

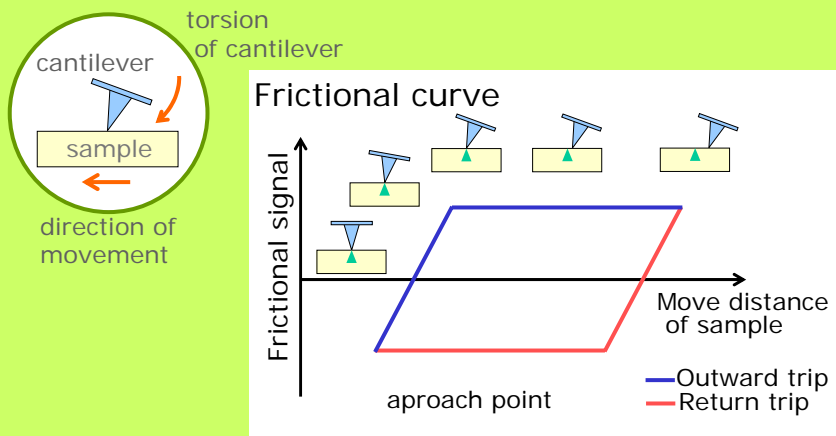
Measurement of epoxy resin thermal curing by the environment controllable SPM

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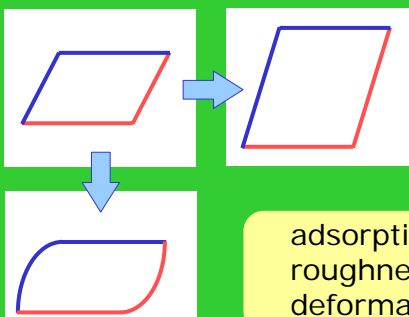
Abstract

Understanding the behavior to temperature is important for developing polymer materials. And in recent years, the needs of physical properties evaluation of polymer thin films or only near the sample surface have been increasing. It is also studied the characteristic by temperature on surface differs from that of inside [1] [2]. And developing material design is expected by evaluating on sample surface physical properties. As a means for understanding the physical properties of polymer materials, the thermal analysis method and the dynamic viscoelasticity measuring method are used widely. However information acquired by these methods is concerned with the whole bulk sample. On the other hand, as the means for understanding the physical properties only on the surface, Scanning Probe Microscope (SPM) is expected. That is because it can observe simultaneously the topography and the physical property on the surface of a sample. Under such a background, we developed the SPM with an environmental control system, "E-sweep" unit. And we have studied the glass-rubber transition temperature on surface of polymer materials, for example, PET [3][4]. In this study, we measured thermal curing temperature and the glass-rubber transition temperature of epoxy resin.

Evaluating the physical property of material by Frictional mode

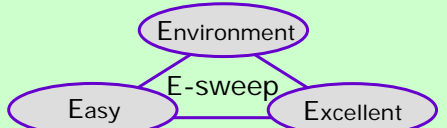


Physical properties of samples can be evaluated from the size and form of the frictional curves.



A changing point of sample is detected by measuring curves continuously under temperature control.

Environment controllable SPM; "E-sweep"

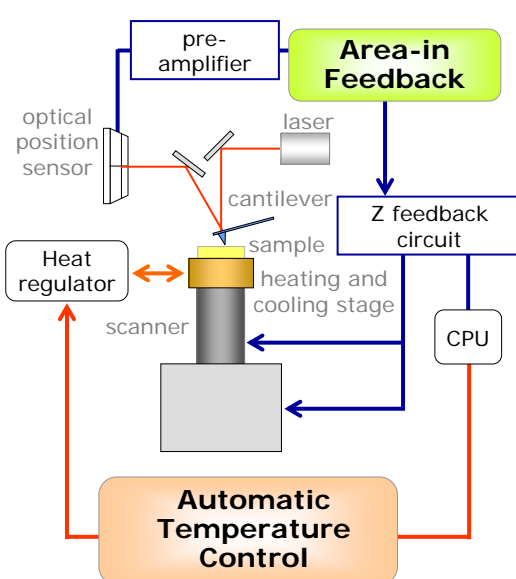


Thermal-sweep

Measure automatically under temperature control

Controllable range; -120 ~ 300
Temperature step; 1 ~

"Thermal sweep" function

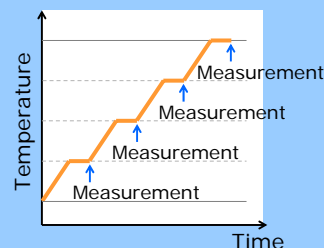


Initial setting

- (1) Sample heating condition
- (2) Frictional curve measurement condition

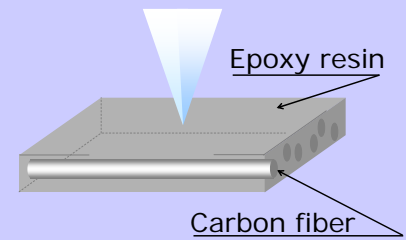
Start

Temperature controlled and Measurement automatically

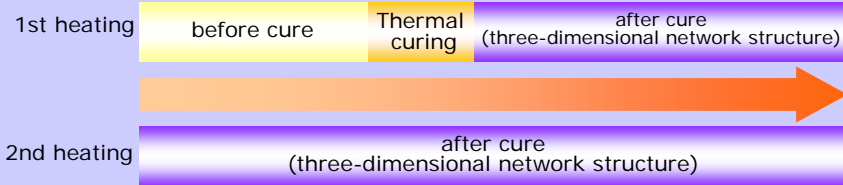


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Sample CFRP (Carbon Fiber Reinforced Plastics)
resin ; Bisphenol A – Epoxy resin
form ; Sheet (t; 0.15mm)



