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Fatigue Characteristics of Vulcanized Natural Rubber for Automotive Engine Mounting (Characteristics of Composition and Mechanical Properties)

by

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Abstract

In order to investigate fatigue characteristics of vulcanized natural rubber (NR), fatigue tests were carried out under various R and variable loadings. In this paper, an indicator for the material design are shown with detailed observation of the fatigue process and it makes rubber more durable against crack initiation. We found that the fatigue crack initiated from the vicinity of $CaCO_3$ by the observation of the fatigue process. Then fatigue tests were conducted using specimens which the content of $CaCO_3$ inclusions were controlled. As a result, the fatigue life has improved in test pieces without the $CaCO_3$ inclusions. The effect of the $CaCO_3$ content on the fatigue life is related only to crack initiation, and the crack propagation was not affected. The continuous observation of the fatigue damages and the measurement of crack growth rate were conducted, and the change of the fatigue process caused by $CaCO_3$ content is discussed by these results.

Keywords: Fatigue, Rubber, Crack propagation, Defect, Fractography, Stress ratio

1. Introduction

NR is used as automotive engine mountings to block the transmission of vibration from an engine to a body. A fatigue characteristic of industrial NR is known that the fatigue life increases with the increase of stress ratio $R(\sigma_{min}/\sigma_{max})$ when R is positive[1)~9)]. Moreover, higher durability of rubber material have risen in recent years for the security mileage of cars,

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and the material design guide has required to improve the durability. It is necessary to clarify the fatigue damage process of NR for that. In a current research, The fatigue damage process is confirmed to the fatigue crack generation, and propagation, and the occurrence of the fatigue crack from the defect of the lying thing etc[1).

Sainter *et al*.[8)] said that about 20% of the fatigue life is crack initiation life by observing the crack that existed in another place on the surface when the length of the main crack became 1mm.

Mars *et al.*[6)] examined the propagation of long cracks (25mm or more), and they proposed the prediction that the fatigue life is considered as the crack propagation life from the defect. However, these researches do not continuously observe the propagation process of small cracks. Therefore, it is not easy to say that the initiation time of the fatigue crack and the crack propagation of a small crack from defect will be clarified enough from the fatigue damage process thought to be the most influence on fatigue life.

In this research, two types of specimens were used. One is the plain specimen, and the other is the specimen with an small artificial defect. The artificial defects are less than 1mm and considered to be equivalent to inclusions inside specimens. So a very small crack growth can be observed. And the fatigue damage process under a constant repetition stress amplitude is observed directly, and clarified. In addition, Ca compound detected as a defect was specified as the origin of fatigue crack from the observation of the fracture surface, and the influence that caused it for the fatigue life was quantitatively evaluated. It is thought that the design manual with high reliability can be derived from these evaluation results.

2. Experimental Procedure

2.1 Material and specimens

The material used in this study is natural rubber vulcanized with sulfur and reinforced by carbon black. The tensile strength was 25MPa and the elongation was 550% using JIS dumbbell test piece. Moreover, the spring hardness was A52. **Figure 1** shows the specimen configuration. Two types specimens were used: One is the plain specimen, and the artificial defect specimens were introduced by an edge of small razors on the surface of the specimens as fatigue crack initiation sites (**Fig.2**).

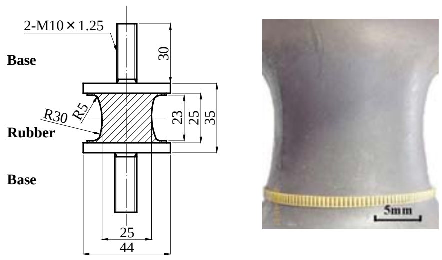


Fig.1 Fatigue specimen and surface of plain specimen.

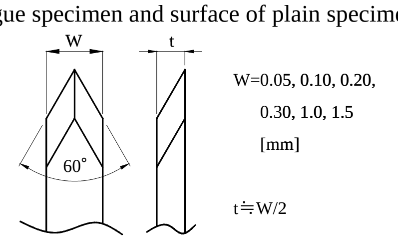


Fig.2 Shape of knife-edge.

In addition, to examine the influence of the $CaCO_3$ on the fatigue life, the NR material of the Ca compound addition existence was prepared in this research.

