

A Review on Creep of Thermoplastic Polymer Composites

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Abstract: Thermoplastic polymer composites (TPCs) have been widely applied as structural materials due to their superior mechanical properties and prolific besides their low costs. On the basis of the nature of viscoelasticity, the deformation of TPCs will inevitably increase with time extension under a constant stress. This phenomenon is called creep, which is followed by irrecoverable damages such as permanent deformation and fracture. Thus, creep can seriously decrease the durability of TPCs and limit their applications. This review discusses the essence and mechanism of creep on TPCs in detail. More importantly, the influencing rules of various factors, such as the molecular structure, fillers, stress, and temperature, and so on, on creep and properties of TPCs are analyzed and summarized. Based on these rules, the negative effect of creep can be controlled reasonably. This review also suggests a suitable methodology for obtaining TPCs with high creep resistance concisely. This is of great theoretical and practical value to fabricate materials with excellent comprehensive properties and achieve the application of them and their products.

Key words: Thermoplastic polymer; Creep; Filler

1 Introduction

Thermoplastic polymer composites (TPCs, composed by polymer matrix and fillers), as one kind of important structural materials, are applied widely around the world^[1]. However, creep of TPCs is inevitable due to their nature of viscoelasticity^[2]. As a result, some irrecoverable damages such as permanent deformation and fracture are often appeared accordingly.

Creep refers to a phenomenon that deformation of materials increases with time extension under a slight stress, usually under room temperature^[3]. In this process, the response to stress is through molecular, structural, and conformational motions. Thus, the materials tend to deform through three stages: the general elastic stage (immediately, completely recoverable deformation caused by the intramolecular alteration of bond length and angle); the viscoelastic stage (time-dependent deformation, aroused by the extension and crimp of chain segments); and the inelastic stage (irrecoverable and time-dependent deformation, related to the slip of the chains mutually).

Many studies have been carried out to reduce the negative effect of creep. Factors such as the synthesis method, processing conditions^[4], the ambient temperature and stress when composites are

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applied^[5], and the dimensions and dispersion of fillers^[6] are considered to be related to the creep of TPCs. The goal of this review is to summarize the creep mechanism and updated progress of TPCs in detail. With the clearly understanding of creep, the negative effect can be expected to avoid and accordingly the methodology to obtain TPUs with high creep resistance can be established.

2 Creep mechanism and impact factors

The essence of creep is the collaborative responses of internal movement units to stress under certain conditions. However, only the slip of chains causes the irrecoverable deformation. If the intermolecular interaction is enhanced, the slip of chains will be restricted. Then, the TPCs can present better creep resistance.

2.1 Structure of TPCs

The structure of TPCs can be classified into molecular structure and aggregate structure. Molecular structure contains the interaction of molecular chains and molecular weight, etc. It has been proved that molecular weight has an influence on creep^[7]. Aggregate structure includes crystalline structure, amorphous structure, orientation structure, and twine structure, etc^[8]. For TPCs, they can get better creep resistance by enhancing the rigidity of chains or forming strongly intermolecular interaction between chains. The former can be achieved by introducing some rigid groups, the latter can be resulted from intermolecular hydrogen bond^[9], hydrophobic associations^[10], metal-ligand interactions^[11], ionic associations^[12], etc.

2.1.1 Molecular structure The results of a study on bio-based shape memory polyurethanes(Bio-SMPUs)^[13] confirmed that the Bio-SMPUs have high crystallinity because of their low density of side chains and good regularity of the polyester chain. Therefore, the slip of chains can be reduced and the Bio-SMPUs express well rigidity. Besides, the hard segment can act as the rein-forcing fillers, leading to well creep resistance. In addition, we have proved that with the non-planar structure, the polymer can obtain higher shape recoverability^[14]. It can be attributed to the rigidity and toughness of the non-planar structure. Moreover, with a rosin-based chain extender, the SMPUs showed wonderful shape recovery at high strain^[15]. As a result, introducing special units to form molecular structures with high rigidity benefits creep resistance.

2.1.2 Aggregate structure Special aggregate structures can be helpful to limit the motions of chains in TPCs, leading to good creep resistance.

