Melt Viscoelasticity, Electrical Conductivity, and Crystallization of PVDF/MWCNT Composites: Effect of the Dispersion of MWCNTs

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ABSTRACT: The melt viscoelasticity, electrical conductivity, and crystallization of poly(vinylidene fluoride)/ multiwalled carbon nanotubes (MWCNTs) composites prepared by melt blending with different shear effects were examined. Rheological characterization indicated that the incorporation of MWCNTs substantially affected the viscoelastic behavior of the composites. A visible rheological network of CNTs was detected in the composites with relatively high content of MWCNTs. The electrical conductivity of the composites differed substantially for the composites prepared with different shear effects because of the different dispersion states of the MWCNTs in the composites. The nonisothermal crystallization of the composites revealed that the samples prepared with high shear intensity and long shear time exhibited a higher crystallization peak temperature and crystallinity. Besides, the heterogeneous nucleation effect of the MWCNTs was found to be significantly dependent on the dispersion state, which dominated the crystallization even when the confinement effect of the nanotube network existed. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 125: E49–E57, 2012

Key words: poly(vinylidene fluoride); multiwalled carbon nanotubes; electrical properties; rheological properties; crystallization

INTRODUCTION

Carbon nanotubes (CNTs) are excellent nanofillers in polymer-based composites because of their nanoscale dimension, large shape anisotropy, good heterogeneous nucleation effect, and remarkable electrical conductivity, thermal conductivity, and mechanical properties.[1,2] Coleman et al.[3] summarized the mechanically reinforcing effect of CNT-filled polymer composites, which was evidently reflected by the increment in tensile strength and Young's modulus. They concluded that both the processing methods and the dispersion state of nanoparticles influenced the effectiveness of reinforcement. For instance, when compared with the mechanical properties of neat polypropylene, the incorporation of modified CNTs caused 110 and 113% increment in tensile strength and Young's modulus of the composites, respectively.[4] Similar improvement has been found in CNT-filled polymer blends.[5] It is well known that the improvement of mechanical properties is dependent on the dispersion state of nanofillers, as well as the interaction between nanofillers and polymer matrix.[6] Furthermore, the conductivity can also be substantially enhanced if uniformly dispersed CNTs can form a conductive network. McClory et al.[7] found that the electrical conductivity of poly(methyl methacrylate) composites was enhanced up to 11 orders of magnitude, with the concentration of multiwalled CNTs (MWCNTs) being below 0.5 wt %. The electrical percolation threshold is commonly used to confirm the formation of conductive network of nanoparticles in the matrix. Generally, high conductivity and low percolation threshold correspond to the good dispersion state of the CNTs at the same filler concentration.[8–10] The crystallization behavior of semicrystalline polymers has also been found to be greatly influenced by CNTs even at low concentrations of CNTs.[11,12] These performance improvements of CNT-filled polymer composites are directly determined by the dispersion state of CNTs in the matrix. Because of the intensive van der Waals interaction between nanotubes, CNTs usually tend to aggregate. The

homogeneous dispersion of nanotubes in a given polymer matrix is fundamental and has been the focus of a number of studies in recent years. Therefore, a number of processing methods such as extrusion, ultrasonic radiation, mixing with high shear, and melt spinning have been used or developed to improve the dispersion of CNTs in the matrix.[13] Recently, Grady[14] summarized several effective methods for the preparation of CNT-filled polymer composites. Among the methods available, melt-powder mixing has been significantly used in practice and was adopted in this work.

As a special semicrystalline and polymorphic thermoplastic functional polymer, poly(vinylidene fluoride) (PVDF) is widely used in the chemical, semiconductor, medical, and construction industries. PVDF exhibits five types of crystal modifications, that is, α-, β-, γ-, δ-, and ε-phases, and the first three modifications are frequently encountered.[15] The β-form of PVDF, which exhibits excellent piezoelectric, pyroelectric, and ferroelectric activities, has been studied more extensively[16–18] than the other phases.

The planar zigzag conformation of CNTs can induce the formation of β-form crystals. Therefore, CNT/PVDF composites have been studied extensively,[19–21] and most CNT/PVDF composites are prepared by solution blending because good dispersion of CNTs and β-form PVDF can easily be obtained by this method. However, melt blending, a frequently used method in the processing industry of CNT-based polymer composites, is less toxic and more environmentally friendly. Besides, the application of PVDF matrix composites filled by CNTs in both chemical and electronic industries is of great interest at present because of its high strength and thermostability. Grady[14] pointed out that melt-powder mixing with the help of high shear can greatly improve the dispersion of CNTs in polymer matrix. Li and Shimizu[22] found that the dispersion of unmodified MWCNTs in a polymer matrix greatly depends on the shear stress exerted during melt processing. Kasaliwal et al.[23] found that the dispersion of MWCNTs in polycarbonate depends significantly on both the mixing temperature and rotation speed in a small microcompounder. High rotation speed and mixing temperature resulted in a lower resistivity and a higher dispersion index. In fact, both the shear effect and mixing time are crucial factors for the dispersion of MWCNTs. Villmow et al.[24] studied the effect of screw configuration, residence time, and specific mechanical energy on the dispersion of MWCNTs in polycaprolactone during extrusion in a twin-screw extruder and found that increasing the rotation speed and prolonging the residence time can promote the size and area decrease of MWCNT agglomerates evidently. Kasaliwal et al.[25] proposed that time-dependent erosion and stress-dependent rupture of the agglomerates are the main

mechanisms for the dispersion of MWCNTs in polymer matrix during melt mixing, and the erosion mechanism was dominant when the applied shear stress was not strong enough or when the composites were processed at a low shear rate.[25]

