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# Improved Dielectric Properties Achieved by Blending Polypropylene and Poly(vinylidene fluoride)

Benhui FAN<sup>1</sup>, Delong HE<sup>1\*</sup>, Yu LIU<sup>1</sup>, Mingyu ZHOU<sup>2</sup>, Chong ZHANG<sup>3</sup> and Jinbo BAI<sup>1\*</sup>

<sup>1</sup>Laboratoire Mécanique des Sols, Structures et Matériaux (MSSMat), CNRS UMR 8579, CentraleSupélec, Université Paris Saclay, 8-10 Rue, Joliot-Curie, 91190, Gif-sur-Yvette, France

<sup>2</sup>Global Energy Interconnection Research Institute Europe GmbH, Kantstr.162, 10623, Berlin, Germany

<sup>3</sup>State Key Laboratory of Advanced Transmission Technology, Global Energy Interconnection Research Institute, Beijing 102211, People's Republic of China

Corresponding authors: Jinbo BAI, jinbo.bai@centralesupelec.fr

Delong HE, delong.he@centralesupelec.fr

## Abstract

Polymer films composed of polypropylene (PP) and poly(vinylidene fluoride) (PVDF) have been fabricated by extrusion with filming system. Due to low content of crystallites and the lubricant of carbon black, the commercial product PVDF 370 can be compatible with PP by simply blending at molten state and the obtained film has improved dielectric constant ( $\epsilon'=4.5$ ) and reduced dielectric loss ( $\tan \delta=0.5\%$ ) at 1000 Hz. The modified dielectric properties result from the high dielectric constant of PVDF and low dielectric loss of PP. In addition, by introducing the third polymer, low-density polyethylene (LDPE), the dielectric constant of the ternary blends (PP/PVDF 370/LDPE) can further be increased.

## 1. Introduction

The application of film capacitors has considerable potentials in the modern high voltage direct current systems (HVDC) which is one of the main strategies in the future for a long distance electricity transmission (over 1500 km) because of its high efficiency and low energy loss.<sup>1, 2</sup> The current state-of-the-art technology uses metallized biaxially oriented polypropylene (BOPP) with the dielectric constant of  $\sim 2.2$  and the dielectric loss of  $\sim 0.2\%$ .<sup>3, 4</sup> But the dielectric constant of BOPP is too low to meet the requirement of HVDC systems in which the dielectric properties are quite strict: extremely low dielectric loss ( $\tan \delta$  of  $\sim 0.5\%$  at 1000 Hz) and improved dielectric constant ( $\epsilon'$  of  $\sim 4$  at 1000 Hz).

One of the common methods for improving dielectric properties of PP based dielectric

materials is to add inorganic fillers with high dielectric constants, such as  $\text{TiO}_2$ ,<sup>5</sup>  $\text{BaTiO}_3$ ,<sup>6</sup>  $\text{Al}_2\text{O}_3$ ,<sup>7</sup> and  $\text{AlN}$ <sup>8</sup> or conductive fillers such as carbon materials.<sup>9, 10</sup> This way usually induces some problems in the practical industry manufacturing because of the less efficient dispersion of fillers in the polymers. Then the formation of aggregations will block the die of the extrusion especially the filter inside which causes the interruption of the manufacture. Hence, using different kinds of polymer blends presents attractive advantages for the beltlines.<sup>11-14</sup>

Most of the polymers are with low dielectric constant (less than 3.5) except poly(vinylidene fluoride) (PVDF) and its copolymers whose dielectric constant is usually over 9.<sup>15</sup> Unfortunately, the high dielectric loss ( $> 2\%$ ) greatly limits the further application of PVDF in the film capacitors. Moreover, PP and PVDF are

generally immiscible which makes their binary blends are quite hard to process into films with reduced thickness. In this work, we have found that a commercial product, PVDF 370 Arkema, can achieve a better compatibility with PP and consequently the binary blends can be stretched into thin film from the extrusion equipment directly. Meanwhile, PP/PVDF 370 samples are with improved dielectric constant of 4.5 and reduced loss of 0.5% at 1000 Hz which can meet the dielectric requirement of HVDC systems. In addition, it is found that by adding low-density polyethylene (LDPE) for adjusting the processing viscosity, the dielectric properties of the ternary blends can be further improved.

