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Electrical and mechanical properties of polyimide–carbon nanotubes composites fabricated by in situ polymerization

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Abstract

Polyimide (PI)–carbon nanotubes composites were fabricated by in situ polymerization using multi wall carbon nanotubes (MWNT) as fillers. It suggested that in situ polymerization is an ideal technique to make a perfect dispersion of carbon nanotubes into matrixes. Besides it, the pre-treatment of carbon nanotubes in solvent to make the networks untied enough and to let solvent percolated into the networks is very important for forming uniform entanglements between carbon nanotubes and polymer molecular chains. The results imply that the percolation threshold for the electric conductivity of the resultant PI–MWNT composites was ca. 0.15 vol%. The electrical conductivity has been increased by more than 11 orders of magnitude to 10^{-4} S/cm at the percolation threshold. The mechanical properties of the polyimide composite were not improved significantly by addition of carbon nanotubes.

Keywords: Polyimide composite; Carbon nanotubes; Electrical property

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1. Introduction

Based on the special chemical, thermal and mechanical characteristics, polyimide has been widely used in the fabrication of aircraft structures, microelectronic devices and so on. However, due to its insulating nature, electrostatic charge may accumulate on the surface of polyimide. This causes local heating, and consequently, premature degradation of material. A surface resistivity in the range of 10^6–10^{10} Ω/cm^2, can be achieved by adding single wall carbon nanotubes (SWNT) to polyimide in order to mitigate the build-up of electrostatic [1,2]. Also, similarly, the surface resistivity of polyimide–carbon black composite has been studies [3]. In this study, we propose an in situ polymerization process of polyimide with multi wall carbon nanotubes (MWNT) for fabricating composite films to study the electrical properties. Their mechanical properties are also investigated.

Carbon nanotubes (CNT) with quasi-one-dimensional structure can be metallic or semiconducting depending on their structural parameters. This makes the CNT as central elements in composite materials for many electronic applications. They also have high stiffness and tensile strength, which make them preferable for the composites with desired mechanical properties [4]. The MWNT have been used in composite materials to improve the electrical properties while reinforcing the mechanical performance of the composites [5–8]. Also, many efforts have been made in exploiting the electrical and mechanical properties of polymer–CNT composite by controlling the orientation of MWNT and polymer molecular chains [9,10]. There are two common processing techniques for fabricating the composites. One is to mix CNT with resin matrix in the melting state to form composites. The other is to disperse CNT into a polymer solution, and casting the solution, then, remove the solvent to obtain the composite. To the best of our knowledge, it is difficult to draw definite conclusions on electrical conductivity from the literatures. This is due to the reported level of CNT loading for a percolation threshold varies widely, ranging from less than 1% to over 10%. Two reasons were thought to account for these discrepancies: the CNT bundles were not well dispersed in the polymer matrix,

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and experimental difficulties resulted in an erroneous detection at the onset of electric conduction [11].

To pursue more perfect dispersion of CNT in polymer matrices, in this study, we pre-treated the CNT in a solvent under sonicating to form loose networks, and adopted in situ polymerization of polyimide with fillers to fabricate the polyimide–CNT composite. This in situ polymerization was firstly reported by Imai et al., where carbon black and nylon-salt-type monomer/carbon black were used as conductive fillers [3]. Ounaies et al. investigated the reinforcement effect of SWNT on polyimide–CNT composite (prepared by the in situ polymerization) as well as electrical properties of the composite [11]. Both of these two studies reported a conductivity jump, with more than eight orders of magnitude, to 10^{-6}–10^{-7} S/cm at a certain critical concentration. The conductivity became almost a constant after the jump. In our experiment, instead of using carbon blacks or SWNT, we used MWNT as fillers to form polyimide–MWNT (PI–MWNT) composites. Our results indicate that the electrical conductivity has been increased by more than 11 orders of magnitude to 10^{-4} S/cm at a critical concentration (ca. 0.15% in volume). It has been further increased to 10^{-1} S/cm when the concentration was raised to 3.7% in volume. The results imply that the percolation threshold for the electric conductivity of the resultant PI–MWNT composites was ca. 0.15 vol%. Although the percolation threshold obtained from this study is higher than that reported in the literature, where the SWNT were used [1,2,11], it is much lower than other polymer composites (using the same MWNT as fillers) prepared with kneading method or dispersing in solution [6,10].