In our previous work, we found that unmodified MWCNTs exhibited a more visible heterogeneous nucleation than carbonylated MWCNTs,[26] and the introduction of chemically modified MWCNTs caused a more complex melt rheological behavior when compared with unmodified MWCNTs because of the interface interactions induced by the van der Waals force and hydrogen bonding.[27] However, the heterogeneous nucleation effect of unmodified MWCNTs relies significantly on their dispersion state. Therefore, an investigation on the effect of melt-mixing conditions, that is, the shear intensity and mixing time, on the dispersion of MWCNTs is comparatively analyzed in this article. Based on the differences in dispersion states of MWCNTs, the rheological properties, electrical conductivity, and crystallization behavior of the resultant composites were comparatively elucidated.

EXPERIMENTAL

Materials and sample preparation

The PVDF powder (FR 901; 3F Co. Ltd., Shanghai) with an average molecular weight $M_w = 286$ kg/mol (polydispersity = 1.9277) was used. MWCNTs with diameters of 10–20 nm and lengths of about 30 μm were purchased from Chengdu Organic Chemistry, Chinese Academy of Science (Chengdu, P.R. China).

PVDF was mixed with 0, 0.5, 1, 2, 3, 4, and 5 wt % of MWCNTs in a 60 ml mixer of a torque rheometer (XSS-300, Shanghai Kechang Rubber Plastics Machinery Set Ltd., Shanghai, P.R. China) at 200°C using two different procedures. The first group of composites was prepared at a rotation speed of 30 r/min and a mixing time of 5 min, and this group was labeled as 1-PVDF/MWCNTs. The other group was first mixed at a rotation speed of 50 r/min and a mixing time of 10 min to obtain the premixture. The premixtures were then melted and remixed for another 5 min at the same rotation speed, and this group was labeled as 2-PVDF/MWCNTs. The composites in the second group were prepared in two steps so that the MWCNTs can be better dispersed, in which the dispersion of MWCNTs would be theoretically better than a continuous melt mixing of 15 min.[28] All the samples were named according to the compositions. For instance, 1-PVDF/0.5% MWCNTs means that the composite contains 0.5 wt % of MWCNTs and was obtained using the first mixing procedure.

