

IMPROVEMENT OF CREEP RESISTANCE OF POLYTETRAFLUOROETHYLENE FILMS BY NANO-INCLUSIONS*

Xue-bo Shi^a, Chun-lei Wu^b, Min-zhi Rong^a, Tibor Czigany^c,
Wen-hong Ruan^{a**} and Ming-qiu Zhang^a

^a Key Laboratory for Polymeric Composite and Functional Materials of Ministry of Education, DSAPM Lab, School of Chemistry and Chemical Engineering, Sun Yat-sen (Zhongshan) University, Guangzhou 510275, China

^b Donghong Rubber & Plastics Products Company Ltd., Guangzhou 510530, China

^c Department of Polymer Engineering, Budapest University of Technology and Economics, H-1111 Budapest, Hungary

Abstract To improve creep resistance of directional polytetrafluoroethylene (PTFE) films, epoxy grafted nano-SiO₂ is mixed with PTFE powder before sintering and calender rolling. The aligned macromolecular chains (especially those in amorphous region) of the composite films can be bundled up by the nanoparticles to share the applied stress together. In addition, incorporation of silica nanoparticles increases crystallinity of PTFE and favors microfibrillation of PTFE in the course of large deformation. As result, PTFE films exhibit lower creep strain and creep rate, and higher tensile strength and hardness. The work is believed to open an avenue for manufacturing high performance fluoropolymers by nano-inclusions.

Keywords: Polytetrafluoroethylene (PTFE); Nano-silica; Directional films; Creep resistance; Crystallinity.

INTRODUCTION

Owing to its unique electrical insulation, chemical inertness and high temperature resistance, polytetrafluoroethylene (PTFE) has gained commercial acceptance in a number of industrial products. However, the inherent cold flow (or high creep) hinders PTFE from applications where dimensional and performance stabilities are critical^[1]. Irreversible plastic strain would be developed in PTFE under low loading, even below glass transition temperature^[2].

So far, many works have been carried out to raise creep resistance of PTFE. The proposed methods can be classified into two categories, namely physical and chemical modifications. The former incorporates micro- or nano-sized fillers into PTFE^[3–5]. Because filler-polymer bonding is less considered, the effect is not significant enough. The latter induces crosslinking of PTFE chains by radiation chemical approaches^[6–11]. Due to the fact that PTFE has low radiation resistance and high oxygen sensitivity, chain scission rather than crosslinking would easily occur during radiation exposure. Mechanical strength of the material has to be decreased accordingly^[10, 11].

The specific properties of PTFE results from its structures with strong bonding between carbon and fluorine atoms, and shielding of the carbon backbone by fluorine atoms. PTFE has a twisting helix configuration consisting of carbon core and fluorine shell^[12]. As a result of mutual repulsion of adjacent fluorine atoms and their relatively large size, intermolecular bonding is negligible. Individual chains can thus slip past each other.

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** Corresponding author: Wen-hong Ruan (阮文红), E-mail: cesrwh@mail.sysu.edu.cn

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On the other hand, the extremely high molecular weight of PTFE leads to a melt viscosity about six orders of magnitude higher than that of most thermoplastic polymers, so that melt-processing is not applicable for fabrication of PTFE. Instead, powder metallurgy is used for manufacturing block items from virgin PTFE powders produced by emulsion or suspension polymerization. PTFE film is yielded from powders subjected to cold press and sintering at 380°C as billet, then cut and calendered to generate directional products. The structure of directional PTFE films is comprised of stacked long crystallites and extended amorphous phase interlayers with defects of chain ends and cavitations^[13]. Although improvement of mechanical properties is possible due to high degree of chain orientation, amorphous regions are still the weak points for withstanding applied stress. In this context, enhancing intermolecular interaction of amorphous phases in oriented PTFE might help to increase creep resistance of the material.

To solve the problem in practice, nanotechnology is believed to be a feasible way. Because the size of nanometer particles is comparable to that of single polymer chains, dynamics of polymers can be greatly influenced by the nanoparticles' boundary interactions, as indicated by both experimental and theoretical studies^[14–18]. Our previous investigation also shows that addition of nanoparticles can effectively improve the creep resistance of polypropylene (PP) so long as the fillers are inter-connected throughout the matrix^[19]. Moreover, the strong interaction between nanoparticles and polymer results in tying up of aligned chain segments in amorphous regions of pre-drawn PP and increase of the concentration of macromolecules that bear loading^[20]. Inspired by these results, it is understood that creep resistance of directional PTFE films can be raised by adding nanoparticles that have high affinity to the matrix. The present paper verifies this idea by using nano-SiO₂ as filler. Different surface pre-treatments of the nanoparticles, including silane modification and graft polymerization, are conducted prior to manufacturing of filmy PTFE nanocomposites. Mechanical properties and microstructure of the materials are characterized and discussed.

