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PVDF-PZT COMPOSITE FILMS FOR STRAIN SENSING APPLICATIONS

Anjana Jain*, P. Siva Subba Rao, Jayanth Kumar .S
CSIR- national Aerospace Laboratories, Bangalore – 560 017
Email: *janjana1@yahoo.com, siva@nal.res.in

Abstract: Piezoelectric polymer-ceramic composites are promising materials for transducer applications. PVDF-PZT composite films with 50% weight fraction of PZT have been developed using solvent cast method. Films are investigated using X-ray diffraction technique, Scanning Electron Microscopy, and Differential Scanning Calorimetry to study crystal structural changes, distribution of particles within the polymer matrix, the changes induced in the melting transition and in the degree of crystallinity of the composites, respectively. The composite films are tested as strain sensor and are compared with conventional strain gauges fixed on CFRP specimens. A load in steps of 1kN was applied at the rate of 0.5 mm / min. It was found that the strain variation was linear to the voltage output from composite PVDF compared to strain values.

Keywords: PVDF-PZT, piezoelectricity, sensors, X-ray diffraction.

1. INTRODUCTION

Studies on piezo-materials have been one of the most dynamic and expanding scientific areas for wide range of applications in the fields such as electrical engineering, electronics, biology and medicine. Among piezoelectric materials, Piezoceramics materials (e.g. PZT) are known for their piezoelectric properties, high dielectric constant and good electromechanical coupling. However, since they are stiff and brittle, monolithic ceramics cannot be bonded on to curved surfaces and thereby limiting their application range. Among piezoelectric polymers, poly (vinylidene fluoride), PVDF is a widely used fluorocarbon polymer with varied range of applications. It is flexible and therefore can easily take the shape of curved surface. In addition, it is chemically inert, creep resistant, and has excellent resistance to sunlight. Unlike PZT, PVDF has good response over a wide frequency range (0.001–109 Hz) [1]. The piezoelectric coefficient values of PVDF, however, are not very high. This problem can be circumvented by using piezopolymer-piezoceramic composite, which derives flexibility from polymeric component and high piezo-properties from Piezoceramics. In the past, piezo-composites have been investigated by various other investigators [2, 3-5]. Different procedures are used to prepare ceramic-polymer composites, e.g. compression molding [6, 7], solvent casting [8, 9] or spin-coating [10]. Compression molding and solvent casting results in films with thicknesses ranging from 30 to 500 µm, while spin-coating usually leads to relatively thin films of only a few µm. If ferroelectric ceramics are embedded in a ferroelectric polymer, the piezo- and pyroelectric transducer properties can be well controlled and adjusted in such a way that either the piezo- or the piezoelectricity is compensated or enhanced due to the parallel or anti-parallel polarization of the ceramic and the polymer dipoles [7, 8]. In this paper, PVDF-PZT piezo-composite films with 50% weight fraction of PZT particles have been developed and characterized for strain sensor applications found superior noise immunity as compared to conventional strain gauges. The comparison was performed only at one frequency, 3Hz by varying the load applied.

2. OPERATION PRINCIPLE AS A STRAIN SENSOR

The composite PVDF films are subjected to high DC electric field at elevated temperature to get permanent polarization. The PVDF-PZT film generates voltage under mechanical deformation. The amplitude and frequency of voltage is directly proportional to the mechanical deformation of the PVDF-PZT [11, 12]. The deformation of the PVDF-PZT by applying mechanical loading causes a change in the surface charge density. The charge q and the voltage generated across the sensor PVDF-PZT electrodes V are related by the capacitance of the sensor C as

$$V = q/C \qquad (1)$$

2.2 2.1 Voltage to Strain Conversion

The PVDF composite films can be treated as parallel plate capacitor, then the capacitance C is given by

$$C = \frac{e^{d}\, l b}{t} \qquad (2)$$

Where, e^d is electrical constant of the PVDF, l is length of PVDF, b is width of the PVDF and t is thickness of the PVDF

Considering the effect of strain only along 1-direction, the voltage generated by the PVDF can be expressed as [13]

$$V = \frac{d_{31}\, Y\, b}{C} \int_{l} \varepsilon \, dx \qquad (3)$$

Assuming the value of ε to be averaged over the gauge length of PVDF and defining the sensitivity parameter S as given as

$$S = d_{31} Y\, l\, b \qquad (4)$$

Where, Y is the Young's modulus of PVDF material. The equation for the strain measurement in terms of voltage generated by sensor, capacitor and sensitivity parameter is given by eq. 5

$$\varepsilon = \frac{V\, C}{S} \qquad (5)$$

The correction factor due to Poisson's ration and shear lag effect is almost unity so it is not considered for measurement of ε in the calculations.

