

Viscoelastic Properties of Short Aramid Fibers-Reinforced Rubbers

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ABSTRACT: Among short fiber-reinforced composites, those with rubber matrices have gained great importance due to the advantages they have in processing and low cost, coupled with high strength. These composites combine the elastic behavior of rubbers with strength and stiffness of fibers. Reinforcement with short fibers offers attractive features such as design flexibility, high modulus, tear strength, etc. The degree of reinforcement depends upon many factors such as: the nature of the rubber matrix, the type of fiber, the concentration and orientation of the fibers, the fiber to rubber adhesion and fiber length. One of the main features related to this class of composites is their hysteretic properties which are of great importance especially in dynamic applications such as tire treads. In this research, short aramid fibers with different kinds of surface treatments: Standard finish and resorcinol formaldehyde latex (RFL)-coating, have been applied in two different carbon black filled rubber compounds based on natural rubber (NR) and ethylene propylene diene rubber (EPDM). The dynamic properties of the compounds have been measured as influenced by the different kinds of fiber treatments on rubber-fiber interaction. Finally, the contribution of the interfaces and/or interphase layers to the loss properties is characterized. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

Fiber-reinforced composites with the best mechanical properties are those with continuous fiber reinforcement. Such materials cannot be adapted easily to mass production and are generally limited to products in which the property benefits outweigh the cost penalty.¹ Alternatively, short fibers are used to reinforce polymers to improve or modify the thermo-mechanical properties of the matrix for specific applications or to reduce the cost of the fabricated article.² By adding suitable fibers and by controlling factors such as the aspect ratio, the dispersion and orientation of the fibers, and the fiber–matrix adhesion, significant improvements in properties can be achieved with thermoplastic, thermosetting, and rubber polymers.¹

Among different short fiber-reinforced composites, those with rubber matrices are gaining increasing importance, due to the advantages they impart in processing and low cost coupled with high strength. These composites combine the elastic behavior of rubber with strength and stiffness of the fibers. Short fiber-reinforced rubbers have been successfully used in production of V-belts, hoses, tire treads, and complex-shaped mechanical goods.^{3,4}

Generally, the degree of reinforcement depends on the nature of the matrix, the type of fibers, the concentration and orientation

of the fibers, fiber to matrix adhesion (generation of a strong interface), and aspect ratio of the fibers.^{3–5} Poor adhesion increases the critical fiber length, which is the minimum length of fibers needed for effective stress transfer, since mechanical friction at the interface must take the role of adhesion. Good adhesion can nearly double the tensile strength and elongation at break compared with a composite in which the adhesion is poor.⁶

A common method to increase the adhesion is by fiber surface treatment, using for example isocyanate or resorcinol formaldehyde latex (RFL); the adhesive treatment for various types of fibers differs. The adhesive layer is applied on a cord by a so-called dipping process. Details of this process can be found elsewhere.⁷

The concept of strength of the interfacial bond is not always clear. In case of perfect adhesion, the applied stress can be properly transferred from matrix to the fibers and failure mode will not be phase separation: the matrix or the fibers break before the interfacial bond. In absence of adhesion, essentially no work is required to separate the surfaces of the matrix and fiber phases even though the two surfaces may appear to be in contact. However, even in the case of no adhesion, work is required to pull a fiber out of a block of the matrix because of the squeezing force exerted on the fiber as a result of mismatch in

coefficient of thermal expansion and cooling down of the composite from the fabrication temperature. Between perfect adhesion and no adhesion there can be many gradations.⁶

Besides the increased strength of the short fiber composites, their viscoelastic properties are also of great importance, especially in dynamic applications. Friction forces of tires are related to the value of the loss angle,⁸ and the relation between rolling resistance and wet grip of tires to the hysteretic properties is also well known. Hess and Klamp⁹ referred to the work done by Khromov and coworkers,¹⁰ who examined a blend of styrene butadiene rubber/butadiene rubber and concluded that a drop of just 0.022 in $\tan \delta$ results in 5% reduction in tire rolling resistance.