EXPERIMENTAL

Surface Treatment of Nano-silica by Coupling Agent

Fumed nano-silica (Aerosil 200) with an average primary particle size of 12 nm and a specific surface area of 200 m²/g was supplied by Degussa Co., Germany. 3-Aminpropyltriethoxysilane (trade name: KH550) produced by Aladdin Co., China was chosen as coupling agent.

The mixture of 10.0 g nano-silica, 3 mL KH550 and 200 mL absolute alcohol was ultrasonically vibrated for 30 min, and then refluxed with stirring for 5 h under N₂ atmosphere. The pretreated silica was recovered by centrifugation, extracted by acetone in a Soxhlet apparatus for 72 h to remove excess KH550 coupling agent, and finally dried under vacuum at 40°C for 48 h, then at 100°C for 12 h. In this way, reactive groups were introduced onto the surface of SiO₂ nanoparticles (for the convenience of discussion hereinafter, the silane pretreated nano-silica is denoted as SiO₂-KH550).

Graft Polymerization onto Nano-silica

Commercially available bisphenol A epoxy resin, obtained from Dongfeng Chemical Company, China, acted as the grafting monomer. 10.0 g SiO₂-KH550, 4.0 g bisphenol A and 200 mL absolute alcohol were refluxed with stirring for 8 h under N₂ atmosphere. The unreacted monomer and homopolymer were removed by acetone in a Soxhlet apparatus for 72 h. Having been dried under vacuum at 140°C for 8 h, the epoxy grafted nano-silica (denotes as SiO₂-g-EP) was grounded for the subsequent usage.

Preparation of Directional PTFE Nanocomposite Films

The untreated, KH550 treated and epoxy grafted SiO₂ nanoparticles were respectively mixed with PTFE powders (suspension polymerization, 831010R-34CGM-16H, molecular weight $\approx 4 \times 10^6$ – 6×10^6 , provided by Zhonghao Chenguang Research Institute of Chemical Industry, China). The mixture was subjected to cold press under 100 MPa for 10 min, then heated to 337°C at a heating rate of 30 K/h, holding for 1 h, heated to 380°C at a heating rate of 10 K/h, and sintered at 380°C for 8 h. After sintering, the billet was cooled to room temperature in a controlled manner: cooled to 320°C at a cooling rate of 25 K/h, holding for 2 h, cooled to 300°C at a cooling

rate of 25 K/h, holding for 2 h, cooled to 280°C at a cooling rate of 30 K/h, holding for 1 h, cooled to 250°C at a cooling rate of 30 K/h, holding for 1 h, and then naturally cooled to room temperature. Finally, the cylinder billet was cut by a skiving machine and calender rolled under 180°C to produce directional films with thickness of 0.05–0.15 mm.

Characterization

The weight increase of nano-silica due to the presence of silane coupling agent KH550 or the grafted epoxy was determined by a thermogravimetric analyzer (TGA, Netzsch TG 209).

Structure of the grafted epoxy was characterized by Fourier transform infrared spectroscopy (FTIR, Nicolet Nexus 670) and nuclear magnetic resonance spectroscopy (NMR, Bruker AVANCE AV 400).

Tensile tests were carried out at room temperature according to GB/T 1040.3-2006 by using a universal testing machine (Hounsfield H10KS) under a crosshead speed of 10 mm/min. Tangent modulus was determined at strain of 2%. Five specimens for each batch were tested in the direction of calender rolling. The dimension of film specimen is 120 mm × 10 mm × 0.10 mm.

Creep behavior was measured in the direction of calender rolling at room temperature according to ISO 899-1:2003 by a thermal mechanical analyzer (TMA, MetraviB DMA-25N) using tensile mode under N₂ atmosphere. Creep stress was set at 1.5 MPa, which is much lower than the tensile strength of PTFE and its nanocomposites by considering cold flow of PTFE. The dimension of film specimen is 60 mm × 8 mm × 0.15 mm. Three parallel samples were tested.

Micro-hardness was tested by Vickers instrumented indentation (SHIMADZU DH-W201S) with load-unload mode with maximum force of 30 mN and holding time of 30 s. The dimension of film specimen is 20 mm × 10 mm × 0.15 mm. Ten parallel data points were recorded.

Non-isothermal crystallization and melting behaviors were studied by differential scanning calorimetry (DSC, TA Q10) under N₂ atmosphere from –20°C to 380°C at a heating rate of 5 K/min.

Crystallinity and crystal orientation were determined by using a wide angle X-ray diffractometer (WAXD, Rigaku D/MAX 2200 VPC) with Cu-K α radiation. The scanning range of Bragg 2θ angle was from 10° to 40° under a scanning rate of 5 K/min. Intensity ratio of (100) reflection ($2\theta = 18.0^\circ$), $I(100)$, to (200) reflection ($2\theta = 36.0^\circ$), $I(200)$, was calculated to evaluate the orientation degree of the a-axis of PTFE crystals^[21].