Characterizations and measurements

Scanning electron microscopy

The morphology of the PVDF/MWCNT composites was characterized by scanning electron microscopy (SEM; JEOL JSM-5900LV, JEOL PTE Ltd., Tokyo,
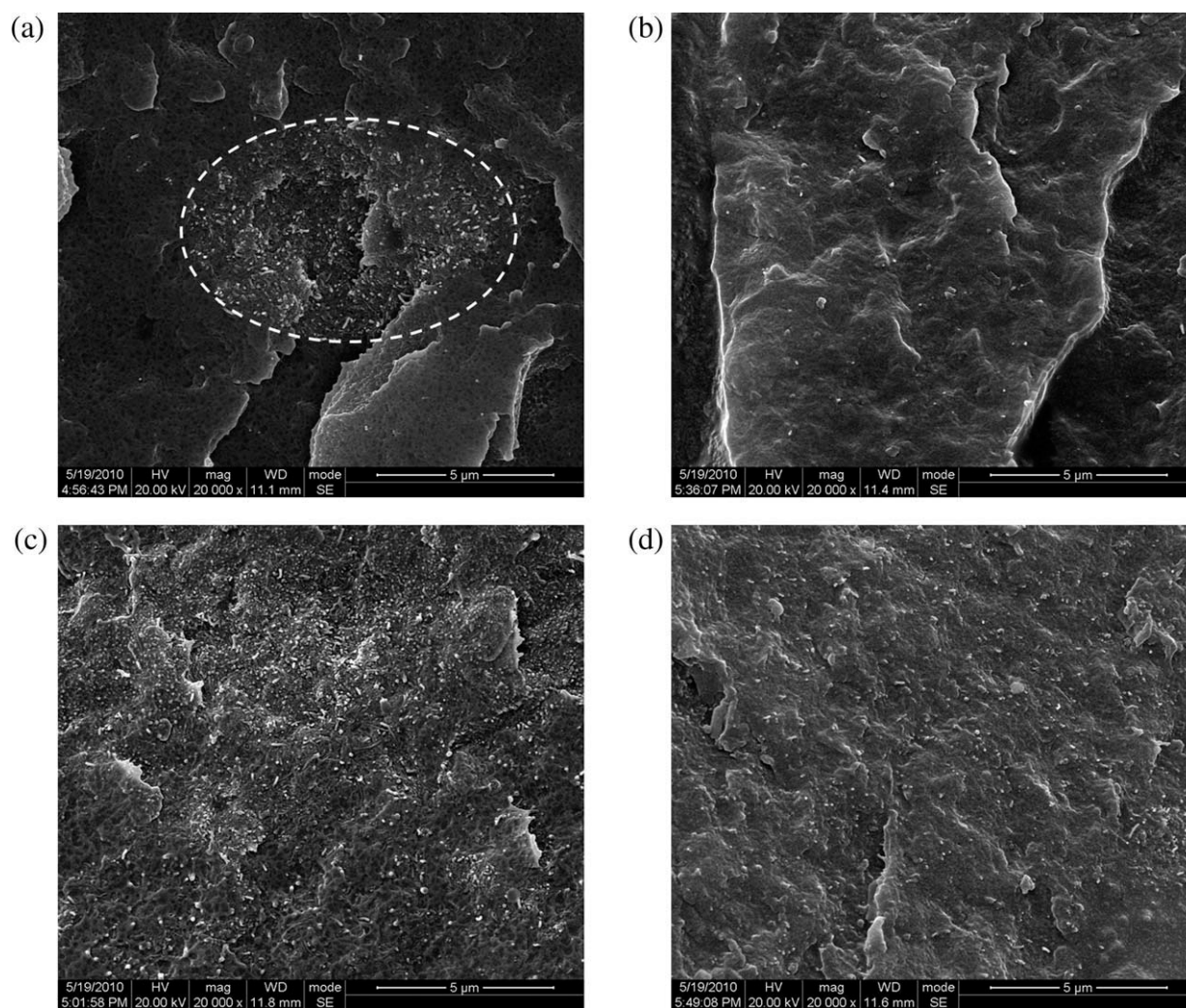
Figure 1 SEM images of the composites: (a) 1-PVDF/0.5% MWCNTs; (b) 2-PVDF/0.5% MWCNTs; (c) 1-PVDF/5% MWCNTs; and (d) 2-PVDF/5% MWCNTs.

Japan) at an accelerating voltage of 20 kV. All the samples were left in liquid nitrogen for 40 min and then impact fractured to obtain a freshly fractured surface for SEM observation. This surface was gold sputtered prior to SEM observation.

Dynamic rheological measurements

The melt viscoelasticity of the PVDF/MWCNT composites was examined using a stress-controlled rheometer (AR2000EX, TA Instruments Inc., New Castle, DE, USA) with parallel plate geometry. The composites were compression molded into disks with a diameter of 25 mm and thickness of 1.5 mm at 8 MPa and 210°C. The frequency sweep was conducted at 200°C with a strain of 1.0% (within the linear viscoelastic region) and a gap of 1200 μm.

Differential scanning calorimeter measurements

The crystallization behavior was studied by differential scanning calorimeter (Q20, TA Instruments Inc.,

New Castle, DE, USA). The samples were heated to 200°C at a rate of 10°C/min and held at 200°C for 5 min to eliminate the thermal history. They were then cooled to 40°C at a rate of 10°C/min and reheated to 200°C at a rate of 10°C/min. The cooling and the second heating curves were recorded to describe the crystallization and melting behaviors of the composites.

Resistance measurements

Electrical conductivity was measured by a Keithley 6517B Electrometer (Keithley Instruments, Inc. Cleveland, Ohio) at room temperature with a voltage of 2 V. All the samples were hot pressed into sheets with dimensions of 17.84 mm × 10.00 mm × 1.12 mm (length × width × thickness). Conductive copper nets and silver binders were used to ensure good contact between the specimen and the electrodes. At least seven samples were used for each measurement, and the average results were reported. It

is noted that the error is less than 10% for the measured resistance of each sample.

RESULTS AND DISCUSSION

Dispersion of MWCNTs in PVDF matrix

The morphology of the PVDF/MWCNT composites prepared by the two different processing procedures is shown in Figure 1. Large MWCNTs aggregates are present in 1-PVDF/0.5% MWCNTs [marked with an oval circle in Fig. 1(a)], whereas a uniform dispersion of MWCNTs is observed in 2-PVDF/0.5% MWCNTs. By increasing the MWCNTs loading, the pristine nanotubes exhibit better dispersion, as shown in Figure 1(c). This is attributed to the better transmission of shear force in the melt with high viscosity during the mixing.[25] However, with an increase in rotation speed and mixing time, that is, with an increase in shear effect, MWCNTs are homogenously distributed in the PVDF matrix as shown in Figure 1(b,d). Therefore, the dispersion of pristine MWCNTs can be greatly improved by increasing the shear effect. This can be attributed to the separation of MWCNTs agglomerates into individual tubes due to the strong shear effect and twice mixing processing.[23–25] Oh et al.[28] found that iterative melt extrusion could promote the disentanglement and dispersion of MWCNTs, resulting in the improvement of conductivity and storage modulus at low frequencies significantly. Therefore, a more homogeneous dispersion of nanotubes can be obtained with stronger shear and twice mixing, which in turn affects the melt viscoelastic properties and electrical conductivity performance in the solid state.

