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16. Abstract
There were two parts to this work: (1) tire aging testing of 10 tire models using oven aging plus a stepped-up-load test, and (2) tire aging testing of 20 tire models using oven aging plus the FMVSS No.139 endurance and low pressure tests

During the first part of this research, 10 tire models were subjected to a 2-hour break-in on a 1.707 m roadwheel at 80 km/h, then inflated with a mixture of 50 percent nitrogen gas and 50 percent oxygen gas (50%N_2/50%O_2) and aged in a circulating air oven for 3 or 5 weeks at 65°C. The fill gas was vented and refreshed weekly to maintain a sufficient supply of oxygen gas in the tire cavity to support oxidative aging. Physical properties of the critical belt edge region of the aged tires were measured and the percentage change from the new tire properties was compared to the percentage change seen in tires retrieved from service in Phoenix. All of the measured physical properties changed in the same direction as the tires from Phoenix service. Oven aging from 3 to 5 weeks at 65°C produced percentage changes in properties similar to those found in tires with 3 to 6 years of service in Phoenix.

After oven aging, these 10 tire models were tested using the stepped-up-load to structural failure (SUL) roadwheel test. The running time of tires aged for 5 weeks at 65°C were similar to those of tires that had been in Phoenix service for 3 to 6 years. Five of the tire models showed failures prior to 34 hours while the load on the tires did not exceed their maximum design load. Nearly all of these failures in the passenger tires were in the critical belt edge and shoulder area. Light-truck tire failures were predominantly separations between the carcass compounds or between the innerliner and ply of the tire.

During the second part of this research, oven aging followed by the FMVSS 139 endurance and low pressure test was used to evaluate the structural durability of new tires. Twenty tire models were aged and tested to determine if this was a valid test of the durability of aged tires. Tires were oven aged for 3, 4, or 5 weeks at 65°C while inflated with the 50%N_2/50%O_2 mixture and then tested according to the FMVSS 139 endurance and low pressure tests. All new tires completed the endurance and low pressure tests. Six of 13 passenger tire models and 3 of 7 models of light-truck tires exhibited no failures after oven aging up to 5 weeks at 65°C. Approximately 70 percent of the failures for passenger tires took place in the critical belt edge and shoulder region of the tire. The light-truck tire failures were predominantly separations between components in the innerliner and sidewall region, including two tires that separated in the oven during aging.

17. Key Words
Tire, tire aging, tire safety, Phoenix, FMVSS 139, TREAD Act, accelerated service life, tire durability, roadwheel oven aging

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EXECUTIVE SUMMARY

The performance tests in Federal Motor Vehicle Safety Standard (FMVSS) No. 139 subject new tires to conditions on a curved indoor roadwheel more severe than normal operating conditions on a flat road surface. These indoor roadwheel tests do not, however, evaluate the structural durability of a tire after experiencing long-term material property degradation under cyclic fatigue. A logical question is: Do thermo-oxidative degradation and fatigue cracking of internal rubber components observed in tires retrieved from service contribute to a decrease in a tire's resistance to operational conditions? For example, would 1-year-old passenger vehicle tires have less chance of a structural failure if operated while underinflated or overloaded, or during long periods of high-speed use, than tires over 8 years of age (7% of passenger vehicle tires)?

Phase 1 addressed the goal of developing a better understanding of service-related tire degradation over time. Phase 2 focused on developing an accelerated, laboratory-based test to simulate tire aging. Phase 3 compared the physical properties and roadwheel durability of 24 additional passenger and light-truck tires which had been oven aged at 60°C to 70°C to those of new tires of the same models.

The initial 34 hours of the SUL test are identical to the endurance portion of the updated FMVSS 139 test for light vehicle tires implemented subsequent to the Phase 3 work. [1] While the new tires ran significantly beyond the 34 hour portion of the SUL test, nearly half of the tires aged for 5 weeks at 65°C or 7 weeks at 70°C failed prior to 34 hours. There was no significant correlation between initial running time and the running time of tires after oven aging.

In the first part of the Phase 4 work, tires from 10 models were subjected to a 2-hour break-in on a 1.707 m roadwheel at 80 km/h, then inflated with a mixture of 50 percent nitrogen and 50 percent oxygen (hereafter $50\%N_2/50\%O_2$) and aged in a circulating air oven for 3 or 5 weeks at 65°C. The fill gas was vented and refreshed weekly to maintain a sufficient supply of oxygen gas in the tire cavity to support oxidative aging. Physical properties of the critical belt edge region of the aged tires were measured and the change from the new tire properties was compared to the change seen in tires retrieved from service in Phoenix. All measured physical properties changed in the same direction as the tires from Phoenix service. These changes indicated that the chemical reaction in the rubber compound was similar to the reaction found in tires during service, specifically oxidative degradation. Oven aging from 3 to 5 weeks at 65°C produced percentage changes in properties similar to those found in tires with 3 to 6 years of service in Phoenix.

Previous work indicated that some tires not subjected to a break-in cycle showed a dramatic increase of indentation modulus in the belt edge compounds during oven aging while the indentation modulus decreased during service in Phoenix. Research showed that a break-in cycle on a roadwheel of 2 hours at 80 km/h was sufficient to eliminate the increase of indentation modulus during oven aging without decreasing the running time.

Ten models of tires were tested using the same stepped-up-load (SUL) to structural failure roadwheel test used in Phases 2 and 3 work. The running times of tires aged for 5 weeks at 65°C were similar to those of tires that had been in Phoenix service for 3 to 6 years. Nearly all these failures in the passenger tires were in the critical belt edge and shoulder area. Failures in the

light-truck tires were predominantly separations between the carcass compounds or between the innerliner and ply of the tire, similar to results from the Phase 3 work.

The SUL test produced failures of oven-aged tires at times similar to those of the tires from service in Phoenix. It also discriminated between tire models, with a wide spread of mean time to failure from 100 percent or less of the maximum rated load for some tire models to significantly greater than the maximum design load for others.

The FMVSS 139 endurance and low pressure tests were designed to evaluate the structural durability of new tires. Twenty different tire models were aged and tested to assess how well these tests measured the durability of aged tires. Tires were oven aged for 3, 4, or 5 weeks at 65°C while inflated with the $50\%N_2/50\%O_2$ mixture, then tested according to the FMVSS 139 endurance and low pressure test. Aging 3 weeks at 65°C is approximately equivalent to 2.4 years of service and aging 5 weeks at 65°C approximates the aging experienced by a tire with 4.0 years of service in Phoenix. All new tires completed the endurance and low pressure test and 46 percent of passenger tire models and 43 percent of light-truck tire models exhibited no failures after oven aging up to 5 weeks at 65°C.

Approximately 70 percent of the failures for passenger tires took place in the critical belt edge and shoulder region of the tire. The light-truck tire failures were predominantly separations between components in the innerliner and sidewall region of the tire, including two tires that separated in the oven during aging. Inspection of the failures indicated that the separations were likely initiated by a buildup of air pressure between the components.

Seven of 13 models (54%) of passenger tires and 2 of 7 models (29%) of the light-truck models completed the endurance and low pressure test after all of the aging conditions. For the 6 models of passenger tires that failed, very few failures were observed prior to 5 weeks of oven aging. The failure times for light-truck tires were, on average, less than those for passenger tires.

Nine of the tire models had no failures after being oven aged for 5 weeks, tires from 2 models failed after 3 weeks of oven aging, 3 additional models did not complete the test after 4 weeks of oven aging, and a total of 11 models did not complete the test after 5 weeks of oven aging.

1.0 INTRODUCTION

On September 12, 2000, the U.S. Senate Committee on Commerce, Science, and Transportation conducted a hearing on the recall of 14.4 million Firestone Radial ATX, Radial ATX II, and Wilderness AT tires on specific models of Ford, Mercury, and Mazda light trucks and SUVs. During these hearings, members of Congress expressed concern that the current FMVSS standards do not evaluate how tires perform when significantly underinflated or after being subjected to environmental variables, such as heat, which accelerate aging. (2) As a result of the committee's actions, the Transportation Recall, Enhancement, Accountability, and Documentation ("TREAD") Act (H.R. 5164, Pub. L. No. 106-414) was enacted on November 1, 2000. Section 10 of the TREAD Act contained provisions mandating NHTSA to "revise and update" the passenger car and light-truck tire safety standards.

The legislation did not mandate specific test requirements. During the consideration and enactment of the TREAD Act, Members of Congress placed emphasis on improving the ability of tires to withstand the effects of factors such as heat build-up, low inflation, and aging (i.e. service-related degradation). With regard to aging, NHTSA was asked to consider whether a required "tire aging test" (i.e. accelerated service life test for tires) was feasible to evaluate the risk of failure later in the tire's life than the current regulation, which only evaluates new tires.

In response to the TREAD Act NHTSA examined the effectiveness of the current passenger vehicle tire safety standards, which had not been substantially revised since their issuance in 1967, and determined the following:

> "While the durability and performance of tires have improved, the conditions under which tires are operated have become more rigorous. Higher speeds, greater loads, extended lifetimes of tires, longer duration of travel and shifting demographics of vehicle sales have all contributed to much greater stresses and strains being placed upon today's radial tires than those endured by earlier generation radial tires. The characteristics of a radial tire construction in conjunction with present usage and purchasing patterns render the existing required minimum performance levels in the high-speed test, endurance test, strength test, and bead-unseating test ineffective in differentiating among today's radial tires with respect to these aspects of performance." (1)

NHTSA conducted tire safety research in support of what would become the new FMVSS 139, "*New pneumatic radial tires for light vehicles*".[i]

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[i] Previously, passenger tires were regulated by FMVSS 109 ("Passenger car tires") and light-truck tires under the separate FMVSS 119 ("Tires for vehicles other than passenger car"). The FMVSS 119 had less severe test conditions than the FMVSS 109 and did not include a high speed or bead unseat test for tires. The FMVSS 139 unifies regulation of the majority of passenger and light-truck tire designs for vehicles with a gross vehicle weight rating of 10,000 pounds or less. This new standard became mandatory on September 1, 2007, for non-snow tire designs and becomes mandatory on September 1, 2008, for designated snow tire designs. Optional compliance is permitted before those dates.

During this effort, NHTSA researchers conducted comprehensive literature reviews and had numerous consultations with industry regarding the long-term effects of service on radial tire durability. NHTSA concluded that while most tire manufacturers conduct some form of accelerated service life testing on their tires, their approaches varied widely and no single industry-wide recommended practice existed. As part of the FMVSS 139 development research, NHTSA evaluated multiple laboratory-based accelerated service life tests for tires that were based on industry submissions or on previous NHTSA test experience. In the March 5, 2002, Notice of Proposed Rulemaking (NPRM) section related to tire aging for FMVSS 139 (67 FR 10050), NHTSA proposed three alternative tests to evaluate a tire's long term durability. These approaches are: (1) 24 hours of roadwheel conditioning followed by an adhesion (peel strength) test between the belts; (2) an extended duration roadwheel endurance test with oxygen-rich inflation gas; and 3) an oven-aging conditioning period followed by a roadwheel endurance test. However, based on the results of an initial evaluation, as well as comments and data from industry, NHTSA decided to defer action on the proposal to add an aging test to the new FMVSS 139 until after further research. This led to the NHTSA Tire Aging Test Development Project in late 2002.

Phase 1 of the NHTSA Tire Aging Test Development Project consisted of the analysis of 6 different tire models collected from service on privately owned vehicles in the Phoenix, Arizona, metropolitan area during the spring of 2003. This study provided a better understanding of service-related tire degradation and served as a "real-world" baseline for the eventual development of laboratory-based accelerated service life test for tires (often referred to as a "tire aging test"). As part of the Phase 1 effort, 109 tires of varying age and mileage were retrieved from service in Phoenix, and their performance was compared to 45 new tires of the same type and model in 1 of 2 laboratory roadwheel tests. Analysis of this data led NHTSA to conclude that peel adhesion decreased systematically as the tires accumulated mileage and age (time in service) in Phoenix. However, the actual peel adhesion value is a complex function of the thickness of the rubber layer between the belts and the physical properties of the rubber. All new tires and most aged tires failed cohesively in the rubber layer but the intrinsic interfacial adhesion was unknown, therefore NHTSA rejected peel adhesion as a primary method of evaluating an aged tire's durability. [3]

The physical and chemical properties of the rubber compound between the belts, known as the skim and wedge compounds, changed in a manner consistent with the mechanism of thermo-oxidative aging. Specifically:

> The level of fixed oxygen (that is the oxygen reacted with the rubber compound) in the rubber compound between the belts systematically increased as the tires were in service in Phoenix. [4]
>
> The hardness and modulus of the rubber compound between the belts changes systematically as the service in Phoenix increased. [3] For 5 of the tire types the hardness increased with service time, for the 6th tire type the hardness and modulus decreased. The latter has been shown to be an effect of the reinforcing resin used in this rubber compound. [5]
>
> The ultimate elongation to break for all tires was significantly reduced as the service time of the tire in Phoenix increased. The tensile strength tended to be reduced and the modulus tended to increase, although the trends were not statistically significant for all tires. [3]

The crosslink density systematically increased as the service in Phoenix increased. A small subset of tires tested for distribution of crosslinks showed systematic changes in the crosslink density as the service life in Phoenix increased. Strong crosslinks increased while weak crosslinks decreased with the effect on intermediate crosslinks being indeterminate. (6)

Kaidou and Ahagon (1990) (7) have shown several types of aging taking place in rubber compounds depending on the temperature and available oxygen. (7), (8) The aging of the Phoenix tires corresponded to aerobic thermo-oxidative aging, as shown by the slope of the log(extension ratio at break) versus log(modulus at 100%, MPA) plots. (3)

Phase 2 focused on developing an accelerated laboratory-based tire test to simulate real world tire aging and to evaluate the remaining structural durability of the aged tires. The 6 tire models previously evaluated after long-term service (aging) on vehicles in Phoenix were evaluated using three candidate methods of laboratory aging:

1. The Long Term Durability Endurance Test, proposed by Michelin. Tires were inflated with a mixture of 50%N_2/50%O_2 and run on a 1.707 m roadwheel for up to 500 hours.

2. The Passenger Endurance Test proposed by Continental, in which the tire is run on the roadwheel for up to 240 hours.

3. An oven-aging method based on tire research by Ford, who recommended that NHTSA use a method in which the tire is inflated using the 50 percent nitrogen and 50 percent oxygen mixture, heated in an oven for a period of time to accelerate the aging process by speeding up chemical reactions, and thus material property changes. The tire may then be studied for material property changes or run on a roadwheel to determine any change in durability.

The material and chemical properties of the tire's structural components were measured for new tires and for tires after each of the candidate aging methods. This was done since over 95 percent of tire failures recorded by NHTSA in its recall and complaint database involve the tire tread and belt area. The wire-coat skim compound that is the rubber compound directly adhered to the steel belts, and the wire-coat wedge compound between the steel belt plies are directly involved in these failures. The shoulder area of the tires was studied in the most detail. Component properties tended to change in the same direction as increased time in service in Phoenix or increased time of laboratory aging of each candidate method. Specifically the hardness, modulus, cross-link density, and oxygen content tended to increase while the tensile strength, ultimate elongation, peel adhesion, and flex properties tended to decrease over time. All of these changes are consistent with the proposed mechanism of thermo-oxidative aging.

The changes in indentation modulus, ultimate elongation, modulus at 100 percent elongation, and peel adhesion in the wire skim-coat and wedge area of the laboratory-aged tires were compared to these changes for the tires taken from service in Phoenix. The changes tended to be progressively greater as the time on a roadwheel test or in an oven-aging test increased. In general, the longest roadwheel test times showed the same level of change as tires with one to three years of service in Phoenix. Oven aging at the most severe conditions tended to show the same level of change in properties as tires with three to over 6 years of service in Phoenix. Oven aging for three weeks produced very little change. Unexpectedly, aging for 3 weeks at 60°C produced a greater change in properties than aging for 3 weeks at 70°C. Oven aging for 6 weeks at 60° to 70°C or 12 weeks at 55°C produced changes similar to those found after roadwheel testing, approximately equivalent to

1 to 3 years of service in Phoenix. Aging for 8 weeks at 65° produced changes approximately equal to changes seen after 4 to greater than 6 years of service. For the type E, H, and L tires, hardness tended to decrease during roadwheel testing or service in Phoenix, while it increased during oven aging. This was found to correlate to the use of a reinforcing thermoset resin in the compounds of these tires, which would be expected to have non-reversible softening during repeated flexing and to harden with increased temperature without flexing. The mechanical energy of a break-in cycle softened these compounds and was incorporated into the Phase 3 testing.

The slope of the plot of log(ultimate elongation) versus log(modulus at 100% extension) has been shown to correspond to the type of aging of rubber compounds [(7).] A slope near -0.75 indicates that the rubber compound has experienced thermo-oxidative aging. All of the tires from Phoenix service showed slopes near -0.75 in the wire skim-coat and wedge compounds. The slopes for most tires exposed to roadwheel aging or oven aging were not close to -0.75. The increase in the oxygen content for the skim-coat, wedge, or innermost tread compounds of tires during roadwheel aging was also significantly less than for tires during service in Phoenix. Researchers determined that the oxygen content of the fill gas in tires after 6 weeks of oven aging decreased from an initial average of approximately 45 percent O_2 to approximately 35 percent O_2. On this basis, the current phase of testing will include venting the fill gas and re-inflating with the 50 percent O_2 gas at two-week intervals.

Selected oven-aged tires were tested using a stepped-up-load test intended to compare the structural integrity of the tire after aging to that of a new tire. Most tires failed at loads much higher than their rated load capacity and are not necessarily expected to show correlation to failures that may happen in normal tire service. Tires aged for 3 weeks showed no failures below 100 percent of the rated load for the tire models. Parallel to the results from the physical property changes, tires aged 3 weeks at 70°C tended to have longer running times to failure than tires aged for 3 weeks at 60°C. Tire models C and L showed no decrease in roadwheel time even after aging at the most severe conditions of 8 weeks at 65°C, even though tire model L showed the greatest loss in physical properties of the skim-coat and wedge compounds during aging. Tire models B and D showed failures below 100 percent load only at aging times of 8 weeks at 65°C. These models also had predicted failure times below 100 percent load after 5 or more years of service in Phoenix. Tire models E and H appear to be most sensitive to oven aging during roadwheel testing, and for service in Phoenix. For tire type E, aging for 8 weeks at 65°C or service in Phoenix for 3 to 4 years produced failures below 100 percent of its maximum rated load. For tire type H, 6 to 8 weeks of aging at temperatures between 60°C and 70°C or service in Phoenix for 2 to 3 years produced failures below 100 percent of its maximum rated load.

A 24-hour roadwheel break-in prior to oven aging tended to decrease the subsequent running time to failure in the test after aging was completed. If the 24 hours are added to the total running, the total running time tends to be longer than the tires without break-in. Since the break-in was done at 100 percent of maximum load, direct comparisons are only possible for tires failing at less than 34 hours on the test after aging, specifically tire models D, E, and H aged 8 weeks at 65°C. Based on these comparisons, the severity of the break-in cycle will be reduced from 24 hours at 120 km/h (75 mph) to 23 hours at 80 km/h (50 mph).

Phase 3 of the research applied the oven-aging procedure to models of tires not previously tested to determine if the physical property changes and changes in whole-tire endurance found in the 6 models tested in Phase 2 applied to a broad range of tires. The physical and chemical properties for 10 passenger and 6 light-truck tire models aged for 10 weeks at 60°C, or 8 weeks at 65°C, while inflated with a mixture of $50\%N_2/50\%O_2$ gas were compared to those of new tires of the same model. The residual integrity of 16 passenger and 9 light-truck tires were measured using a stepped-up-load test to structural failure after aging for 7 weeks at 60°C, or 5 weeks at 65°C. The effect of a 23 hour break-in cycle on a 1.707 m roadwheel at 80 km/h prior to oven aging was also investigated.

Since there were no in-service tires available for comparison, values were compared to the relative properties – the percent of the new tire property retained after aging – of the tires from service in Phoenix and oven aged in Phase 2. The physical properties of all tire models changed in the same manner as the tires investigated in Phase 1 and 2. Specifically, the indentation modulus of the compounds in the wire-coat and the shoulder area of the tire tended to increase during aging to levels greater than that of the average tire with approximately 3 years of service in service in Phoenix. The modulus of the wire coat compounds at all levels of strain increased to levels greater than tires with 3 years of service in Phoenix. The tensile strength and ultimate elongation decreased to values less than the retained values from the average tires from service in Phoenix. This is consistent with previous estimates published by Ford Motor Company and the calculated rate of reaction from molecular kinetics. The ratio of log(modulus at 100% elongation) versus log(ultimate elongation) indicates that the aging was Type 1 – oxidative aging.

Based on measured changes in material properties, oven aging tires at 60°C to 70°C while inflated with a mixture of $50\%N_2/50\%O_2$ gas produces thermo-oxidative aging effects qualitatively similar to those found in tires after long-term service in Phoenix. When measured as a percentage of new tire properties, all properties were within the range of values found for the Phase 1 and Phase 2 work. For certain tires, achieving values for modulus that were found during service requires a break-in cycle prior to oven aging, found to correlate to the use of a thermoset reinforcing resin in the rubber compounds. Researchers determined, that, the less-severe break-in cycle of 23 hours at 80 km/h still reduced the running time of the tires after oven aging compared to tires with no break-in.

Roadwheel testing using a stepped-up-load to catastrophic failure of tires oven aged for 7 weeks at 60°C or 5 weeks at 65°C (calculated to be equivalent to 3.5 to 4 years of Phoenix service) produced failures in a significantly shorter time than that of new tires. These values were within the range of values found in the Phase 1 and Phase 2 testing. Prior to aging only a single light-truck tire failed prior to the completion of the 100 percent load step of the FMVSS 139 Endurance test method. After aging 49 percent of passenger tires and 76 percent of light-truck tires failed prior to the completion of the 100 percent load step. The majority of these failures occurred in the belt and shoulder area of the tires.

The fourth phase of the work, reported here, investigates 20 tire models. 10 tire models were aged for 5 weeks at 65°C while inflated with the $50\%N_2/50\%O_2$ gas mixture, then were tested using the stepped-up-load to structural failure (SUL) test that was used in Phases 2 and 3. Three of these tire models were models used in Phase 3, and the other seven were additional models. The time to failure was compared to new tires of the same model. These 10 tire models were oven aged for 3 or 5 weeks at 65°C and the physical properties were compared to new tires of the same model. An additional 10 tire models were oven aged for 3, 4, or 5 weeks at 65°C. The aged tires were tested for roadwheel durability using the procedures specified in the FMVSS 139 endurance and low pressure regulation and compared to the results of the new tires subjected to the same tests.

2.0 METHODOLOGY

The 20 tire models studied in the Phase 4 testing are shown in Table 1. Selected models were evaluated after laboratory aging for the material properties shown in Table 2 and compared to new tires of the same model. Ten tire models were aged for 5 weeks at 65ºC and then tested using the stepped-up-load to failure protocol used in Phases 2 and 3. The test protocol is shown in Table 3. Additional tires of all 20 models were tested for durability after aging using the endurance and low pressure test methods in the FMVSS 139, and results were also compared to new tires of the same model. The first 34 hours of the FMVSS 139 method are the same as the first 34 hours of the stepped-up-load to failure protocol (SUL) shown in Table 4. Oven aging was carried out in a circulating air oven for 3, 4, or 5 weeks at 65ºC after tires were subjected to a break-in cycle of 2 hours at 80km/h (50 mph) on a 1.707 m roadwheel. A description of the tests employed in the study is shown in Appendix 1. The complete list of tires referred to in the material properties phase of the study is shown in Table 6. All raw data used in the report are available in the public NHTSA Phoenix Dataset 5.0 (9)

Table 1. Tire Models Selected for Physical Property and Roadwheel Testing

Tire Type	Application	Market	Brand	Model	Size	Load Index	Speed Index
B1	Passenger	Replacement	Bridgestone	Blizzak DM-Z3	235/70R16	105	Q
B7[2]	Passenger	Replacement	Firestone	Wilderness AT I	P265/75R16	114	S
B8	Passenger	OE	Bridgestone	B450	P205/65R15	92	S
B9	Light Truck	OE	Bridgestone	Duravis M773 II	LT265/75R16	123	-
C7	Passenger	OE	Continental	Contitrac TR	P265/70R17	113	S
C8	Passenger	OE	General	Ameri G4S	P205/65R15	92	T
D6	Passenger	Replacement	Mohave [Discount Tire]	RS	P205/65R15	92	R
G1	Passenger	Replacement	Goodyear	Ultra Grip	P235/75R15XL	108	S
H3	Light Truck	OE	Hankook	DynaPro AS	LT245/75R16	120	-
M1	Passenger	Replacement	Michelin	Cross Terrain SUV	P265/75R16	114	S
M3	Passenger	Replacement	Michelin	X-Ice	205/65R15	94	Q
M10	Light Truck	OE	Michelin	LTX A/S	LT245/75R16	120	R
N1	Light Truck	Replacement	Nokian	Hakkapeliitta 10LT	LT235/85R16	120	-
N2	Light Truck	Replacement	Nokian	Hakkapeliitta LT	LT265/75R16	119	-
O4	Light Truck	Replacement	Big O [Big O Tire]	X/T BIG FOOT (356)	LT265/75R16	123	N
P3[ii]	Passenger	Replacement	Futura [Pep Boys]	Scrambler A/P (P-XL)	P235/75R15XL	108	S
S1	Metric	Replacement	Sumitomo	HTR+	225/60R16	98	V

[2] Model also studied in Phase 3 work

Tire Type	Application	Market	Brand	Model	Size	Load Index	Speed Index
T2[ii]	Passenger	Replacement	Toyo	800 Ultra	P235/60R16	99	T
U2[ii]	Passenger	OE	Dunlop	SP Sport 4000 DSST (Run Flat)	P225/60R17	98	T
Y2	Light Truck	Replacement	Yokohama	Geolandar A/T+II	LT285/75R16	122	Q

Table 2. Material Properties Evaluated[3]

Material Property Test	Tire Component
Indentation Modulus	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Tread/Shoulder Wedge
	Shoulder
Tensile Strength and Elongation	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
180º Peel Adhesion	Skim Area of Belt
	Wedge Area of Belt
Fixed Oxygen	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound

2.1 Oven Exposure

Tires were inflated with a mixture of 50%N_2/50%O_2 to their maximum rated pressure and exposed to a constant temperature of 65ºC in a circulating air oven. Tires analyzed for material properties were aged for either 3 weeks at 65ºC or 5 weeks at 65ºC. Tires for roadwheel testing were aged for 3, 4, or 5 weeks at 65ºC, following a 2-hour break-in cycle on a roadwheel at 80 km/h. The tires were vented and refilled weekly with the 50%N_2/50%O_2 gas to maintain a sufficient supply of oxygen to support oxidative aging. After oven exposure, durability on a roadwheel test and material properties were compared to the same model of new tire.

