MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2014A0009 実験課題名 Title of experiment Studies on Dynamcs of Highly Crosslinked Rubber by μSR 実験責任者名 Name of principal investigator Toshiji Kanaya 所属 Affiliation Institute for Chemical Research, Kyoto University	装置責任者 Name of responsible person Yasuhiro Miyake 装置名 Name of Instrument/(BL No.) D1 実施日 Date of Experiment June 19–22, 2014

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Materials and sample preparation

The details of materials used in the present study are summarized in Table 1. We used polybutadiene (PB) as a matrix rubber. Zinc diacrylate (ZDA) and dicumyl peroxide (DCP) were used as a crosslinking agent and a polymerization initiator of butadiene, respectively. The components were mixed on a 6-inch two-roll mill, and they were molded into sheet 1 mm thick at 170 ℃ for 20 min. The glass transition temperature (T_g) of Sample A and B are 168.6 K and 168.1 K, respectively. The Elastic modulus at 293 K of Samples A and B are 4.0 MPa and 104.6 MPa, respectively.

Table 1.: Detail of components of samples used in present study.

	Sample A	Sample B
Polybutadiene / vol%	99.2	83.6
Zinc diacrylate / vol%		15.7
Dicumyl peroxide / vol%	0.8	0.7
Elastic modulus / MPa	4.0	104.6

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Measurements

The Longitudinal field (LF) μSR experiment was performed using a beamline D1 instrument installed at J-PARC in the temperature range between 70 and 280 K. External magnetic field range is between 10 to 2000 G. Each sample is set onto the sample holder. The obtained raw asymmetry data were corrected for the instrumental constant and for the backgrounds from the sample holder.

Results

The LF-μSR spectra of Samples A at 70 K and 280 K are shown in Fig. 1 and 2, respectively. We used the double stretched exponential fitting function (1) as first trial.

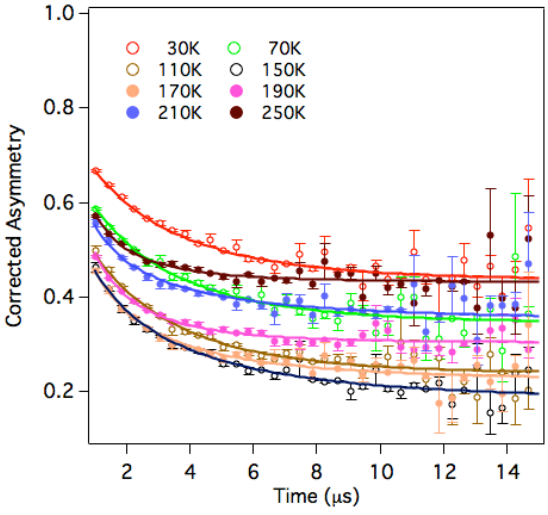
$$f_{(t)} = A_1 \exp\left(-\left(\Gamma_1 t\right)^{\beta_1}\right) + A_2 \exp\left(-\left(\Gamma_2 t\right)^{\beta_2}\right) + B \quad (1)$$

where Γ_1 and Γ_2 are relaxation rates, β_1 and β_2 are the stretch parameters, A_1, A_2 and B are the fitting parameters. Fig. 1 and 2 show the observed and calculated spectra of Sample A at 70 K and 280 K, respectively. The observed μSR spectra were well fitted to Equation 1. According to Pratt et al.[1] and our previous sutdy[2], the dynamics of muon is thought to correspond to the dynamics of crosslinked polymer.

The obtained relaxation rate Γ_1 of Sample A at 70 K and 280 K by fitting is shown in Fig. 3. At 70 K, below glass transition temperature of polymer T_g (=168.6 K), Γ_1 is almost constant between 10 to 500 G, and decrease above 500 G. This result means that the dynamics of polymer is not affected by external magnetic field below 500 G because micro-Brownian motion of polymer chain is frozen below T_g. On the other hand, at 280 K, above T_g, Γ_1 decrease with increasing external magnetic field, suggesting that dynamics of polymer is affected by external magnetic field because the frozen micro-Brown motion of polymer chain is thawed and the polymer chain is able to move actively above T_g. Furthermore, the value of Γ_1 at 280 K is higher than that of Γ_1 at 70 K. This result is also caused by higher mobility of polymer chain above T_g.