2.2 Testing method

Fatigue tests were carried out under load controlled in the air at room temperature. As for the examination, the load levels were mainly chosen from 10^4 to 10^5 cycles of fatigue life. The testing frequency *f* of all the specimens were at 3Hz. Specimens were air-cooled with blower. In order to observe the fatigue damage, photographs of the surface of the specimens were taken at suitable intervals. The photographs were taken under the state that the specimens were stretched 2mm for the observation of crack tips.

3. Experimental Results and Discussions

3.1 *S-N* diagram and constant life diagram

Figure 3 shows *S-N* diagram. The stress in the actual experiment adopts the true stress in the minimum cross-sectional area. *R* in the figure is stress ratio of the maximum stress and the minimum stress($R=\sigma_{min}/\sigma_{max}$).

Figure 4 shows the constant life diagram. It was made by using plain specimens. In the area *A* of the figure, the fatigue cracks propagated along the loading direction. In the area *B* and *C* of the figure, the fatigue cracks propagated along the direction perpendicular to the maximum principal strain (not stress) in one loading cycle. The fatigue lives in the area *C* were as long as the fatigue lives in the area *B* although high mean stresses were applied. The positive mean stress as in the area *C* is known that it induces the crystallization and reinforces natural rubbers[1)2)]. In order to simplify the phenomena of the fatigue characteristics, testing loads to investigate were selected in the area *B* and *C*.

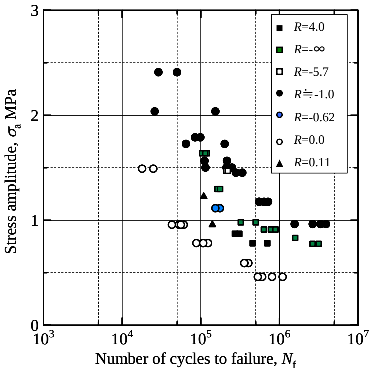


Fig.3 Relationship between σ_a and N_f.

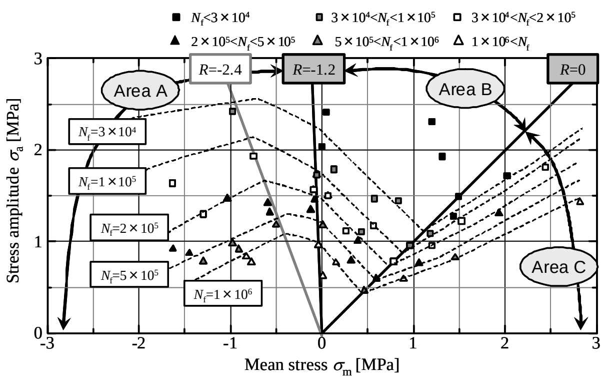


Fig.4 Constant life diagram.
(Relationship between σ_a and σ_m)

3.2 Fatigue characteristics of plain specimen

Figures 5 and **6** show the fatigue crack behavior of surfaces of test B and C, respectively. In the both tests, the fatigue cracks were observed on the surfaces of the specimens at about 80% of the fatigue life. **Figures 7** and **8** show fracture observation result of both examination result. The fatigue cracks are nucleated from defects like Ca or Si compound.

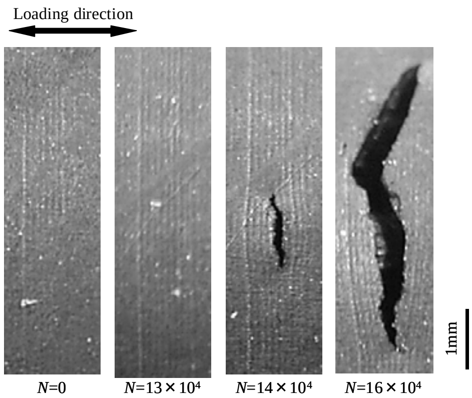


Fig.5 Fatigue crack of plain specimens and behavior of fracture surface in Test *B*.