Orientation factors of amorphous and crystalline phases in PTFE and its nanocomposites were determined in terms of infrared dichroism method by Fourier transform infrared spectroscopy (FTIR, Bruker Equinox55). The specimens were tested in the direction vertical and parallel to the rolling direction, respectively. The bands representing oriented amorphous (1510–2080 cm^{–1}) and crystalline regions (2040–2750 cm^{–1}) of PTFE were used for the calculation^[22].

Microstructure of PTFE and its nanocomposites before and after tensile test was observed by scanning electron microscopy (SEM, JEOL-5400). The samples were split in parallel to the tensile direction in liquid nitrogen in advance.

RESULTS AND DISCUSSION

Surface Modification of Nano-silica

To enhance interaction between nanoparticles and PTFE matrix, introducing graft polymer onto nanoparticles surface is necessary, as entanglement between molecular chains of grafted polymer and surrounding matrix proves to be a useful measure^[23]. Considering that PTFE powder has to be sintered at 380°C during processing, the grafted polymer should be stable enough at the very temperature to keep from thermal degradation. Therefore, heat resistant bisphenol A type epoxy serves as the graft monomer in this work (see the Experimental).

First, nano-silica is pretreated by a silane coupling agent (KH550) for introducing reactive amino-groups onto the surface of nano-silica. On the basis of optimization, SiO₂-KH550 with 4.97 wt% KH550 attached to nano-SiO₂ surface (determined by TGA measurement) is chosen for further graft polymerization. Since the

amino groups of KH550 can react with epoxy groups, epoxy is thus polymerized onto silica nanoparticles (Fig. 1).

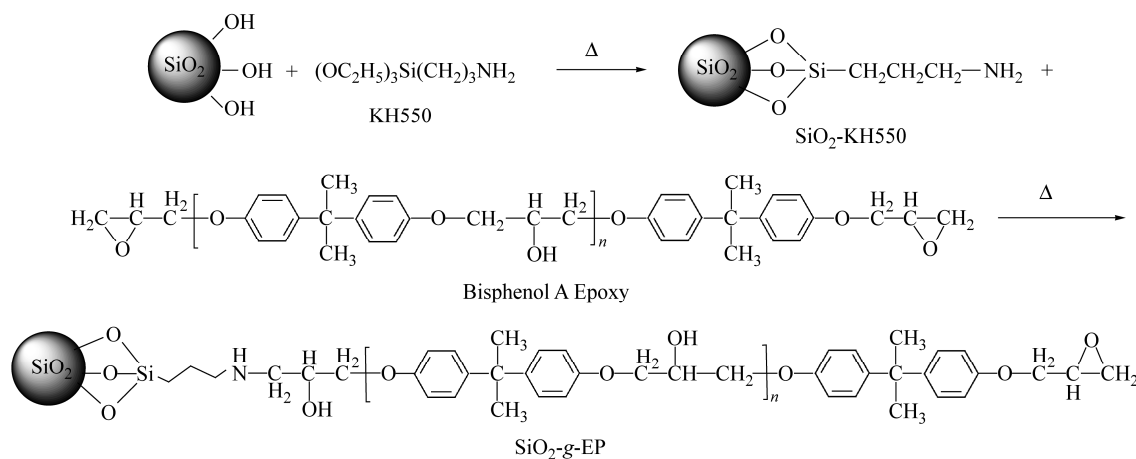


Fig. 1 Schematic drawing of surface modification and graft polymerization onto nano-silica

Figure 2 gives the infrared spectra of nano-silica as-received and its treated versions. In comparison with the spectrum of untreated nano-silica, —OH adsorption at 3340 cm^{-1} disappears in the spectrum of SiO₂-KH550, while the peak at 1557 cm^{-1} that belongs to —NH— appears. It means that amino double bonds have been introduced onto the nanoparticles as expected. In the spectrum of SiO₂-g-EP, multiple peaks representing benzene ring are perceived from 1360 cm^{-1} to 1751 cm^{-1} (see the inset of Fig. 2). Additionally, weak adsorption at 2965 cm^{-1} related to methyl and methylene groups appear. These suggest that the amino groups on the surface of KH550 treated nano-silica have reacted with the reactive monomers, and bisphenol A epoxy has been chemically bonded to nano-SiO₂.

