methods have been used to measure the artifacts developed for stylus contact methods (note that this attempted comparison has been carried out for roughness standards. If the surface is rough the optical and mechanical probe gives the surface geometry fairly adequately. The difference between optical and mechanical techniques is not usually significant. But if the surface is smooth then even small differences can cause a serious divergence of the two methods.

Although it seems sensible to use optical interferometry to measure position, displacement and step height, typical results with interferometry where d is the measured height would give an uncertainty of measurement of 10^{-4} d nm + 2.5nm where as the stylus method gives 1.5×10^{-3} d nm + 0.7nm. The errors associated with the stylus method or nanostep method can be attributed to small non-linearities ion the transducers, where as the errors in the optical methods are usually attributed to difficulties in fringe position measurement, surface contamination and roughness of the surface of the steps¹⁰.

The true way to compare instruments properly is to measure along exactly the same track by using kinematic methods together with track identification techniques for example the double cross method as shown in the figure.

Using the relocation method there is no ambiguity of trace position. The yaw angle is adjusted until just the two marks appear on the trace. There is sometimes the possibility of occurrence of noise. This occurrence of noise may lead to the burial of the signal because there is a mismatch between what is being measured and the metrology unit. One example of this mismatch is the use of light having nominal wavelength of $0.5\mu m$ to measure nano detail. The mismatch in this case is about 500:1. This gives a very low signal to noise ratio. Also there is a limit to which what can be extracted from the attenuated signal.



Fig.4. Relocation method for comparing different instruments

X-RAY INTERFEROMETER



Fig. 5(a) Interferometer structure (b) Arrangement of silicon blocks inside the setup

In this arrangement the interferometer is a monolithic block of silicon. This is machined to project three blades from one face. The third blade is a part of the ligament hinge. X-rays enter from the left (as shown in the figure) and are focused via the second blade B onto C which is the movable blade. The movement of C varies with the absorption of X-rays by the blade. This is then detected by a scintillation counter.

If movable blade C is moved linearly upward the voltage output varies sinusoidally. This signal is the master which, when related back to the lattice spacing can be used to compare with the test transducer signal whose contact point or focal point is positioned on the top of the moving ligament hinge configuration of the crystal. The actual value of the x-ray wavelength is not critical but the most important factor is the positioning of the parallel faces of the blades¹¹. Interpolation of the sinusoidal signal would give much better sub-nanometer accuracy and resolution.

NOISE

The most common source of noise in many instruments is due to electronic or thermal effects.

ELECTRONIC

This is due to the electronic fluctuations inherent in the input stage of the amplifier or the probe itself. This noise is also commonly referred to as Johnson noise and Shot noise. The equation for RMS voltage for Johnson noise is

$$E (V_j^2) = KT\Delta B R^{-1}$$

Where

 ΔB = bandwidth gap

K= constant which depend upon the dimensions of the tip

Shot noise is the variation in the current due to the random movement of electrons. It has a same bandwidth ΔB as above and give about the same level of equivalent noise as Johnson noise.

THERMAL EFFECTS

The deflection of the end of an AFM cantilever beam may be caused by the Brownian motion. For a simple detection system based on viewing the end of the cantilever, the expected variation in z is given by

$$E (\Delta z^2) = 0.065 \text{ K}^{-0.5}$$

 $K = 0.25 \text{ E wh}^2 \text{ L}^{-3}$

Where h is thickness, w is width and L is the length of the cantilever.

In cases where the deflection is measured by a laser beam reflected from the cantilever end, an optical cantilever, then dz(L)/dx is the operative measurand.

$$Z(L) = 0.667L dz(L)/dx$$

By applying thermal vibration theory to cover the two cases when (a) the one end of the cantilever is free and (b) when it is making contact with the surface it is possible to deduce the different modes of vibration of the beam using standard above given formulae.

When all the vibration modes are taken into account the root mean square deflection $\sqrt{z^2}$ is given that the optical lever is used for measurement

$$\sqrt{(4kt/rK)}$$
 for free end
 $\sqrt{(kt/3rK)}$ for contact end

Where k is boltzman's constant and t is temperature

For a temperature of 22° C and a spring constant such as given by K in Nm the values of the resultant thermal noise are just less than 10^{-10} m. This means that the mechanical noise and the electronic noise are about the same and are uncomfortably close to the desired resolution. For small structures ex small cantilevers for AFM, the internal friction has been broken down into surface effects and bulk effects, both of which dissipate energy independently.

CALIBRATION ARTEFACTS

It is always very difficult to provide specimens with which to calibrate precision instruments. In engineering applications the obvious example of the use of artifacts is the use of gauge blocks, which are used as a practical intermediary between the test piece and length standards whose values can be traced back to the standard meter. However these gauge block concept cannot be extended to ranges of micrometer and nanometer.

One of the problems is the fact that there is a need for an artifact of regular pattern. This type is needed for nano-structure spacing as well as for lateral information in scanning microscopes and for filter characteristics in micro-roughness. The most common technique for measuring the roughness of the artifacts is by diffraction grating. The diamond turning is used to generate sine waves in copper. These are usually chrome plated to give hard-wearing properties¹². However this technique cannot be used as effectively used for stylus or optical methods. This is because the stylus method tends to ignore remnant turning marks in the surface (as shown in fig) and light in optical techniques gets diffracted.

