Synthesis, characterization and thermal properties of tetramethyl bipheno-based epoxy resin for electronic encapsulation

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Introduction

Encapsulation (packaging) is the last step of intergraded circuits fabrication. Encapsulating material must protect components from chemicals and mechanical stress, ensure a good electrical insulation, and offer a good thermal conductivity. Owing to the lower cost and easier processing, over 90% of encapsulation materials are polymers. Among these polymers, epoxy resins loaded with 70-90% silica micro-particles are most widely used. Currently, glycidyl ether of o-cresol novolac epoxy(CNE) is the most widely used encapsulating material. However, the high moisture absorption and melt viscosity of the resin have limited its usage in ultra large-scale integrated circuits(ULSI). An epoxy with low viscosity can be loaded with higher amount of silica micro-particles, and thus reduce the moisture absorption, internal stress increase encasulant's conductivity. However, low viscosity epoxy resins are often low functionality with high epoxy equivalent weight, and cause low heat resistance compared with CNE(1).

Mesogene-containing epoxy, such as biphenol-based, when cured at proper condition, can enhaned the thermal and mechanical properties of cured epoxy. Furthermore, due to the hydrophobic properties of methyl group, an epoxy with high substitution will reduce its moisture absorption. In this paper, combining the mesogene and methyl effects, two tetra methyl ketone-based epoxy were synthesized from the epoxidation of tetramethyl biphenol-4,4-difluorobenzophenone. The structure of epoxy resin was characterized by IR spectra and differential scanning

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calorimeter(DSC). These datas were compared with those of commercial tetramethyl biphenol(TMBP) epoxy system.

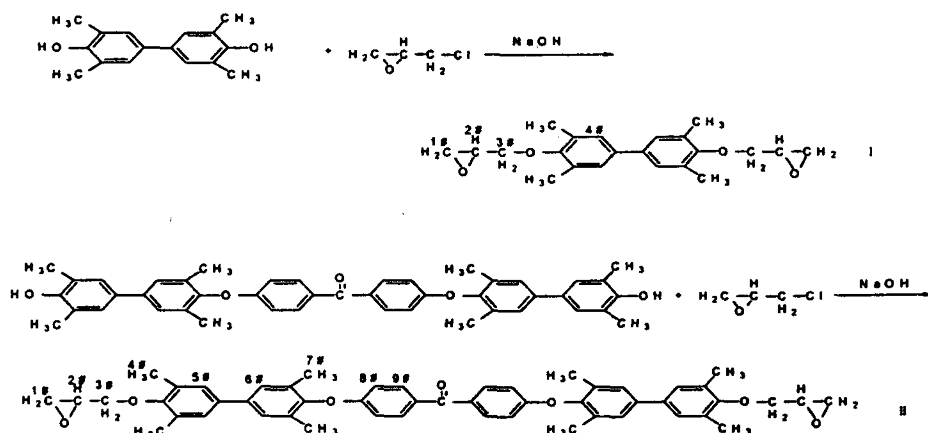
Experimental

Material

Tetramethyl biphenol (m.p 224 ℃) was made by our laboratory. 4,4'-difluorobenzophenone (m.p 105 ℃) was relieved from Yanjin chemical plant. Potassium carbonate (anhydrous, A.R) was relieved from Beijing chemical reagent plant. The reaction solvent 1,4-dioxane was relieved from Tianjin chemical reagent plant.

Synthesis of epoxy resin.

Polycondensation reaction was conducted in a simple 3-neck flask equipped with mechanical stirrer, thermometer and condenser. Ordinal monomer, epichlorohydrin and sodium hydroxide were added to the flask. The reaction mixture was heated to 80 ℃, then reaction mixture was maintained at this temperature for 4 hours. The solution was cooled, poured into water, washed with boiling water twice. And solid product was dried in vaccum.



Characterization

FT-IR was carried out on the Nicolet impact 410 Fourier transform inferred spectrometer. ^{1}H-NMR was carried out on BRUKER AVANCE 500. The thermal behavior of the epoxy resin was investigated by differential scanning calorimetry (DSC) using a Mettler Toledo DSC 821e at a heating rate of 10k/min in nitrogen, melting point was measured in the range 25-300℃.

Results and discussion.

Fig.1 (a) and (b) show the FT-IR spectra of the epoxy resin Ⅰ and Ⅱ, respectively.

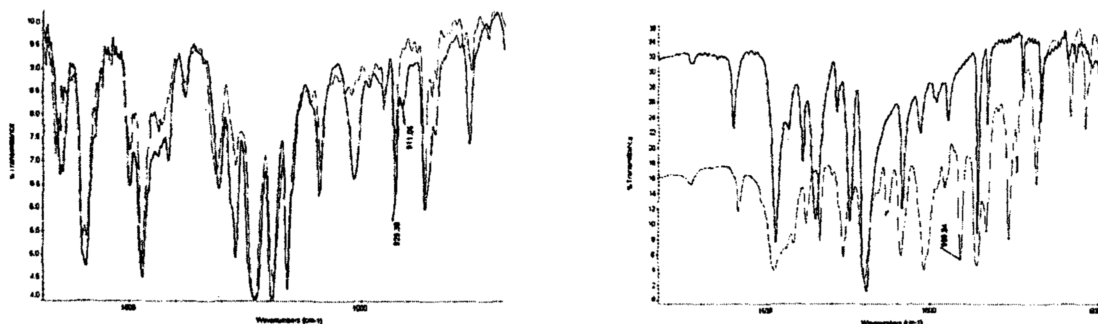


Fig.1 (a) and (b) FT-IR of the epoxy resin Ⅰ and Ⅱ.

From Fig.1 (a), the similar absorption was found between monomer and the epoxy resin Ⅰ except for the stretching of epoxy group at 910 cm^{-1} in the epoxy resin, and the same absorption at 910 cm^{-1} in Fig.1 (b) has been found. To further conform the structure, ^{1}H-NMR was carried out in the epoxy resin Ⅰ and Ⅱ as shown in Table.1 and Table.2.

Table.1 ^{1}H-NMR of the epoxy resin Ⅰ

	CH_3	1#H	2#H	3#H	4#H
δ	2.3	2.7~3.0	3.3~3.5	3.6~4.2	7.2

Table. 2 ^{1}H-NMR of the epoxy resin Ⅱ

	1#H	2#H	3#H	4#H	5#H	6#H	7#H	8#H	9#H
δ	2.73~2.92	3.39~3.41	3.78~4.11	2.20	7.23	7.28	2.73	6.86~6.87	7.76~7.78

From the tests of FT-IR and ^{1}H-NMR the expected productions have been obtained. The thermal properties were tested by DSC. The melting point of the epoxy resin Ⅱ is higher than that of the epoxy resin Ⅰ, which shows that the ketone group in the backbond of epoxy resin has made its thermal stability improved. With the methyl group substitution increasing, the decreasing of moisture absorption was expected.

References

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