Rheological properties

Generally, the shear viscosity of pure polymer is characterized by two distinct regions: the Newtonian region and the shear thinning region.[29] Figure 2 shows the complex viscosity $|\eta^*|$ curves of the neat PVDF and PVDF/MWCNT composites. The rheological behavior of neat PVDF shows two distinct regions: the Newtonian region at low frequencies ($\omega = 0.01$–0.02 Hz) and the shear thinning region at high frequencies.

The incorporation of MWCNTs has a dramatic effect on the rheological behavior of the PVDF/MWCNT composites. With an increase in MWCNTs loadings, the Newtonian region becomes increasingly weaker and disappears from the 1-PVDF/5% MWCNTs. The complex viscosity increases slightly with an increase in MWCNTs, and the 1-PVDF/5.0% MWCNT composite exhibits a visibly higher value of $|\eta^*|$ than the other samples. Similar rheological behaviors were also reported in the literature.[30]
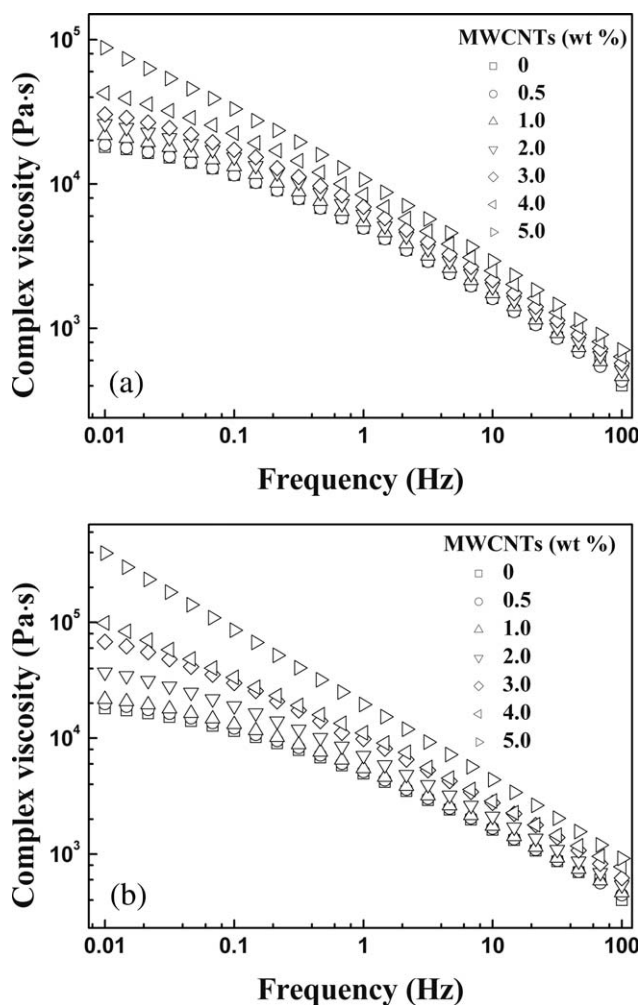

Figure 2 Complex viscosity of 1-PVDF/MWCNT (a) and 2-PVDF/MWCNT composites (b).

The $|\eta^*|$ curves of 2-PVDF/MWCNT composites are similar with those of the 1-PVDF/MWCNT composites, as shown in Figure 2(b). However, an increase in $|\eta^*|$ with an increase in MWCNTs content is more evident, which can be attributed to a more homogeneous dispersion of MWCNTs due to the stronger shear effect.

