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Influence of Indium Tin Oxide (ITO) poling temperature in vacuum on surface roughness of Polyvinylidene Fluoride(PVDF) film

Suprapto[1*], Lisyanto[1], Eswanto[1], Selamat Riadi[1], Safri Gunawan[1], Henry Iskandar[1], Jubaidah[2], Muhtadin[3]

[1]Mechanical Engineering, Universitas Negeri Medan, Medan, 20221, Indonesia
[2] Physic, Universitas Negeri Medan, Medan, 20221, Indonesia
[3]Mechanical Engineering, National Taiwan University of Science and Technology, Taiwan (NTUST), Taiwan
*Corresponding author: suprapto@unimed.ac.id

Abstract

Surface roughness is a significant parameter of the performance of PVDF thin film sensors. In this work, the effect of ITO poling in vacuum at various temperatures on the surface roughness of uniaxially stretched PVDF film was investigated. The influence of poling temperature on the surface roughness of PVDF film was examined using Atomic Force Microscopy (AFM). In this work, the AFM data were obtained through the database, and topography was analyzed using Gwydion Software (GS). GS has characterized surface roughness in terms of average roughness (R_a), root mean square (R_{rms}), and arithmetic average height (R_z). The results show that the GS can analysis and measure profile thickness and roughness on a nanoscale with reliable accuracy. The results confirm that the increased poling temperature can reduce the roughness of the surface.

Keywords:
PVDF, ITO poling, roughness surface, AFM, Gwydion software.

1 Introduction

Rapid advancements in materials science and engineering have created innovative materials with specialized qualities to fulfill the ever-changing demands of diverse sectors. PVDF has received much attention because of its exceptional piezoelectric characteristics, flexibility, great thermal stability, and high mechanical strength [1, 2]. Polyvinylidene Fluoride (PVDF), as a type of piezoelectric material, possesses a diverse array of applications such as sensors, actuators, energy harvesting, storage devices, and various other functions[3, 4]. The efficacy of PVDF in these applications is often decided by its surface qualities, particularly its surface roughness. Investigating surface roughness and topography has become essential for comprehending and enhancing the efficiency of PVDF films in this domain. The performance of PVDF thin sensors and other electric device applications is substantially affected by surface roughness[5, 6].

Roughness can significantly affect materials dielectric and electrical properties, particularly in the context of surfaces and interfaces. These effects are particularly relevant in microelectronics, thin films, and materials science roughness effects on dielectric and electrical properties [7]. Rough surfaces can lead to enhanced electric field concentrations at surface irregularities. It can increase surface leakage currents during poling, a problem in high-voltage applications. Additionally, sharp, rough features can act as sites for electrical breakdown, creating conducting paths where they should not exist. In high-frequency applications, the surface roughness can lead to increased losses and decreased performance due to additional scattering and absorption of electromagnetic waves.

Previous studies [5, 8, 9]examined the impact of surface roughness on the piezoelectric properties of PVDF. Research has shown that precise control of surface roughness can enhance the piezoelectric response of PVDF, making it well-suited for applications in sensors, actuators, and energy-harvesting devices. Methods for surface texturing, such as micro or nano-structuring, have been studied to create controlled roughness. Previous research [10] found that PVDF films with roughness surfaces exhibited high piezoelectric phase content and demonstrated good dynamic and static response. Poling temperature and electric field significantly affect the piezoelectric properties of PVDF films. Ting, Y [11] found that Indium Tin Oxide(ITO) glass is used as an electrode to transmit a high voltage into PVDF for the poling process can enhance piezoelectric response compare than conventional poling method. According to Porter [12], increased higher β-phase content in PVDF films was related with lower extrusion temperatures, faster extrusion rates, and higher voltages.

