



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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**Plastics - Epoxy resins - Determination of degree of
crosslinking of crosslinked epoxy resins by Fourier Transform
Infrared (FTIR) Spectroscopy**

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(English Translation)

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1 Scope

This document describes a method for the determination of the degree of crosslinking of epoxy resins by Fourier transform infrared spectroscopy.

This document applies to the determination of the degree of crosslinking of epoxy resins by Fourier transform infrared spectroscopy.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1

degree of crosslinking

Value indicating the degree of crosslinking of an epoxy resin.

NOTE 1: See formula (4) in Clause 8.

NOTE 2: It is expressed as a percentage.

3.2

height of absorption peak

Maximum distance on the ordinate direction between the virtual baseline and the Fourier transform infrared spectral absorption peak.

NOTE 1: The virtual baseline is simply drawn by connecting the peak start and peak end point by a straight line.

NOTE 2: When a specified peak cannot be completely separated from other peaks, the virtual baseline is drawn by optionally selecting a peak start and a peak end point from among multiple peaks, including the specified peak, and connecting them by a straight line.

3.3

peak height ratio

Value calculated by dividing the peak height of the epoxy group absorption (wave number 900 cm^{-1} to 915 cm^{-1}) by the peak height of the internal standard group absorption, which is not changed by a crosslinking reaction.

NOTE: It is recommended to use the methylene group absorption band (approximately 2930 cm^{-1}) as the internal standard group absorption peak.

4 Principle

It is determined by the peak height ratio of the absorption peak of the crosslinked epoxy resin to the non-crosslinked epoxy resin measured by Fourier transform infrared spectrometer (FTIR).

The height of the epoxy group absorption peak (Pa1) and the height of the internal standard group absorption peak (Pa2) in a non-crosslinked epoxy resin sample (see

Figure 1) are measured by FTIR.

The peak height ratio (X) of the non-crosslinked state is calculated in accordance with formula (1).

The height of the epoxy group absorption peak (Pb1) and the height of the internal standard group absorption peak (Pb2) in a crosslinked epoxy resin sample (see Figure

2) are measured by FTIR.

The peak height ratio (Y) of the crosslinked state is calculated in accordance with formula (2).

The degree of crosslinking is determined in accordance with formula (4) in Clause 8.

Figure 1 -- Spectrum of non-crosslinked epoxy resins

Figure 2 -- Spectrum of crosslinked epoxy resins

5.1 Materials

5.1.1

Epoxy resin, as specified in the test resin system formulation.

5.1.2

Hardener, as specified in the test resin system formulation.

Wave number/cm-1

Wave number/cm-1

5.1.3

Catalyst, as specified in the test resin system formulation.

5.2 Crosslinking conditions

Crosslinking conditions are set according to the characteristics of the resin system formulation and application requirements, including crosslinking temperature and crosslinking time.

NOTE: For examples of resin system formulation and crosslinking conditions, refer to Annex A.

6 Apparatus

6.1

Fourier transform infrared spectrophotometer, with the following characteristics.

- a) an operating wavenumber range. $4000\text{ cm}^{-1} \sim 650\text{ cm}^{-1}$;
- b) the capable cumulative peak count shall exceed 16.

6.2

Micropipette, with capacity $1\text{ }\mu\text{L} \sim 10\text{ }\mu\text{L}$.

6.3

Silicon wafer, with the following characteristics.

- a) single-sided polished monocrystalline silicon wafer;
- b) thickness of $625\text{ }\mu\text{m} \pm 15\text{ }\mu\text{m}$;
- c) total thickness variation (TTV) of $10\text{ }\mu\text{m}$;
- d) total dimensions of the wafer (150 mm wafer);

e) dimensions of the square silicon wafer for testing. 15 mm (± 2 mm) x 15 mm (± 2 mm).

NOTE: The square silicon wafer for testing is cut from a wafer (see 6.3 d) or a finished silicon wafer meeting specifications can be used.

6.4

Analytical balance, with accuracy 0.1 mg.

7.1 Preparation for determination

To ensure the accurate and effective use of the Fourier transform infrared spectrophotometer, please follow the operating manual or other relevant guidelines.

Before the test, check the instrument according to the instrument procedures and confirm that the instrument works properly. Turn on the power and preheat the instrument to a stable state. If necessary, use a standard substance for measurement and confirm that the measured spectral peaks, their repeatability and other values are within the specified range.

7.2.1 General

This document applies to crosslinked epoxy resin systems where two non-crosslinked test samples are determined at the same time and under the same conditions using the same FTIR spectrophotometer to obtain an acceptable peak height ratio.

7.2.2 Test procedure

7.2.2.1

Mix all the components (epoxy resin and other ingredients) of the epoxy resin system thoroughly to prepare a mixture of the non-crosslinked epoxy resin system to be tested.

7.2.2.2

Using a micropipette, immediately measure 5 μL ~10 μL of the specimen from this mixture, and put it on the polished surface of a silicon wafer for testing [see 6.3 e)].

If a micropipette is not applicable, immediately weigh 5 mg ~ 10 mg of the specimen using an analytical balance, and put it on the polished surface of the silicon wafer. Stack a second silicon wafer [see 6.3 e)] on top of the first silicon wafer, with the polished surfaces facing each other. Wipe away any excess sample that has spilled around the edges of the silicon wafer; this sample serves as the test sample.

7.2.2.3

Scan two uncoated silicon wafers that are stacked with the polished surfaces opposite each other [see 6.3 e)] as the background spectrum for the Fourier transform infrared (FTIR) spectrum.

NOTE: These two silicon wafers may differ from those used to prepare the specimen in 7.2.2.2.