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Review

Atomic force microscopy studies on sulfur-, selenium- and tellurium-based metal chalcogenide thin films: A review

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Sulfur, selenium and tellurium-based metal chalcogenide films have been prepared using various deposition methods. Investigation of morphological properties of the generated surface structures on chalcogenide thin films using atomic force microscopy technique was reported. The purpose of this work is to describe past important research findings that are related to atomic force microscopy technique.

Key words: Atomic force microscopy, surface roughness, film thickness, grain size.

INTRODUCTION

Sulfur-based films (Ho et al., 2013; Saravanan et al., 2010; Mohd et al., 2011; Abdullah et al., 2010; Dhandayuthapani et al., 2017; Huse et al., 2017; Ahmad et al., 2010; Garcia et al., 2017), selenium based films (Ham et al., 2008; Xue et al., 2006; Rajesh et al., 2013; Kassim et al., 2010; Wen et al., 2017), and tellurium-based metal chalcogenide films (Laxman et al., 2012; Pandiaraman et al., 2011; Camacho-Espinosa et al., 2014; Chen et al., 2009; Yang et al., 2017) possess useful electrical, optical and physical properties. These films can be found in many applications such as optoelectronic devices, laser devices, photovoltaic cell, microelectronic, and nano electronics. They could be prepared by a variety of deposition methods including chemical bath deposition, electro deposition, thermal evaporation, chemical vapor deposition, molecular beam epitaxy, metal organic vapor phase epitaxy, pulsed laser deposition, spray pyrolysis, successive ionic layer adsorption, and reaction. There are a number of papers that report the results of morphological, structural, compositional, functional group, and optical characterization of thin films. These films were characterized using range of characterization techniques such as X-ray photoelectron spectroscopy (Lisco et al., 2015; Meng et al., 2015; Zhang et al., 2001; Subramanian et al., 2001), scanning electron microscopy (Remigijus et al., 2012; Anuar et al., 2010; Yazid et al., 2009; Murilo and Lucia, 2016; Salh et al., 2017; Amira and Hager, 2017), X-ray diffraction (Zulkefly et al., 2010; Saravanan et al., 2008; Kamoun et al., 2007; Ho et al., 2010; Sall et al., 2017; Kiran et al., 2017; Anitha et al., 2017; Kassim et al., 2011), transmission electron

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microscopy (Chen et al., 2016; Mukherjee et al., 2016a; Gallardo et al., 2016; Ghribi et al., 2016), energy dispersive X-ray analysis (Jelas et al., 2011; Deshmukh et al., 2017; Khan et al., 2017; Bakiyaraj and Dhanasekaran, 2013), Fourier transform infrared spectroscopy (Sahuban et al., 2016; Dedova et al., 2005; Taj and Tayyaba, 2012), and UV-Visible spectrophotometer (Ho et al., 2011; Thirumavalavan et al., 2015; Ersin and Suleyman, 2015; Ramesh et al., 2014).

ATOMIC FORCE MICROSCOPY

The first atomic force microscopy was operated in contact mode by Binnig et al. (1986) in Fiorani and Sberveglieri (1994). The AFM technique has been used for problems in a wide range of fields of the natural sciences such as semiconductor, medicine, molecular biology, solid state physics, polymer chemistry and nanomaterials (Xie et al., 2011). Applications of the AFM technique include the identification of atoms at a surface (Sergei and Whangbo, 1996), the study of changes in physical properties (Gernot and Henning, 2011), the evaluation of interactions between a specific atom and its neighboring atoms (Jaroslaw and Kash, 2005).

Atomic force microscopy works by bringing an atomically sharp tip close to a surface (Franz and Calvin, 2006). AFM tips were made from silicon, silicon nitride (Alan, 2015), silicon oxide (Enrico and Ernst, 2015), and quartz (Rebecca and Lisa, 2000). Basically, there are few main parts in AFM. Microscope stage is used for moving AFM tip (Maurice et al., 2007), sample holder and force sensor. Controls electronics consist of optical microscope (Peter and Paul, 2010) and vibration controller. The control electronics usually takes the form of a large box interfaced to both the computer and microscope stage. The computer is employed for setting the scanning parameters such as scan speed and scan size. Lastly, frame supports the whole AFM microscope. It must be very rigid (Georg et al., 2006), so that, it does not let vibrations between the surface and tip.