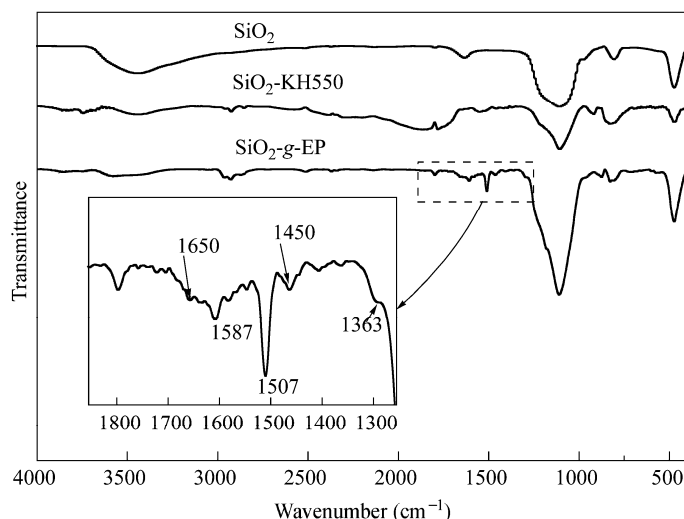


Fig. 2 FTIR spectra of nano-SiO₂ as-received, SiO₂-KH550 and SiO₂-g-EP

To further confirm the existence of grafted epoxy on surface of the nanoparticles after the aforesaid treatment, solid state NMR spectrum was collected (Fig. 3). It is seen that the peaks assigned to hybridized carbon of sp^2 appear from $\delta = 114.4$ to 157.5 , showing that benzene ring carbon is present on nano-silica. The

peaks between $\delta = 48.8$ and 58.2 originate from amino carbon, which implies that tertiary or secondary amino groups are attached to the nanoparticles. Moreover, the peaks at $\delta = 14.8$, 31.0 and 71.6 represent $\text{C}-\text{CH}_3$, methyl groups and carbon of $\text{O}-\text{CH}_2$ groups, respectively. Clearly, the NMR analysis also demonstrates that epoxy has reacted with amino on silane pretreated nano-silica and introduced onto silica nanoparticles.

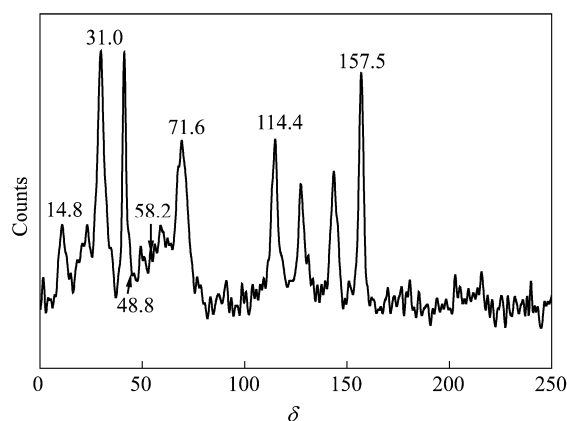


Fig. 3 ^{13}C -NMR spectrum of SiO_2 -g-EP

Mechanical Properties of PTFE Nanocomposites

At the beginning of mechanical properties characterization, tensile tests were performed to reveal differences in mechanical behavior between unfilled PTFE and PTFE nanocomposites films. Figure 4 shows typical stress-strain curves of the materials, in which SiO_2 -g-EP contains 17.8 wt% grafted epoxy (the grafted nano- SiO_2 with the same grafting percentage is included in SiO_2 -g-EP/PTFE composites in the following discussion). We can see that irreversible plastic strain exists in the early stage of deformation (refer to the insert of Fig. 4). Linear segment of the curves corresponding to elastic strain was not recorded at all. Nonlinear response of PTFE in low strain region is related to its structure, *i.e.*, linear chain configuration, high degree of crystallinity and chain orientation without intermolecular bonding, *etc.* These factors give rise to cold flow. Nevertheless, directional PTFE films become much stronger and stiffer after incorporation of the nanofillers, especially in the case of SiO_2 -g-EP. With a rise in nano- SiO_2 content, as shown in Table 1, modulus increases and elongation at break decreases like the case of particulates filled rubbery materials. With respect to tensile strength, there is an

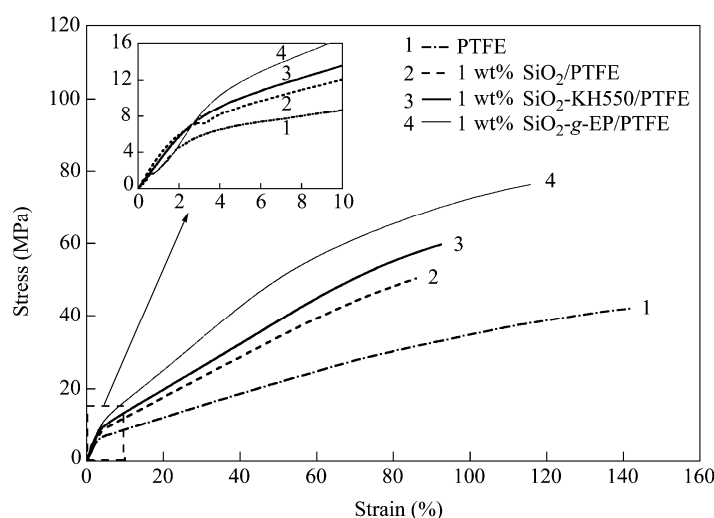


Fig. 4 Typical tensile stress-strain curves of directional films of PTFE and its nanocomposites

optimal filler content in connection with particles dispersion status. SiO₂-g-EP filled systems have the highest reinforcement effect, obviously owing to the enhanced interaction between grafted nanoparticles and the matrix, as mentioned in the beginning of last section.

