Journal of Materials and Electronic Devices 1 (2023) 1-11

JOURNAL OF MATERIALS AND ELECTRONIC DEVICES
FYTRONIX JMED http://dergi-fytronix.com/

Consecutive Imaging and Image Processing Techniques Using Non-Contact Atomic Force Microscopy

Naim ASLAN[1,2], Mümin Mehmet KOÇ[3,4*]

[1]Faculty of Engineering, Department of Metallurgical and Materials Engineering, Munzur University, Tunceli, Turkey
[2] Rare Earth Materials Research Institute, Munzur University, Tunceli, Turkey
[3] Kırklareli University, Faculty of Art and Science, Department of Physics, Kırklareli, Turkey
[4] Kırklareli University, School of Medical Service, Kırklareli, Turkey

Probe Microscopy widely used in the investigation of the surface properties of ultra-thin films, nanostructures, crystal structures etc. Atomic force microscope is an important tool to reveal different properties of such structure since atomic force microscopy is able to provide alternative solutions for desired investigations. Atomic microscopy was mostly used to assess stable and static structures. Especially contact microscopy or tapping microscopy techniques helps researchers to reveal mechanical properties or electrical properties of the thin films and nanostructures since such techniques find opportunity to have intermittent or continuous contact with surface. Such contact obtains valuable data about the mechanical and electrical properties of surface. On contrary, non-contact AFM does not have contact with surface where the forces between AFM tip and surface has important role on surface characterisation. Since AFM tip does not have contact with surface, it was believed that the tip does cause alteration on surface. In this chapter, the consecutive imaging technique used by non-contact AFM will be discussed.

Keywords: *Non-contact AFM; Probe Microscopy; Consecutive Scanning; Image Processing*
Submission Date: 18 December 2022
Acceptance Date: 10 February 2023

**Corresponding author: muminmehmetkoc@klu.edu.tr*

1. Introduction

Atomic force microscopy (AFM) is one of the most popular branch of scanning probe microscope (SPM) family. Conventional microscopes are mostly produce image by light where photons interact with surface and reflected photons were obtained by collective lenses. Quite similar working mechanism was also exist for electron microscopy, in that case electrons interact with investigated structure instead of light. In both light microscopy and electron microscopy, range of microscope and resolution of the microscope was limited with the wavelength of light or electron. On contrary, working mechanism of scanning probe microscopes are quite different than conventional optical or electron microscopes; SPMs are mostly working like a touching hand where the probe either directly touches the investigated media or closely interact with it. Such interaction gives valuable information about the surface. Unusual working mechanism of probe microscopy help us to obtain information in outstanding resolution and accuracy where the atomic structure of the investigated samples or surface properties could be acquired.

Scanning Probe Microscopy was first investigated and used by Binning and Rohrer in early 80s [1]. A short duration after the invention, the achievement was rewarded with Nobel Prize in 1986. The first prototype was then named as scanning tunnelling microscope (STM). SPM family is quite large and different types of tools were developed in years for desired applications where the tools could work in extreme conditions. For examples ultra-high vacuum (UHV) systems were developed to work in ultra-clean environment, cryogenic systems were developed to work in extremely cold conditions to investigate the motions of molecules and agglomeration processes of atomic structures [2] or special tools were developed to work in wet conditions to investigate the properties of living organisms such as cells or tissues. Since various models were developed for specific applications, the popularity of SPM was increased day by day.

Atomic force microscopy has important place in scanning probe microscopy family since AFM is superior than the optic or electron microscopy technique. AFM can provide valuable data about both morphological and structural information about investigated samples. Specialized tools may give information about electronic, magnetic, capacitive properties beside morphology and topography of the surface. In addition, different modes provided by the manufacturers enable researcher to obtain different type of information from investigated sample. For example, Contact mode may give information about tribological properties of the surface while tapping mode can give information about stiffness or elastic modulus; non-contact mode can provide information about topography of the surface without damaging it.

In this chapter brief introduction about the structure and the working mechanism of the AFM will be given. In addition, consecutive imaging method using AFM non-contact mode will be discussed using specific examples.

2. Basics of Probe Microscopy

2.1. What is Probe Microscopy?

Probe microscopy is a special technique which was developed at early 80's. The technique basically uses a probe in nanometre size which closely interact with surface. Unlike the electron or optical microscopy, the image obtained from the investigated sample does not reflect the real image of the sample. The image gives information about the interaction between the surface and the tip; but this does not mean the image is irrelevant with real data. The interaction between the probe and surface was processed with processors of SPM. Regarding the method applied the surface an image was compiled by the computer. Such image can give information about the structure or the properties of the surface. For example, in contact AFM probe directly touches the surface where the topographic image obtained from this measurement may not give the real topography of the investigated surface. In this method tip raster on the surface while having constant contact. This case may result in alteration in the shape of the tip of the probe. Such alteration may alter the image obtained from the surface. However, the tribological information or tribological (friction) mapping obtained from contact measurement may give much better information about the tribological properties of the surface where details cannot be obtained from not contact microscopy, optic microscopy or electron microscopy could be achieved. Regarding the interaction between the probe and the investigated samples different techniques were developed. One of the most common techniques are, Scanning tunnelling microscopy and Atomic force microscopy. Details are described in the following section.

2.2. Probe Microscopy Types

As it was previously described probe microscopy is a wide family and various types of microscopes were developed regarding to the needs of the researchers two common types were known as

2.2.1 Scanning Tunnelling Microscope (STM)

STM measures the electric current between tip and the specimen ; the current was called tunnelling current; therefore, the technique was called as scanning tunnelling microscopy. The probe of the microscope was moved back and forth over the investigated specimen and current was obtained from specific points. The measured data was evaluated by the computer system and the image was obtained from investigated region. STM is one of the simplest techniques but it has some drawbacks. For example, it cannot be used in aqueous media and investigated sample should be conductive since the current between the tip and sample was measured to obtain an image. The tip moves up and down while moving on the surface to keep the current stable where the piezo actuators help them to make such a move. The data obtained from piezo actuators and the currents obtained from the tip was also used to improve the image obtained from the measurement.

