Epoxy thermoset resins with high pristine thermal conductivity

Ying Lin, Xingyi Huang, Jin Chen, Pingkai Jiang
Shanghai Key Laboratory of Electrical Insulation and Thermal Ageing, Shanghai Jiao Tong University, Shanghai 200240, People’s Republic of China
E-mail: xyhuang@sjtu.edu.cn

Abstract: Heat dissipation becomes a critical problem because of the miniaturisation and the increase of power density in electronic devices and electric equipment, which calls for electrical insulating materials with high thermal management capability. Epoxy thermosets have been widely used as electrical insulating materials, but suffer from their low thermal conductivity. This study reviewed the research progress on the development of epoxy thermosets with high pristine thermal conductivity. First, the thermal conduction mechanism of polymers was briefly introduced. Second, the approaches used to enhance the thermal conductivity of epoxy thermosets were summarised, which mainly dealt with the formation of microscopically anisotropic but macroscopically isotropic structure in the epoxy thermosets. Third, the applications of high thermal conductivity epoxy thermoset resins were reviewed. Finally, the review provided the existing challenges and the future directions for the development of epoxy thermosets with high pristine thermal conductivity.

1 Introduction
Polymeric electrical insulation materials have been widely used in Electronic devices and electrical equipment because of their light weight, flexibility, ease of processing and most importantly, low cost. Epoxy resin, one of the most important thermoset polymers, has been used as electrical insulating materials for more than 70 years [1]. Now epoxy resins serve as electrical insulation from the smallest printed circuit board (PCB) to the largest generators. Currently, the rapid development of electrical and electronic technology has resulted in an increase of power dissipation as well as escalation of heat flow at the devices due to the requirement of miniaturisation in components [2]. This calls for high-thermal conductivity electrical insulating materials for improved thermal management capability. However, most of conventional epoxy thermoset resins are amorphous polymer, typically possessing quite low thermal conductivity at a range of 0.17–0.21 W m⁻¹K⁻¹ [3], which cannot fulfil the thermal management requirements of high power in a small space. This review article summarises the recently development of novel epoxy thermoset resins with pristine high-thermal conductivity, and aimed to raise new ideas to design and synthesise new thermally conductive epoxy resins for thermal management applications.

For the sake of discussion, the term ‘epoxy resin’ is defined as uncured materials, which usually consist of epoxy, hardener and other additives, while ‘epoxy thermoset’ will refer to cured polymers.

2 Importance of high pristine thermal conductivity of epoxy thermosets
Bulk epoxy thermosets were rarely used in practical electrical insulation applications. In most cases, epoxy composites were utilised because of their excellent comprehensive properties [4]. For increasing the thermal conductivity of epoxy composites, thermally conductive filler such as oxide (SiO₂, MgO, Al₂O₃) and nitride (AlN, BN, Si₃N₄) particles were usually added into the epoxy thermoset matrix to prepare composites [3, 5–11]. A simple parallel model shown in (1) can be used approximately to evaluate the contribution of pristine thermal conductivity of epoxy matrix on the thermal conduction enhancement of the composites [12, 13]:

\[ k_c = (1 - \phi)k_e + \phi k_f \]  (1)

where \( k_c \) and \( k_e \) are the thermal conductivity of polymer composite, epoxy thermoset matrix and filler, respectively. \( \phi \) represents the filler volume fraction. One can see according to (1) that at a fixed filler loading, a resin matrix with higher thermal conductivity can more effectively increase the thermal conductivity of the composites. Recent research by Giang and Kim [14] on the epoxy/alumina composites documented that the influence of thermal conductivity of epoxy thermosets on the thermal conductivity of composites. As shown in Fig. 1, the thermal conductivity of the composites is dependent on the thermal conductivity of epoxy matrix at the same alumina loading, and resin matrix with higher thermal conductivity are more effective to enhance the thermal conductivity of the composites.

According to (1), the thermal conductivity of polymer composites is highly depends on the content of fillers and a high thermal conductivity enhancement can only be achieved at high filler loadings. However, when the filler loading becomes high, many properties will severely deteriorate, including the processing (e.g. significant increase of viscosity), mechanical (e.g. decrease of toughness, strength and flexibility), electrical (e.g. significant decrease of dielectric strength and increase of dielectric loss) properties and other unique merits of polymeric materials (increase of density). Considering these limitations of the development of thermally conductive composites, epoxy thermosets with high pristine thermal conductivity are highly desirable.