Table 1. Tensile properties of directional films of PTFE and PTFE nanocomposites as a function of nano-SiO₂ content

Materials	Modulus (MPa)	Tensile strength (MPa)	Elongation at break (%)
PTFE	318.2	42.7	137.5
0.5 wt% SiO ₂ /PTFE	406.6	43.8	96.3
1.0 wt% SiO ₂ /PTFE	452.6	52.4	87.6
1.5 wt% SiO ₂ /PTFE	500.3	54.9	74.6
2.0 wt% SiO ₂ /PTFE	523.3	53.2	67.7
0.5 wt% SiO ₂ -KH550/PTFE	416.4	52.0	94.3
1.0 wt% SiO ₂ -KH550/PTFE	504.3	61.8	89.6
1.5 wt% SiO ₂ -KH550/PTFE	526.3	63.8	81.3
2.0 wt% SiO ₂ -KH550/PTFE	538.3	63.4	69.5
0.5 wt% SiO ₂ -g-EP/PTFE	446.6	51.8	106.7
1.0 wt% SiO ₂ -g-EP/PTFE	574.9	75.2	104.7
1.5 wt% SiO ₂ -g-EP/PTFE	587.3	67.5	99.3
2.0 wt% SiO ₂ -g-EP/PTFE	593.5	60.6	94.6

On the other hand, since the composite with 1.0 wt% SiO₂-g-EP has acquired balanced mechanical properties, the subsequent creep study is focused on the composites with the same filler fraction (*i.e.* 1.0 wt%) no matter whether the particles have been treated or not.

Figure 5 illustrates typical time dependences of tensile creep of directional films of PTFE and its nanocomposites. In general, a creep strain versus time curve of plastics material consists of four stages^[24]: (i) initial rapid elongation, which is independent of time and is attributed to the elastic and plastic deformation of the polymer; (ii) primary creep, the creep rate starts at a relatively high value, and then decreases with time; (iii) secondary creep and (iv) tertiary creep, in which the creep rate increases rapidly and final creep fracture occurs. For the PTFE films exhibited in Fig. 5, there is nearly no initial rapid elongation stage on the creep curves. The above-mentioned primary creep is observed as a result of slippage and reorientation of polymer chains under persistent stress. The secondary creep occurs at a rate almost identical to that of the last stage. Unfilled PTFE has higher creep strain than the other materials at the same time level. Creep strain of the directional films of SiO₂-g-EP/PTFE is significantly reduced over the chosen period due to the restriction effect of the nanoparticles. It is noteworthy that SiO₂-g-EP/PTFE system also possesses the lowest creep rate in the primary and secondary stages. In practice, reduction of creep strain and creep rate is very important for PTFE. The above experimental

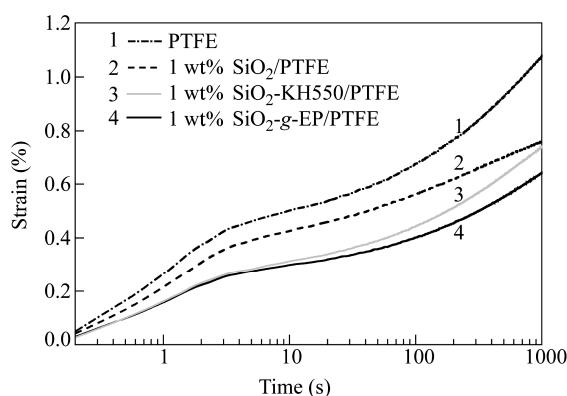


Fig. 5 Typical tensile creep strain-time curves of directional films of PTFE and PTFE nanocomposites

results suggest that a proper design of microstructure and interfacial effect of the nanocomposites would inhibit cold flow feature of PTFE.

In addition to the tensile creep study that is correlated to bulk property, Vickers micro-hardness tester can also be used to characterize surface creep habit of the materials by monitoring loading-unloading curves of the indenter. During the indentation, the load linearly increases for a time of duration, then keeps constant at the maximum force for a constant time, and finally decreases linearly to zero over a time period (Fig. 6a). The maximum depth (depth 1) is reached under the maximal applied load. Due to plastic deformation, residual indentation displacement, depth 2, remains after unloading. Figure 6(b) indicates that the PTFE nanocomposites have smaller values of depth 1 and depth 2 than unfilled PTFE. Table 2 lists the quantitative data and demonstrates that the addition of nano-silica shortens both depths 1 and 2, and improves the hardness because of the hardening effect of silica nanoparticulates. The SiO₂-g-EP/PTFE system has the smallest depth 1 and highest hardness, meaning that SiO₂-g-EP has strong intermolecular bonding to restrict chains movement.