2. Experimental

Two kinds of MWNT with different shapes and distribution in diameter were used as fillers in the present work. One named as MWNT-I furnished by Hyperion Co. Ltd, which was synthesized by catalytic decomposition method. The MWNTs are 10–20 nm in diameter (D), 10–20 μm in length (L) and the L/D ratio is $1–2\times10^3$. The other one, MWNT-II with an average diameter ca. 20 nm was produced by vapor ablating technique. Fig. 1 shows the scanning electron micrographs (SEM) for these two kinds of MWNT. Photo (a) shows that MWNT-I has a uniform distribution in diameter and with a high purity, and nanotubes seem to entangle each other. The aggregates of the large quantities of entanglements have been a big issue to be dispersed in matrices. On the other hand, as shown in photo (b), MWNT-II has a wide distribution in diameter and a few catalysts could be observed, and MWNT self-organized in bundles as like SWNT reported [12,13].

2.1. Fabricating of CNT-contained polyimide composite films

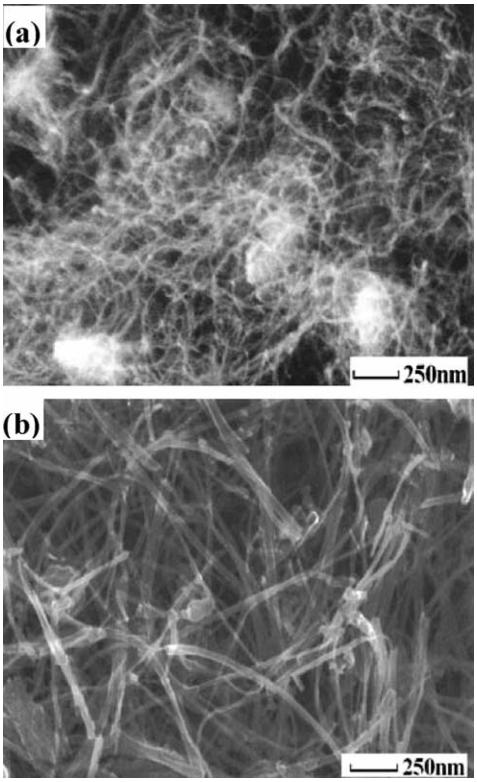
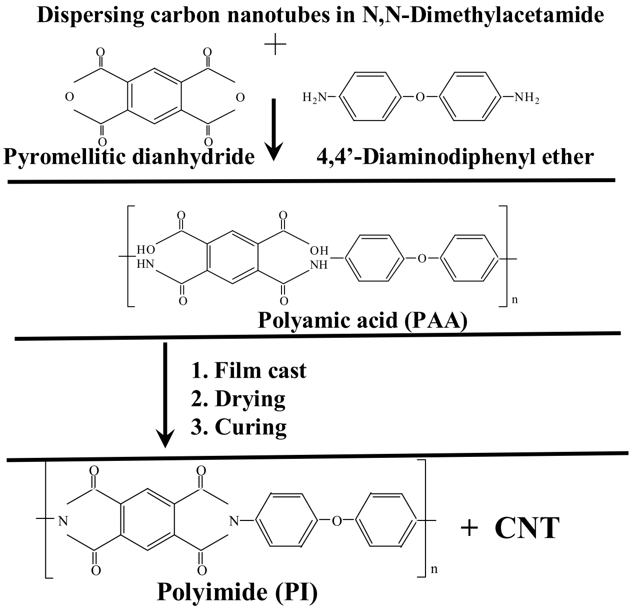


Fig. 1. SEM patterns of two kinds of carbon nanotubes (a) MWNT-I (b) MWNT-II.



Scheme 1. The strategy for the preparation of polyimide composite film with carbon nanotubes.