3 Basics of thermal conduction in polymers
In solids, heat transfer may be realised by charge carriers (i.e. electron) or by phonons. Since most polymers are electrical insulators with the absence of free electrons, thus, heat dissipation is mainly determined by phonon transport mechanism [15]. The thermal conductivity (\( \kappa \)) of polymers can be obtained theoretically by Debye equation [16, 17]

\[ \kappa = \frac{C_p \nu l}{3} \]  (2)

where \( C_p \) denotes the specific heat capacity per unit volume, \( \nu \) is the average phonon and \( l \) denotes the phonon mean free path. \( l \) of most polymers is an extremely small constant due to numerous defects which cause scattering of phonons in amorphous states.
The heat would be transferred slowly due to tortuous path and scattering is caused by polymer structure itself. Heat reaches the surface of polymer chain, and then spreads to the adjacent atom, and to the next one, and so on. Therefore, most polymers exhibit a quite low thermal conductivity at the range of 0.1–0.4 W m⁻¹K⁻¹, as shown in Fig. 2.

The thermal conduction mechanism is quite distinct in amorphous polymers and crystalline polymers [19]. For amorphous polymer, since the existence of numerous defects and random orientation structures in polymer chains, a tortuous path was formed for the propagation of vibrational waves. This random curvature and sequence of bends would severely impact the phonon transmission, promoting phonon scattering [20].

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Crystalline polymers show different heat transfer mechanism. In the highly ordered regions, an individual polymer chain traverses one end of the crystal to another, or highly aligned chains are extended and share a same axial orientation but not necessarily head–tail [21]. In these regions, the polymer chains can act as effective thermal conductors along the axis direction because of the stiff covalent bonding. In order to illuminate the heat conduction mechanism for crystalline polymers, Newton pendulum can be used as an analogy, which can be seen as a crystalline phase in a material [19]. As shown in Fig. 3a, initial kinetic energy transfers quickly from one side to the opposite side in the ball without additional irregular vibration, just as heat spreads rapidly across the lattice atom without phonon scattering. Most crystalline polymers are treated as semi-crystalline systems consisting of crystalline and amorphous regions [22]. The highly ordered structure of crystalline phase can promote phonon transmission, which is helpful to the thermal conductivity enhancement, whereas the disordered phases with many defects contribute greatly to the so-called ‘thermal resistance’, which cause phonon scattering, slowing the thermal conduction significantly.

The introduction of highly ordered structures in polymer chain can promote phonon transmission, and thus this strategy has been known as the main method to enhance the intrinsic thermal conductivity of a polymer [12, 23, 24]. In the following, we briefly review the recent progress of the development of thermally conductive epoxy thermosets by the introduction of highly ordered structures. Fig. 4 depicts the thermal conductivity of various epoxy thermosets, including both amorphous and liquid-crystalline ones. The thermal conductivities of conventional epoxy thermosets are in a quite low range of 0.17–0.21 W m⁻¹K⁻¹ since they are mainly isotropic amorphous [27, 30–32], while the crystalline ones exhibit higher thermal conductivity up to 1.05 W m⁻¹K⁻¹, which is almost 5.5 times that of DGEBA/DDM epoxy thermoset. In addition, the epoxy thermosets with different mesogenic groups display various thermal conductivities, which can be affected by many factors, such as the size and content of crystalline domain, curing temperature and orientation by external fields. These factors will be discussed in the following section.