Currently, AFM is an important device for investigating surface morphology and mechanical properties. Analysis with an AFM has been widely utilized to characterize material properties for PVDF nanofiber[13], surface chemistry [14], semiconductor [15]. It is a dependable instrument for providing helpful information about a material's surface morphology, pore size distribution, surface roughness, and pore density. AFM provides 3D scanning topography of diverse materials' contact surfaces at micro and nano levels, resulting in high-resolution 3D pictures [16, 17]. Unlike Scanning Electron Microscopy (SEM), AFM may be utilized at room temperature without any surface preparation, such as coating with conductive material (gold). It can benefit coating materials with critical applications in various fields, including piezoelectric devices and sensors. AFM allows researchers to image the surface of piezoelectric materials such as PVDF and piezoceramic (PZT) at high resolution, allowing visualization of surface features, such as grain boundaries, grain size, defects, and domains, which can have significant implications for material performance [18]. AFM can perform nanomechanical measurements, such as nanoindentation and force spectroscopy, on piezoelectric materials [19]. These measurements can provide valuable information about the material's mechanical properties, including hardness, elastic modulus, and adhesion forces [20].

Characterization of piezoelectric devices can also use AFM to help optimize device design and increase efficiency and sensitivity. In the context of AFM, the magnitude of the surface roughness or irregularity on the sample surface may be anticipated using the Root Mean Square (RMS) value [5]. It is usually used to measure variations in elevation or deviation from the average surface elevation. RMS is an essential parameter in surface characterization, as it can provide valuable information about the overall surface roughness at the nanoscale[21]. The RMS value is just one of many factors used to measure surface roughness. Additional parameters, such as average roughness (R_a), root mean square roughness (R_{rms}), and peak-to-valley height (R_z), are commonly employed to provide insights into the surface's topography and roughness properties.

The Root Mean Square (RMS) value provides a precise measurement of surface roughness, where a lower RMS value indicates a smoother surface and a larger RMS value indicates a rougher or more uneven surface. AFM's action at the nanoscale necessitates measuring RMS in nanometers (nm). Examining the surface roughness of materials is of significant importance in several components, including PVDF thin film sensors. An inherent challenge in thin film sensors is the presence of surface roughness resulting from various mechanical polishing procedures.

Surface roughness has a substantial influence on the performance of thin film sensors. Based on those mentioned earlier, this study's objective is to observe and investigate the surface roughness of stretched PVDF and the effect of Indium Tin Oxide (ITO) poling conditions in a vacuum oven with various temperatures using AFM. Using Gwydion software, all of the data obtained is evaluated in the form of certain roughness parameters (R_a, R_{rms}, and R_z) and photographic photos in 2D-3D roughness profile.

2 Materials and Methods

2.1 Material

The unstretched commercial PVDF (Kynar®720) with a thickness of 130 mm was acquired from the company for this study. The PVDF film was divided into rectangular pieces measuring 15 cm ×20 cm and affixed to a uniaxial stretching mechanism. An oven chamber is equipped with a purpose-built uniaxial stretching apparatus capable of bi-directional movement (x-y). The apparatus is fitted with heaters located at the top and bottom. During the stretching operation, the clamps undergo opposing directional movement as the machine shaft is rotated, as depicted in Fig. 1. The PVDF sample underwent a fivefold stretching ($R = 5$) at a temperature of 80°C. The stretching process was regulated by circulating hot air at a velocity of 0.5 m/s, and the stretching rate remained constant at approximately 40 mm/h. The stretching ratio (R) is the quotient of the final length divided by the original length of the PVDF film. The formula for determining the stretching ratio (R) is Eq. 1.

$$R = \frac{Final\ Length}{Original\ Length} = \frac{(L_o)}{(L_i)} \tag{1}$$

where L_o is final length of PVDF film, L_i is original length of PVDF film.

