Technical

Material composition effects on OE car tire performance

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Executive summary

This joint paper by Ford Motor Co. and Arlanxeo investigates the impact of material composition of original equipment tires as it relates to their tested performance. This work combines the global expertise of the largest synthetic rubber producer in the world with that of one of the leading global automobile producers.

The study includes a wide range of OE tires sourced from various regions and manufacturers. Tire composition analysis was performed on various parts of the tire using the expertise of the Arlanxeo Technical Center in London, Ontario, combined with tire performance data measured by Ford in Dearborn, Mich.

The presented data will highlight the need for high performance materials to attain the demanding performance requirements of original equipment applications.

This paper summarizes the results of an original equipment passenger car radial (PCR) tire survey conducted jointly between Arlanxeo's Tire and Specialty Rubber (TSR) business unit and Ford Motor Co. The survey provides a global overview of tire technology, with tires sourced from most of the top manufacturers and regions.

As tire technology continues to improve, driven by the specific requirements of automobile manufacturers, it is important to understand these developments on a global basis. The Arlanxeo TSR business unit has a unique position, supplying synthetic elastomers used in all areas of the tire to producers worldwide from a truly global manufacturing footprint. Ford is a leading global automobile manufacturer that specifies the performance requirements for the tires fitted on its new vehicles to improve fuel economy, performance and safety as part of the entire vehicle system. Together, these companies can offer a very unique perspective to the developments in PCR tires.

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The current tire survey is focused on OE PCR tires from a whole tire perspective, including material analysis of various parts of the tire and overall performance testing of the tire. Over the last several decades, significant focus has been put on the improvement of tread technology in order to lower the rolling resistance to meet new fuel efficiency requirements for new vehicles while maintaining the grip requirements of performance and safety.

This has been driven by the ever increasing fuel economy and emissions regulations imposed on new vehicles around the world, such as the CAFE standards in the U.S.[1] The original "Green Tire" patent by Michelin is seen as one of the key milestones in the improvement of the tire properties, achieving a better balance of rolling resistance and wet traction.[2]

Since then, improvements in synthetic elastomers for treads such as neodymium polybutadiene and functionalized SSBR materials in combination with silica technology have allowed for significant rolling resistance improvements without detrimentally affecting treadwear or grip, the other corners of the well-known "magic triangle for tire technology."[3]

Tire producers will continue to push technology of the tread in order to further increase fuel economy to meet existing or new regulations without affecting durability or safety (grip) of the tire. Significant focus was put on analysis of the treads in the selected OE tires because of its importance in the overall performance of the tire. Additionally, other areas of the tires also were investigated, including the sidewall, apex, rim cushion and innerliners.

The PCR and TBR survey presented herein provides a global perspective of tire technology, with tires sourced from all major manufacturers and regions. As much as possible, proportional representation of tires included in the survey was based on global sales ranking of each manufacturer and the major regions of production.

Analysis of tire segments was performed using various analytical techniques to understand the composition of the various portions of the tires included in the survey. Dynamic mechanical analysis of specific components within the tire was performed in order to predict their effect on the overall tire performance.

This material analysis and performance predictive testing were then compared to the actual rolling resistance and traction data measure for each tire, and attempts were made to correlate the information.

Fig. 1: Regional OE fitment distribution of 35 tires selected for the PCR survey. (SA = South America, NA = North America, EU = Europe, CN = Greater China, IN = India).

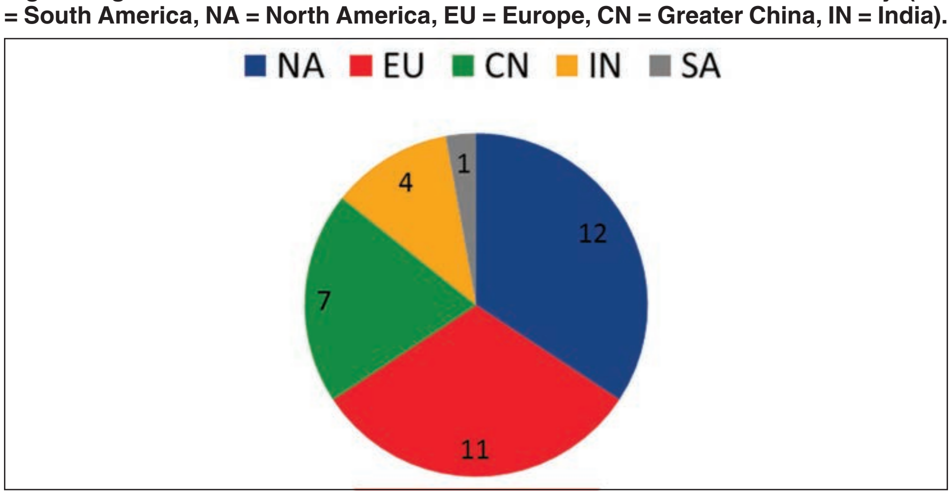
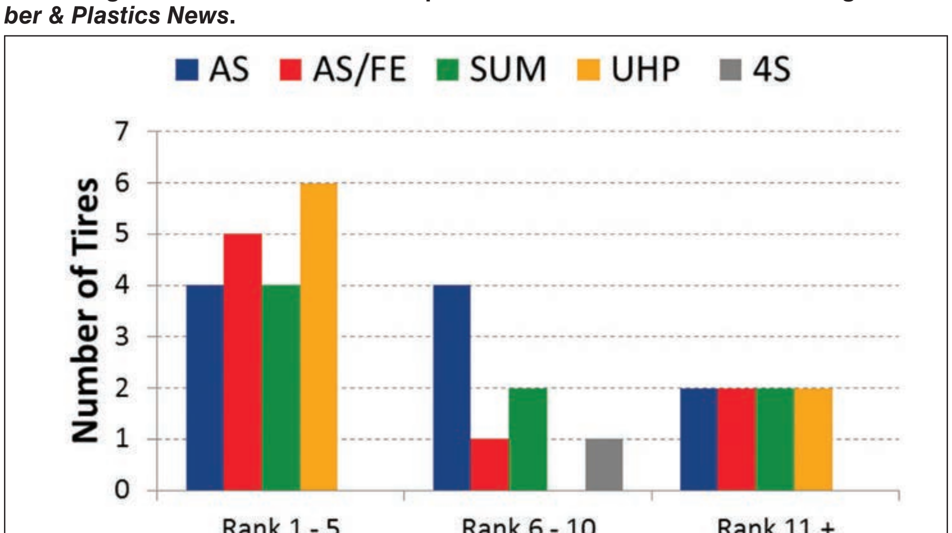


