EVALUATION OF PHYSICAL PROPERTIES AND FATIGUE DURABILITY OF RUBBER-CLAY NANO-COMPOSITES

C. S. Woo[a*], H. S. Park[a], D. H. Joe[b]

[a]Korea Institute of Machinery & Materials, Department of nano-mechanics, 171 Jang-dong, Yuseong-gu, Daejeon, Korea, 305-343.
[b]Diamond R. Tech. Co. Ltd.,91-23 Paho-dong, Dalseo-gu, Daegu, Korea, 704-220.
*cswoo@kimm.re.kr

Keywords: Nano-composites, Rubber-clay, Aging, Fatigue test, Lifetime prediction.

Abstract

We performed static and dynamic tests of rubber-clay nano-composites synthesized by inserting nano-filler between silicate layers at the high temperature of +70 ~+100°C, and verified that their mechanical properties were superior to the existing rubber material. In addition, a new method was developed to estimate fatigue lifetime of rubber parts in a short period in the initial stage of design, assuming that the fatigue damage parameter was Green-Lagrange strain generated at the weak points of parts. As results of estimation of fatigue durability, it was verified that the fatigue lifetime obtained by fatigue tests on actual engine mounts and the expected lifetime relatively match.

1. Introduction

Environment-friendly parts of automobiles have been actively promoted for the purposes of profit increase and environment-related regulations including vehicle exhaust gas reduction and fuel efficiency. To secure quality competitiveness through warranty extension and reduced repair expense of automobiles, technical development of anti-vibration rubber parts that are most important components for every function of automobiles is now required to enhance their durability and reliability [1, 2]. In this study, we developed rubber material that is environment-friendly and superior in physical property using rubber-clay nano-composites. Thermal resistance was estimated through material tests of developed material at room temperature and aged condition. Fatigue durability was estimated after we developed a new method that could estimate fatigue lifetime of rubber parts in a short period in the initial stage. As results, lifetime estimated by the fatigue lifetime estimation equation was exactly consistent with that obtained by fatigue tests of actual engine mounts. In addition, we verified that the developed material was superior in fatigue durability as well as mechanical properties because the lifetime of engine mounts made by the developed material was longer than the existing material.

2. Development of rubber-clay nano-composites

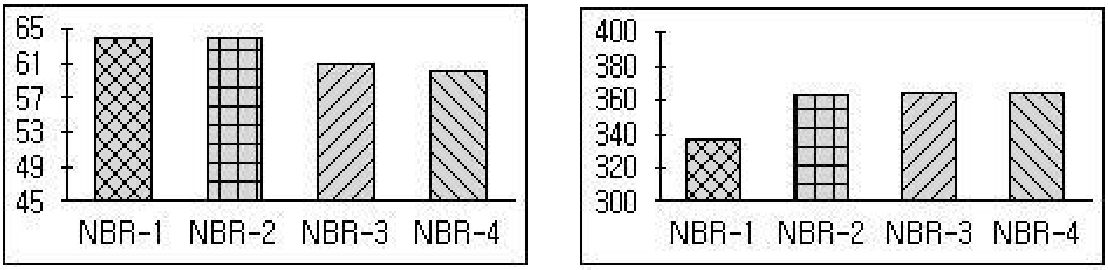
2.1 Rubber-clay nano-composites

Typical modifiers to enhance dynamical properties of rubber material were carbon black and silica. Recently, nano-clay was popular as modifiers. Many researches on nano-composites are being actively carried out because they are excellent in modification even with a small quantity of them while nano-clay is difficult to diffuse [3, 4]. Fillers or modifiers used when manufacturing polymer nano-composites include layered silicate, POSS nanoparticles, CNT and nanoparticles of metal or inorganic matters, among which layered silicate is now being most actively developed as polymer nano-composites. The key technology of development of polymer nano-composites is how to change layered clay so as to easily insert polymers into it [5]. When organic matters are inserted using inorganic material like clay silicate that has a uniform structure with nano-scale, in particular, nano-composites are attracting great concerns in their application. The basic structure of clay, as it is well known, consists of silica tetrahedral and alumina octahedral sheets: it is classified into several groups including vermiculite and montmorillonite depending on its negative charge.

