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Poly (vinylidene fluoride) Thin Film Prepared by Roll Hot Press

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Abstract: *Various methods have been developed in the manufacture of Poly (vinylidene fluoride) (PVDF) thin films such as coating, spreading, evaporation and calendaring. The method of making this film certainly has many advantages and disadvantages as in the case of operational processes and also the cost of the required treatment. In this paper, an alternative method to preparation of PVDF thin films has been done through the development calendaring method by using roll hot press. The advantages of roll hot press are simple in terms of operation and relative low cost. PVDF thin film has been produced for several temperatures of roll hot press with several different thicknesses. The PVDF thin films are characterized using X-Ray Diffraction and IR spectra. In addition to determine the surface resistivity are caried out using I-V meter. We found that an increase in β fraction of PVDF thin films with increasing temperature at fixed film thickness, while the surface resistivity of PVDF film are decreased. This shown that the piezoelectric property of PVDF films has been improved.*

Keywords - *β fraction, piezoelectric, PVDF film, Roll hot press, Surface resistivity*

I. INTRODUCTION

Poly(Vinylidene Fluoride) (PVDF) polymer has three forms dense molecular structure of the α phase, β phase and γ phase [1]. Preparation of PVDF with β structure recently has been developed, it is because the molecular structure provides the greatest piezoelectric effect compared with other phase [2]. Currently, the development of PVDF thin film technology and its utilization continues to increase. Several methods have been carried out by previous researchers in the PVDF film were: annealing [3], spreading [4], evaporation [5, 6, 7] and calendaring [8].

PVDF is extensively used in smart structure sensors, actuators, and transducers because of its good piezoelectric and pyroelectric properties [9]. Until now, research on PVDF piezoelectric properties is still being developed, both in terms of increasing the piezoelectric material and the analysis of physical parameters related to the amount of beta fraction of the sample as well as in its application to sensors. Piezoelectric properties of PVDF are largely dependent on the content of β phase [10,11,12] and therefore have made efforts to develop the β phase films.

In this paper, we present an alternative method for prepared thin film of PVDF by using Roll Hot Press machine. Roll Hot Press machine is a tool used to flatten PVDF material into a thin film. The flatten process is called calendaring process. This tool consists of two cylinders in which there is heating, speed control for controlling the speed of rotation of the cylinder, thermocouples and temperature sensors that are useful for adjusting the temperature given to the cylinder.

II. EXPERIMENT

In this experiments we performed and characterization of PVDF film using such equipment; Roll Hot Press machine, micrometer screw, I-V meter, FTIR and X-Ray Diffraction aparatus. Profiles of Roll Hot Press machine are shown in Fig.1. The operating system of the roll hot press machine is very simple compared other methods. Initially, the PVDF samples in powder form were placed on the cylinder engine and then heated until it melts furthermore roll hot press machine is operated to obtain a thin film. The PVDF thin film are perform for several different temperatures and various thickness. Film thickness was measured with a micrometer screw, the surface resistivity of film obtained by four point probes, and to determine the crystalline phase is determined from FTIR spectrum and pattern X-Ray Diffraction.

Fig. 1. Roll Hot Press Machine

III. RESULT AND DISCUSSION

From the experimental results obtained some pieces sample of PVDF films with various thicknesses for three different temperatures as listed in Table 1. XRD patern of PVDF film with various film thickness, for roll hot press temperature; 140^0C, 150^0C 160^0C are shown in Fig. 2. As we can see in Fig 2, for fixed temperature the film thickness influence the amount of β fraction, this is caused by the PVDF thin films, will cause the dipole-dipole more properly oriented so that a larger of β fraction . Theoretically, it is known that the film thickness effect on the surface resistivity of the PVDF films where with increasing PVDF thin films, the smaller the resistivity or otherwise. The lower the resistivity indicates that the greater the of β fraction.

