The rod-coil block polyimide thermoplastic/ epoxy resin blend

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Abstract: A series of rod-coil block polyimides (*b*-CPI) have been synthesized by introducing Jeffamine D2000 into polyimide chain and completely characterized by FT-IR, NMR and TGA. These rod-coil polyimides can form different pattern by self-assembly, but also can enhance the properties of epoxy resin. Dynamic Mechanic Analysis (DMA) indicated that *b*-CPI can toughen the epoxy resin and improve its thermal endurance obviously. The T_g increased more than 15 ℃with b-CPI as toughen agent. AFM indicated *b*-CPI' dispersed homogeneously in the cured resin as nanoscale granule, which could separate stress, toughen epoxy resin and improve its thermal endurance effectively. After developing in chloroform to remove rod-coil polyimides, micro & nano porous of different scale can be obtained in the crosslinked epoxy film through control of the type and content of block polyimides, and the curing conditions. This is a very facile approach to prepare micro & nano- porous epoxy film, which would find wide applications such as chemical and biological separations, catalysis, photonic devices and micro-electronic materials.

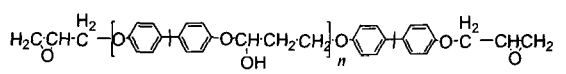
KEY WORDS: Rod-coil block Polyimide, epoxy resin, toughen, Micro&nano-pattern

Introduction

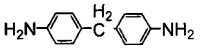
Polyimides (PIs) are high-performance polymer materials with exceptional thermal stability and mechanical and electrical properties, which have been widely applied in the aerospace, electrical, and microelectronics industries. The epoxy resin has some excellent adhesive and mechanical properties, but with bad thermo-stability and brittleness. The thermo-stability and brittleness of epoxy resin can be enhanced by introduction of polyimide into the epoxy resin system.

Experimental

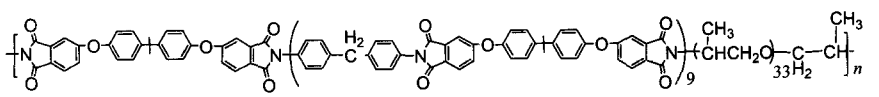
The structures of epoxy resin and b-CPI are shown in Figure 1. Block rod-coil polyimide (b-CPI) was synthesized as following: DDM (5.213g) was dissolved in 100 ml NMP, and the solution was added BISA-DA(15.179g) and pyridine (1.0g). The mixture was stirred at room temperature for 2 h, and then added Jeffamine D2000 (5.85g). The solution was added 40 ml toluene and then heated at 180℃ for 5h. The mixture was cooled to room temperature and poured into water/methanol. The precipitation was filtered and dried in vacuum. Mn=5.4×10^4 (determined using DMF as eluent). FT-IR (KBr): 3 100-3500(O-H), 1778, 1721, 1376 (imide ring)



DGEBA



DDM



b-CPI

Figure 1 Structure of epoxy resin and b-CPI

Results and Discussion

Fig.2 shows the DMA tgδ - *t* curves of epoxy resin using b-CPI as toughen agent. The peak temperature related to T_g increased with the increasing of b-CPI content. In comparison to pure epoxy resin, Tg increased about 15℃ for epoxy resin system with b-CPI content 60%.

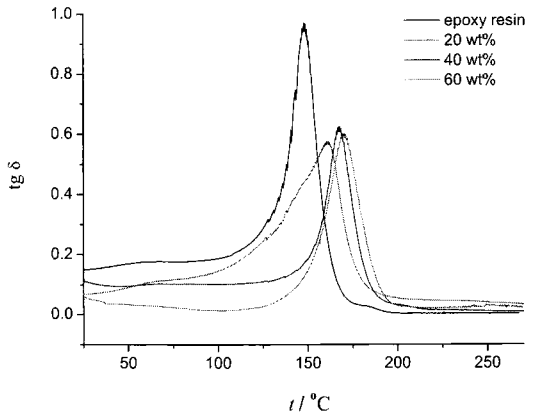
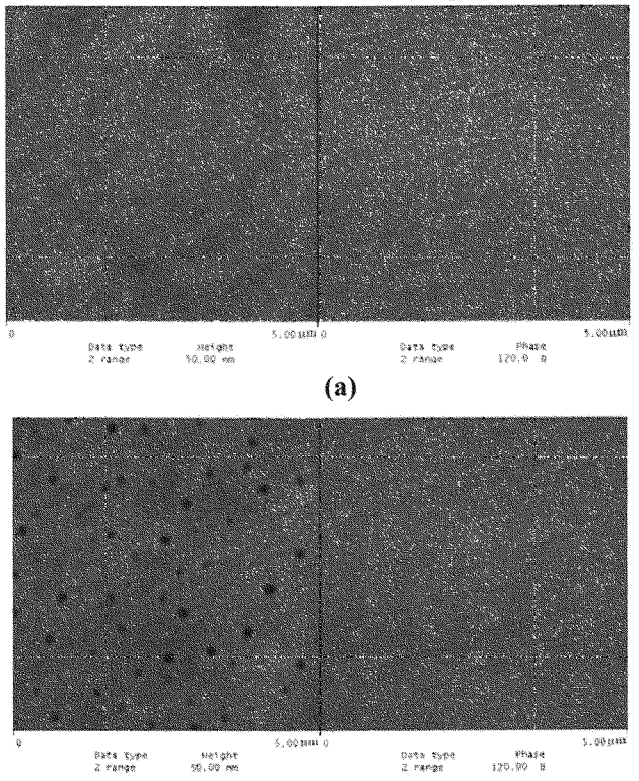


