AC conductivity and dielectric constant/loss measurement in pristine and swift heavy 100 MeV Ag-ion irradiated poly (vinylidene fluoride) films

D. S. RANA, D. K. CHATURVEDI, J. K. QUAMARA^a

Institute of Instrumentation Engineering, Kurukshetra University, Kurukshetra, India ^aDepartment of Physics, National institute of Technology, Kurukshetra, India

AC conductivity[$\sigma(\omega)$], dielectric constant [$\epsilon'(\omega)$], tangent loss factor (tan $\delta = \epsilon''/\epsilon'$) and dielectric loss [$\epsilon''(\omega)$] have been measured in pristine and 100 MeV Ag-ion (fluence; $1.8X10^{11}$ ions/cm²) irradiated 20 µm Poly (Vinylidene Fluoride) (PVDF) thin films in the temperature region 30° C -170 °C at different frequency ranging from 1kHz -1MHz. Dielectric conductivity [$\sigma(\omega)$] was derived from the dielectric constant and loss tangent data in the frequency range 1kHz-1MHz. The frequency dependent ac conductivity described by Jonscher power law $\sigma(\omega) = A(\omega)^{N}$, has been observed in the low temperature region where N<1 and decreases with increase in temperature for pristine PVDF. The $\epsilon'(\omega)$ of pristine film in low temperature region. Trapped charge carriers make a large contribution to the dielectric parameters at lower frequencies. The variation of ac conductivity for Ag-ion irradiated PVDF thin film with frequency also obeys Jonscher power law except a small deviation in the low frequency region. There is an overall increase in $\epsilon'(\omega)$ in ion irradiated sample as compare to the pristine sample. This shows the dominance of interfacial polarization arising from the large number of radiation induced defect sites and free radicals. Two relaxations; the α_c - and the α_a - relaxations, appearing from high temperature side to low temperature side in the dielectric loss versus temperature spectrum have been observed in present investigation.

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1. Introduction

Dielectric spectroscopy has been turned out to be indispensable tool for investigating molecular motions in polymer usually know as dielectric relaxation process during the application of the electric field. These dielectric relaxation processes not only affect the important physical and chemical properties but also the electrical, electromechanical, and piezoelectric properties of dielectric material such as PVDF. Dielectric property of PVDF is largely governed by strong electric dipoles in the –CH2-CF2- monomer.

The Poly (Vinylidene Fluoride) (PVDF) is a semicrystalline high molecular weight polymer with -CH₂-CF₂-as repeating unit having at least five phases known as α , β , γ , δ and ε [1-3]. Earlier reports have shown that α phase comprises of helical structure with chain conformation- trans-gauche-trans-gauche (TGTG), while β- phase posses all-trans planar zigzag conformation. The PVDF in its β - phase exhibits piezo- and pyroelectric properties. The PVDF polymers have attracted attention in the last few decades because of its unique piezoelectric, pyroelectric, ferroelectric, and nonlinear optical properties, which promote its use in many technological applications in micro sensors and actuators, biomaterials and implantable medical devices, nonlinear optical components, ferroelectric memory and many more [4-9]. It is also one of rare electroactive polymers which have tendered his strong candidature to be the smart material due to its piezoelectric properties. The term smart materials are one who changes their one or more properties in response to external stimuli in controlled manner. The energetic ion irradiation method is relatively new techniques for tailoring the properties of the materials for different technological applications including sensors and actuators.

The energetic heavy ion also termed as swift heavy ion (SHI), irradiation effect on PVDF have attracted much attention during the last few years for its special applications as a biocompatible material with important applications in the pharmaceutical and food industries[10]. The behavior of PVDF irradiated to different types of ions has been studied by several groups [11-16]. These studies reveal change in crystallinity of PVDF [12-16]. The decrease in crystallinity has been reported under electron and low-energy ion irradiation [12-14] whereas an increase in crystallinity has been reported under electron, X-ray and γ -ray irradiations [11, 14-16]. The crystallinity plays a crucial role in determining the piezoelectric, mechanical, optical, electrical and even thermal properties of polymers [12]. The conduction mechanism in ion irradiated polymer has not yet fully understood due to the complexity of the polymer structure. A few reports are available on the ion irradiation effect on various dielectric relaxation behaviors of polymers [17]. These dielectric relaxation processes not only affect the important physical chemical properties but also electric. and electromechanical, and piezoelectric properties of dielectric material such as PVDF. Dielectric property of PVDF is largely governed by strong electric dipoles in the -CH2-CF2- monomer. Dielectric relaxation behavior of PVDF has been investigated by several groups in pristine form [18]. However, swift heavy ion induced effect on AC conductivity and dielectric relaxation behavior of PVDF thin films are not yet explored.