2.2.2 Atomic Force Microscope (AFM)

Atomic force microscopy was divided to three major branch which are

- Contact Mode Atomic Force Microscopy
-Non-Contact Mode Atomic Force Microscopy
-Tapping Mode Atomic Force Microscopy
Besides,
-Dynamic Contact Mode Atomic Force Microscopy
-AFM-Infrared Technique was also considered as an AFM branch.

The detailed working mechanism of AFM is discussed in the next chapter.

Other known types of force microscopy techniques derived from these AFM modes are listed as follows:

2.2.2.1 Chemical Force Microscopy (CFM)

Chemical force microscopy often referred as a branch of atomic force microscopy where a special tip was used in microscopy method. A special tip was functionalized with functional groups before the measurement. Therefore, the chemical interactions between the tip and the sample was measured and the behaviour of the sample was characterized. The technique was mostly used to characterize the polymers and organic molecule, especially heterogeneity and surface chemical of such samples were determined [3].

2.2.2.2 Electrostatic Force Microscopy (EFM)

Electrostatic Force Microscopy is a kind of non-contact probe microscopy which uses electric force to map the surface. Dynamic non-contact mode was used in this mode where the tip of the microscope itself both oscillates and does not contact with the surface. Th tip basically scans the surface and maps the electrostatic force occurs between the tip and the investigated surface.

2.2.2.3 Conductive Atomic Force Microscopy (C-AFM)

Conductive Atomic Force Microscopy (C-AFM) is a special mode of probe microscopy where a conductive tip scans the surface of the sample and when voltage ranging from couple of pA to 500nA was applied between surface and sample. The set up both generates topographic image of the sample and maps the current voltage characteristics of the sample. In addition, IV curves were also obtained for specific points in the images. Diamond coated silicon tips were preferred in the measurements since the conductive tips were required to make such measurements.

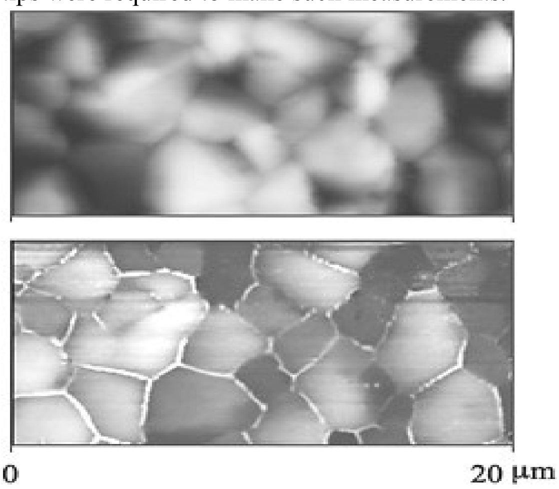

Figure 1: AFM (Top) and Conductive AFM (bottom) images of $CdTe/CdS/SnO_2$/glass composite sample were illustrated in the image. Conductivity map was obtained in the C-AFM investigations and regions with higher conductivity looks brighter in the image which is at the bottom.

2.2.2.4 Kelvin Probe Microscopy (KPM)

Kelvin probe microscopy is a kind of non contact-atomic force microscopy where contact potential difference between the tip of AFM and the sample was measured and mapped in 2D. The measurement was measured in thermal equilibrium and then, work function of the material is able to be calculated. The tip oscillating in resonance frequency first scans the surface and then, atomic force and long range electrostatic force between the sample was measured by applying a dc voltage to the tip.

2.2.2.5 Scanning Capacitance Microscopy

Scanning probe microscopy is a branch of atomic force microscopy method in which contact mode scanning was used in the investigations. Ultra-high frequency resonant capacitances sensors was attached to a grounded tip to make measurement. The system mostly used to analyse dielectric and charge carrier properties of semi conductive materials.

2.2.2.6 Magnetic Force Microscopy (MFM)

Magnetic force microscopy is also atomic force microscopy technique which allows users to measure magnetic force between the tip and sample which is showing magnetic properties. The system work on non-contact mode where a topography image was obtained by processing the alterations in the tip amplitude and frequency (Figure 2a) another magnetic force image was obtained by oscillation from the magnetic force between the surface and magnetic tip. The interactions between the tip and sample was detected and magnetic structure of the sample was reconstructed (Figure 2b).

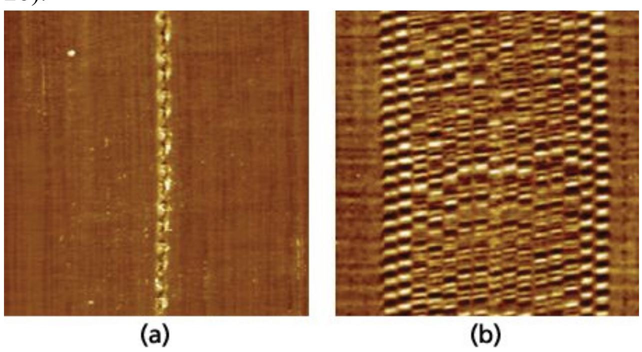

Figure 2: Topographic image (a) and magnetic force image (b) obtained from the MFM measurement was presented. Magnetic domains in the surface could be seen in Figure 2b.