2.2 Roadwheel Testing

After oven exposure, tires were tested using either the SUL procedure used in Phases 2 and 3 of this work or the Endurance and low pressure test of the FMVSS 139. (10) The tire is inflated, stabilized at the ambient laboratory temperature, then run continuously on a 1.707 m (67 inch) roadwheel at 120 km/h (75 mph) for 4 hours at 85 percent maximum load, followed by 6 hours at 90 percent maximum load, and then 24 hours at 100 percent maximum load. If the tire completes the initial roadwheel test intact (i.e. no catastrophic structural failures or significant loss of inflation pressure), the tire is stopped for a 1-hour cool-down period and inspected.

For the SUL test, the load is increased in 10 percent increments each hour until the tire displays a visible failure. For those tires tested using the FMVSS 139 protocol the low-pressure portion of the test is run. The inflation pressure is reduced and the tire is run for an additional 90 minutes at 100 percent of maximum load. This test is a stop-finish test at the end of the low pressure step with a total running time of 35.5 hours. The initial load and incremental loads are proportional to the

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[3] Not every test was run on every tire. The complete list of tires and all results are included in Reference (9)

maximum load rating for each tire. The Endurance test is the same as the first 34 hours of the stepped-up-load to failure (SUL) test used in the Phase 2 and Phase 3 testing. The test conditions are shown in Table 3 and Table 4 respectively. Tires that were tested using the SUL protocol are shown in Table 6 and those tested using the endurance and low pressure procedure are shown in Table 7.

Table 3. Stepped-Up-Load to Structural Failure Roadwheel Test Conditions

Endurance Test S6.3		
Inflation Pressure		
	Passenger Car Tire, Standard Load	220 kPa
	Passenger Car Tire, Extra Load	260 kPa
	Light Truck Tire, Load Range C	260 kPa
	Light Truck Tire, Load Range D	340 kPa
	Light Truck Tire, Load Range E	410 kPa
Temperature		38°C
Speed		120 km/hr
Load		
	85% Maximum	4 hrs
	90% Maximum	6 hrs
	100% Maximum	24 hrs
	Increase Load by an Additional 10% Each Hour	1 Hour Steps Until Visible Tire Failure

Table 4. FMVSS 139 Endurance and Low Pressure Test Conditions

Endurance Test S6.3		
Inflation Pressure		
	Passenger Car Tire, Standard Load	220 kPa
	Passenger Car Tire, Extra Load	260 kPa
	Light Truck Tire, Load Range C	260 kPa
	Light Truck Tire, Load Range D	340 kPa
	Light Truck Tire, Load Range E	410 kPa
Temperature		38°C
Speed		120 km/hr
Load		
	85% Maximum	4 hours
	90% Maximum	6 hours
	100% Maximum	24 hours

FMVSS 139 Endurance and Low Pressure Test Conditions (Continued)

Low Pressure Test S6.4		
Inflation Pressure		
	Passenger Car Tire, Standard Load	140 kPa
	Passenger Car Tire, Extra Load	160 kPa
	Light Truck Tire, Load Range C	200 kPa
	Light Truck Tire, Load Range D	260 kPa
	Light Truck Tire, Load Range E	320 kPa
Temperature		38°C
Speed		120 km/hr
Load		
	100% Maximum	1.5 hours

The tires analyzed in the Phase 4 materials properties testing and the oven-aging conditions are shown in Table 5.

Table 5. Tires and Tests Used in Phase 4 Material Properties Aging Study

Tire Type	Temperature	Weeks	Barcode	DOT TIN Number
P3	No Oven Aging		2056	UTHLPAN2806
	65	3	2059	UTHLPAN2806
	65	5	2060	UTHLPAN2806
B8	No Oven Aging		2286	0BURB411606
	65	3	2289	0BURB411606
	65	5	2290	0BURB411606
B9	No Oven Aging		2517	7XW8P7M1806
	65	3	2520	7XW8P7M1806
	65	5	2521	7XW8P7M1806
C7	No Oven Aging		2542	A3T645RW2206
	65	3	2545	A3T645RW2206
	65	5	2546	A3T645RW2206
C8	No Oven Aging		2567	P5UR4421806
	65	3	2570	P5UR4421806
	65	5	2571	P5UR4421806
H3	No Oven Aging		2592	T7XD5JNH4905
	65	3	2595	T7XD5JNH4905
	65	5	2596	T7XD5JNH4905
M10	No Oven Aging		2617	B72K2EHX1106
	65	3	2620	B72K2EHX1106
	65	5	2621	B72K2EHX1106
S1	No Oven Aging		2642	V4A64MCR5005
	65	5	2646	V4A64MCR5005
	65	3	2649	V4A64MCR5005
T2	No Oven Aging		2667	9TKU93A0706
	65	3	2670	9TKU93A0706
	65	5	2671	9TKU93A0706
U2	No Oven Aging		2081	EUFC3TMR4705
	65	3	2084	EUFC3TMR4705
	65	5	2085	EUFC3TMR4705

Table 6. Tires Tested on Roadwheel With Stepped-Up-Load to Structural Failure Test

Tire Type	DOT TIN Number	Aging Time, Weeks at 65°C	Barcode
B8	0BURB411606	None	2283
B8	0BURB411606	None	2284
B8	0BURB411606	5	2287
B8	0BURB411606	5	2288
B9	7XW8P7M1806	None	2514
B9	7XW8P7M1806	None	2515
B9	7XW8P7M1806	5	2524
C7	A3T645RW2206	None	2539
C7	A3T645RW2206	5	2543
C7	A3T645RW2206	5	2544
C8	P5UR4421806	None	2564
C8	P5UR4421806	None	2565
C8	P5UR4421806	5	2568
C8	P5UR4421806	5	2569
H3	T7XD5JNH4905	None	2599
H3	T7XD5JNH4905	5	2593
H3	T7XD5JNH4905	5	2594
M10	B72K2EHX1106	None	2624
M10	B72K2EHX1106	5	2618
P3	UTHLPAN2806	None	2053
P3	UTHLPAN2806	None	2054
P3	UTHLPAN2806	5	2057
P3	UTHLPAN2806	5	2058
S1	V4A64MCR5005	None	2639
S1	V4A64MCR5005	None	2640
S1	V4A64MCR5005	5	2643
S1	V4A64MCR5005	5	2644
T2	9TKU93A0706	None	2664
T2	9TKU93A0706	None	2665
T2	9TKU93A0706	5	2668
T2	9TKU93A0706	5	2669
U2	EUFC3TMR4705	None	2078
U2	EUFC3TMR4705	None	2079
U2	EUFC3TMR4705	5	2082
U2	EUFC3TMR4705	5	2083

Table 7. Tires Tested to FMVSS 139 Endurance and Low-Pressure Conditions

Tire Type	Vehicle Type	DOT TIN Number	Aging Time, Weeks at 65°C	Barcode
P3	Passenger	UTHLPAN2806	None	2045
		UTHLPAN2806	None	2046
		UTHLPAN2806	3	2047
		UTHLPAN2806	3	2048
		UTHLPAN2806	4	2049
		UTHLPAN2806	4	2050
		UTHLPAN2806	5	2051
		UTHLPAN2806	5	2052
U2	Passenger	EUFC3TMR4705	None	2070
		EUFC3TMR4705	None	2071
		EUFC3TMR4705	3	2072
		EUFC3TMR4705	3	2073
		EUFC3TMR4705	4	2074
		EUFC3TMR4705	4	2075
		EUFC3TMR4705	5	2076
		EUFC3TMR4705	5	2077
G1	Passenger	PDHLVEHR2005	None	2105
		PDHLVEHR2005	None	2106
		PDHLVEHR1705	3	2107
		PDHLVEHR2205	3	2108
		PDHLVEHR2005	4	2109
		PDHLVEHR2005	4	2110
		PDHLVEHR2305	5	2111
		PDHLVEHR2305	5	2112
Y2	Light Truck	CCLF8AK1305	None	2145
		CCLF8AK1405	None	2146
		CCLF8AK1405	3	2147
		FBLFTDT2005	3	2148
		FBLFTDT2005	4	2149
		FBLFTDT2005	4	2150
		FBLFTDT2005	5	2151
		FBLFTDT2005	5	2152
N1	Light Truck	UP0RNA81105	None	2157
		UP0RNA81105	None	2158
		UP0RNA81105	3	2160
		UP0RNA81105	4	2161
		UP0RNA81105	4	2162
		UP0RNA81105	5	2163
		UP0RNA81105	5	2164
N2	Light Truck	YL6R2905	None	2170
		YL6R2905	None	2171

Tire Type	Vehicle Type	DOT TIN Number	Aging Time, Weeks at 65°C	Barcode
		YL6R2905	3	2172
		YL6R2905	3	2173
		YL6R2905	4	2174
		YL6R2905	4	2175
		YL6R2805	5	2176
		YL6R2805	5	2177
B7	Passenger	VN73WM00105	None	2242
		VN73WM00105	None	2243
		VN73WM00105	3	2245
		VN73WM00105	3	2246
		VN73WM00105	4	2247
		VN73WM00105	4	2248
		VN73WM03105	5	2251
		VN73WM03205	5	2256
B1	Passenger	EP6RDJF1105	None	2261
		EP6RDJF1205	None	2262
		EP6RDJF1205	3	2263
		EP6RDJF1205	4	2265
		EP6RDJF1105	4	2266
		EP6RDJF1205	5	2267
		EP6RDJF1105	5	2268
B8	Passenger	0BURB411606	None	2275
		0BURB411606	None	2276
		0BURB411606	3	2277
		0BURB411606	3	2278
		0BURB411606	4	2279
		0BURB411606	4	2280
		0BURB411606	5	2281
		0BURB411606	5	2282
O4	Light Truck	UTW8T3W3205	None	2357
		UTW8T3W3205	None	2358
		UTW8T3W3205	3	2359
		UTW8T3W3205	3	2360
		UTW8T3W3205	4	2361
		UTW8T3W3205	4	2362
		UTW8T3W3205	5	2363
		UTW8T3W3205	5	2364
D6	Passenger	PBURBTKR5004	None	2396
		PBURBTKR2705	None	2397
		PBURBTKR2505	3	2398
		PBURBTKR2005	3	2399
		PBURBTKR2505	4	2400

Tire Type	Vehicle Type	DOT TIN Number	Aging Time, Weeks at 65°C	Barcode
		PBURBTKR5004	4	2401
		PBURBTKR2505	5	2402
		PBURBTKR5004	5	2403
M1	Passenger	B74NFN3X0405	None	2461
		B74NFN3X0405	None	2462
		B74NFN3X0405	3	2463
		B74NFN3X0405	3	2464
		B74NFN3X0405	4	2465
		B74NFN3X0405	4	2466
		B74NFN3X0405	5	2467
		B74NFN3X0405	5	2468
M3	Passenger	EDPA2MNX2405	None	2474
		EDPA2MNX2405	None	2475
		EDPA2MNX2405	3	2476
		EDPA2MNX2405	3	2477
		EDPA2MNX2405	4	2478
		EDPA2MNX2405	4	2479
		EDPA2MNX2405	5	2480
		EDPA2MNX2405	5	2481
B9	Light Truck	7XW8P7M1806	None	2506
		7XW8P7M1806	None	2507
		7XW8P7M1806	3	2508
		7XW8P7M1806	3	2509
		7XW8P7M1806	4	2510
		7XW8P7M1806	4	2511
		7XW8P7M1806	5	2512
		7XW8P7M1806	5	2513
C7	Passenger	A3T645RW2206	None	2531
		A3T645RW2206	None	2532
		A3T645RW2206	3	2533
		A3T645RW2206	3	2534
		A3T645RW2206	4	2535
		A3T645RW2206	4	2536
		A3T645RW2206	5	2537
		A3T645RW2206	5	2538
C8	Passenger	P5UR4421806	None	2556
		P5UR4421806	None	2557
		P5UR4421806	3	2558
		P5UR4421806	3	2559
		P5UR4421806	4	2560
		P5UR4421806	4	2561
		P5UR4421806	5	2562

Tire Type	Vehicle Type	DOT TIN Number	Aging Time, Weeks at 65°C	Barcode
		P5UR4421806	5	2563
H3	Light Truck	T7XD5JNH4905	None	2581
		T7XD5JNH4905	None	2582
		T7XD5JNH4905	3	2583
		T7XD5JNH4905	3	2584
		T7XD5JNH4905	4	2585
		T7XD5JNH4905	4	2586
		T7XD5JNH4905	5	2587
		T7XD5JNH4905	5	2588
M10	Light Truck	B72K2EHX1106	None	2606
		B72K2EHX1106	None	2607
		B72K2EHX1106	3	2608
		B72K2EHX1106	3	2609
		B72K2EHX1106	4	2610
		B72K2EHX1106	4	2611
		B72K2EHX1106	5	2612
		B72K2EHX1106	5	2613
S1	Passenger	V4A64MCR5005	None	2631
		V4A64MCR5005	None	2632
		V4A64MCR5005	3	2633
		V4A64MCR5005	3	2634
		V4A64MCR5005	4	2636
		V4A64MCR5005	5	2637
		V4A64MCR5005	5	2638
T2	Passenger	9TKU93A0706	None	2656
		9TKU93A0706	None	2657
		9TKU93A0706	3	2658
		9TKU93A0706	3	2659
		9TKU93A0706	4	2660
		9TKU93A0706	4	2661
		9TKU93A0706	5	2662
		9TKU93A0706	5	2663

3.0 RESULTS

3.1 Physical Property Changes

In Phase 2, the physical property changes in the laboratory could be directly compared to the changes seen in the Phase 1 tires retrieved from service in Phoenix. In order to compare the property component changes of these tires to those in Phase 1 and 2, data was normalized by setting the value of the property of a new tire equal to 100 for each model. This allows trends to be compared at a glance to ascertain if laboratory oven aging was consistent with the type of aging seen in service and by laboratory-aged tires in Phase 2.

For instance, the peel adhesion strength for all tire models from Phoenix service declined with time in service as well as with laboratory aging. If the models tested in this work declined similarly in peel adhesion, it supports the hypothesis that the aging types are similar. Additionally, the tires from Phoenix service showed different rates and amounts of peel adhesion loss with aging for different models. This allows a comparison for rates of aging in these models to the models previously tested.

Extensive studies by Ford Motor Company compared the rate of tire aging in a circulating air oven to that of tires in service. [11] Ford calculated the acceleration factor for oven aging tires inflated with a mixture of 50% N_2/50% O_2 at 65°C was 36. That is, each week of oven aging under those conditions corresponds to approximately 36 weeks of field service. Based on this work, the physical properties studied after 3 weeks at 65°C would be approximately equivalent to 2.1 years of field service, and aging for 5 weeks at 65°C would be approximately equivalent to 3.5 years of field service.

3.1.1 Indentation Modulus

The indentation modulus of rubber components of selected tires was measured using the method shown in Appendix 1. The results, indexed to the new tire of the same model equal to 100, are shown in Table 8. For the tires removed from service in Phoenix, the average index for indentation modulus ranged from 84 to 145, with most tires showing little average change. Most tires tested in Phase 4 showed indexed modulus values from 95 to 157, which are very similar to the tires from Phoenix service. The exceptions are tire models C7 and C8 (the only Continental/General Tire models in the study) which showed relative modulus values of 162 to 204. Earlier work [5] has shown that the modulus tends to increase dramatically with oven aging unless the tires are first subjected to an extended break-in cycle. Further, Continental tires had the highest estimated resin content in the wedge area of the tires studied. Examples of the indentation modulus measurements are shown in Figure 1, with the shoulder area circled on the plots.

Table 8. Change of Indentation Modulus in Shoulder Area of Tire

Aging Condition	Tire Type	Aging Time, Years	Relative Indentation Modulus
Phoenix Service	B	1.37	110.6
		2.51	110.8
		6.04	145.3
	C	1.81	91.6
		5.45	98.0
	E	0.53	96.9
		3.27	116.2
	H	1.36	83.8
		1.99	90.9
		2.99	103.0
		5.96	91.2
	L	1.24	103.6
		2.65	92.6
Circulating Air Oven atat 65°C	B8	3	157
		5	132
	B9	3	98
		5	95
	C7	3	170
		5	162
	C8	3	186
		5	204
	H3	3	108
		5	102
	M10	3	144
		5	142
	P3	3	137
		5	131
	S1	3	143
		5	150
	T2	3	115
		5	115
	U2	3	NA
		5	153

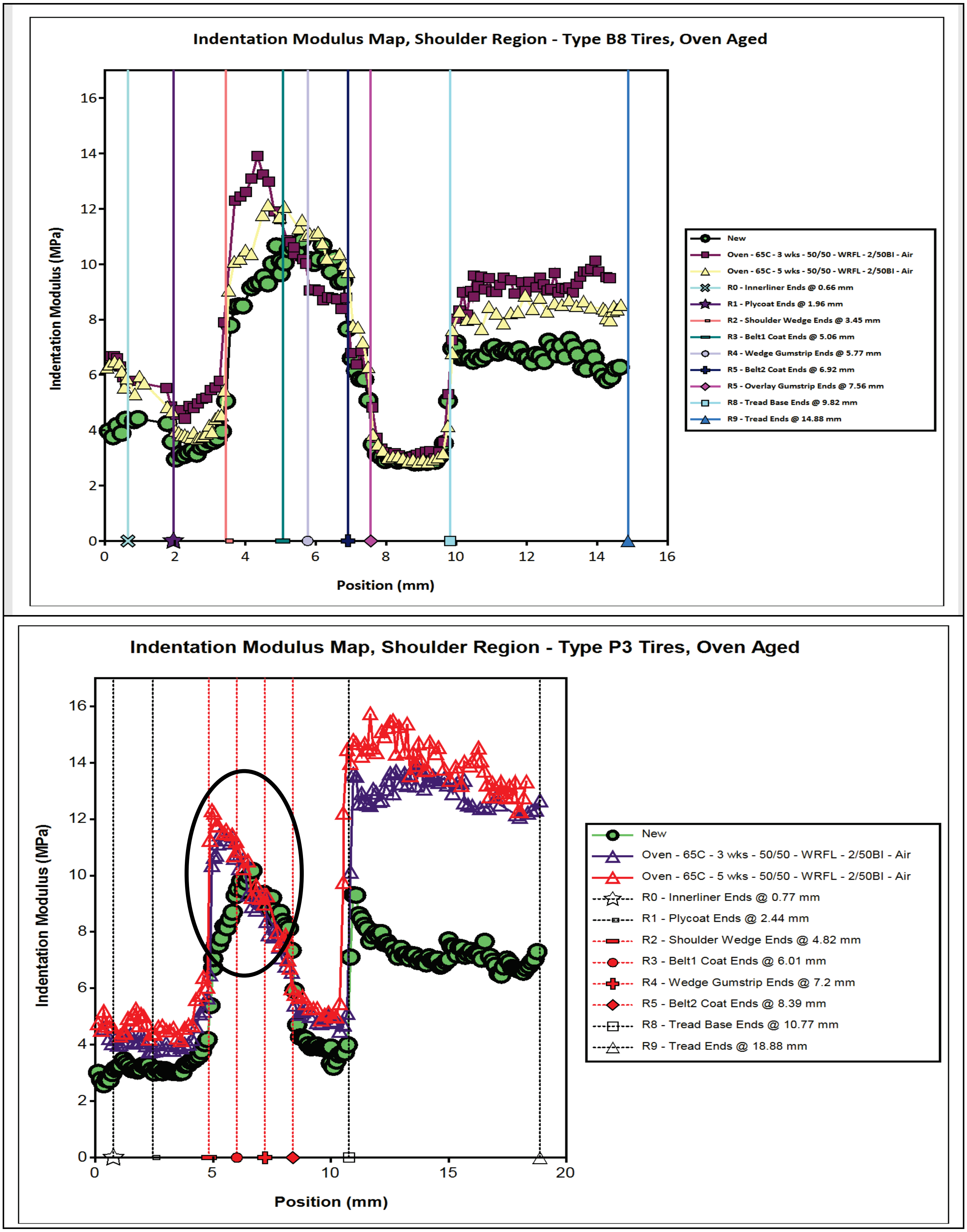

Figure 1. Indentation Modulus Profiles of New Tires and Tires Aged 3 & 5 Weeks at 65°C

3.1.2 Tensile Strength and Elongation of Skim-Coat Compound

Tensile strength and elongation properties of the rubber compound were measured according to the method in ASTM D 412. The modulus of the skim-coat compound for tires in the Phase 1 study tended to increase during service in Phoenix, with an average modulus value of 145 percent after 2.9 years of service. Samples at the longest service time (5 to 7 years) reached up to 200 percent of the modulus found in a new tire. The average modulus values of the skim-coat compounds are shown in Table 9. After 3 weeks at 65°C, the modulus values of the Phase 4 tires increased to an average of 160 percent of the corresponding new tires, with one model increasing to 200 percent of the original modulus values. After 5 weeks the modulus increased to an average of 190 percent of the corresponding new tires, with one model increasing to 300 percent of the original modulus values. The tensile strength values for the Phoenix tires retained an average of 70 percent of their original elongation and 90 percent of their original tensile strength values, while the oven-aged tires retained 45 percent to 60 percent of their elongation and 75 percent of their tensile strength to break values.

The rate of a chemical reaction is described by the Arrhenius equation[4] shown in Equation 1. **(12)** From this equation, the rate constant for an average service-year in Phoenix can be calculated. Assuming the tire is driven 4 percent of the time **(13)** and the average temperature during operation is 60°C, **(14)** and that the remaining time is an at rest temperature of 25°C, then the exponential term of the rate constant is 1.59×10^{-15}. For aging 3 weeks at 65°C, the exponential term of the rate constant is 3.85×10^{-15} or the equivalent of 2.4 years of average service in Phoenix. For aging 5 weeks at 65°C, the term is 6.42×10^{-15} or the equivalent of 4.0 years of average service in Phoenix. The relative increase for the modulus, reduction in tensile strength to break, and reduction in ultimate elongation for the oven-aged tires compared to the tires in Phoenix are consistent with this calculation. The average results for each aging condition are compared in Table 10. Consistent with the state of aging predicted from the Arrhenius equation, aging 5 weeks at 65°C resulted in significantly higher modulus and lower ultimate elongation values than did aging 3 weeks at 65°C. For Phoenix service tires the tensile strength showed the least average change over time in service. This is consistent with the measurement of the average tensile strength to break for the oven-aged tires that was not significantly different between 3 weeks and 5 weeks of aging.

Equation 1. Arrhenius Equation for Oxidation of Natural Rubber

$$\mathbf{k = Ae^{-(Ea/RT)}}$$

Where: k = Rate Constant for Reaction
A = Prefactor
Ea = Energy of Activation (for Natural Rubber ~96,400 joules / mole) (15)
R = Ideal Gas Constant (8.3145 joules / kelvins / mole)
T = Temperature, kelvins

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[4] The rate of oxidation is also proportional to the concentration of reactants. In addition, there are numerous processes taking place in the rubber compounds, such as reaction with antioxidants and diffusion of O_2. However, since most processes can be described by an exponential function similar to that proposed by Arrhenius, the relative rates of aging were compared using this term.