Modes of AFM

There are different imaging modes of AFM, namely, contact mode, non-contact mode and tapping mode (Nikodem and Kuan, 2011). The first and original mode of operation is contact mode (Bharat et al., 2008). It has the ability to produce high resolution images. Tip of the probe always touching the sample (less than a few angstroms) in this mode. In this mode, the deflection of the cantilever is sensed (Seungbum, 2004) and compared in a DC feedback amplifier to some desired value of deflection.

The first non-contact mode was developed by Martin et al. (1987) in Baldeschwieler et al. (1996). In non-contact mode, a sharp probe is moved close (ranging from tens of angstroms to hundreds of angstroms) to the surface under study. It uses detection of a cantilever resonant frequency as an indirect measure of sample topography. Finally, the image is constructed from the force interactions during the scan (Kantorovich et al., 2000).

Tapping mode is used by researcher when operating in ambient conditions or in liquids (Yang, 2009). In this mode, the cantilever is driven to oscillate up and down at or near its resonance frequency. This mode allows high resolution topographic imaging of sample surfaces including on surfaces that are easily damaged and loosely held to their substrate. Furthermore, it overcomes some problems associated with friction, adhesion and electrostatic forces (Fernando et al., 2004) that can plague other scanning methods.

Scanning electron microscope (SEM)

Scanning electron microscope (SEM) is a type of electron microscope scans, a focused electron beam over a surface to create an image (Nidal et al., 2017). The electrons in the beam interact with the sample, producing various signals that can be used to collect useful information (Patrick, 2009) such as surface topography and composition (Ahmad et al., 2015). Transmission electron microscopy (TEM) is employed to observe the features of very small specimens such as structure and morphology (Jozef, 2017). It uses an accelerated beam of electrons (Olga and Dieter, 2012), which passes through thin specimen to produce images. Comparison between scanning electron microscopy, atomic force microscopy and transmission electron microscopy as listed in Table 1.

Here, this review will focus upon morphology applications of AFM technique in sulphur-, selenium-, tellurium-based metal chalcogenide films. In this article, author discusses the analysis results (published literature), advantages and limitations of AFM.

LITERATURE SURVEY: METAL CHALCOGENIDE THIN FILMS

The author performed extensive searches in Google Scholar, Scopus, ISI and international refereed journals, using a combination of the search terms: atomic force microscope, film thickness, surface roughness, grain size, and metal chalcogenide thin films.

Atomic force microscopy (AFM) is a highly sensitive technique (Shivprasad et al., 2005) can give the images on the nanometer scale (Lehr, 2000). AFM has found increasing use in the material sciences and engineering fields (Last et al., 2010). Since 1990, the number of research publications making use of AFM to investigate the morphology of samples has increased (Ho, 2014; Mukherjee et al., 2016b; Soumya et al., 2014; Daniel et al., 2016; Siang et al., 2011; Kelvin et al., 2011). The

Table 1. Comparison between SEM, TEM and AFM.

Parameter	Scanning electron microscopy	Atomic force microscopy	Transmission electron microscopy	References
Sample type	Conductive	Conductive/Insulating	Conductive	Bruno and Khatib (2008)
Magnification	2-Dimensional	3-Dimensional	2-Dimensional	Kholoud et al. (2010)
Sample environment	Vacuum	Vacuum/Air/Liquid	Vacuum	Peter and Paul (2010)
Time for image	Up to 60 s	60 - 300 s	Up to 60 seconds	Carter and David (2016)
Relative cost	Medium	Low	High	Li and Wu (2014)
Horizontal resolution	5 nm	0.2 nm	0.2 nm	John (2013)
Field of view	1 mm	100 µm	100 nm	Linda et al. (2008)
Depth of field	Good	Poor	good	Peter and Paul (2010)
Contrast on flat sample	Poor	Good	poor	Xie (2013)
Maximum sample size	30 mm	Unlimited	2 mm	Bryant et al. (1993)

characterization of obtained films is an important step in order to identity and improve the quality of films. AFM has been traditionally evaluated as a powerful tool to carry out an accurate measurement of surface roughness of films (Victor, 2012). Surface roughness can affect the optical properties of films (reflectivity and scattering) and electrical conductivity as well.

