2013 3rd International Conference on Instrumentation, Communications, Information Technology, and Biomedical Engineering (ICICI-BME)
Bandung, November 7-8, 2013

Preparation of PVDF Film Using Deep Coating Method for Biosensor Transducer Applied

Ambran Hartono[1*], Mitra Djamal[2], Suparno Satira[2], Herman[2], Ramli[3]
[1]Department of Physics Faculty of Science and Technology, Islamic State University Syarif Hidayatullah, Jl. Ir.H. Juanda 95 Ciputat Jakarta, Indonesia
[2]Theoretical High Energy Physics and Instrumentation Research Group, Faculty of Mathematics and Natural Sciences, Institut Teknologi Bandung, Jl. Ganesa 10 Bandung 40132, Indonesia
[3]Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, Jl. Prof Hamka, Padang 25131, Indonesia
*ambranhartono@yahoo.com

Abstract

Fabrication of PVDF films has been making using Deep Coating Machine. Preparation of samples carried out for three different solvent concentrations, five of different temperature and three different heating times. This condition is carried out to see the effect of its on piezoelectric properties of PVDF films. The piezoelectric properties like as β fraction are discussion focus in this paper. Piezoelectric properties of PVDF can be improved by solvent concentrations various, temperature and heating time. To obtain the diffraction pattern of sample characterization is performed using X-Ray Diffraction. The β fraction of PVDF films calculate from broadening pattern of X-Ray Diffraction.. Has been obtained an increase piezoelectric properties of PVDF films that characterized by increasing β fraction. Have been obtained β fraction increased 31 %, 39 % and 44.5% for solvent concentrations of 10%, 15% and 20%, respectively. For the heating temperature from 70 ^{0}C up to 110^{0}C have been obtained β fraction 37%, 38%, 44%, 50% and 58% respectively. Furthermore for the heating times 10 minute, 20 minute and 30 minute the β fraction are 34%, 48% and 60%. These results indicate that PVDF film results using deep coating method is good. These indication shown as from the β fraction obtained. From the results of sample characterization is known that a good percentage of solvent was 20%, heating temperature 110^{0}C and long heating time is 30 minutes.

Keywords: Biosensor, Concentration various, Deep Coating, Heating temperature, Heating Time, PVDF, β fraction

1. INTRODUCTION

Lately, technological and industrial development is very rapid. Industry especially the sensor plays a very important, especially in Indonesia. With a growing range of industrial sensors would require a system of planning instruments and material good to be able to support the passage of the sensor manufacturing process and supporting the development of sensor materials. Surely this kind of material development than is needed in the sensor industry is also very necessary in other systems such as transducers, isolators and other systems.

Technological development of PVDF thin film as biosensor materials continues to grow, so does the method of manufacture. Various existing methods such as casting method, calendaring, spreading and hot press produces thin film samples with various advantages and disadvantages in terms of the characteristics of the sample and also the costs.

In this research, the development of an alternative method other than the method described previously. The method was the method of deep coating. This method is very simple and it is possible to obtain films with good enough quality. This is a deep coating method is a simple method of system equipment and how it works. In this method the sample was made with the technique of dyeing a substrate in a solution of PVDF. Motor driving up and down is done by allowing the speed setting that has obtained a sample of homogeneous thickness.

The filming done some experiments with varying concentrations of solvent manufacture, temperature variation and the variation of heating long heating time. This was done to see the trend of PVDF film quality. To characterize the sample was examined by FTIR and XRD.

2. BASIC THEORY

2.1. Polivinylidene Fluoride (PVDF)

Since the discovery of the piezoelectric effect in polymer polyvinylidene fluoride by Kawai in 1969, when it also began investigating the properties of molecules and co-polymers with well [1]. Polivinylidene Fluoride (PVDF) is a specialty plastic material in the family of fluorine polymers, commonly used in applications requiring high purity, strong and resistance to solvents. These polymers are easily formed because it has a relatively low melting point of about 177^0C. Low density and low cost compared with other fluorine polymer [3].