Table 9. Relative Values of Modulus, Elongation, and Tensile Strength for Skim-Coat Compound

Tire Type	Years in Service	Modulus at 50%	Modulus at 100%	Modulus at 200%	Elongation at Break	Ultimate Tensile Strength
B	0.44	91.9	106.7	110.1	100.1	107.8
	0.93	123.3	131.8	132.9	89.2	107.1
	1.36	133.9	143.0	139.9	85.7	105.8
	2.26	103.9	120.9	125.7	77.7	89.5
	2.51	166.3	174.9	162.4	64.3	88.9
	2.53	114.0	136.1	143.0	70.9	90.6
	4.66	179.5	198.6	185.1	60.4	95.0
	5.54	147.2	172.5	169.0	63.8	91.3
	6.04	153.2	177.7	171.5	58.0	85.4
	6.1	136.8	149.6	146.6	73.6	93.2
C	1.92	151.4	162.9	153.1	77.1	98.6
	2.05	159.8	160.4	160.9	69.1	101.4
	4.55	199.5	207.6	193.6	53.5	77.9
	4.56	150.2	172.7	173.3	76.2	104.6
	6.8	212.8	212.9	187.2	54.2	76.8
D	1.58	123.0	124.7	118.4	82.0	94.1
E	1.43	156.9	155.7	148.5	73.8	93.9
	2.83	189.4	181.1	160.9	62.3	85.1
	3.02	178.9	174.8	158.0	67.2	90.4
H	1.36	95.8	109.6	117.5	79.2	93.9
	1.5	123.3	132.1	134.4	72.8	94.1
	1.55	81.6	114.3	112.2	67.8	82.7
	1.99	93.8	110.1	117.2	68.4	79.0
	2.99	138.8	150.9	153.2	54.3	79.2
	4.7	166.2	182.9	.	25.4	45.6
L	1.51	131.2	128.3	119.8	70.2	81.9
	1.53	127.8	124.5	115.3	80.9	92.0
	1.93	101.1	97.7	94.8	96.3	89.9
	4.47	146.6	152.9	.	55.9	76.7
Type	**Weeks in Oven at 65°C**	**Modulus at 50%**	**Modulus at 100%**	**Modulus at 200%**	**Elongation at Break**	**Ultimate Tensile Strength**
B8	3	150	162	164	53	73
	3	158	162	159	48	63
	5	176	184	186	50	76
C7	3	147	152	152	52	69
	5	142	159	170	54	79
C8	3	148	163	170	57	82
	5	184	194	192	48	76
H3	3	175	193	187	43	64
	5	184	208	203	46	75
M10	3	124	131	130	72	97
	5	163	182	181	46	86

Type	Weeks in Oven at 65°C	Modulus at 50%	Modulus at 100%	Modulus at 200%	Elongation at Break	Ultimate Tensile Strength
P3	3	124	133	136	57	70
	5	166	190	188	38	62
S1	3	143	169	199	61	97
	5	238	313	337	25	55
T2	3	136	150	155	55	75
	5	185	211	NA	36	68
U2	3	174	208	210	57	87
	5	158	154	147	66	89

Table 10. Relative Measurement of Properties of Skim-Coat Compound After Aging

Property Measured	Aging Condition		Statistically Different at 95% Confidence
	3 Weeks at 65°C, % of Original Value	5 Weeks at 65°C, % of Original Value	
Modulus at 50% Elongation	148	177	Yes
Modulus at 100% Elongation	162	199	Yes
Modulus at 200% Elongation	166	200	Yes
Tensile Strength to Break	78	74	No
Ultimate Elongation	56	45	Yes

The slope of log(Ultimate Elongation at Break, %) versus log(Modulus at 100% Strain, MPa), often referred to as Ahagon slopes, was calculated for the Phase 4 tires. Slopes near -0.75 for these plots have been shown to correlate to oxidative (Type I) aging of rubber compounds. [8] Reductions in elongation with less change in modulus are associated with anaerobic (Type II) aging, which would be indicated by a less-steep negative slope or even a positive slope. Anaerobic aging associated with chain scission (Type III) is usually associated with temperatures in excess of 90ºC. The slopes of the Ahagon plots for the skim-coat compounds are shown in Table 11. All of the tire types from Phoenix service have slopes near -0.75, confirming that during service in Phoenix the skim-coat compound of tires tends to age oxidatively. The oven-aged samples also had slopes near -0.75 associated with oxidative aging.

Table 11. Ahagon Slopes for Skim-Coat Compound

Tire Type	Ahagon Slope, From Log(Elongation) Vs. Log(100% Modulus)	
	Phoenix Tires	Oven Aged at 65°C
B	- 0.76	
C	- 0.81	
D	- 0.91[5]	
E	-0.86[6]	
H	- 1.00	
L	- 0.44	
B8		-0.75
B9		-0.76
C7		-0.68
C8		-0.89
H3		-0.85
M10		-0.78
P3		-0.66
S1		-0.81
T2		-0.73
U2		-1.10

3.1.3 Tensile Strength and Elongation of Wedge Compound

The wedge compound is the compound between the wire belts in the shoulder area. The shoulder area has been shown to be where 95 percent of tire failures have been reported to NHTSA in the published defects reports. (16) This compound may be the same composition as the skim-coat compound or it may be a special formulation placed between the wire skim-coat compounds in the shoulder area during the building process.

The modulus of the wedge compound for tires in the Phase 1 study tended to increase during service in Phoenix, with an average modulus value of 140 percent after 2.9 years of service. Samples at the longest service time (5 to 7 years) reached up to 180 percent of the modulus found in a new tire. The average modulus values of the skim-coat compounds are shown in Table 12. After 5 weeks at 65ºC, the modulus values of the Phase 4 tires increased to an average of 185 percent of the corresponding new tires. As with the skim compound, the modulus of tire model S1 increased to over 300 percent of the original modulus values. The tensile strength values for the Phoenix tires retained an average of 70 percent of their original elongation and 85 percent of their original tensile strength values, while the oven-aged tires retained 40 percent of their elongation and 60 percent of their tensile strength values after aging 5 weeks at 65ºC. Like the skim-coat compound, this is consistent with the aging rates calculated from the Arrhenius equation.

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[5] Based on a single tire in Phoenix service

[6] Modulus decreases during service in Phoenix, therefore slope does not include new tire data.

Table 12. Relative Values for Modulus, Tensile Strength, and Elongation for Wedge Compound

Tire Type	Years in Service	Modulus at 50%	Modulus at 100%	Modulus at 200%	Elongation at Break	Ultimate Tensile Strength
B	0.44	93.7	104.9	109.5	97.9	107.4
	0.93	117.9	130.9	131.2	83.9	100.3
	1.36	128.6	138.6	139.5	83.7	105.6
	2.26	105.4	122.8	128.5	75.9	90.1
	2.51	156.0	168.3	162.3	65.7	92.1
	2.53	117.1	135.4	140.3	71.8	90.6
	4.66	170.2	187.2	175.3	60.7	90.1
	5.54	145.3	169.4	168.5	65.6	95.2
	6.04	163.0	186.4	182.1	55.4	85.3
	6.1	159.1	172.1	168.3	70.8	101.9
C	1.92	132.2	137.0	131.7	74.1	86.4
	2.05	166.3	166.5	158.6	52.9	78.3
	4.55	164.0	163.9	139.2	43.6	55.6
	4.56	140.4	149.2	142.6	66.1	81.8
	6.8	154.4	155.2	146.3	60.2	73.6
D	1.58	108.4	123.5	118.5	76.6	90.0
E	1.43	150.5	148.0	139.0	58.5	72.9
	2.83	143.8	143.7	134.3	64.9	78.7
	3.02	167.3	167.5	150.7	54.5	73.4
H	1.36	157.8	159.4	143.5	59.5	76.1
	1.5	93.4	106.9	115.1	84.7	98.2
	1.55	118.7	131.2	136.7	68.5	90.4
	1.99	87.0	106.9	117.1	79.0	92.3
	2.99	96.1	109.3	118.0	73.7	86.2
	4.7	130.4	146.0	150.8	58.2	85.0
L	1.51	147.7	141.1	120.7	62.6	77.2
	1.53	150.3	146.7	133.4	62.6	80.3
	1.93	131.5	128.1	119.6	74.7	87.6
	4.47	177.7	175.1	.	40.9	61.0
B8	3	148	154	157	57	76
B9	3	141	148	149	61	78
	5	155	165	169	60	87
C7	3	124	134	135	61	76
	5	139	157	162	55	81
C8	3	138	149	155	63	87
	5	161	178	182	49	77
H3	3	138	152	155	51	67
	5	168	190	193	47	75
M10	3	113	118	121	69	84
	5	148	166	175	53	88
P3	3	122	131	133	54	66
	5	156	179	183	36	58

Tire Type	Years in Service	Modulus at 50%	Modulus at 100%	Modulus at 200%	Elongation at Break	Ultimate Tensile Strength
S1	3	155	187	207	57	90
	5	275	344	.	28	63
T2	3	141	154	157	49	67
	5	182	209	.	36	63
U2	3	143	155	159	70	94
	5	206	235	244	46	86

Table 13. Relative Measurement of Properties of Wedge Compound After Aging

Property Measured	Aging Condition		Statistically Different at 95% Confidence
	3 Weeks at 65°C, % of Original Value	5 Weeks at 65°C, % of Original Value	
Modulus at 50% Elongation	136	177	Yes
Modulus at 100% Elongation	148	202	Yes
Modulus at 200% Elongation	153	187	Yes
Tensile Strength to Break	79	75	No
Ultimate Elongation	59	46	Yes

The slopes of the Ahagon plots for the wedge compounds are shown in Table 14. All of the tire types from Phoenix service have slopes near -0.75, indicating that during service in Phoenix the wedge compound of tires tends to age oxidatively. The wedge compounds for the Phase 4 oven-aged tires also had slopes near -0.75, evidence that the aging was predominantly thermo-oxidative.

Table 14. Ahagon Slopes for Wedge Compound

Tire Type	Ahagon Slope, From Log(Elongation) Vs. Log(100% Modulus)	
	Phoenix Tires	Oven Aged 8 Weeks at 65°C
B	- 0.77	
C	- 1.39	
D	- 0.80[7]	
E	- 1.15	
H	- 1.17	
L	- 1.56	
B8		-0.77
B9		-0.90
C7		-0.70
C8		-0.81
H3		-0.76

[7] Based on a single tire in service in Phoenix

Tire Type	Ahagon Slope, From Log(Elongation) Vs. Log(100% Modulus)	
	Phoenix Tires	Oven Aged 8 Weeks at 65°C
M10		-0.77
P3		-0.56
S1		-0.95
T2		-0.70
U2		-1.10

3.1.4 Peel Adhesion

Peel adhesion was considered as a test to determine the residual durability of tires after aging or time in service, based on the observed reduction in peel adhesion force for the tires recalled during the Ford/Firestone investigation. The peel adhesion behavior of the 6 tire types recovered from service in Phoenix has been reported. [3] While peel adhesion decreased with time in service, researchers concluded that peel adhesion could not be used as a predictive measure because: i) most samples did not fail at the wire/rubber interface, but failed cohesively in the rubber compound; and ii) due to the physics of the peel sample, the actual force measured is a function of rubber properties such as thickness and modulus. For the tires removed from service in Phoenix after an average of 2.99 years, both the skim-coat and the wedge areas retained an average peel adhesion of 53 percent of the original tires. At the maximum service time of 5 to 7 years, the retained adhesion ranged from approximately 25 percent to 60 percent of the original adhesion. The oven-aged tires retained approximately 50 percent of the adhesion for both the skim-coat and wedge areas, with a range of approximately 30 percent to 80 percent.

Table 15. Relative Peel Adhesion Values for the Skim-Coat and Wedge Compounds

Tire Type	Years in Service	Peel Adhesion of Skim-Coat, % of New Tire	Peel Adhesion of Wedge, % of New Tire
B	0.44	87.8	93.2
	0.93	60.9	75.4
	1.36	56.5	64.4
	2.26	56.7	57.8
	2.51	43.5	46.7
	2.53	58.6	62.4
	5.54	48.9	54.8
	6.04	43.3	53.7
	6.1	52.6	72.4
C	1.92	55.1	41.5
	2.05	72.7	61.6
	4.55	44.3	37.8
	4.56	58.9	52.2
	6.8	55.7	47.2
D	1.58	56.0	71.4
E	1.43	64.4	68.4
	2.83	42.7	38.9
	3.02	49.1	48.6
H	1.36	78.1	62.3
	1.5	46.8	53.5
	1.55	42.3	45.0
	1.99	69.0	67.3
	2.99	44.3	47.1
	4.7	24.5	16.8

Tire Type	Years in Service	Peel Adhesion of Skim-Coat, % of New Tire	Peel Adhesion of Wedge, % of New Tire
L	1.51	41.0	31.9
	1.53	35.1	26.8
	1.93	68.2	67.4
	4.47	35.4	31.6
Type	**Weeks in Oven**	**Peel Adhesion of Skim-Coat, % of New Tire**	**Peel Adhesion of Wedge, % of New Tire**
B8	3	31	36
	5	38	45
B9	3	64	59
	5	57	52
C7	3	46	51
	5	58	67
C8	3	65	55
	5	54	47
H3	3	37	28
	5	45	40
M10	3	54	54
	5	43	46
P3	3	49	61
	5	44	64
S1	3	77	65
	5	38	39
T2	3	50	51
	5	37	62
U2	3	81	54
	5	48	48

Table 16. Relative Measurement of Peel Adhesion After Aging

Property Measured	Aging Condition		Statistically Different at 95% Confidence
	3 Weeks at 65°C, % of Original Value	5 Weeks at 65°C, % of Original Value	
Peel Adhesion of Skim-Coat, % of New Tire	54.9	46.3	Yes
Peel Adhesion of Wedge, % of New Tire	51.4	51.0	No

3.1.5 <u>Fixed Oxygen Level</u>

Fixed oxygen is the amount of oxygen that is chemically combined with the rubber compound. As noted earlier, changes in physical properties may result from thermal reactions (anaerobic aging) or from reaction with oxygen (aerobic aging). NHTSA examined the level of fixed oxygen in the skim, wedge, and innermost tread of 6 tire models collected from on-vehicle use in Phoenix after varying amounts of service. These results confirmed the aerobic chemical reactions in the shoulder region of light vehicle tires during service. The normalized fixed oxygen level for varying service or aging time is shown in Table 17. As noted, the level of fixed oxygen increases in all components for tires in service. Most tire types showed a small increase in fixed oxygen level to an average of approximately 110 percent of the original value. Tire types C and H showed more dramatic increases, to approximately 120 percent and 150 percent respectively, after 5 to 7 years of aging. The fixed oxygen content for 6 tire models of the Phase 4 tires was measured and the averages were approximately 100 percent to 165 percent of the levels of the original tires of the same model. This suggests that there was sufficient oxygen available from the 50%N_2/50%O_2 inflation gas to provide oxygen to the internal components of the tire during the oven aging time. This was also supported by the modeling of diffusion-limited oxidation in a number of tire models and has been reported separately. [(4)] The level of oxidation experienced in these components of the Phase 4 tires appears to be similar to that experienced by tires after 5 to 7 years in Phoenix service.

Table 17. Normalized Fixed Oxygen Level over Time

Tire Type	Time in Service, Years	Shoulder Area	Wedge Compound	Tread Compound
B	0.44	98.3	96.6	99.3
	0.93	101.2	99.3	113.0
	1.36	104.5	100.3	112.9
	2.26	103.5	99.9	101.0
	2.51	102.7	106.8	100.7
	2.53	110.8	109.9	106.0
	4.66	108.4	105.4	100.0
	5.54	113.6	111.5	113.7
	6.04	113.6	110.6	135.9
	6.1	109.8	110.4	128.7
C	1.92	106.1	106.3	106.5
	2.05	102.1	98.9	106.7
	4.55	109.3	123.5	114.9
	4.56	117.4	122.9	116.9
	6.8	112.3	127.9	122.7
D	1.41	104.1	99.2	102.9
	1.58	105.3	102.3	112.5
	3.87	115.2	115.5	124.5

Tire Type	Time in Service, Years	Shoulder Area	Wedge Compound	Tread Compound
E	1.43	108.6	109.2	100.1
	2.83	105.6	107.8	95.0
	2.91	109.2	104.8	108.7
	3.02	104.0	105.6	94.5
H	1.36	117.0	101.0	107.2
	1.5	93.8	91.5	122.2
	1.55	100.8	96.4	119.9
	1.99	104.8	97.6	118.8
	2.99	100.9	97.4	122.3
	4.7	152.1	148.8	125.2
	5.96	144.6	139.4	138.0
L	1.06	105.8	107.9	114.9
	1.24	102.7	98.5	101.9
	1.51	106.9	92.1	120.5
	1.53	98.9	97.3	128.0
	1.93	109.3	104.5	114.0
	2.65	109.6	105.5	102.2
	4.47	106.7	104.3	119.4
Tire TypeB8	3	113	121	110
	5	113	102	99
B9	3	133	136	109
	5	130	137	128
C7	3	120	110	115
	5	138	125	157
C8	3	122	113	85
	5	116	114	100
H3	3	141	134	96
	5	157	152	120
M10	3	109	107	112
	5	130	123	167
P3	3	95	100	93
	5	110	108	100
S1	3	122	129	120
	5	143	144	94
T2	3	127	134	102
	5	131	135	97
U2	3	140	110	81
	5	151	117	101

3.2 Roadwheel Testing

3.2.1 Testing Using Stepped-Up Load to Failure Test

Ten tire models were oven aged for 5 weeks at 65°C while inflated with the $50\%N_2/50\%O_2$ mixture then subjected to testing on a 1.707 m roadwheel using the stepped-up load to failure (SUL) protocol used in Phases 2 and 3 of the aging work. The SUL test results for the aged tires were compared to new tires of the same model. The test conditions are shown in Table 3. The results and discussion of the Phoenix service tires have been reported separately. [13] Using the Arrhenius formula in Equation 1 and service conditions discussed earlier, aging 5 weeks at 65°C is approximately equivalent to the aging experienced by a tire with 4.0 years of service in Phoenix. The results for the SUL testing are shown in Table 18. Failures in less than 34 hours happened while the load on the tire was at or below its maximum stated load capacity and are emphasized in bold type.

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Table 18. Results for Stepped-Up-Load to Failure of Tires

Tire Type	Vehicle Fitment	Barcode	Oven Aging, Weeks at 65°C	Failure Time, Hours	Failure Code
B8	Passenger	2283	None	64.6	Tread Shoulder Lug + Belt Edge / Bead Turn Up Cracking
		2284	None	53.33	Bead Turn Up Cracking
		2287	**5**	**18.23**	**Sidewall Cracking**
		2288	5	54.65	Tread Shoulder Chunking / Belt Edge Separation
B9	Light Truck	2514	None	50.38	Sidewall Rupture
		2515	None	56.62	Innerliner Split / Bead Turn Up Rupture
		2524	5	56.62	Sidewall Rupture+Delamination
C7	Passenger	2539	None	61.25	Belt Edge Loosened
		2543	5	60.92	Bead Turn Up Rupture
		2544	5	58.87	Tread Shoulder Pocket Cracking
C8	Passenger	2564	None	74.62	Sidewall Rupture
		2565	None	72.22	Bead Turn Up Cracking
		2568	5	69.4	Tread Shoulder Cracking+Belt Edge
		2569	5	57.63	Bead Turn Up Cracking
H3	Light Truck	2599	None	60.82	Sidewall Rupture
		2593	**5**	**25.25**	**Plycoat Delamination – Interface**
		2594	**5**	**10.88**	**Plycoat Delamination – Interface**
M10	Light Truck	2624	None	40.75	Complete Tread and Belt 2 Detachment
		2618	**5**	**16.92**	**Complete Tread and Belt 2 Detachment, Carcass Rupture**
P3	Passenger	2053	None	48.33	Tread Lug Chunking+Belt EdgeBead Turn Up Cracking
		2054	None	51.42	Tread Lug Chunking+Belt Edge
		2057	**5**	**27.5**	**Complete Tread and Belt 2 Detachment**

Tire Type	Vehicle Fitment	Barcode	Oven Aging, Weeks at 65°C	Failure Time, Hours	Failure Code
		2058	**5**	**13.78**	**Belt Edge Separation**
S1	Passenger	2639	None	61.62	Sidewall Rupture
		2640	None	55.5	Tread Lug Chunking
		2643	**5**	**13.53**	**Tread Shoulder Pocket Cracking**
		2644	**5**	**23.75**	**Tread Shoulder Cracking+Belt Edge**
T2	Passenger	2664	None	67.1	Bead Turn Up Rupture
		2665	None	66.83	Bead Turn Up Cracking
		2668	5	46.78	Belt Edge Separation
		2669	5	51.33	Belt Edge Separation
U2	Passenger	2078	None	63.55	Innerliner Delamination Shoulder Rupture
		2079	None	63.27	Shoulder Rupture
		2082	5	59.52	Innerliner Delamination
		2083	5	61.34	Innerliner Delamination

The results of the stepped-up-load test failures for the tires tested by the SUL procedure (shown graphically in Figure 2a to Figure 2j) are compared to the predicted values from linear regression of the tires of varying ages taken from Phoenix service. The mean predicted time to failure for the Phoenix tires is indicated by the solid black line. The average failure times of the tire models showing the least change in time to failure, and those showing the greatest reduction in time to failure are indicated by the dashed light blue lines. The green line at 34 hours represents the maximum time tested at 100 percent or less of the tire's rated load. Testing at loads above this level causes stress on parts of the tire, such as the sidewall and bead, beyond what is expected during normal operation, often causing failures in these components.

At failure times of less than 34 hours, the load on the tire was equal to or less than the rated load of the tire, therefore the stresses on individual components could have been experienced in normal service conditions. The 5 of 14 aged passenger tires (36%) exhibiting visible failures in less than 34 hours of roadwheel testing are summarized in Figure 3. Approximately two-thirds of these failures occurred in the belt area, with another 22 percent of failures taking place in the shoulder area. As discussed previously, over 90 percent of failures that consumers reported to NHTSA happened in the belt and shoulder area. Failures by tread cracking accounted for 6 percent of the visible failures in the oven-aged tires, with a small number of carcass and sidewall failures.

