

A Study on a Visualization of Fatigue Behavior near the Glass Transition Region

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ABSTRACT

This study aimed to visualize crack initiation and propagation of rubber at low temperatures. Typical fatigue behavior such as cracking at only the compressed part of rubber products like dustcovers during oscillation tests at ultra-low temperatures have been reported [1]. Rubber products are usually used at temperatures where good rubber elasticity can be obtained. However, in some cases, they are used near glass transition temperatures at which rubber elasticity is poor. Fatigue failures of rubbers generally occur due to existing defects in the rubber material, and rubber tends to fracture easily near glass transition temperatures due to cracking on the surface of the compressed side of the rubber. The observation of the crack initiation process of rubber is difficult at low temperatures because the fracture patterns on rubber disappear when elasticity is recovered at room temperature. For this reason, in this study, we aimed to observe the fatigue behavior of rubber near the glass transition temperature by testing rubber materials whose T_g is above room temperature.

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INTRODUCTION

Rubber is generally known to be a material with good elasticity in the rubbery plateau region. However, rubber elasticity becomes poor when exposed around the glass transition temperature, at which rubber becomes brittle as glass. Meanwhile recently, due to increased demands for the material in extremely cold countries, use of rubber near glass transition temperatures is also increasing. In some cases, rubber products such as dustcovers are damaged because of deformation at ultra-low temperatures. Hosen et al. reported that cracks occurred at the compressed part of dustcovers during oscillation tests at ultra-low temperatures (Figure 1) [1]. Growth of these cracks is an area of interest to the authors. General evaluation methods of low temperature rubber characteristics include Temperature-Retraction test, Gehman's torsion test, impact test, etc. However, these methods do not show dynamic properties but static properties of rubber at low temperatures. It is difficult to estimate the fatigue behavior of rubber at low temperatures from only these information because the conditions of rubber change during the fatigue test per cycle.

On the other hand, it is known that fatigue failure of rubber consists of two main processes: crack initiation and crack propagation. We therefore decided to observe these processes by oscillating near glass transition temperatures as a model test. There are two possible observation methods for this. The first method is testing rubber whose glass transition temperature (T_g) is below room temperature. With this method,

the rubber specimens need to be tested at low temperature (below room temperature) and observed from outside the thermostat chamber.

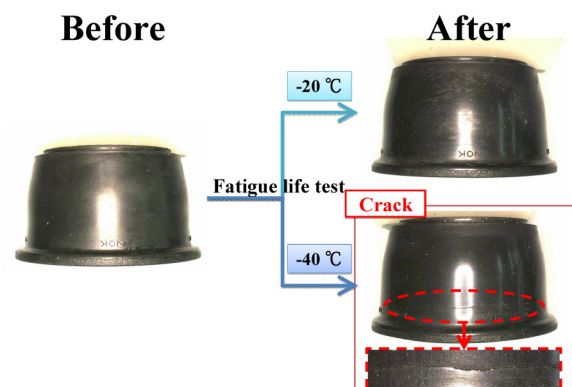


Figure 1. Observed dustcover cracks after low temperature oscillation test.

We initially tried to evaluate ACM rubber specimen whose T_g is around -10°C (see the specimen shape and oscillation test method in Figures 3 and 4 respectively). Cracks appeared at the compressed side of the rubber specimen surface during oscillation test in a chamber set to -10°C . It was not possible to observe the crack initiation process of the rubber specimen in this chamber from outside due to condensation on the chamber window. Observation of the rubber specimen at room temperature after the oscillation test was inadequate because of the temperature-retraction property of the specimen. Signs

of fracture of the rubber specimen disappeared. The cracks on the rubber specimen surface at room temperature do not provide sufficient information to understand the crack initiation process (Figure 2). For this reason, it is difficult to observe the crack initiation process by testing rubber whose T_g is below room temperature.

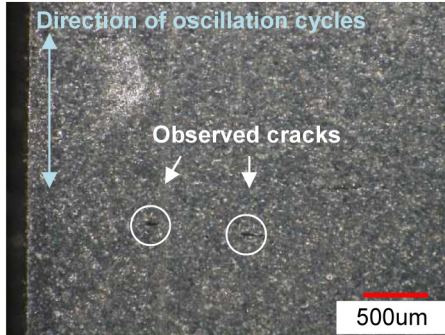


Figure 2. Optical microscope image of ACM specimen compressed side surface after oscillation test at room temperature.

