Preparation and Research of High Heat Resistance and Flame Retardance Polyimide Foam

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ABSTRACT Polyimide (PI) foam has excellent performances of heat resistance and flame retardance, so it is widely applied in fields like aerospace, aviation, transportation, wind power generation, etc. This paper discussed performances of heat resistance and flame retardance of PI foam with benzophenone tetracarboxylic acid dianhydride (BTDA), diaminodiphenyl methane (MDA), diaminodiphenyl ether (ODA) and diaminodiphenyl sulfone (DDS) as the main raw materials, methanol and tetrahydrofuran (THF) being the mixed solvents by using the esterification method. The flame retardance and heat resistance of PI foam were characterized by vertical combustion test, limiting oxygen Index (LOI) , differential scanning calorimetry (DSC) and thermogravimetic analysis (TGA) . The research results show that the glass transition temperature (T_g) of PI foam is more than 240 ℃; 5% weight loss temperature (T_{d5}) is above 420℃, limiting oxygen index (LOI) is over 40%. Meanwhile, comprehensive analysis shows that heat resistance and flame retardance of PI foam perform best when BTDA and MDA being the reacted monomer.

Keywords polyimide; flame retardance; heat resistance; foam

Preparation of PI foam

PI foam is prepared by the following steps: firstly, a certain amount of BTDA was put into the mixture solution of THF and methanol, dialkylester diacid was formed after refluxing for 2 hours at 70℃, then the equimolar amounts of MDA was added into the solution and stirred at 80℃ for 3 hours to form polyester ammonium salt (PEAS) precursor solution; secondly, the PEAS precursor solution was heated so as to remove the extra solvent, the PEAS precursor powder would be obtained by using pulverizer; finally, the PEAS precursor powder was spreaded out evenly on the mold and kept the mold in the oven for 3 hours at about 260℃ for foaming, thus PI_{BM} foam is prepared. The preparations of PI_{BO} foam and PI_{BD} foam are as above.

RESULTS

Heat resistance

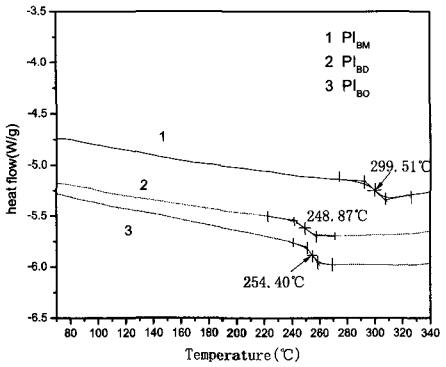


Fig. 1. DSC curves of PI foams

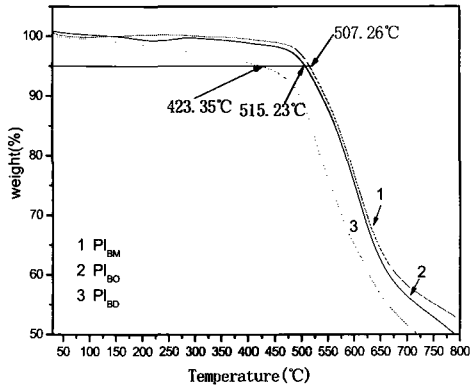


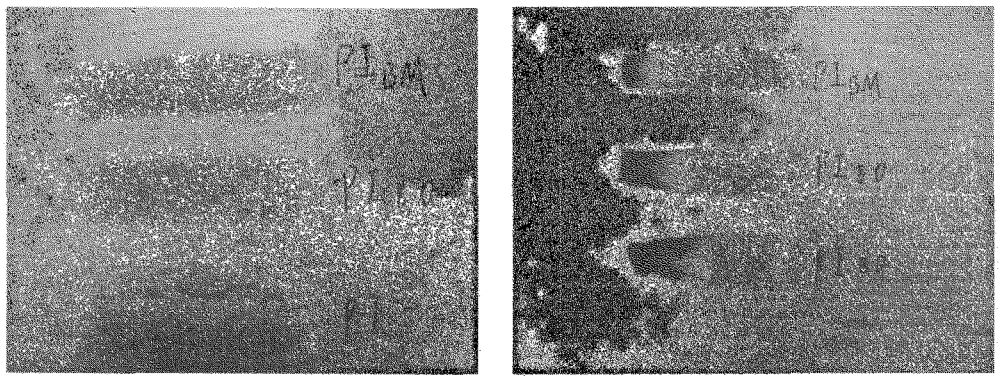
Fig. 2. TGA curves of PI foams

As shown in figs. 1 and 2, glass transition temperature (T_g) and 5% weight loss temperature (T_{5d}) of three different kinds of PI foams are all over 240℃ and 420℃ respectively. among which the T_g and T_{5d} of PI_{BM} are the highest.

Flame retardance

Tab. 1. Flame retardance of PI foams

Foam variety	LOI /%	Vertical combustion		
		Burning time /s	Burning length /mm	dropping
PI_{BM}	46	0	5	None
PI_{BO}	43	0	7	None
PI_{BD}	41	0	11	None



(a) **(b)**

Fig. 3. Vertical burning test of PI foam

(a) pre-combustion (b) post-combustion

Tab. 1. shows that those three kinds of PI foams extinguish immediately while being away from the fire without any flaming dropping, of which LOI are all over 40%. Fig. 3. shows that PI_{BM} has the best flame retardance performance.

CONCLUSION

In this paper, PI foam was successfully prepared based on dianhydride and diamine as the main raw materials by adopting esterification. The glass transition temperature (T_g) of prepared PI foam reaches up to 240℃, 5% weight loss temperature (T_{d5}) is above 420℃, limiting oxygen index (LOI) is more than 40%. Meanwhile, heat resistance and flame retardance of PI foam perform best when BTDA and MDA being the main materials.

Therefore, PI foam is a highly-performanced foam with excellent heat resistance and flame retardance which is widely used in fields like aerospace, aviation, transportation, wind power generation, etc.

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