There are studies^{2,11,12} which show that adding short fibers generally results in an increase in storage modulus of rubbers in a considerable range of fiber loadings and temperatures. It has been also observed that increase in adhesion increases the storage modulus and mechanical loss per cycle under dynamic conditions.¹³

Furthermore, the interface and interfacial bonding affects the loss properties of composites.^{2,13} It has been suggested that the quality of the interfacial adhesion in the composites can be evaluated by measuring that part of energy dissipation which is contributed by the interfaces, which can be obtained by subtracting the loss of fiber and matrix from the total loss of the composites.¹⁴ Gibson¹⁵ found higher damping of aramid fibers than glass fibers in an epoxy resin. Nielson and Landel⁶ observing higher damping of the aramid fibers compared with the glass fibers in an epoxy resin, concluded that this may be due to poor adhesion of aramid fibers, but at least some of the damping is due to the high damping of the aramid fiber itself compared with the negligible damping of a glass fiber.

Tensile test results and a study on fracture mechanics of natural rubber (NR) and ethylene propylene diene rubber (EPDM) compounds reinforced with short aramid fibers, treated with Standard Finish (St), an oily substance used to facilitate spinning, and RFL were presented elsewhere.¹⁶ It was shown that adding fibers to the compounds causes a drop in ultimate tensile strength and elongation at break, but also results in higher stresses in both low and high strain regimes. Although in all cases RFL-treated fibers showed to lead to the best mechanical properties, reinforcement in sulfur-cured NR especially with RFL-treated fibers was significantly less than in peroxide-cured EPDM. It was argued that clear chemical adhesion only happened in the case of peroxide-cured EPDM reinforced with RFL-treated fibers, and that in the other cases reinforcement was due to mechanical interaction. The results of fiber length studies¹⁷ in EPDM and NR compounds, without carbon black, showed that in both matrices RFL-treated fibers ended up in slightly higher fiber length after mixing. A scanning electron microscopy (SEM) study on the fracture surfaces of the carbon black filled rubbers showed that the fibers treated either with St or RFL coating did not break as the result of applied tensile force, which shows that they were still above their critical length.¹⁶ So, fiber breakage was not a critical factor in determining the final properties of the composites.

Table I. Compound Recipes (phr)

Component	NR	EPDM
NR	100	-
EPDM	-	100
Carbon black N-220	55	-
Carbon black N-550	-	105
Oil	8	60
Stearic acid	2	1
ZnO	5	-
6PPD	2	-
TMQ	1.5	-
Wax	2	-
PEG2000	-	2.5
TBBS	1.5	-
Sulphur	1.5	-
Perkadox 14/40	-	7.5
TRIM	-	4

In this work, in relation to the previous research, the viscoelastic properties of these two types of widely used rubbers, EPDM and NR are investigated in the form of typical radiator hose and truck tire tread compounds, reinforced with short aramid fibers. The aim was to study how the viscoelastic properties of these rubbers are affected by adding short aramid fibers and how the two types of reinforcement, chemical and mechanical, work out in these viscoelastic properties.

EXPERIMENTAL

Materials

Twaron[®] aramid short fibers were supplied by Teijin Aramid B.V., the Netherlands, with average initial length of 3 mm and fiber diameter of 10–12 microns. Two types of elastomers were used: NR: SMR CV60; and EPDM: Keltan[®] 8340A with 5.5 weight percent ethylidene norbornene (eNB) content, and 55 and 39.5 weight percents of ethylene and propylene respectively, from DSM Elastomers B.V., The Netherlands. Carbon blacks N220 and N550 were obtained from Evonik GmbH (formerly Degussa), Germany. The oil types added to the EPDM and NR compounds were Sunpar[®] 2280 paraffinic oil from Sun Oil Company and Nyltex 840 naphthenic oil from Nynas, respectively. ZnO was Red Seal quality from Union Minière, Belgium, and stearic acid was a technical quality grade. Polyethyleneglycol (PEG2000) was obtained from Merck, Darmstadt, Germany. Sulfur and *t*-butyl-benzothiazol-sulfenamide (TBBS) were provided by Rhein Chemie, Germany. Trimethylolpropane-trimethacrylate (TRIM) and Perkadox 14/40 peroxide were obtained from AkzoNobel, Deventer, The Netherlands. Polymerized 1,2-dihydro-2,2,4-trimethylquinoline (TMQ) came from Flexsys, Belgium.