For crystalline and semicrystalline TPCs, they have crystalline phase and amorphous phase. The crystalline phase plays a role as cross-link point. So it helps composites to form the analogous crosslinked structure and restricts the motions of polymer chains. Increasing the crystallinity is analogous to increasing the numbers of cross-link points. Thereby, it can increase the resistance to deformation and perform well stiffness and creep resistance. Besides, the small size distribution of crystals and the perfect regular crystalline array also result in good creep resistance. The crystallization of composites is mainly determined by the internal regularity of structure. The more regular structure forms, the better the crystallization characteristics produces.

Different molecular structure is also exhibited in the composite of graphene reinforced ultra-high molecular weight polyethylene (UHMWPE)^[16]. Comparing the pre-reduction method (R-1) and the in situ method (R-2), the former leads to less variation in crystal size and higher crystallinity, so the composite has better network structure and creep resistance; the latter results in thinner graphene

flakes and better dispersion, so it restricts the polymer chains to organize in particular arrangement and hinders the crystallization (Fig. 1).

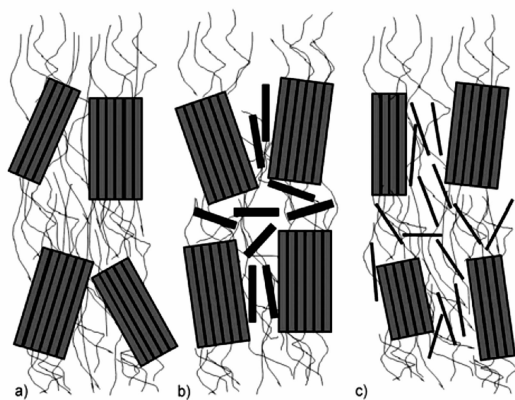


Figure 1 Schematic of proposed morphology for (a) pure UHMWPE film, (b) nanocomposite film by R-1, and (c) nanocomposite film by R-2^[16].

Molecular orientation can also affect creep, especially in amorphous phase. As study^[17] shows, chain orientation in amorphous phase is really strong, because of the effectively interact between nanoparticles and the amorphous phase. Combining the reasons above, aligned chains is tied to bear higher load and intermolecular repulsion, so the sliding of polymer chains is well restricted. These all bring benefit to better creep resistance.

Supramolecular structure is another kind of network structure. It can be reversibly broke and reformed^[18]. For some TPCs, they can form supramolecular structure if fillers can help to create strong interaction between chains. Once the supramolecular structure is formed, the creep resistance will be improved.

For example^[19], high-quality titanate nanotubes (TiNT) were mixed with modified polypropylene (PP*) to be compared with the analogous systems filled with micro-(mTiO₂) and nano-(nTiO₂) particles. The morphology of the composites with TiX in Fig. 2 indicates that TiNT was penetrated by the matrix, leading to form supramolecular structure. The supramolecular structure formed by nucleated crystallization is one reason for inducing improved mechanical properties.

2.2 Fillers

Choosing appropriate fillers can also improve the creep resistance properties of TPCs^[20]. Different kinds of fillers have been used to improve creep resistance of TPCs, including organic fillers (one polymer coated by another^[21], woodflour^[22], fiber^[23, 24], microcrystalline cellulose^[25, 26], etc) and inorganic fillers (CaCO₃^[27], SiO₂^[28], AlN^[29], MgCO₃^[30], BaTiO₃^[31], montmorillonite (MMT) and halloysite nanotubes^[32], nanoparticles^[33], etc). Yang and coworkers^[34, 35] have demonstrated that titania nanoparticles are efficient in reducing the creep compliance of nylon 66. Siengchin^[36] has proved that alumina nanoparticles can effectively reduce the creep compliance of polystyrene. However the creep resistance is not always enhanced by the addition of fillers. If the addition of fillers improve the motion of chains, creep may be easier to occur^[37].