Fig. 2: Distribution of the 35 OE tires selected for the PCR survey by classification according to the 2016 Global Tire Report for tire manufacturers according to *Rubber & Plastics News*.



Experimental

For this PCR OE tire survey, a total of 37 tires produced in 2015 or 2016 were selected from the global top 20 tire producers according to overall tire sales figures for 2016 as reported by *Rubber & Plastics News* in its 2016 Global Tire Report.[4] For the most part, a proportional number of tires were chosen from each manufacturer according to their global ranking, and from each major manufacturing region wherever possible.

The composition of both tread and IL was determined via thermo-gravimetric analysis (TGA) to quantify the proportion of volatiles/extractables, polymer, carbon black and ash in each tire segment. The measurement was performed by heating a sample of the tread or IL compound at 20°C/minute under nitrogen to 550°C, followed by cooling to 300°C, and finally heating in air to 875°C.

Elemental composition of the ash was measured by SEM-EDX (external lab) in order to determine the inorganic filler type in both tread and IL samples. The halobutyl rubber (HIIR) type was determined by XRF analysis. The trace metal analysis was performed by ICP high

Table 1: Estimated weight percentage of rubber in various parts of a PCR tire.

Tire Area	Rubber (wt%)[5]	Effect on RRc (%)[6]	Effect on Grip (%)
Tread	32.6	54	~100
Tread Base	1.7	13	n/a
Sidewall	21.9	10	n/a
Bead Apex	5.0	6	n/a
Bead Insulation	1.2		n/a
Fabric Insulation	11.8	13	n/a
Steel Cord Insulation	9.5		n/a
Undercushion	3.9		n/a
Innerliner	12.4	5	n/a
Total	100	100	100

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Teertstra started his career 11 years ago with Lanxess as a research scientist in the Global Butyl research and development group. He worked on various new product development projects of butyl and butyl-like materials for both tire and non-tire applications.

In 2011, he relocated to Singapore as part of the commissioning team for a new Lanxess butyl rubber plant. Following a successful plant start-up in 2013, Teertstra relocated back to Canada, taking on a manufacturing support role with particular focus on the Singapore plant during its first year of operation. More recently, he made a transition to his current technical marketing role with the formation of the TSR business unit in early 2015.

Teertstra received both his bachelor's and doctorate from the University of Waterloo, in Waterloo, Ontario.

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Tardiff has been at Ford for 25 years, conducting project work focused primarily on developing and implementing advanced materials technologies into automotive materials. Her positions within Ford have varied from research to manufacturing, giving her experience in the development, implementation and maintenance of new technologies in the automotive industry.

temperature and pressure closed system microwave dissolution of the samples followed by inductively coupled plasma atomic emission spectroscopy (ICP) analysis using a Varian 725 Radial ICP-AES (external lab).

The proportion of BR/SBR/NR in various parts of the tire was determined using pyrolysis-GCMS. This technique involves pyrolyzing samples in an inert atmosphere and analyzing the pyrolyzate by GCMS, where characteristic polymer decomposition signals are quantified using a calibration curve established with rubber compounds of known composition run under the same conditions.

The temperature dependent modulus and loss factor were determined for samples taken from the tires. Tension clamps were used to test rectangular specimens cut from the tread and sidewall areas of tire segments in a temperature sweep experiment performed at 0.1 percent dynamic strain and a frequency of 10 Hz. Additionally, strain sweep experiments were performed with double shear clamps at 10 Hz under isothermal conditions (60°C) using cylindrical specimens cut from the tread and sidewall areas.

The rolling resistance of the tires was measured according to SAE J2452 on a MTS rolling resistance measurement system. The MTS Flat-Trac III Tire Test System was used to measure the lateral and longitudinal forces on the tire, providing the coefficient of friction (µ) values, which describes the grip or traction characteristics of the tire in each direction.