The storage moduli (G') of the 1-PVDF/MWCNTs and 2-PVDF/MWCNT composites are shown in Figure 3. The introduction of MWCNTs to PVDF leads to an increase in G'. Generally, homopolymer chains can be fully relaxed and exhibit a terminal behavior of $G' \propto \omega^2$ and $G'' \propto \omega^1$ at low frequencies.[31] However, the power for the dependence of G' (G'') on the frequency is usually lower than the theoretical values because of different polydispersity. As shown in Figure 3, neat PVDF melt exhibits approximately a terminal-like behavior in the low-frequency region. However, this terminal-like behavior becomes invisible when the MWCNT content is higher than 2 wt %. As shown in Figure 3 and Table I, the power for G' at low frequencies remarkably decreases as the
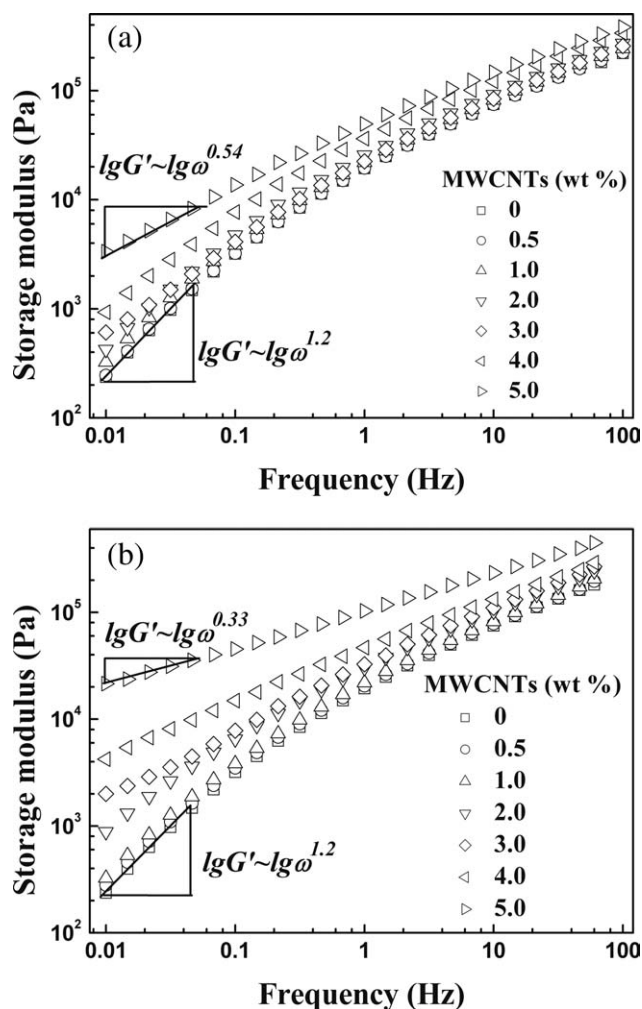
Figure 3 Storage modulus of 1-PVDF/MWCNT composites (a) and 2-PVDF/MWCNT composites (b) with increasing frequency.

MWCNT loading increases. The addition of MWCNTs changes the frequency dependence of the dynamic moduli. The dependence of G' on ω, which becomes increasingly weaker, indicates a transition from a liquid-like to a solid-like viscoelastic behavior.[10] This can be attributed to the formation of a rheological percolation network when the nanotubes

TABLE I
Terminal Slopes of G' and G for PVDF/MWCNT Nanocomposites

1-PVDF/MWCNTs			2-PVDF/MWCNTs		
MWCNTs (wt %)	G'	G''	MWCNTs (wt %)	G'	G''
0	1.20	0.80	0	1.20	0.80
0.5	1.18	0.79	0.5	1.15	0.78
1	1.14	0.78	1	1.11	0.75
2	1.07	0.76	2	0.92	0.69
3	0.81	0.75	3	0.53	0.63
4	0.69	0.70	4	0.46	0.51
5	0.54	0.57	5	0.33	0.34

exceed a critical content, confining the long-range motion of the PVDF chains to a great extent.[32,33] The detailed data for the slopes, that is, power, G', and G'', are listed in Table I. The network in the 2-PVDF/5% MWCNT composites is more compact because it has a smaller power at low frequencies.

Viscoelastic behavior is also considerably related to the dispersion of the MWCNTs in the matrix. As shown in Figure 3, a more visible enhancement of G' at low frequencies can be seen in Figure 3(b) than that in Figure 3(a) when the content of the MWCNTs is higher than 1 wt %, and the 2-PVDF/MWCNT composites exhibit a lower power for G' and G'' with the same loading of MWCNTs. The 2-PVDF/MWCNTs samples achieved a visibly better dispersion of MWCNTs than 1-PVDF/MWCNTs samples, which verifies the SEM observations that a strong shear effect facilitates the dispersion of MWCNTs.

Relative storage modulus, which is the ratio of G' at low frequency of composite to that of neat polymer, is a valuable parameter to describe the rheological percolation of composites.[34] To have a further study on the effect of MWCNTs on the rheological behaviors, the dependence of relative storage modulus on the content of MWCNTs is shown in Figure 4. It is clear that composites of 2-PVDF/MWCNTs exhibit a much more visible increment of relative storage modulus than that of 1-PVDF/MWCNTs, which is ascribed to the better dispersion of MWCNTs in PVDF matrix. Besides, the rheological percolation threshold of 2-PVDF/MWCNTs is below 1 wt %, whereas that of 1-PVDF/MWCNTs, it is between 2 and 3 wt %. It reveals that the dispersion state of MWCNTs affects the rheological percolation threshold significantly.