Scheme 1 shows the strategy for the preparation of polyimide composite film with MWNT. At first, MWNT were pre-treated in *N*,*N*-dimethylacetamide (DMAc) under ultrasonic for 2.5 h to make them untied and to form loose networks. Here we have to emphasize this step is quite important for making a uniform dispersion of MWNT with polyimide. MWNT is not soluble in DMAc. However, the treatment under sonicator made the MWNT networks become loose. The viscosity of solution containing MWNT became very high and even the pure DMAc solvent could not observed indicating that a large amount of solvent percolated into MWNT networks. Then the monomers, pyromellitic dianhydride (PMDA) and with equimolar amounts of 4,4′-diaminadiphenyl ether (DADPE) were added into the DMAc solution containing MWNT. After stirring the solutions for 3 h in the nitrogen gas at ambient temperature, PMDA–DADPE polyamic acid (PAA)–MWNT solution was obtained. The concentration of solution was 10 wt% oligomer against DMAc. MWNT's content against PAA was 0.1–5 wt%. The PAA solution with MWNT was cast onto clean, dry plate glass and dried to tack-free state at 60 °C for 3 h under nitrogen gas flow. And then composite PAA films were cured at 220 °C in vacuum for 3 h to form PI–MWNT composite films. The films were subsequently removed from the glass plate and characterized. The thickness of films is ca. 50 µm. The true density of MWNT is 2.0 g/cm^3. Based on the PMDA–DADPE crystal structure, $a = 0.631$, $b = 0.402$ and c(chain axis) = 3.258 nm (space group $P2/c$), the density of pure PI was calculated to be 1.54 g/cm^3 [14]. For the weight content of 0.1–5% MWNT against matrix, the corresponding volume ratio of MWNT was 0.077–3.71%.

2.2. Measurements

The MWNT dispersion of the composites was observed under with field emission scanning electron microscopes (FE SEM JSM-6700F of JEOL) at accelerating voltage of 1 kV. Differential scanning calorimetry (DSC) was performed with DSC6200 (SII EXSTAR 6000 of Seiko Instrument) with at a heating rate of 10 °C/min under nitrogen. The electrical conductivity of the composites was measured by a two-terminal method. The specimens of electrical resistivity above $10^6\ \Omega$ cm were measured by applying $10\ V_{DC}$ of voltage, while those of low-resistivity ($< 10^6\ \Omega$ cm) were measured under the constant current of $0.1 \sim 1$ mA. Tensile modulus was measured with Instron TENSILON/STM-H-500BP, at a speed of 2 mm/min at room temperature. The size of the specimens was 20 mm in length and 2 mm in width. The temperature dependence of the dynamic tensile modulus of PI–MWNT composites was estimated with a visco-elastic spectrometer (VES-F) (Iwamoto Machine Co. Ltd) at a fixed frequency of 10 Hz over the temperature range of −150–300 °C [15].

3. Results and discussion

Fig. 2 shows DSC curves measured for as-cast poly(amic acid) films containing different contents of MWNT-I. With increasing the content of carbon nanotubes, the endothermic peak assigned to be imidization became broader and the commencement of imidization shifted to higher temperature. The poly(amic acid) films first lose solvent at low temperature with little or no imidization, and followed by thermal imidization in which additional solvent is lost. Significant crystallization occurs only when the imidization is nearly complete [16]. During thermal imidization, a reverse reaction of acylation (depolymerization) occurs partially to form amino and anhydride terminal groups and leads to a tentative molecular weight decrease, although the terminal groups recombine by additional annealing at higher temperature [17]. The peak profiles of Fig. 2 may imply that the anhydride groups are formed in a wider temperature range for the composites than that for neat polymer, and the loss of solvent is slower because of the restriction by the loose carbon nanotubes networks. It may be suggested that the treatment of carbon nanotubes in DMAc solvent even leads to the solvent partly penetrating into the hollow of nanotubes. The releasing of solvent from the inner of nanotubes was reasonable to be much slower than from the outside parts. The other reason for the lower imidization speed of composite films is thought to be due to the lower local mobility at the reactive sites because of the restriction by nanotubes and there is additional curing in the film interior, because the trapped solvent acts as a plasticizer. However, for all specimens, the endothermic peaks ended almost at ca. 220 °C, so it can be considered that the annealing at 220 °C in vacuum for 3 h promoted entirely recombination reaction and the imidization was completed.

Fig. 3(a) shows the electrical resistivity against MWNT content measured at room temperature for PI–MWNT-I composites. The value of electrical resistivity of the films decreased more than eleven orders of magnitude from 10^{15} to $10^3\ \Omega$ cm in the range from 0.077 to 0.38 vol% of MWNT

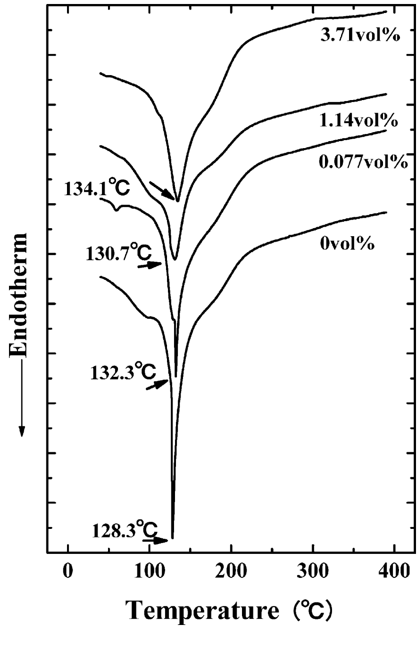


Fig. 2. DSC curves for the PAA–MWNT-I films.

content. The resistivity decrease with MWNT content fraction indicates a typical percolation transition behavior. The resistivity showed a slightly linear decrease and reached 10^1 Ω cm corresponding to conductive material when the MWNT content was 3.71 vol%.