Methods

Two master batches were made in a 150 L industrial internal mixer. The compositions of the two compounds are presented in Table I. The curatives and short fibers were added on a

laboratory two roll mill. Two different kinds of surface treatments have been used for the aramid fibers. The treatments were St and RFL coating. It was shown elsewhere that the standard finish has no negative influence on the adhesion of cords to rubbers.¹⁸

The cure characteristics of the compounds were measured with a Rubber Process Analyzer (RPA) of Alpha Technologies, and according to the results the compounds were cured for their $t_{90} + 2$ min in a Wickert hot press WLP1600, at 100 bar pressure in sheets of 1.9 mm thickness. To obtain a preferred orientation of the fibers, 20 g of each compound—the amount needed to fill the mold—was passed several times through a two roll mill. The milling direction was considered as longitudinal direction of fiber orientation.

Dynamic mechanical analysis were done using a Metravib Viscoanalyser DMA+150, in tension mode; storage moduli and loss angles of the samples were measured in temperature sweep, strain sweep, and frequency sweep tests. To reach minimum error, the tests were performed at least on three samples of every compound. For NR compounds, the standard deviation was usually low; for example in temperature sweep tests above the glass transition temperature range, it was below 1%, and in strain sweep tests, below 3%. For EPDM in some cases, to reach a minimum level of error up to six to seven samples of the same compound had to be tested until at least three to four similar curves close to each other with a standard deviation less than 3%, were obtained. The first tests were done without the application of a prestrain which is closest to practical use of most rubber goods in dynamic applications. A second set of tests were done with application of a static elongation, prestrain, larger than the applied dynamic strain values, to remain in the elongation regime. In any case, only the data with an acceptable standard deviation are reported.

Elongation set tests have been done on three samples of each compound containing 5 phr fibers. Stripes with a length of 82 ± 1 mm and width of 10 mm were cut out of the cured sheets with thickness of 1.9 mm in parallel direction of fiber orientation. The samples were stretched for $30\% \pm 1.5\%$ of their initial length. After 24 h at room temperature the samples were released and after 30 min their lengths were measured. The elongation set is reported as the permanent increase in length in percent divided by the applied elongation.

RESULTS

The carbon black filled compounds were loaded with 3 phr fibers, to avoid possible interactions between the short fibers, which happens at higher concentrations resulting in a significant contribution to the measured viscoelastic properties. In fact, previous results on model systems (without carbon black) showed that this does not happen till 5 phr loading.¹⁷ The viscoelastic properties of the samples have been evaluated by tests in temperature, strain, and frequency sweep modes, to give a rather complete picture of the viscoelasticity of the composites under different practical conditions.

Temperature Sweep Measurements

The storage moduli of NR and EPDM compounds in temperature sweep tests, without prestrain are shown in Figure 1. It can

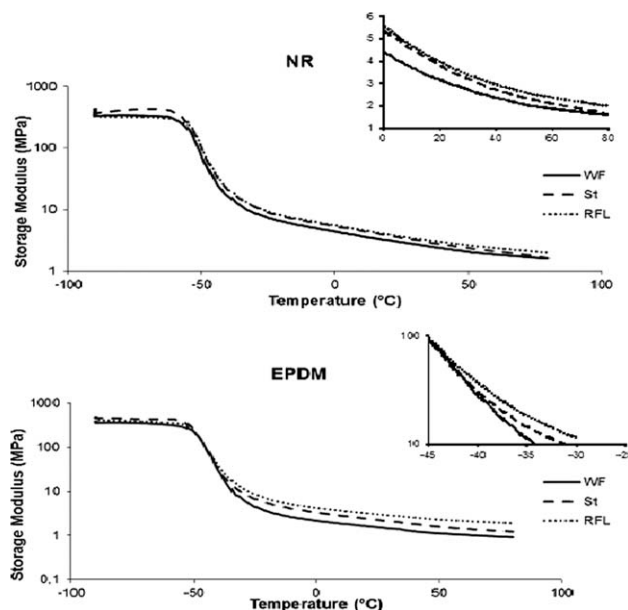


Figure 1. Storage moduli from temperature sweep DMA measurements at 0.1% strain, without prestrain, frequency of 10 Hz for various fiber treatments. WF, without fiber; St, standard finish treated; RFL, RFL-treated.

be seen that the addition of fibers generally results in an increase in storage modulus, especially in the temperature range above T_g . For NR this is even more clear in the temperature range above 0°C than at lower temperatures. It also can be seen that RFL treatment leads to more reinforcement compared with St-fibers. The storage moduli of the EPDM compounds also increase considerably with inclusion of RFL-treated fibers.