2.2.2.7 Piezoresponse Force Microscopy (PFM)

Piezoelectric force microscopy is a branch of atomic force microscopy which allows imaging and investigating piezoelectric properties of investigated samples. The system simply makes intermittent contact with investigated surface voltage of which was measured. The tip applies constant force to the sample and the measured voltage assess the piezoelectric properties of the investigated sample. The operation repeated for the whole investigated sample and a map showing piezoelectric properties is obtained [4].

2.2.2.8 Scanning SQUID Microscopy (SSM)

Scanning superconducting quantum interference device (SUQID) microscopy system which enables researchers to image surface magnetic field. Main purpose of the method is to be able to image the local magnetic flux occurs on the surface of the sample. Typically, superconducting vortices, ferrimagnetic and ferromagnetic samples were investigated using SSM. In addition, SSM allows researcher to generate the image of the magnetic field originated from current distribution and magnetic contamination occurs on non-magnetic surfaces.

2.2.2.9 Scanning Thermal Microscopy (SThM)

Scanning Thermal Microscopy is advanced technique that developed to obtain thermal properties and topographic image of the nanostructures. Such thermal properties may be thermal conductivity, heat capacity, transition temperature, temperature distribution, heat transport and thermal-resistance. The key part of the technique is specially designed tip. The tip resistance is changed regarding to the temperature of the investigated sample the alteration of the resistance is used in the thermal mapping.

3. Atomic Force Microscopy

3.1. Structure of the AFM

AFM is essentially quite complex system; all the components of the system work precisely. The motion of almost any part of the system was controlled with feedback systems where electronic systems play very important role. Basically, AFM consist of three main components optical components (*i*), tip and cantilever (*ii*), and AFM electronics (*iii*). The position and/or structure of the system may show slight difference device by device. However, each system contains these basic components. In Figure 3, a basic structure of the AFM was illustrated. The AFM system sketched in the figure scans the sample placed at the top of the scanner tip. Such position is not a common a type of AFM; the example shows that the structure may chance but basic components stays.

Each part works coordinal and their coordinal motion effects the quality of the image and measurement. Therefore, the system needs to be calibrated before the measurement. Special calibration tools were used to calibrate the system. Such tools help researcher to make more correct assessment, increase the precision of the tool and provide higher quality results.

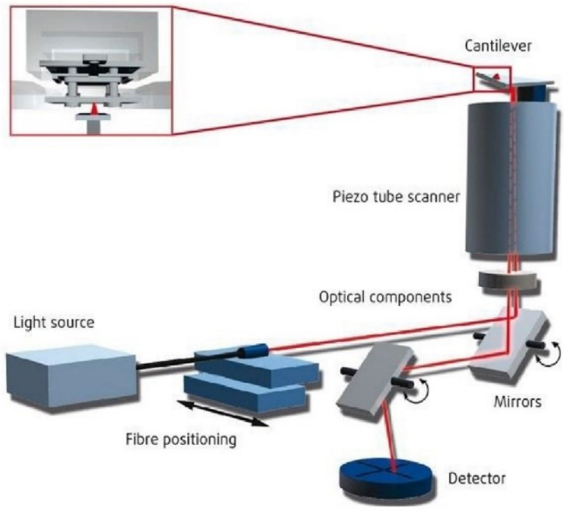

Figure 3: A schematic or an AFM system was shown in the figure. In this type of AFM, sample was placed at the top of cantilever which was not a common way to place the sample. Sample was mostly placed below the AFM tip in common AFM systems [5].

3.2. Optical Components

3.2.1 Light Source

Laser source was used a light source in the optical parts. The source should provide stable light. Wavelength and should be pre-defined since the slight frequency shifts in the light may result in deterioration in image quality. Most of the AFM manufacturers use visible laser light but some others prefer invisible light source. The systems which offer invisible lights, mostly provide automated calibration or positioning system that parameters such as quality and/or position of the light was set by automated software. In these systems, laser light was positioned on the shiny side of the tip system automatically. Moreover, the tip automatically approaches to the position where the investigation will be performed. For many systems, laser source was well protected and do not let user to make manipulation on it.

3.2.2. Mirrors and Photodetector

Mirrors used in the AFM system helps laser light to be adjusted to the desired point on position sensitive photo diode (PSPD). A sample schematic of the mirrors and photodetectors for an AFM system was presented in Figure 3. The laser light needs to be set at the top of the AFM tip and reflected light should be positioned at centre of photodetector. Therefore, multiple mirrors were used in a regular AFM system which were used to reflect the laser light to work in higher efficiency to obtain image in high quality. AFM producers mostly use the four segmented photodetectors (see Figure 4). Photo detector measures and detects the position change and properties of laser light such as frequency, intensity etc. When AFM cantilever was scanning the surface obstacles and/or structures on the surface cause deflection on the AFM tip. Slight deflection or movement on the tip results a big alteration in the position of the laser light. In non-contact mode AFM tip was also attracted by the features on the sample surface. Such position change and alteration of the frequency of the laser light was evaluated by the AFM electronics and attractive forces and the shape of the samples scanned on the surface was produced. At this point, positioning and calibration of the laser light is essential to have high quality images. Therefore, the light should be positioned at the middle of the four segmented photo detector (see the Figure 4 where red circle represents laser light positioned at the middle of photo detector).

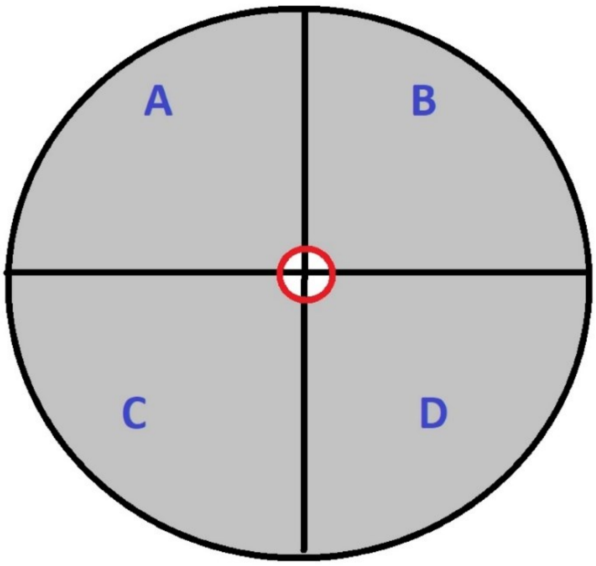

Figure 4: A schematic of the four segmented photodetector. Red circle represents the laser light focused on the middle of the detector.