Results and discussion

OE PCR Tire Selection

The tires were selected from most major manufacturing regions as shown in **Fig. 1**, with proportional representation reflective of the total global PCR tire sales data.[4] Only OE tires were selected to be included in this survey, with 15 of the top 25 global tire companies represented in these selections.

A greater proportion of tires were selected from the top producers, as these companies represent a significant proportion of the total global tire sales. Tires were sourced from at least two different regions for the top five global producers.

The tires can be further distinguished into all season (AS), all-season fuel efficient (AS/FE), summer (SUM), ultra-high performance (UHP) and four-season tire classes (4S). For the purpose of this study, AS tires are considered as three-season tires in comparison to a true 4S tire designed also for use in winter driving conditions (snow and ice).

As much as possible the selection of the different tire classes was distributed among the various company rankings, as shown in **Fig. 2**. A larger number of tires were sourced from the top five globally ranked tire companies as these combined account for nearly 50 percent of the global tire sales for 2016.[4]

Material analysis was performed on five areas of the tire, namely the tread, sidewall, apex, rim cushion and innerliner. These areas of the tire combined represent about 70 wt-percent of the overall tire as illustrated in **Table 1**, and thus have a significant effect on the overall performance of the tire.

The material analysis results of these five areas of the tire are presented in this paper, with attempts made to correlate the findings with the actual measured performance of the tires.

Tire Treads

Significant focus has been given by tire manufacturers to development of tire tread technology. It is clearly seen in **Table 1** that the estimated contribution to the rolling resistance of a PCR tire is more than 50 percent because of the tread area,[6] and the contribution of the tread to the grip of the tire on the road is about 100 percent. This is particularly important for OE tires, where the rolling resistance and traction performance of the tire is specified for the particular vehicle fitment.

The tread sections from the 35 tires included in the survey were analyzed for composition by TGA analysis, to determine the proportions of carbon black (CB), polymer, volatiles and ash. Treads with high ash content contain silica, and this was confirmed by SEM-EDX elemental analysis of the residual ash left after TGA analysis.

The levels of other inorganic additives such as zinc oxide (ZnO) also could be determined through this analysis. In general, the majority of the tire treads analyzed in this study contain high levels of silica. Silica technology in the tread was introduced several decades ago, and was a step-change improvement in tire technology.

Further improvements in the technology since that time have provided overall benefits in tire grip, treadwear and rolling resistance (fuel economy), and usage of hydrophibized silica with silanes in particular with high performance rubber such as neodymium butadiene or solution styrene-butadiene rubber have become the industry benchmark.[7,8]

Only one tire of the 35 included in the survey contained an all-CB tread compound, and two others were approximately 50-50 blends of silica and CB. The average filler content of the tread compounds for the five classifications of tires included in this survey as determined by TGA analysis was compared to the rolling resistance performance measured of the entire tire in **Fig. 3**. Both the ash content, consisting mainly of silica, and the total filler content (ash content + CB) were plotted.

When looking at the data in **Fig. 3**, it is evident that the filler content is very similar for both the AS and AS/FE tires, even though the RRc measured for the AS/FE tires is significantly lower. This could indicate the use of optimized materials in the tread compound, including higher performance polymers (eg. functionalized). Of course the RRc is measured on the entire tire, and the AS/FE tires may have features in areas other than the treads that also are helping to increase their overall fuel efficiency rating.

The SUM tires have a similar level of total filler, but a lower level of silica and more CB. This is likely combined with a higher T_g polymer in order to increase the traction of these tires and results in the higher average RRc observed for these tires.

The total filler and silica contents measured for the UHP tires is significantly higher than that seen in either the AS or SUM tires, and the proportion of inorganic filler (silica) used is observed to be significantly higher than the other tire classes. As a result the RRc measured for these tires is significantly higher, but this is not surprising because these tires have been optimized for performance (grip) requirements rather than fuel economy.

These tires also have a very wide contact patch on the road, likely also increasing their measured RRc values. The 4S tire included in this survey is also showing a high filler content with high amounts of silica, likely a result of the requirement of ice and snow traction as a true four-season tire.

The average filler content of the tread compounds for the five classifications of tires included in this survey was further compared to the tire traction performance measured in the lateral direction for each tire in **Fig. 4**. Similarly to **Fig. 3**, both the ash content, consisting mainly of silica, and the total filler content were plotted.

In this case the grip of the tire can be much more directly linked to the materials used in the tread compound, as it is the only part of the tire responsible for traction. The AS and AS/FE tires show comparable grip for similar filler levels. This is not unexpected as these tires should have very similar requirements for these tire classes. It is interesting to note that the AS/FE tires were able to maintain traction with an overall improved RRc value, as observed in **Fig. 3**.

The SUM tires included in the survey do show a slightly higher lateral grip than the AS tires at similar total filler levels, likely due to differences in the polymers. The UHP tires show a significant increase in traction at the higher silica loadings, as expected for these performance tires designed for traction and handling. This can be a result of higher silica loading and/or increased level of silanization, or the usage of a higher T_g or functionalized polymers for enhanced grip.

The 4S tire included in this survey has dry grip behavior similar to the level of the SUM tires included in this survey, a result of its focus on improved traction in snow and ice conditions.