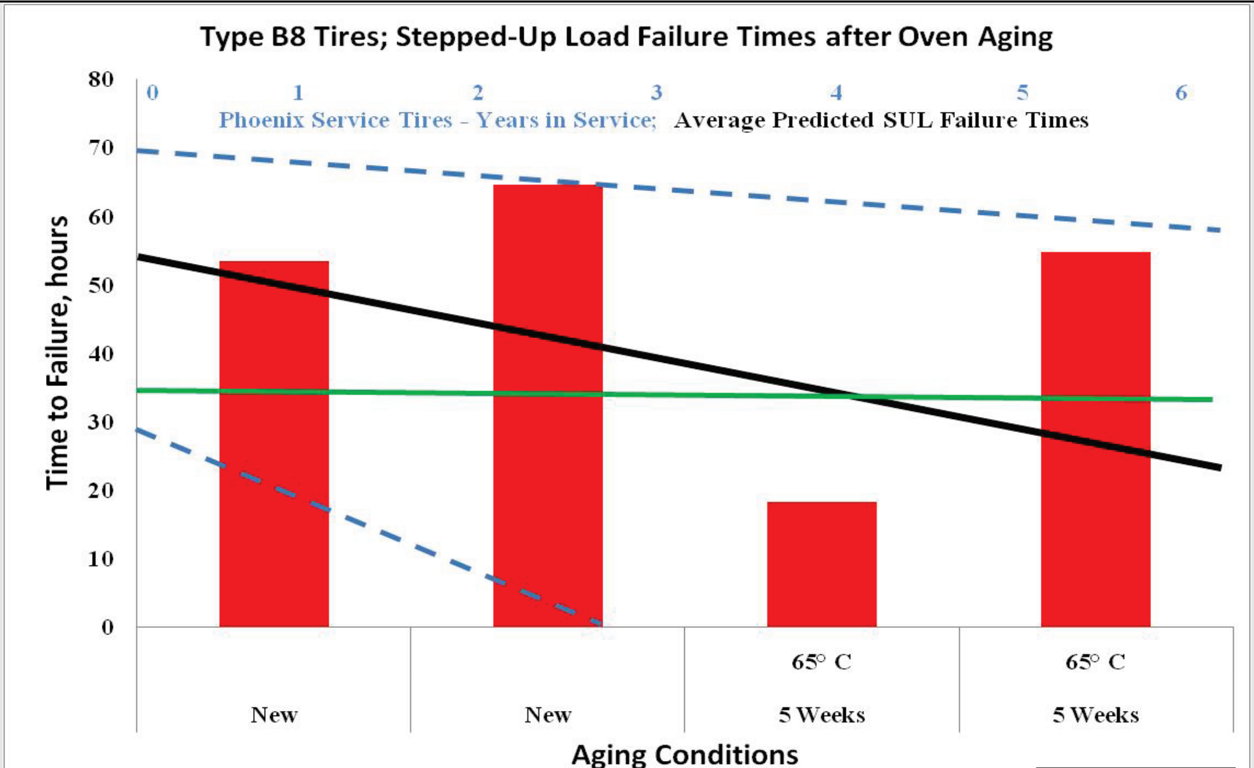


Figure 2a

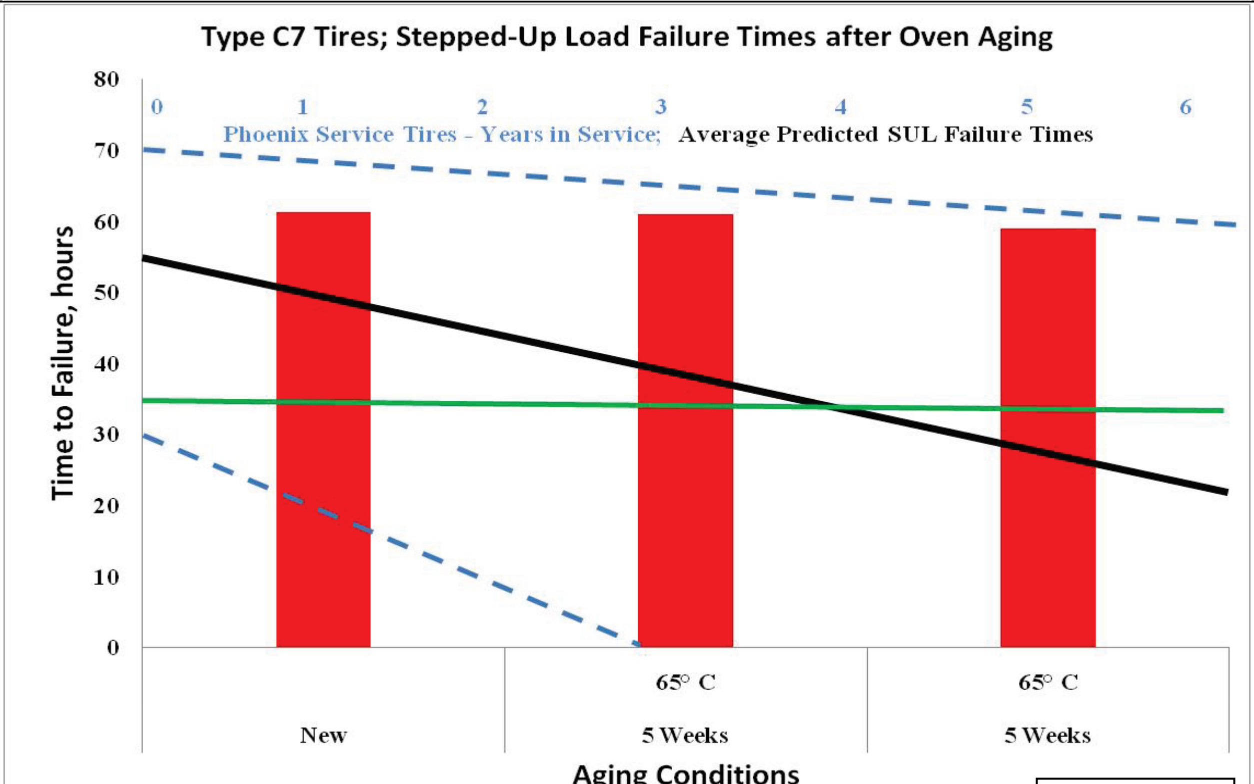


Figure 2b

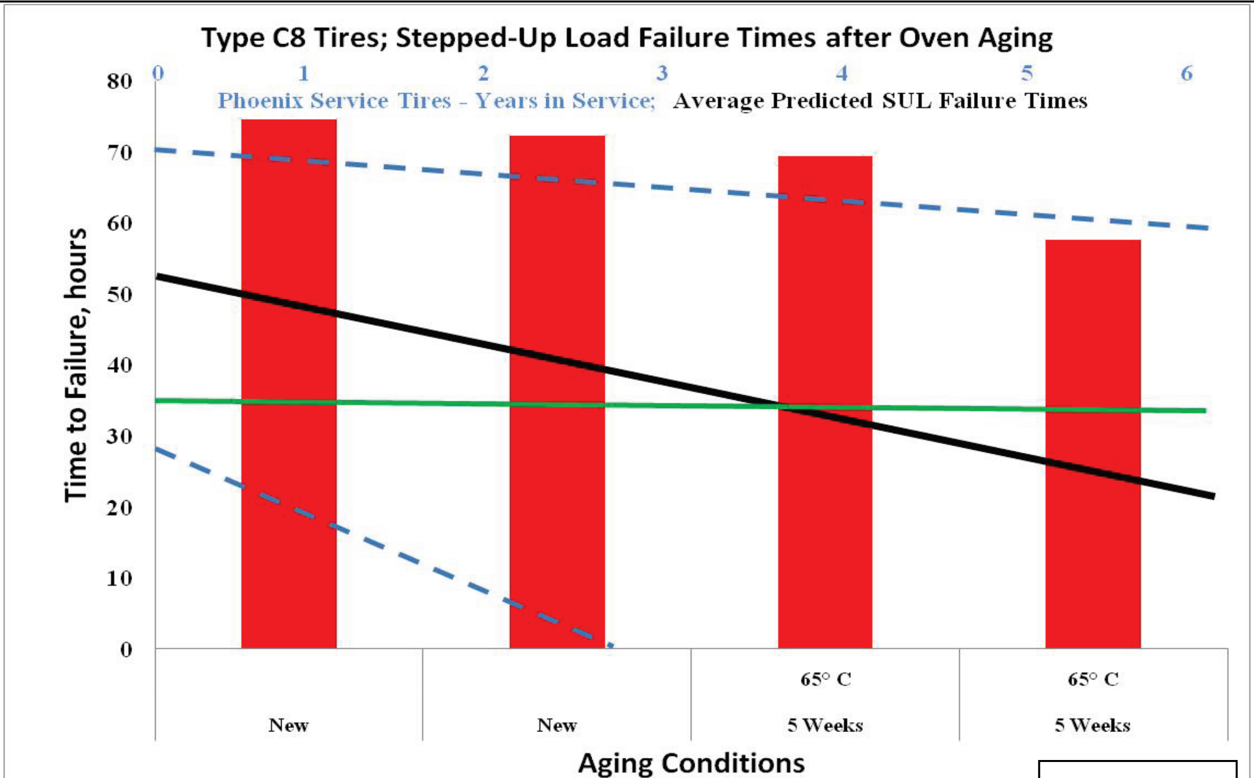


Figure 2c

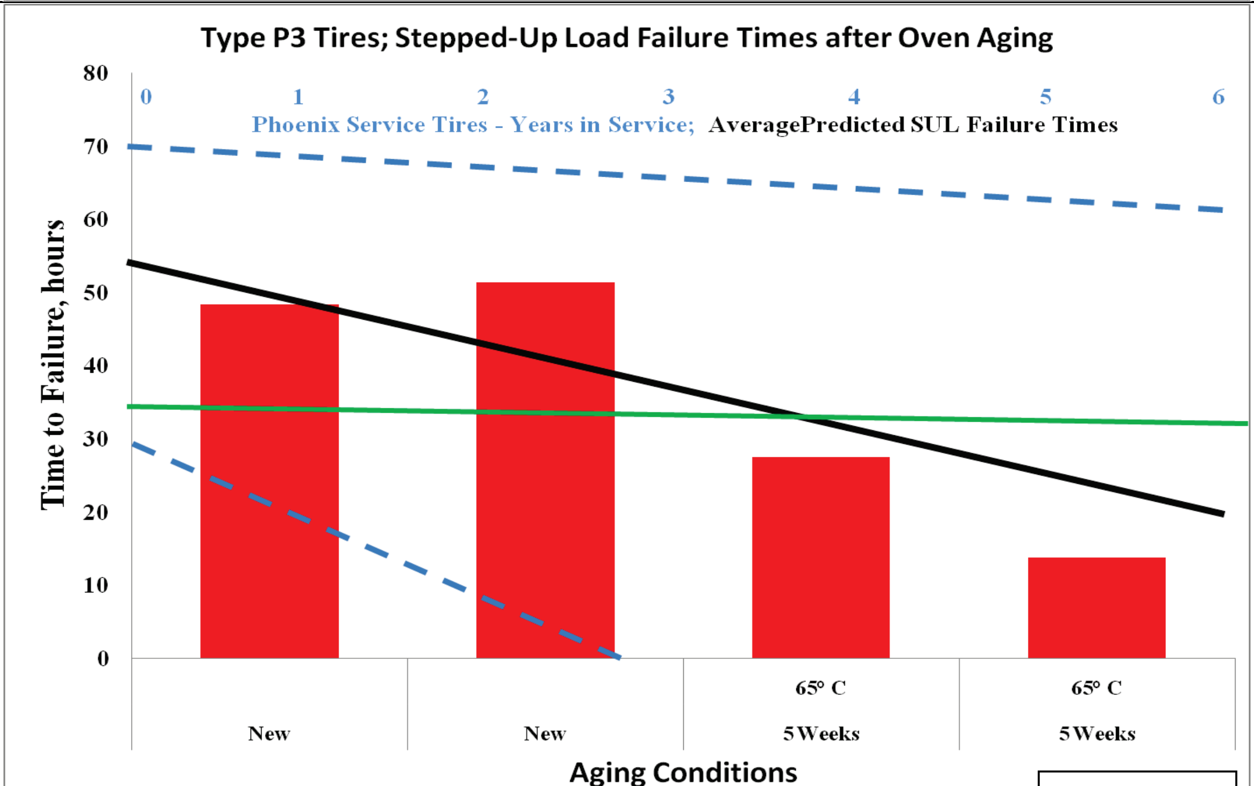


Figure 2d

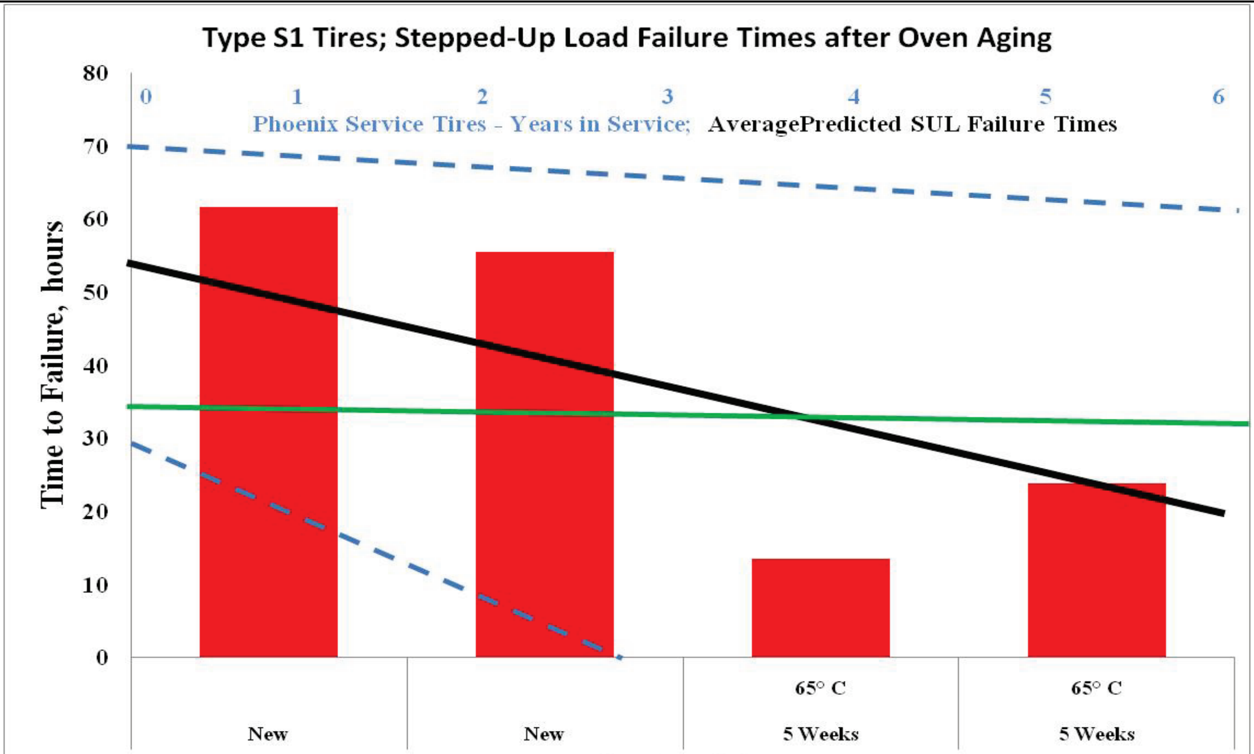


Figure 2e

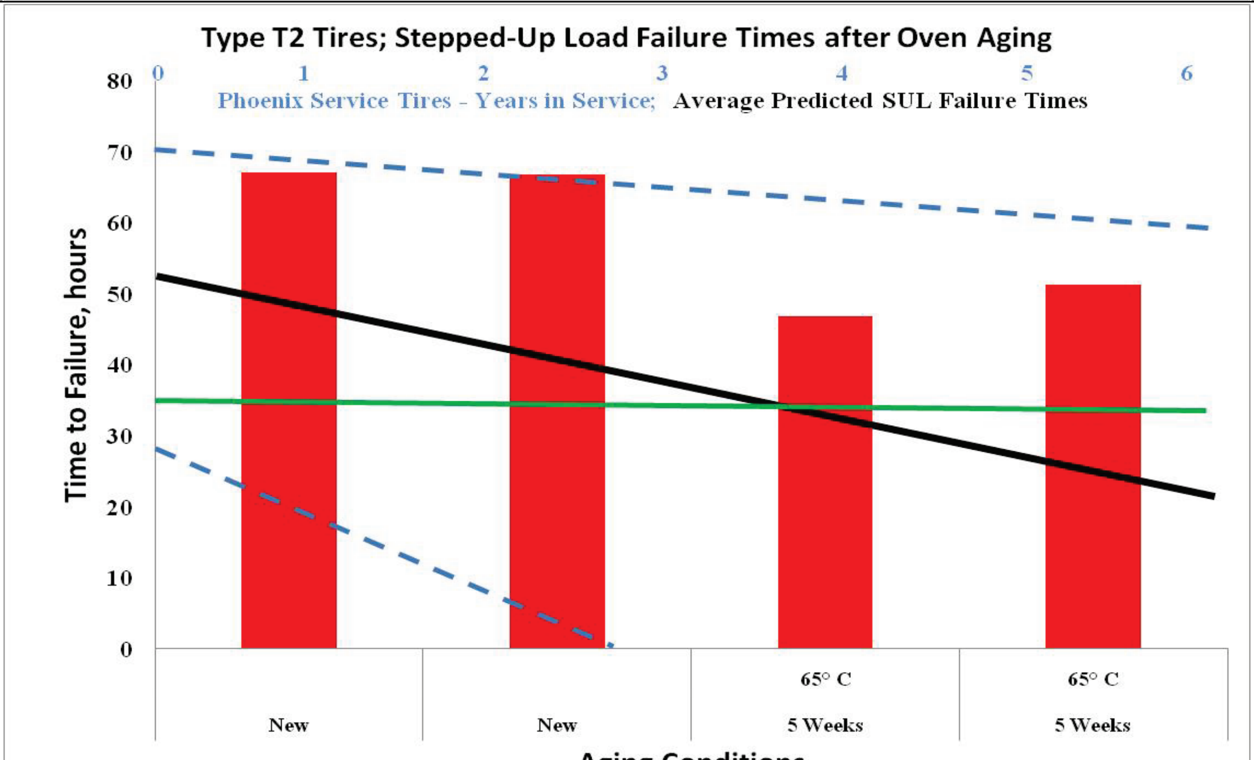


Figure 2f

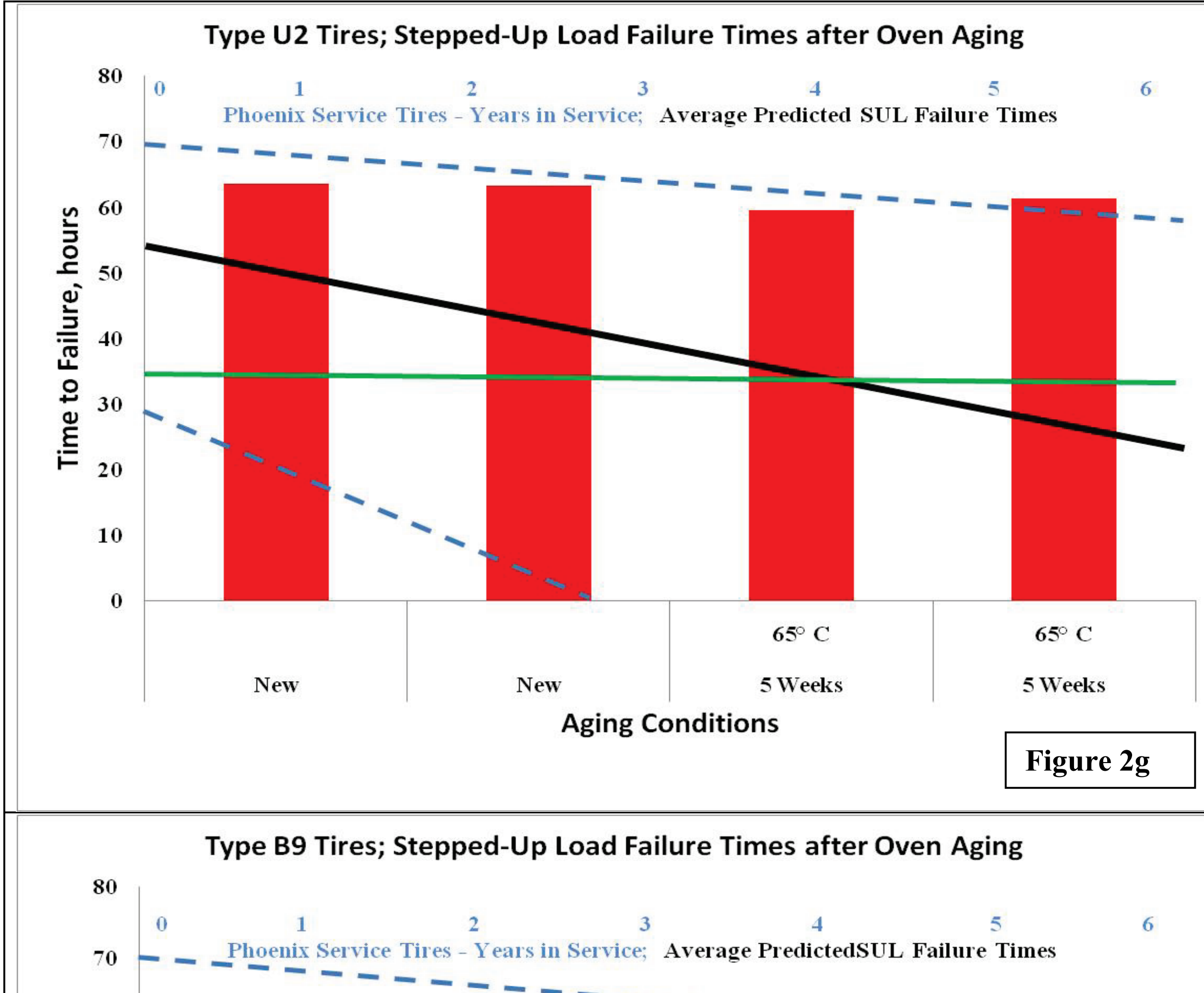


Figure 2g

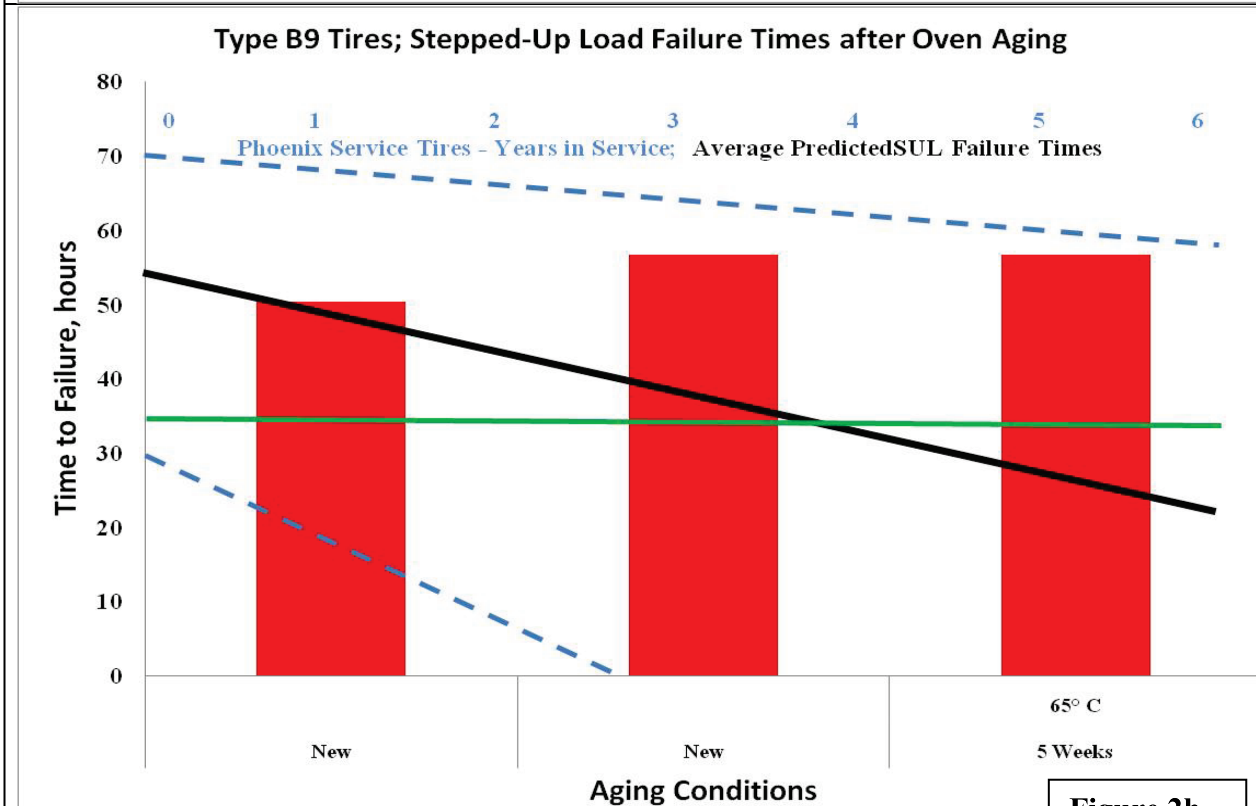


Figure 2h

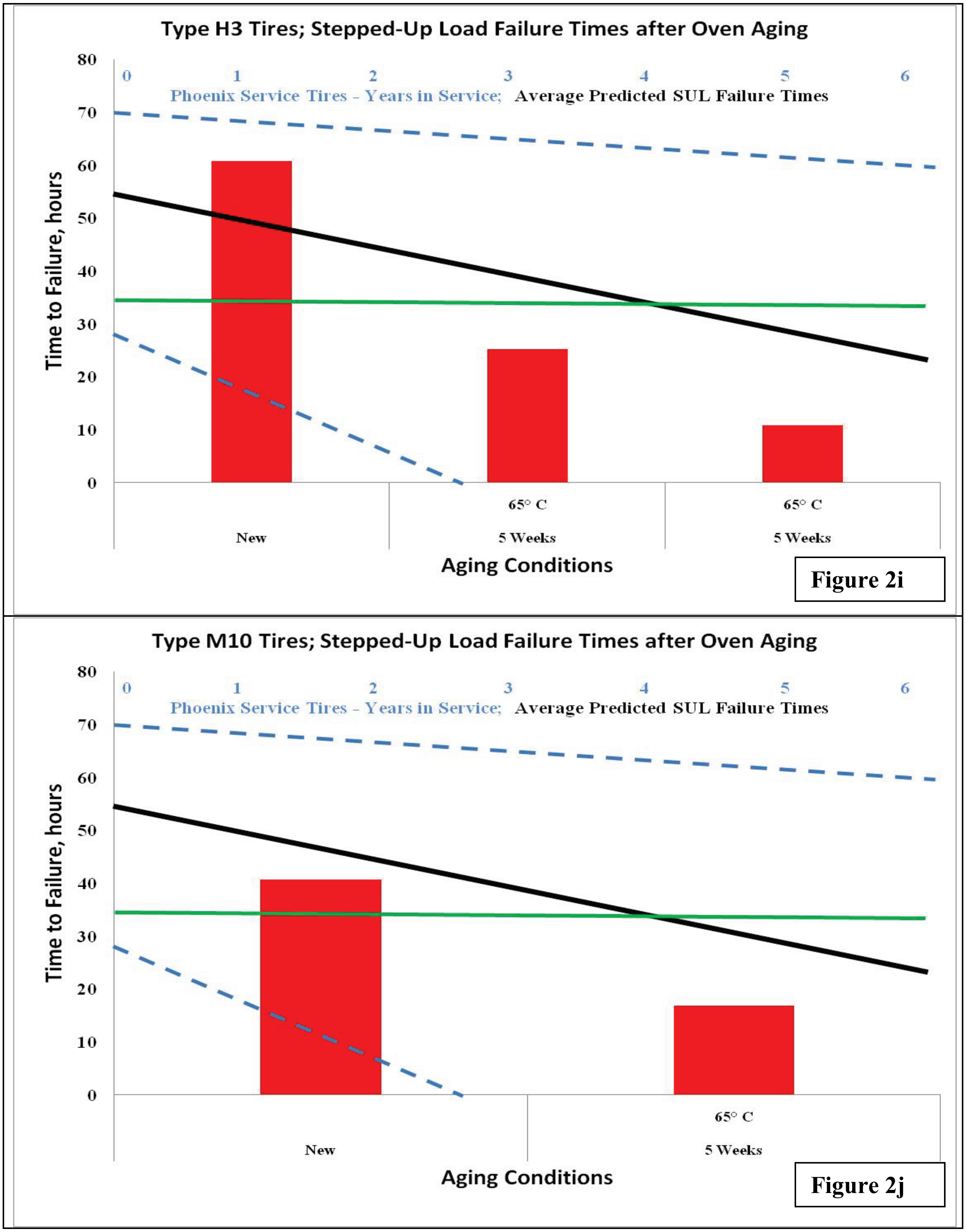


Figure 2. Tire Failure Times for Stepped-Up-Load to Failure (SUL) Test

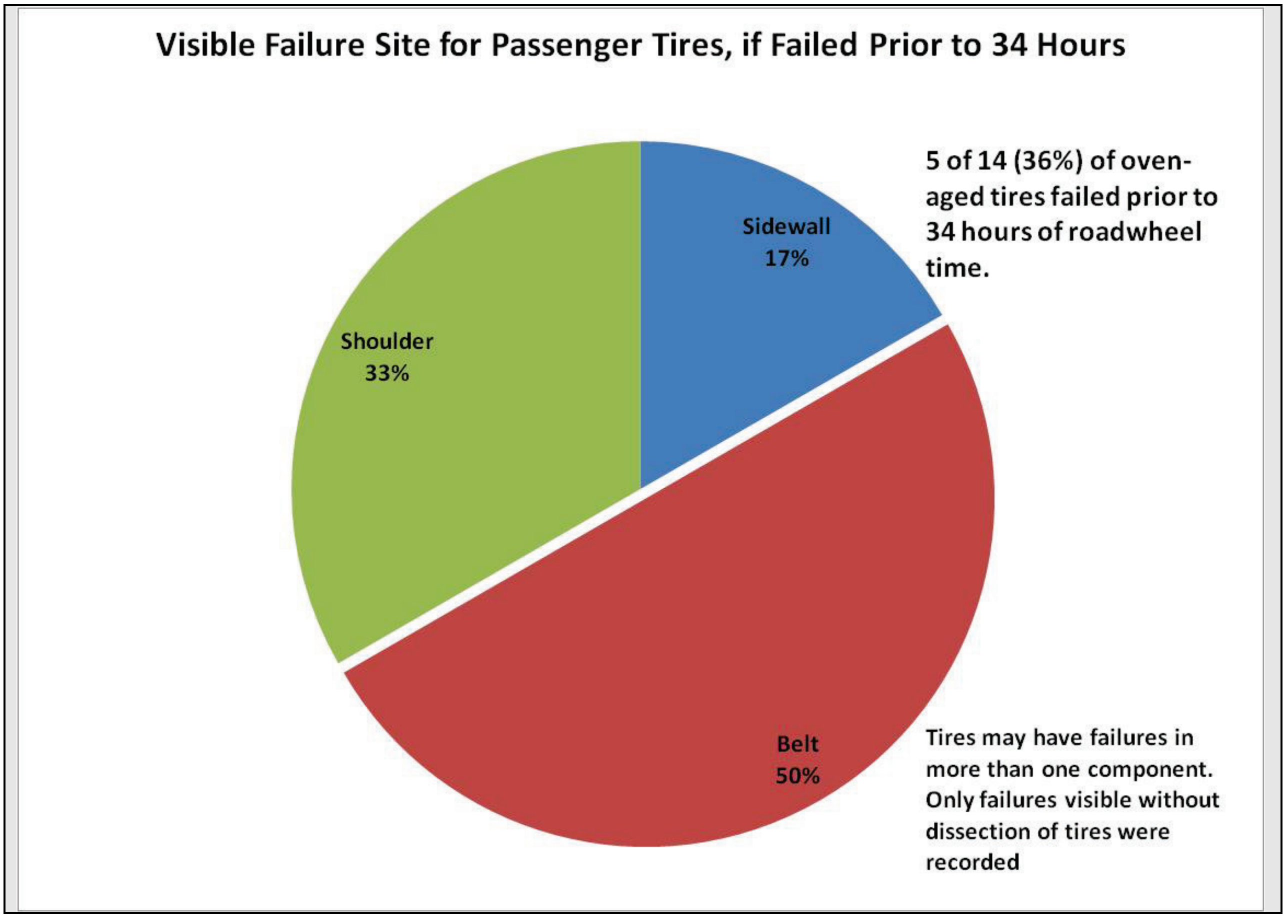


Figure 3. Failure for Passenger Tires, if Failure Time Was Less Than 34 Hours

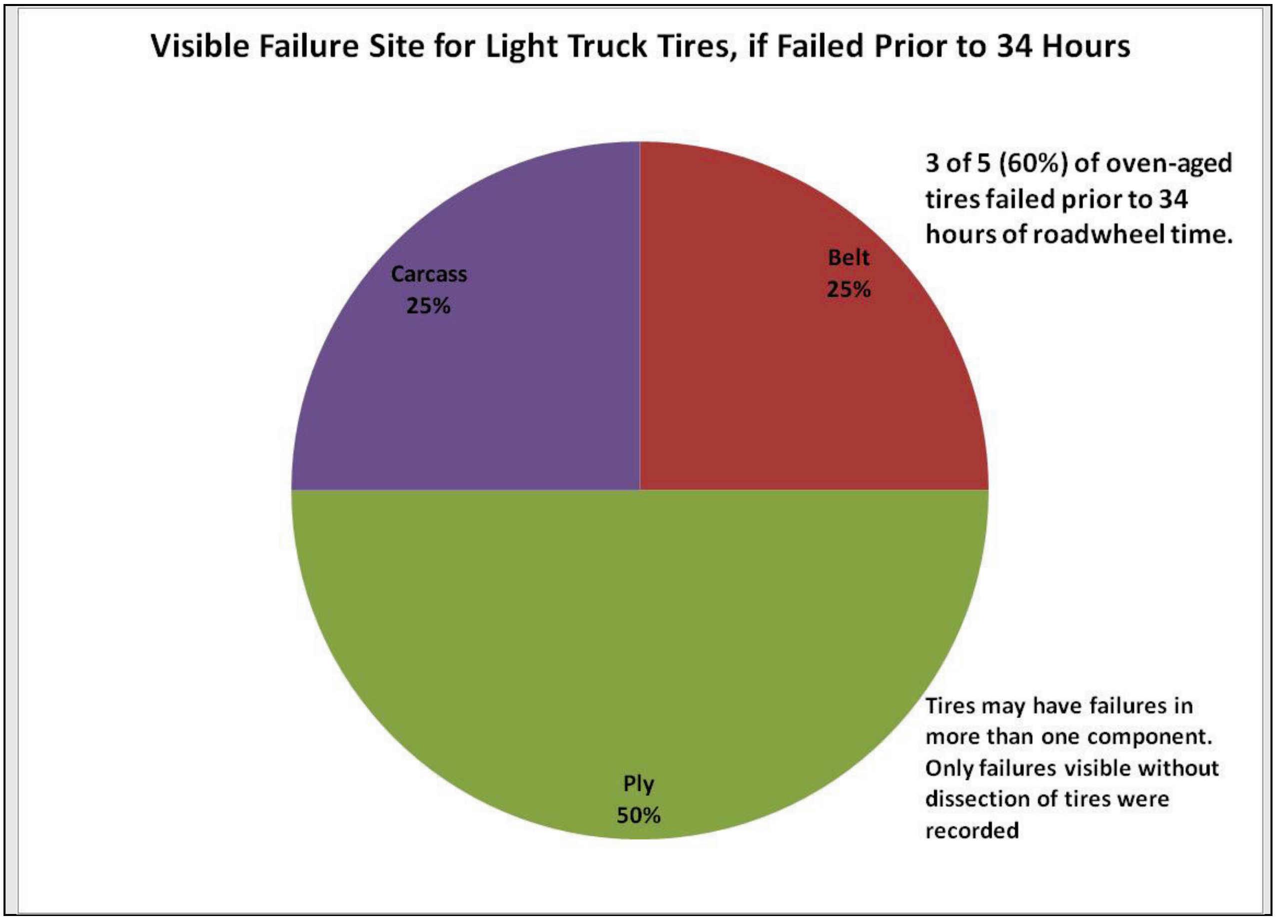


Figure 4. Site of Failure for Light Truck Tires, if Failure Time Was Less Than 34 Hours

3.2.1 Testing to Conditions of the FMVSS 139 Standard

Tires were oven aged for 3, 4, or 5 weeks at 65°C while inflated with the $50\%N_2/50\%O_2$ mixture and then tested according to the FMVSS 139 endurance and low pressure test. The first 34 hours of the FMVSS 139 Endurance Test, [17] are identical to the first 34 hours of the stepped-up-load to structural failure (SUL) test used in Phase 2, Phase 3, and for selected tires from Phase 4. This is followed by the FMVSS 139 Low Pressure test. The test conditions are shown in Table 3. The results and discussion of the Phoenix service tires have been reported separately. [13] Using the Arrhenius formula in Equation 1 and service conditions discussed earlier, aging 3 weeks at 65°C is approximately equivalent to 2.4 years of service, and aging 5 weeks at 65°C is equivalent to the aging experienced by a tire with 4.0 years of service in Phoenix. The FMVSS 139 test was designed to ensure that tires had sufficient structural integrity to perform under the stringent conditions required for service on passenger, SUV, and light-truck vehicles. The low pressure test was added after studies indicated that more than 50 percent of passenger vehicles in service had at least 1 tire that was underinflated by 25 percent or more [18]. It should be noted that these tires were produced in 2005–2006, prior to the full adoption of the FMVSS 139 standard in September, 2007, so they were not required to meet the requirements of FMVSS 139. The results are shown in Table 19.

Table 19. Data Tires Tested to FMVSS 139 Endurance and Low-Pressure Protocol

Tire Type	Aging Time, weeks	Barcode	FMVSS 139 Endurance, hrs	FMVSS 139 Low Pressure, hrs	Failure Code
P3	New	2045	Completed	Completed	No Visible Failure
	New	2046	Completed	Completed	No Visible Failure
	3	2047	Completed	Completed	No Visible Failure
	3	2048	Completed	Completed	No Visible Failure
	4	2049	34 (Completed)	0	Belt Edge Exposed
	4	2050	32.83	NA	Complete Tread and Belt 2 Detachment
	5	2051	16.38	NA	Sidewall Cracking
	5	2052	19	NA	Complete Tread and Belt 2 Detachment
U2	New	2070	Completed	Completed	No Visible Failure
	New	2071	Completed	Completed	No Visible Failure
	3	2072	Completed	Completed	No Visible Failure
	3	2073	Completed	Completed	No Visible Failure
	4	2074	Completed	Completed	No Visible Failure
	4	2075	Completed	Completed	No Visible Failure
	5	2076	Completed	Completed	No Visible Failure
	5	2077	Completed	Completed	No Visible Failure
G1	New	2105	Completed	Completed	No Visible Failure
	New	2106	Completed	Completed	No Visible Failure
	3	2107	Completed	Completed	No Visible Failure
	3	2108	Completed	Completed	No Visible Failure
	4	2109	Completed	1.33	Tread Shoulder Cracking+Belt Edge
	4	2110	Completed	Completed	No Visible Failure
	5	2111	21.1	NA	Tread Shoulder Cracking+Belt Edge
	5	2112	23.2	NA	Tread Chunking
Y2	New	2145	Completed	Completed	No Visible Failure
	New	2146	Completed	Completed	No Visible Failure
	3	2147	Completed	Completed	No Visible Failure
	3	2148	Completed	Completed	No Visible Failure
	4	2149	Completed	Completed	No Visible Failure
	4	2150	Completed	Completed	No Visible Failure
	5	2151	Completed	Completed	No Visible Failure
	5	2152	Completed	Completed	No Visible Failure
N1	New	2157	Completed	Completed	No Visible Failure
	New	2158	Completed	Completed	No Visible Failure
	3	2160	Completed	Completed	No Visible Failure
	4	2161	Completed	Completed	No Visible Failure
	4	2162	Completed	Completed	No Visible Failure

Tire Type	Aging Time, weeks	Barcode	FMVSS 139 Endurance, hrs	FMVSS 139 Low Pressure, hrs	Failure Code
	5	2163	11.97	NA	Complete Tread and Belt 2 Detachment
	5	2164	9.5	NA	Complete Tread and Belt 2 Detachment
N2	New	2170	Completed	Completed	No Visible Failure
	New	2171	Completed	Completed	No Visible Failure
	3	2172	Completed	Completed	No Visible Failure
	3	2173	Completed	Completed	No Visible Failure
	4	2174	Completed	Completed	No Visible Failure
	4	2175	Completed	Completed	No Visible Failure
	5	2176	26.46	NA	Plycoat Delamination - Interface
	5	2177	18.73	NA	Innerliner Separation
B7	New	2242	Completed	Completed	No Visible Failure
	New	2243	Completed	Completed	No Visible Failure
	3	2245	Completed	Completed	No Visible Failure
	3	2246	Completed	Completed	No Visible Failure
	4	2247	Completed	Completed	No Visible Failure
	4	2248	Completed	Completed	No Visible Failure
	5	2251	Completed	Completed	No Visible Failure
	5	2256	22.45	NA	Sidewall Cracking
B1	New	2261	Completed	Completed	No Visible Failure
	New	2262	Completed	Completed	No Visible Failure
	3	2263	Completed	Completed	No Visible Failure
	4	2265	Completed	Completed	No Visible Failure
	4	2266	Completed	Completed	No Visible Failure
	5	2267	Completed	Completed	No Visible Failure
	5	2268	Completed	Completed	No Visible Failure
B8	New	2275	Completed	Completed	No Visible Failure
	New	2276	Completed	Completed	No Visible Failure
	3	2277	Completed	Completed	No Visible Failure
	3	2278	Completed	Completed	No Visible Failure
	4	2279	Completed	Completed	No Visible Failure
	4	2280	Completed	Completed	No Visible Failure
	5	2281	Completed	Completed	No Visible Failure