3.3 AFM Electronics

3.3.1 Piezo Tube, Micromotor & Tip

Piezo tube system consists of two components piezo tube and micromotor. At the top of the system AFM cantilever was attached. The AFM tip was attached to the cantilever. Tip may be in many different forms, but most common forms are the tips in conical or pyramidal shapes. Some cantilevers may contain more than one where each tip may contain different stiffness and vibration frequency. Piezo system has two major objective approaching tip to the surface (*i*) and arrange the distance between the tip and surface (*ii*). Tip approach mechanism is essential to obtain a good image.

In contact mode, the tip should touch the surface properly. However, contact moment is very important. If the contact occurs softly, both properties and shape of the tip remain stable. Hard contacts or crashes may result in alteration in tip properties moreover the tip may get broken or damaged where double tip or tip related failures may be seen. At this point, micromorts and piezo actuator tube was used to provide soft contact. The micromotor approaches tip to the surface with micrometre size steps. The motor was used in rough approaches. When the distance between the tip and surface in couple of micrometres range, piezo actuators tube was used. The tube approaches the tip to the surface in nanometre susceptibility. Such susceptibility was arranged with the voltage applied to the piezo tube that piezo materials change their form with applied electric voltage. When the voltage applied to the piezo tube the tube expands and the vibration frequency of the tip was checked with photodiode. Regarding to the vibration frequency of the tip, system interprets whether the tip landed to the surface, or it is far away from it. If the distance is longer than the expansion limit of the tube, the tube was retracted and micromotor step forward. The process is repeated until the tip was softly landed to the surface.

In non-contact mode the AFM tip does not contact with surface. Therefore, the piezo actuators were used to provide stable distance between surface and tip. In this mode, the attraction force between the surface and the tip was measured. The attraction was correlated with the vibration frequency of the tip. When the tip was attracted with the surface the vibration frequency is changed. The alteration in the vibration frequency was measured with the photo detector. Piezo tube helps the alter the position of the tip to keep vibration frequency stable which was associated with the surface topography. The data obtained from, tip and piezo tubes were processed with processors and the image was obtained.

3.3.2 Processors and Feedback System (PAFS)

Processors and feedback system are essential to maintain the continuity of the scanning. The processor and feedback system (PAFS) both provide image and prevent tip and cantilever from crashing. The system also help tip to approach surface safely without crashing. PAFS work coordinatively with the detector and piezo tube. When the tip was scanning the surface, every motion of the tip was evaluated assessed by the photo detector. The data obtained from photo detector helps to maintain the position of the piezo tube which also help to change the position of the tip. These changes were coordinatively works and assessed by the feed bac system: The excessive increase or decrease detected by the detector stimulates feedback mechanism that the changes were compensated by the feedback system. All the data (frequency change, position change, alteration of piezo tube, alteration of the position of the tip etc.) evaluated by the processor (a schematics showing the working mechanism of processor and feedback system for non-contact mode was presented in Figure 5). Then the processor processes the topographic and map. Besides, attraction force curves and friction map, etc additional data about the evaluated sample was also created by the processor.

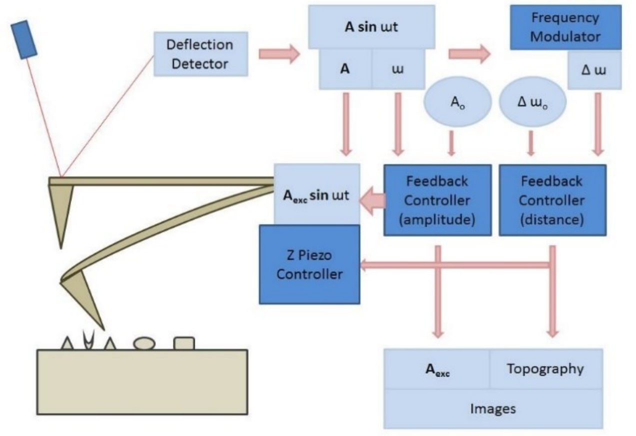

Figure 5: Schematic illustrates the working mechanism of processors and feedback system for non-contact mode AFM [5].

3.4. Forces Affecting the AFM Tip

Different types of forces such as magnetic force, van der Waals forces, electrostatic forces, capillary forces and friction forces may occur between the tip and sample surface. The intensity of such forces may affect the quality of the data obtained from the investigation.

Van der Waals forces named after Dutch physicist Johannes Diderik van der Waals which describes the attraction between the molecules in nano size regime. Dipole dipole forces was also attributed to describe these forces. Van der Waals forces are described in three main topic Dbye forces (*i*), Keesom forces(*ii*) and London forces (*iii*). Debye forces describes the attraction between one permanent and one induced dipole, Keesom forces assess the attraction between two permanent dipole and London forces describes the attraction between two induced dipoles. Van der Waals attractions occur due to the electronic charge fluctuations between the molecules. The distance between the molecules are expected to be in nanometre range to be attracted from such forces. As the forces are effective in nm range the forces were also referred short range forces. Since the tip distance between the tip and surface in nanometre range in both tapping mode and non-contact mode the tip was affected by such forces. Moreover, the tip geometry is also important where the forces acting a spherical tip can be described as

$$F = \frac{HR}{6D^2}$$

where F is the force, H Hamaker constant, R radius of the tip, D is the distance between the tip and the surface.