In addition to the performance testing of the overall tire, sample pieces also were cut out of the tread and analyzed by

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Fig. 3: Inorganic filler (ash) and total filler (ash + CB) for the various tire classes compared to the rolling resistance (RRc) of the tire. The errors bars denote 1 standard deviation of the data.

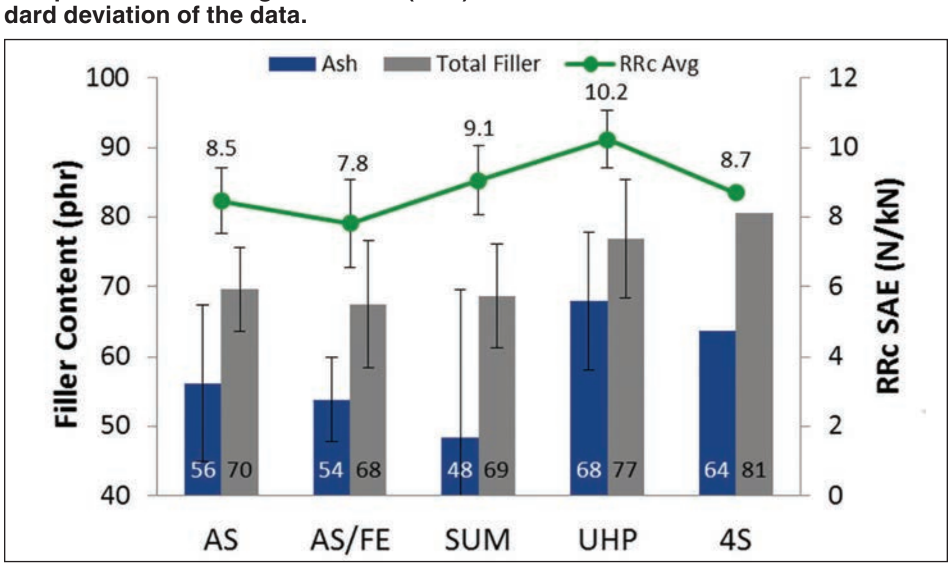


Fig. 4: Inorganic filler (ash) and total filler (ash + CB) for the various tire classes compared to the lateral grip (Lat µ) of the tire. The errors bars denote 1 standard deviation of the data.

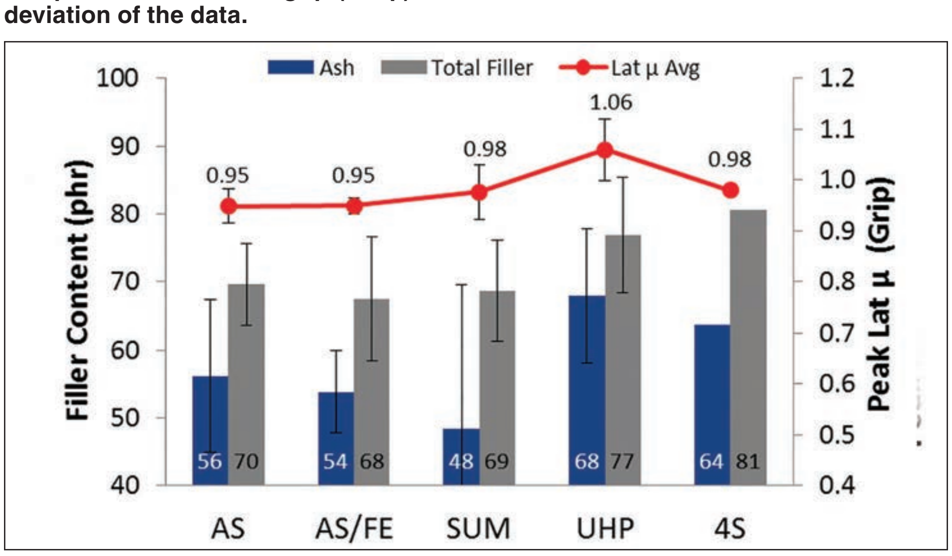
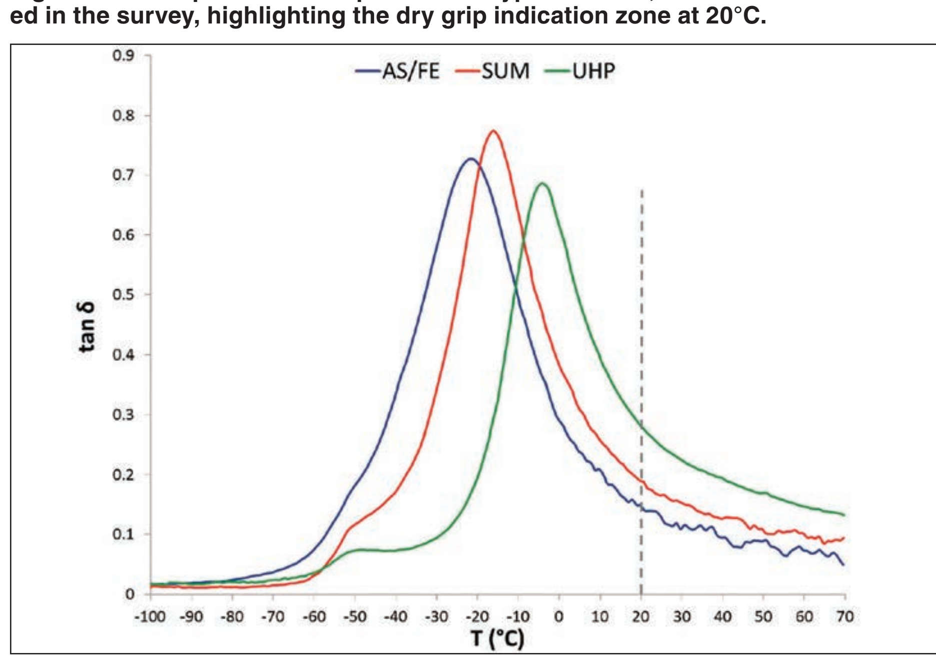