(a)(c) ההההההההההההההההה (b)

Fig. 6.Attempt to calibrate optical instrument with tactile standard

NANOMETER METROLOGY SYSTEMS

SCANNING FORCE MICROSCOPE- NANOMETROLOGY VERSION

The term metrology in this and similar devices refers to the possibility of pinpointing the position of the probe or tool in all three axes, and making this position traceable to the length standard. Many STM and AFM can be used to produce realistic pictures of surface with very fine resolutions. Along with achievement of higher resolution we should also obtain a corresponding higher accuracy. If the quantitative information from all the three axes is possible then the instrument can be accepted as a measuring instrument at the atomic level.

Most of the AFM use piezoelectric actuators (PZT) for both x, y and z profiles. Commonly the translators are in the form of either a tube or tripod for small scans. A major problem with PZT elements is their inherent non-linearity caused by small amounts of creep, tilt etc. The result is that the voltage applied to PZT does not translate into position readily. Sometimes the picture image can be highly distorted, even approaching 50% distortion. So we would have the problem of not linearizing the movement. One way of linearizing is by a compensating non-linear drive voltage, which has to have some knowledge of distortion so that the problem can be matched¹³.

A charge drive can also be used for linearizing. In this method each axes namely, x, y and z directions are separately linearized. For this one of the best way is to use an integrated metrology loop that uses the coordinates of the probe directly. The positions of x, y, z is obtained by using a capacitance gauging. In this technique it involves the use of the kinematic principles and the elastic compliance in various parts of the instrument. The thermal effects also play an important role. Due to the sudden temperature change there would be thermal expansion or contraction of the cantilever probe which would lead to errors in reading. So it is necessary to keep the temperature within the limits of $\pm 0.2^{0}$ C.

COORDINATE MEASURING MACHINE

This is also called the molecular measuring machine or M³ machine. To achieve subnanometer-scale metrology over centimeter scale areas, a specialized instrument design is required. Key design requirements that must be accentuated because of the large rangeover-resolution goal are vibration isolation, machine stiffness and low noise in the control electronics, which together are needed to minimize the positional noise¹⁴. For measurement accuracy, care must also be taken to design for motion accuracy and repeatability, and high-stability temperature control to minimize measurement uncertainty due to thermal expansion effects. In addition, we have to operate in a vacuum environment, both to reduce sample contamination and adsorbed over layers, and to remove measurement errors in the interferometers due to changes in the refractive index of the ambient air.

The specification for this is to obtain a point to point resolution of 0.1nm or the distance between any points within 50mm x 50mm x 100 μ m volume with a net uncertainty of 1nm. This is more of a planar measuring device aimed at surface masks, thin films and wafer characteristics than it is mini CMM¹⁵. The real criteria which have to be met in order to get atomic capability are:

- 1) The ability of the material to be stable in the presence of its thermal conductivity and coefficient of expansion
- 2) The ability of the material to absorb thermal shock. This is determined by diffusivity- the ratio of the thermal conductivity and the density multiplied by specific heat.
- 3) Satisfactory response of the structure to dynamic and static loading. (This effect is minimized by the material having large specific stiffness.)
- 4) Response to transient loading the device has to have controlled damping



Fig.7. Schematic showing various components of a MMM

Long range motion of the probe relative to the specimen is obtained by mounting the probe assembly and the specimen on separate carriages. These carriages would move on crossed linear hinges. These are monolithically part of the core assembly. There can be some additional features such as design to reduce the mechanical and acoustic vibration and thermal effects. The datum for displacement measurement is shown in fig; also shown is the integration with the interferometers for the two major axes. The inside differential interferometer configuration combined with the metrology box would enable the measurement of the probe position relative to the specimen mounted on the box independently of spurious drift or strain. The external differential interferometer design is as shown in the figure..



Fig.8. External design of an interferometer

A scanning probe microscope is used as the sample imaging probe system for M^3 . The probe system is comprised of coarse and fine motion stages, a capacitance gauge position sensor, and the probe itself (figure). It was in-house designed to meet the various constraints, notable among them the very limited available space¹⁶. The coarse motion stage is a cylindrical piezo-ceramic linear stepper motor that operates in the vertical axis with a 3mm range. The grounded or open circuit clamping force is sufficient to keep the motor slug from sliding down when the power is removed. Built into this motor slug is the vertical, 5 μ m range fine motion stage, having flexure-guided motion, and driven by a piezo-ceramic through a decoupling mechanism of a ball between two flats. The elements of the fine motion stage had to be folded back through the coarse motion stage to accommodate the space constraints. There is no horizontal (scan) motion built into the probe system, because any such motion would not be measured by the interferometer system; all horizontal scanning either drives the metrology box containing the sample or drives the entire upper assembly that includes the probe system along with the interferometer optics, as already described. The vertical fine motion is measured relative to the vertical coarse motion stage using a capacitance gauge that is calibrated *ex situ* against an interferometer. For a given measurement, the vertical range is limited to that provided by the guided fine motion stage. The vertical coarse motion is only crudely guided by the cylindrical housing in which the motor slug runs, and is not considered to be sufficiently characterized or repeatable to use during measurements, since it is within the metrology loop.