	5	2282	Completed	Completed	No Visible Failure
O4	New	2357	Completed	Completed	No Visible Failure
	New	2358	Completed	Completed	No Visible Failure
	3	2359	3.13	NA	Innerliner Separation
	3	2360	7.6	NA	Innerliner Separation
	4	2361	2.35	NA	Sidewall Cracking
	4	2362	1.1	NA	Tread Lug Chunking

Tire Type	Aging Time, weeks	Barcode	FMVSS 139 Endurance, hrs	FMVSS 139 Low Pressure, hrs	Failure Code
	5	2363	0	NA	Failed During Oven Aging
	5	2364	0	NA	Failed During Oven Aging
D6	New	2396	Completed	Completed	No Visible Failure
	New	2397	Completed	Completed	No Visible Failure
	3	2398	Completed	Completed	No Visible Failure
	3	2399	Completed	Completed	No Visible Failure
	4	2400	Completed	Completed	No Visible Failure
	4	2401	Completed	Completed	No Visible Failure
	5	2402	11.57	NA	Tread Shoulder Cracking+Belt Edge
	5	2403	Completed	Completed	No Visible Failure
M1	New	2461	Completed	Completed	No Visible Failure
	New	2462	Completed	Completed	No Visible Failure
	3	2463	Completed	Completed	No Visible Failure
	3	2464	Completed	Completed	No Visible Failure
	4	2465	Completed	Completed	No Visible Failure
	4	2466	Completed	Completed	No Visible Failure
	5	2467	Completed	Completed	No Visible Failure
	5	2468	28.5	NA	Sidewall Rupture+Delamination
M3	New	2474	Completed	Completed	No Visible Failure
	New	2475	Completed	Completed	No Visible Failure
	3	2476	Completed	Completed	No Visible Failure
	3	2477	Completed	Completed	No Visible Failure
	4	2478	Completed	Completed	No Visible Failure
	4	2479	Completed	Completed	No Visible Failure
	5	2480	Completed	Completed	No Visible Failure
	5	2481	Completed	Completed	No Visible Failure
B9	New	2506	Completed	Completed	No Visible Failure
	New	2507	Completed	Completed	No Visible Failure
	3	2508	Completed	Completed	No Visible Failure
	3	2509	Completed	Completed	No Visible Failure
	4	2510	Completed	1.17	Sidewall Separation
	4	2511	Completed	0.77	Sidewall Separation Innerliner Separation
	5	2512	Completed	Completed	No Visible Failure
	5	2513	Completed	Completed	No Visible Failure
C7	New	2531	Completed	Completed	No Visible Failure
	New	2532	Completed	Completed	No Visible Failure
	3	2533	Completed	Completed	No Visible Failure
	3	2534	Completed	Completed	No Visible Failure
	4	2535	Completed	Completed	No Visible Failure
	4	2536	Completed	Completed	No Visible Failure

Tire Type	Aging Time, weeks	Barcode	FMVSS 139 Endurance, hrs	FMVSS 139 Low Pressure, hrs	Failure Code
	5	2537	Completed	Completed	No Visible Failure
	5	2538	Completed	Completed	No Visible Failure
C8	New	2556	Completed	Completed	No Visible Failure
	New	2557	Completed	Completed	No Visible Failure
	3	2558	Completed	Completed	No Visible Failure
	3	2559	Completed	Completed	No Visible Failure
	4	2560	Completed	Completed	No Visible Failure
	4	2561	Completed	Completed	No Visible Failure
	5	2562	Completed	Completed	No Visible Failure
	5	2563	Completed	Completed	No Visible Failure
H3	New	2581	Completed	Completed	No Visible Failure
	New	2582	Completed	Completed	No Visible Failure
	3	2583	Completed	Completed	No Visible Failure
	3	2584	Completed	Completed	No Visible Failure
	4	2585	Completed	Completed	No Visible Failure
	4	2586	31.23	NA	Sidewall Rupture+Delamination
	5	2587	22.37	NA	Belt to Belt Separation
	5	2588	31.58	NA	Sidewall Rupture+Delamination
M10	New	2606	Completed	Completed	No Visible Failure
	New	2607	Completed	Completed	No Visible Failure
	3	2608	Completed	Completed	No Visible Failure
	3	2609	Completed	Completed	No Visible Failure
	4	2610	Completed	Completed	No Visible Failure
	4	2611	Completed	Completed	No Visible Failure
	5	2612	Completed	Completed	No Visible Failure
	5	2613	Completed	Completed	No Visible Failure
S1	New	2631	Completed	Completed	No Visible Failure
	New	2632	Completed	Completed	No Visible Failure
	3	2633	Completed	Completed	No Visible Failure
	3	2634	Completed	0.5	Belt Edge Separation
	4	2636	Completed	Completed	No Visible Failure
	5	2637	Completed	0.17	Sidewall Separation
	5	2638	15.77	NA	Belt Edge Exposed, Belt Edge
T2	New	2656	Completed	Completed	No Visible Failure
	New	2657	Completed	Completed	No Visible Failure
	3	2658	Completed	Completed	No Visible Failure
	3	2659	Completed	Completed	No Visible Failure
	4	2660	Completed	Completed	No Visible Failure
	4	2661	Completed	Completed	No Visible Failure
	5	2662	Completed	Completed	No Visible Failure
	5	2663	Completed	Completed	No Visible Failure

All new tires completed the endurance and low pressure tests. Forty-six percent (6/13) of passenger tire models and 43 percent (3/7) of light-truck tire models exhibited no failures after oven aging up to 5 weeks at 65°C. The percentage of individual oven-aged tires failing prior to completion of the test is shown in Figure 5. Over 40 percent of passenger tires (11/26) and nearly 60 percent (8/14) of light-truck tires failed before the end of the test.

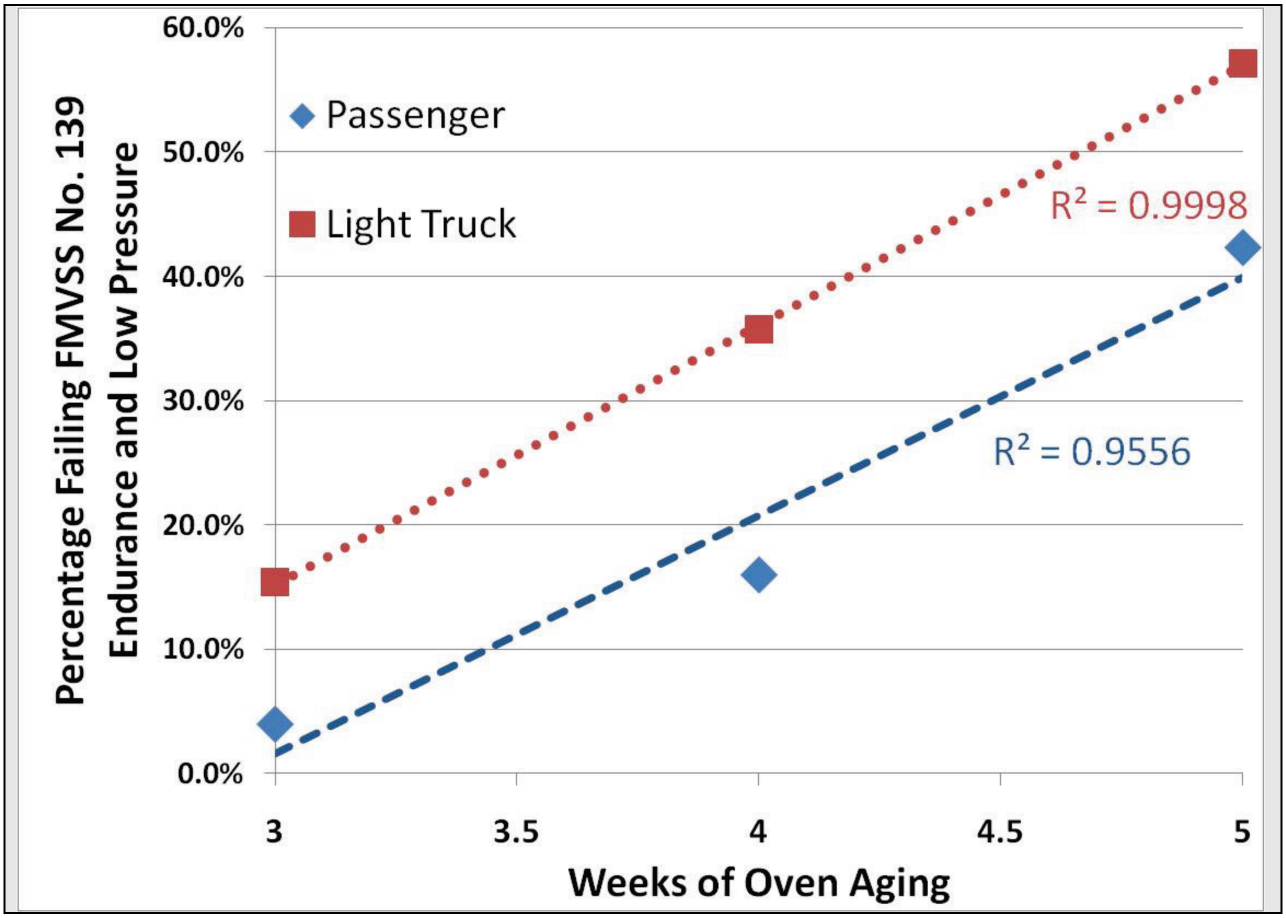


Figure 5. Percentage of Tires That Failed Before the Completion of the FMVSS 139 Endurance and Low Pressure Test Versus Weeks of Oven Aging at 65°C

Figure 6 shows the failure sites for passenger tires. Approximately 70 percent of the failures occurred in the critical belt edge and shoulder region of the tire. Figure 7 shows the failure sites for the light-truck tires. Failures in light-truck tires consisted predominantly of separations between components in the innerliner and sidewall region of the tire, including two tires that separated in the oven during aging. Inspection of the failures indicated that the separations were likely initiated by a buildup of air pressure between the components. Figure 8 shows a failure that took place at the ply end of the sidewall, and Figure 9 shows a separation between the innerliner and the ply coat that took place during oven aging of the tires.

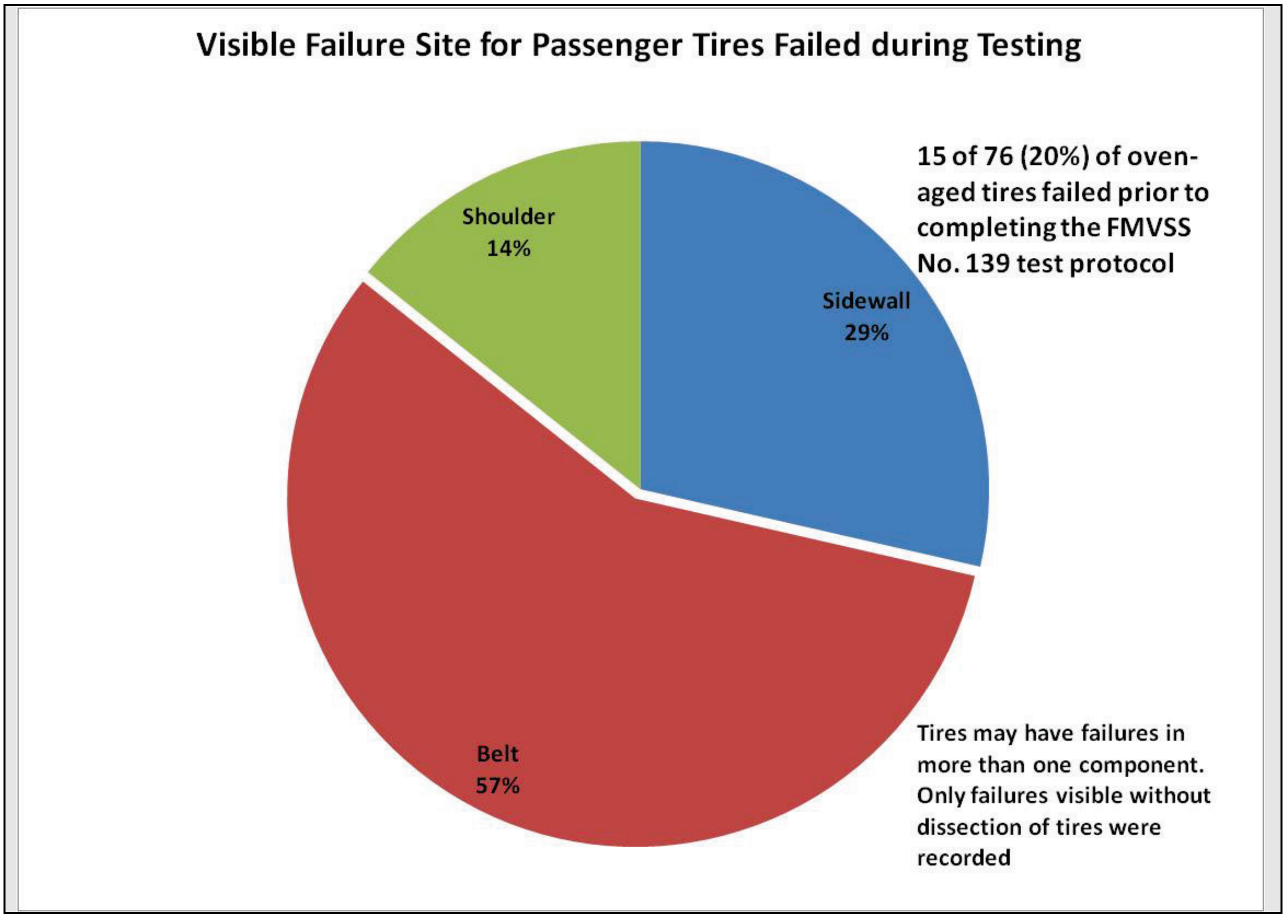


Figure 6. Failure Site for Passenger Tires That Did Not Complete the FMVSS 139 Endurance and Low Pressure Test Protocol