In this study, acrylonitrile butadiene rubber (NBR) was used as rubber in combination; ZnO and stearic acid were used as vulcanization activators; and 3C was used as an additive; sulfur of purity 99.9% was used as a vulcanizing agent; TT and CZ were used as vulcanization accelerators; and carbon black, clay, and nano-clay were used as reinforced compound. Polymer layered silicate was made by the melted intercalation method in which polymers in the melted state were inserted between silicate layers: this method is advantageous in mass production and does not need to use solution.

2.2 Estimation of physical properties of rubber-clay nano-composites

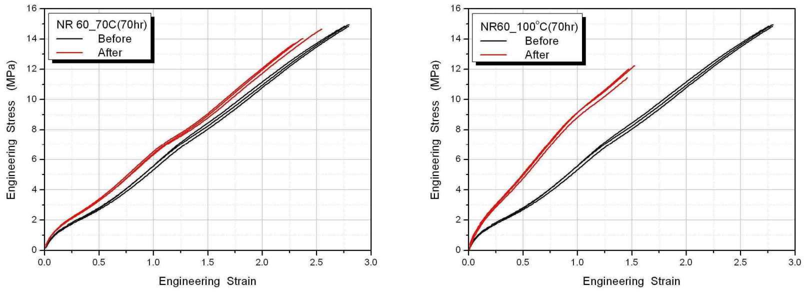
Physical properties [6] of NBR-1, NBR-2, NBR-3, and NBR-4 nano-composites developed according to the kind of nano-clay and the fraction of additives showed that their hardness and elongation at break depended on the clay as in figure 1. Figure 2 and figure 3 show the results of aging tests for 70 hours at 70° and 100°C to estimate thermal properties of natural rubber (NR) that have been used and the developed material (NBR-2). The change in the tensile strength of the developed material was 1.86% at 70°C and 7.44% at 100°C: these results are less than 5% and 20% for natural rubber, respectively. The change in elongation of the developed material was 8.8% at 70°C and 15.36% at 100°C: these results are remarkably less than 13.3% and 46.6% for natural rubber, respectively. In addition, the changes in 25% modulus of the developed material were smaller than the existing material. From the results, we found that the thermal properties of the developed material were superior to the existing material. Figure 4 and figure 5 show the fluctuations in storage modulus and loss factor at room temperature and aged condition. The dynamic properties of the developed material were superior to natural rubber because the fluctuation in its dynamical properties depending on temperature was less than the existing material.