Sulphur is a chemical element (S) and has a pale yellowish color. It has atomic number 16, with a chemical formula S_8. In pure form, it is completely tasteless and odorless. Abundant multivalent non-metal sulphur happens in many sulfide and sulfate minerals. Non-toxically enhanced sulfur reaction for formation of chalcogenide thin films and have been widely employed for high performance optoelectronic devices. Table 2 shows the surface roughness, film thickness and grain size of sulfur-based metal chalcogenide films measured by AFM technique.

Selenium is a non-metal, with symbol Se and atomic number 34 (Valadabadi et al., 2010). It is rare and in the Group 16 of the period table. Generally, it is a mineral found in the soil. The used selenium has good photovoltaic and photoconductive properties (Christopher et al., 2016); the most important uses of selenium in optoelectronic devices and solar cells (David, 2013). This is due to the fact that it has good photovoltaic and photoconductive properties. For example, the electrical conductivity increases more than 100 fold in illumination conditions. Additionally, the electrical resistivity of selenium varies over a tremendous range, depending upon experimental conditions. Table 3 indicates the surface roughness, film thickness and grain size of selenium-based metal chalcogenide films measured by AFM technique.

Tellurium has an atomic number of 52 and the symbol Te. Tellurium has a silvery-white appearance and is found in native form as elemental crystals. Tellurium is an element in Group VI of the Periodic Table, which has been intensively studied because of its unique optical and electrical properties. Tellurium films show p-type conduction (Begona et al., 2015), because of lattice defects acting as acceptors. The band gap about 0.34 eV (Chen et al., 2017) and the carrier concentration at room temperature is in the range of (1-5) × 10^{18} cm^{-3}. Tellurium is used in cadmium telluride (CdTe) solar panels, a rapidly growing and increasingly important market. Massive commercial production of CdTe by First Solar Company has significantly increased tellurium demand. However, CdTe is classified as harmful if inhaled and harmful to aquatic life with long lasting effects. Table 4 displays the surface roughness, film thickness and grain size of tellurium-based metal chalcogenide films measured by AFM technique.

Researchers expressed some challenges (Ampere, 2011) of atomic force microscopy technique. For instance, there is need for sharp probe (Dufrene, 2011) for high resolution in order to achieve good results. The feedback controller should have a fast control in order for adjust topographic film to be produced. A high speed computer is needed to produce the images in real time. Lastly, the force between probe and sample should be 1 nN or less because of noise and stability considerations.

CONCLUSION

Current research literature suggests that atomic force microscopy is one of the most widely used instruments in research in the material sciences and engineering. Characterization of morphological metal chalcogenide thin films could be done using this instrument effectively. Three-dimensional AFM images of topography provide nanoscale information on film structure including surface roughness, film thickness, and grain size.

CONFLICT OF INTERESTS

The author has not declared any conflict of interests.

Table 2. Surface roughness, film thickness and grain size of sulfur-based metal chalcogenide films measured by AFM technique.

