MECHANICAL PROPERTIES OF SILTEM MODIFIED BISMALEIMIDE RESINS

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Abstract

Mechanical properties such as G_{IC} (critical strain energy release rate), K_{IC} (critical stress intensity factor), tensile and flexural strength of bismaleimide (BMI) toughened by commercially available polyetherimide Siltem STM 1700 were investigated. Dependence of the mechanical properties on the Siltem content was explored. Further investigation of morphological properties by optical microscopy was provided.

1 Introduction

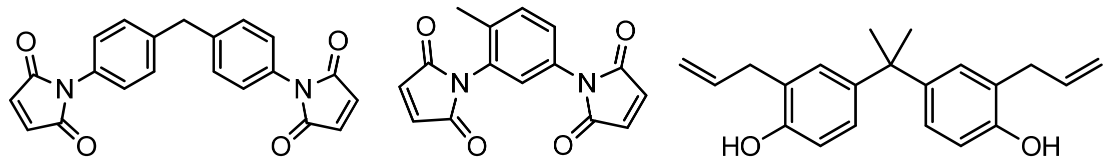
High temperature resins like bismaleimide resins are widely used for deriving high performance composites since their high glass transition temperature (Tg), good tensile properties and excellent processability**.** However, inherent brittle behavior of BMI has restricted their using in high performance application. To improve fracture properties of BMI resins several conventional methods were applied, namely: incorporating in BMI ductile thermoplastics [1-5]; modification by liquid rubber [6]; using of chain-extended monomers [7].

There are limited number of high performance thermoplastic which can be conveniently used as toughening agent due to pure miscibility of thermoplastics with BMI resins [6, 8]. The aim of this work was to investigate the influence of new commercially available type polyehherimides Siltem on mechanical and thermal properties.

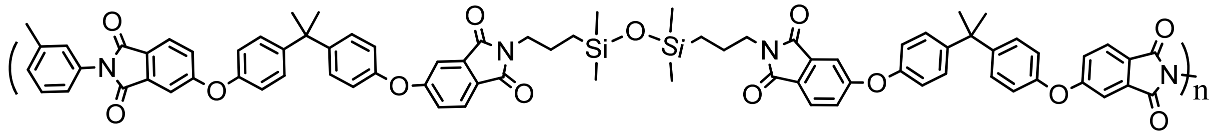
2 Experimental

2.1 Using materials and equipment.

All materials were received from commercial sources, and were used without further purification. Chemical formulas of using materials are shown on fig. 1. Components of BMI blend: 1,1'-(methylenebis(4,1-phenylene))bis(1H-pyrrole-2,5-dione) (MDA BMI), 1,1'-(4-methyl-1,3-phenylene)bis(1H-pyrrole-2,5-dione) (TDA BMI), 4,4'-(propane-2,2-diyl)bis(2-allylphenol) (DABA) were obtained from HOS-Technik GmbH Austria. Siltem STM 1700 was obtained from Sabic Innovative Plastics™ . Mechanical tests were provided using Hounsfield H100KS and H5KS testing machines. The glass transition temperature of the matrix samples was measured in the temperature range between 50 and 300°C on a DMA Q800 (TA Instruments) at a heating rate of $5°C·min^{-1}$. Constant strain amplitude of 0.05% and a fixed frequency of 1 Hz were used. **E´´** peak was assigned as Tg. Micrographs were obtained by Olympus **BX51** optical microscope.



MDA BMI TDA BMI DABA



Siltem

Figure 1 Components of BMI blends.