3. EXPERIMENTAL DETAILS

3.1 PVDF-PZT composite films

In the present study, pellets of PVDF have been procured from Pennwalt India Ltd and PZT is obtained from Sparkler Ceramics Pvt Ltd, Pune, India. PVDF-PZT composite films are prepared by solvent cast method. The PZT is dispersed thoroughly in the polymer PVDF solution. The prepared solution is then cast on the glass plate and heated in the oven at about 65°C. When the solvent evaporates completely, PVDF-PZT composite films are obtained. These films are characterized for structural, surface and mechanical properties by X-Ray diffraction (XRD), scanning electron microscopy (SEM), DSC, and tensile testing techniques. X-ray diffraction pattern is taken using the diffractometer D/max Ultima 2200 from Rigaku International Corporation, Japan. Cukα radiations with Graphite monochromator and scintillation counter were used. Scanning electron micrographs is recorded using a Jeol scanning electron microscope to evaluate composite microstructure and dispersion of ceramic powder in polymer matrix. All the specimens are coated with a conductive layer of sputtered gold. Differential scanning calorimetric measurements are conducted with a Perkin Elmer DSC-7 to evaluate the degree of crystallinity. In all experiments, the samples are subjected to temperatures ranging from 130-190°C, at a constant heating rate of 10°C/min under nitrogen atmosphere. Tensile properties of the film were also recorded using INSTRON 5500 series upgraded to Instron Load frames 1175. The films are electroded on both sides with silver and poled at high voltage and high temperature. Piezoelectric coefficients have been measured using piezometer PM300.

3.2 Strain measurement setup and procedure

An aluminum beam of dimensions- 350 X 25 X 2 mm was taken as a specimen. a strain gauge (KYOWA KFG-5-120-C1-11) of resistance of 120 Ω, and of gauge factor 2.16 and 5mm gauge length was fixed on the Al beam. On the exact other side of the strain gauge on the specimen, a composite film PVDF of dimensions 15 X 15 mm was fixed. The film edge connections were made by using the flexible circuit connector developed in CSIR- NAL. The properties of composite PVDF film were shown in the Table 1. INSTRON 8000 100kN UTM was used for conducting the test to measure the strain. The specimen was held for loading as shown in figure 1 and aligned properly using hydraulic grips. Initially, the static test was done on the specimen to understand the strain and PVDF voltage pattern. The static load was applied on the specimen in the position control mode, up to 5kN & 10kN, in the steps of 1kN with the rate of 0.5mm/min. After the static test, the cyclic loads of 0.5KN to 5kN with frequencies (sine wave) of 1, 3, 5, 10, 15 and 20Hz was applied on the specimen. System5000 (Measurements Group Inc.) was used to record strain data at data sampling rate of 10 samples per second. Constant frequency (3 Hz) with variable load was applied to compare the stain and the PVDF output voltage as shown in the Table.2 The PVDF voltage was recorded using Tektronix digital storage oscilloscope model No TDS2024C.

Figure 1: Mounting of the specimen on Instron machine.

Table 1 PVDF Composite film properties

PVDF – PZT properties	Values
Young' modulus (E) GPa	1.5-2.0
Piezoelectric properties (d_{13} d_{32}, d_{33}) pC/N	2.2×10^{-11} , 0.3×10^{-11} , $-0..3 \times 10^{-11}$
Relative permittivity	12
Thickness (t) mm	0.205
Length (l) mm	42.5
Width (b) mm	16
Capacitance pF	290-310

Table 2 Strain and voltage generated - PVDF-PZT at 3Hz

Load @3Hz	Strain	Voltage (Pk to Pk)
2kN	533 $\mu\varepsilon$	0.05 V
5kN	1378$\mu\varepsilon$	0.12 V
8kN	2187 $\mu\varepsilon$	0.20 V
10kN	2786 $\mu\varepsilon$	0.26 V

4. RESULTS AND DISCUSSIONS

4.1 X-Ray Diffraction (XRD)

Figure2 presents the PVDF-PZT composite film prepared by solvent cast method. The PVDF crystalline phases are the non-polar α-phase and the polar β-phase. The PVDF-PZT composite film, however, is in all Trans TTTT conformation and is in polar form.