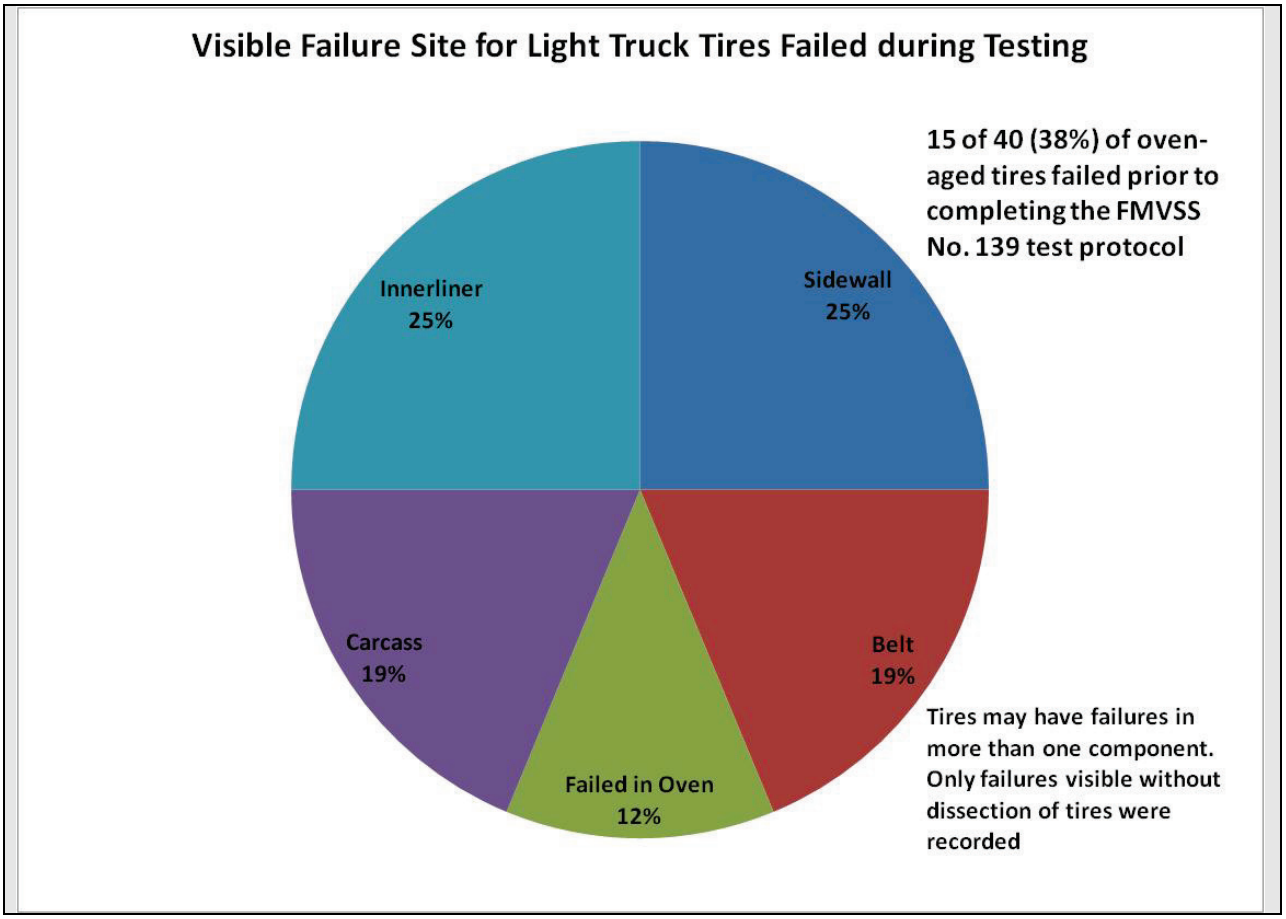


Figure 7. Failure Site for Light Truck Tires That Did Not Complete the FMVSS 139 Endurance and Low Pressure Test Protocol

Figure 8. Failure at Ply End of Sidewall During Oven Aging

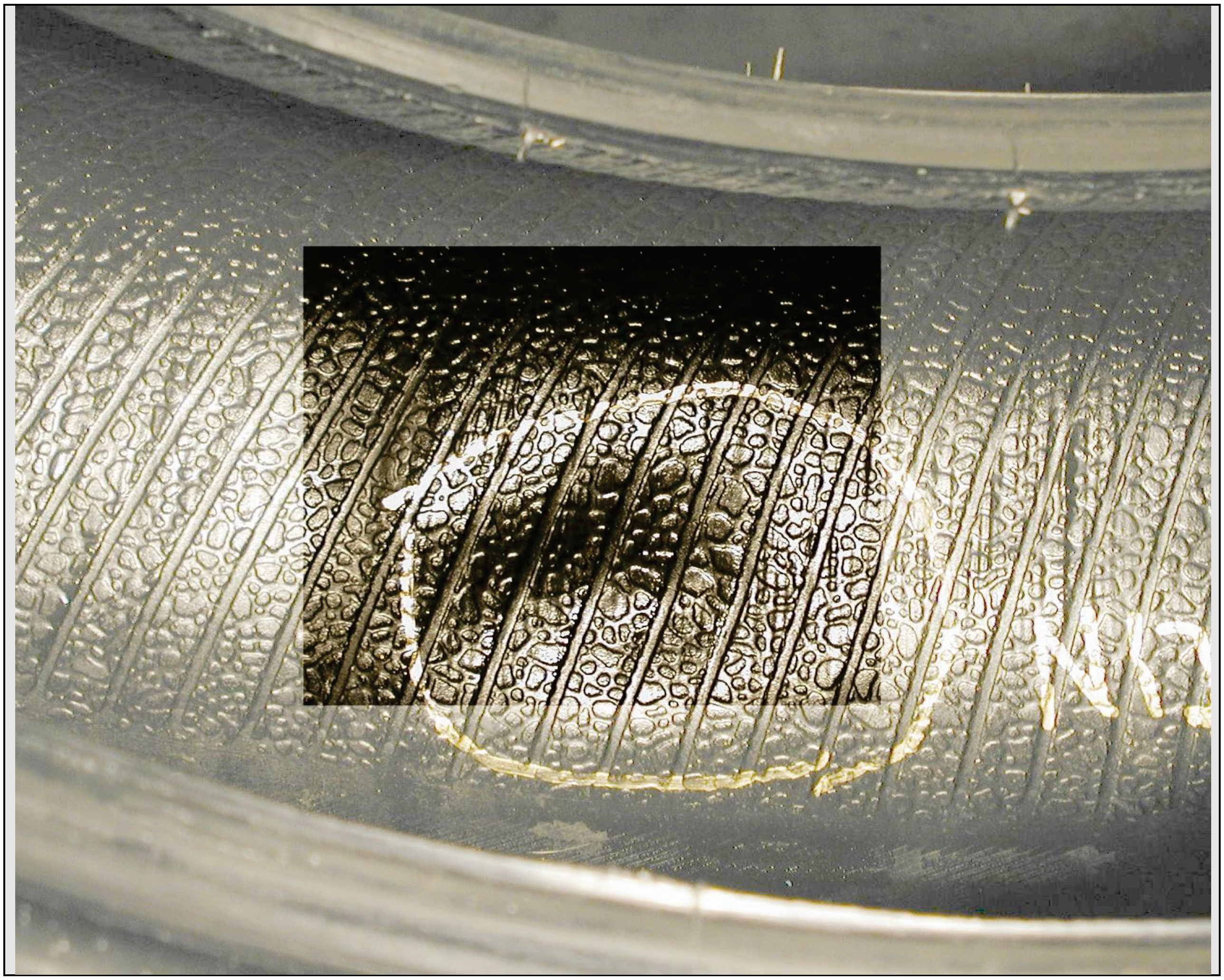


Figure 9. Separation of Innerliner From Ply During Oven Aging

Seven of 13 models (54%) of passenger tires and 2 of 7 models (29%) of the light-truck tires completed the endurance and low pressure test after all of the aging conditions. For the 6 models of passenger tires that failed prior to completing the test, the failure times versus weeks aging are shown in Figure 10. Very few failures were observed prior to 5 weeks of oven aging. For light-truck tires the failure times are shown in Figure 11. Like the passenger tires, most light-truck tire models also exhibited few failures after aging times of less than 5 weeks. However the failure times were generally less than those seen on passenger tires.

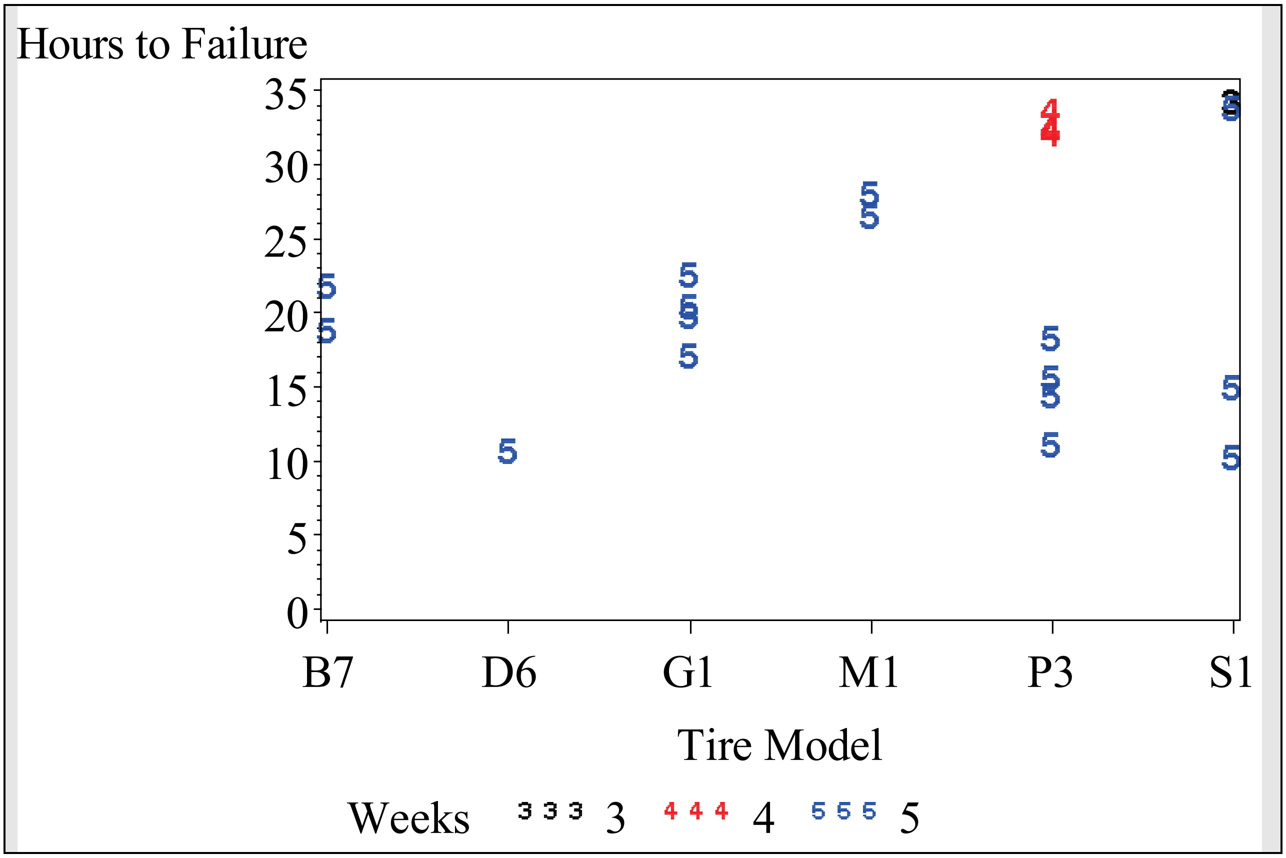


Figure 10. Passenger Tire Failure Times by Tire Model and Weeks of Oven Aging

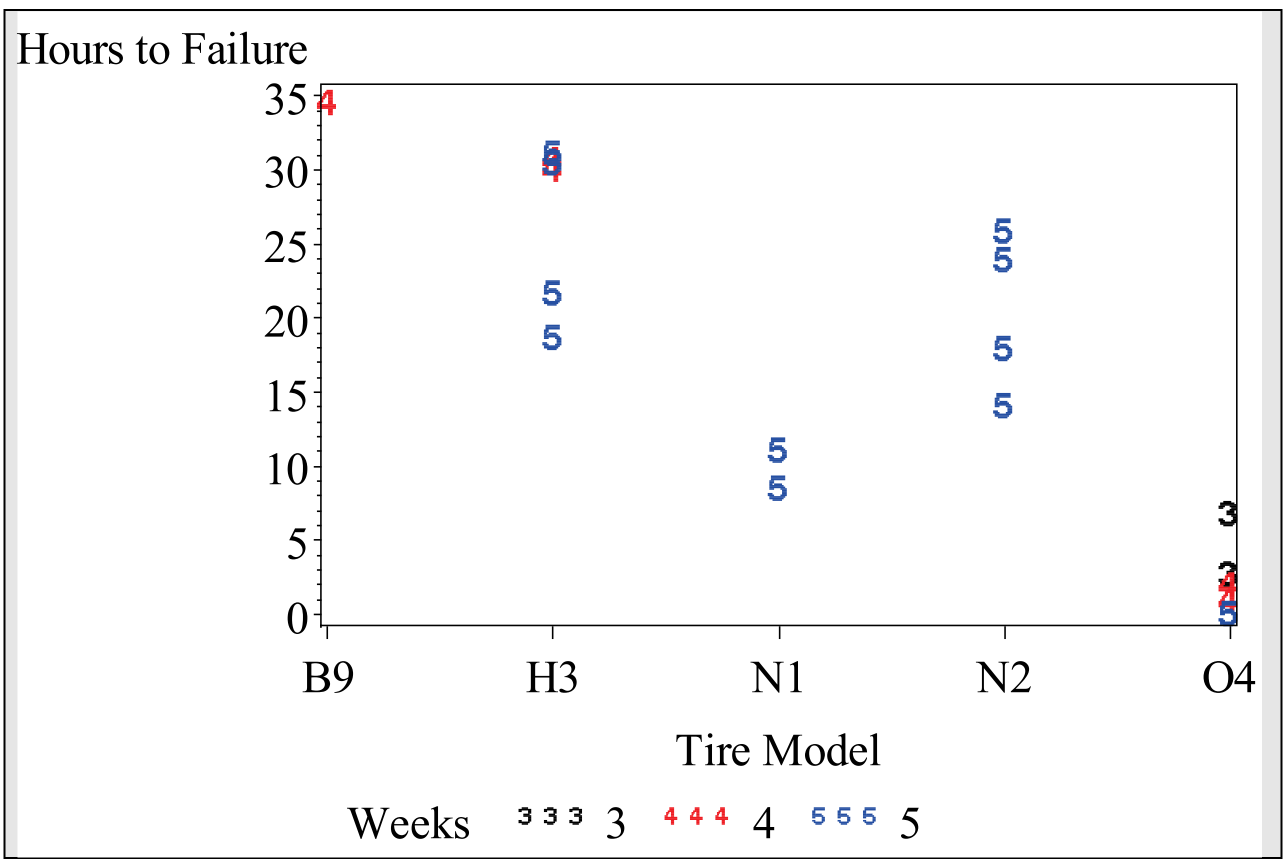


Figure 11. Light Truck Tire Failure Times by Tire Model and Weeks of Oven Aging

4.0 SUMMARY AND CONCLUSIONS

The performance tests in FMVSS 139 subject new tires to conditions on a curved indoor roadwheel more severe than normal operating conditions on a flat road surface. Still, the indoor roadwheel tests in the safety standard do not evaluate the structural durability of a tire after it has experienced long-term material property degradation under cyclic fatigue. In this context, it is logical to ask whether the thermo-oxidative degradation and fatigue cracking of internal rubber components observed in tires retrieved from service contribute to a decrease in a tire's resistance to operational conditions. For example, would 1-year-old passenger vehicle tires have less chance of structural failure while operated underinflated or overloaded, or during long periods of high-speed use, than tires over 8 years of age (7% of passenger vehicle tires)?

Accordingly, the first goal of tire aging research was to develop a better understanding of service-related tire degradation over time. Phase 1 addressed this goal and its research reported that there are two mechanisms operating to produce changes in tire properties, particularly in the critical belt-edge region: (1) degradation of the rubber compound and material interfaces due to the effects of heat and reaction with oxygen (thermo-oxidative aging), and (2) the effect of cyclic fatigue during tire deformation.

Phase 2 focused on developing an accelerated laboratory-based test to simulate tire aging using the same 6 tire models that were evaluated from service in Phoenix. The physical and chemical properties of the belt coat stocks and the remaining structural durability of the laboratory-aged tires were compared to the 6 tire models previously evaluated after long-term service (aging) on vehicles in Phoenix. The most promising method was a method in which the tire is inflated using a 50%N_2/50%O_2 mixture, and heated in an oven for a period of time to accelerate the aging process by speeding up chemical reactions, and thus material property changes. The tire may then be studied for material property changes or run on a roadwheel to determine any change in durability.

The material and chemical properties of the belt compounds showed that the properties of the components tended to change in the same direction with increased time in service in Phoenix or with increased time of laboratory aging. Particularly, the hardness, modulus, cross-link density, and oxygen content tended to increase while the tensile strength, ultimate elongation, peel adhesion, and flex properties tended to decrease over time. All of these changes are consistent with the proposed mechanism of thermo-oxidative aging.

The oven-aged tires were also tested using a stepped-up-load test intended to compare the structural integrity of the tire after aging to that of a new tire. Oven aging tires for 3 weeks did not cause a significant decrease in structural integrity compared to a new tire of the same model. Oven aging tires for 6 weeks at 60°C to 70°C decreased the running time on the stepped-up-load test to that of a tire with approximately 1 to 3 years of service in Phoenix, while oven aging for 8 weeks at 60°C to 70°C gave running times consistent with Phoenix service tires with 5 or greater years of service.

The third phase of the research compared selected physical and chemical properties for 10 passenger and 6 light-truck tire models after aging for 10 weeks at 60°C, or 8 weeks at 65°C, while inflated with a mixture of 50%N_2/50%O_2 gas, to those of new tires of the same model. The wire-coat compound between the belts and the wedge compound in the belt edge region were studied in detail. The residual integrity of the tires was measured using a stepped-up-load test to structural failure after aging for 7 weeks at 60°C, or 5 weeks at 65°C. The effect of a 23 hour break-in cycle on a 1.707 m roadwheel at 80 km/h prior to oven aging was also investigated.

No in-service tires were available for comparison, so values were compared to the relative properties – the percent of the new tire property that was retained after aging – of the tires from service in Phoenix. The physical properties of all tire models changed in the same manner as the tires investigated in service in Phases 1 and 2. Specifically, the indentation modulus of the wire-coat compounds and shoulder area of the tire tended to increase during aging to levels greater than the average values of tires that had experienced 3 years of service in Phoenix. The tensile strength and ultimate elongation decreased to values less than the retained values from the average tires from service in Phoenix.

The rates of the chemical reactions occurring during oven aging were calculated using literature referenced values for activation energy of natural rubber. The oxidation reaction in the natural rubber wire-coat compounds of tires aged for 10 weeks at 60°C, or 8 weeks at 65°C, was calculated to be approximately equivalent to service in Phoenix of 4.8 and 6.5 years, respectively. Aging studies conducted at Ford Motor Company estimated that aging for 8 weeks at 65°C produces changes in physical properties in tires approximately equivalent to 5.5 years of service in Phoenix. Ahagon and co-workers showed that the slope of the plot of log(modulus at 100% elongation) versus log(ultimate elongation), referred to as the Ahagon number, correlates to the mechanistic type of aging. The wire-coat compounds of the oven-aged tires had slopes near -0.75, which correspond to Type 1 oxidative aging.

Tires aged in the oven for 7 weeks at 60°C, or 5 weeks at 65°C, were tested on a 1.707 m roadwheel using the stepped-up-load test to structural failure used in Phase 2 testing. The aging conditions were calculated using the Arrhenius equation to be approximately equal to 3.4 and 4.0 years of service in Phoenix, respectively. Twenty-eight of 57 aged passenger tires (49%) exhibited visible failures at times less than 34 hours. Approximately two-thirds of these failures occurred in the belt area, and 22 percent of failures took place in the shoulder area. This is similar to the customer reported tire failures received by NHTSA, with over 90 percent of them occurring in the belt and shoulder area. Thirty-five of 46 aged light-truck tires (76%) exhibited visible failure in less than 34 hours of roadwheel testing. Approximately one-third of these failures occurred in the belt area, with another 10 percent of failures taking place in the shoulder area.

Based on measured changes in material properties, oven aging tires at 60°C to 70°C while inflated with a mixture of 50%N_2/50%O_2 gas produces thermo-oxidative aging effects qualitatively similar to those found in tires after long-term service in Phoenix. When measured as a percentage of new tire properties, all were within the range of values found for the Phase 1 and Phase 2 work. For certain tires, it was found that achieving the modulus found during service requires a break-in cycle prior to oven aging, which was found to correlate to the use of a thermoset reinforcing resin in the rubber compounds. A break-in cycle of 23 hours at 80 km/h still reduced the running time of the tires after oven aging compared to tires with no break-in.

In the first part of the Phase 4 work tires from 10 models were subjected to a 2-hour break-in on a 1.707 m roadwheel at 80 km/h, then inflated with a mixture of 50%N_2/50%O_2 and aged in a circulating air oven for 3 or 5 weeks at 65°C. The fill gas was vented and refreshed weekly to maintain a sufficient supply of oxygen gas in the tire cavity to support oxidative aging.

Physical properties of the critical belt edge region of the aged tires were measured and the percentage change from the new tire properties compared to the percentage change seen in tires retrieved from service in Phoenix. All of the measured physical properties changed in the same direction as the tires from Phoenix service. The modulus and the percent of fixed oxygen increased, while the elongation to break, the tensile strength, and the peel adhesion strength decreased. The increase in fixed oxygen and the slope of the log(modulus at 100% elongation) versus log(ultimate elongation) both indicated that the chemical reaction in the rubber compound was similar to the reaction found in tires during service, specifically oxidative degradation. Oven aging from 3 to 5 weeks at 65°C produced percentage changes in properties similar to those found in tires with 3 to 6 years of service in Phoenix.

Previous work had shown that some tires not subjected to a break-in cycle showed a dramatic increase of indentation modulus in the belt edge compounds during oven aging while the indentation modulus actually decreased during service in Phoenix. A break-in cycle eliminated the increase in indentation modulus, however even the break-in cycles of 24 hours at 120 km/h or 23 hours at 80 km/h in Phases 3 and 4 reduced the running time of the tires compared to those with no break-in. The break-in cycle of 2-hours at 80 km/h was sufficient to eliminate the increase of indentation modulus during oven aging without a noticeable reduction in running time.

Ten models of tires were tested using the stepped-up-load to structural failure (SUL) roadwheel test used in Phase 2 and 3 work. The running time of tires aged for 5 weeks at 65°C were similar to those of tires that had been in Phoenix service for 3 to 6 years. Five of the tire models failed prior to 34 hours, even though the load on the tires did not exceed their maximum design load. Nearly all of the failures in the passenger tires were in the critical belt edge and shoulder area. The failures in the light-truck tires consisted mainly of separations between the carcass compounds or between the innerliner and ply of the tire. Overall, the results were similar to those from the Phase 3 work.

The SUL test produced failures of oven-aged tires at times similar to those of the tires from service in Phoenix. It also discriminated between tire models, since all tires of some models did not fail until the loads significantly exceeded their maximum design while others failed at loads of 100 percent or less of their maximum rated load.

The FMVSS 139 endurance and low pressure test was designed to evaluate the structural durability of new tires. Twenty tire models were aged and tested to determine if this was truly an appropriate test to determine the durability of aged tires. Tires were oven aged for 3, 4, or 5 weeks at 65°C while inflated with the 50%N_2/50%O_2 mixture and then tested according to the FMVSS 139 endurance and low pressure test. Aging 3 weeks at 65°C is approximately equivalent to 2.4 years of service and aging 5 weeks at 65°C approximates the aging experienced by a tire with 4.0 years of service in Phoenix. All new tires completed the endurance and low pressure test and 46 percent (6/13) of passenger tire models and 43 percent (3/7) models of light-truck tires exhibited no failures after oven aging up to 5 weeks at 65°C.

Approximately 70 percent of the failures for passenger tires took place in the critical belt edge and shoulder region of the tire. The light-truck tires failures were predominantly separations between components in the innerliner and sidewall region of the tire, including two tires that separated in the oven during aging. Inspection of the failures indicated that the separations were likely initiated by a buildup of air pressure between the components.

Seven of 13 models (54%) of passenger tires and 2 of 7 models (29%) of the light-truck models completed the endurance and low pressure test after all of the aging conditions. For the 6 models of passenger tires that failed, few failures occurred prior to 5 weeks of oven aging. The failure times for light-truck tires were, on-average, less than those for passenger tires.

Nine of the tire models had no failures even after being oven aged for 5 weeks, tires from 2 models failed after 3 weeks of oven aging, 3 additional models did not complete the test after 4 weeks of oven aging and a total of 11 models did not complete the test after 5 weeks of oven aging.

Appendix 1 – Detailed Contract Service Specifications

Notes:

- 0° degrees corresponds the “D” in the DOT number on the sidewall. “SS” refers to the Serial Side (side with DOT number). “OSS” refers to the Opposite Serial Side.
- GFE refers to Government Furnished Equipment
- The contractor shall individually label every test sample (when possible) and tire remnant. Tire test samples shall be stored in safe storage conditions in clearly labeled polyethylene or Mylar bags provided by the contractor for each test sample.

All units shall be SI unless otherwise specified in the task order. All data shall be submitted in a MS Excel format unless otherwise specified in the task order requirements.