2.2. General procedure:

A 2000 ml three-necked round bottom flask was charged with 360 g (1.17mole) of DABA and solution of 100 g Siltem in 400 ml CH_2Cl_2. Methylene chloride was distilled off under reduced pressure, vigorous mechanical stirring (1000rpm) to reduce stable foam formation and heating up to 140 °C. After cooling down to 80 °C 252 g (0.7 mole), MDA-BMI 252 g (0.89 mole) were added and then mixture was heated up to 130 °C under reduce pressure to remove all volatile impurities. Clear amber-brown solution was obtained. Then the resin was cast into three stainless steel molds coated by release agent (for tensile tests 450x270x2 mm, for flexural tests 450x270x3 mm, for fracture tests 170x170x4 mm) followed by curing for 3 h. at 180 °C and postcuring for 4 h. at 230 °C in air circulated heated oven. Resulting plates were cut by CNC machine in to samples for mechanical testing according to corresponding ASTM specified below.

3. Results and discussion

The fracture toughness measurements of neat and modified BMI were conducted according to ASTM D5045 [10]. Flexural strength was measured on an unnotched specimen according to ASTM D790 [11]. Tensile strength was measured according to ASTM D638 [12]. The mechanical property values reported are based on an average of at least five tests. Fracture, tensile, flexural and thermal properties of neat and Siltem-modified BMI at room temperature are shown in Table 1.

Siltem content, %	**Tensile strength, MPa**	**Flexural strength, MPa**	**K_{IC}, MPa*m$^{1/2}$**	**G_{IC}, J/m^2**	**Tg, °C (E´´)**
0	106±5	150±2	0.9±0.10	240±35	287
6.5	102±5	168±8	1.34±0.07	480±36	276
10	70±14	140±10	1.36±0.08	540±48	284
15	44±6	70±10	1.3±0.16	570±68	278

Table 1. Mechanical and thermal properties of obtained BMI resins

The K_{IC} and G_{IC} of the neat resin was found to be about 0.9 MPa·m$^{1/2}$ and 240 respectively, indicating that the BMI resin is not very brittle in comparison with the one and two component BMI systems due to low time and temperature cure schedule. All of the Siltem-modified samples displayed increasing of K_{IC} values over that of the neat BMI resin. The higher Siltem content correspond to greater improvement in fracture characteristics (Fig. 2 and Fig. 3). However, when the Siltem content is above 10 wt %, the further toughening effect appears to be negligible meanwhile flexural and tensile strength are significantly decreased. The addition of thermoplastic particles did not affect the Tg of BMI blends. Figure 2 shows the fracture properties of the Siltem-modified resins as a function of modifier concentration.

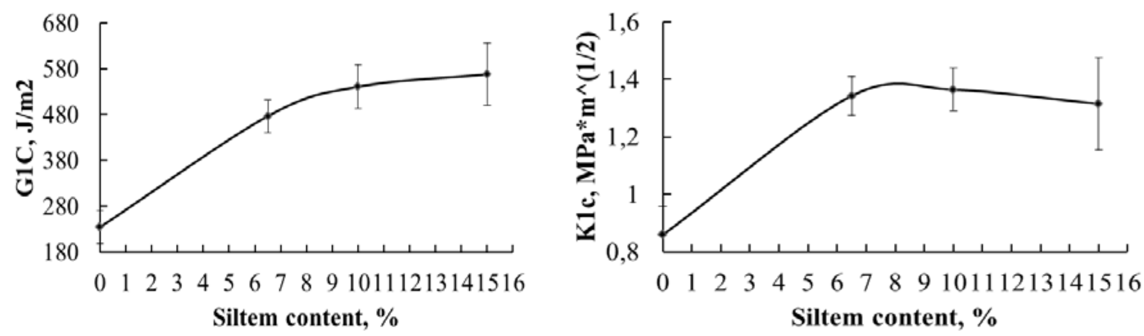


Figure 2 Fracture properties K_{IC} G_{IC} as function on Siltem content

As it is shown on Fig. 2 increasing of Siltem content causes increasing of fracture properties, however at Fig.3 it is shown that increasing Siltem content above 10% causes dramatically decreasing tensile and flexural strength.