The second method is observing the crack initiation process of rubber at room temperature. For this, rubber whose T_g is above room temperature is required. Observation of the crack initiation process can be facilitated using transparent rubber specimens. Thus, we prepared transparent cross-linked rubber specimens made of SBS and SBBS with glass transition temperatures above room temperature. Furthermore, these specimens enabled observation of crack initiation and crack propagation using an optical microscope at room temperature.

EXPERIMENT

1. Observation of Transparent SBS and SBBS Rubber Materials

Table 1. Details of transparent SBS and SBBS rubber specimens.

	SBS	SBBS-1 (St:57.8wt%)	SBBS-2 (St: 53.3wt%)	SBBS-3 (St 48.5wt%)
Tufprene™ 126S [phr]	100	-	-	-
Tuftec™ P2000 [phr]	-	75	63	50
Tuftec™ P1500 [phr]	-	25	37	50
DCP [phr]	1			
Perhexa 25B [phr]	1			
Roll mixing condition	6 inches two roll mill. @80°C			
Press cure condition	6 min. @180°C			

Polymer

Styrene/butadiene block copolymer (SBS)

Tufprene™ 126S : Styrene cont.: 40wt%, Asahi Kasei Chemicals corporation.

Hydrogenated styrene/butadiene block copolymer (SBBS)

Tuftec™ P2000: Styrene cont.: 67wt%, Asahi Kasei Chemicals corporation.

Tuftec™ P1500: Styrene cont.: 30wt%, Asahi Kasei Chemicals corporation.

Curing agent

Coagent

Tricyclodecane dimethanol Dimethacrylate

DCP: Shin-Nakamura Chemical Co., Ltd

Peroxide

2, 5- Dimethyl- 2, 5- di (t-butylperoxy) hexane

Perhexa 25B: NOF Corporation

Mixing process

Raw materials were mixed with 6 inch two rolls mixing mill preheated at 80°C.

Curing process

Each compound was cured by press molding at curing condition of 180°C/6min.

Specimens for oscillation test

Specimens were prepared by punching into predetermined shapes (Figure 3) from cured SBS and SBBS sheets (Thickness: 2mm). Original dumbbells were made for testing small samples

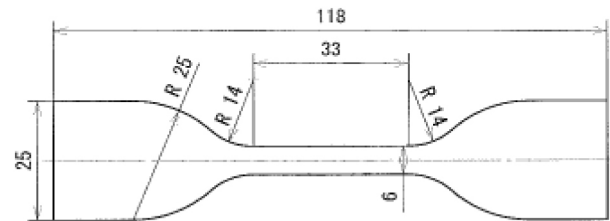


Figure 3. Dumbbell shape for punching rubber specimens.

2. Temperature-Retracton Test

The testing method is based on ASTM1329 but in this review the gauge length specimens were measured using a vernier calipers.

Specimen size: Thickness: 2mm, Width: 2mm, Length: 40mm.

Initial elongation: Specimens were elongated by 25% in thermostat chamber at 120°C.

Measurement of gauge length: Specimens were elongated by 25% elongation at room temperature for 10 minutes. Then the specimens were released from grips. The gauge length of the specimens were measured after putting them in the oven for 5 minutes in turn (25, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120°C).

$$Retraction(\%) = \frac{(l_{25\%} - l_t)}{(l_{25\%} - l_0)} \cdot 100$$

(1)

$l_{25\%}$:gauge length at 25% elongation

l_t :gauge length at each temperature

l_0 :original gauge length(20mm)

Retraction (%) is calculated from Equation (1).

3. Oscillation Test

As shown in Figure 4, the SBS and SBBS dumbbell shape specimens were oscillated using the L5kN Servopulser (Shimadzu Corporation) in cycle tests. They were then observed using an optical microscope at room temperature. The retention rate of loop areas (A_1 , A_{100}) corrected according to the thickness of each specimen was calculated from the loop area obtained using the software provided with the L5kN Servopulser. The ratio of the loop area of the Stroke-Load curve (%) was calculated using Equation (2).