Electrical conductivity

The electrical conductivity of the composites was examined so that the microstructure in PVDF/MWCNTs can be further understood. The resistance
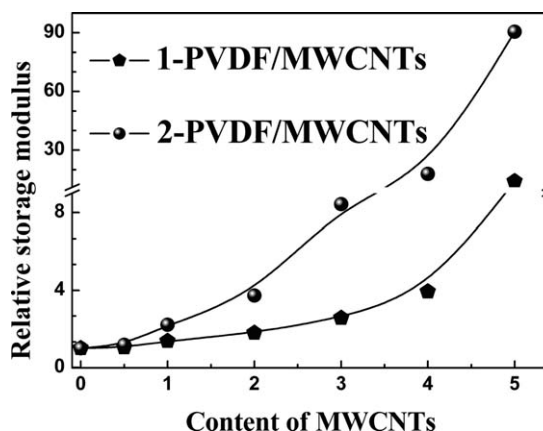

Figure 4 The dependence of relative storage modulus on the content of MWCNTs for PVDF/MWCNT composites.
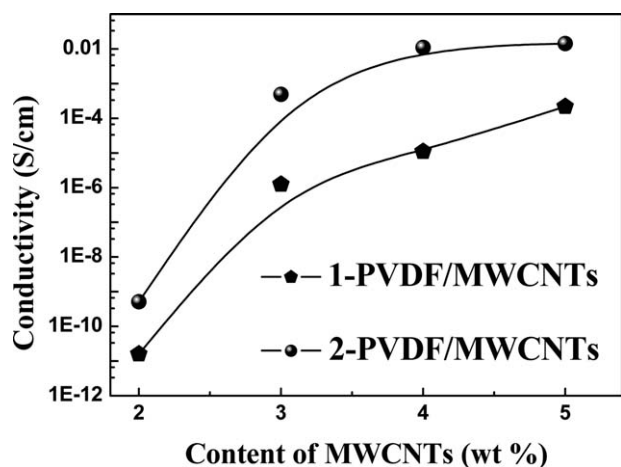
Figure 5 The dependence of electrical conductivity on the content of MWCNTs for PVDF/MWCNT composites.

of the samples was converted into volume resistivity using the following equation[35]:

$$\rho_v = WDR_v/L, \tag{1}$$

where W is the width, D is the thickness, L is the length of the sample, and R_v is the measured resistance. Resistance values higher than 10^{12} Ω were inaccurate and were not used. It should be noted that the resistances of composites containing 0.5 and 1 wt % of MWCNTs are higher than 10^{12} Ω, and thus, they are not recorded. The conductivity of the composites is shown in Figure 5, and it is evident that the conductivity of the 2-PVDF/MWCNT composites is visibly higher than that of 1-PVDF/ MWCNT composites with the same MWCNT loading, indicating a relatively better dispersion of

MWCNTs in the 2-PVDF/MWCNT composites. Besides, it is clear that the percolation threshold of 1-PVDF/MWCNT composites is between 2 and 3 wt % as well. It indicates that the content of MWCNTs also has a significant influence on the electrical conductivity of the composites. If the content of MWCNTs is high enough to form a network by the connection of agglomerates, the composites will be conductive. However, when compared with samples with homogeneously dispersed MWCNTs, a lower conductivity is obtained. Similar result has been reported in Ref. 36. Pan et al. found that the conductivity of MWCNT-filled polymer composites was mainly determined by the formation of conductive network. Homogeneous dispersion of MWCNTs caused by the strong interaction between nanotubes and polymer has little contribution to the enhancement of electrical conductivity but contributes greatly to the mechanical reinforcement.[36] As a result, the fact that the composites have the same content of MWCNTs but exhibit visibly different electrical conductivity reveals that the dispersion state of MWCNTs in 2-PVDF/MWCNTs is assuredly better when it was processed under stronger shear. In addition, the conductivity obtained in this work is visibly higher than that in the work by Martins et al.[30] It is probably that the differences in aspect ratio of MWCNTs and mixing conditions led to the difference.

A conductive nanotube network can be formed only when the loading of the nanotubes reaches the conductivity threshold. Besides, the aggregation can decrease the density of the random connection of nanoparticles when the content of nanoparticles is identical. Thus, for the same loading level of MWCNTs, a compact network of conductive
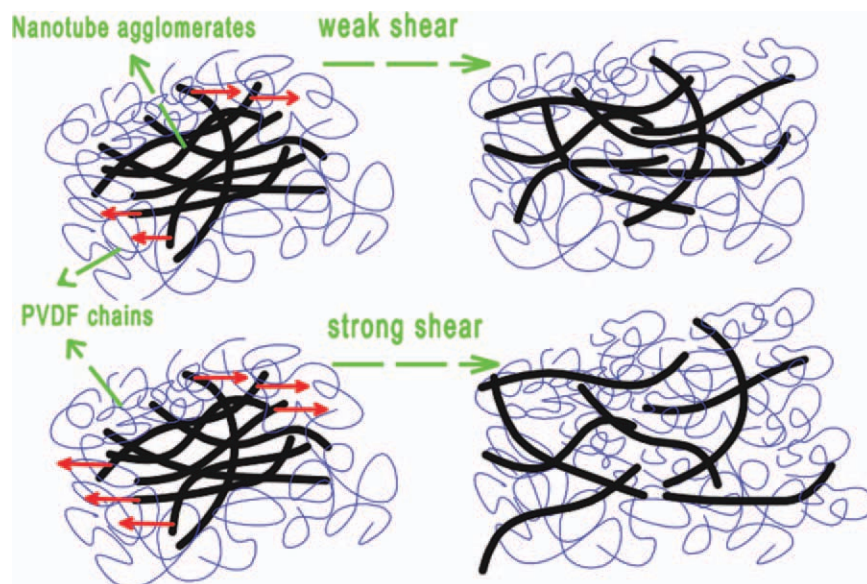