Thin films	Deposition technique	Results	Reference
ZnS	Thermal evaporation	The surface roughness and grain size (24.2 to 31.4 nm) increase with the increase of film thickness (310 to 1240 nm).	Wu et al. (2008)
MnS	Chemical bath deposition	AFM study indicates spherical grains having coalescences between them. Mountainous like structures have extended characteristics with planar crest as shown in 3-dimenstional	Sunil et al. (2017)
CuS	Thermal co-evaporation	The increased in grain size (32 to 61 nm) and RMS roughness values (24 to 42 nm) as film thickness increases from 100 to 200 nm.	Sahoo et al. (2015)
CdS	Spray pyrolysis method	Some pin holes could be seen in AFM images. The grain sizes vary between 100 and 300 nm and surface roughness about 45 nm.	Baykul and Balcioglu (2000)
SnS	Thermal evaporation	AFM studies showed that the grain size and roughness increased from 51-265 nm and 2.3-6.8 nm, respectively with increase of substrate temperature (50 to 300°C).	Hegde et al. (2011)
CoS	-	Film thickness (458-1354 nm) increased with increase in ammonia concentration up to 15 mL, then, thickness decreases for higher concentration of ammonia (18-25 mL). RMS roughness (123.3 to 21.4 nm) reduced with increase in TEA concentration (2-6 mL)	Kamble et al. (2015)
FeS	Electrodeposition and hydrothermal method	The films are compact and have cubic structures distributed irregularly in the film surface. The average grain size of 192 nm and average RMS value about 251 nm could be obtained.	Henriquez et al. (2016)
NiS	Dip coating method	RMS roughness value was 19.2 and 14.3 nm for the NiS films prepared on the porous-TiO_2 and compact TiO_2 layers.	Kang et al. (2017)
PbS	Chemical bath deposition	Larger average particle size (37-137 nm) could be found with increasing the deposition temperature (22-50°C).	Hajar et al. (2016)

Table 3. Surface roughness, film thickness and grain size of selenium-based metal chalcogenide films measured by AFM technique.

Thin films	Deposition technique	Results	References
CuSe	Chemical bath deposition	AFM image shows the uniform distribution of agglomerated CuSe nanoparticles on the surface of substrates.	Soundararajan et al. (2015)
CdSe	Electron beam evaporation	Average roughness properties of CdSe films prepared at room temperature (0.34 nm), 100 (0.21 nm), 200 (0.25 nm) and 300°C (0.35 nm) were investigated using AFM.	Rani and Shanthi (2014)
SnSe	Spin Coating Method	It is found that as the thickness is increased from 1 to 4 µm, the average roughness is reduced (9.88 to 3.97 nm)	Keyur et al. (2016)
PbSe	Electro chemical atomic layer epitaxy	A number of smaller crystallites (300 nm) could be seen in AFM image of a 50 cycle electrodeposited PbSe thin films on annealed Au substrate.	Raman et al. (2004)
FeSe	Electron beam deposition	AFM images indicate that film thickness (29.7 to 270 nm) and roughness (6.3 to 30.8 nm) are directly related to the temperature changes (room temperature to 300°C).	Segu et al. (2017)
ZnSe	Molecular beam epitaxy	RMS value is 2.9 nm and rough surface is seen.	Jung et al. (2006)

Table 4. Surface roughness, film thickness and grain size of tellurium-based metal chalcogenide films measured by AFM technique.

Thin films	Deposition technique	Results	References
CdTe	Close space sublimation	The grain sizes of CdTe films deposited at 450 and 620°C were 0.3 µm and 2 to 5 µm. Due to the higher surface mobility during deposition.	Al-Jassim et al. (2001).
SnTe	Molecular beam epitaxy	Atomic force microscopy image of 7.09 µm SnTe layer grown on (111) BaF_2 surface revealed spirals with monolayer steps formed around threading dislocations. There are some small black pits, called holes (left during growth) could be detected.	Mengui et al. (2006)
PbTe	Electro deposition	As shown in AFM images, the crystallite size (70 to 200 nm) and film thickness (60 to 150 nm) increased with an increase in deposition time (6 to 15 h)	Ibrahim et al. (2009)
ZnTe	Molecular beam epitaxy	With increasing film thickness (10.8 to 797. 6 nm), the grain radius (95 to 297 nm) and RMS value (0.79 to 13.2 nm) gradually increase	Klapetek et al. (2003)
CuTe	Thermal evaporation	The grain size and roughness were 40 and 3.2 nm, respectively.	Neyvasagam et al. (2007)

CONFLICT OF INTERESTS

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