The aim of the present work is to investigate the ac conductivity and dielectric Constant/loss measurements in 20 μ m PVDF thin film before and after the swift heavy 100 MeV Ag-ion irradiation.

2. Experimental details

The poly-vinylidene fluoride used in the present study was procured form DuPont (USA) in film form of thicknesses 20 µm. The samples of size 1 sq. cm were mounted on a ladder for the irradiation in a vacuum chamber. The ladder was loaded in a chamber kept in a high vacuum of the order of 10-6 Torr. The films were then irradiated with 100 MeV Ag-ion beam at fluences; 1.8X10¹¹ ions/cm² using the PELLETRON facility at Inter University Accelerator Centre (IUAC), New Delhi. The ion beam fluence was measured by integrating the ion charge on the sample ladder with time. Ion energies were selected in such a manner that they can easily pass through the PVDF films. The depth profiles were estimated using SRIM calculations. The projected range of 100 MeV Agion beams in the PVDF was calculated to be 25.55 µm, using the SRIM-08 code (Ziegler 2008), which is larger than the thickness of PVDF samples.

The electric contacts were made by vacuum evaporation of high quality silver on the both surface of samples. The samples were fitted in a dielectric cell and specially prepared temperature controlled furnace. The furnace was shield against the stray pickups. The dielectric constant/ loss was measured from room temperature 30°C to 170°C with the help of HIOKI 3532-50 LCZ meter and Fluke PM 6306 programmable LCR meter in the frequency range from 1kHz- 1MHz. Dielectric conductivity $\sigma(\omega)$ was derived from the dielectric constant and loss tangent data in the frequency range from 1KHz-1MHz. AC conductivity calculations were carried out based on derived formula.

The dielectric constant $[\epsilon'(\omega)]$, loss factor $[\epsilon''(\omega)]$ and tangent loss factor $(\tan \delta = \epsilon''/\epsilon')$ and ac $\sigma(\omega)$ conductivity of pristine and SHI irradiated PVDF thin films have been investigated in the temperature range 30°C -170 °C and in the frequency range 1kHz-1MHz. The dielectric parameters were evaluated by measuring equivalent capacitance C_p and tangent loss factor tan δ . The real part of the dielectric constant (ϵ') was calculated from

$$C_{p} = \varepsilon' \varepsilon_{o} A/d \qquad (1)$$

where, ε_o is the dielectric constant (permittivity; 8.86 X 10⁻¹⁰ F/cm) for free-space, 'd' (in cm) is the thickness and 'A' (in cm²) is the cross-sectional area of PVDF thin films.

The real part of dielectric conductivity $\sigma(\omega)$ was obtain from the data of the dielectric constant $[\epsilon'(\omega)]$ and tangent loss factor $(\tan \delta = \epsilon''/\epsilon')$ using the relation:

$$\sigma(\omega) = \varepsilon'(\omega) \varepsilon_0 \quad \omega \tan \delta = \omega \varepsilon_0 \varepsilon''(\omega) \tag{2}$$

where, $\omega = 2\pi$ f ('f' being the frequency) is the angular frequency. The dielectric parameters are represented in terms of dielectric constant [$\varepsilon'(\omega)$] and dielectric loss ε'' [(ω)].