4 Epoxy thermosets with highly ordered structure

Much attention is focused on introducing highly ordered structure in epoxy thermosets to enhance the heat transport performance, which were realised by combining mesogenic groups with epoxy chains [18, 38–40]. Fig. 5 depicts the AFM and TEM images showing the microstructure of conventional bis-phenol A/MDI thermoset and twin-mesogenic epoxy thermosets, which were prepared by curing the glycidyl ether type diepoxide monomer with DDM hardener. The AFM image shows that the mesogenic epoxy
thermoset exhibits highly ordered lattice structure, and large domains of several micrometres can be observed. It should be noted that there exist no ordered domains in AFM images of the conventional bis-phenol A/DDM thermoset resin, indicating that the highly ordered lattice structures result in high thermal conductivity. That is to say, phonon transport was significantly promoted in the domains where the mesogenic groups were self-arranged lattice structure. As a result, the thermal conductivity of the twin-mesogenic epoxy thermosets can reach as high as 0.96 W m\(^{-1}\) K\(^{-1}\) [33], which is \(\sim 5.0\) folds higher than that of conventional...
bis-phenol A/DDM epoxy thermoset. This value is much higher even than that of HDPE, which possesses the highest thermal conductivity among the commercially available macroscopically isotropic polymeric insulating materials (shown in Fig. 2) [33, 41].

4.1 Role of disorder-to-order transition

Introducing highly ordered structure can increase the phonon mean free path, and thus high thermal conduction enhancement can be expected in epoxy thermosets. Akatsuka and co-authors [33] prepared a mesogen epoxy resin by reacting 4, 4′-biphenol diglycidyl ether (BPE) with curing agent (DDM) (see Fig. 6A for the molecular structure of BPE and DDM). The cured BPE/DDM thermoset exhibited a thermal conductivity at 0.30 W m⁻¹K⁻¹, which was 1.5 times greater than that of conventional epoxy thermoset resin. TEM images show that the self-ordering process (Fig. 6B) of the mesogenic groups (biphenyl) leads to the formation of nanodomains in the BPE/DDM thermoset, which can be observed in Fig. 6C.

By introducing 4, 4′-dihydroxydiphenyl (DHDP) into a biphenyl F epoxy (BFE) thermoset, the microstructure dependent thermal conductivity of the modified thermosets was documented by Huang and co-authors [27]. As can be seen from Fig. 7, the pristine BFE thermoset exhibits a homogeneous and disordered structure. As the DHDP content increases, the microstructure of the modified thermosets shows a disorder-to-order transition from sphere-like nanostructure feature to long-distance ordered nanodomain morphology. Accordingly, the thermal conductivity of the epoxy thermosets increases from 0.21 W m⁻¹K⁻¹ for the pristine BFE thermoset to 0.4 W m⁻¹K⁻¹ for the BFE thermoset with 25 wt% DHDP. The ordered microstructure formed in the modified epoxy thermosets originates from the strong π–π interaction among the biphenyl groups introduced in the epoxy molecular, which should result in suppressed phonon scattering.

4.2 Role of mesophase size and content

Since the thermal conduction is significantly enhanced in the ordered mesophase (liquid crystal) domains, the thermal conductive performance of mesogenic epoxy thermosets should be associated with the size and content of mesophases. Actually, a strong connection between the size and content of mesogen domain in epoxy thermosets exists and these two factors sometimes work together on thermal conductivity due to the ‘thermal percolation’ [12]. It was found that thermal conductivity was associated with the ratio and size of anisotropic structure (i.e., liquid crystal domain) in the epoxy thermosets. As shown in Fig. 8, when the maximum diameter of the liquid crystal domains is more than 400 nm, the thermal conductivity increased with the content of anisotropic structure (the ratio <50 vol%), especially when its content exceeding the threshold (25 vol%). When the maximum diameter of anisotropic structure is less than 400 nm, however, only a minor enhancement of thermal conductivity can be achieved.
Therefore, there should exist a threshold of liquid crystal size and content in determining the thermal conductivity enhancement. Akatsuka et al. [42] also found there is existence of a threshold of mono-mesogenic (bipenyl) group content in the thermal conductivity enhancement of mono-mesogen/DDM thermosets system. One can see from Fig. 9 that the thermal conductivity shows a marginal enhancement when the bipenyl group content is lower than the threshold (about 35 wt%), but starts to increase as the bipenyl group content increases to about 50 wt% from the threshold. When the bipenyl group content exceeded 50 wt%, no apparent thermal conductivity enhancement can be observed. This may be explained by the facts that the size of the liquid crystal domains formed in mono-mesogen epoxy thermosets are small when the bipenyl group content is lower than the threshold.