The effect of fillers on improving creep resistance is due to many reasons: (1) as narrated above, they can increase the crystallinity of polymer composites by the hetero-nucleation effect; (2) they can strengthen the amorphous area through hindering the disentanglement and stretching of polymer

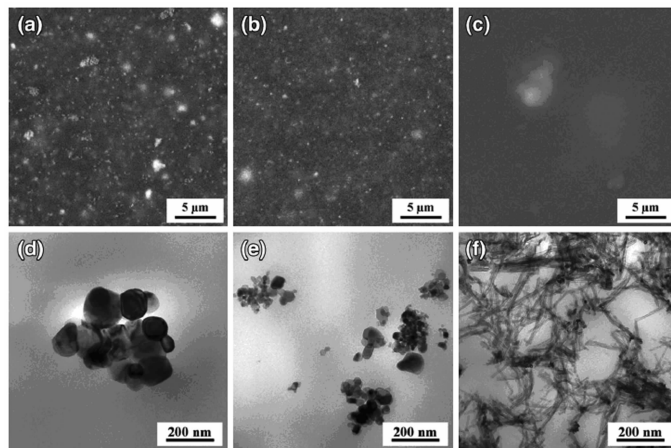


Figure 2 Dispersion of (a, d) mTiO_2 , (b, e) nTiO_2 , and (c, f) TiNT particles in PP^* matrix; (a-c) SEM/BSE micrographs of cut surfaces; (d-f) TEM/BF micrographs of ultrathin sections^[19].

chains; (3) they can provide more support and better distribution of stress and extend the response time of chains by transferring stress from themselves to chains rather than from stress to chains directly. The efficiency of fillers is restricted by their size, structure and surface activity, etc.

PP nanocomposites were reported to be reinforced by particles on base of a layered organoclay (MMT), halloysite nanotubules (HNT), respectively^[38]. MMT occurs in PP copolymer matrix in form of isolated exfoliated lamellas and simultaneously as well intercalated clusters of montmorillonite [Fig. 3(a)]. HNT is equally dispersed in PP copolymer matrix. Great numbers of HNT are oriented parallel with direction of melt flow [Fig. 3(b)]. Thus, the improving influence of HNT to creep resistance is more obviously than that of MMT.

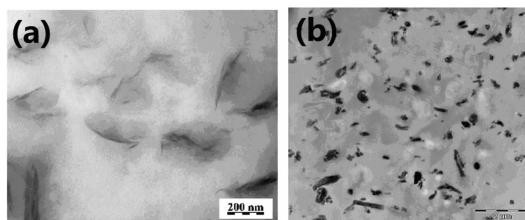


Figure 3 SEM micrographs of PP nanocomposites: (a) PPC/MMT and (b) PPC/HNT^[38].

Siengchin^[6] has examined the effect of nanofillers, sodium fluorohectorite (FH) and boehmite alumina (BA), in a hydrogenated nitrile rubber (HNBR) toughened polyamide-6 (PA-6). FH is preferentially located in the PA-6 phase [Fig. 4(a)]. So it adheres well to PA-6 matrix and can strengthen the amorphous phase. Thus the stretching of the amorphous phase and entangled chains occur with considerable time delay. While BA is mostly located in the HNBR phase [Fig. 4(b)], it just reinforces the HNBR particle by reducing the difference in the moduli between PA-6 and HNBR without affecting the amorphous phase.

To get better dispersion quality and interfacial bonding between fillers and matrix, surface modifications of fillers have been studied^[39, 40]. The ozone functionalization of MWCNT has better dispersion and interfacial adhesion in the thermoplastic polyurethane (TPU) matrix which results in better creep resistance and recovery^[41]. Another study indicates that when the alkalinization, silanization, and impregnation with PE treatments applied to fibers, the fiber-matrix adhesion was

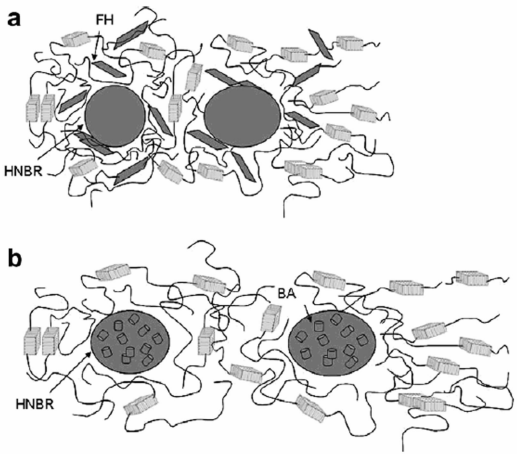


Figure 4 Morphological sketches showing the reinforcing effect of nanofillers; (a) FH and (b) BA^[6].
enhanced, leading to better creep resistance (Fig. 5)^[42].