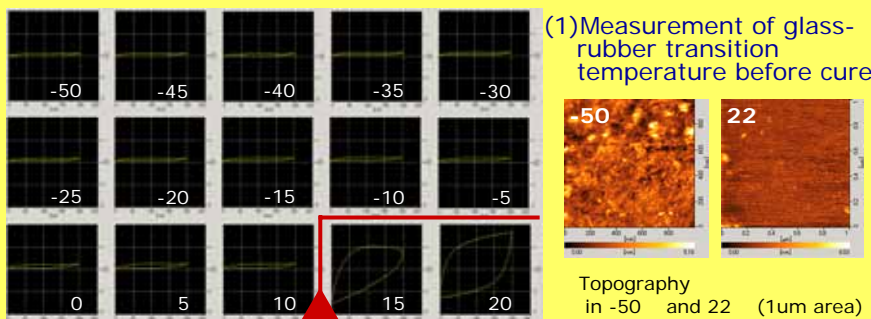
Change of resin while heat process



In this experiment

- (1) glass-rubber transition temperature before cure
- (2) thermal curing temperature
- (3) glass-rubber transition temperature after cure are measured.

Results and discussion



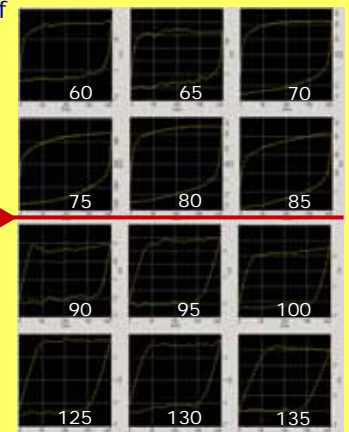
E-sweep

Measurement condition

- cantilever: SI-AF01 (spring constant 0.09N/m)
- scan distance: 200nm
- scan Speed: 1Hz
- system condition: in vacuum ($\sim 0.5 \times 10^{-5}$ Pa)

Change point; 10

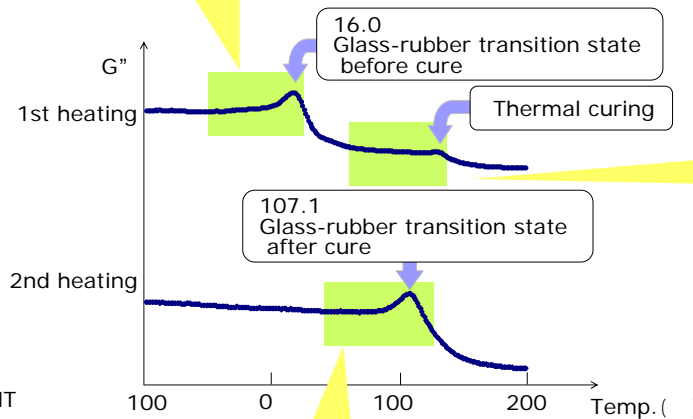
(2) Measurement of thermal curing temperature



Change point; 90

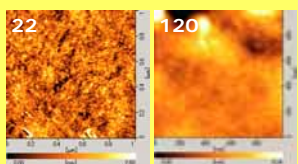
DMS

Measurement condition
temperature ; -100 ~ 200
frequency ; 1Hz



Instrument; DMS6100; SIINT

(3) Measurement of glass-rubber transition temperature after cure



Change point; 105

DMS

Information from the whole bulk sample

E-sweep

Information on the surface of sample

Summary

We successfully specified automatically the glass-rubber transition temperature and the thermal curing temperature on epoxy resin surface by the frictional mode with "Thermal sweep function" in environmental control unit "E-sweep". These results of SPM concur with the results of DMS measurement approximately.

"Thermal sweep function" is powerful tool for investigating polymer surface in nanometer scale.

Reference

- [1] T. Kajiyama, K. Tanaka, I. Ohki, S. -R. Ge, J. -S. Yoon, A. Takahara, *Macromolecules*, 27, 7932 (1994)
- [2] K. Tanaka, K. Hashimoto, T. Kajiyama, A. Takahara, *Langmuir*, 19, 6573 (2003)
- [3] A. Nihei, K. Ando, K. Watanabe, Y. Shikakura, *The Japan Society of Applied Physics*, 51, No.3, 1367 (2004)
- [4] Application Brief SPI No.51 & 52, SII NanoTechnology Inc. (2004)