(a) Hardness

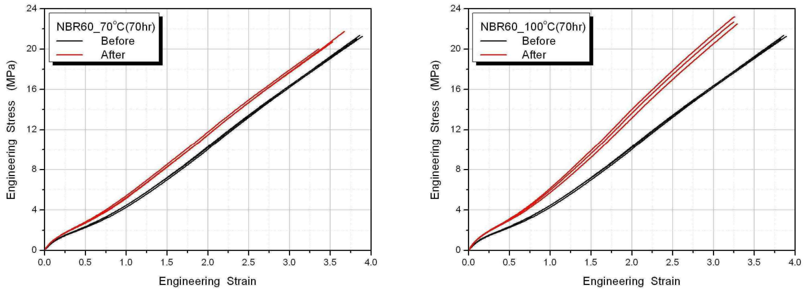
(b) Elongation at break

Figure 1. Physical properties of developed material



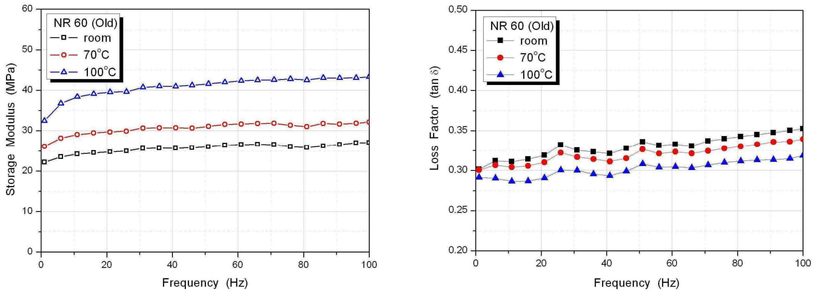
(a) 70°C (b) 100°C

Figure 2. Stress-strain curve of existed material



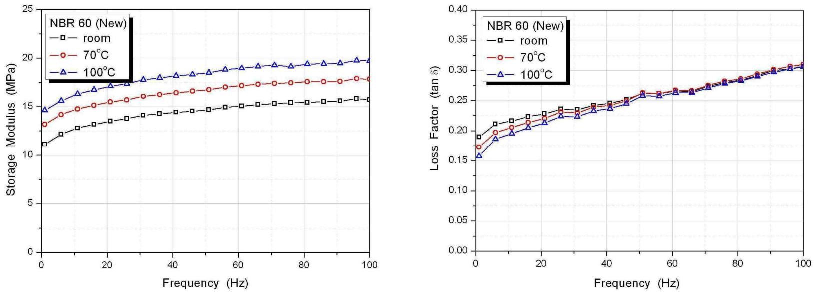
(a) 70°C (b) 100°C

Figure 3. Stress-strain curve of developed material



(a) Storage modulus (b) Loss factor

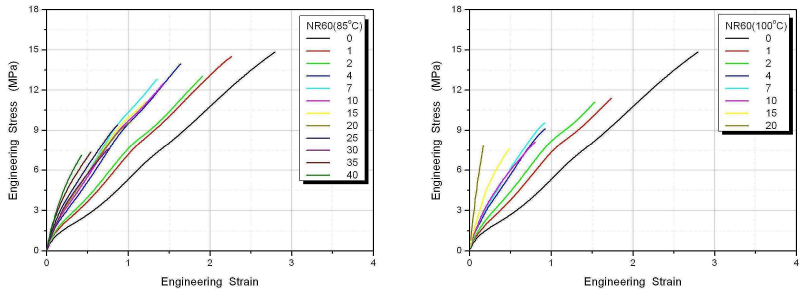
Figure 4. Dynamic properties of existed material



(a) Storage modulus (b) Loss factor

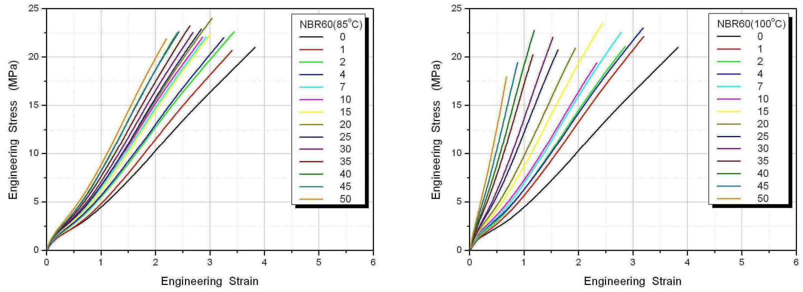
Figure 5. Dynamic properties of the developed material

After the developed material was processed through thermal aging for 1, 2, 4, 7, 10, 15, 20, 25, 30, 35, 40, and 50 days at 70°C, 85°C, and 100°C to investigate its long term thermal aging properties, changes in its hardness, tensile strength, elongation, and modulus were compared with those of the existing material. Figure 6 ~ figure10 show the results of aging property test of the existing material and the developed material for 50 days. The developed material was not damaged after 50 days at all temperatures while the natural rubber was damaged after 40 days at 85°C and 20 days at 100°C: the thermal properties of the developed material were excellent. Moreover, aging properties of the developed material including tensile strength, elongation, and change in 25% modulus were superior to those of natural rubber.