3. Results and discussion

3.1 Pristine PVDF

The variation in dielectric constant $[\varepsilon'(\omega)]$ with temperature for 20 µm pristine PVDF thin film at the frequencies; 1kHz, 10 KHz, 100 kHz and 1MHz have been illustrated in Fig. 1. It has been observed from $\varepsilon'(\omega)$ - T characteristics of pristine PVDF thin film that the behavior of variation of $\varepsilon'(\omega)$ with temperature is almost similar for all frequencies range. At low temperature $\varepsilon'(\omega)$ show little dependence on temperature for intermediate frequencies, but at higher temperature it show strong dependence on temperature for all frequencies and increases at higher pace for low frequencies (1kHz and10 KHz). In polymers, the thermal response of the dynamics of molecular motions and Brownian motion of main chain segment results in various relaxation processes. These relaxation process have direct relationship with the frequency and temperature dependence of dielectric parameters such as dielectric constant $[\varepsilon'(\omega)]$, tangent loss factor tan δ [(ω)] and dielectric loss [ϵ "(ω)]. In pristine PVDF, three type of relaxation process namely α , β , γ have been recognized respectively in the descending order of temperature [19]. The α -relaxation process is attributed to the rotation diffusional motion of the molecules from one quasi-stable state to another involving conformational rearrangement of main chain. The β -relaxation is associated with the orientation of the dipoles in the range of local environment where the rearrangement of the main chain is frozen.

The increase in $\varepsilon'(\omega)$ is due to the total polarization of PVDF. A rapid decrease in $\varepsilon'(\omega)$ is also observed after around 140°C for all frequencies. It can be easily observed from the figure 1 that the dielectric constant $[\varepsilon'(\omega)]$ is higher for the lower frequencies and lower for the higher frequencies across the temperature spectrum. This behavior of dielectric constant $[\varepsilon'(\omega)]$ is consistent with a Deby-type dielectric dispersion characterize by a relaxation frequency. The decrease of $\varepsilon'(\omega)$ with increase in frequency may be attributed to the electrical relaxation processes. We observed a well define peak in $\varepsilon'(\omega)$ -T characteristics for 10 kHz frequency at 120°C which could be due to the presence of α_{c} relaxation of phase II PVDF(α - phase) and the sharp decrease in $\varepsilon'(\omega)$ after 140°C. This decrease in $\varepsilon'(\omega)$ can be associated with change in the geometry of the sample (shrinkage) owing to

release of the molecular orientation at start of dynamic melting temperature of the PVDF.

Figs. 2 and 3a show the variation of tangent loss factor tand $[(\omega)]$ and dielectric loss $[\varepsilon''(\omega)]$ respectively, as a function of temperature in the range 30°C -170 °C at four fixed frequencies; 1kHz, 10 kHz, 100 kHz and 1MHz for pristine PVDF. Fig. 3 (b) shows the frequency dependence of dielectric loss $[\varepsilon''(\omega)]$ measurement for pristine PVDF. It has been observed from tan $\delta(\omega)$ - T and $\varepsilon''(\omega)$ - T characteristics that the behavior of variation in $\tan \delta$ (ω) and $\varepsilon''(\omega)$ with temperature is similar for frequencies: 1kHz, 10 kHz, and 100 kHz i.e. both tan $\delta(\omega)$ and $\varepsilon''(\omega)$ increases at higher pace with increase in temperature, but decreases with increase in temperature at the higher frequency of 1MHz. The $\varepsilon''(\omega)$ - T characteristics at 100 kHz show initial decrease of tan\delta (ω) and $\varepsilon''(\omega)$ with increase in temperature in low temperature region and show increase in tan $\delta(\omega)$ and $\varepsilon''(\omega)$ with temperature in higher temperature region. The initial decrease in $\varepsilon''(\omega)$ with temperature at higher frequencies i.e 100kHz and 1 MHz (Fig. 3 (a)) could be the post-peak portion of the α_{a-} relaxation. The loss maxima would occur at still lower temperature. This dielectric absorption is broadly, in the region of α_{a} relaxation of α -phase PVDF.

The higher values of $\tan\delta(\omega)$ and $\varepsilon''(\omega)$ at low frequency is attributed to trapped charge carriers at the crystalline amorphous interface [free charge motion] within the phase II PVDF polymer at higher temperature.

Two loss valley were observed in tan $\delta(\omega)$ - T and $\varepsilon''(\omega)$ - T characteristics at 10 kHz frequency. One is around 70°C arising from the loss of molecular motion in the folds on the surface of crystallites and other at 120 °C which may be related to loss in the α_{c} relaxation associated with the molecular motion of PVDF chain in the crystalline region of α -phase due to orientation of dipoles. This is in conformity with dielectric relaxation observed in the $\varepsilon'(\omega)$ - T characteristics of pristine PVDF.