Electrostatic forces are the forces occurs due to the static charges exist or localize on the tip and surface. Such charges can make attractive or repulsive effect on tip. Many different factors may cause such charge localizations such as ion sputtering occurs due to sample preparation, friction between the tip and surface, electric voltage applied to the AFM during the measurement, charge localization in polymer samples etc. Such localization may produce attraction of repulsion between the tip and localized surface and affect the quality of the measurement.

Magnetic forces are mostly observed in ferromagnetic samples which contains Fe, Co or Ni in their structure. Especially in magnetic force microscopy, the localized magnetic forces may alter the surface properties. Ferromagnetic domains can interact with magnetic tip used in the measurement and the quality of the measurement may be altered.

AFM measurements in aqueous environment is more challenging than the measurement in vacuum environment and/or ambient environment. Variety of aqueous environment related factor effect the AFM performance; two major aqueous environment related factors can be explained as capillary effect (*i*) and double layer forces (*ii*).

Capillary effect is a strong effect that traps the tip which occurs not only in aqueous but also ambient conditions. Such effect is basically the trapping of AFM tip to a water bubble. The AFM tip is quite small compared to the sample surface. Therefore, the humid may be condensed between the tip and the surface or the tip could be trapped in a bubble on the surface. Such bubble catches the tip and the tip often are not able to get rid of the surface tension of the bubble. The bubble either crash the tip to the surface or the tip can not respond to the comments of AFM electronics. Hence, capillary effect results in deterioration in the obtained image.

Double layer forces can be observed in the investigations performed in aqueous environment. In aqueous measurements, the tip was submerged in aqueous solution. Sometimes the solution may contain ionic molecules. If the solution consists more than one ionic molecule, positive and negative charged molecules may accumulate in different side of the aqueous solution: For example, negative ions may accumulate on the surface and positive ions may accumulate on the tip. Such case starts to produce attraction between the surface and the AFM tip. Then, the oscillation frequency of the tip may be changed, or the tip may crash to the surface, depending on the attraction between the tip and surface. Therefore, the attraction deteriorates the data obtained from investigations in aqueous environment.

Friction forces also affect the image quality and the topographical image obtained from contact mode. Since AFM tip is continuously contact the surface tribological properties of the sample surface is important in contact mode investigations. Therefore, special tips produced with different stiffness and/or materials can provide relatively lower friction and may provide better topographic image. Even if contact mode is affected from friction forces, contact mode can use this as an advantage and the tribological properties of the sample can be calculated by assessing such forces [6]. The technique has used since the 80s which was first proposed by the Meyer et al.

3.5. Artefacts Affecting Image Quality

Sample or instrument related limitations can affect the quality of the AFM investigations. Such limitations may be caused by environmental issues, sample issues or AFM component related problems. Major problems experienced in the AFM measurements can be described as multiple tip effect, temperature change related thermal drifts, sample mobility, optical interference problems occur on optical components of AFM.

Thermal drift is one of the most common problem experienced in AFM investigations. Slight temperature change in the room where AFM was kept or temperature changes occur in AFM system may result in alteration scanned surface. Since the AFM tip scans very small piece of sample surface in nm range, slight change may result in expansion on sample in mm range. Such expansion my seem small but result in a drift on scanned region. In addition, expansion occurs on AFM components may also result alteration in scanned region. Especially, in consecutive images thermal drifts were often experienced since, the consecutive imaging may take up to several days.

AFM is capable of imaging the topographical features of the investigated sample in nanometre range resolution. However, it should be noted that the image obtained in the AFM investigation does not reflect the true image of the data since the obtained image is tip convoluted version of real image. Therefore, the obtained image was highly related to the shape of the tip (see Figure 6). During the AFM investigation, the shape of the tip may change due to the experimental factor. The alteration of the shape of the tip result in alteration of the obtained image. At this point couple of problem occurs. One of the most frequent problems is bifurcation of the AFM tip. When the tip was bifurcated or a dirt on the surface attached to it, doubled image was obtained; such case was called as double tip affect (see Figure 7). The double tip effect can be understood from image which looks shifted and every feature in the images which obtained by bifurcated tip looks twinned.

Un-adjusted laser light in the AFM measurement is also problem for measurement. Especially, unfocused laser light may disperse on the mirror like part of the tip. Such dispersion may unnaturally stimulate the photo detector of the AFM which results in deterioration in obtained image.

Lastly, the motion of the sample has deleterious effect on obtained image. Since the AFM tip result a pre-set region, a small motion in the sample result in alteration in the scanned region. Especially the samples in the liquids, tend to move. Constantly moving sample result un-healthy and unrealistic images.

3.6. Contact Mode

Contact mode is a method where the deflection in the laser light obtained by static motion of the AFM tip was used. In this method, AFM tip is put in a direct contact with the investigated sample. The method allow investigator to obtain many properties such as friction forces between the tip and surface, lateral contact stiffness, velocity dependent tribological properties of investigated system.

In the contact mode investigations, the position of the tip, the force applied to the tip, deflection amount obtained from photo detector was recorder. Then the data was evaluated, and all these data was processed by the processor. After the data process, many different properties mentioned above was obtained.

In the image processing, processors assess the deflection of the tip, the voltages applied to piezo tubes and the feedback obtained from comparison of these data. Deflection of the tip was assessed by the motion of the laser light reflected from the top bright side of the cantilever. At this mode attractive and repulsive forces might be prominent due to close distance between tip and surface where jumps can be seen while the tip was scanning the surface. The aim of probe microscopy is to achieve true atomic resolution. However, it is almost impossible to obtain such resolution using contact mode, since direct contact of the tip result in alteration in the contact area between the tip and the surface. Such size increase of the tip, decrease the resolution.