## 2. Materials and experiments

PP was provided from Borealis, Belgium. PVDF 370 and PVDF 761 were provided from Arkema, France. Polymethylmethacrylate (PMMA) was from LGPMMA Corporation, Korea. Polystyrene (PS), polypropylene grafted maleic (PP-g-MA) and LDPE were purchased from Sigma Aldrich.

All the polymers were stored in the oven at 80 °C overnight before using in the experiments. Polymer binary blends and ternary blends were prepared by the co-rotating, conical, two-screw micro-extruder/compounder (Micro 5 cc Twin Screw Compounder, DSM) at 230 °C for 20 min with the speed of 60 rpm. If without specially reminding, the blending order of polymers was that PP was firstly put into the compounder, afterwards PVDF and finally the third polymer. The obtained film was stretched by the filming system directly (Xplore) at room temperature.

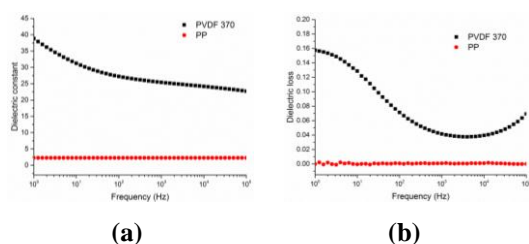
The fracture surface of film samples were characterized by scanning electron microscope (SEM) (LEO Gemini 530). The films were broken after impregnated in liquid nitrogen for 5 min. The fractured surfaces were coated by a thin layer of gold to increase the surface conductivity.

X-ray diffraction (XRD) was measured by D2 Phaser X-ray Powder Diffraction, Bruker with  $2\theta$  from 10 to 30°. Impedance measurement was conducted by Solartron modulab XM at room temperature. The surface area of the sample was 10 mm × 10 mm. The thickness of the sample was measured by thickness gauge way on an iron plate (Surfix Pro, PHYNIX). Before measuring, silver pastes were applied on both faces of the sample for reducing contacting resistances. The tested samples were considered as a plane capacitor and described by parallel resistor-capacitor (RC) circuit systems. The complex dielectric permittivity ( $\epsilon^*$ ) was calculated as follows:  $\epsilon^* = \epsilon' - j\epsilon''$ , where  $\epsilon'$  and  $\epsilon''$  corresponded to the real and imaginary parts of the complex  $\epsilon$ , respectively.  $\omega$  ( $\omega = 2\pi f$ ) was the angular frequency, and  $j = (-1)^{\frac{1}{2}}$ , imaginary unit. The dielectric loss tangent ( $\tan \delta$ ) was defined as  $\tan \delta = \frac{\epsilon''}{\epsilon'}$ .

## 3. Results and discussions

Firstly, frequency dependence of dielectric properties for the neat PP and PVDF 370 are presented in Figure 1 (a) and (b), respectively. It can be found that the dielectric constant of PP is about 2.4 and the dielectric loss is less than 0.3%. The dielectric properties of PP are stable in the whole measuring range. The dielectric constant of PVDF 370 is about 25 from 100 Hz to 10<sup>5</sup> Hz and over 30 at low frequency (1-10 Hz). The dielectric loss is from 0.04 to 0.08 from 100 Hz to 10<sup>5</sup> Hz and over 0.1 at low frequency (1-10 Hz). It can be found that the dielectric constants of PVDF 370 are much higher than that of a commonly used PVDF product, PVDF 761 (761 Kynar Arkema, the dielectric constant is about 10 and the dielectric loss is about 0.02<sup>16</sup>). One of the possible reasons is with the carbon black fillers charged in PVDF 370. Indicated by the dielectric loss plateau at low frequency, the

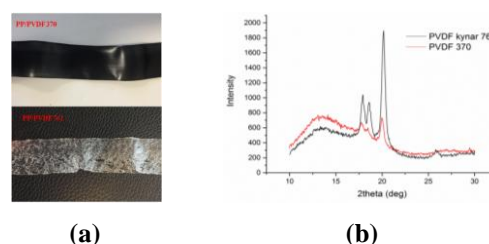
strong interfacial polarization is visibly aroused by the percolated behavior of carbon black in PVDF matrix.<sup>17</sup>