The $\tan \delta$ results for the temperature sweep tests, without prestrain, Figure 2, show that for NR the compound with RFL-treated fibers generally has the lowest $\tan \delta$ throughout the whole temperature range, even lower than for the compound without fibers. It can also be seen that in both NR and EPDM compounds in the range above room temperature St-fibers generally result in the highest loss angle. An interesting point here is the difference between the loss angle curves of the compounds at the $\tan \delta$ peak or glass transition temperature, where RFL shows the lowest $\tan \delta$ peak and the compound without fibers (WF) the highest for both polymers. Having a closer look at Figure 1, it turns out that with passing the glass transition temperature the drop in storage modulus of fiber-filled composites is less compared with the WF-compounds. This trend is similar to the results of tensile tests: at elongations up to 100%, RFL-treated fibers lead to the highest stresses, and St-treated fibers still result in higher stress compared with WF-compounds.¹⁶

Figure 3 shows that for NR compounds, without prestrain, at a higher dynamic strain of 5%, the difference between the various $\tan \delta$ curves is more pronounced in the range above room temperature. This test was done on the NR compounds mainly to represent rolling resistance as an important tire property in a more severe conditions of higher strain. The rolling resistance of a tire is represented by the loss angle of the compound in the range between 30 and 70°C which is the running temperature range of tires.¹⁹ It can be seen that the NR-compound

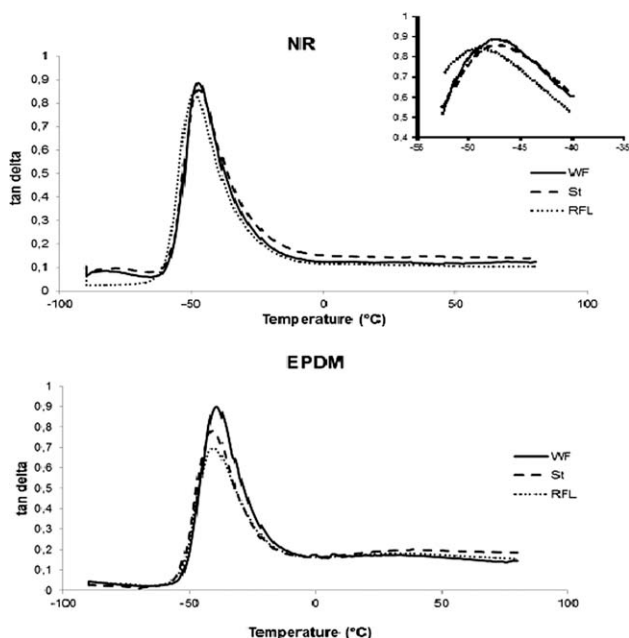


Figure 2. Loss factor $\tan \delta$ derived from temperature sweep DMA measurements at 0.1% strain, without prestrain, frequency of 10 Hz for various fiber treatments. WF, without fiber; St, standard finish treated; RFL, RFL-treated.

containing RFL-treated fibers has a considerably lower loss angle, despite the fact that the reinforcement mechanisms involved in this case are mainly mechanical of nature¹⁶ and, unlike for RFL-treated cords, no chemical bonds occur between rubber and the coating on the fibers. It can also be seen that at 5% of strain, St-treated fibers do not negatively affect rolling resistance relative to the compound without fibers. An attempt was also made to perform a temperature sweep test on EPDM compounds at 5% of strain, but the scatter in the data was too high to be presented here.

The same temperature sweep tests in low strains have been done on the samples with application of 0.2% of static prestrain and the results of storage modulus and $\tan \delta$ were comparable

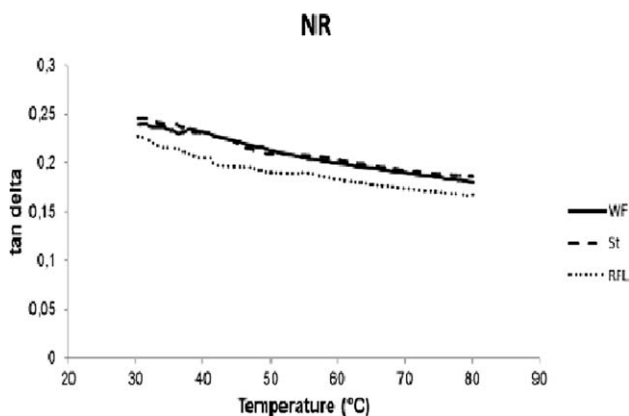


Figure 3. Loss factor $\tan \delta$ derived from temperature sweep DMA measurements at 5% strain, without prestrain, frequency of 10 Hz for various fiber treatments.