When compared with other fluoropolymer, PVDF melt easier because having a low melting point. PVDF is usually available in powder, pellet, sheet, and wire used for insulators. One advantage of PVDF is more easily established and commonly used in chemistry, semiconductor, medical and electronics industries such as lithium ion batteries [3].

PVDF can be synthesized from VDF monomer gas through a free radical polymerization process, after which it can proceed with the formation of films with pressings or also by other processes such as spin coating and spreading. PVDF is the name given to the structure of the polymer 1.1 difluoroethylene repetition is (CH2 - CF2) while the monomer is CH2 = CF2 (Figure 1) [4].

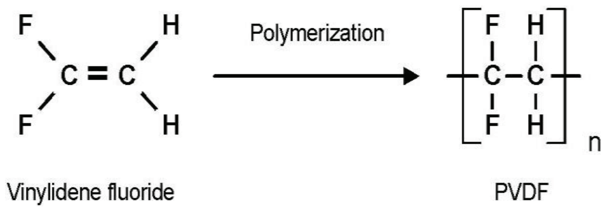


Figure 1 Polymerization process

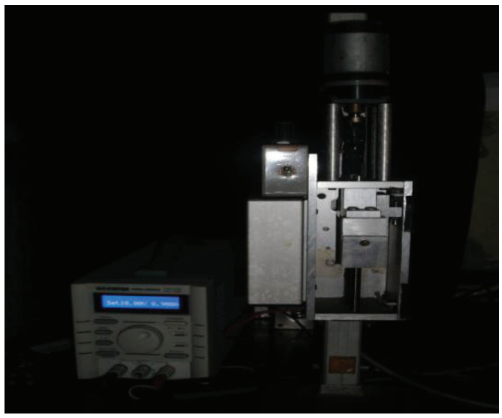
Kawai and colleagues observed the piezoelectric effect in polyvinylidene fluoride and piezoelectric coefficients obtained from the PVDF films for 6-7 PCN-1, 10 times greater than other polymers that have been observed [1].
PVDF solid molecular form is one of the things that greatly affect the piezoelectric effect of the material. This is due to the piezoelectric effect of a material is influenced by the spontaneous polarization of the material, while the spontaneous polarization of a material is influenced by the orientation of the dipole moments of the material that eventually the material orientation of the dipole moment by the form of dense molecular material. Solid form of polyvinylidene fluoride molecules can be grouped into 3 phases form the structure of the beta phase (YYYY), alpha phase (TGTG ') and gamma phase (TTTGTTTG') [4].
Polyvinylidene fluoride that has been developed is because the structure of the beta phase has good piezoelectric properties. PVDF with good piezoelectric properties is further developed as a biosensor material. Biosensor basically consists of three elements, namely the biological element (biological receptor), transducers, electronics and signal processing systems.

To the transducer, which is widely used in an electrochemical biosensor is the transducer, optoelectronic, crystal piezoelectric, field effect transistors and thermostat. Processes that occur in the transducer can be calorimetric biosensors, potentiometric biosensors, amperometric biosensors, optical biosensors and piezoelectric biosensors. The signal coming out of the transducer is then in the process in an electronic system such as recorder or computer [5].

2.2. Deep Coating

Many methods have been developed in the manufacture of thin film PVDF include: methods calendaring [6], coating, spreading [7] and evaporation [8, 9]. In this study tried to develop a fabrication method named Deep Coating. This method is a method of dyeing a substrate with a pull system for PVDF coating solution to be made into a movie. The advantage of this method is the coating process is done by a machine (motor) driving up and down. Up and down motion of the motor is controlled by the speed control system.

This method allows for a deep coating made of thin film samples with a thickness of a homogeneous, while also using this method can be expected to obtain thin films which have characteristics quite well. Deep coating device has three main components: supply power systems, motors and speed control. Set of deep coating equipment as shown in Figure 2.