Standards Referenced:

ASTM D 412-98a(2002)$^{\epsilon 1}$	ASTM D5992-96(2001)e1
ASTM D 413-98(2002)$^{\epsilon 1}$	ASTM E1131-98
ASTM D430-95(2000)	ASTM F 421-00
ASTM D 638-02a	ASTM F 538-99
ASTM D813-95(2000)	ASTM F 551-89
ASTM D 1171-99	ASTM F 1112-00
ASTM D 1434-82(1998)	SAE J1561 (February 2001)
ASTM D 2240-02b	SAE J1205-J1206

Required tests and services are specified in the following table.

The contractor may add additional contract items or labor categories to the end of the table.

Specifications

Item 1 – Tire Storage **Pricing:** Price is per tire - per month. Storage costs may be prorated on a per time basis. Price includes all costs associated with this service. **Labor Costs:** Included in the price. **GFE:** New and used tires. **Specifications:** All tires in the contractor's possession shall be stored in a secure, out-of-the-weather, storage area. The storage area does not have to be heated, but temperatures shall not exceed 32°C (90° F) at any time. The tires shall be stored in a dry, well-ventilated environment away from direct sunlight and electric motors. Untested tires shall be turned every 4-6 weeks to prevent flat spots. Tires shall be stored vertically, on racks, and in an un-mounted condition. Tires may temporarily be stored in horizontal stacks during testing and handling, but the stacks shall never exceed four tires in height. **Deliverables:** Tire storage per tire per month (may be prorated) under specified conditions.
Item 2 – Wheel Storage **Pricing:** Price is per wheel - per month. Storage costs may be prorated on a per time basis. Price includes all costs associated with this service. **Labor Costs:** Included in the price. **GFE:** New wheels. **Specifications:** All wheels in the contractor's possession shall be stored in a secure, out-of-the-weather, storage area. **Deliverables:** Wheel storage per wheel per month (may be prorated) under specified conditions.
Item 3 - Tire Mounting on Wheel, Dry Air Inflation, New Valve Stem **Pricing:** Price is per tire mount. Price includes all costs associated with the service, including valve stem assembly, and tire lube. **Labor Costs:** Included in the price. **GFE:** New and used tires, new wheels. **Specifications:** The tire shall be mounted on the wheel size specified by the COTR in the task order: A new tire valve assembly (stem and core), provided by the contractor, shall be installed in the wheel every time a tire is mounted. The valve stem rubber must be an EPDM compound and must be certified as meeting SAE J1205 & J1206 standard requirements. The tire shall be mounted using ready-to-use Murphy's Tire and Tube Mounting Lubricant (no mixing from

concentrate and absolutely no petroleum or water based bead lubricants). Tires shall be seated in accordance with all OSHA, RMA, tire manufacturer, and internal lab procedures. Tires shall be inflated to test specified inflation pressure with air that has had the moisture removed such that the outlet pressure dew point at rated conditions is 38°F (3°C). For tests that require inflation pressures that exceed sidewall maximum inflation pressure, tires shall be inflated in a safety cage (similar to ones used for truck tire inflation).

Deliverables: One tire mounted and inflated on wheel with a new tire valve and inflated with dry air to the specified test pressure.

Item 4 - Tire Mounting on Wheel, 50%N_2/50%O_2 **Inflation, New Valve Stem**

Pricing: Price is per tire mount. Price includes all costs associated with the service, including valve stem assembly, and tire lube.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: The tire shall be mounted on the wheel size specified by the COTR in the task order using air to seat the bead: A new tire valve assembly (stem and core), provided by the contractor, shall be installed in the wheel every time a tire is mounted. The valve stem rubber must be an EPDM compound and must be certified as meeting SAE J1205 & J1206 standard requirements. To assist in mounting, the tire shall be mounted using ready-to-use Murphy's Tire and Tube Mounting Lubricant (no mixing from concentrate and absolutely no petroleum or water based bead lubricants). Tires shall be seated using compressed air in accordance with all OSHA, RMA, tire manufacturer, and internal lab procedures. Tires shall then be completely deflated and re-inflated to the specified test pressure with a 50%N_2/50%O_2 certified blend with a certified
accuracy of at least ± 0.5%. For tests that require inflation pressures that exceed sidewall maximum inflation pressure, tires shall be inflated in a safety cage (similar to ones used for truck tire inflation).

Deliverables: One tire mounted and inflated on wheel with a new tire valve and inflated with a 50%N_2/50%O_2 certified blend to the specified test pressure.

Item 5 - Tire Mounting on Wheel, 100% N_2 Inflation, New Valve Stem

Pricing: Price is per tire mount. Price includes all costs associated with the service, including valve stem assembly, and tire lube.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: The tire shall be mounted on the wheel size specified by the COTR in the task order: A new tire valve assembly (stem & core), provided by the contractor, shall be installed in the wheel every time a tire is mounted. The valve stem rubber must be an EPDM compound and must be certified as meeting SAE J1205 & J1206 standard requirements. The tire shall be mounted using ready-to-use Murphy's Tire and Tube Mounting Lubricant (no mixing from concentrate and absolutely no petroleum or water-based bead lubricants). Tires shall be seated using compressed air in accordance with all OSHA, RMA, tire manufacturer, and internal lab procedures. Tires shall then be completely deflated and re-inflated to the specified test pressure with a 100% N2 certified gas with a certified accuracy of at least ± 0.5%. For tests that require inflation pressures that exceed sidewall maximum inflation pressure, tires shall be inflated in a safety cage (similar to ones used for truck tire inflation).

Deliverables: One tire mounted and inflated on wheel with a new tire valve and inflated with a 100% N2 inflation gas to the specified test pressure.

Item 6 - Cut Tires into Sections

Pricing: Price is per cut. Price includes all costs associated with this service.

Labor Costs: Included in the price.

GFE: New and used tires.

Specifications: Some tires will have to be cut into sections and the sections distributed per the direction of the COTR. Pricing is per radial cut, perpendicular to the tread, from tread through the two beads. The number of cuts will be specified in the task order. This service is different from the sectioning work required for sample preparations.

Deliverables: One complete radial tire cut from the surface of the tread through the two beads.

Item 7 - Tire & Tire Section Transport: Inter-laboratory, Greater Akron Ohio Area

Pricing: Price is per tire per mile. Price includes all costs associated with this service.

Labor Costs: Included in the price.

GFE: New and used tires, new wheels.

Specifications: The contractor may be required to arrange either pickup or drop-off of tires or tire sections at multiple locations in the Greater Akron Ohio area (including Akron, Massillon, and Ravenna). Prices shall be quoted per tire, per mile. Travel without tires is not chargeable. For instance, Lab A delivers 10 tires to Lab B 20 miles away. This is 200 tire-miles. The return trip, if empty is not charged. If the truck brings back 5 tires, this is an additional 100 tire-miles. Deliveries outside of the Greater Akron area, if required, will be arranged by the government, at the government's expense.

Deliverables: One roundtrip delivery or pickup of tires or tire sections from another location in the Greater Akron Ohio area.

Item 8 - Move Tire-Wheel Assembly Between Oven and Roadwheel, or Vice Versa

Pricing: Price is per tire per move. Price includes all costs associated with this service.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: During the Hybrid Oven and Roadwheel Aging Method, it is anticipated that mounted tires will have to be moved from the oven to a roadwheel, or vice versa. During each move, the tire shall be:

- Uninstalled from the roadwheel or oven
- Allowed to cool if necessary
- Deflated
- A new tire valve assembly, provided by the contractor, installed (new stem and core, valve
- stem rubber must be an EPDM compound and must be certified as meeting SAE J1205 & J1206 standard requirements)
- If a bead unseats, reseated beads with compressed air inflation and then completely deflate tire
- Re-inflated with fresh 50%N2/50%O2 gas (± 0.5% certified blend)
- Reinstalled in the new location

For tests that require inflation pressures above sidewall maximum inflation pressure, tires shall be inflated in a safety cage (similar to ones used for truck tire inflation).

Deliverables: One tire moved from oven to roadwheel, or vice versa, per specifications.

Item 9 – Tire & Remnants Disposal

Pricing: Price is per tire. Tire sample and remnant disposal may be prorated based on weight or volume. Price includes all costs associated with the service.

Labor Costs: Included in the price.

GFE: New and used tires.

Specifications: All tires, test samples, and tire remnants shall be preserved by the contractor in appropriate safe storage conditions until the contractor either receives written notice from the Government authorizing disposal, or the items are returned to the Government at the Government's option. Disposal of tires, test samples, and tire remnants shall comply with all applicable tire disposal regulations. Any whole tires leaving the test facility for disposal shall be made unusable by the contractor before departure to prevent possible reuse by the public (even tires sent to a third party for disposal).

Deliverables: One tire, or tire remnant on a prorated basis, properly disposed of per specifications.

Item 10 – Tire Inspection, Used Tires

Pricing: Price is per tire inspection. Price includes all costs associated with the service and data report.

Labor Costs: Included in the price.

GFE: Used tires.

Specifications: The contractor shall provide qualified personnel to inspect or measure seven used tire properties: Average tread depth,[1] surface crack coding,[2] innerliner crack coding,[3] inspection for number of punctures,[4] repair type coding,[5] Shore durometer readings of the tread,[6] and macro tire damage coding.[7] All data shall be submitted in an MS Excel format.

[1]Average tread depth measurement shall be per ASTM F 421-00. Follow Section 9.2.1 *Preferred Method* and Section 9.3.1 or 9.3.2 (depending on the tread width). Follow Section 9.4.2 *Minimum Requirement* regarding measurements and spacing. Report raw and average values for each groove (void).

[2]Surface crack coding shall be per ASTM D 1171-99, Section 7.3 *Rating of Exposed Specimens*. Notes or comments may be added.

[3]Innerliner crack coding shall be per ASTM D 1171-99, Section 7.3 *Rating of Exposed Specimens*. Notes or comments may be added.

[4]Count the number of punctures, with the location of each puncture clearly marked on the tire and documented.

[5]Repair type coding shall follow standard industry nomenclature (plug, patch, not repaired, etc.). Notes or comments may be added.

[6]Shore durometer readings on the Shore A scale shall be completed per ASTM D 2240-02b. Measurements shall be conducted per Section 9.1.8 or 9.2.5 on the tire tread (5 determinations of hardness at least 6.0mm (0.24 in) apart at a location the corresponds to one of the tread depth measurement locations). Report raw and average values.

[7]Macro tire damage, such as belt separation, chunking, etc., shall be coded per SAE J1561 (February 2001) or ASTM F 538-99.

Data Units: mm

Deliverables: One tire inspection that produces the following measurements. All data shall be submitted in an MS Excel format.

- Six raw groove depth data points per groove
- Average groove depth on a circumferential line for each groove
- One average tread depth for the entire tire
- Surface crack coding of outer tire surfaces
- Innerliner crack coding
- Number of tire punctures with the location of each puncture marked on the tire and documented (if any)
- Repair type coding (if any)
- Five raw shore hardness numbers for the tire tread
- One average shore hardness number for the tire tread
- Documentation of macro tire damage (if any)

Item 11 – Tire Inspection, Post Test

Pricing: Price is per tire inspection. Price includes all costs associated with this service and data report.

Labor Costs: Included in the price.

GFE: New and used tires.

Specifications: Any tire tested on a roadwheel shall be inspected after each complete roadwheel sequence for tire damage. The tire shall be inspected by qualified personnel and categorized as passing the inspection or failing the inspection. [Failed tires shall not progress to further testing. The Government shall be notified of a tire that failed inspection so that a suitable replacement can be obtained. The contractor shall take three digital photographs from various angles, per Item 12 specifications, of the failed tire. The cost of the photography will be charged separately under Item 12.] Data should be submitted in a MS Excel format. For failed tires, detailed descriptions of the failures shall be annotated per SAE J1561 (February 2001) and/or ASTM F 538-99. Tires purposely run to failure shall have a clearly annotated description of the failure type and location.

Deliverables: Documented tire rating of pass or fail per each inspection. For failed tires, detailed documentation of failure type and location, as well as three digital pictures of the failure are required. All data shall be submitted in a MS Excel format, pictures in a jpeg format.

Item 12 – Digital Photo

Pricing: Price is per photo. Price includes all costs associated with the service and media storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Specifications: The contractor shall take a clear, 300 dpi or better digital photograph, using sufficient lighting and focal length. The photograph shall contain the view of the object as requested by the task order. The digital camera used for the photography shall have a minimum of 3.0 megapixels. Photographs shall be saved and submitted to the Government in a digital jpg format.

Deliverables: One clearly viewable 300 dpi digital photograph per task order specifications in a digital jpg format.

Item 13 – Shearography

Pricing: Price is per bead-to-bead shearography inspection. Price includes all costs associated with the service, data report, and media storage.

Labor Costs: Included in the price.

Predecessors: Inspection for used tires.

GFE: New and used tires.

Specifications: Shearography shall be conducted per the procedure specified by the equipment manufacturer. The tires shall undergo bead-to-bead shearography, a qualified expert shall interpret the results, and suspect areas of the tire shall be clearly marked on the tire and their existence annotated in a MS Excel spreadsheet. A qualified expert for shearography interpretation is someone who has been trained by the shearography machine manufacturer on the proper shearography techniques and shearography interpretation. The expert must have billed at least 500 hours of previous shearography interpretation work to qualify as an expert. Resultant images of the individual sections recorded shall be stored in the native format of the equipment and a copy submitted to the Government. If the native format of the machine is not a pdf, also supply a copy of the images in a pdf format.

Deliverables: The complete set of digital tire section images stored in the native format of the machine as well as a digital copy of those images stored in a pdf format if pdf is not the native machine format. Results of analysis of shearography images, done by a qualified expert, shall be submitted in a MS Excel format on CD or DVD media. In addition, any accompanying notes or data shall be submitted.

Item 14 - Microscopy

Pricing: Price is per microscopy inspection. Price includes all costs associated with the service, including test sample preparation, data report, and sample, data, and media storage.

Labor Costs: Included in the price.

Replicates: Four.

Sample Regions: Radial cross sections at 0, 90, 180, and 270 degrees.

GFE: New tires.

Specifications: Section tire into appropriate test specimens. Use microscopy to measure the following component thickness:

Component gauges at center of crown
- Innerliner
- Squeegee/gumstrips
- No. 1 body ply
- No. 2 body ply
- No. 1 belt
- No. 2 belt
- Skim rubber between belts
- Cap ply (if any)
- Undertread

- SW/Base
- Tread

Component gauges belt edge
- Innerliner
- Wedge
- #1 Belt Width
- #2 Belt Width
- Belt Step
- Inter Belt Gauge
- Wedge Gauge/Location
- Buttress Gauge
- Base Gauge
- W7 Gauge
- Squeegee (barrier)
- Plycoat
- Shoulder wedge
- Beltcoat #1
- Beltcoat #2
- Tread base
- Tread (under lug)
- Tread (under groove)

Data Units: mm

Deliverables: A digital picture of the tire cross-section with a traceable rule and each measured component clearly identified. In addition, the following data points shall be submitted:
- Four sets of raw thickness measurements, one data point for each component listed
- One average thickness measurement for each component listed

All data shall be submitted in an MS Excel format on CD or DVD media.

Item 15 – Tire Air Permeability per ASTM F 1112-00, 21°C (70°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and data storage.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: Measure whole tire air permeability per ASTM F 1112-00. Per Section 9.6 of the standard, the test period may be shorter that the commonly used 180 days depending on the precision level of the data. If a shorter test period is desired, the contractor shall obtain written authorization by the Government to use a test period less than the specified 180 days. Proof of data accuracy and precision must be submitted if the contractor recommends a reduced test duration period. The test chamber shall be controlled to provide a mean ambient temperature that is within ± 0.6°C (1.1°F) of the 21°C (70°F) nominal test temperature and with overall variation within ±3°C (±5°F) over the course of the test.

Data Units: days, kPa, °K

Deliverables: Tire air permeability data per ASTM F 1112-00, Section 11 *Report* submitted in an MS Excel format.

Item 16 – Tire Air Permeability per ASTM F 1112-00, 70°C (158°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and data storage.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: Measure whole tire air permeability per ASTM F 1112-00. Per Section 9.6, the test period may be shorter that the commonly used 180 days depending on the precision level of the data. If a shorter test period is desired, the contractor shall obtain written authorization by the Government to use a test period less than the specified 180 days. Proof of data accuracy and precision must be submitted if the contractor recommends a reduced test duration period. Disregard Section 8.2 (nominal test temperatures). The test chamber shall be controlled to provide a mean ambient temperature that is within ± 0.6°C (1.1°F) of the 70°C (158°F) nominal test temperature and with overall variation within ±3°C (±5°F) over the course of the test.

Data Units: days, kPa, °K

Deliverables: Tire air permeability data per ASTM F 1112-00, Section 11 *Report* submitted in an MS Excel format.

Item 17 – Innerliner Air Permeability per ASTM D 1434-82(1998), 21°C (70°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: Section tire into appropriate test specimens. Measure tire innerliner air Gas Transmission Rate (GTR), permeance, and permeability per ASTM D 1434-82(1998).

Data Units: °C, mm, $mol/(m^2 \cdot s)$, $mol/(m^2 \cdot s \cdot Pa)$, $mol/(m \cdot s \cdot Pa)$

Deliverables: Tire innerliner air GTR, permeance, and permeability data measured per specifications and submitted in an MS Excel format.

Item 18 – Innerliner Air Permeability per ASTM D 1434-82(1998), 70°C (158°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New tires, new wheels.

Specifications: Section tire into appropriate test specimens. Measure tire innerliner air Gas Transmission Rate (GTR), permeance, and permeability per ASTM D 1434-82(1998).

Data Units: °C, mm, $mol/(m^2 \cdot s)$, $mol/(m^2 \cdot s \cdot Pa)$, $mol/(m \cdot s \cdot Pa)$

Deliverables: Tire innerliner air GTR, permeance, and permeability data measured per specifications and submitted in an MS Excel format.

Item 19 – Intracarcass Pressure 21°C (69.8°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

Replicates: Four needles per tire.

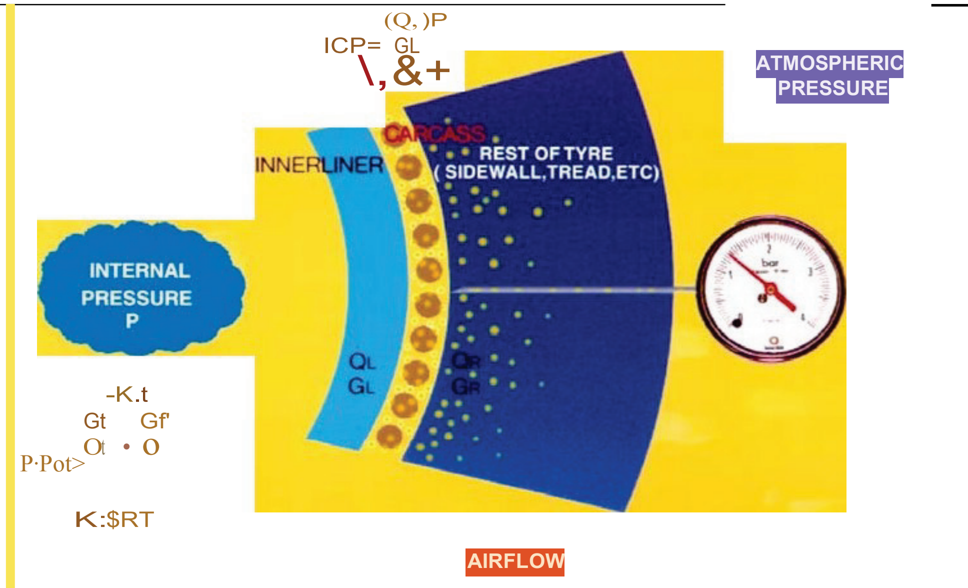
Sample Regions: Carcass, SS @ 0, 90, 180, and 270 degrees.

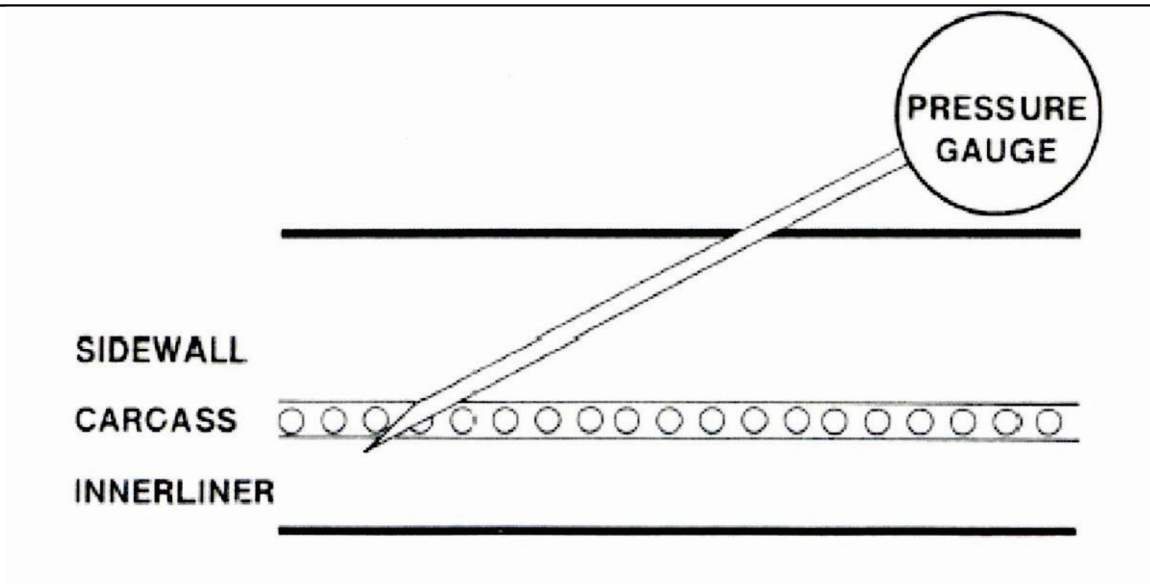
GFE: New tires, new wheels.

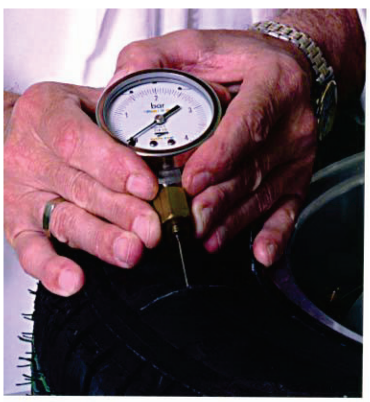
Specifications: A detailed test procedure is available upon request.

General Process:

- Measure tire intracarcass pressure at 21°C ±3°C (69.8°F ±5°F) with the following method:
    - Equilibrate tires at 21°C
    - Inflate tires to 2.2 bar and maintain pressure with a pressure manifold system
    - Fill hypodermic needle / pressure gauge with water
    - Insert 4 needles into tire sidewall until carcass cords are touched
    - Rest gauge on inclined support tray
    - Monitor ICP
    - Equilibrium is usually reached within 20 days


ATMOSPHERIC PRESSURE
CARCASS
INNERLINER
REST OF TYRE
(SIDEWALL,TREAD,ETC)
INTERNAL PRESSURE P
AIRFLOW





Data Units: °C, bar

Deliverables: Tire intracarcass pressure, in bar, measured per specifications and submitted in an MS Excel format.
**Pictures courtesy of Exxon-Mobil Chemical (EMC) Company. Full test procedure available from EMC.*

Item 20 – Peel Strength per ASTM D 413-98(2002)∊1 23°C (73.4°F)

Pricing: Price is per test. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

Predecessors: Inspection and shearography for used tires.

GFE: New and used tires.

Replicates: Four repeated measurements per tire sample region

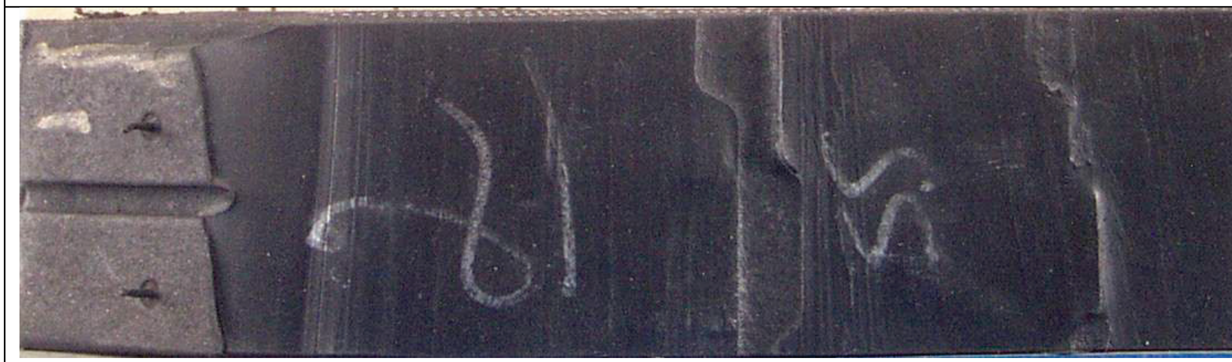
Sample Regions: Skim & wedge rubber, 0° & 180°.

Specifications: General - Section tire into appropriate test specimens. Measure peel strength of the tire skim rubber between the steel belts as well as the wedge rubber (belt edge) per ASTM D 413-98(2002)$^{\epsilon 1}$ *Standard Test Methods for Rubber Property—Adhesion to Flexible Substrate*.