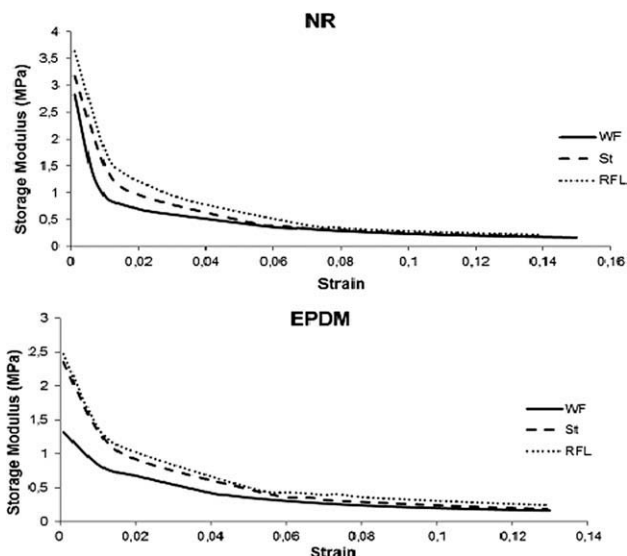


Figure 4. Storage moduli from strain sweep DMA measurements on 3 phr fiber-loaded NR and EPDM compounds at ambient temperature, without prestrain, frequency of 10 Hz, for various fiber treatments.

with the results of the same test without prestrain, which are not presented to avoid unnecessary additional pictures.

Strain Sweep Measurements

Figure 4 shows storage moduli measured in strain sweep tests, without prestrain at ambient temperature of 20°C. It can be seen that again in both cases, NR and EPDM, over a wide strain range RFL-treatment of the fibers results in the highest storage modulus, although this difference is not always very significant because of the small amount of fibers added. In the NR compound, St-fibers perform better than WF for strains till 5%, while this range is wider for EPDM. In all cases, increase in strain results in less difference between the fiber-filled compounds and the compound without fibers. The loss angles obtained from the strain sweep tests, without prestrain at ambient temperature, Figure 5, show that in a considerable range of strains, the NR compound reinforced with RFL-treated fibers has the lowest $\tan \delta$, while there is almost no change in loss angle of the NR compound as a result of adding St-treated fibers in comparison with WF.

In the $\tan \delta$ graphs for EPDM, Figure 5, without prestrain and at very low dynamic strains, adding fibers results in almost no change in $\tan \delta$. This is in accordance with the results presented in Figure 2 in the temperature range between 0 and 20°C. At slightly higher strain of about 1%, the interface effect is pronounced and St-fibers show higher loss compared with the compound without fibers, while RFL-coated fibers give a lower $\tan \delta$ than St-fibers. This can be due to the absence of chemical bonding for the St-fibers. Where a high $\tan \delta$ can be an indication of mechanical loss/hysteretic effects due to mutual friction between rubber and the fibers on the interface, the lower level of $\tan \delta$ for the RFL-coated fibers suggests that there is less mechanical loss than with St-coating.

Strain sweep tests after applying a prestrain of 10%, larger than the dynamic strain values of up to 9%; have also been performed on the samples: Figures 6 and 7. The main difference between