Percolation theory predicts the relationship between the composite resistivity and volume content of the conductive filler as [18]

$$\rho = \rho_0(\phi - \phi_c)^{-t} \tag{1}$$

where ρ, composite resistivity (Ω cm); ρ_0, resistivity of conductive filler (Ω cm); ϕ, volume fraction of filler; ϕ_c, percolation threshold; t, critical exponent.

This equation is valid at the concentration above the percolation threshold, i.e. when $\phi > \phi_c$. The value of the critical exponent, t, is dependent on the dimension of the lattice and the aspect ratio of the filler with a value in the range of 1–3.1 [18,19]. Based on Fig. 3(b), ϕ_c was determined to be 0.15 vol%, and t was determined as to be 1.56 obtained from the slope of the regression analysis. The percolation threshold value of 0.15 vol% which is much lower than that for any resins filled with the same MWNT reported till now [6,10,20].

As mentioned in the introduction part, there are several reports containing the polyimide composite used carbon nanotubes as fillers [1,2,11,20]. Ounaies et al. [11] investigated the electrical properties for reinforced polyimide–SWNT composites. In their research, purified laser ablated SWNT were used as fillers and an aromatic polyimide as polymer matrix. They obtained the percolation threshold of 0.05 vol% SWNT. The electrical conductivity of polyimide composites increased more than 7 orders of magnitude, and reached to 10^{-8}–10^{-6} S/cm corresponding to semiconductor field when the SWNT concentration was beyond 0.2 vol%. Smith et al. [1,2] adapted two-step process for preparing the PI–SWNT composites film: as the first step, the amide acid polymer was synthesized, and secondly adding carbon nanotubes to the pre-made polymer and the mixture was then stirred and cast to form films. They got a similar result in the electrical properties as reported by Ounaies et al. [11]. They pointed out that the percolation threshold had been reached at a load of 0.05 wt% SWNT, the further addition of SWNT, till to 0.08 wt%, exhibited similar surface resistivity and volume resistivity as the films containing 0.05 wt% SWNT, without further increase in conductivity. The volume resistivity was at a level of 10^{10} Ω cm. The decrease in electrical resistivity in the percolation threshold was about 10^5 Ω cm. For polyimide and MWNT composite system, Ogasawara et al. prepared the films by mixing MWNT with imide oligomers using a mechanical blender, in which MWNT content was 3.3–14.3 wt% [20]. They pointed out that the electrical conductivity of the composites was 10^{-4}–10^{-2} S/cm in the CNT concentration range from 5 to 15 vol% and the percolation threshold was calculated to be 2.2 vol%.

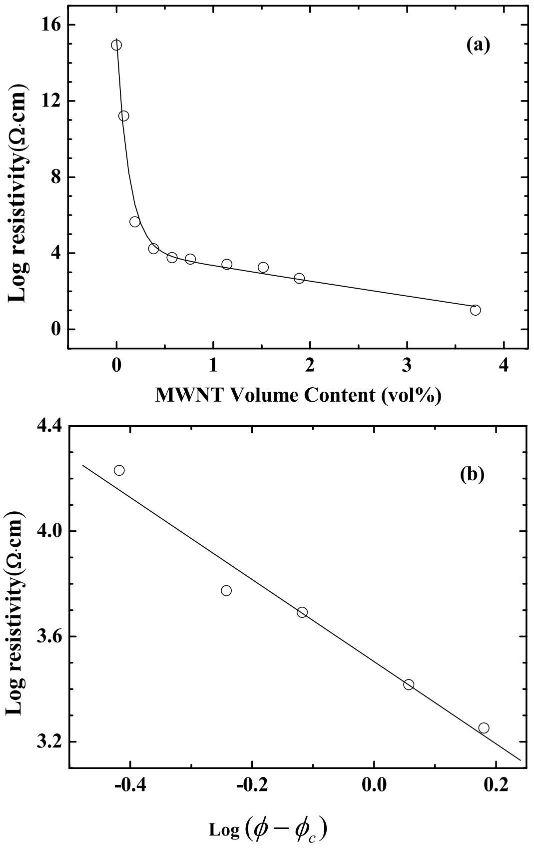


Fig. 3. (a) Dependence of the Dc resistivity at room temperature on MWNT-I content for PI composite; (b) plot of resistivity against $\phi - \phi_c$ with $\phi_c = 0.15$ vol% according to Eq. (1).