Fig. 3 (b) shows increasing dielectric losses $[\varepsilon"(\omega)]$ with decrease in frequency for fixed temperature. This low frequency dispersion of $\varepsilon"(\omega)$ is generally correlated to the trapped charge carriers. Ionic impurities have been observed to get trapped at the crystalline-amorphous boundaries, leading to the large losses at low frequencies in pristine PVDF [20]. The initial decrease of tanð (ω) and $\varepsilon"(\omega)$ with increase in temperature at higher frequencies ; 100kHz and 1MHz (Figs. 2 and 3 (a)) could be post portion of the α_a relaxation i.e. the loss peak would occur at still lower temperatures.

It can be concluded from the above discussion of dielectric constant and dielectric loss measurements (Figs. 1, 2 and 3 (a)) that there are signs of presence of two relaxations designated as α_a and α_c -relaxation appearing from low temperature side to high temperature side of dielectric spectra. The α_c -relaxation observed in the present investigation has been attributed to the molecular motions in the crystalline regions of the polymer main chain whereas the α_a -relaxation is associated with the molecular motion in the folds on the surface of crystallites- amorphous interface.



Fig. 1. Variation of dielectric constant (ε') of pristine PVDF thin film with temperature at four fixed frequencies; (a) 1 kHz (b)10 kHz (c) 100 kHz and (d) 1 MHz.



Fig. 2. Variation of tangent loss factor $(tan\delta)$ in pristine PVDF thin film with temperature at four fixed frequencies.



Fig. 3. (a). Variation of dielectric loss (ε ") in pristine PVDF thin film with temperature at four fixed frequencies; (b) Variation of dielectric loss (ε ") of pristine PVDF thin film with frequencies at various fixed

temperature.

Fig. 4 (a) shows the variation of ac conductivity $[\sigma(\omega)]$ as a function of temperature in the range 30°C -170 °C at four fixed frequencies; 1kHz, 10 kHz, 100 kHz and 1MHz of 20 µm pristine PVDF. It is clear from the Fig. 4 that in low temperature region the measured ac conductivity $[(\sigma(\omega))]$ increases with increase in frequency. It has been observed from Fig. 4 that the behavior of variation of $[\sigma(\omega)]$ with temperature is almost similar for frequencies; 1kHz, 10 kHz, and 100 kHz. The ac increases with increase in conductivity $[(\sigma(\omega))]$ temperature for the fixed frequencies; 1kHz, 10 kHz, and 100 kHz while $\sigma(\omega)$ decreases with increase in temperature for frequency 1MHz. It is evident from Fig. 4 (a) that at very high temperature the ac conductivity $[(\sigma(\omega))]$ will saturate and seems to be equal for all frequencies.





Fig. 4. (a) Variation of dielectric conductivity (σ) of pristine PVDF thin film with temperature at four fixed frequencies; (b) Variation of dielectric conductivity (σ) of pristine PVDF thin film with frequency at various fixed temperature.

Fig. 4 (b) show the variation of ac conductivity $[\sigma(\omega)]$ as a function of frequency in the range 1kHz - 1MHz[representing low frequency $\sigma(\omega)$ dispersion as well as high frequency $\sigma(\omega)$ dispersion(inset)] at various fixed temperature. It clear from the Fig. 4 (b) that in low frequency region ac conductivity $[(\sigma(\omega)]$ increases with increase in temperature but $\sigma(\omega)$ decreases with increase in temperature in high frequency region. It is also observed that at room temperature (30°C) the calculated ac conductivity $[\sigma(\omega)]$ shows an increase with increasing frequency. This trend is also observed for higher temperature. The frequency dependent ac conductivity $[\sigma(\omega)]$ in present case may be describe by equation;

$$\sigma(\omega) = A(\omega)^{N} \tag{3}$$

where the exponent 'N' is observed to be less than unity and 'A' is structural parameter which usually constant. This behavior can be explain in term of correlated barrier hopping model (CBH) [21]. This model considers the hopping of carriers between two neighbouring sites over a coulombic barrier separating them. Also, according to this model, the frequency exponent 'N' is temperature dependent and is given by [21]:

$$N = 6 \text{ KT} / \text{E}_{\text{m}} - [\text{KT} \ln (1/\omega\tau_{\text{o}})]$$
(4)

where E_m is maximum height of energy band and τ_o is characteristic relaxation time. It can very easily conclude from this equation (4) that 'N' decreases with increasing temperature at large E_m /KT. This agrees well with obtained result shown in Fig. 4 (b).