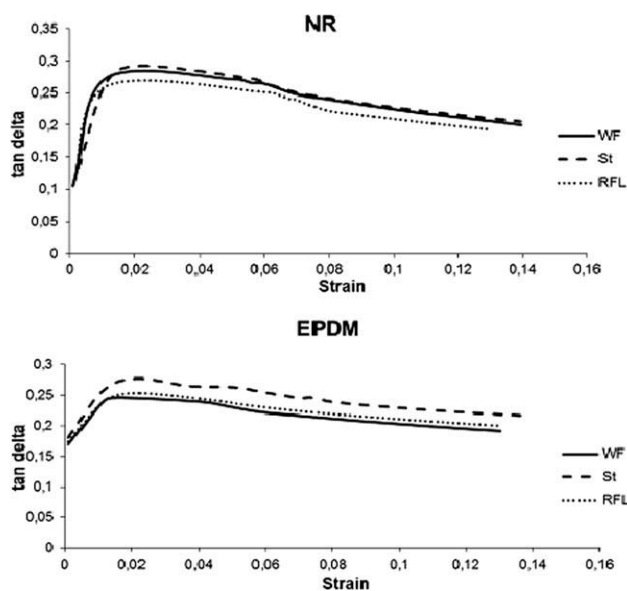


Figure 5. Loss factor $\tan \delta$ derived from strain sweep DMA measurements at ambient temperature, without prestrain, frequency of 10 Hz, for various fiber treatments.

the storage modulus results here is seen for NR, while the storage modulus of EPDM shows the same trends as the tests without prestrain. For NR the application of a prestrain, reduces the benefits of RFL-treated fibers. While for NR the interaction with all fibers is mainly mechanical and not chemical of nature, it is clear that this kind of interaction mainly acts at low elongations. This was also confirmed by the results of tensile tests, as presented in our previous work.¹⁶ It was shown there that in a static tensile test the reinforcement factor, defined as the stress of the compos-

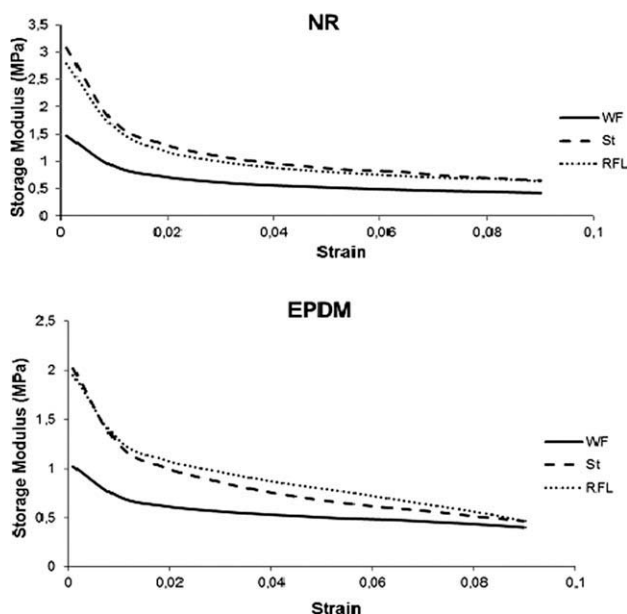


Figure 6. Storage moduli from strain sweep DMA measurements, with application of 0.1 (=10%) prestrain, on 3 phr fiber-loaded NR and EPDM compounds at ambient temperature, frequency of 10 Hz, for various fiber treatments.

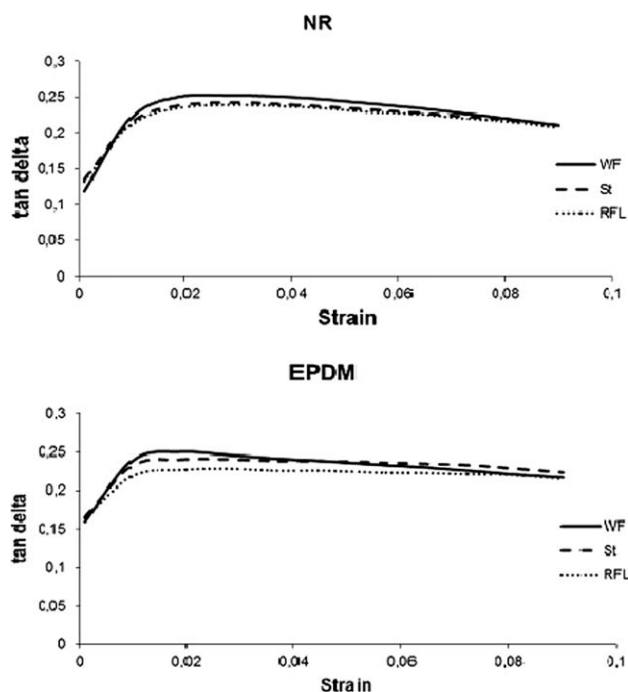


Figure 7. Loss factor $\tan \delta$ derived from strain sweep DMA measurements at ambient temperature, frequency of 10 Hz, Prestrain of 0.1 (=10%), on 3 phr fiber-loaded NR and EPDM compounds for various fiber treatments.