The stretching force (F) applied to a PVDF film can be calculated using Hooke's Law for linear elasticity, which relates the force tensile to the PVDF films properties and the deformation. The formula for stretching force is Eq. 2.

$$F = k \cdot \Delta L \tag{2}$$

Where k is the material's stiffness or Young's modulus ($k =$ 1.7 GPa). ΔL is the change in length, which is the difference between the final length ($L_o = 1$ m) and the original length of the PVDF film ($L_i = 0.2$m). The stretching force (F) is approximately $1.36{\times}10^9$N. The final thickness of PVDF films was between 17and 50 mm, which shows a dependence on stretching parameters (dimension origin of PVDF, speed rate, and temperature).

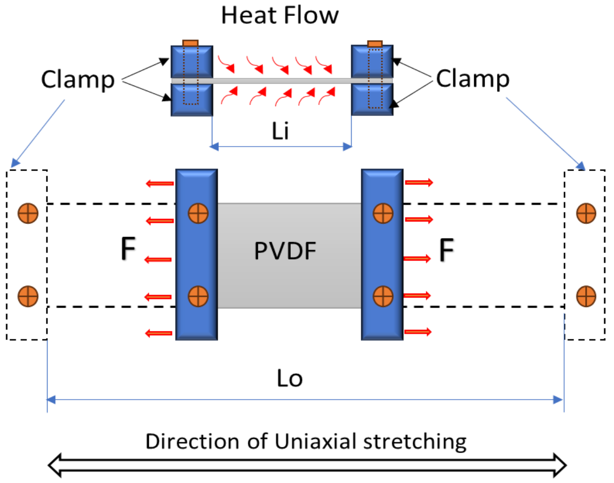

Fig. 1. Schematic illustration uniaxial stretching of the PVDF film.

2.2 Polarization

Polarization is a crucial factor in enhancing the piezoelectric characteristics of the PVDF layer. The process involves subjecting the polymer's molecular dipoles to a strong electric field in order to align them in the desired orientation. A domain refers to a small region within a crystal that exhibits uniform polarization. These domains are not in alignment in their inherent state. Polarization can be achieved by delivering a Direct Current (DC) electric voltage for a specific duration, which aligns the domains in a unified direction[22]. Domain direction dependsupon the direction of polarization[23, 24]. This alignment greatly enhances the piezoelectric charge coefficients and sensitivity of the device, improving its efficiency in transforming mechanical stimuli into electrical signals. Traditional polarization approaches commonly involve the attachment of metal electrodes on PVDF films. Although effective, these approaches have limitations, especially in obtaining significant electric field strengths during polarization. Flashover and arcing are serious issues that might impede the polarization process, lowering PVDF's overall performance.

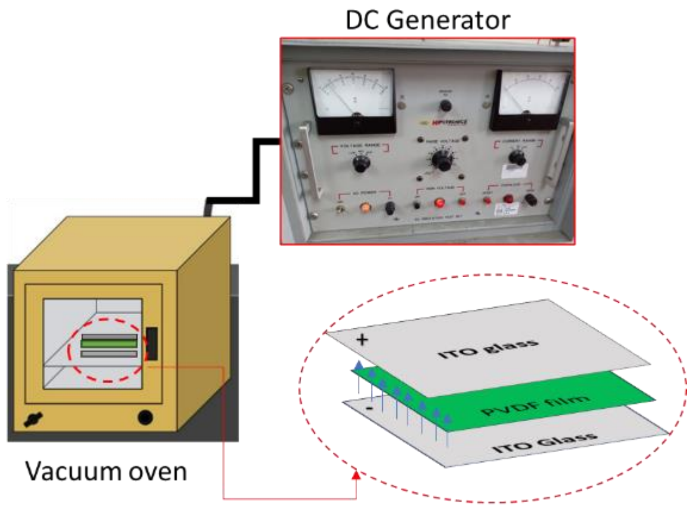

Fig. 2. Thickness poling using ITO glass.

The investigation involved the application of thickness poling utilizing ITO glass poling as a conductor. This was done in a strong electric field of 3 kV/cm for a duration of 15 minutes. The process took place in a vacuum oven at temperatures ranging from 80°C to 120°C. The temperature range of 80°C–120°C is used for the poling process as it includes the Curie temperature of PVDF. This range enables the alignment of molecular dipoles without causing any thermal harm to the material[25, 26].