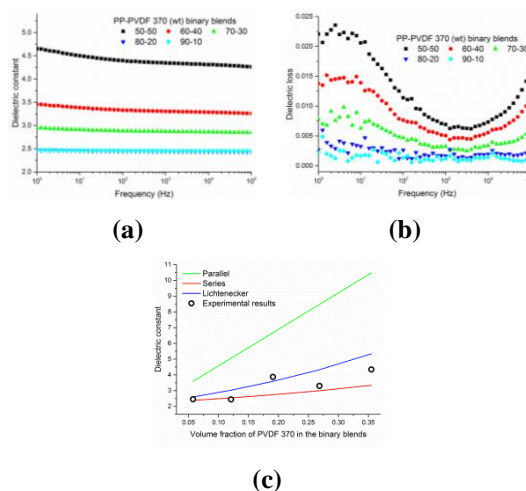
**Fig. 1** Frequency dependence of dielectric properties for PP and PVDF 370: (a) for frequency-dielectric constant and (b) for frequency-dielectric loss.

It is known that the common product PVDF (such as 761 Kynar, Arkema) is not compatible with PP and their binary blends cannot be stretched into film as the below image presented in Figure 2 (a). But PVDF 370 is the exception, it can be compatible with PP and the obtained film is with homogeneous thickness and smooth surface as presented in Figure 2 (a). If comparing to XRD patterns between two kinds of PVDF, it can be found that PVDF 761 has much more crystalline structures than that of PVDF 370 as inferred by higher and sharper peaks in PVDF 761. Considering the stretching process of a polymer from extrusion, the flow induced crystalline behavior usually largely affects the quality of the film. Different semi-crystalline structures and flow rates of crystallites of PP and PVDF will cause the mismatch in the mechanical strength and consequently result in a poor quality of binary blending films. But PVDF 370 has much more amorphous regions than that of PVDF 761 which benefits to better match with PP during the stretching. Meanwhile, if comparing with the rheological parameters between the two PVDF products, the melt flow index of PVDF 370 (230 °C/10min: 5-17) is much higher than that of PVDF 761 (230 °C/10min: 2-6). A higher flow rate of PVDF 370 is easy to stretch with PP

during the filming process and help to improve the quality of the film. Additionally, the existence of carbon black in PVDF 370 products can be viewed as lubricant which favors to reduce the friction between PP and PVDF macromolecular chains during the blending process and consequently to achieve better compatibility. Hence, because of its lower crystallinities and higher melting flow rate, as well as the existence of carbon black, PVDF 370 can be more compatible with PP than PVDF 761.



**Fig. 2** (a) Images for PVDF370/PP and PVDF Kynar 761/PP samples, (b) XRD patterns for PVDF 761 and PVDF 370



**Fig. 3** Frequency dependence of dielectric properties for PP/PVDF 370 films with different weight fractions: (a) for frequency-dielectric constant and (b) for frequency-dielectric loss (c) Experimental results of dielectric constants with different volume fractions of PVDF 370 in PVDF 370/PP binary blends fitted by series and parallels modes and the logarithmic mixing law.

Frequency dependences of dielectric properties

of PP/PVDF 370 binary blends with different weight fractions are presented in Figure 3. It can be found that as the weight fraction of PVDF 370 increasing, the dielectric constant and loss increase simultaneously, especially when the weight fraction of PVDF 370 reaches 50%, the dielectric constant of the binary blends has reached 4.5 and the peak at 5 Hz in the dielectric loss from interfacial polarization is visible in Figure 3 (b). If using series/parallels modes as presented in equation (1) and the Lichtenecker mixing law in equation (2)<sup>15</sup> to fit dielectric constants at 1000 Hz of the binary blends with five volume fractions of PVDF 370 (the density of PP used in the calculation is 0.95, PVDF 370 is 1.84 g/cm<sup>3</sup>, respectively), it can be found that as presented in Figure 3 (c), the experimental results of binary blends are between series and parallels modes, and better fitted by Lichtenecker formula. This phenomenon infers that the dispersion of PVDF 370 is relatively random and homogeneous in PP matrix which is still far away from the percolation threshold.

$$\varepsilon_{\text{eff}}^n = (1 - \varphi)\varepsilon_m^n + \varphi\varepsilon_f^n \quad (1)$$