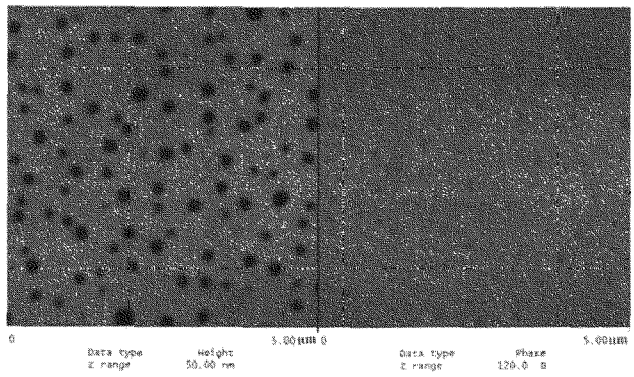
Fig. 2 DMA tgδ - *t* curves of DGEBA/DDM/ *b*-CPI' cured resin

To understand the mechanism for epoxy resin toughened by b-CPI, we studied the epoxy resin/b-CPI systems by AFM. The thin film was prepared by spin-coating, and then developed in chloroform after curing at 150℃ for 4h. From the Fig. 3 (a) and (b), the micro-poles with 300 nm diameter appears after developing, which indicates that b-CPI was dispersed as micro-particle in epoxy resin systems. This should be ascribed to reaction-induced phase separate (RIPS) of epoxy resin/b-CPI system. Before curing, b-CPI can be dissolved in èpoxy resin to form homogeneous phase. The curing reaction between DBGDA and DDM leads to the smaller entropic contribution to the Gibbs free energy, resulting in a positive Δ G. Therefore, b-CPI was extruded from the crosslinked network of epoxy resin, to form the

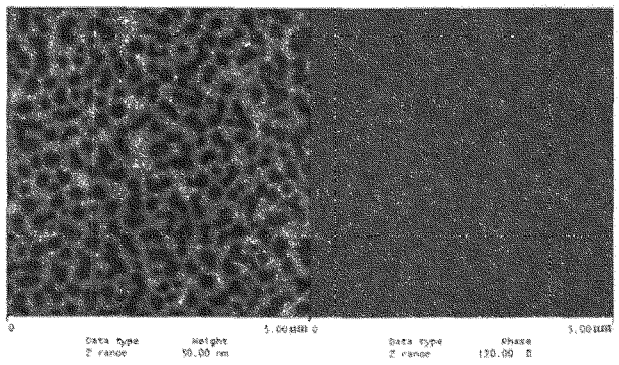


(a)

(b)



(c)



(d)

Figure 3 (c) and (d). It is micro&nano-particles of b-CPI dispersed uniformly in the crosslinked network of epoxy resin that enhance properties of epoxy-resin.

Fig. 3 AFM images comparison of DGEBA/DDM/ *b*-CPI before and after developing in chloroform. Sample: (a) – 20 wt% before developing; (b) – 20 wt% after developing; (c) – 40 wt% after developing; (d)-60 wt% after developing. Left: Height image: Scan size is 5 μm×5 μm×50 nm, Right: Phase image: Scan size is 5 μm×5 μm×120°

Conclusions

By introducing Jeffamine D2000 into the polyimide chain, we synthesized the rod-coil polyimide, Epoxy resin can be toughened by the rod-coil co-polyimide (b-CPI), which can enhance the properties of epoxy resin. After cuing of epoxy resin, b-CPI dispersed uniformly as micro & nano-particles in the crosslinked network of epoxy resin.

Acknowledgement

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