2.3 AFM Setup

AFM-SPA-400/Nano Navi by Seiko instruments was used to observe PVDF films' morphological and surface roughness at nanoscale levels. To achieve good image resolution, an AFM SPA 400 cantilever with a sharp tip, high displacement and high force sensitivity was utilized in the experiment. The cantilever probes were selected based on the recommendation of the microscope producer (Hitachi-High-Tech). A summary of the technical specifications of cantilever-type SI-DFM40P is shown in Table 1.

Table 1. Summary of technical specification cantilever

AFM Cantilever SI-DF40P	
Resonance frequency (f)	: 285 kHz
Spring constant (C)	: 26 N/m
Shape	: Pyramidal

Fig. 3 illustrates the major components of an AFM setup. AFM with a resolution of 512×512 points and a scan size of 5×5 μm^2 with a frequency of 265.855 kHz is used to observe the surface morphology and roughness of the PVDF film. A micromachined probe positioned at the end of a cantilever is an essential apparatus component. This mode provides high-resolution data such as roughness, pore size distribution, and density [28]. This study used contact mode to characterize and evaluate the surface roughness of PVDF film. In the test, a tip cantilever travels over the sample, maintaining a constant force between the tip and the sample while allowing the z-direction to shift or change. The deflection of laser light reflected on the cantilever is translated into 2D or 3D images in nano-scale measurement. The purpose of observing the appearance of objects in AFM, both in 2D and 3D, is to gain valuable information about the PVDFfilms morphology, such as roughness, height variation of roughness, physical properties, and the presence of surface features. Observing objects' appearance in AFM, both in 2D and 3D, is to gain valuable information about the PVDF films morphology, such as roughness, height variation of roughness, physical properties, and the presence of surface features. In some cases, knowing the height and shape of surface features is not enough if only observe in 2D images. 3D image still needs to extend the capabilities of 2D imaging by providing a 3D representation of the sample's surface to obtain more comprehensive information on the PVDF film topography, including the PVDF film's valley, peak, and shape surface structures.

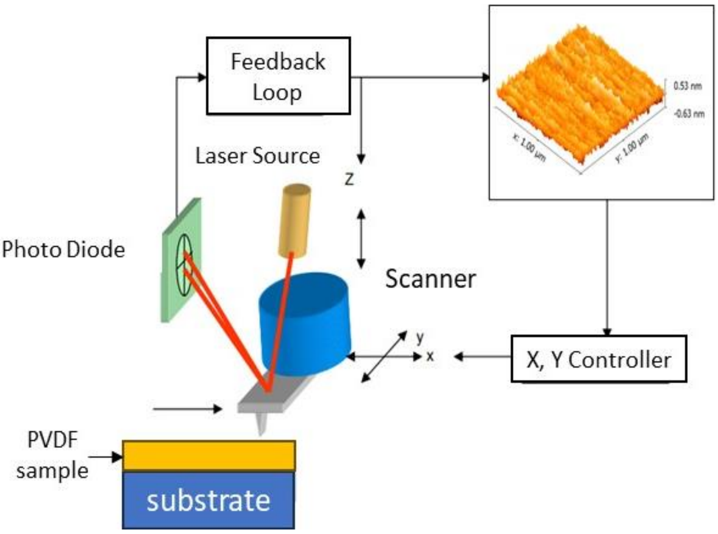

Fig. 3. Setup of AFM operation [27].