However, the results in the present study show a different tendency. The electrical conductivity of polyimide composites had a higher jump at the percolation threshold compared with the above cases. The value reached to 10^{-4} S/cm when the MWNT concentration beyond 0.37 vol% and to 10^{-1} S/cm while MWNT was 3.71 vol%. This suggested indicate a higher conductivity compared with PI–SWNT composites, and a quite low percolation threshold compared with PI–MWNT film prepared by Ogasawara et al.

Here it may be noted that the electric properties of polymer composites was sensitive to the kinds of polymer matrices and the preparation methods, even the same MWNTs were used. Shaffer et al. reported the percolation threshold for the poly(vinyl alcohol) composite with the same kind of carbon nanotubes used here, MWNT-I, lies between 5 and 10 wt% [21]. The percolation threshold for the polycarbonate composite reported elsewhere was about 2 wt% [6]. This means that the dispersing of the MWNT in matrixes is not as sufficient as in the present system. In situ polymerization of poly(amic acid) may make molecular chains formed in MWNT networks instead of the dispersion of MWNTs into polymer known as a normal technique. The following imidization of poly(amic acid) also occurred within the MWNT networks. The process yielded a large of entanglements between polyimide molecular chains and nanotubes. It is reasonable to get a quite low percolation threshold and high conductivity.

Fig. 4 shows the SEM patterns observed on the cross-section of PI–MWNT with 1.14 vol% MWNT-I. In photo (a), the dispersion of MWNT seems to be not very uniform in whole. A part of entangled MWNT networks exist implying the difficulty of the untying of MWNTs aggregated in the as-prepared state (see the photo (a) in Fig. 1). However, the further observation in photo (b) which is the enlargement of MWNT aggregated part in photo (a) indicated that carbon nanotubes were well wetted by polyimide matrices.

The microscopes of the cross-section of the polyimide–MWNT-II composite film was shown in Fig. 5. Compared with Fig. 4, the dispersion of MWNT-II in polyimide matrix seems to be more uniform. However, the photo (b), the enlargement of photo (a), indicates that the bundles of MWNT which were observed in photo (b) in Fig. 1 were not separated perfectly during pre-treatment and mixing. This leads to one order of magnitude difference in conductivity for the composite as listed in Table 1. The electrical conductivity for PIMWNT-II composites was 4.35×10^{-5} S/cm which is lower than 3.83×10^{-4} S/cm for PI–MWNT-I at the same content of 1.5 wt% (1.14 vol%) CNT. The separation of the bundles of MWNT-II in solvent must be taken into consideration in the further research.

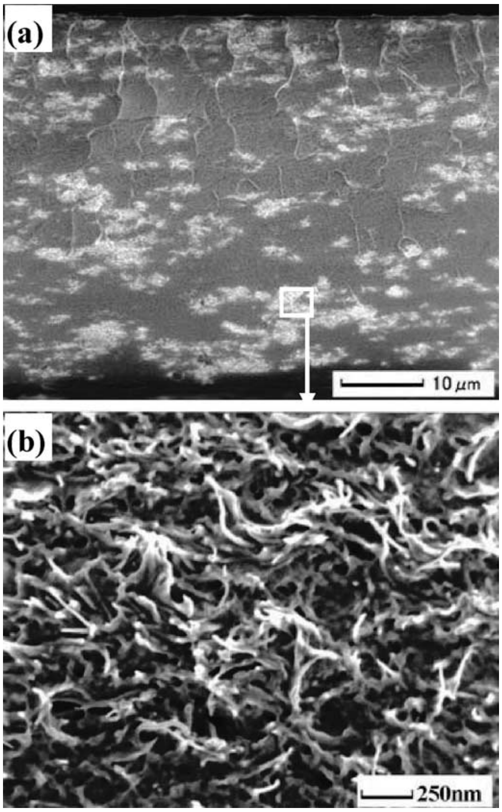


Fig. 4. SEM patterns of the cross-section of PI–MWNT-I (MWNT = 1.14 vol%) composite film.