Fig. 5: DMA temperature sweep data for a typical AS/FE, SUM and UHP tire included in the survey, highlighting the dry grip indication zone at 20°C.



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DMA in order to assess the predicted performance of tread compound.

Fig. 5 displays the tan delta curves generated during a temperature sweep experiment for one tire chosen from each of the AS/FE, SUM and UHP classifications. This data was chosen to be included in this paper as it represented a good average tan delta/temperature curve within each tire classification.

It can be seen that the main polymer tan delta peak is shifted to higher temperatures when moving from AS/FE to SUM to UHP, as higher T_g polymers are being used to enhance grip. This can be achieved by using increased levels of SSBR, high styrene or high vinyl containing SSBR materials in the tread compound.

It is well known that the tan delta at 20°C can be used to predict the dry grip performance of a tread compound, with better grip or traction indicated by a higher tan delta value. The curves in **Fig. 5** show the UHP tire has the best predicted grip, followed by the SUM tire and the AS/FE. This is expected based on the tire types and was observed in the actual tire testing data discussed in the previous section.

A shoulder also can be observed on the tan delta versus temperature curves in **Fig. 5** at about -50°C, and is attributed to the presence of NR. This was confirmed by analysis of the treads using pyrolysis-GCMS, where signals from the pyrolytic decomposition products of NR could be detected. In fact, NR was detected in the treads of all but three of the tires included in the survey. The usage of NR has been increasing in PCR tire treads in general, likely a result of continuing low prices.

The average tan delta values measured at 20°C are compared to the average tire lateral grip data measured for each tire class in **Fig. 6**. It clearly is seen that the predicted performance based on the DMA measurement fits the trend of the actual grip data generated for each classification of tire and follow the expected trend for each type of tire.

The UHP tires display the highest predicted and actual grip followed by the SUM, while the AS, AS/FE and 4S tire are similar. These results are not unexpected considering the end-use performance requirements of each classification of tire.

Analysis of SBR and BR Rubber Type

The type of SBR and BR rubber contained in the tread could be determined from the trace element analysis by ICP. The presence of trace metals related to particular catalyst types indicates the type of SBR and/or BR rubber contained in the tread, sidewall, apex and rim cushion areas of the tire. In the tread area, it clearly could be seen that all but one tire contained trace lithium signatures. These are likely arising from the usage of SSBR, which is produced by anionic polymerization using alkyl-lithium initiators.

The presence of SSBR is not surprising as the majority of tires also contained high silica levels. Normally, silica technology is combined with a high performance material such as SSBR to achieve the best properties of fuel economy and grip for a tire.[9]

The other areas of the tire displayed the opposite trend, as lithium was only detected in 10 sidewalls, four apex and five rim cushions. In these other areas of the tire, compounds typically are NR or NR/BR blends. The presence of trace lithium could suggest work-away of other compounds or the addition of SSBR or Li-BR for particular performance benefits.

The presence of trace neodymium was of particular interest, and it was seen in various parts of the tire. As there are no other sources of Nd normally found in a tire, the presence of this trace metal is likely to indicate the usage of Nd-BR in the compound. Nd-BR is considered a higher performance BR rubber due to its highly linear nature, narrow molecular weight distribution and extremely low vinyl content, which result in improved dynamic performance (lower rolling resistance) as compared to standard high cis-1,4 BR rubber (Ni, Ti or Co catalyzed).[8]

The percentage of tires which were found to contain Nd-BR in their treads, sidewalls (SW), apex or rim cushion (RC) is shown in **Fig. 7**. The data is arranged according to grouping of the global tire manufacturer rankings. It was observed that Nd-BR was more prevalent in tires from the top five ranked tire manufacturers than for those ranked six and higher, indicating that these tires are utilizing this high performance material to improve the performance of their tires.

Sidewall

The sidewall sections from the 35 tires included in the survey were analyzed for composition by TGA analysis, to determine the proportions of carbon black, polymer, volatiles and ash. The sidewalls were found to all be CB compounds, as the ash content was less than 5 phr for all of the tires tested.

The level of CB ranged between 31 to 54 phr and the volatiles ranged between 7 and 17 phr. Unlike tread compounds, which can be very different based on the requirements of the specific type of tire in terms of both fuel efficiency and traction, the sidewall compounds are much more consistent as its function is similar in the different tire types.

Samples of each sidewall were analyzed by DMA in order to predict their influence on the overall performance of the tire. For this purpose, a strain sweep experiment was performed at constant temperature (60°C) and frequency (10 Hz). In this way, the contribution to the rolling resistance of the sidewall could be estimated from the tan delta, with a lower value indicating improved RRc.