3 Results and Discussion

The PVDF films were subjected to stretching at a temperature of 80 degrees Celsius, with a stretching ratio of 5. Subsequently, the films were poled at different temperatures and examined using the AFM technique. The Gwydion software, which utilizes nanoscale analytics, was employed to identify and quantify the alteration in the microstructure of a stretched PVDF sheet. It is important to highlight that numerous surface roughness measurements were selected for analysis. Gwydion program provides an extensive range of tools and capabilities for assessing the surface roughness of PVDF polymer and other materials. This software provides many kinds of tools for measuring and analyzing surface features, such as arithmetic average roughness (R_a), root mean square roughness (R_q), and surface height (R_z).

Eq. 3, 4 and 5 show the formulas for these parameters and some other often derived roughness parameters. Average roughness (R_a) refers to the mean height value computed across the entire surface area under examination. R_a is commonly employed to characterize the surface roughness (of PVDF film and is typically expressed as [29]:

$$R_a = \frac{1}{n}\sum_{i=1}^{n}|y_i| \tag{3}$$

Where *n* is the point quantities on the *x* and *y* axes, respectively, *y* varies from 1 to *n*, representing the surface profile deviation from the mean line.

Height roughness (R_z) signifies the vertical gap encompassing the uppermost and lowest points within the assessed length or area, comprehensively depicting the surface's overall roughness.

$$R_z = \left|\frac{min y_i}{1 \le i \le n}\right| + \left|\frac{max y_i}{1 \le i \le n}\right| \tag{4}$$

wherey is varies from 1 to *n*.

The square root of the surface height distribution is the root mean square roughness (R_q). It is more sensitive than average roughness to substantial deviations from the mean line/plane and is also used to calculate the skew and kurtosis parameters.

$$R_q = \sqrt{\frac{1}{n}\sum_{i=1}^{n}|y_i^2|} \tag{5}$$

wherey is varies from 1 to *n*.

In statistical analysis, skewness and kurtosis are frequently used in together to depict the shape ofdata distribution and evaluate its adherence to the normal distribution assumption in various statistical analyses. These two measures, skewness, and kurtosis, are standard tools in statistical analysis for describing data distribution and assessing its conformity to the assumption of a normal distribution in specific statistical investigations. The roughness parameter is analyzed using the roughness tool in Gwydion software, as shown in Fig. 4.

The comprehensive roughness parameters of all PVDF samples are outlined in Table 2. The data shown in Table 2 unambiguously demonstrates that the surface skewness of the annealed PVDF film is favorably inclined, signifying an asymmetrical characteristic of the surface that encompasses a greater number of peaks than valleys [30]. Kurtosis quantifies the degree of susceptibility of the data to outliers. A higher kurtosis number suggests that the data is concentrated towards the tails, indicating the presence of extreme values. Conversely, a lower kurtosis value suggests a more uniform distribution of data. The observations indicated that the kurtosis moment was compared to another sample at various poling temperatures. The kurtosis value obtained from the test results (Table 2) indicates that it is nearly equal to 3 at a temperature of 80°C. These findings suggest that the number of peaks in Fig.5 is greater than the number of valleys detected in the 3D and 2D images. Based on the collected samples, it is evident that the kurtosis value is consistently below three, which indicates a flat surface.

Table 2. Comparison roughness of stretched PVDF film with various poling temperature

Parameter	Poling temperature (°C)				
	80	90	100	110	120
Scanning area in μm	512×256	512×256	512×256	512×256	512×256
Root mean square (R_{rms}) in nm	19.875	47.17	28.89	32.14	14.18
Average roughness (R_{rms}) in nm	711.4	728.5	735.5	713.0	1.320
Arithmetic average height (Z) in nm	104.3	251.4	218.5	283.5	0.100
Surface skewness	0.6299	-0.2140	0.1012	0.2279	0.07888
Coefficient of kurtosis	2.924	0.03927	2.085	1.040	0.1998