Figure 6 Schematic diagram describing the variation of nanotube agglomerates with different shear effects. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

nanotubes is more likely to form if MWCNTs are well dispersed. As stated in the "Rheological properties" section, a rheological network is detected. Therefore, high shear intensity and long mixing time can facilitate the homogeneous dispersion of the MWCNTs and thus result in the formation of an effective conductive network more easily if they are better dispersed in the PVDF matrix. To better understand this conclusion, a schematic is proposed in Figure 6. As shown in Figure 6, it is visible that the agglomerates of MWCNTs cannot be completely broken when the applied shear is not strong enough. Therefore, they are just partly disentangled. However, they can be well disentangled if the applied stress is strong enough under an intense shear effect. Thus, the dispersion of MWCNTs in the matrix will be better.

Nonisothermal crystallization

Generally, the incorporation of MWCNTs into crystalline polymers can significantly affect their crystallization behavior, which depends on the dispersion and content of MWCNTs. When the content of MWCNTs is low, heterogeneous nucleation effect is dominant, and therefore, the crystallization peak shifts to higher temperature when compared with neat polymer. However, there is a confinement effect of MWCNTs on the crystal growth, and this confinement will be stronger if a network of MWCNTs is formed.[26,37,38] Therefore, we selected some samples to elucidate the influence of MWCNTs on the nonisothermal crystallization of PVDF.

The nonisothermal crystallization behavior of the composites is shown in Figure 5. The crystallinity of the composites is calculated based on eq. (2)[37]:

$$X_c = (\Delta H_m)/\phi(\Delta H_m^0) \times 100\%, \qquad (2)$$

where ΔH_m is the heating enthalpy of the fusion of the first scan, and ϕ is the PVDF content in the PVDF/MWCNT composites. The value of 104.6 J/g is used as the heat of fusion of 100% crystallized PVDF.[39] The related data are summarized in Table I.

The cooling curves of PVDF/MWCNTs are shown in Figure 7, and it is evident that the incorporation of MWCNTs leads to higher crystallization peak temperatures ($T_{cp}s$) for both 1-PVDF/MWCNTs and 2-PVDF/MWCNT composites. The crystallization peak is obviously sharpened, which is indicative of the effective heterogeneous nucleation effect of the MWCNTs and is in accordance with reported results.[25,40,41] To understand comprehensively the influence of shear effect on the crystallization of PVDF composites, the dependence of T_{cp} and X_c on the MWCNTs loadings is shown in Figure 8. As shown in Figure 8(a,b), the composites of 2-PVDF/MWCNTs have higher crystallinities ($X_c s$) and $T_{cp}s$
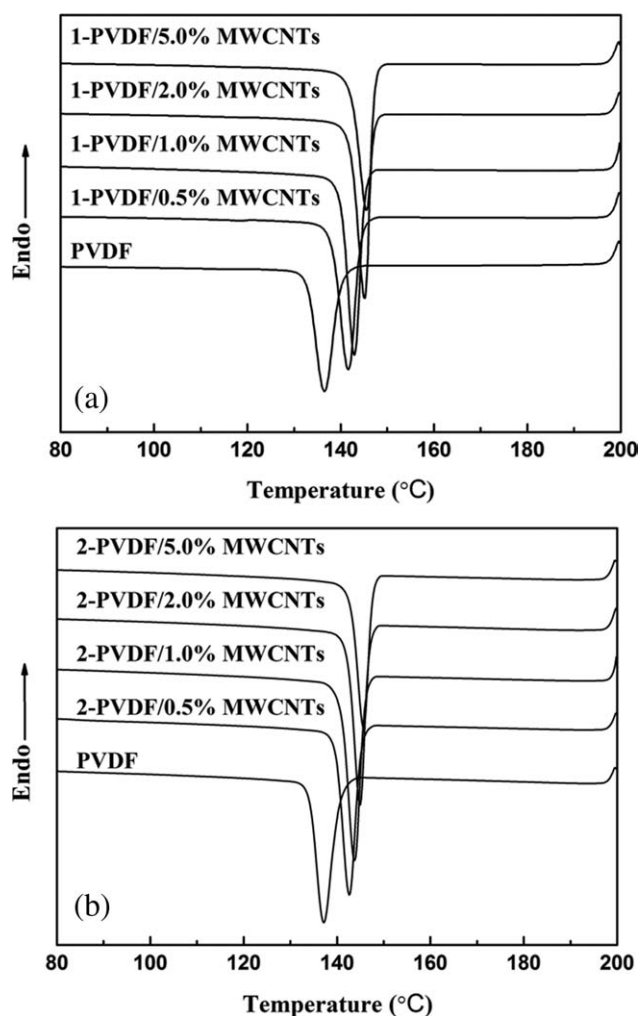

Figure 7 Differential scanning calorimeter scans of PVDF/MWCNT composites during cooling for 1-PVDF/MWCNTs (a) and 2-PVDF/MWCNTs (b).