ite at a certain elongation divided by the stress of the corresponding WF compound at the same elongation, is higher for low elongations. That is because in processing of the composites, the rubber can form itself around the roughness of the fiber before being cured. So, after curing, it is difficult to pull the fiber out of the first position. At higher strains, after changing the mutual position of the fiber-surrounding matrix, the reinforcement may still take place due to frictional forces between fibers and rubber, but to a relatively lesser extent. Another important factor is that: the RFL-coated fibers used in this research had a dip pick up of 12–15 weight percent, so that using the same weight, the number of RFL-treated fibers is 12–15% less than St-fibers. So, it is readily understood that by lack of chemical adhesion, and by removing the most effective part of mechanical interaction as a result of applying a large enough prestrain, St-fibers can even lead to a slightly higher storage modulus than RFL-treated fibers. Loss angle results show again no advantage of RFL-treated compared with St-fibers fibers in NR, in spite of a general decrease in $\tan \delta$ for both fiber-filled compounds compared with the compound without fibers.

For EPDM, the chemical adhesion still gives an advantage to RFL-treated fibers, even with this amount of prestrain. It can also be seen that with increasing strain, the RFL-curve approaches the St-curve as the result of breakage of the chemical bonds at higher strains.

Frequency Sweep Measurements

Frequency sweep tests have been performed on both NR and EPDM samples at 0.5% strain without prestrain and the results are presented in Figure 8. It has been tried to do the tests also

with prestrain, but the data scattering was outside an acceptable range, so the results are not presented. For EPDM, at increased frequencies, the mechanical interaction between fibers and rubber prevents or limits mutual sliding with the corresponding energy dissipation and becomes the dominant effect: $\tan \delta$ values of these fiber-filled compounds are lower than for the compound without fibers. For NR, there is not much difference between the compound without fibers and the compound containing RFL-treated fibers, but in this low strain regime of 0.5% RFL leads to lower loss angles in the whole frequency range compared with St-fibers. Comparing this with the strain sweep test results it can be seen that in the stiffer matrix of NR, the degree of reinforcement is more sensitive to strain rather than to frequency.

Elongation Set

To investigate the rubber–fiber interaction and sliding of rubber along the fiber surface by a different method, elongation set tests have been performed on the samples. As this is a rather primitive mechanical test with lower sensitivity compared with DMA, the fiber contents had been increased slightly, to 5 phr. The results are presented in Figure 9. For both NR and EPDM, adding St-treated fibers results in a considerable increase in the set. This is more pronounced for EPDM compared with NR. The interesting point is that replacing the St-treated fibers with RFL-treated, the set decreases to a large extent. St-fibers are not able to form chemical bonds, resulting in weaker interfaces. So the rubber can more readily slide along the fiber surface, which in the static test of elongation set is reflected in a higher value, and in dynamic tests in a lower storage modulus and higher loss angle compared with RFL-treated fibers.

DISCUSSION

Adding fibers in fact means generating interfaces (and in some cases interphases) into a material. If the rubber would be per-

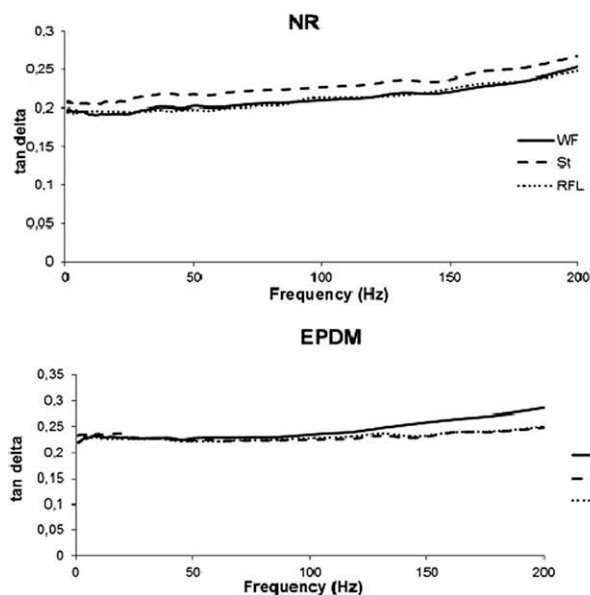


Figure 8. Loss factor $\tan \delta$ derived from frequency sweep DMA measurements at ambient temperature, without prestrain, strain of 0.5%, on 3 phr fiber-loaded NR and EPDM compounds for various fiber treatments.