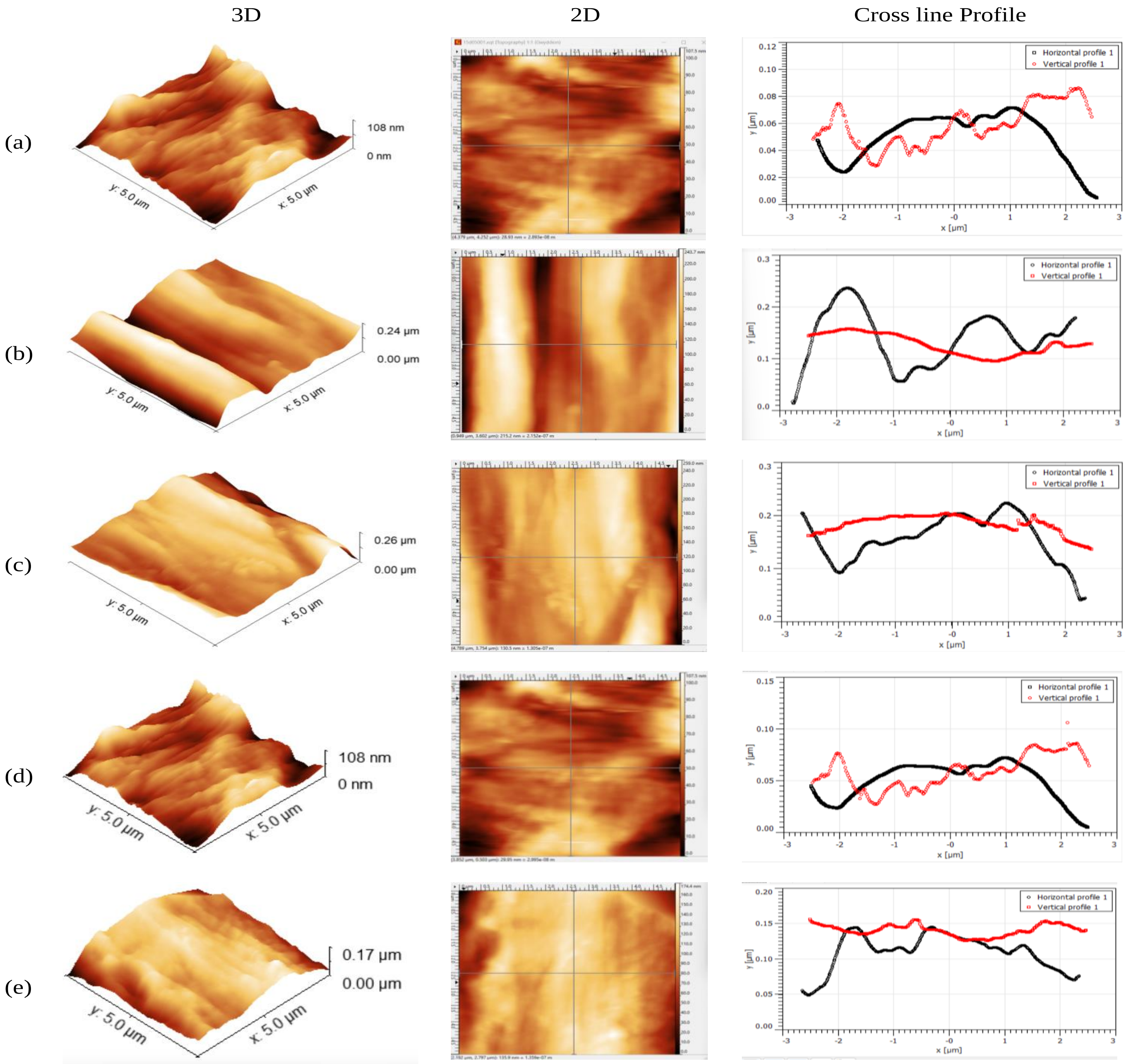

Fig. 4. AFM image topography of stretched PVDF film at (a) 80ºC, (b) 90ºC, (c)100ºC, (d)110ºC and (e)120ºC.

Variations in the kurtosis coefficients for all PVDF films in different poling temperatures suggest that a change in functional groups will alter surface chemistry and surface structure, consequently affecting both films' surface energy.