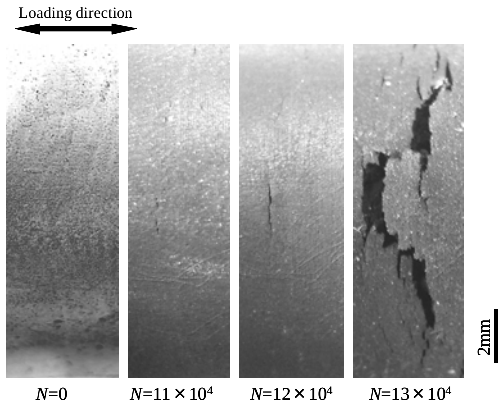


Fig.6 Fatigue crack of plain specimens and behavior of fracture surface in Test *C*.

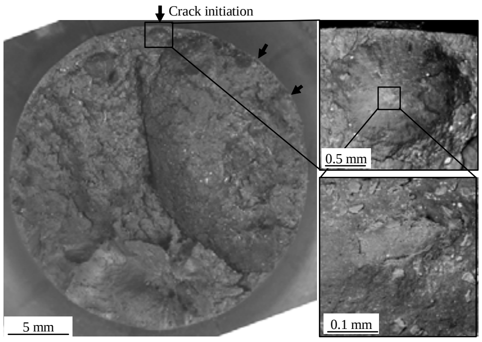


Fig.7 Fracture origin in the test *B*.

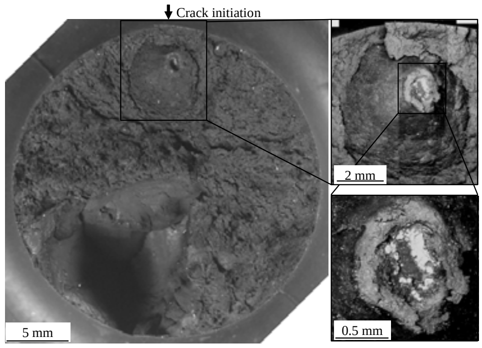


Fig.8 Fracture origin in the test *C*.

3.3 Influence of Ca compound on fatigue life

In this research, the influence of the Ca compound on the fatigue life is investigated. The purpose is to develop the material design indicator that can achieve higher durability.

The test was carried out with test *B* and test *C*. Moreover, two examinations were done respectively.

The fatigue lives are shown in **Table 1**. 'Without Ca compound' in the table means the test piece that removes Ca compound, and 'With Ca compound' means the test piece that used Ca compound so far. The fatigue lives in the case of the Ca removed rubber has improved by 33% in the test *B*, and by 21% in the test *C*. It is understood that the removal of the Ca compound contributes to the improvement of fatigue life. The improvement mechanism of fatigue life is considered in the next paragraphs.

Table 1 Test condition and result.

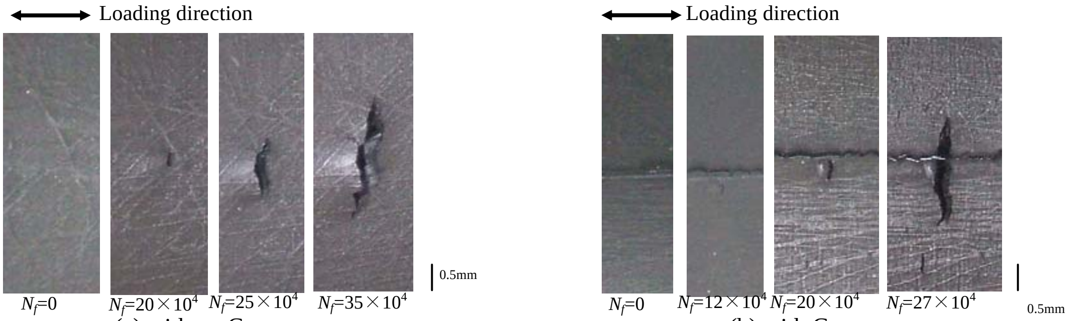
	R	σ_a [MPa]	σ_m [MPa]	With Ca compound N_f^{Ca} [cycle]	Without Ca compound N_f^{noCa} [cycle]	N_f^{noCa}/Nf^{Ca} [-]
Test B	-0.62	1.110	0.26	279×10^3	209×10^3	1.33
Test C	0.11	0.956	1.20	430×10^3	355×10^3	1.21

3.4 Influence of Ca compound on crack propagation

Figure 9 shows the crack progress situation on each surface of the test piece in test B. About ten cracks were nucleated on the surface, several propagated among them, and each specimen were broken from a main crack. Moreover, a main crack comes out on the specimen surface with about *N/Nf*=0.6~0.7, and the difference by the Ca compound existence is not seen on surface.