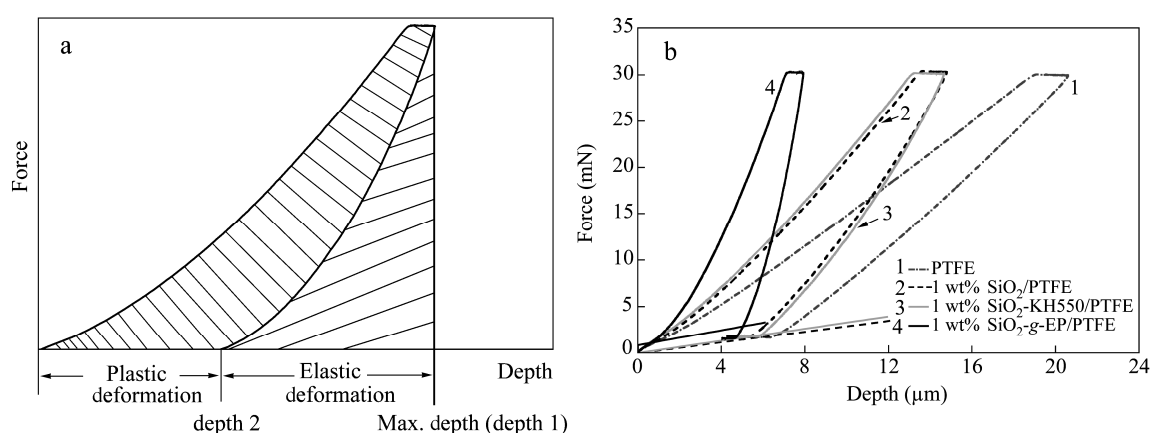


Fig. 6 (a) Characteristic parameters of a pair of loading-unloading curves tested by instrumented indentation and (b) typical loading-unloading curves of directional films of PTFE and PTFE nanocomposites

Table 2. Indentation parameters of directional films of PTFE and its nanocomposites

Materials	Force (mN)	Depth 1 (μm)	Depth 2 (μm)	HV* (GPa)
PTFE	30	20.4	5.3	2.90
1.0 wt% SiO ₂ /PTFE	30	14.4	4.3	3.12
1.0 wt% SiO ₂ -KH550/PTFE	30	14.7	4.9	3.20
1.0 wt% SiO ₂ -g-EP/PTFE	30	8.0	4.2	3.26

*HV: Vickers micro-hardness

According to the operation principle of instrumented indentation, indentation creep is defined as the relative change of indentation depth under constant load (*i.e.* holding segment on force-depth curve). The corresponding creep displacement versus time curves of PTFE and its nanocomposites are plotted in Fig. 7. It should be noted that these curves have been set to zero at the start of the holding period. Similar to the results given in Fig. 5, addition of SiO₂ nanoparticles significantly increases indentation creep resistance of PTFE films. The creep strain and creep rates of the materials are ranked in the following order: PTFE > SiO₂/PTFE > SiO₂-KH550/PTFE > SiO₂-g-EP/PTFE. The results offer more information about the interaction between SiO₂ nanoparticles and PTFE matrix from viscoelastic consideration, manifesting the anchoring effect of the grafted nano-SiO₂ characterized by collecting aligned molecular chains^[20].

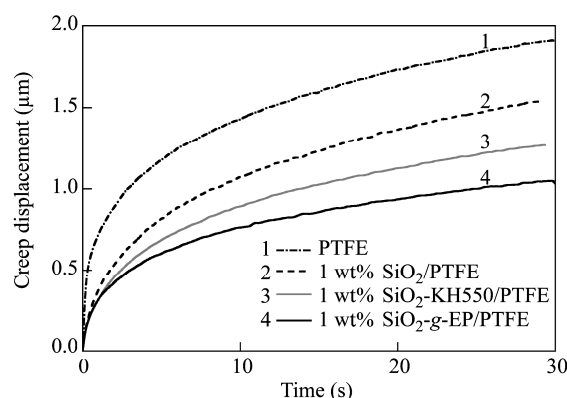


Fig. 7 Typical indentation creep curves of directional films of PTFE and its nanocomposites

Structural and Morphological Characteristics of PTFE Nanocomposites

To have deeper understanding of the significant improvement of creep resistance and mechanical properties of directional films of PTFE nanocomposites, a series of structural characterization were made. Crystalline behavior and crystallinity of the films before and after calender rolling were measured by DSC and WAXD (Table 3). It is found that solid-state rolling has little effect on melting and crystallization temperatures of PTFE films, although orientation induced crystallization increases crystallinity of the directional PTFE films. Nano-SiO₂ slightly accelerates the crystallization rate of PTFE probably due to hetero-nucleation effect of the nanoparticles. With the addition of silica nanoparticles, crystallinity of PTFE films increases, especially for the composites with KH550 treated nano-SiO₂ and epoxy grafted nano-SiO₂. It is worth noting that both directional SiO₂-g-EP/PTFE and SiO₂-KH550/PTFE films have almost the same crystallinity, while the above mechanical tests show that the former has much higher tensile strength and creep resistance than those of the latter. We believe that the stronger interaction between epoxy grafted nano-SiO₂ and amorphous portions of PTFE makes the main contribution, which increases load bearing capability of the bulk material and lowers mobility of PTFE chains.