3.2 100 MeV Ag-ion irradiated PVDF

Figs. 5, 6 (a) and (b) show the temperature dependence of dielectric parameters (dielectric constant $[\varepsilon'(\omega)]$, loss tangent factor tan δ $[(\omega)]$ and dielectric loss $[\varepsilon''(\omega)]$) measured for 100 MeV Ag-ion (fluence; 1.8X10¹¹ ions/cm2) irradiated 20 µm PVDF thin film. From the $\varepsilon'(\omega)$ - T characteristics (figure 5) of Ag-ion irradiated PVDF sample, we observed an overall increase in $\varepsilon'(\omega)$ in irradiated samples as compared to pristine PVDF samples over the entire temperature region. Unlike pristine samples in ion irradiated samples, the $\varepsilon'(\omega)$ is almost temperature independent in low temperature region 30-40°C for the frequencies; 1kHz, 10 kHz, 100 kHz. Thereafter, $\varepsilon'(\omega)$ increases gradually and show peak around 138°C for frequency of 1kHz. This peak get broaden and shifted to higher temperature side for higher frequencies. A rapid decrease in $\varepsilon'(\omega)$ is also observed after around 140°C for all frequencies.

The main factors which can effect the $\varepsilon'(\omega)$ - T characteristics in irradiated samples are radiation induced cross linking, chain scission, production of free radical and the formation of unsaturated double bonds [22-23]. In addition, the swift heavy ion irradiation may also change crystalline morphology of polymer. Fascinatingly, all radiation effects have their own role to affect $\varepsilon'(\omega)$ in different way. The overall increase in $\varepsilon'(\omega)$ can be associated with the dominance interfacial polarization also known as ionic relaxation in irradiated samples. The ionic relaxation comprises ionic conductivity and space charge relaxation. The increase in $\varepsilon'(\omega)$ at low temperature is mainly contributed by the (i) α_a relaxation process which is mainly dominated in low temperature region and (ii) chain scission of various functional groups. The chain scissioning process accelerate the movement of functional groups which would increase the $\varepsilon'(\omega)$.

In ion irradiated samples owing to the production of large number of defect sites results in significant enhancement in the interfacial polarization and hence α_a relaxation. The presence of α_a relaxation is also manifested in the form of loss maxima in tan $\delta(\omega)$ – T and $\varepsilon''(\omega)$ - T characteristics (figure 6a and 6b) at lower temperature side. This α_a relaxation process arises due to molecular motion in the folds on the surface of crystallite amorphous interface.

The tanb (ω) – T and $\varepsilon''(\omega)$ - T characteristics (figure 6a and 6b) of irradiated sample show dielectric loss maxima correspond to the α_a relaxation process at the temperature; 38°C, 32°C for the frequencies; 100 kHz and 1 MHz respectively and at around 40°C for frequencies; 1 kHz and 100 kHz. The increase in $\varepsilon'(\omega)$ with temperature can be resolve on the fact that at higher temperature there is a significant enhancement in interfacial polarization due to the production of large number of defect sites on irradiation. Further increase in temperature would increase molecular mobility and therefore large numbers of dipole orient themselves in field direction. The peak in $\varepsilon'(\omega)$ - T characteristics at higher temperature may due to the α_c relaxation process. The α_c -relaxation observed in the present investigation has been attributed to the molecular motions in the crystalline regions of the polymer main chain. The presence of α_c relaxation is also manifested in the form of loss maxima in tan $\delta(\omega)$ – T and $\varepsilon''(\omega)$ - T characteristics (Fig. 6 (a) and (b)) at very high temperature.