Even though the wetting of polyimide and carbon nanotubes in the composites prepared with in situ polymerization is absolutely different from those prepared by normal blending technique such as mixing in melt state or mixing in solutions, it should be noticed that the electrical resistivity for the composites was not lower than 10^0 Ω cm even when the content reaches 3.71 vol% which is much higher than the percolation threshold. This value is much lower than the intrinsic conductivity of MWNT. The SEM patterns in Figs. 4 and 5 shown the most of conductive fillers were covered by a layer of insulating matrixes. The MWNT are expected to promote nucleate crystallization on its surface as reported for the cured polyimide and carbon fibers composites [22,23]. The crystallites formed on the CNT surface may interrupted the direct contact of a part of CNT, thus restrained the improvement of conductivity of the composites.

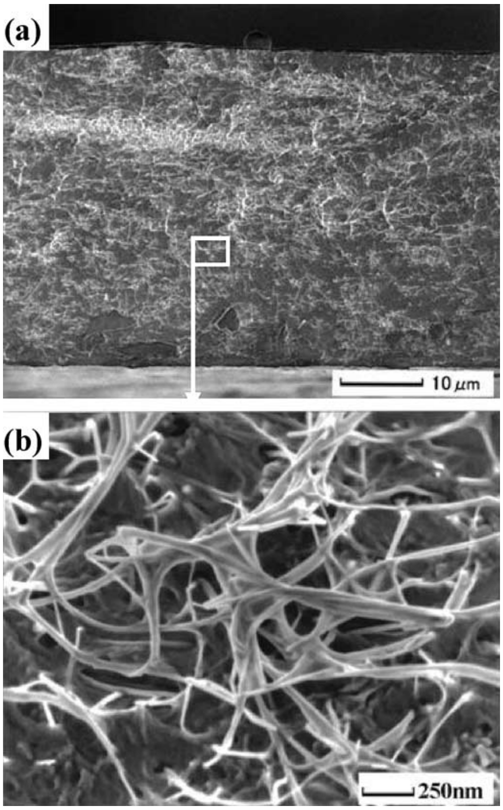


Fig. 5. SEM pattern of the cross-section of PI–MWNT-II (MWNT = 1.14 vol%) composite film.

To check the effect of the aspect ratio, besides MWNT, vapor growth carbon fiber (VGCF) with an average diameter ca. 150 nm furnished by Shouwa Denkou Co. Ltd, and low structure carbon black (CB) produced by Cobat. Co. Ltd were also used as fillers to prepare the polyimide composites with the same fabrication method.

Table 1 lists the electric conductivities for the polyimide composites filled with four kinds for carbon fillers. The content of filler was fixed to be 1.5 wt%. The conductivities for the CB and VGCF composites were 9.83×10^{-15} and 1.02×10^{-14} S/cm, respectively. They were almost the

Table 1
The electrical conductivity of polyimide with different carbon fillers at a content of 1.5 wt%

Filler	Electrical conductivity (S/cm)
No filler	1.17×10^{-15}
MWNT-I	3.83×10^{-4}
MWNT-II	4.35×10^{-5}
VGCF	1.02×10^{-14}
CB	9.83×10^{-14}

same as pure polyimide (1.17×10^{-15} S/cm). But the electric resistivity reached 3.83×10^{-4} and 4.35×10^{-5} S/cm for PI–MWNT-I and PI–MWNT-II, respectively, indicating the significant effect of high aspect ratio of carbon nanotubes on promoting the electric conductivity.

The well wetting of matrix and fillers was expected to improve the mechanical properties of polymers. So Young's modulus and viscoelastic behavior of the above PI–MWNT composite films were also investigated. Through a series of experiments, it was found that there is no evident difference in the mechanical properties for the composites with MWNT-I or MWNT-II. Thus here just report the results of PI–MWNT-I composite films.

Fig. 6 shows the Young's modulus for the composite films of PI with different MWNT contents measured by Instron Tensile. Only about 6% increase in Young's modulus was observed when MWNT content was 0.077–1.89 vol%. And the Young's modulus decreased slightly when MWNT content further increase.