The crack growth rate *dl/dN* is shown in **Fig.10**. The crack growth rate of main cracks are scattering, but the gradient is almost 1.

Therefore, it is thought that the Ca compound doesn't influence the crack propagation characteristic.



(a) without Ca (b) with Ca

Fig.9 Fatigue crack of the with/without Ca specimens in test *B*.

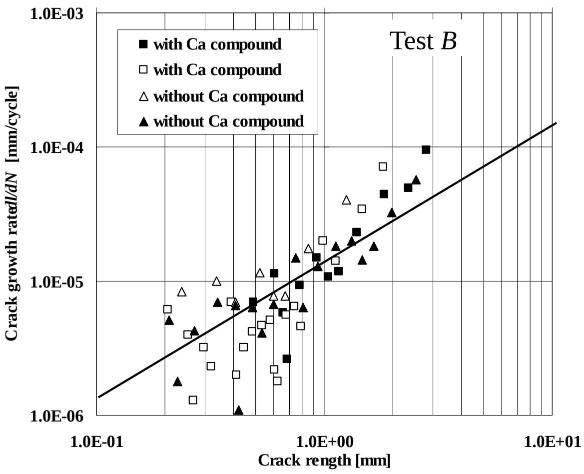


Fig.10 Relation between *dl/dN* and *l*.

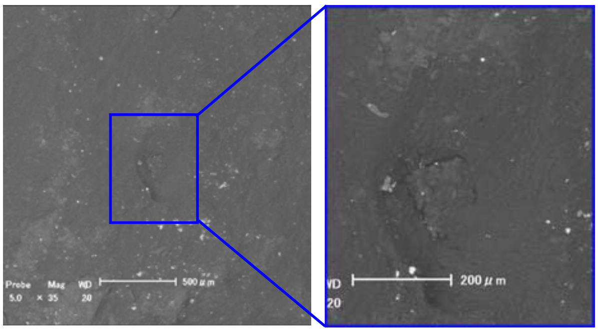
3.5 Fractography

To investigate the influence on the fatigue life with the Ca compound, the fracture origin was investigated by the fracture observation. **Figure 11** shows the vicinity of the fracture origin of both test pieces. As for the entire fatigue fracture, the difference is not seen in both test pieces. However, the substances around the crack origins of both test pieces were different. In the 'with Ca test piece', Ca with a white surface was confirmed. On the other hand, the black substances was confirmed in the 'without Ca test piece'.

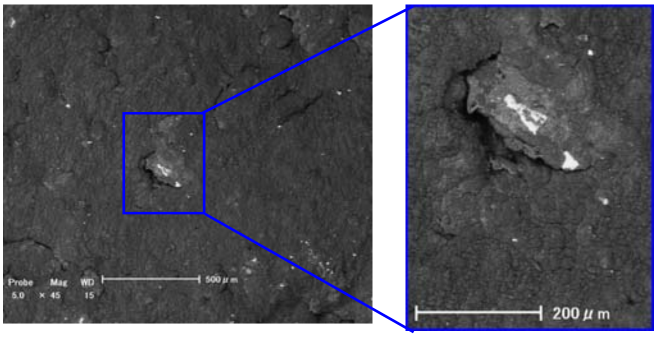
Figure 12 shows the result of analyzing the element by using EDX of these vicinity of the fracture origin.

The white substance on the fracture surface of the 'with Ca test piece' is thought to be Ca composed by the elemental analysis. Moreover, the majority of these white substance is considered to be covered with thin rubber. On the other hand, the black substances of the 'without Ca test piece' is considered to be the same element as the surroundings matrix, and they had been formed when the rubber had been vulcanized.

From the above-mentioned result, it has been understood that the change takes place in the fatigue fracture origin because of the Ca compound addition.

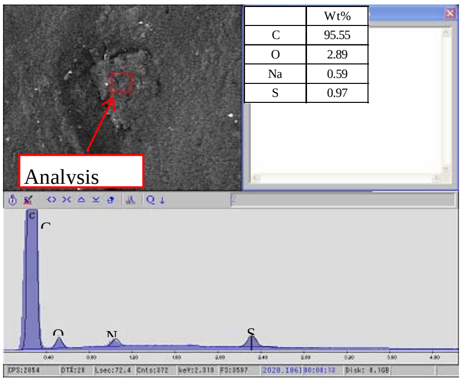


(a) Fracture origin of ‘without Ca specimen'.