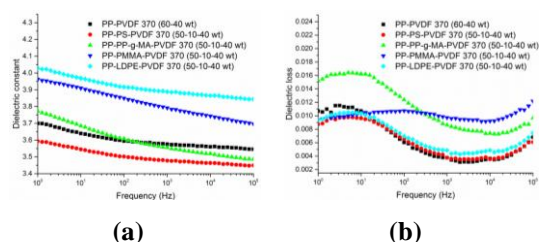
$$\log\varepsilon_{\text{eff}} = (1 - \varphi)\log\varepsilon_m + \varphi\log\varepsilon_f \quad (2)$$

where  $\varphi$  is the volume fraction of PVDF 370,  $\varepsilon_m$  and  $\varepsilon_f$  are the dielectric constants of PP (2.26 at 1000 Hz) and PVDF 370 (25.4 at 1000 Hz), respectively. In equation (1)  $n$  is either “+1” for the parallel case or “-1” for the series case.

Because the dielectric loss of PP is very low, the values of loss for five binary blends samples are always less than 0.025 at low frequency. In order to further improve dielectric properties of the binary blending samples, the third polymer including PP-g-MA, PMMA, PS and LDPE with the weight fraction of 10% has been introduced in the system of PP/PVDF370 (60-40 wt) to study their influences on the dielectric behaviors. The dielectric properties of the ternary blends are presented in Figure 4 (a) and (b). It can be found that introducing the third polymer affects

the dielectric properties of the PP/PVDF 370 blends and the influence depends on the type of the third polymer used. As indicated in Figure 4, the highest dielectric constant of the ternary blends is achieved by adding LDPE. This infers that the combination of LDPE and PVDF 370 may help to improve the compatibility of ternary blending. It is known that LDPE is hard to be miscible with PP or with PVDF if without adding certain copolymers due to the semi-crystalline structure.<sup>18</sup> But PVDF 370 is a product with less crystalline structure as we have discussed. Using this kind of PVDF with more amorphous segments can help to improve the compatibility with PP and LDPE by reducing the crystallinity during the flow from the extrusion. Moreover, LDPE has branching structures which differs from the linear chains. They may modify the aggregated state of PVDF 370 in the ternary blends and consequently promotes the percolation of PVDF 370 in the ternary blends. Thus, a higher dielectric constant will appear comparing with others ternary blends. For the case of PP/PVDF370/PP-g-MA ternary blends, PP-g-MA can be miscible with PP due to the existence of PP grafting structure. Hence, the dispersion of PVDF 370 may not change a lot in the ternary blends which makes the dielectric constant of the sample similar with that of PP/PVDF 370 binary blends. But it can be found that the dielectric loss of the ternary blends PP/PVDF370/PP-g-MA is the highest among all the ternary blends. A possible reason may be that PP-g-MA is a commonly-used plasticizer with low molecular weight. Moreover, PP-g-MA is with polar units ranged on the sides of the main polyolefin chain. The dangling MA side units introduce dipoles which can reorient in response to an alternating electric field. Such a reorientation from the dipoles in a solid is known as orientational polarization which suffers from certain friction with the polymer matrix and causes the relaxation and dielectric loss.<sup>19</sup> Consequently, it can be found that the

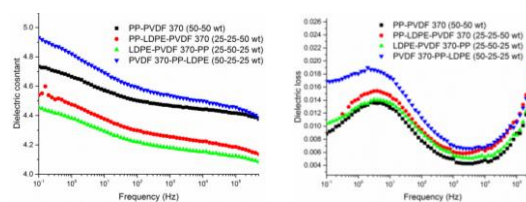
dielectric loss is not only large in low frequency regions aroused by the interfacial polarization, but also large in high frequency regions caused by the orientational polarization. In addition, it can be found that the dielectric constant and loss of ternary blends PP/PVDF 370/PMMA are higher than that of PP/PVDF 370/PS. This variety of the dielectric properties may result from the different polarity between PMMA and PS. In fact, PMMA has higher dielectric constant (4.3 at 1000 Hz)<sup>20</sup> than that of PS (2.65 at 1000 Hz)<sup>21</sup> because the dangling methyl side units have stronger polarity which arouses more orientational polarization. Similarly, the increased orientational polarization unavoidably brings higher dielectric loss as we have mentioned before. Hence, the dielectric constant and loss of PP/PVDF 370/PMMA are higher than that of PP/PVDF 370/PS.