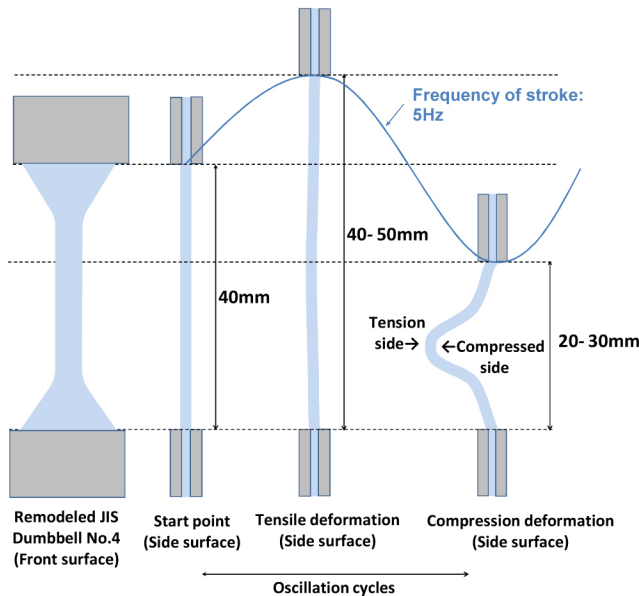


Figure 4. Illustration of oscillated specimens.

$$\text{Retention rate of loop area (\%)} = \frac{(A_{100})}{(A_1)} \cdot 100 \quad (2)$$

A_{100} : Loop area of stroke versus load curve at 100 oscillation cycles

A_1 : Loop area of stroke versus load curve at initial oscillation cycle

4. Dynamic Mechanical Analysis (DMA)

The RheoGel-E4000 (UBM, Tension mode, 2K/min., 10Hz, thickness: 2mm, width: 2mm, length: 20mm, amplitude: $\pm 10\mu\text{m}$) was used for dynamic mechanical analysis.

5. Observation of Specimens

The VH7000 digital video microscope (KEYENCE Corporation) was used for the observation.

RESULTS AND DISCUSSION

1. Evaluation of SBS Compound

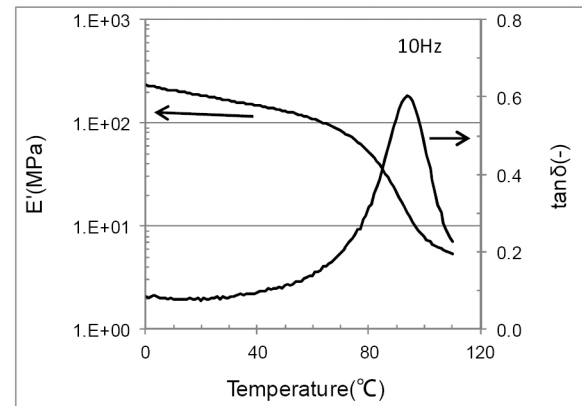


Figure 5. Dynamic mechanical analysis of SBS specimen.

The glass transition region of rubber material generally exists around the $\tan\delta$ peak obtained by Dynamic Mechanical Analysis (DMA). In this region, some rubber characteristics show significant changes such as hardness, flexibility and hysteresis, including fatigue behavior, etc. Transparent SBS specimens show the $\tan\delta$ peak above 80°C at 10Hz (Figure 5). The temperature of 50% elastic recovery (TR50) and the temperature of 100% elastic recovery (TR100) have been confirmed to exist above 70°C and 100°C respectively, from the results of the temperature-retraction test (Figure 6). Good elastic recovery at high temperature results from rubber crosslinking of SBS and low elastic recovery at room temperature (TR5) from the glass transition temperature of SBS. The max stress ($\text{Stress}_{\text{max}}$) for TR5 (@ 23 °C) or TR50 (@ 70 °C) temperature in the oscillation test, becomes high with increasing deformation speed or decreasing elastic recovery (Figure 7). It is therefore important to understand how these characteristics obtained from DMA and the temperature-retraction test are related to fatigue behavior. To understand the fatigue behavior near the glass transition region, it is necessary to observe the crack initiation process determining the fatigue life of rubber.

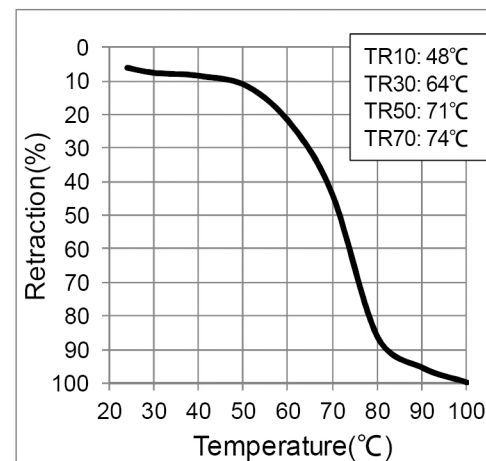


Figure 6. Temperature-retraction curve of SBS specimen.