Fig.9. The gauge system depicting coarse and fine motion mechanisms.

Here in this instrument we would also involve the use of low expansion ceramics and glasses such as Zerodur, which considerably reduce thermal problems. The problem of expansion of mechanical loop due to heat can be solved by the use of ebonite which has very low thermal expansion coefficient. This would make the loop stable but dynamic rather than static.

TRACEABILITY

Since both x and y axes are of linear position on the surface there is no very prominent problem in making the positions along the x and y axes traceable. However it is not straightforward to make the z-axis traceable. If the z-axis is for measuring topography of a surface then traceability is readily possible. The movement of the probe will be measuring height and there should be little difficulty in relating surface height at x, y with the vertical position of the probe at xy. There would be some amount of discrepancy if the probe touches the surface with a force sufficient to cause elastic deformation. In principle it should always be possible to make the vertical position of the probe traceable by using interferometers or other calibrated transducer.

METHODS FOR MEASURING LENGTH AND SURFACES TO NANOSCALE RESULTS WITH INTERFEROMETERS AND OTHER DEVICES

CONVENTIONAL METHOD- MICHELSON INTERFEROMETER

The Michelson interferometer produces interference fringes by splitting a beam of monochromatic light so that one beam strikes a fixed mirror and the other a movable mirror. When the reflected beams are brought back together, an interference pattern is produced.



Fig.10. Simple schematic diagram of a Michelson interferometer.

ELECTRON AND X-RAY DIFFRACTION

X-ray diffraction used as the basis for height or length calibration has been very successful. However the specimen and the diameter of the x-ray beam, are millimeters in size which severely restricts the applications and usefulness of this technique. Using intense synchrotron source brings down the size to about 1 μ m but the diffraction patterns of the object still require long durations, ex- tens of minutes. The only way to investigate small objects is by using electron beams instead of x-rays. Electrons are used in transmission electron microscopes and scanning electron microscopes but they suffer sometimes from strong scatter produced in the bulk. Surface scatter gets lost at normal incidence and the surface resolutions can also be very poor¹⁷.

The major use of the electron diffraction methods as far as the nanosurface metrology is concerned is lateral crystal structure of periodic nature. So to extend the effective range and reducing the penetration into the bulk the reflection mode is used. In this the angle of incidence is less than the Bragg angle. This has to be for dominant lattice spacing on the surface (or suspected to be on the surface). This configuration produces diffraction spots perpendicular to the surface and at right angles to the incident beam. Sometimes as a result of the incoherent scattering we would have the Kikuchi lines formation. To get an image reconstruction from the diffraction pattern requires all the diffraction spots. The method is to form an image by selecting one or more diffraction spots and then retransforming this diffraction spot.

CAPACITATIVE METHODS

The capacitance methods were initially used to measure the roughness. The big advantage over stylus method was the extreme sensitivity of the method coupled with the fact that an area assessment was possible. The method faltered due to practical considerations usually associated with the difference in tilt and shape of the reference electrode relative to the test surface. Capacitance micrometers are essentially made up of two conducting electrodes separated by a millimeter or less. A typical capacitance is 2pF with a 10 mm diameter. Any change in the overlap area or the separation changes the capacitance. The use of three terminal capacitors and shaped electrodes gives improved performance. However the shaped capacitors can be a disadvantage if the capacitative micrometer is intended for general use because the shape may not correspond to the object.

These sensors are most useful for measuring small gaps to high accuracy. This gap can be increased or decreased as the applications warrants. The bandwidth of frequency varies from 50Hz to 5KHz. Linearity for capacitative sensor is 0.01%. However by utilizing a small part of the range of a large gap sensor a linearity of 0.005% is possible.

The most comprehensive treatment of capacitative transducers is due to the Queensgate instruments. The working formula for capacitance between two plates is given by

$$C = \varepsilon_R \varepsilon_0 A/d$$

Where d = separation between the plates

A = area of the electrode

 ε_{R} = relative permittivity of the medium between the plates

 $\varepsilon_{o} = permittivity in vacuum$

By changing the value of A or d would change the value of the capacitance C. However, changing d is more sensitive as compared to changing A.

$$\delta C = \varepsilon_R \varepsilon_0 \, \delta A/d \quad \dots \quad [a]$$
$$\delta C = -\varepsilon_R \varepsilon_0 \, A \delta d/d^2 \, \dots \quad [b]$$

From the above expressions we find that the equation [b] would have greater amount of change in δC value as compared to in equation [a]. So by making use of this principle we would use δd as the mechanism for small range high sensitivity and to use δA for long range low sensitivity. Using the standard value of permittivity we can conclude that the sensitivity of the short range variant is about 100 times that of the long range version, adding the condition that the range is proportionately shorter.

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