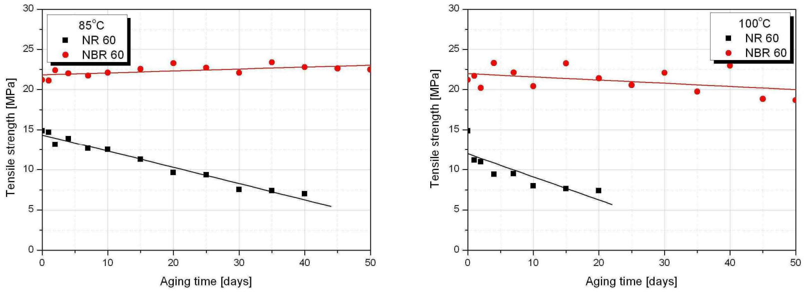
(a) 85°C (b) 100°C

Figure 6. Heat aging property of existed material



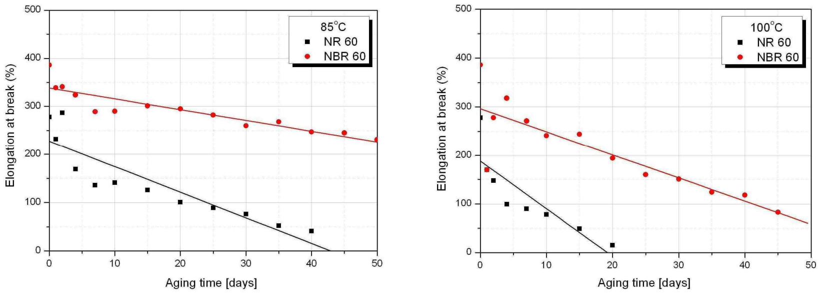
(a) 85°C (b) 100°C

Figure 7. Heat aging property of developed material



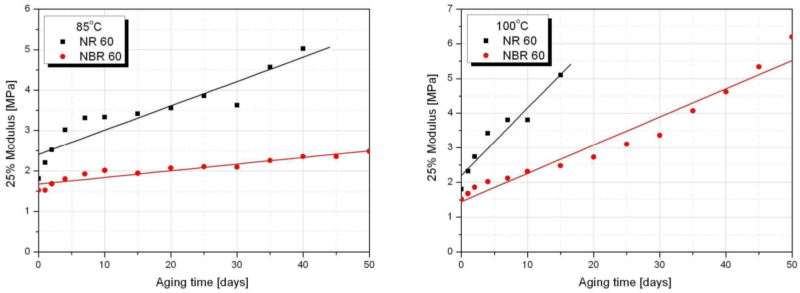
(a) 85°C (b) 100°C

Figure 8. Change of tensile strength of existed and developed material



(a) 85°C (b) 100°C

Figure 9. Change of elongation at break of existed and developed material



(a) 85°C (b) 100°C

Figure 10. Change of 25% modulus of existed and developed material

2.3 Estimation of fatigue durability of rubber-clay nano-composites

As demands for guarantee of quality and durability of products have been recently increased, estimation of fatigue lifetime and durability of rubber parts that have difficulties in reliability attracted many concerns. Anti-vibration rubber parts of automobiles, in particular, might cause fatigue damages by cyclic load while driving. Estimation of fatigue durability, therefore, is compulsory as warranty period of reliability tends to increase. In this study, we developed a new method that can exactly estimate fatigue lifetime of rubber parts in a short time in the initial stage of design and then estimated the fatigue durability of the developed material [7]. The method of estimation of fatigue lifetime of rubber parts suggested in this study was performed as follows: (1) the finite element analysis of rubber parts was done using the results of material tests of rubber material; (2) the relation between the maximum Green-Lagrange strain and displacement was obtained; (3) fatigue tests of 3-dimensional fatigue specimens that have the same physical properties was performed to obtain the relation between the maximum Green-Lagrange strain and fatigue lifetime; (4) the fatigue lifetime of rubber parts was estimated using the results of finite element analysis of the parts and fatigue tests on specimens. Comparing estimated fatigue lifetime with the results of fatigue tests on rubber parts, validity of the procedures of fatigue lifetime estimation suggested in this study was verified. To estimate fatigue lifetime of rubber parts, fatigue lifetime diagrams of the same rubber materials are required. We designed and made 3-dimensional fatigue specimen that could reproduce the maximum tensile strain as shown in figure 11 and carried out fatigue tests to estimate lifetime of rubber parts.

