

# TECHNICAL INFORMATION

T00142

## Hardening or cross-linking of resins.

It is possible to compare relatively the hardening behavior and hardness of resins by the change of NMR spectral shape and relaxation time reflecting molecular mobility.

#### Hardening behavior of epoxy resin in FRP

To investigate the cure behavior of FRP (fiber reinforced plastic) which impregnated glass cloth with epoxy resin, solid-state <sup>13</sup>C NMR measurements of two samples, that were precured one (R.T.× 20hr) and post-cured one (R.T.× 20hr + 60°C × 2hr), were performed. The <sup>13</sup>C NMR spectra indicated that the epoxy ring was reduced but remained after post-curing. And, the decrease of relaxation time  $T_{1p}^{H}$  values suggested the hardening was unfinished in the post-cured sample.



### Degree of cross-linking of flexible polyurethane resins

In order to compare the degree of cross-linking of the flexible urethane resins A and B having different crosslinking conditions, solid-state <sup>13</sup>C NMR measurements were carried out. Since peak intensities are A<B in <sup>13</sup>C CP/MAS NMR spectra, and these relaxation times  $T_1^{C}$  are A>B, it is considered that the resin B is harder than A.



## 🏘 Toray Research Center, Inc.

T00142構造化学第2研究室20141010 STC:開(20141121)

# Evaluating degradation of polymers.

The change of chemical structure by degradation of rubbers or resins can be evaluated from solid-state NMR spectra and/or relaxation time reflecting molecular mobility.

#### Thermal degradation of silicone rubber

In order to analyze the structural changes due to thermal degradation for cross-linked silicone rubber, solid-state <sup>29</sup>Si NMR measurements were performed.

Polysiloxane structure was reduced by thermal degradation, and the formation of cross-linked structure by condensation was estimated from <sup>29</sup>Si NMR spectra. And, Changes of relaxation time  $T_2^{H}$  suggested constraint of molecular motion was increasing with crosslinking points.



### Degradation of automotive resin parts

To investigate the structural change of a bumper made of polyethylene-propylene copolymer by the accelerated degradation test with sunshine weather meter, the solid-state <sup>13</sup>C NMR measurements were carried out.

While there was no little difference in the spectra for bulk samples, the peaks derived degradation structure were observed in the surface layer of the tested sample.



Surface analysis technique such as XPS and TOF-SIMS, FTIR-ATR is very useful for the analysis of the surface layer. When a cross-section piece is prepared, depth analysis is also possible by these techniques.

Since solid-state NMR is the bulk measurement, chasing the local change in the surface layer part is usually difficult. But, if only a surface part can be shaved off, analysis focusing to the surface part can be conducted.

Since molecular mobility evaluation is possible by solid-state NMR unlike other techniques, there is an advantage which allows to discuss about molecular chain scission and cross-link formation due to degradation.

## 称 Toray Research Center, Inc.