Table 1. PVDF film samples produced with some thickness for three different temperatures

No	Roll hot press temperatures (^{0}C)	Film thickness (μm)
1	140	18 13 11
2	150	15 13 8
3	160	16 13 10

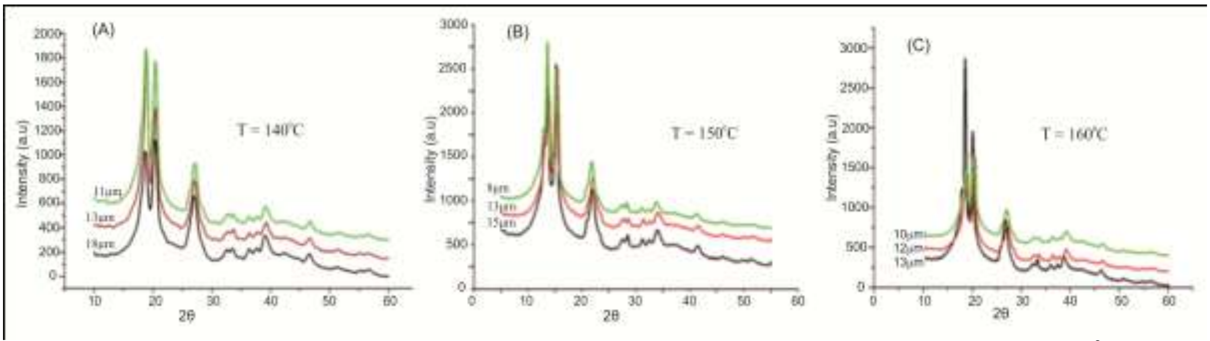

Fig. 2. XRD patern of PVDF film with various thickness for roll hot press temperature: (A). 140^0C, (B). 150^0C and (C). 160^0C.

Effect of temperature at the same thickness is 13 μm to β fraction are shown in Fig. 3 [13]. The amount of β fraction of each sample can be calculate using the equation (1):

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

Obtained that β fraction for the temperature 140^0C, 150^0C and 160^0C are 27.51%, 29.56% and 39.13% respectively. These results show an increase in the β fraction with increasing temperature. Graph of increasing β fraction with increasing temperature is shown in Fig. 4. The increase in temperature causes an increase in polarity due to the dipole orientation resulting in the transformation of the structure occurs. It is characterized by the addition of β fraction [14].

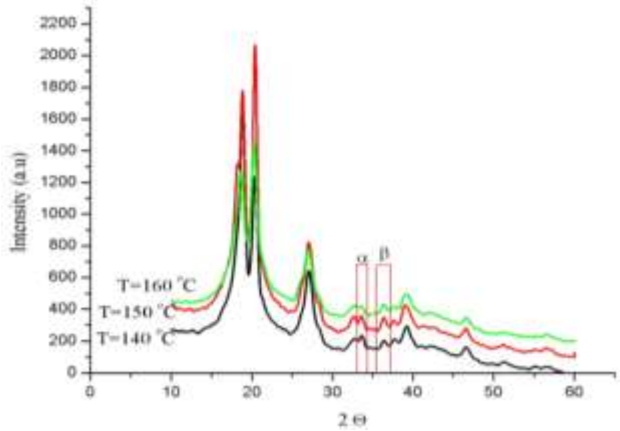

Fig. 3. Diffraction pattern of sample with film thickness of 13 µm, for temperature of 140^{0}C, 150^{0}C and 160^{0}C [13].

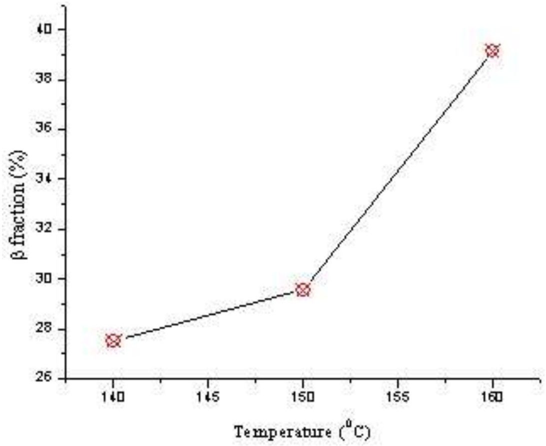

Fig. 4. Graph of increasing β fraction to temperature in PVDF film prepared by roll hot press.

IR spectrum for the PVDF film at a thickness of 13 µm with the variation of temperature 140^{0}C, 150^{0}C and 160^{0}C are shown in Fig. 5. IR spectrum showed an increase of β fraction with increasing temperature, at wave number region of 600 cm^{-1}. These results are consistent with the results of XRD measurements that have been described previously.