Fig. 7 displays the temperature dependence of storage and loss moduli for the PI film and PI–MWNT composite with 1.14 and 3.71 vol% MWNT performed at a fixed frequency of 10 Hz. The mechanical properties were not sensitive to the amount of MWNT generally. The storage modulus for the composites containing a small amount of MWNT (1.14 wt%) was about 20% higher than that of pure PI film in the given temperature range from −150–300 °C. However, the modulus for the composites with 3.71 vol% MWNTs was lower than that of neat PI as temperature lower than 200 °C, and a little higher at high temperatures. The loss modulus shows three dispersion peaks associated with α, β and γ relaxation mechanisms of PI, but the three peaks were no significantly influenced by adding MWNT.

There are several reports on carbon fiber (CF) reinforced polyimide composites concerning the laminate thickness, the degree of curing of the matrix, crystallization conditions, and on the mechanical properties of the polyimide [23,24]. The fiber was thought to be nucleate crystallization on the surface and to induce a highly ordered trans-crystallinity, which may result in improved mechanical properties of the composites material in the fiber direction. The nucleating ability is dependent strongly on the precursor of carbon fibers and the content. The storage modulus of the PI–CF composite increased nearly one order of magnitude when the volume of carbon fiber increased to 72% [24]. The high aspect ratio for the carbon nanotubes

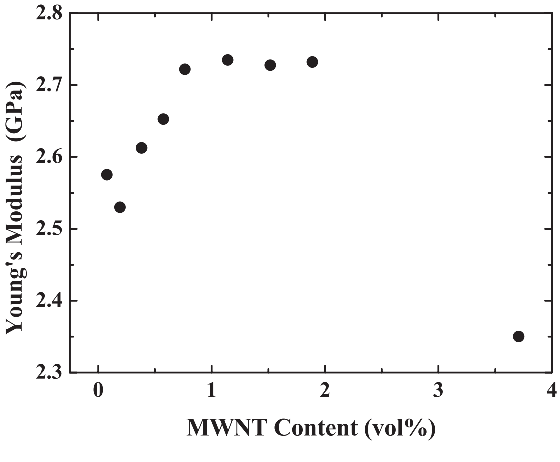


Fig. 6. Young's Modulus of PI–MWNT-I composites films against the MWNT content.

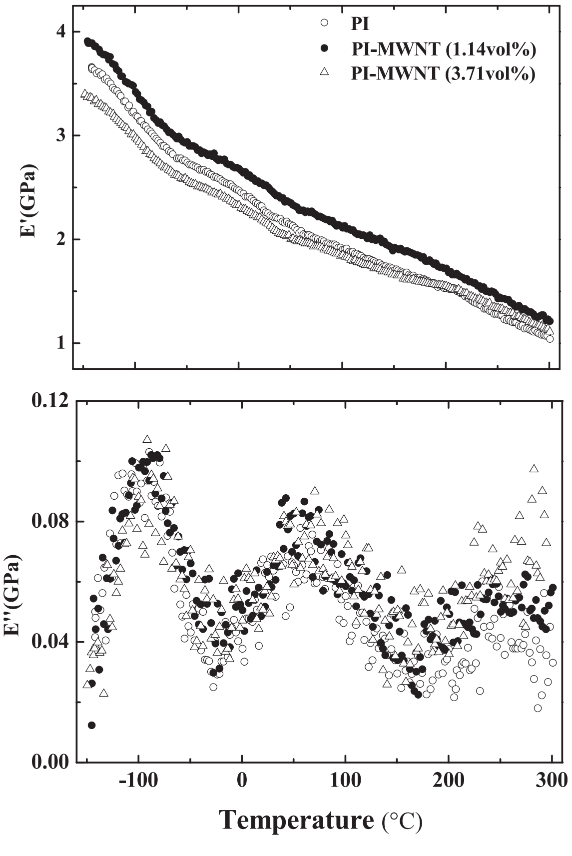


Fig. 7. Temperature dependence of storage and loss modulus of PI, PI–MWNT-I (1.14 and 3.71 vol%) composite films.

and well wetting between matrix and nanotubes are expected to be in favor of improving the mechanical properties of the composites. According to the report by Ogasawara et al. [20], the addition of CNT caused an increase in the elastic modulus and yield strength, and decrease in the failure strain. But the resulting polyimide composites showed only 37% increase in the elastic modulus for the composite containing 14.3 wt% MWNTs.