3.7. Tapping Mode (Hybrid Mode)

Tapping mode (also known as hybrid mode) is a method where AFM tip both oscillates above the surface and makes intermittent contact with surface at certain periods. Such method was developed to avoid tip-surface interaction related problems (problems occurs due to attractive or repulsive forces) and/or to reduce the friction related problems. Tip oscillates 20nm to 100nm above the surface and the vibration frequency is measured with photodetector. Electronic system of the AFM processes the position of the tip, vibration frequency and attractive and repulsive forces and produce the image. The resolution of the mode is limited with the radius of the tip, however this mode provides various advantages. For example, reduced contact with the surface result in reduced friction forces where increased image quality obtained. In addition, intermittent contact can provide information about the physical properties of the surface such as stiffness and elastic modulus.

3.8. Non-Contact Mode

In non-contact mode (also known as true non-contact mode), no contact between the surface and the tip occurs. The oscillates above the surface and the vibration frequency of the tip was measured with photodetector. Since, feedback system and electronic configuration is complex, a detailed schematic showing the mechanism of AFM electronics was illustrated in figure 5. Vibration frequency of the AFM tip is essential in noncontact mode. All feedback mechanism and AFM electronics constantly assess the vibration frequency of the tip. When the tip moves above the surface any feature on the surface interacts with vibrating tip. Such interaction results in alteration in vibration frequency of the tip. Alteration was assessed by the AFM electronics and the distance between the surface and the tip was arranged by piezo actuator. Changing in the distance, position of the tip and vibration frequency was processed, and the AFM image was produced (see figure 5). Since AFM tip does not have a direct contact with surface, no surface deformation was obtained in non-contact mode. Moreover, non-contact mode is able to provide atomic resolution which was previously evidenced by Giessible in 1995 for silicon (111) 7x7 [7]. Such resolution can be obtained by STM as well. However, STM is not able to work on the surfaces showing insulator characteristics. In addition, non-contact mode can be used in the investigation in aqueous media especially for the investigation of organic molecules such as DNA and proteins in their characteristic environment. Exposing such organic molecules to dry environment may decompose their structure, making measurement in aqueous environment help researcher to obtain high quality data about their research.

3.9. Data Acquisition

Most of the AFM producers provide their commercial AFM operating system with their product. The operating systems may vary depending on the producers. However, most of the operating systems can provide images in common image formats such as Jpeg, tiff, etc. Moreover, AFM also produce raw data file which can be processed by special software. Such software can either be provided by AFM manufacturer or free non-profit software can also be available in the market. WSXM is one of the most common AFM data process software [8]. Such software is able to process raw AFM data. Raw data can reflect many different properties obtained from the measurement. As it was described above, contact and non-contact data can provide different properties. Raw AFM data may contain friction, electric properties, surface topography, deflection of AFM tip, vibration alteration amount of the tip etc. User of the software can choose the requested data and process the data using such software. The software enables users to correct distorted and data, flatten the surface tilt, optimize image quality, obtain topographic information about surface such as root mean square roughness, height distribution, height profiling.

4. Consecutive Imaging

Consecutive imaging technique is a special technique that sequential image is taken at certain place of interest where dynamical changes was observed. Sometimes couple of hundreds of images were taken for certain places that the procedure may take couple of hours to days. Such technique was often used to investigate the agglomeration processes of atoms and early thin and ultra-thin film formation processes. Since the image of certain place was repeatedly taken, the technique is not suitable for contact mode imaging. Contact mode has direct contact with the surface and consecutive imaging of certain places using contact mode either damage the investigated surface and/or thin films. Moreover, direct contact of the tip cleanses the single atoms or molecules on the surface. In addition, single atoms or molecules attaches the tip of the AFM that bifurcation and/or double tip formation was occurred. At this point, doubled tip does not reflect the real data on the surface.

The technique helps to comprehend the behaviour of single atoms, early thin film formation processes, defect – mobile particle interactions, mobile particle – mobile particle interactions, mobile particle – stable particle interactions, film grow parameters, diffusion of single atom, diffusion of multiple atoms, step diffusion, jump diffusion, trace diffusion, collective diffusion etc. The technique often used in STM investigations that STM is an efficient tool to observe such small molecules in high efficiency. Swartzentruber evidenced that direct measurement of surface diffusion using atom-tracking scanning tunnelling microscopy was possible [9]. Brune et al uses the technique in the investigation of metal atoms and tin film formation processes where cryogenic ultra-high vacuum STM was used at around -170 ˚C [2]. Such temperature was chosen because the motion of atoms slows down at such cold environment that STM can find opportunity to observe them. Mitsui uses same technique to asses the diffusion of CO molecules and pair interactions on Pd (111) at 41K [10]. Diffusion of hydrogen atoms on Si (111)-(7 3 7) surfaces was assessed at 310˚C using STM by Lo and his group [11]. Bikondoa uses the same technique to illustrate the defect-mediated dissociation of water on TiO2(110) [12]. Mobile point defects and structural alteration and formation processes were evidenced by Hwang using STM (Hwang 1994). One and two dimensional diffusion of metal atoms was assessed Gan using STM [13]. Rosso et al investigated the self - diffusion of defects on pyrite(100) where UHV STM was used [14]. Si diffusion on pit free graphene was investigated by Sun and his group (sun 2011). Mobility of Silicane defects was illustrated by Gao and his group by using both theoretical and probe microscopy techniques [15]. Adsorption of phthalocyanine on graphite surfaces was assessed by Ahlund et al [16] . Koc and his group was focused on assessing thin films and graphite surface defects by using AFM consecutive imaging in non-contact mode. Self-healing mechanism of silicon nanoparticles was evidenced [17]. Diffusion mechanism of single layer graphite defects was illustrated [18]. At the end of this chapter, consecutive imaging method using non-contact AFM will be illustrated for highly oriented pyrolytic graphite (HOPG).