than their counterparts. This difference can be attributed to the greater number of heterogeneous nucleation sites caused by the more homogeneously dispersed MWCNTs in the 2-PVDF/MWCNTs. It is usually accepted that the better the dispersion of the nanofillers, the higher is the nucleation effect.[12,42,43] However, when compared with 1-PVDF/MWCNTs, the improvement of T_{cp} in 2-PVDF/MWCNTs becomes weaker when the MWCNTs loading exceeds 1 wt % [see Fig. 8(a)]. The formation of a compact network restrains the motion of PVDF chains and influences the crystallization process of the 2-PVDF/MWCNT composites. Obviously, this restraining effect is also related to the dispersion of MWCNTs in PVDF matrix.

As shown in Figure 8(b), the crystallinity of 2-PVDF/MWCNT composites is higher than that of 1-PVDF/MWCNT composites. The crystallinity of the former increases with the increasing MWCNTs content; however, this increase slows down at high loadings, indicating the constraint effect of the
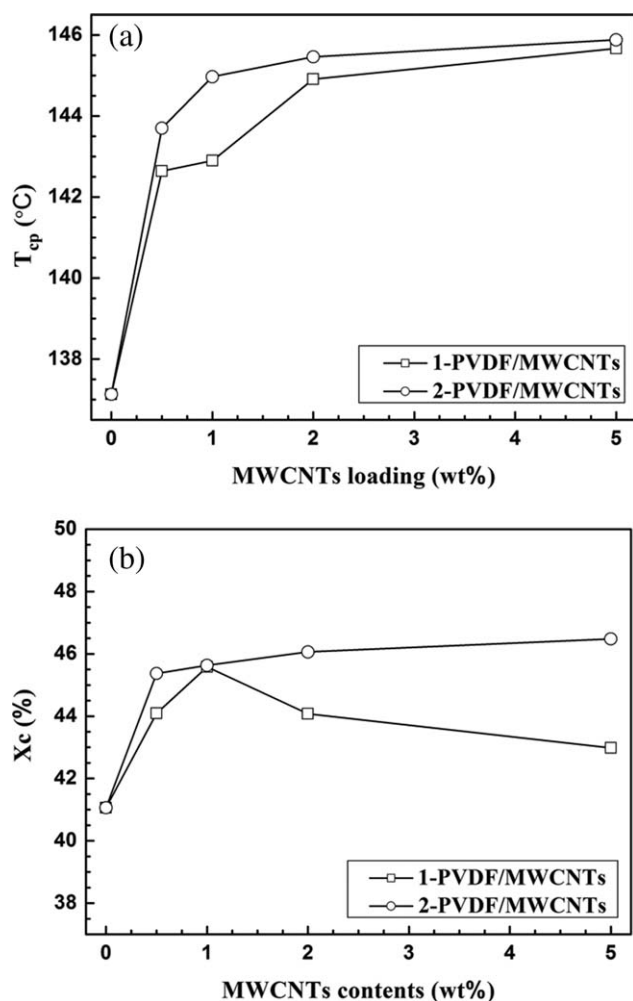
Figure 8 MWCNTs loading dependence of T_{cp} (a) and X_c (b) for the PVDF/MWCNT composites.

nanotube network on the motion of the PVDF chains during crystallization. On the other hand, the crystallinity of 1-PVDF/MWCNT composites initially increases with increasing MWCNT loading, and then decreases subsequently. The reasons can be illustrated as follows. For 2-PVDF/MWCNT composites, the contribution of heterogeneous nucleation by nanotubes to the crystallinity, which is dependent on the dispersion of nanotubes, is stronger than that of 1-PVDF/MWCNT composites. As stated previously, 2-PVDF/MWCNT composites prepared with a stronger shear effect possess a better dispersion of CNTs and can provide more heterogeneous nucleation sites for crystallization when compared with 1-PVDF/MWCNTs.

The comparison of the two groups of composites prepared with different shear effects reveals that the dispersion state of CNTs plays an important role in the crystallization of PVDF composites. Well-dispersed MWCNTs can offer more nucleating sites and contribute to the increase in T_{cp} and X_c of PVDF

composites. Therefore, the nucleation effect seems to be dominant even when the network is formed. However, further investigation of crystallization kinetics of the composites is necessary for studying the influence of network of MWCNTs on the confinement of crystal growth.

CONCLUSION

A melt-powder blending method was used to produce PVDF/MWCNT composites with different dispersion states of MWCNTs. The dispersion state of MWCNTs substantially affects the crystallization behavior, melt rheology, and electrical properties of the composites. A strong shear effect can facilitate the dispersion of MWCNTs in PVDF, resulting in more visible effects on viscoelasticity at low frequencies and higher electrical conductivity of the composites. In addition, comparisons of the crystallization behavior of the two groups of PVDF/MWCNT composites indicate that the contribution of heterogeneous nucleation by MWCNTs to crystallinity exceeds that of the confinement effect by the network of nanotubes.

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