4.3 Role of curing temperature

The morphology/microstructure of epoxy thermosets may be significantly affected by the curing conditions. Some mesogen-containing epoxy monomers may form crystal-like structures in thermosets during the curing process [12]. It is worth noting that the curing condition of the mesogenic epoxy is also considered as a vital factor for the thermal conductivity enhancement in terms of the correlation between liquid crystalline phase transition and curing state [43, 44]. Generally, the reaction rate is proportional to the curing temperature, thereby when the curing temperature becomes too high, the curing reaction of epoxy may proceed faster than the formation of liquid crystal domains, finally resulting in an isotropic amorphous thermoset [12, 38].

Song et al. [38] have studied the effect of the curing temperature on thermal conductivity of mesogenic epoxy thermosets. It was found that many spherulite crystals were formed during the curing process and the spherulite size is closely associated with the curing temperatures. As shown in Fig. 10, with the increase of curing temperature from 100 to 140°C, the size of spherulites decreased obviously. Specifically, there are no spherulites at a high curing temperature of 140°C. Accordingly, the epoxy thermoset possesses the lowest thermal conductivity of 0.22 W m⁻¹K⁻¹. The epoxy thermoset cured at 100°C exhibited the highest thermal conductivity of 0.33 W m⁻¹K⁻¹ because of the formation of spherulites with the largest size.

4.4 Role of mesogenic group orientation

For the mesogenic epoxy thermosets, much larger thermal conductivity can be observed when the content of highly ordered structures becomes higher, which should be attributed to suppressed phonon scattering in the highly ordered area [33]. Apart from increasing the area of ordered structure by increasing the content of mesogenic groups, the improved orientation of mesogenic groups may further increase the degree of order, resulting in further enhanced thermal conductivity. Since the liquid the mesogenic groups can be easily oriented by magnetic field [36, 45–47], mechanical stress [46] and electrical field, resulting in alignment of mesogen groups through the direction along the applied field [12, 45, 48], further enhanced thermal conductivity may be expected in such cases.

The effect of magnetic field on thermal conduction property of liquid crystalline DGETAM/DDE epoxy thermosets had been documented by Harada et al. [36]. It was shown from Fig. 11 that in the direction along the applied field, the thermal conductivity of the epoxy thermoset increases with the increase of magnetic field strength. The thermal conductivity increased to about 0.8 W m⁻¹K⁻¹ in the 2 T magnetic field strength from 0.43 W m⁻¹K⁻¹ of the untreated thermoset sample. Further increase of magnetic field results in marginal enhancement of thermal conductivity, indicating that the order of degree cannot improve after a critical magnetic field strength. The much higher thermal conductivity in the direction along the applied field should originate from the alignment of mesogenic groups along the magnetic field, as shown in Fig. 11, which led to smectic phase on the macroscopic scale. However, it was also found that in a direction perpendicular the applied field, the thermal conductivity decreased to 0.32 W m⁻¹K⁻¹ because of the enhanced phonon scattering along the crossed direction of the main chains.
5 Applications of epoxy resin with high thermal conductivity

5.1 Vacuum pressure impregnation (VPI)

VPI denotes a process through which an electrical unit such as wound coil is completely immersed in a resin tank. The viscosity of the resin should be maintained at a level allowing the resin to be assimilated into all the openings of the insulation system. After the thermally curing process, the impregnated insulation system becomes homogenous. Such as process can allow the insulation have high dielectric and mechanical strength, and improved protection against the containments, water and chemicals. Fig. 12a shows the globally vacuum pressure impregnation of an entire stator of a 300 MW generator in a large epoxy resin tank.

For turbo generators using the indirect hydrogen gas or air cooling systems, the heat generated by the electrical losses in the conductors should be passed through the main mica insulation impregnated by epoxy resin to the coolant (Fig. 12b). The low thermal conductivity restricted the improvement of cooling efficiency of the system and thus the increase of capacity and power density of the generators is limited. The application of high thermal conductivity insulation could not only improves the generator performance without changing of the current coil design and main wall insulation thickness, but also enhance the long-term reliability of the generators. Fig. 12c illustrates the impact of two times of thermal conductivity enhancement of the main insulation on the design of turbo generators with indirect cooling system.