However, the reinforcement effect was not evident in the present experiment. Compared with the above researches [20,24], the content of nanotubes was much lower in the present experiment. Another reason may be considered that the addition of carbon nanotubes may be an obstacle to ensure the degree of polymerization and orientation of polyimide. Hatori et al. researched the in-plane orientation of polyimide [25]. They pointed out that resultant PI oriented in-plane by casting PAA on the substrate and subsequent imidization without taking off from the substrate in vacuum. It may be considered that the in-plane orientation of PI molecular chains in the composite prepared by in situ polymerization with CNT would be destroyed somewhat because of the formation of entangled structures between PI molecular chains and carbon nanotubes.

4. Conclusion

Polyimide–carbon nanotubes composites were prepared by in situ polymerization. It suggests that in situ polymerization is most powerful in the fabrication of PI composites with a certain level of electric conductivity by addition of a very small amount of carbon nanotubes. Besides it, the pre-treatment of carbon nanotubes in solvent to make the networks untied enough and let solvent percolated into the network is very important for forming large mount of entanglements between carbon nanotubes and polymer molecular chains. The results imply that the percolation threshold for the electric conductivity of the resultant PI–MWNT composites was ca. 0.15 vol%. Although the percolation threshold obtained from this study is higher than that reported in the literature, where the SWNT were used, it is much lower than other polymer composites (using the same MWNT as fillers) prepared with kneading method or dispersing in solution. The electrical conductivity has been increased by more than 11 orders of magnitude to 10^{-4} S/cm at the percolation threshold. It has been further increased to 10^{-1} S/cm when the concentration was raised to 3.7% in volume. The mechanical properties of the polyimide composite were not improved significantly by addition of carbon nanotubes.

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References

[1] Smith Jr JG, Connell JW, Delozier DM, Lillehei PT, Watson KA, Lin Y, et al. Polymer 2004;45:825.
[2] Smith Jr JG, Delozier DM, Connell JW, Lillehei PT, Watson KA. Polymer 2004;45:6133.
[3] Imai Y, Fueki T, Inoue T, Kakimoto M. J Polym Sci, Part A: Polym Chem 1998;36:1031.
[4] Popov VN. Mater Sci Eng 2004;R43:61.
[5] Thostenson ET, Ren Z, Chou TW. Compos Sci Technol 2001;61:1899.
[6] Potschke P, Fornes TD, Paul DR. Polymer 2002;43:3247.
[7] Curran S, Davey AP, Coleman J, Dalton A, McCarthy B, Maier S, et al. Synth Met 1999;103:2559.
[8] Cadek M, Coleman JN, Barron V, Hedicke K, Blau WJ. Appl Phys Lett 2002;81(27):5123.
[9] Bin Y, Kitanaka M, Zhu D, Matsuo M. Macromoleculs 2003;36:6213.
[10] Koganemaru A, Bin Y, Agari Y, Matsuo M. Adv Funct Mater 2004;14:842.
[11] Ounaies Z, Park C, Wise KE, Siochi EJ, Harrison JS. Compos Sci Technol 2003;63:1637.
[12] IBM Almaden Research Center. Phys B: Condens Matter 2002;323(1–4):90.
[13] Borowiak-Palen E, Pichler T, Liu X, Knupfer M, Graff A, Jost O, et al. Chem Phys Lett 2002;363(5/6, 16):567.
[14] Freilich SC, Gardner KH. In: Feger C, Khojasteh MM, Mcgrath JE, editors. Ploymides: materials, chemistry, and haracterization. Amsterdam: Elsevier; 1989. p. 513.
[15] Matsuo M, Sawatari C, Ohhata T. Macromolecules 1988;21:1317.
[16] Ojeda JR, Mobely J, Martin DC. J Polym Sci, Part B: Polym Phys 1995;32:559.
[17] Hasegawa M, Sebsui N, Shindo Y, Yokota R. Macromolecules 1999;32:387.
[18] Weber M, Kamal MR. Polym Comp 1997;18(6):711.
[19] Xi Y, Ishikawa H, Bin Y, Matsuo M. Carbon 2004;42:1699.
[20] Ogasawara T, Ishida Y, Ishikawa T, Yokota R. Compos: Part A 2004;35:67.
[21] Shaffer MSP, Windle AH. Adv Mater 1999;11:937.
[22] Yudin VE, Svetlichnyi VM, Gubanova GN, Grigor'ev AI, Sukhanova TE, Gofman IV, et al. Polym Sci Series A 2002;44:148.
[23] Nuriel H, Klein N, Marom G. Compos Sci Technol 1999;59:1685.
[24] Labronici M, Ishida H. Polym Compos 1999;20(4):515.
[25] Hatori H, Yamada Y, Shiraishi M. Carbon 1992;30:763.