The composition of the sidewall cannot be effectively compared to the overall RRc measured on the tire, since this part of the tire contributes < 10 percent to the overall value.[5]

A plot of the tan delta value measured during the strain sweep experiment at 5 percent strain as compared to the polymer content of the sidewall as measured by TGA (**Fig. 8**) shows a trend toward lower tan delta values at higher rubber contents. This is expected, as decreased filler and/or volatile loadings in a compound generally improve its dynamic properties.

The data in **Fig. 7** indicated that a number of tires included in this survey were found to contain Nd-BR in their sidewalls. The predicted (DMA) and actual measured performance (RRc) of the tires which contain Nd-BR in their sidewall were compared to those tires where Nd was not detected in their sidewalls in **Fig. 9**.

It is clearly seen that the measured tan delta value at 5 percent strain for the sidewalls that contain Nd-BR is lower than that of the tires without Nd-BR, showing the benefit of this high-performance rubber. It is interesting to note that the opposite trend is observed when looking at the actual RRc measured for the tire, where the tires without Nd-BR

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Fig. 6: Comparison of the average predicted dry grip by DMA (tan δ @ 20°C) for each tire class to that of the actual lateral grip measured of the tire (peak Lat μ). The errors bars denote 1 standard deviation of the data.

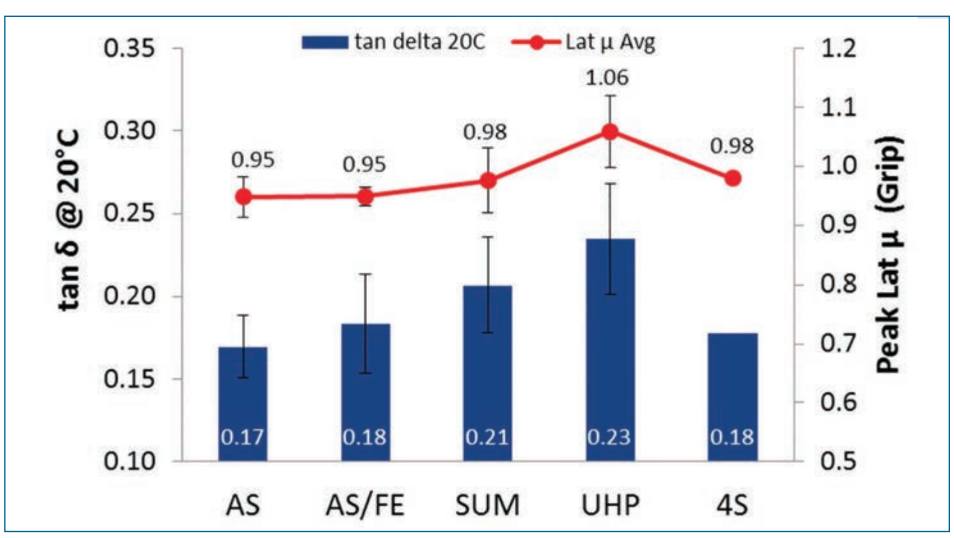


Fig. 7: Percentage of tires within select ranges of tire company global rankings that contain Nd-BR in the tread, sidewall, apex and rim cushion portions of the tire.

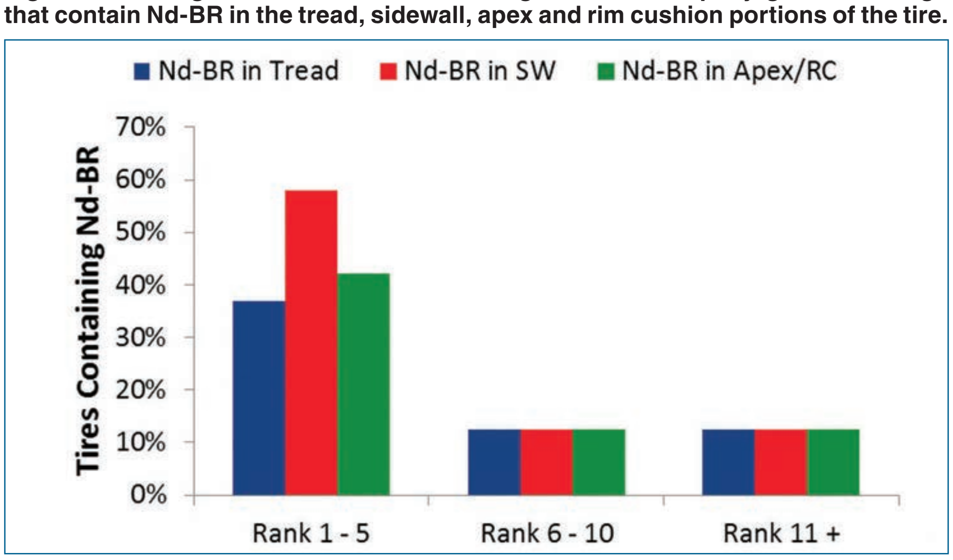


Fig. 8: Plot of tan delta value at 5 percent strain measured during a constant temperature (60°C) and frequency (10 Hz) strain sweep experiment compared to the polymer content of the sidewall as measured by TGA.