Table 3. Crystalline characteristics of PTFE films before and after calender rolling

Materials	Processing	T_m (°C)	T_c (°C)	ΔT (°C)	X_c (%)
PTFE	Before calender rolling	329.9	310.7	19.2	58.2
1 wt% SiO ₂ /PTFE		331.9	311.4	20.5	68.3
1 wt% SiO ₂ -KH550/PTFE		332.4	312.9	16.2	70.8
1 wt% SiO ₂ -g-EP/PTFE		334.0	314.4	19.6	75.1
PTFE	After calender rolling	331.4	312.7	18.7	63.7
1 wt% SiO ₂ /PTFE		332.1	314.0	18.1	72.5
1 wt% SiO ₂ -KH550/PTFE		330.3	313.4	16.9	89.7
1 wt% SiO ₂ -g-EP/PTFE		330.1	313.3	16.8	88.9

T_m : melting temperature; T_c : crystallization temperature; ΔT : supercooled temperature;

X_c : crystallinity measured by WAXD

Molecular orientation is also an important parameter related to mechanical performance of polymers. Figure 8(a) reveals that there is no evident difference between unfilled PTFE and the nanocomposites, despite the fact that the degree of orientation of directional films are higher than that of unidirectional films because PTFE chains have been extended after calender rolling. On the other hand, orientation factor, f , of amorphous phase of the composites is slightly higher than that of crystalline phase (Fig. 8b), implying that the treated nanoparticles interact with the amorphous phase of PTFE more effectively. This coincides with our previous observation that inorganic nanoparticles used to be expelled from crystalline region to amorphous phase in the course of matrix crystallization^[25]. Since calender rolling of PTFE films was conducted just above the glass

transition temperature of PTFE, it is in fact in the solid-state processing stage. The crystallite domains preserve their ordering to a certain extent and form long crystallites during shear and compression. In contrast, the amorphous interlayers partly acquire ordering due to high degree of chain orientation. Under these circumstances, SiO₂ nanoparticles would be able to collect and bind more aligned chains in amorphous region.

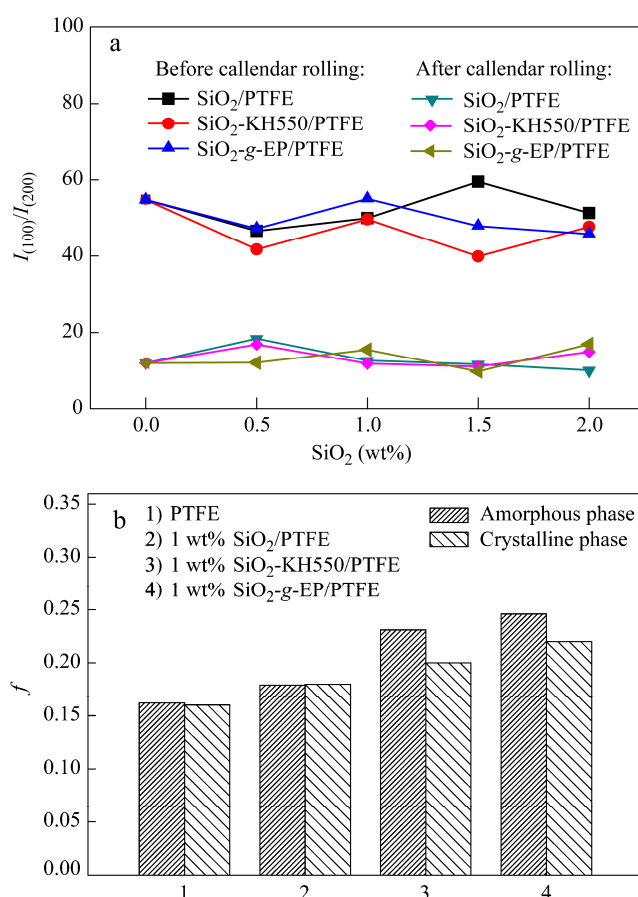


Fig. 8 (a) Degree of orientation of PTFE films before and after calendar rolling determined by WAXD and (b) orientation factor, f , of amorphous and crystalline phases of directional films of PTFE and its nanocomposites