4.1. Experimental Procedure

Omicron (GMBH) UHV STM/AFM was used to obtain imaging process in non-contact mode. 9 mm × 3 mm × 2 mm Mosaic Spread Pi-Kem HOPG substrate with orientation degree of 0.5°–2.0 was used in the assessment. HOPG was cleaned with isopropanol. Sample was placed to UHV load chamber of the AFM and vacuumed there for one day. The sample was then transferred to the main chamber of the AFM where the non-contact investigation was conducted at room temperature. Investigated region was chosen and tip was approached. Constant force, noncontact mode was applied in the investigation process. Fine adjustment was made, and scanning was started. Consecutive 165 and 33 images were taken for two different HOPG defect (It should be noted that image taking processes were performed for two different HOPG crystal). Sometimes, drifts were observed in the image taking process. It was seen that the region where image was taken was slightly shifted. Drift was manually corrected with visual inspection in the image taking process. The real drift correction was made after finishing the investigation using drift correction tool of WSXM software. When the image process was finished, raw AFM data was processed with WSXM software [8]. Z profile images were chosen and were put in sequential order. A video was obtained by combining those images. Drift correction was made using WSXM software. Then dynamical changes occurred on the investigated region was assessed.

4.2. Evaluation of the Results

In this section, two different HOPG defects were investigated using consecutive AFM imaging. Consecutive images obtained from different HOPG surfaces were illustrated some of the investigations revealed that some HOPG defects show mobile characteristic. In Figure 6, motion of a mobile HOPG defect (defect 1) was illustrated. 165 consecutive images were obtained, and certain images were chosen to illustrate the motion of mobile defect. Height profile analysis showed that the thickness of the defect is about 0.35 nm which was illustrated at the Figure 6 f. The region where height profile was taken is showed with green line in Figure 6 e. Height profile indicate that the defect consists of single layer HOPG. It can be seen that the defect moves in a horizontal position which was pointed with white arrow. In addition, an active particle like formation was also defined in the surface and pointed with white arrow. It was seen that white particles having tail like structure moves horizontally on the surface. At the last image particle collide with another particle (see figure 6 e).

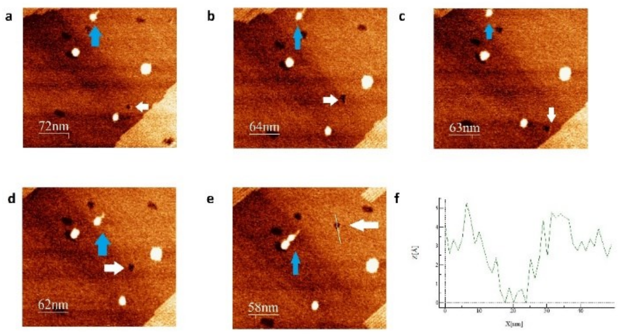

Figure 6: Motion of a defect 1 and a particle like structure was evidenced with consecutive imaging technique. Images sequence belonging 16 (a), 30 (b), 41 (c), 78 (d), 126 (e) and height profile of defect (f) were showed in the schematics. White arrow points single layer defect and blue arrow points particle like structure. White line in the (e) shows the line where the height profile was taken.

Another consecutive imaging application of an HOPG defect (defect 2) was illustrated in Figure 7. 33 consecutive images were obtained in this investigation. Images were taken using non-contact AFM in constant force mode. Height profile of the defect indicate that the defect is a single layer HOPG defect (see Figure 7e and f). Investigation of consecutive images revealed that the defect moves in horizontal direction. In figure 7, motion of the defect was tracked by using C/C++ open CV library image processing tool [19]. In the tracking process, the images were converted to grey scale to allow programme to track the defect using the tone difference in the images. The software tracked the path of the defect and trace of the motion was produced (See figure 7b). The distance taken each step was calculated and distribution was plotted. It was seen that distribution show almost Gaussian like shape which indicate the motion of the defect was random motion. Trace obtained from the motion of the defect was also support such idea (see Figure 7d).

As can be seen in two different examples presented in the section, consecutive imaging used by AFM technique can give information about the dynamic changes occurred on the surface. It was evidenced and showed that non-contact AFM can be used to asses the dynamical alterations occurred on the surface. It should be noted that consecutive imaging is a complex technique and different parameters should be taken into consideration such as thermal drifts or shifts occurs in the imaging process. However, the technique still can be used to assess the dynamical alterations occurs on the surface.

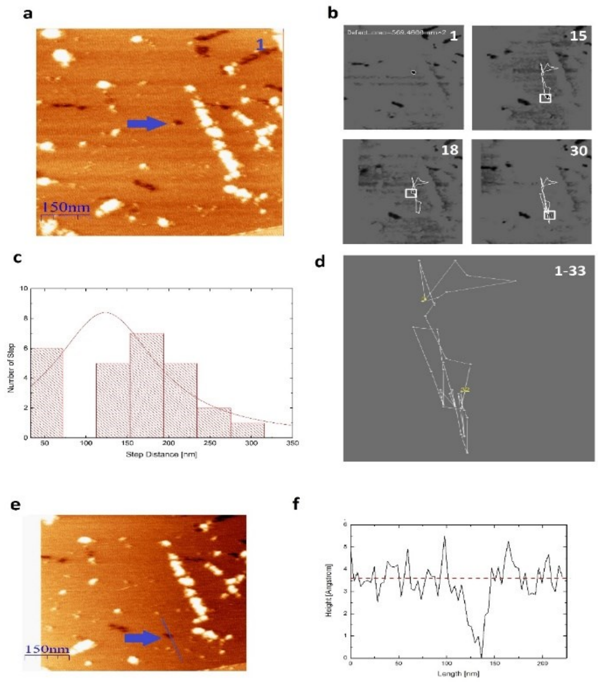

Figure 7: First image of defect 2 (a), tracking process of defect motion in grey scale(b), step distance distribution of defect 2(c), trace path of defect 2 (d) and height profile defect 2 (e, f) was presented in the figure. Blue line in Figure 7 (e) was presented in the figure.