5.2 Thermal interface materials (TIMs)

The continuous increase of power density and miniaturisation of electronic devices had resulted in big challenges in heat dissipation of thermal management materials. In many power devices, the heat produced during operation should be efficiently transferred to the heat sink and dissipated into the ambient environment. Therefore, TIM between the device and heat sink (see Fig. 13) should have high enough thermal conductivity and decreased interfacial thermal resistance. The trend in TIM is to develop materials with high thermal conduction performance, ease of processing, low cost and lightweight. Some epoxy composites can fulfil these requirements and thus they have been used as TIMs. However, the traditional way to enhance thermal conductivity by increasing filler loading becomes inappropriate because the processing, mechanical and dielectric properties will severely deteriorate, as mentioned earlier. In this case, epoxy resin with high pristine thermal conductivity is highly desirable for developing high thermal conductivity TIMs.

This is because that in this case, the filler loading could be significantly decreased in composites with the same target of thermal conductivity enhancement. Fig. 13b shows that for resin matrices, even a small increase of thermal conductivity can result in larger enhancement of thermal conductivity in the composites at comparable filler loading levels. In this case, the filler loading can be reduced and thus the merits (ease of processing, strong adhesion and light weight) of the resin matrices can be maintained [23].
Printed circuit boards (PCBs)

PCBs are designed to mechanically support and electrically connect the electronic components onto an electrical insulating substrate, which typically are layered composites consisting of copper foil and glass fibre reinforced thermosets such as epoxy. The thermal management of PCBs becomes increasingly important because of the continuous increase of power density of circuits and components. Thermal conductivity of the electrical insulating substrates plays a critical role in thermal management, and thus it should be fully considered for PCB design. Using epoxy resins with pristine high thermal conductivity, electrical insulating substrates with ultrahigh thermal conductivity could be produced, which may significantly improve the thermal management of many devices, such as automobile electronics [18, 51].

Other applications

Epoxy composites are also used as electronic packaging materials. For example, integrated circuits are usually encapsulated by epoxy thermosets, which can provide pathways for heat dissipation [52–54]. In addition, epoxy composites have been widely used electrical insulating adhesive for high power devices, solid insulation for gas-insulated switchgears and reactor coil, and insulating for rotating machines, which all require high thermal conduction performance. Therefore, epoxy resin with high pristine thermal conductivity is promising in the future applications of many devices, such as automobile electronics [18, 51].

6 Conclusions

In this review, we introduced the thermal conduction mechanism in polymers, the state-of-the-art progress and emerging applications of epoxy thermosets with high pristine thermal conductivity. For enhancing the intrinsic thermal conductivity of epoxy thermosets, the main approach is the formation of highly ordered structure in the epoxy thermosets by introducing mesogenic groups. The size of liquid crystal domain, curing conditions and orientation of mesogenic groups can dramatically affect the thermal conductivity of the developed epoxy thermosets.

In spite of the achievements made in the past years, many challenges remain in the further development of high thermal conductivity epoxy thermosets with high comprehensive performance. More attention could be focused on the following points.

Dielectric, mechanical, processing and other properties should be comprehensively considered when developing high thermal conductivity epoxy thermosets. For electrical insulation applications, the dielectric constant, volume resistivity, dielectric loss tangent and breakdown strength of the epoxy thermosets should be fully evaluated. When used in VPI systems, the viscosity of the newly developed epoxy resins should be low enough and their shelf life time should be as long as possible.

The research of the correlation between thermal conductivity and microstructure of epoxy composites containing mesogenic groups is still at its early stage. More efforts should be made to reveal the impact of nanostructure and microscopic molecular arrangement on the thermal conductivity of epoxy thermosets.

Controlling alignment of mesogenic groups can tailor the thermal conductivity at relatively low content of mesogenic groups, due to the formation of high-ordered structures. Therefore, it is useful to develop new proper techniques to align mesogenic groups during the practical engineering applications of the epoxy thermosets. Also, it is essential to figure out how external conditions affect the arrangement of molecular chains.

The innovation of processing and synthesis technology is of great importance for achieving the goal of low cost and large-scale production of the epoxy thermosets with high pristine thermal conductivity.

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