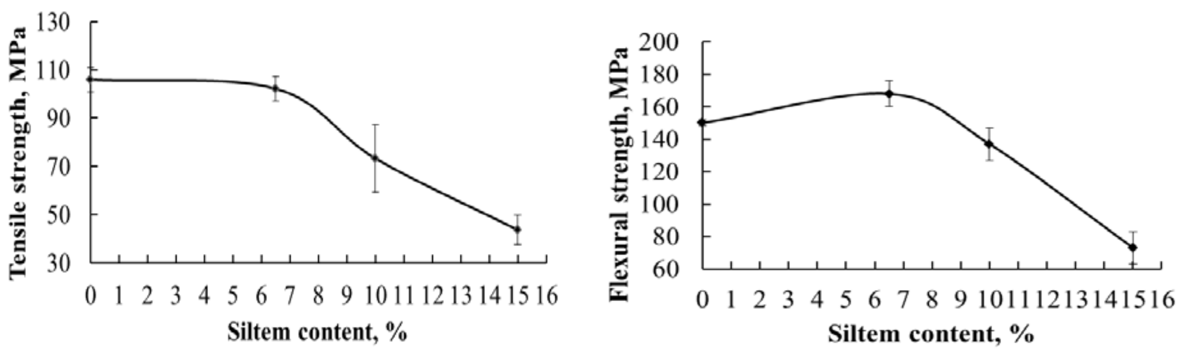
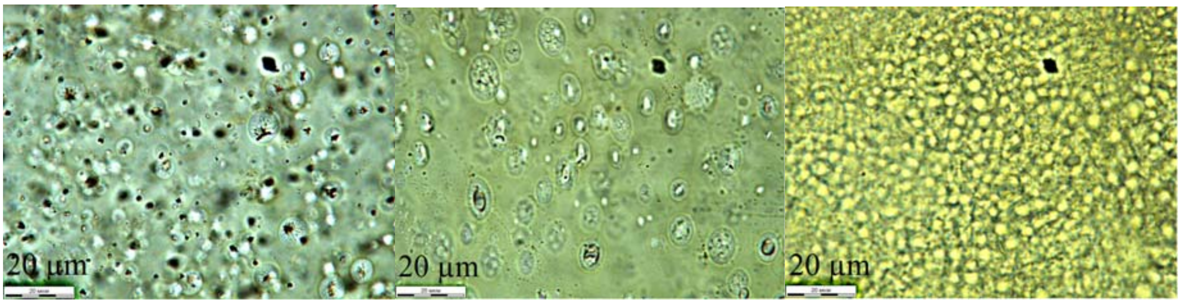


Figure 3 Dependence of tensile and flexural strength of the modified BMIs on the Siltem content

Loss of the strength apparently is caused by different phase separation behavior(Fig.4).



6.5% **10%** **15%**

Figure 4 Optical micrographs of specimens with different Siltem content

Particles size in blends with 6.5 and 10% Siltem content were in range 5 -10 µm. Increasing of Siltem content up to 15% lead to particle size decreasing to the 3-7 µm level. Moreover in this case bicontinuous phase separation with formation of reverse phase zones was observed (Fig. 5). Probably, formation of second continuous phase enriched by thermoplastic causes significant change in mechanical properties.

Figure 5 Bicontinuous phase separation at 15% Siltem content

This proposal confirming by the fact that tensile strength of 15% Siltem modified BMI is close to those of neat Siltem (Siltem STM 1700 tensile strength is 53 MPa [9]).

4. Conclusions

A thermoplastic, Siltem STM 1700, was employed to modify a bismaleimide resin. Mechanical properties such as tensile strength, flexural strength, critical strain energy release rate and critical stress intensity factor were investigated. The results show that fracture properties K_{IC} and G_{IC} can be significantly improved by incorporating of the Siltem without deterioration of other mechanical and thermal properties in case of Siltem content up to 10%. Increasing of Siltem content above 10% causes dramatically loss in tensile and flexural strength. Investigation of morphological properties by optical microscopy shows phase separation with spherical particles formation and bicontinuous phase separation in blends with 15% Siltem content

5. Acknowledgement

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6. References

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