(a) Three dimensional dumbbell specimen (b) fatigue tester

Figure 11. Fatigue test of three dimensional dumbbell specimens

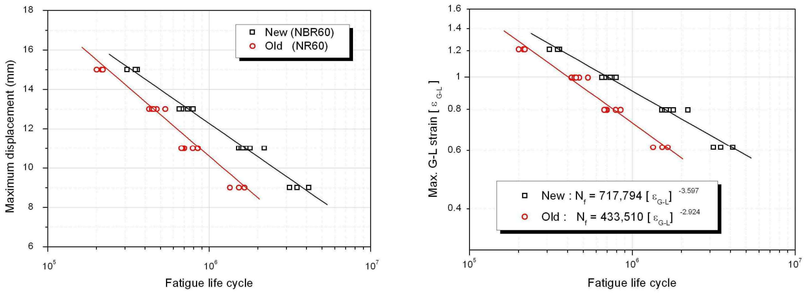
To investigate fatigue lifetime of the existing material and the developed material, fatigue tests on 3-D fatigue specimens were performed with the displacement control tester. Figure 12(a) shows a diagram that represents the relation between fatigue lifetime and the maximum tensile displacement of the existing material and the developed material: fatigue lifetime decreases as the tensile displacement increases. This diagram well represents fatigue lifetime regardless of test conditions when the maximum tensile displacement is the fatigue damage parameter. Using the relation of the maximum tensile displacement and strain obtained from finite element analysis on fatigue specimen, the diagram of the maximum tensile displacement and fatigue lifetime obtained by fatigue tests could be well represented by the relation diagram of the maximum strain and fatigue lifetime as shown in figure 12(b). Estimation of fatigue lifetime of rubber material was derived by the relation between the maximum strain and fatigue lifetime as in equation (1) and (2). The fatigue durability of the developed material was superior to the existing material as shown in figure 12.

Equation of fatigue lifetime prediction for existed material:

$$N_f = 433{,}510 \cdot [\epsilon_{G-L}]^{-2.924} \tag{1}$$

Equation of fatigue lifetime prediction for developed material:

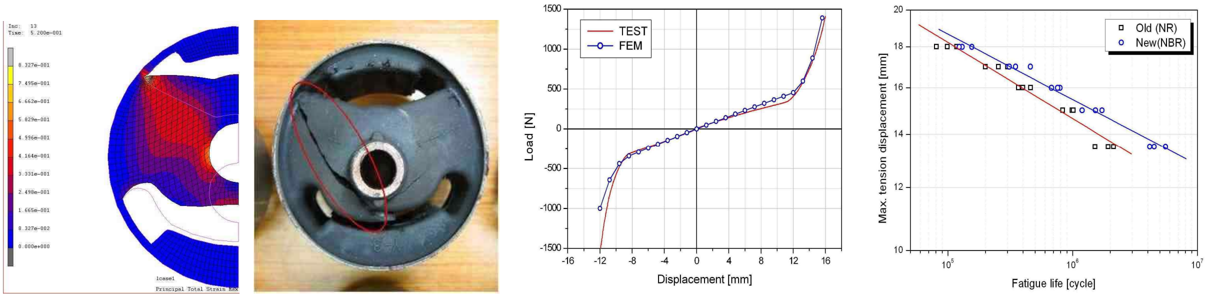
$$N_f = 717{,}794 \cdot [\epsilon_{G-L}]^{-3.597} \tag{2}$$