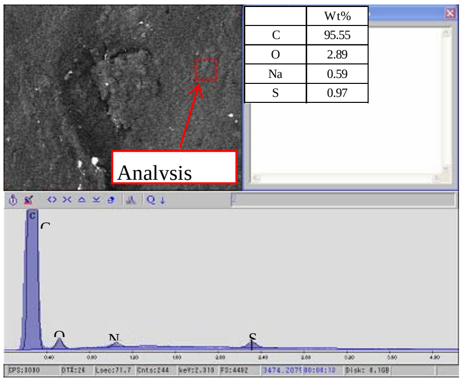


(b) Fracture origin of ‘with Ca specimen’.

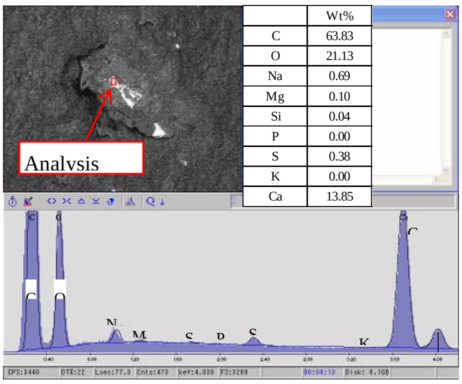
Fig.11 Fatigue fracture surface.



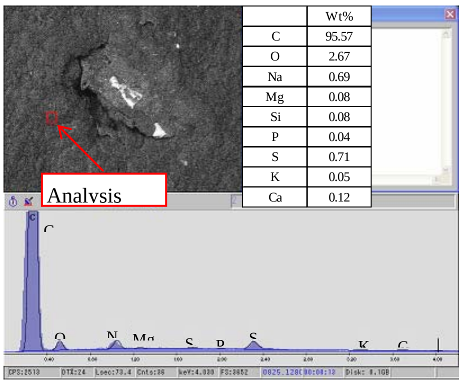
(a) On of the without Ca specimen



(b) Beside of the without Ca specimen



(c) On the with Ca specimen



(d) Beside of the with Ca specimen

Fig.12 Element analysis result of fatigue origin point.

The sizes of the fracture origin are shown in **Table 2**. The fatigue life of the 'without Ca test piece' is longer than that of the 'with Ca test piece' though rubber defect is larger than the Ca compound inclusion. On the other hand, there is no difference between the crack growth rate from the Ca inclusion and that from the rubber defect. The longer life of rubber defects than that of Ca inclusions cannot be explained by the size of the defects. It may be described by the late crack initiation and the material element of composition affects the crack initiation.

Table 2 Size of fatigue origin.

	With Ca compound	Without Ca compound
Fatigue origin of main crack	Ca compound	rubber
size [μm]	216	400

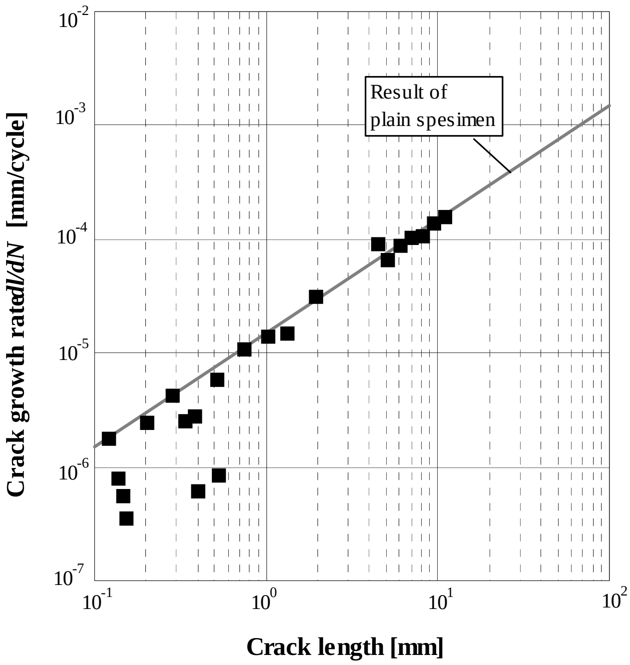


Fig. 13 Relation between crack growth rate *dl/dN* and crack length *l*.