[1] F. L. Pratt *et al*., Physica B 289-290(2000)625.

[2] T. Kanaya *et al*., KEK-MSL Report 2013, 2013B0016, in press.



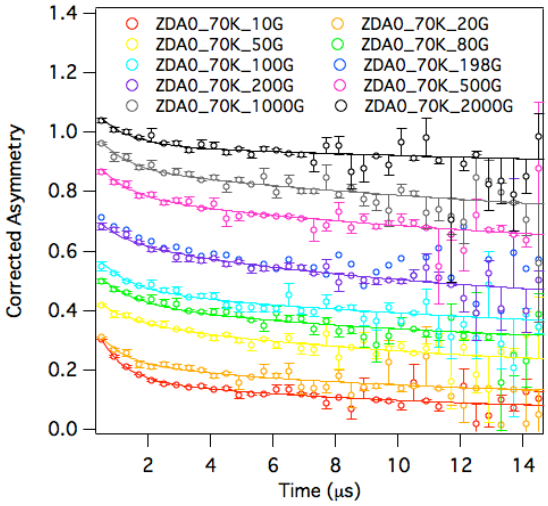


Fig. 1.: LF-μSR spectra of Sample A at

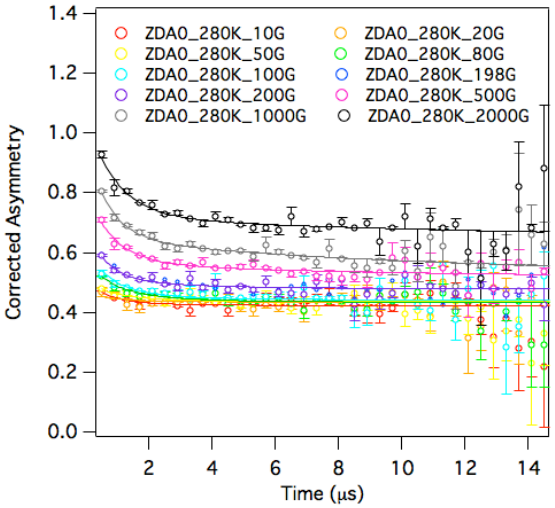


Fig. 2.: LF-μSR spectra of Sample A at 280K. Solid curves are the results of fits.

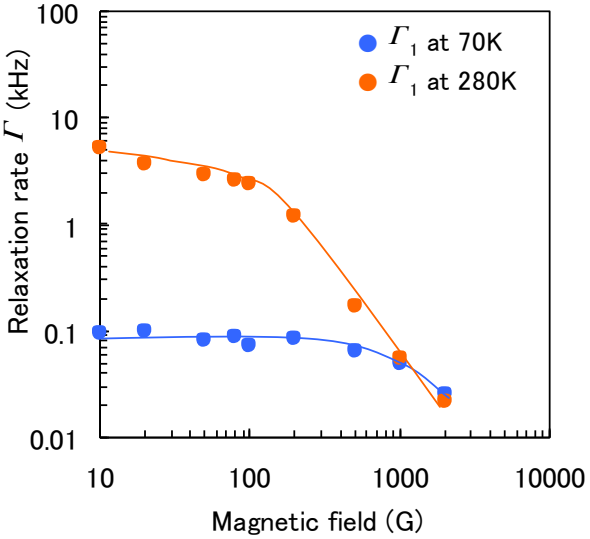


Fig. 3.: Relaxation rate Γ_1 and Γ_2 of Sample A at 70K and 280K as a function of magnetic field.