The presence of irradiation induced ions and free radicals responsible for the increase in tan $\delta(\omega)$ and $\varepsilon''(\omega)$ at higher temperature. The decrease in $\varepsilon'(\omega)$ can be explained on the basis of irradiation induced free radical cross-linking. Heavy ion irradiation induced free radical plays a dual role. On one hand they try to increase the α_c -relaxation which would result in an increase in $\varepsilon'(\omega)$ and on other hand, they may undergo a radiation induced free radical cross linking at higher temperature. The free radical cross linking at higher temperature would not only ceases the orientation of molecular segment but also reduces the population of irradiation induced free radicals results in a decrease in $\varepsilon'(\omega)$ for ion irradiated samples.











Fig. 7 (a) shows the variation of ac conductivity $[\sigma(\omega)]$ as a function of temperature in the range 30°C -170 °C at four fixed frequencies; 1kHz, 10 kHz, 100 kHz and 1MHz for 20 µm 100MeV Ag-ion irradiated pristine PVDF. It is clear from Fig. 7 (a) that in low temperature region the measured ac conductivity $[(\sigma(\omega))]$ increases with increase in frequency. It has been observed from figure 7a that the behavior of variation of $[\sigma(\omega)]$ with temperature is almost similar for frequencies; 1kHz, 100 kHz, and 1MHz i.e., the ac conductivity $[(\sigma(\omega))]$ increases with increase in temperature while $\sigma(\omega)$ first increase then decreases with increase in temperature for frequency 10 KHz at the low and intermediate temperature range respectively. Further the ac conductivity at the frequency 10 KHz shows increase at very high temperature.

Fig. 7 (b) show the variation of ac conductivity $[\sigma(\omega)]$ as a function of frequency in the range 1kHz - 1MHz at various fixed temperature. It clear from the Fig. 7 (b) that in low frequency region below 10 kHz the ac conductivity

 $[(\sigma(\omega))]$ increases with increase in temperature similar trend is obtained at the frequencies above 10kHz. Thus it is clear that $\sigma(\omega)$ increases with both temperature and frequency. This increase in ac conductivity in ion irradiated sample is not difficult to understand. An enhancement in α_c -relaxation due to interfacial polarization and irradiation induced ions and free radical is held responsible for increase in ac conductivity as compared to the pristine sample. The frequency dependent ac conductivity $[\sigma(\omega)]$ in ion irradiated samples may be describe by equation (3) and equation (4), it is worth noting here that inspite of the decrease in 'N' with temperature, it increases for the ion irradiated samples. This may be attributed the constant 'A' in equation (3) which structural dependent.

Thus it can be safely to conclude that the ac conductivity of ion irradiated samples is more larger than that pristine sample.





(D)

Fig. 7. (a) Variation of dielectric conductivity (σ) with temperature at four fixed frequencies (1 kHz, 10 kHz, and 100 kHz and 1 MHz) for 100 MeV Ag-ion irradiated PVDF; (b)Variation of dielectric conductivity (σ) with frequency at various fixed temperature for 100 MeV Ag-ion irradiated PVDF.

4. Conclusions

AC conductivity and dielectric properties such as dielectric constant and dielectric loss in pristine and Agion irradiated PVDF thin films have been investigated in the temperature region 30°C -170 °C at different frequency ranging from 1KHz -1MHz.. Charge carriers trapped at crystalline –amorphous interface make a large contribution to the dielectric parameter of pristine film at low frequencies. Estimated ac conductivity for pristine PVDF can described by Jonscher power law $\sigma(\omega) = A (\omega)^N$, where N<1 and decreases with increase in temperature.

The estimated ac conductivity for Ag-ion irradiated PVDF can be described by Jonscher power law $\sigma(\omega) = A(\omega)^N$. The effectively increase in $\sigma(\omega)$ with temperature and frequencies is strongly dependent on structural parameter 'A'. An overall increase in $\varepsilon'(\omega)$ in Ag- ion irradiated sample as compared to the pristine sample has been described by dominance of interfacial polarization arising from the large number of radiation induced defect sites and free radicals. Two relaxations; the α_c - and the α_a -relaxations in the form of loss peaks in $\varepsilon''(\omega)$ - T characteristics have shown their presence in ion irradiated sample, appearing from high temperature side to low temperature side.

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*Corresponding author: dineshsrana24@rediffmail.com dineshsrana@yahoo.com