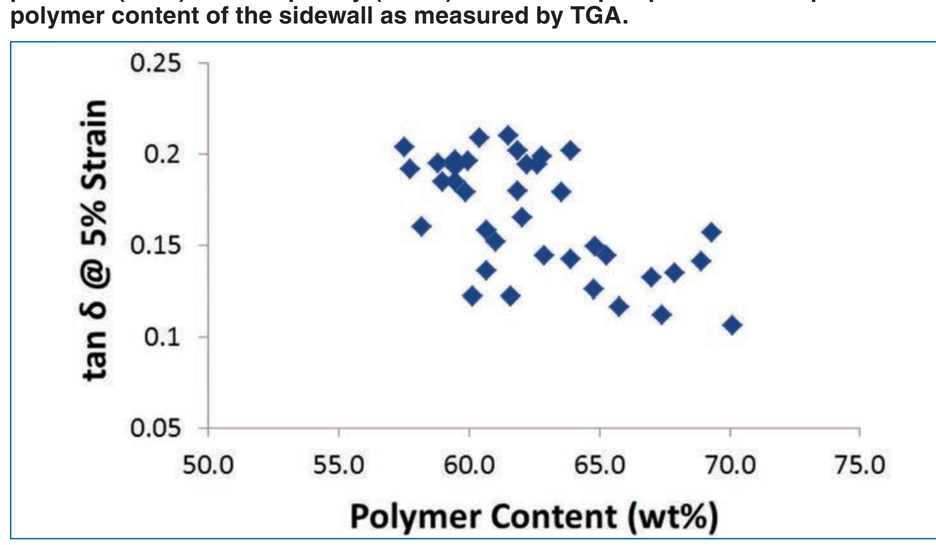
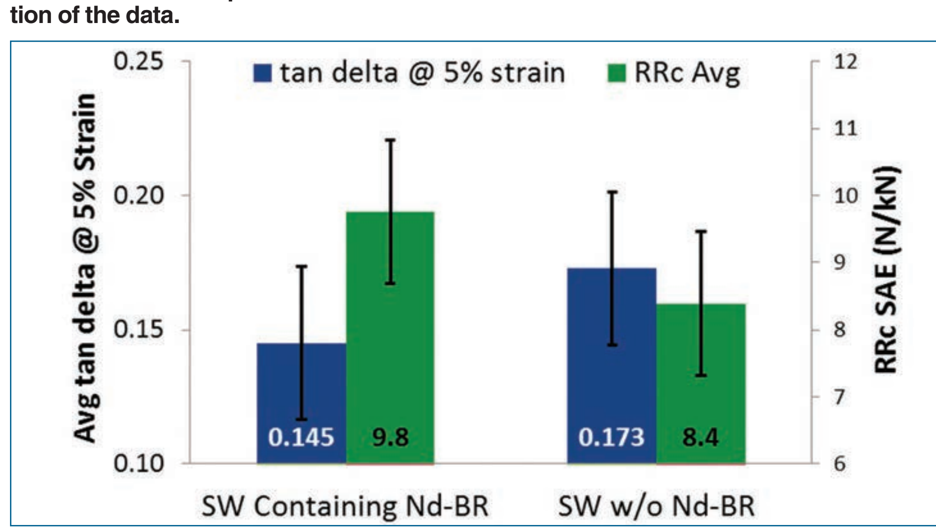


Fig. 9: Comparison of the average predicted rolling resistance behavior (tan δ @ 5 percent from a strain sweep experiment) to the average actual RRc measured for tires with and without Nd-BR present in the sidewalls. The errors bars denote 1 standard deviation of the data.



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were found to have the lower overall values and better fuel efficiency.

To further explain this difference, the distribution of the tires containing Nd-BR in their sidewalls is shown in **Fig. 10**, which shows that the Nd-BR is mainly being used in the sidewalls of SUM, UHP and 4S tires, with only one tire in both the AS and AS/FE segments found to contain Nd.

It is likely that the Nd-BR is being used in these tires to improve the overall RRc, as the tread has been optimized in order to increase grip for performance requirements. The AS and AS/FE tires in general have tread formulations that already are optimized toward improved RRc for the tire, and therefore improved performance of the sidewall is not necessary.

Innerliner

The innerliner is a key component in the tire acting as an air barrier for retaining the proper pressure during the operation of the tire. The amount and type of rubber used in the IL is a key factor determining its barrier properties. In general, a higher proportion of halobutyl rubber (HIIR) will result in improved air retention of the tire.[10]

Air retention is critical to the performance of the tire, as it has been effectively shown that the performance of the tire in terms of both fuel efficiency and stopping distance (safety) are negatively affected if the proper air pressure of the tire is not maintained.[11, 12] The best performing tires only do so when at or near the proper recommended air pressure.

The type of halobutyl rubber used in the innerliners of the tires included in the survey was determined by X-ray fluorescence spectroscopy. The innerliners normally contain either bromobutyl (BIIR) or chlorobutyl (CIIR).

NR is typically used with CIIR to achieve the required cured adhesion properties. It is known for such polymer blends that the air retention capability of tires containing higher levels of NR will be decreased due to the increased permeability of the innerliner.[13]

Tires that use BIIR do not require any NR, and therefore will have better air retention properties. Additionally these innerliners can be made thinner in order to reduce the weight and rolling mass of a tire for improved fuel efficiency while maintaining the same air retention ability as a thicker CIIR/NR blended innerliner.