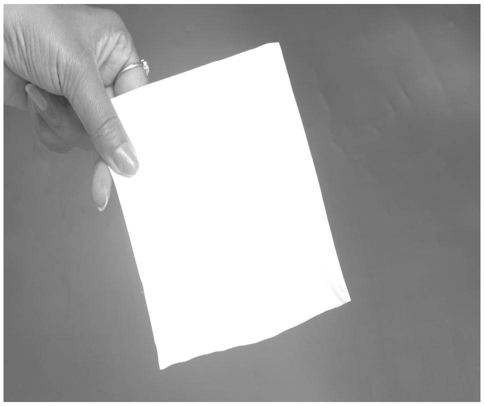

Figure 2: PVDF-PZT composite film.

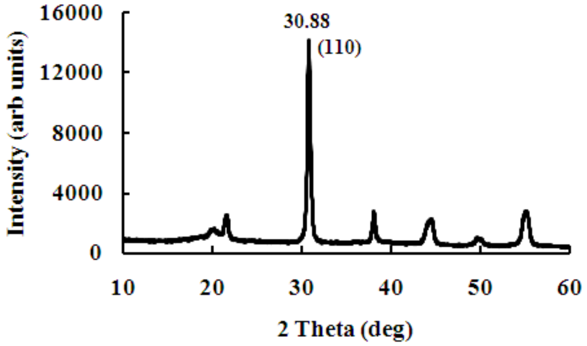

Figure 3: XRD pattern of PVDF-PZT composite film.

The film is characterized using X-ray diffraction technique. Figure 3 presents the X-ray pattern of the PVDF-PZT composite film. It is clear from the patterns that most intense reflection (110) occurs at 2θ~30.88°, which is very close to the value mentioned in literature [14] for PVDF-PZT composite.

4.2 Scanning Electron Microscopy (SEM)

SEM was performed in order to evaluate the composites microstructure and the dispersion of the ceramic powder within the polymer matrix. Figure 4 shows the SEM photograph of PVDF-PZT composite film. Addition of PZT powder leads to uniform distribution of PZT particles in the PVDF solution. As density of PZT is more than PVDF, the PZT particles tend to settle down. At same magnification, top surface (figure 4a) shows less concentration of PZT particles as compared to bottom surface (figure 4b). PZT powder starts settling down during the time of solvent evaporation itself. This feature has also been observed by earlier investigators [3,15,16].

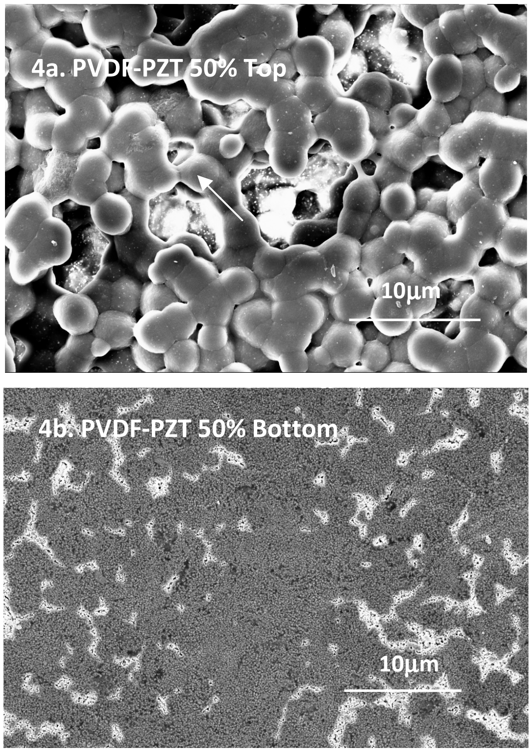

Figure 4: SEM of PVDF-PZT composite film.

4.3 Differential Scanning Calorimetry

Figure 5 shows typical DSC traces of PVDF-PZT composite films. The melting peak occurs at 168°C for the PVDF-PZT composite.

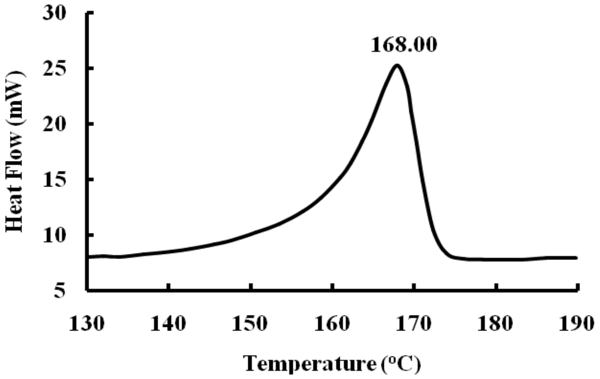

Figure 5: DSC curves of PVDF-PZT composite film

4.4 Tensile Properties

To check the mechanical properties, the films were subjected to standard tensile tests in the INSTRON tensile testing machine. Figure 6 presents the tensile properties of PVDF-PZT composite film. It is found that modulus increases and tensile strength reduces with increase in percentage of PZT. At 50% weight fraction of PZT, Young's modulus was found to be 1574 MPa and tensile strength was 8.5MPa.