Figuer 2. Deep coating machine

3. EXPERIMENTS

3.1. Used Equipment

Device / equipment used in this study are as follows:

a. Deep coating machine
b. ATR FTIR NZSE
c. magnetic stirrer
d. glass preparations
e. measuring cup
f. Digital analytical balance
g. XRD
h. Plate hot press

3.2. Research procedures

In this research, the steps or stages as following:

a. Prepare equipment and materials
b. Make a solution of PVDF with several concentrations
c. Dissolution process by providing appropriate heating
d. Preparing deep coating machine
e. Placing glass preparations (substrate) on the machine deep
f. Placing PVDF solution in the space provided on the machine deep coating..
g. Implement deep coating process
h. drying the sample
i. PVDF films characterization by FTIR and XRD

4. RESULT and DISCUSS

PVDF thin films obtained for three concentrations of solvents, namely 10%, 15% and 20%. Subsequently the samples were heated to the temperature variation of the hot press plates. Result of making the film can be seen in the Figure 3.

Figure 3 PVDF film sample

From Film sample obtained further testing to look at the characteristics of the sample Film sample obtained from further testing to look at the characteristics of the sample.

4.1. FTIR Characterization

PVDF thin films produced from three concentrations of solvent 10%, 15%, and 20% were examined by FTIR alpha Bruckner to see beta fraction of film. IR pattern results of three samples are shown in Figure 4.

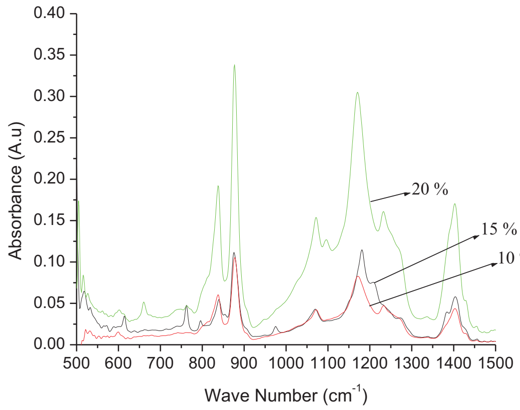


Figure 4 Spectrum IR samples for three variations of the concentration of solvent

4.2. XRD Characterization

After initial testing of IR PVDF film sample is then performed using XRD characterization. The test results of the samples are shown in a graph below: After initial testing of IR PVDF film sample is then performed using XRD characterization. The test results of the samples are shown in figure 5.

Figure 5 shows that for the sample with 20% solvent concentration showed the greatest degree of crystalline, it is shown with peak intensities greater than 10 and 15% concentrations.

Further characterization of the results of XRD and IR and optimization performed on films with a concentration of 20% by hot press for several variations of temperature and do well for some time-varying warming. XRD characterization results of samples with temperature variation are shown in Figure 6. Of Figure 6 shows that the sample with warming 1100C showed better results than the temperature below it.

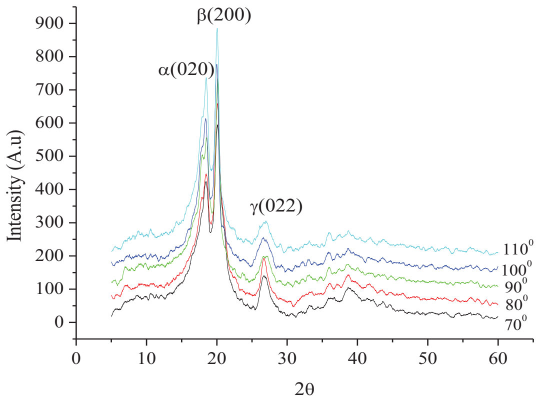


Figure 6 Diffraction pattern of the sample with temperature variations

The next step was the optimization sample to 110^0C temperature variations by performing warm-up time is: 10 minutes, 20 minutes and 30 minutes. Characterization results for these conditions are shown in Figure 7 below:

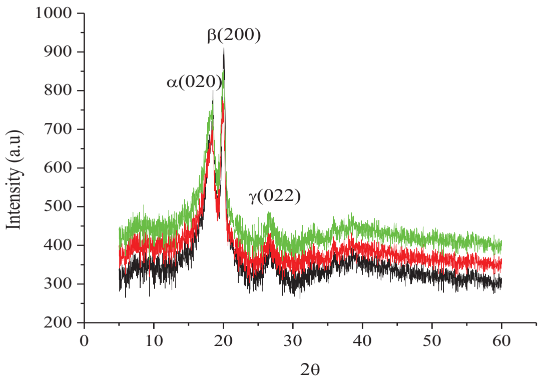


Figure 7 Diffraction pattern of the sample with time-varying warming to temperatures 110^0 C

Characterization of experimental results and calculations performed to determine the beta fraction of the sample using equation 1 [7].

$$F(\beta) = \frac{A_\beta}{1.26A_\alpha + A_\beta} \times 100\% \text{.................................} (1)$$

Beta Fraction calculation for PVDF films with solvent concentration of 10%, 15% and 20% respectively were obtained for 31%, 39% and 44.5%. While the results of the calculations for the beta fraction PVDF films with variations in temperature heating up to temperatures of 70 0C to 110 0C obtained respectively by 37%, 38%, 44%, 50% and 58%. While the calculation of beta for a fraction of the time variation of heating 10 minutes, 20 minutes and 30 minutes respectively obtained by 34%, 48% and 60%. From the calculation of the beta fraction is clearly seen that the greater the concentration of the solvent, the greater the fraction Betha and the greater the heating temperature the greater the beta fraction obtained.

5. CONCLUSION

Of experiments and calculations have been done beta fractions obtained that the fraction of beta optimum solvent concentration obtained for 20% of 44.5%, for heating temperature 1100C in the amount of 58% and for the variation of warming time is 30 minutes with a beta value of the fraction of 60%.
This shows that the greater the concentration of the solvent, the greater the fraction of beta and the greater the heating temperature, the greater the beta fraction obtained. Obviously the result made PVDF films exhibit good piezoelectric properties.

6. Acknowledgements

Our thanks to the **ASAHI GLASS FOUNDATION** on financial support in the implementation of this research.

REFERENCE

[1] H. Kawai, "Piezoelectricity of polyvinylidene fluoride", Jpn. J. Appl. Physics. 8 (1969), pp. 975-976.)
[2] en. Wikipedia. Org/wiki/polyvinylidene_fluoride)
[3] Kawai. H, Jpn. J. Appl. Phys.**8**, 1969, 1975
[4] Lovinger Andrew, Poly-(vinylidene Fluoride), Bell Lab. New Jersey, 1981
[5] Wikipedia.com. Biosensor. Situs Web Wikipedia English
[6] ambran et. al.AMPC Journal, Effect of Mechanical Treatment Temperature on Electrical Properties and Crystallite Size of PVDF Film, (2013), pp
[7] V. Sencadas, V.M. Moreira, S. Lanceros-Mendez, A. S. Pouzada and R. Gregorio Jr. α- to-β Transformation on PVDF Films Obtained by Uniaxial Stretch, *Materials Science Forum* Vols. 514-516 (2006) pp 872-876
[8] Harsha: Principles of Physical Vapor Deposition of Thin Films Ch02 Final Proof page 11 18.11.2005 11:55am Harsha: Principles of Physical Vapor Deposition of Thin Films Ch02 Final Proof page 11 18.11.2005 11:55am
[9] Kwang Min Kim et.al. Effect of Evaporation Temperature on the Crystalline Properties of Solution-Cast Films of Poly(vinylidene fluoride)s, *Korean J. Chem. Eng.*, **20**(5), 934-941 (2003)