References

[1] G. Binnig, H. Rohrer, C. Gerber, E. Weibel, Surface Studies by Scanning Tunneling Microscopy, Phys. Rev. Lett. 49 (1982) 57–61. doi:10.1103/PhysRevLett.49.57.

[2] H. Brune, Microscopic view of epitaxial metal growth: nucleation and aggregation, Surf. Sci. Rep. 31 (1998) 125–229. doi:10.1016/S0167-5729(99)80001-6.

[3] T. Ito, S. Ibrahim, I. Grabowska, Chemical-force microscopy for materials characterization, TrAC Trends Anal. Chem. 29 (2010) 225–233. doi:10.1016/J.TRAC.2009.12.008.

[4] E. Soergel, Piezoresponse force microscopy (PFM), J. Phys. D. Appl. Phys. 44 (2011) 464003. doi:10.1088/0022-3727/44/46/464003.

[5] M. Koc, Non Contact Atomic Force Microscopy Investigation of Silicon Nanoparticles Deposited on HOPG, (2015). https://lra.le.ac.uk/handle/2381/36124 (accessed May 22, 2018).

[6] E. Meyer, H. Heinzelmann, P. Grütter, T. Jung, H.-R. Hidber, H. Rudin, H.-J. Güntherodt, Atomic force microscopy for the study of tribology and adhesion, Thin Solid Films. 181 (1989) 527–544. doi:10.1016/0040-6090(89)90522-1.

[7] F. Giessibl, True atomic resolution by atomic force microscopy through repulsive and attractive forces., Science (80-.). 267 (1995) 68–71. doi:10.1126/science.267.5194.68.

[8] I. Horcas, R. Fernández, J.M. Gómez-Rodríguez, J. Colchero, J. Gómez-Herrero, A.M. Baro, WSXM : A software for scanning probe microscopy and a tool for nanotechnology, Rev. Sci. Instrum. 78 (2007) 013705. doi:10.1063/1.2432410.

[9] B.S. Swartzentruber, Direct Measurement of Surface Diffusion Using Atom-Tracking Scanning Tunneling Microscopy, Phys. Rev. Lett. 76 (1996) 459–462. doi:10.1103/PhysRevLett.76.459.

[10] T. Mitsui, M.K. Rose, E. Fomin, D.F. Ogletree, M. Salmeron, Diffusion and Pair Interactions of CO Molecules on Pd(111), Phys. Rev. Lett. 94 (2005) 036101. doi:10.1103/PhysRevLett.94.036101.

[11] R.-L. Lo, I.-S. Hwang, M.-S. Ho, T.T. Tsong, Diffusion of Single Hydrogen Atoms on Si(111)-(7 × 7) Surfaces, Phys. Rev. Lett. 80 (1998) 5584–5587. doi:10.1103/PhysRevLett.80.5584.

[12] O. Bikondoa, C.L. Pang, R. Ithnin, C.A. Muryn, H. Onishi, G. Thornton, Direct visualization of defect-mediated dissociation of water on TiO2(110), Nat. Mater. 5 (2006) 189–192. doi:10.1038/nmat1592.

[13] Y. Gan, L. Sun, F. Banhart, One- and Two-Dimensional Diffusion of Metal Atoms in Graphene, Small. 4 (2008) 587–591. doi:10.1002/smll.200700929.

[14] K.M. Rosso, U. Becker, M.F. Hochella, Surface defects and self-diffusion on pyrite {100}: An ultra-high vacuum scanning tunneling microscopy and theoretical modeling study, Am. Mineral. 85 (2000) 1428–1436. doi:10.2138/am-2000-1011.

[15] J. Gao, J. Zhang, H. Liu, Q. Zhang, J. Zhao, J.Z.-Nanoscale, undefined 2013, Structures, mobilities, electronic and magnetic properties of point defects in silicene, Nanoscale. 5 (2013) 9785. doi:10.1039/c3nr02826g.

[16] J. Åhlund, J. Schnadt, K. Nilson, E. Göthelid, J. Schiessling, F. Besenbacher, N. Mårtensson, C. Puglia, The adsorption of iron phthalocyanine on graphite: A scanning tunnelling microscopy study, Surf. Sci. 601 (2007) 3661–3667. doi:10.1016/J.SUSC.2007.06.008.

[17] M. Koç, M.J. McNally, K. von Haeften, M.J. Watkins, AFM induced self-assembling and self-healing mechanism of silicon oxide nanoparticle linear array domains templated on Moiré superlattice patterns on HOPG, Surf. Sci. (2019)

230–239. https://www.sciencedirect.com/science/article/pii/S0039602818305235 (accessed January 2, 2019).
[18] M.M. Koç, G.E. Ragkousis, AFM induced diffusion of large scale mobile HOPG defects, Appl. Nanosci. (2018) 1–10. doi:10.1007/s13204-018-0929-z.
[19] G. Bradski, A.K.-D.D. journal of software tools, undefined 2000, OpenCV, Ros.Exbot.Net. (n.d.). http://ros.exbot.net/wiki/attachments/Events(2f)ICRA2010Tutorial/ICRA_2010_OpenCV_Tutorial.pdf (accessed May 22, 2018).