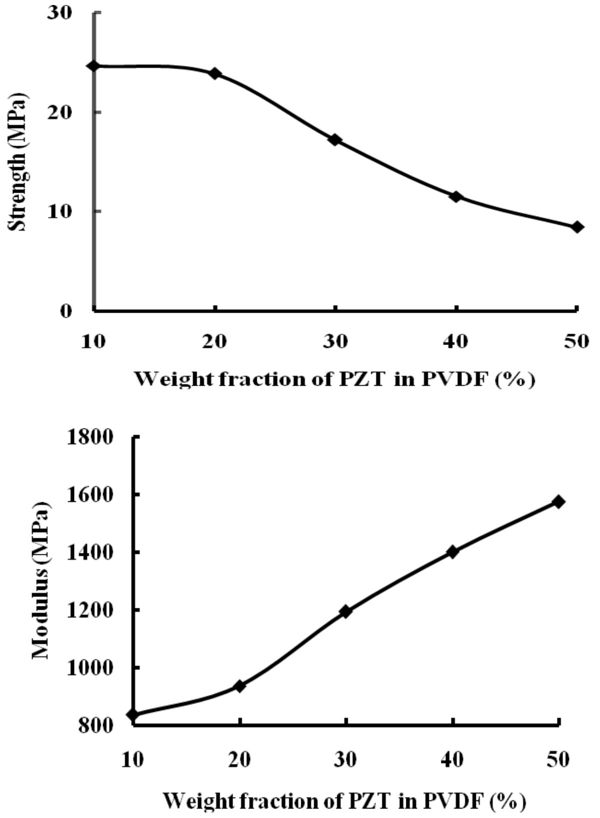

Figure 6: Variation in tensile strength and modulus with weight fraction of PZT in PVDF.

The increase in tensile modulus and degree of crystallinity (discussed earlier) lead to improved piezoelectric properties

[15,17]. Piezoelectric coefficients have been measured using piezometer PM300. The average value of piezoelectric charge coefficient d_{33} obtained is 40pC/N and the maximum value obtained is 100pC/N.

4.5 Strain Measurements

The static and cyclic tests were conducted successfully on the specimen. The strain readings and the voltage generated by composite PVDF film were measured. These were recorded with different data acquisition systems. It is found from the Table.2 that the voltage recorded is proportional to the applied load and comparable to the strain values. The voltage values were converted to equivalent strain values by eq.7 and using the maximum values from the Table 1 and 2. The values of the strain gauge and the converted strain values are plotted with reference to load and are presented in the figure 7. The variation in strain from strain gauge and voltage from PVDF composite film follows the same trend.

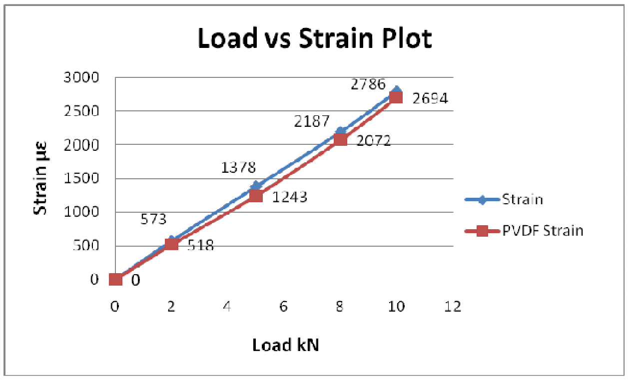

Figure 7: Load vs. Strain measured -strain gauge and PVDF

5. CONCLUSIONS

PVDF-PZT films are prepared using solvent cast method. XRD analysis shows that as prepared films are in β-phase which are in all trans TTTT conformation. Surface features show that PZT is uniformly distributed in PVDF. The DSC scans indicate that the degree of crystallinity increases with PZT weight fraction, and the tensile modulus and strength with 50% weight fraction of PZT are found to be 1574 and 8.5MPa respectively, which matches well with the literature value. The composite PVDF films were tested for strain applications and results were compared with conventional foil strain gauge at a constant frequency 3Hz and variable amplitude loads. It is found that the strain recorded during the test was linear with applied loads and voltage generated from composite PVDF film sensor is proportional to strain. The voltage from PVDF was converted to strain values. It has good correlation with foil strain gauge measurements.

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