The distribution of tires that contained BIIR innerliners among specific groupings of tire manufacturer global rankings is shown in **Fig. 11**. It is clearly seen that the top five global manufacturers are almost exclusively using BIIR in the innerliner, as only one out of the 19 tires included in the survey did not contain BIIR. The companies ranked six to 10 also were using BIIR at a higher level, as six out of eight tires were found to contain BIIR.

However, the lower ranked companies in the survey (rank 11+) were found to be using predominantly CIIR/NR blends. These tires are expected to have poorer air retention characteristics.

Summary

This paper provides information gathered from a survey of OE passenger car tires manufactured in 2015 and 2016. Five areas of the tire were analyzed to determine their composition and individual predicted performance, and compared to the actual measured rolling resistance (fuel efficiency) and grip (tire traction) of each tire.

Particular focus was given to the tire treads in this study, as this portion of the tire accounts for a large percentage of the rolling resistance (~50 percent) and all of the grip performance of the tire. The tire treads of the OE tires included in this survey were almost exclusively high silica compounds, with only one tire found to contain an all-CB compound and 2 tires found to contain a 50-50 CB/silica blend.

The total filler content was found to be similar for the AS, AS/FE and SUM tire classes, with the SUM tires containing on average more CB and less silica. The UHP and 4S tires were found to contain higher levels of total filler, and the UHP tires contained the highest proportion of silica. These different proportions and levels of fillers are a result of the SUM and UHP tires being optimized to enhance their dry grip performance, while the 4S tire also is optimized for snow and ice traction.

The average measured RRc for each class of tire follows the trend of filler level (UHP>SUM>4S~AS>AS/FE) and fits with the intended performance requirements. The measured lateral grip performance of the tire follows a similar trend (UHP>>SUM~4S>AS~AS/FE) with the higher silica filler level treads showing the best traction performance.

DMA analysis of the treads showed a progression to higher Tg compounds as the traction performance requirement of the tire was increased (UHP>SUM>AS). Additionally, it was observed in the DMA and confirmed by pyrolysis-GCMS measurements that NR was present in a majority of the tread compounds for the tires included in this survey.

The sidewalls of the OE tires included in this survey all were found to be CB compounds that were fairly consistent in their proportions. The estimated rolling resistance contribution as measured by DMA for samples taken from the sidewall was observed to decrease as the total polymer content of the sidewall was increased.

Interestingly, the sidewalls that contained Nd-BR rubber showed lower estimated rolling resistance on average than those that did not contain this high performance rubber. The use of Nd-BR in the sidewalls also was more prevalent in the UHP, 4S and SUM tires, presumably to gain some additional RRc improvement for the tires where the treads have been optimized more toward improved traction.

Overall, the usage of Nd-BR was more prevalent in the OE tires from the top five manufacturers as compared to tire manufacturers ranked six and lower.

The type of halobutyl used in the innerliner is an important factor in its ability as a barrier material to retain proper air pressure in a tire and maintain its expected performance.

The usage of BIIR rubber allows for 100 percent butyl to be used in the innerliner, leading to improved impermeability. CIIR rubber requires blending with NR, which lowers the ability of the innerliner to retain air at a similar thickness. A greater proportion of tires from the top 10 global manufacturers were found to contain BIIR in their innerliner, and will have improved air retention.

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Fig. 10: Distribution of sidewalls containing Nd-BR among the tire classifications.

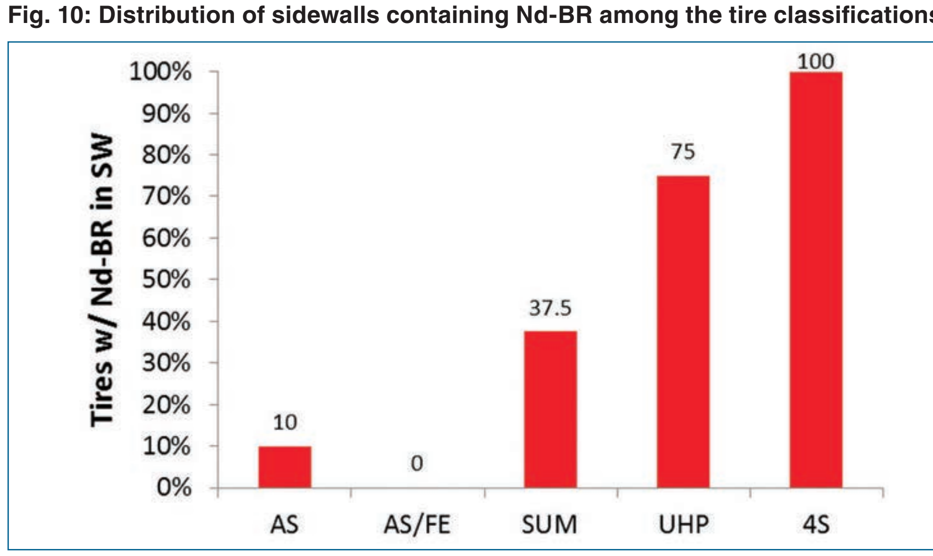

Fig. 11: Percentage of tires within select ranges of tire company global rankings that contain BIIR in their innerliner.