(a) Maximum displacement (b) Maximum G-L strain

Figure 12. Fatigue lifetime diagram of existed and developed material

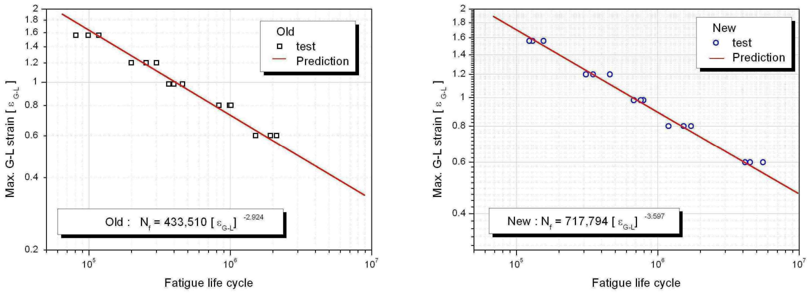
To verify the validity of the fatigue lifetime estimation method of rubber parts suggested in the above, finite element analysis and fatigue tests of engine rubber mounts that are used as anti-vibration rubber parts of auto mobiles were carried out. Figure 13(a) shows the results of finite element analysis on engine mounts: the maximum strain and stress were the maximum at the center and on the surface where rubber and metal meets (the weakest section). The results of analysis were reliable because the results of finite element analysis were consistent with those of characteristics test in the load-displacement diagram as shown in figure 13(b). Figure 13 (c) and (d) show the results of fatigue tests on engine mounts: the results of finite element analysis were well consistent with those of tests because fatigue cracks were occurred at the section where strain was the maximum in the analysis. The lifetime of engine mounts made of the developed material appeared to be longer than that of the existing material: the developed rubber material has an improved durability.



(a) FEA (b) Fatigue crack (c) Load-displacement curve (d) Fatigue lifetime prediction

Figure 13. Finite element analysis and fatigue lifetime diagram of engine rubber mount

We verified that the estimated lifetime using the fatigue lifetime prediction equation of rubber specimen and the fatigue lifetime obtained by fatigue tests on actual engine rubber mounts was exactly consistent as shown in figure 14. With the results of finite element analysis of rubber parts using the fatigue lifetime estimation method suggested in this study, the lifetime can be estimated without fatigue tests on rubber parts. Therefore, we can save development time and expense and achieve good quality and reliability of rubber parts.



(a) Existed material (b) Developed material

Figure 14. Comparison of estimated lifetime with that in experiments

3. Conclusions

We developed anti-vibration rubber material made of rubber-clay nano-composites that are environment-friendly and excellent in mechanical properties and obtained the following conclusions.

(1) Polymer layered silicate was made by the melted intercalation method in which polymers in the melted state were inserted between silicate layers: this method is advantageous in mass production and does not need to use solution.
(2) Mechanical tests on the developed material were performed at room temperature and aging condition, and we verified that mechanical properties of the developed material such as tensile strength, elongation, and modulus change were superior to the existing material.
(3) Fatigue durability was estimated after we developed a new method that could estimate fatigue lifetime of rubber parts in a short period in the initial stage.
(4) Fatigue lifetime of rubber specimens estimated by the fatigue lifetime estimation equation was exactly consistent with that obtained by fatigue tests of actual engine mounts. In addition, we verified that the developed material was superior in fatigue durability as well as mechanical properties because the lifetime of engine mounts made by developed material was longer than the existing material.
(5) In this study, advance of related technologies was achieved by constructing complete technologies including design, analysis, and estimation of rubber parts. We expect that these results will contribute to enhance performance and reliability of rubber parts.

Acknowledgements

This study is a part of "the small and medium business technology innovation project" supported by the Small and Medium Business Administration.

References

[1] A. N. Gent and G. J. Lake. *Engineering with rubber*, Hanser publication, 1992.
[2] C. S. Woo, S. S. Choi and S. B. Lee. Useful lifetime prediction of rubber components using accelerated testing. *IEEE Trans.*, Vol. (59), 11-17, 2010.

[3] M. S. Kirchhoff, U. Specht and G. Veser. Syntheis and characterization of novel high temperature stable nano-composite catalysts, *Nanotech*, Vol. (3), 268-271, 2004.
[4] A. Ochsner, W. Ahmed and N. Ali. Nanocomposite coatings and nanocomposite materials. *Material Science Foundations*, Vol. (54), 402, 2009.
[5] M. Kawasumi, M. Hasegawa, A. Kato and A. Usuki. Preparation and mechanical properties of polypropylene-clay hybrids, Vol. (30), 6333-6338, 1997.
[6] R. P. Brown, *Physical testing of rubber elastic*, 3rd ed., Chapman & Hill, 1990.
[7] C. S. Woo, W. D. Kim and J. D. Kwon. A study on the material properties and fatigue life prediction of natural rubber component, *Materials Science and Engineering*, Vol. (483-484), 376-381, 2008.