**Fig. 4** Frequency dependence of dielectric properties for PP/PVDF 370 (60-40 wt), PP/PVDF 370/PP-g-MA (50-40-10 wt), PP/PVDF 370/PMMA (50-40-10 wt), PP/PVDF 370/PS (50-40-10 wt) and PP/PVDF 370/LDPE (50-40-10 wt).

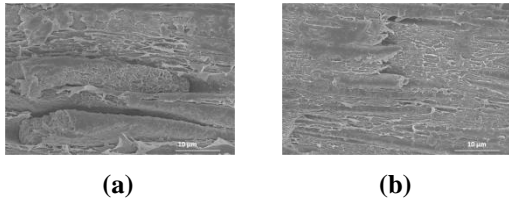
As mentioned before, adding 10 wt % LDPE in the PP/PVDF 370 (60-40 wt) blends can help to improve the dielectric properties. If further modifying the weight fractions of each component, the dielectric properties of the ternary blends will further increase as presented in Figure 5 (a) and (b). Meanwhile, it can be found that the blending order of three polymers will change the dielectric properties of ternary blends. With the same weight fractions, the sample indicated by green line (blending PVDF

370 and LDPE for 5 min at first and then adding PP into PVDF 370/LDPE system for another 15 min) is with the lowest dielectric constant while the sample indicated by blue line (blending PP and PVDF 370 for 5 min at first and then adding LDPE into PP/PVDF 370 for another 15 min) is with the highest dielectric constant. One of the possible reasons may be caused by different processing viscosities of these three polymers which causes various micro-structures in the ternary blends. SEM is used to characterize the fracture surface of PVDF 370/PP/LDPE (50-25-25 wt) (the blue sample in Figure 5) and LDPE/PVDF 370/PP (25-50-25 wt) (the green sample in Figure 5) which are presented in Figure 6 (a) and (b). If comparing with the micro-structures of the two samples with different blending orders, it can be found that mixing PP and PVDF 370 at first will form larger agglomerate of PVDF 370 than that of mixing LDPE and PVDF 370 at first. Since PP has the highest processing viscosity among the three, mixing PP and PVDF 370 at first and then adding LDPE will dilute the viscosity of the mixture by shearing while mixing PVDF 370 and LDPE at first and then adding PP is the process of increasing viscosity. From this study, experimental results infer that blending with a reduced viscosity by mixing PP and PVDF 370 at first and then adding LDPE can help to increase the size of agglomerate of PVDF 370 and reduce the adjacent distances between carbon black fillers in the ternary blends. As a result, stronger interfacial polarizations will be aroused which increase the dielectric constant and loss of the sample. Therefore, the sample of PVDF 370/PP/LDPE (50-25-25 wt) can have the highest dielectric constant among the three.



(a) (b)

**Fig. 5** Frequency dependence of dielectric properties for different adding sequences, (a) for dielectric constant and (b) for dielectric loss; In two plots: the black line: blending PP and PVDF 370 for 20 min; the red line: blending LDPE and PP for 5 min at first and then adding PVDF 370 into LDPE/PP for another 15 min; the green line: blending LDPE and PVDF 370 for 5 min at first and then adding PP into LDPE/PVDF 370 for another 15 min; the blue line: blending PP and PVDF 370 for 5 min at first and then adding LDPE into PP/PVDF 370 for another 15 min



**Fig. 6** SEM images for PVDF 370/PP/LDPE ternary blends with different blending orders: (a) for PVDF 370-PP-LDPE (50-25-25 wt) and (b) for LDPE-PVDF 370-PP (25-50-25 wt).

#### 4. Conclusions

PVDF 370 can be compatible with PP and their binary blends can be fabricated into thin films due to fewer crystalline structures and less processing viscosity of PVDF 370 as well as the lubricant of charged carbon black fillers. The dielectric properties of the obtained binary blends films depend on the weight fractions of PVDF 370. Especially when the weight fraction of PVDF 370 reaches 50%, the dielectric constant and dielectric loss at 1000 Hz are 4.5 and 0.5%, respectively because of the combination of high dielectric constant of PVDF 370 and low dielectric loss of PP. Moreover, by introducing the third polymer, LDPE, the dielectric properties of the ternary blends of PP/PVDF 370/LDPE can be further increased. The samples with the highest dielectric

properties have been achieved by blending PP and PVDF 370 at first and then adding LDPE because this blending order can effectively adjust processing viscosity.

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