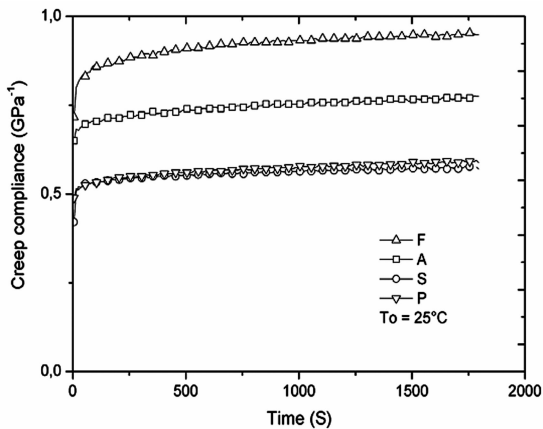


Figure 5 Short-term compliance curves for 30% fiber LDPE-Al treatments F (LDPE-Al and fiqu without treatment), A (fiqu fibers with alkali treatment), S (fiqu fibers with alkali treatment and silanization), and P (fiqu fibers with alkali treatment, silanization, and impregnation with LDPE)^[42].

Besides, the process of blending can also contribute to enhance the interaction of matrix and fillers, it will be helpful to increase creep resistance. In a study of polyoxymethylene/polyurethane/alumina ternary composites, the composites produced by the advanced masterbatch (MBa) technique outperformed those made by direct melt compounding in most of creep properties. Because alumina can get markedly better dispersion with rubber particles through the MBa method^[4]. In another study, different kinds of carbon nanofibers (CNF)/polydimethylsiloxane (PDMS) nanocomposites were obtained^[43]. According to the results (Fig. 6), the ex-situ prepared nanocomposites show prominent agglomeration. However, in the in situ prepared nanocomposites, monomer molecules entrapped in the CNFs tend to deagglomerate during polymerization. As a result, the conventional ex situ nanocomposites exhibit higher value of creep compliance (Fig. 7).

2.3 External factors

2.3.1 Stress Stress can affect creep of composites by affecting the motion of chains. When the load is larger than the yield strength of composites, creep rupture can occur because of the break of chains^[44]. Fig. 8 shows the results of the creep analysis on the original HDPE under different values of

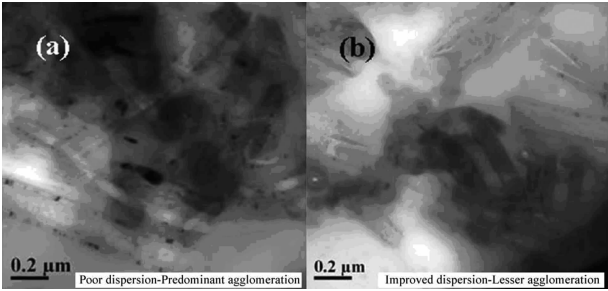


Figure 6 Representative HRTEM images of (a) PD C4E (ex situ prepared nanocomposite with unmodified CNF), (b) PD C4 (in situ prepared nanocomposite with unmodified CNF)^[43].