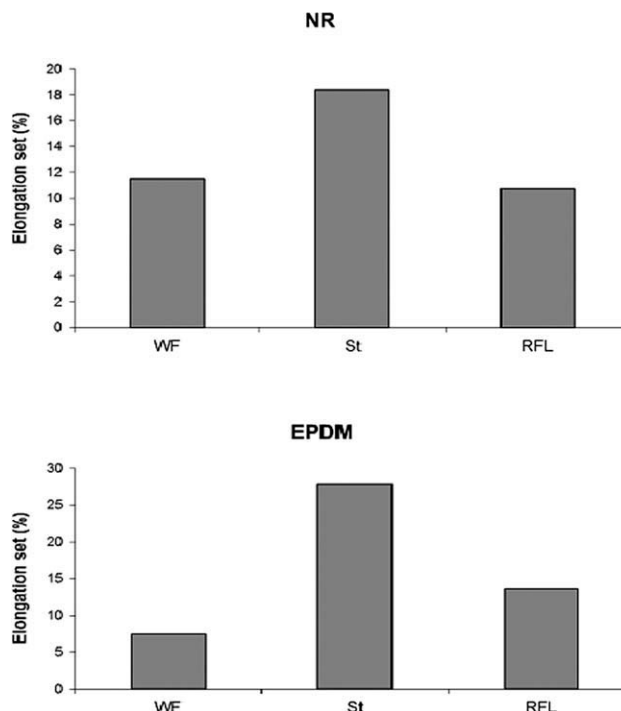


Figure 9. Elongation sets, 24 h, ambient temperature, NR and EPDM compounds with 5 phr fiber loading.

fectly and ideally bonded to the fibers' surface, the storage modulus would increase and the contribution of the interface to the loss modulus would not be significant. But in practice there is no perfect and totally uniform bonding taking place and reinforcement happens by combined effects of several mechanisms. In this study, for St-fibers there is no chemical bond, but for RFL-fibers part of the fibers which are properly coated, depending on the curing system, are able to generate chemical bonds to the rubber matrix.¹⁶ Rubber which is not chemically attached to the fibers can partially slide along the fiber surface and so create friction. This results in increasing elongation set or additional losses in dynamic loading. Despite that, in a considerable range of strains, temperatures and frequencies, reinforcement still has a possible effect in the sense that it keeps the loss values at the same level as for the compound without fibers or even lowers these to some extent. It is also possible to decrease the set properties by improving fiber–rubber interaction.

An example of using St-fibers to increase the storage modulus of rubbers without negative effects on loss properties is NR with prestrain. If in actual practice, a NR compound is under permanent load and subjected to a dynamic stress, 3 phr of fibers without treatment can enhance the dynamic properties. For RFL treated fibers with higher degree of reinforcement the situation is even better. Generally, adding RFL-treated fibers to a compound result in increasing storage modulus, with almost no negative effects or even a decrease in $\tan \delta$. This can be attributed their higher interaction with the rubber matrix, chemical as well as mechanical.

CONCLUSIONS

The viscoelastic properties which are of great importance in dynamic applications, especially in tires, have been investigated in different modes. It was shown that mechanical and chemical interactions between short fibers and a rubber matrix, which are the reasons for an increase in storage modulus, can affect loss properties in different ways, depending on the type of matrix, temperature, dynamic strain and the possible application of a static prestrain.

There are two main influencing factors which should be considered: (1) reinforcement as a result of interaction between fibers and rubber which results in an increase in storage modulus of the composite; (2) if there is no perfect interphase and fibers and rubbers are not fully bonded, sliding of the rubber matrix along the fiber surface causes additional losses due to friction.

In general, it will not be possible to obtain similar reinforcement with short fibers compared with long ones; considering the advantage of the lower processing costs of short fiber-reinforced composites, there is still room for improvement in their applications. It has been shown that chemical interaction is not the only mechanism for reinforcement of rubbers with short fibers, but that mechanical interactions are also of great importance.

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