3.6 Crack initiation life

An artificial defect was introduced into the 'with Ca test piece', and a detailed propagation process of the crack was observed from the surface. The defects were introduced on the surface of the specimen by the knife in **Fig.2**. The loading was done on the same condition as the test *B*. **Figure 13** shows the relation between crack growth rate *dl/dN* and crack length *l*. The plain specimen results are also shown in the figure. It is understood that *dl/dN* of the artificial defect material and the plain specimen doesn't depend on the stress level and *dl/dN* exist almost in *l* proportion (The gradient is 1). The ratio of the fatigue crack initiation life to total fatigue life of the plain specimen is estimated by using the crack growth rate obtained from this result. The crack growth rate of the fatigue crack is considered to be proportional to the crack length from **Fig.10** and **Fig.13**, and the relational expression is shown by the equation (1).

$$dl/dN=\alpha l \quad (1)$$

Here, α is a proportion constant parameter that depends on the stress level and the material.

$$(N_2 - N_1) = \frac{1}{\alpha}\ln\frac{l_2}{l_1} \quad (2)$$

If the equation is integrated, it becomes an equation (2).

Here, l_1, and l_2 are the crack length at repetition number N_1 and N_2 , respectively.

The fatigue crack initiation life from the Ca compound assumed to be N_1. N_2 is the cycle that the occurring crack reaches the surface. The size of the Ca compound was assumed to be l_1 and progress like the concentric circular crack, and the twice the distance from the Ca compound center to the surface was assumed to be l_2.

In case of test *B* using the 'with Ca', it was α=1.4 and l_2=6.8 mm, and $N_2=310\times10^3$.

Figure 14 shows the relation between N_1 and l_1 obtained from Equation (2). The crack initiation life N_1 becomes almost 0 if there is a lying thing of about 100μm from this figure. In **Fig.12**, the rubber part of about 100μm size around the found $CaCO_3$ inclusions has flaked off in the 'with Ca test piece'. In a word, the crack initiation life of the 'with Ca test piece' is estimated to be near almost 0. On the other hand, 20~30% has improved by the fatigue life in the test piece that has not added the Ca compound. It is thought that the 'without Ca test piece' contributes to making the fatigue life longer by controlling the fatigue crack initiation.

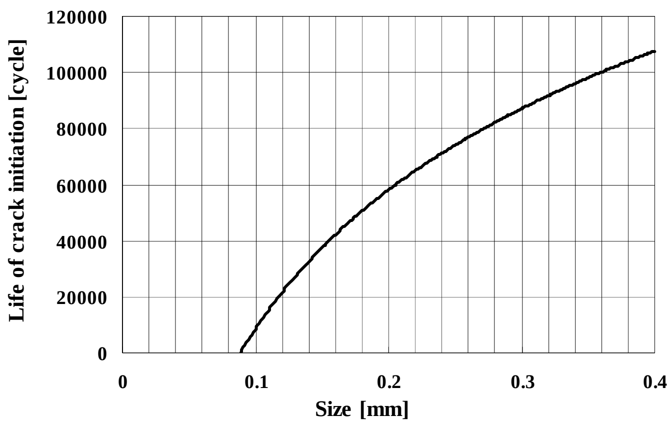


Fig.14 Relation between size of compound in the with Ca and crack initiation life.

4. Conclusion

The following conclusions were obtained from the fatigue testing that used natural rubber vulcanized with sulfur and reinforced by carbon black.

(1) The fatigue crack of the plain specimen is nucleated from the an internal Ca compound .

(2) As for the test piece without Ca, the improvement of the durability of 20~30% was seen for the test piece with Ca.

(3) The relationship between the crack growth rate and the crack length *l* of both test pieces

almost agree, and *dl/dN* is proportional to *l*.

(4) It is thought that the reason for the mechanism of making the life longer by not adding the Ca compound is that the fatigue crack nucleation was controlled.

(5) It is thought that this result can be used as one of the indicators of the rubber mixing design, and further high durability rubber material development can be promoted by progressing this idea.

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