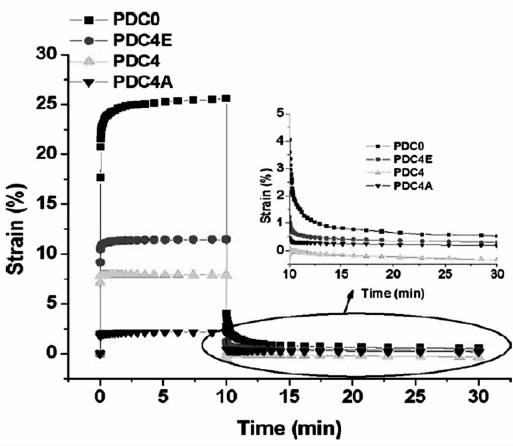


Figure 7 Comparison of strain versus time curves for various CNF loaded PDMS nanocomposites [PDC0 (neat PDMS), PD C4A (in situ prepared nanocomposite with amine modified CNF)] (under a constant stress of 0.025MPa and at 30°C temperature) ^[43].

applied stress at $T=30^{\circ}\text{C}$. At the lowest stress value, $\sigma_0\leqslant0.1\text{MPa}$, the compliance curves obtained with different stress values are not easy to be distinguished and almost superimposed (as show in Fig. 8 (a)). In this stress range, the compliance is constant regardless of stress value. For larger stress value, $\sigma_0>0.1\text{MPa}$, the creep behavior is more affected by stress^[45]. The larger the stress, the more creep compliance it will show.

2.3.2 Temperature Larger creep deformation can be observed under higher temperature, due to the higher mechanical flexibility aroused by the enhanced chains mobility and the increased free volume. In a creep test, a constant load is applied to the specimens, and the variation of deformations with time under different temperatures are recorded^[46]. Fig. 9 shows that at the same value of imposed stress, the strain and creep compliance increase as the test temperature rises.

Generally, creep is affected by temperature and stress simultaneously^[47]. At higher stress level and temperature, creep will be aggravated^[48].

2.3.3 Other factors Other factors, including relative humidity (RH)^[49], ultraviolet (UV) radiation, experimental selected solvent, etc, also have influence on creep of TPCs.

A study on the mechanical properties of PU lacquer films (Fig. 10) shows the strain increases with the increasing RH under the same stress^[50]. Hence, it indicates that the PU lacquer become more flexible with the absorbed moisture serving as a plasticizer. Besides, as the exposure of UV radiation increased (Fig. 11), the elastic region of the stress-strain curve is shortened while the failure stress

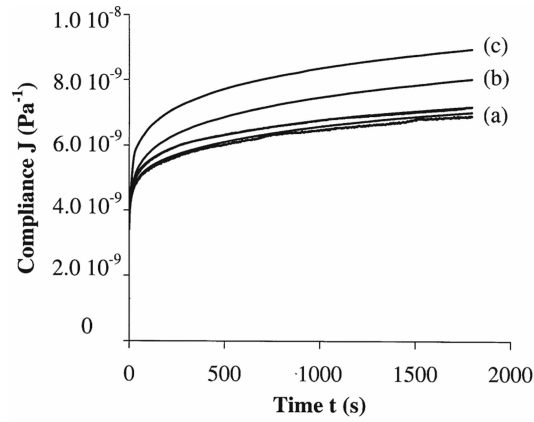


Figure 8 Compliance curves of the original HDPE registered at $T=30^{\circ}\text{C}$ for different values of applied stress: (a) $5\text{ kPa}\leq\sigma_0\leq 0.1\text{ MPa}$, (b) $\sigma_0=0.5\text{ MPa}$, and (c) $\sigma_0=1\text{ MPa}$ ^[45].

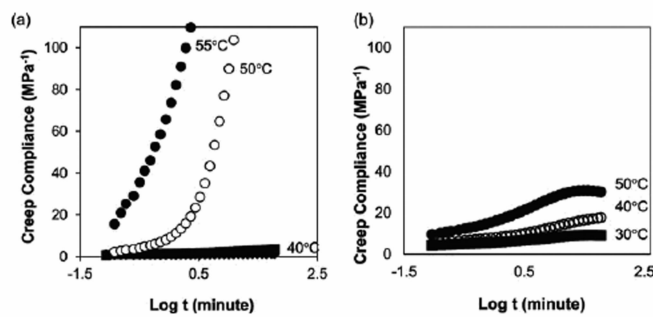


Figure 9 Creep compliance curves in time function at different test temperatures for (a) dry specimens and (b) specimens contain 6% liquid uptake^[46].

significantly increases. As a conclusion, increasing RH can significantly reduce the creep resistance of the PU lacquer, while with the increasing time of UV irradiation, the opposite trends are seen.