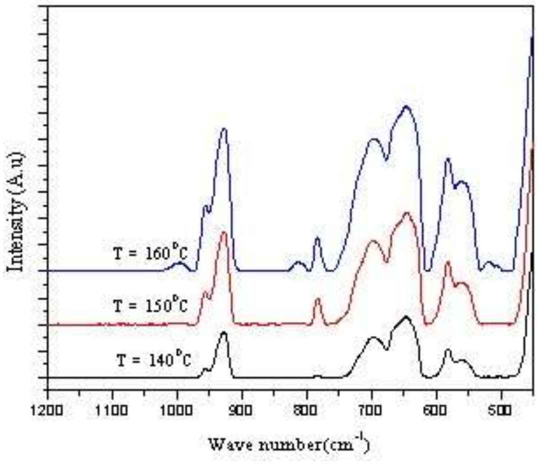

Fig. 5. Graph of IR spectrum for sample at film thickness of 13µm with the variation of temperature 140^{0}C, 150^{0}C and 160^{0}C.

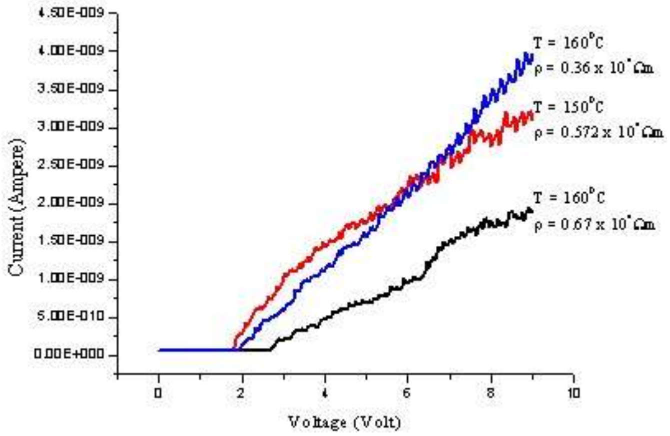

Fig. 6. I-V curves for samples with a thickness of 13 μm with the variation of temperature 140^{0}C, 150^{0}C and 160^{0}C.

Surface resistivity of PVDF film to temperature variation at the same thickness at 13 μ m is shown in Fig. 6. The results of resistivity measurements show that the applied voltage is proportional to the increase in current, in accordance with Ohm's law.

$$R = \frac{V}{I} \tag{2}$$

where *R* is resistance (Ω), *V* is applied voltage (V) and *I* is current (A). The value of *R* can obtained from I-V curve gradient. Resistivity of PVDF film is calculated by equation (3):

$$\rho = 2\pi RS \tag{3}$$

where ρ is resistivity (Ω m) and *S* is distance between the electrodes (5mm). We found that surface resistivity values for temperature 140^{0}C, 150^{0}C and 160^{0}C are 0.67 x 10^{4}Ωm, 0.572 x 10^{4}Ωm and 0.36 x 10^{4}Ωm, respectively. Graph of resistivity versus temperature are shown in Fig. 7.

In Fig.7 seen that the increase of temperature causes a decrease in the surface resistivity value. Decrease in resistivity indicates that the dipole orientation of the PVDF films getting better. This means there has been an increase of β fraction. These results indicate that the properties of piezoelectric PVDF films is improving.

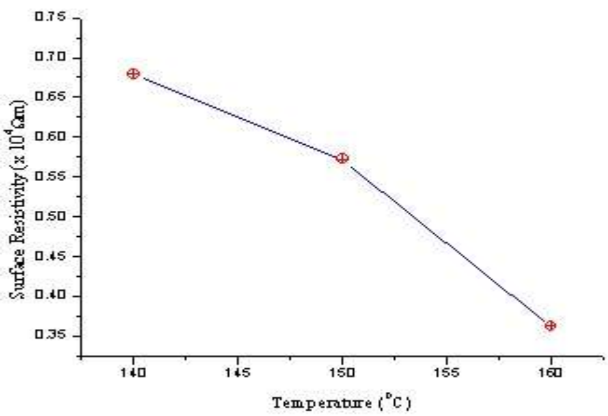

Fig. 7. Graph resistivity as a function of temperature for PVDF film

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IV. CONCLUSION

The PVDF thin films have been successfully prepared by Roll Hot Press machine. The increase in temperature causes an increase in polarity due to the dipole orientation resulting in the transformation of the structure occurs. It is characterized by the increase of β fraction. While the temperature increase causes a decrease in the surface resistivity value, which indicates that the properties of piezoelectric PVDF films is improving.

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