Microscopic morphology observation of PTFE films after the tensile test shows the dispersion of nanoparticles in matrix and another role of the nano-inclusions and orientation. As shown in Fig. 9, fracture in the directional PTFE films starts with longitudinal delamination of layers parallel to the drawing plane and spreads all over the gauge length of the sample. Microfibrils are formed in the final stage of delamination, which would certainly favor the increase of the ultimate strength. Compared to SiO₂/PTFE (Fig. 9b) and SiO₂-KH550/PTFE films (Fig. 9c), the sizes of agglomerated SiO₂-g-EP in PTFE are smaller and more evenly dispersed (Fig. 9d). The evenly dispersed nanoparticles might have helped to split the microfibrils into finer ones in the course of large deformation, which benefits improvement of the composites mechanical properties as a result of reduction of defects concentration. It should be noted that the morphological feature of PTFE manufactured by powder metallurgy technology is porous. Interdiffusion and entanglement between molecular chains of the grafting polymer on silica nanoparticles and matrix would weaken intermolecular repulsion and interfere with mutual sliding of PTFE chains.

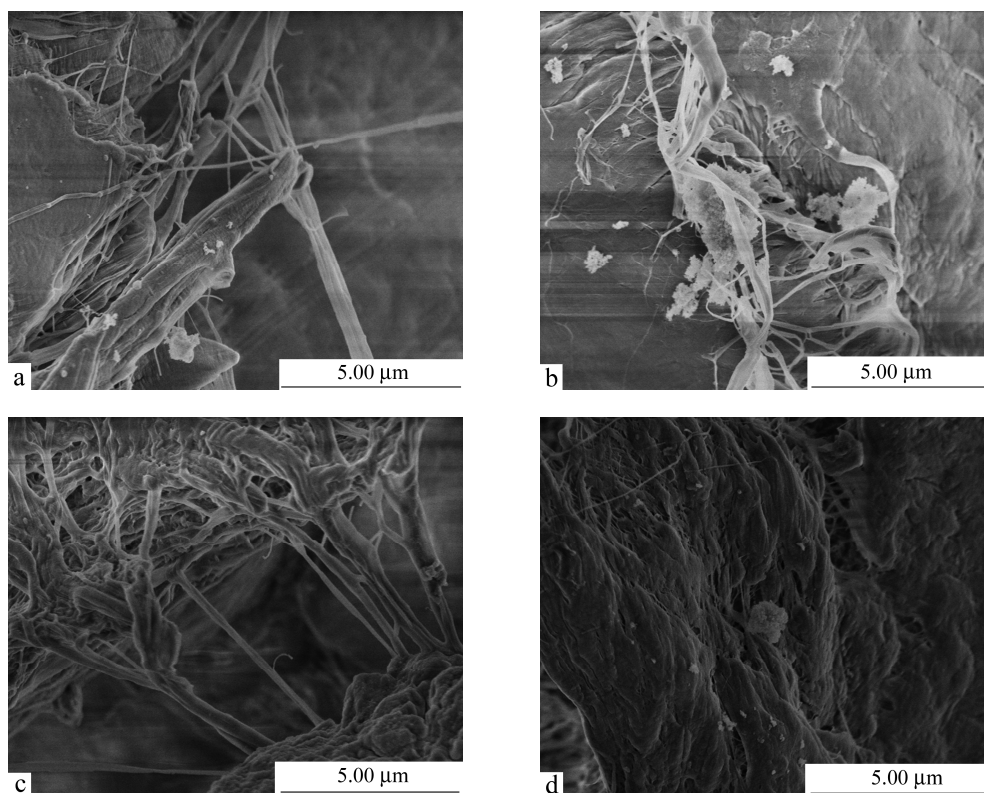


Fig. 9 SEM photographs of fractured surface parallel to tensile direction of directional PTFE and its nanocomposites (nano-SiO₂ content = 1 wt%): (a) PTFE, (b) SiO₂/PTFE, (c) SiO₂-KH550/PTFE and (d) SiO₂-g-EP/PTFE

The specimens were first subjected to tensile test along the calender rolling direction, and then split in parallel to the tensile direction in liquid nitrogen.

CONCLUSIONS

Creep resistance and mechanical strength of directional PTFE films can be improved by adding nano-SiO₂ particles, which is particularly true when the nanoparticles are grafted with epoxy in advance. The grafted nano-SiO₂ effectively interacts with PTFE matrix (mainly the amorphous portion) through molecular entanglement at the interface. Aligned chains are tied to bear higher loading. Intermolecular repulsion and mutual sliding of PTFE chains are restricted. Moreover, the addition of silica nanoparticles increases crystallinity of directional PTFE films and favors microfibrillation of the PTFE matrix during tensile test. As a result, the nano-SiO₂ filled PTFE directional film exhibits reduced creep strain and creep rate, higher tensile strength and hardness. It is believed that further exploration of (i) interfacial design for enhancing interaction between nanoparticles and polymer matrix, and (ii) approaches to prevent nanoparticles from agglomeration at high content would lead to fluoropolymers with much higher performance.

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