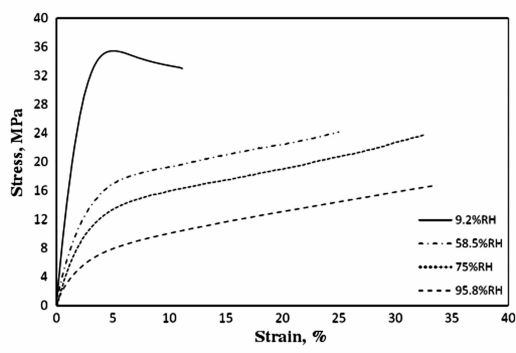


Figure 10 Stress-strain curves from tensile tests at different relative humidities^[50].

According to another study, the creep life of silane-TiO₂/HDPE in saline solution is shortened greatly compared with that in air, while the creep strain remains almost the same (Fig. 12)^[51]. Once stress concentrations on sample surface are enough to break the chemical bond of Ti—O—Si, Ti—OH and Si—OH would form subsequently. In saline solution, the process can be sped up because of the surrounding water, while in air, the forming process is very slow.

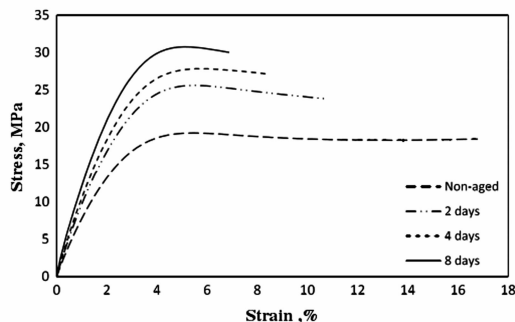


Figure 11 Stress-strain curves from tensile tests on PU samples after UV irradiation^[50].

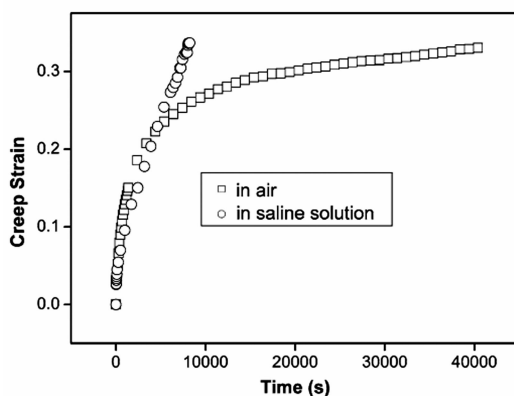


Figure 12 Experimental creep curves obtained in saline solution and air under 45MPa^[51].

3 Conclusion and outlook

The flexibility of chains is the reason for creep, and only the slip of chains causes the irrecoverable deformation. So we can build TPCs with high creep resistance by changing structures of TPCs, such as introducing rigid units into chains or forming strong intermolecular interactions; adding appropriate fillers with suitable method; controlling the external factors during using, etc.

At presents, the research progress on creep of TPCs has been concentrating on fillers modified polymer systems. But the reasons for choosing the fillers are not discussed. Besides, there is no systemic method to build specific TPCs with high creep resistance. Thus, the creep behavior of TPCs needs to be further studied.

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热塑性聚合物复合材料蠕变性能的综述

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摘要:热塑性聚合物复合材料(TPCs)由于其力学性能优异、产量巨大,并具有价格优势,已被作为重要的结构材料得到了广泛的应用。然而,由于聚合物的粘弹本性,其不可避免地会在外力的作用下产生随着时间延长而增加的形变,即蠕变。这种作用将对材料造成永久形变以及不可恢复的破坏,从而限制材料的应用。本文通过对 TPCs 蠕变的研究综述,详细探讨了 TPCs 蠕变的机理特征,分析了各类因素(诸如分子结构、填料、应力、温度等)对 TPCs 蠕变作用的影响规律。在此基础上,简明扼要的分析并提出了有助于获得抗蠕变性能优异的 TPCs 的方法。

关键词:热塑性聚合物; 蠕变; 填料

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