The dark and bright colors in an AFM image in Fig. 5 represent the surface's specific properties.These colors (dark and bright) are used to represent the topography of the surface, explicitly identifying the heights and depths of its features. Dark color often refers to lower areas or valleys on the PVDF film's surface. These dark regions correspond to areas that are lower or deeper than the surrounding surface. Dark areas indicate that the AFM tip is closer to the sample's character in these spots. In contrast, bright areas indicate weaker interactions and correspond to features higher in elevation or thickness. These contrasts provide valuable information about the topography and surface characteristics of the sample being examined with AFM. Bright color in an AFM image represents higher regions or peaks on the surface of the PVDF film. These light areas indicate relatively higher areas than the surrounding surface. Bright regions suggest that the AFM tip is further away from the sample's character in these areas. This information helps researchers and scientists to analyze the topography and roughness of the surface being studied, such as the PVDF thin film. Under poling in vacuum conditions, the poling temperature can affect the PVDF film's ohmic contact resistance and surface shape. The surface roughness becomes smoother as the poling temperature rises [31].

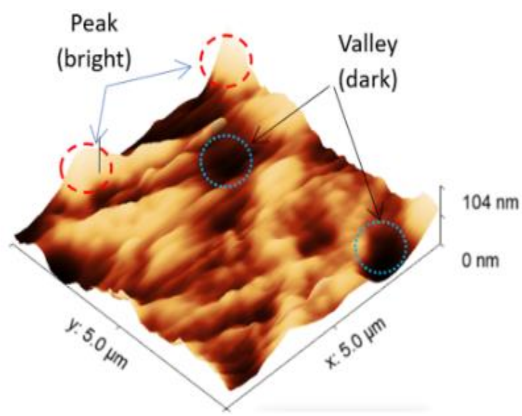

Fig. 5. 3-D topography image of stretched PVDF film with poling temperature of 80ºC.

PVDF's molecular chains can restructure and achieve a more ordered crystalline structure during annealing. The polymer chains are more active at higher temperatures, allowing them to adapt and settle into a more uniform and stable structure [32]. This restructuring minimizes wrinkles and defects, resulting in a smoother finish [33]. Another factor is that thermal stresses can develop within the PVDF film during the initial film formation and cooling, leading to surface deformations and roughness, so annealing allows these internal stresses to relax, minimizing the possibility for surface distortions and promoting a more even surface and smoothness. It is consistent with previous research[34] that generally, PVDF film annealed between Curie and melting temperature will lead to higher chain mobility in the paraelectric phase than in the ferroelectric phase. In addition, the mobility of the chain will increase as the temperature increases.

The chain's increased mobility promotes the lowest energy conformation (all trans), enhancing its ferroelectricity[35]. Previous studies reported [36, 37] that the poling temperature significantly affects the degree of crystallinity in PVDF. The lower poling temperature may result in lower crystallinity due to reduced thermal energy, which inhibits the creation of crystalline areas. Reduced crystallinity can result in decreased piezoelectric properties. In contrast, poling at higher temperatures can result in enhanced crystallinity. The increased thermal energy allows polymer chains to align better and produce crystalline areas.

4 Conclusion

This study highlights the significance of controlling the ITO poling temperature in vacuum conditions for determining the surface roughness of PVDF film. Combining AFM with Gwydion software offers a highly efficient approach to analyze surface roughness properties at the nanoscale. To thoroughly examine the surface topography alterations caused by the annealing treatment in the ITO poling process, one can employ measures such as mean roughness (R_a), root mean square (R_{rms}), and arithmetic mean height (R_z). From the examination of roughness, surface skewness, and coefficient of kurtosis characteristics, it can be deduced that the PVDF film with a low poling temperature has a higher number of peaks than valleys on its surface. An increase in poling temperature leads to a reduction in surface roughness, resulting in a smoother surface.

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