For used tires, the contractor shall use previously conducted tire shearography images to locate two regions approximately 180 degrees from one another around the circumference of the tire that are not damaged or separated. For new tires, the first region shall be located at the DOT number (0°), with the second region located 180 degrees around the circumference of the tire (180°). Prepare the sample according to the following guidelines:

- For each of the two chosen tire regions, cut one 63.5 mm (2½") wide radial section bead to bead. The section should resemble the following photo:

- Trim off the sidewall portion of each section approximately 31.75 mm (1¼") down the sidewall from the end of the first (bottom) belt. Discard sidewall portions.
- Buff the tread flat on each radial tread sections.
- Bisect each radial tread section into two 31.75 mm (1¼") wide radial strips.
- Bisect the radial strips, circumferential to the tire at the centerline of the tread, to produce two test specimens (1-SS and 1-OSS) per strip. This will produce a total of eight 31.75 mm (1¼") wide test specimens in total (4-SS and 4-OSS) from the two original radial sections.
- Mark each sample for identification e.g. SS0a, SS0b, OSS0a, OSS0b, SS180a...
- Buff the edges of all samples until smooth, paying close attention to minimizing heat generation.
- Cut each sample with a razor knife from the skim end of the test strip, midway between the belts, for a length of 25.4 mm (1") to facilitate gripping the ends in the stress/strain tester jaws.
- Score the sides of each specimen at a point midway between the belts to a depth of 3.175 mm (1/8"). The scoring will extend from the end of the gripping surface to the end of belt #2 in the shoulder area, providing a final 25.4 mm (1") wide peel section. The final section should look like this:



- Testing is conducted per ASTM D 413 “Type A 180° peel.” Testing is restricted to the Section 3.1.2 *Machine Method*, in which the force required to cause separation between adhered surfaces is applied by means of a tension machine. A mark shall be made on the side of the sample where the wedge material begins and a separate result shall be recorded for the wedge region to the end of the belts. The peel test shall be performed at 0.8 mm/s (2 in/min). Test shall be conducted under the following environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5 %, with the specimens being conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test. Peel strength values shall be submitted in units of both N/m and lbf/in.

Data Units: N/m, lbf/in, °C, %

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:
- SS, 4 average & 4 peak peel strengths for skim and wedge compounds in both N/m & lbf/in units.
- OSS, 4 average & 4 peak peel strengths for skim and wedge compounds in both N/m & lbf/in units.
- Temperature and relative humidity readings for each test.

Item 21 - Peel Strength per ASTM D 413-98(2002)∈1, 100°C (212°F)

Peel strength per Item 20 specifications at 100°C (212°F) instead of 23°C (73.4°F).

Item 22 - Variable Speed Peel Strength

Peel strength per Item 20 specifications using variable peel speeds ranging from 0.00001 m / sec to 5 m /sec. Sample preparation and dimensions may be modified to accommodate test procedure.

Item 23 - Total Crosslink Density

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

GFE: New and used tires.

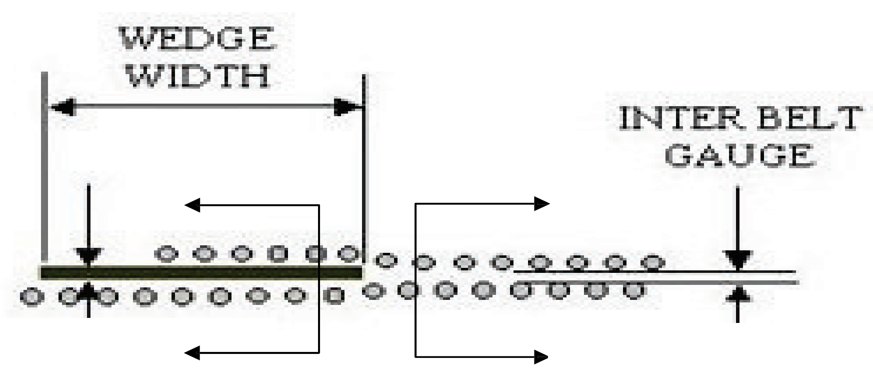
Labor Costs: Included in the price.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS, Wedge Rubber 0° SS, Wedge Rubber 180° OSS, Tread Rubber 0° SS, Tread Rubber 180° OSS.

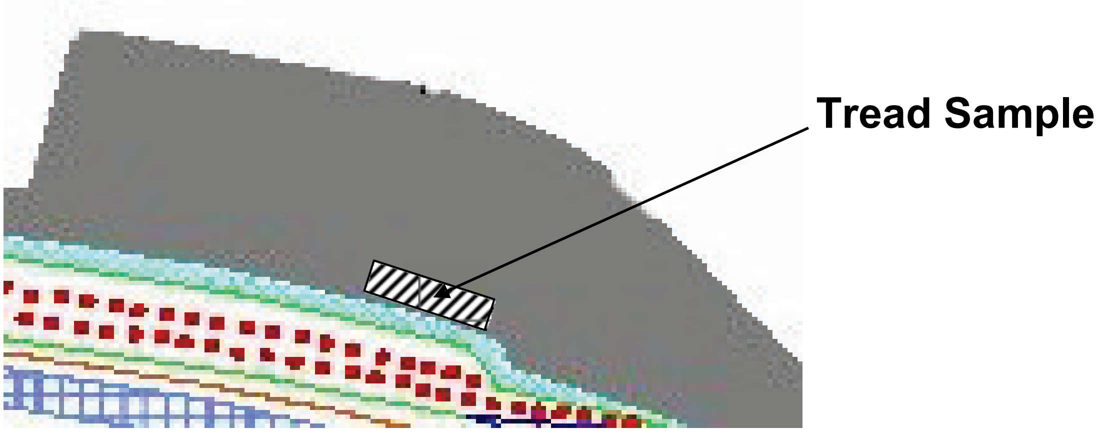
Specifications: General - Section tire into appropriate test specimens. Measure total crosslink density in six regions, with five repeated measurements on a sample taken from each region. Remove a large enough sample from each section to accommodate the skim, wedge, and tread rubber excisions, accounting for replicates.

- Skim rubber sample: Locate the region between the 2 steel belts, inboard of the end of the 2nd (top) belt. Come inboard to the 2nd cable of the top belt beyond the wedge ending (beyond the flare) and excise the rubber sample.
- Wedge rubber sample: Samples shall be taken from the belt wedge rubber outboard of the flare in the inter-belt rubber to a thicker gauge (under the edge of the 2nd (top) belt). Care should be taken to excise only the belt wedge compound, as tires occasionally have a belt wedge compound that differs from the skim coat.



Wedge Sample **Skim Sample***

- Tread rubber sample: Samples shall be taken from the tread rubber above the edge of the 2nd (top) belt at the innermost depth of the tread rubber compound (above any skim, under-tread, or cap-ply layers). The sample is excised from under a tread block (not in a void or groove).



Measure total crosslink density on samples swollen to equilibrium in toluene after 24 hours. If Coefficient of variation (COV) between 5 replicates is above 4%, remove outlier and recalculate. Repeat if COV is above 4% for 4 samples. If COV is above 4% for 3 samples, repeat test. Results shall be reported as mol/cc^3 using the Flory-Rehner equation.

Data Units: mol/cc^3

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:
- 5 raw & 1 average (adjusted per COV) total crosslink density

**Picture sampled from Summary Root Cause Analysis Bridgestone/Firestone, Inc., Slide 1*

Item 24 – Crosslink Density Distribution (S1-S8)

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS, Wedge Rubber 0° SS, Wedge Rubber 180° OSS, Tread Rubber 0° SS, Tread Rubber 180° OSS.

Specifications: General - Section tire into appropriate test specimens (see Item 23 for sample region description). Measure crosslink density distribution (S1-S8) in six regions, with five repeated measurements on a sample taken from each region.

Test Procedure Adapted From Akron Rubber Development Lab (ARDL) Procedure:

1. SCOPE: This work instruction covers the necessary steps to measure the crosslink density and percent S1-S8 of a vulcanized rubber specimen.

2. PURPOSE: This procedure describes the testing procedure and the calculation procedure to measure the crosslink density of a sample.

3. EQUIPMENT: Chemical solvents A (37.6 ml of propane-2-thio and 39.5 ml of piperidine to toluene diluted with 1 liter of toluene.), solvent B (118 ml of hexane-1-thio to piperidine and diluted with 1 liter of piperidine), Toluene, precision scale.

4. PROCEDURE:
   4.1. A new sample is treated with the Toluene solution to produce a swollen rubber network.
   4.2. The density of unswollen and swollen network yields q, the swelling ratio.
   4.3. Using the correct interaction parameter *x1* and the molar volume of the solvent *V1*, we go to step 4.4
   4.4. Use the Flory-Rehner's equation Ve = (0.5-*x1*) / (*V1*) / ($q^{5/3}$) to calculate total crosslink density.
   4.5. Repeat procedure 4.1 through 4.4 on a new sample using a solution of solvent A. The solvent A solution will cleave only the poly-sulfidics from the rubber sample to yield the crosslink density of the remaining mono and di-sulfidics.
   4.6. Repeat procedure 4.1 through 4.4 on a new sample using a solution of solvent B. The solvent B solution will cleave both the poly-sulfidics and di-sulfidics to yield the crosslink density of the remaining mono-sulfidics.
   4.7. Calculate the % poly-sulfidics, di-sulphidics, and mono-sulpfidics based on the numbers obtained from 4.1 through 4.3

If COV between 5 replicates is above 4%, remove outlier and recalculate. Repeat if COV is above 4% for 4 samples. If COV is above 4% for 3 samples, repeat test.

Data Units: mol/cc^3, %

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:
- 5 raw & 1 average swelling ratio (q)
- 5 raw & 1 average total crosslink density with Tolune
- 5 raw & 1 average S1 crosslink density
- 5 raw & 1 average S2 crosslink density
- 5 raw & 1 average S8 crosslink density
- 5 raw & 1 average % Monosulfidic crosslinks
- 5 raw & 1 average % Disulfidic crosslinks
- 5 raw & 1 average % Polysulfidic crosslinks
- 5 raw & 1 average total crosslink density from sum of S1-S8 components

Item 25 – Fixed Oxygen by Weight

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS, Wedge Rubber 0° SS, Wedge Rubber 180° OSS, Tread Rubber 0° SS, Tread Rubber 180° OSS.

Specifications: General - Section tire into appropriate test specimens. Measure percent oxygen by weight of skim, wedge, and tread rubber (see Item 23 for sample region descriptions). Once the sample is removed, care shall be taken that sample is placed in an inert atmosphere so that further oxidation does not occur. If COV between 5 replicates is above 4%, remove outlier and recalculate. Repeat if COV is above 4% for 4 samples. If COV is above 4% for 3 samples, repeat test. Results shall be reported as percent oxygen by weight.

Data Units: $\%O_2$ by weight

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:
- 5 raw & 1 average percent oxygen by weight

Item 26 – Tensile Properties per ASTM D 412-98a(2002)∈1

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, data report, and sample & data storage.

Labor Costs: Included in the price.

Predecessors: Inspection and shearography for used tires.

GFE: New and used tires.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS, Wedge Rubber 0° SS, Wedge Rubber 180° OSS

Specifications: General - Section tire into appropriate test specimens. Perform tensile & elongation measurements of the skim and wedge material per D 412-98a(2002)$^{\epsilon 1}$ *Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension*.

Under D412, certain test parameters require additional specifications:

- Samples of at least 100 mm (4 in) in length and 25 mm (1 in) in width shall be removed from the skim and wedge rubber (see Item 23 for sample region details) for each test. Samples shall be buffed to uniform thickness of 0.5 to 1.0 mm (0.02 to 0.04"). Care must be taken to minimize heat generation during buffing.
- Since the test specimen sizes available in D 412 are too large for tire sample purposes, specimens shall be die-cut using an ASTM D 638-02a Type V dumbbell die (see following photo):

- Test shall be conducted per D 412-98a(2002)$^{\epsilon 1}$, Section 9.1 environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5%, with the specimens being at conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test.
- The rate of jaw separation is 500 ± 50 mm/min (20 ± 2 in/min).
- The 5 samples from each area shall be evaluated and 1 or 2 outliers may be removed. If less than 3 good samples remain, test must be repeated.
- Record the modulus in MPa @ 50%, 100%, 200%, 300%; ultimate elongation and tensile strength, and provide the raw data curves.

Data Units: MPa, mol/cc^3, °C, %

Deliverables:
For each tensile test, provide the raw data curves. From the measurements, provide the following data points for each sample region in an MS Excel format:

- 5 raw & 1 average modulus at 50%
- 5 raw & 1 average modulus at 100%
- 5 raw & 1 average modulus at 200%
- 5 raw & 1 average modulus at 300%
- 5 raw & 1 average tensile strength
- 5 raw & 1 average ultimate elongation
- Temperature and relative humidity for each test

Item 27 – Shore A Hardness per ASTM D2240-02b

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Innerliner 0° SS, Innerliner 180° SS, wedge rubber 0° SS, wedge rubber 180° SS.

Specifications: Section tire into appropriate test specimens. Measure tire innerliner hardness at the centerline of the crown. Wedge rubber samples shall be per Item 23 sample descriptions. Measure innerliner and wedge rubber hardness using the Shore A scale per D2240-02b *Standard Test Method for Rubber Property—Durometer Hardness.* Measurements shall be conducted per Section 9.1.8 or 9.2.5 (5 determinations of hardness at least 6.0mm (0.24 in) apart). Test shall be conducted under the following environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5 %, with the specimens being conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test. Report raw and average values.

Data Units: Shore A Hardness #.

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:

- 5 raw & 1 average shore hardness numbers

Item 28 – Micro Hardness per ASTM 1415-88 (Reapproved 1999)

Pricing: Price is per tire. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Five repeated measurements in each sample region.

Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS

Specifications: Section tire into appropriate test specimens per Item 23. Measure skim rubber micro hardness per 1415-88 (99) *Standard Test Method for Rubber Property—International Hardness.* Test specimens shall be per Section 6.3 *Micro Tester.* Test shall be conducted under the following environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5 %, with the specimens being conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test. Report raw and average values.

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:

- 5 raw & 1 average micro hardness numbers

Item 29 – Indentation Modulus Profiling

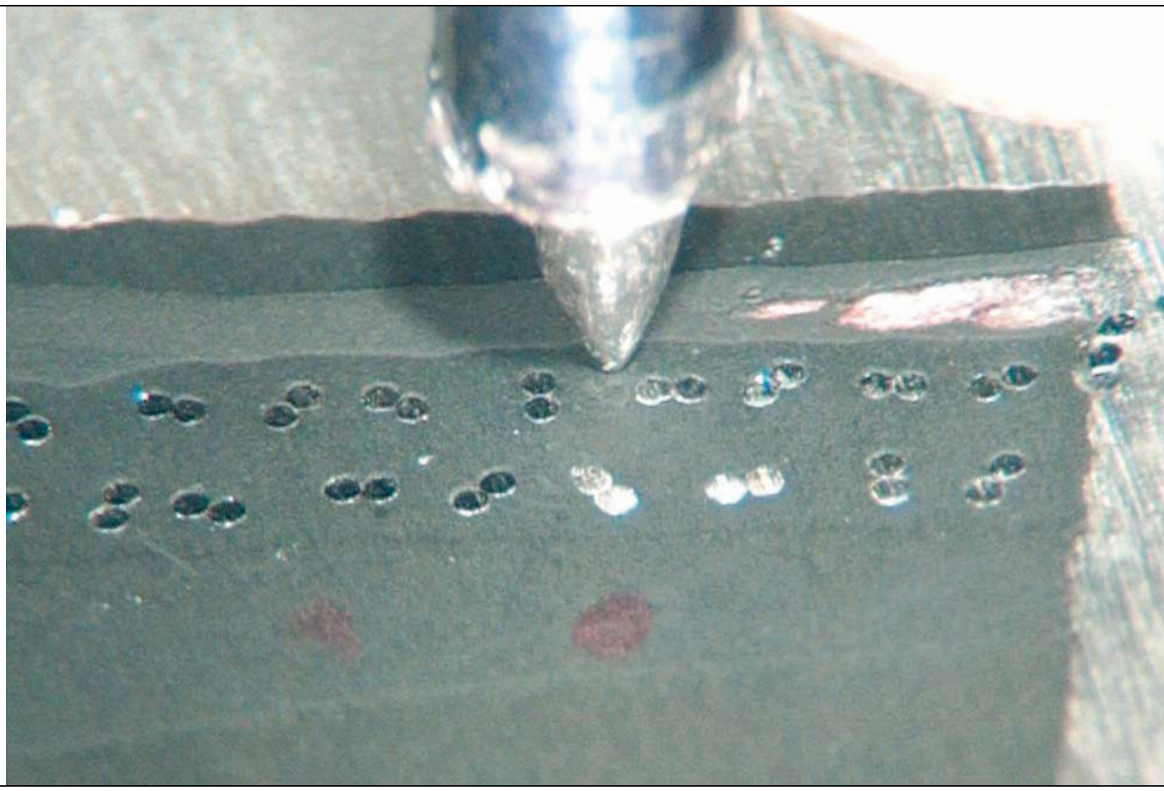
Pricing: Price is per tire per sample region, either shoulder or bead region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: One complete radial scan per sample region.

Sample Regions: Specified by COTR in task order.



Specifications: Section tire into appropriate test specimens. A nano-indenter in accordance with the methodology contained in RC&T, 74, No.3, pp. 428ff (2001) shall be used to acquire indentation modulus measurements of the rubber components in 0.1mm increments from interior to exterior surfaces (innerliner to tread surface of the sample). Test specimens shall be prepared with minimal heat input in an embedment medium suitable for grinding/polishing in order to obtain a flat surface for measurement. A single radial scan shall performed on each specimen. Test shall be conducted under the following environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5 %, with the specimens being conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test. Data shall be submitted to the Government in a MS Excel format.

Deliverables: A chart containing the modulus profile trace with accompanying data in a MS Excel format.

Item 30 ‑ Roadwheel Time ≤ 120 km/h (75 mph), per ASTM F 551-89

Pricing: Quotes for road wheel testing may be quoted in two forms: hourly use/yearly rental

- Per tire-hour of road wheel time. Fractional hours may be prorated.
- Per year rental of a road wheel. The per year quote shall be on the basis that the road wheel is available for NHTSA testing at least 80 percent of time (i.e., at least 7,000 hours per year). Price reductions, in the event that the 80 percent availability goal cannot be met should be clearly indicated in the yearly rental quotation.

All prices quoted for road wheel tests shall include all costs of operating and maintaining the road wheel. Tire mounting and inflation with air or enriched inflation mixtures will be charged separately.

Labor Costs: Labor for general road wheel testing is included in the hourly price. The yearly rental price should indicate if labor will be charged separately.

GFE: New and used tires, new wheels.

Specifications: General – Test tires on a tire dynamometer per ASTM F 551-89(2000) *Standard Practice for Using a 67.23-in. (1.707-m) Diameter Laboratory Test Roadwheel in Testing Tires*.

All tests shall be performed according to standard commercial tire road wheel testing practice. The following test parameters will be provided to the contractor when tests are ordered:

- Mark the direction of rotation on the test tires
- Tire Inflation Pressure – an initial cold inflation pressure will be specified by the COTR in writing for each test. Dynamic pressure regulation is not required.
- Tire Load – will be specified by the COTR in writing for each test, but will not exceed the 200% of passenger car or LT tire rated loads (no medium or heavy truck tires).
- Test Speed – will be specified by the COTR in writing for each test, but will not exceed 120 km/h (75 mph) for any test.
- Duration – will be specified by the COTR in writing for each test.

Deliverables: Tire road wheel testing per task order specifications.

Item 31 ‑ Roadwheel Time ≤ 120 km/h (75 mph), Dynamic Press. Maintenance

Same specifications as Item 30, but with dynamic tire pressure maintenance (rotary air union).

Item 32 ‑ Oven Aging, Contractor's Ovens

Pricing: Quotes for oven aging time shall be quoted in two forms: hourly use/yearly rental

- Per tire-hour of oven aging. Fractional hours may be prorated.
- Per year rental of an oven . The per year quote shall be on the basis that the oven is available

for NHTSA use at least 80 percent of time (i.e., at least 7,000 hours per year). Price reductions, in the event that the 80 percent availability goal cannot be met should be clearly indicated in the yearly rental quotation.

Prices quoted for tire oven aging shall include all costs of operating and maintaining the oven.

Tire mounting and inflation with air or enriched inflation mixtures will be charged separately.

Labor Costs: Labor for general oven aging is included in the price. The yearly rental price should indicate if labor will be charged separately.

GFE: New and used tires, new wheels.

Specifications: General – Tire oven aging under task order specified conditions.
- Oven Temperature - 70°C ± 2°C (158°F ± 3.6°F) unless otherwise specified in the task order.
- Tire Inflation Pressure – an initial cold inflation pressure will be specified by the COTR in writing for each test. Dynamic pressure regulation is not required.
- Duration – will be specified by the COTR in writing for each test.

Deliverables: Tire oven aging per task order specifications.

Item 33 - Oven Aging, Government's Ovens

Pricing: Quotes for oven aging time shall be quoted in two forms: hourly use/year use

- Per tire-hour of oven aging. Fractional hours may be prorated.
- Per year operation of aging oven.

All prices quoted for oven aging shall include all costs of operating ovens, including energy costs, in a safe in-door test space.

Tire mounting and inflation with air or enriched inflation mixtures will be charged separately.

GFE (ovens) will be shipped to and from the contractor's location, and installed and uninstalled by VRTC at no cost to the contractor. The VRTC will be responsible for the preventative maintenance, calibration, and repair of the ovens.

The contractor will be responsible for the correct operation and day-to-day maintenance of the GFE while in its possession. The government will be responsible for shipping the GFE back to VRTC at no cost to the contractor.

Labor Costs: Labor for general oven aging is included in the price. The yearly operation price should indicate if any labor will be charged separately.

GFE: New and used tires, new wheels, ovens.

Specifications: General – Tire oven aging under task order specified conditions.

- Oven Details (total of two ovens available):
  - Thermcraft Oven:
    - Model 48-48-60-REC-0V, Serial # 83439
    - 480 V, 3 phase, 24000 watts, 29 amps (oven)
    - Controller – Model 1Z3-480-50, Serial # 84080-C
    - Interior dimensions – 4' wide, 4' deep, 5' tall (80 ft^3)
    - Exterior dimensions – 7' wide, 5'3"deep, 8'4" tall
  - GS Blue M Electric Oven:
    - Model 806 Batch Oven, Serial #15X-402-90
    - 480V, 3 phase, 52 amps (oven), 60 Hz
    - Interior dimensions – 4' wide, 4' deep, 5' tall (80 ft^3)
    - Exterior dimensions – 7'9" wide, 5'2" deep, 7'10" tall

- Oven Test Temperature - 70°C ± 2°C (158°F ± 3.6°F) unless otherwise specified in the task order.
- Tire Inflation Pressure – an initial cold inflation pressure will be specified by the COTR in writing for each test. Dynamic pressure regulation is not required.
- Duration – will be specified by the COTR in writing for each test.

Deliverables: Tire oven use per task order specifications.

Item 34 - Analysis of Innerliner Compound by FTIR

Pricing: Price is per analysis. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.
Labor Costs: Included in the price.

GFE: New tires.

Replicates: One measurement per tire.

Sample Regions: Innerliner 0° SS.

Specifications: Section tire into appropriate test specimens. Identify innerliner compound polymer composition by pyrolysis Fourier Transform InfraRed (FTIR) technique in accordance with ASTM D 367700, *Standard Test Methods for Rubber – Identification by Infrared Spectrophotometry.* The vendor shall estimate the relative percentage of the polymer composition based on control curves run at vendor's expense.

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:

- Innerliner polymer type
- % Halo-butyl rubber
- % Natural rubber
- % other rubber polymer (specify polymer)

The vendor shall retain and shall archive copies of sample spectra and reference spectra in pdf format and shall provide them to the Government upon request.

Item 35 - Inflation Gas Oxygen Concentration

Pricing: Price is per measurement.

Labor Costs: Included in the price.

GFE: New and used tires, new wheels.

Replicates: One measurement per tire.

Sample Regions: Tire inflation gas.

Specifications: Non-dynamic, pre or post-test; measure the percent oxygen concentration in the tire's inflation gas mixture with a gas analyzer accurate to within ±0.1%.

Data Units: %

Deliverables: Percent oxygen concentration in tire inflation mixture in an MS Excel format.

Item 36 - Tire Aging Simulations with Finite Element Software

Pricing: Price is per simulation per region.

Labor Costs: Included in the price.

Sample Regions: Specified by COTR in the task order.

Specifications: Use tire component temperatures, gauges (see **Item 14 - Microscopy**), oxygen partial pressures (inside tire and outside tire), effect of partial pressure on oxygen consumption rate, density, diffusion limited oxygen rates, and known polymer properties to simulate the effects of different aging tests on the tire by finite element simulation. Simulations should attempt to quantify changes in the modulus, crosslink density, and fixed oxygen for the sample region. Methodology should follow that described in Tire Technology International, Annual Review, 46 (2001). Vendor shall provide diffusion parameters, solubility constants and other necessary measures in order to quantify the simulation constituents. Controls shall be run as necessary in order to obtain FEA convergent models.

Deliverables: A finite element simulation of tire aging per specified conditions. Simulations should quantify oxygen consumption rate with about 0.2 mm resolution performed at specified location along with input parameters. A chart containing the oxygen consumption rate profile with accompanying data in a MS Excel format.

Item 37 - Micro Demattia

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Eight repeated measurements per sample region.

Sample Regions: Specified by COTR in the task order.

Specifications: Prepare the necessary test samples to conduct Demattia rate of crack growth testing. Use a test machine scaled down from the ASTM specifications D813-95(2000) *Standard Test Method for Rubber Deterioration-Crack Growth* and ASTM D430-95(2000) *Standard Test Methods for Rubber Deterioration-Dynamic Fatigue* to give less variation in testing. This is otherwise referred to as "Micro Demattia" testing.

Data Units: Rate of crack growth in SI units.

Deliverables: Eight rates of crack growth and one average rate of crack growth for each region tested.

Item 38 - Torsional Test on Belt Ply

Pricing: Price is test. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

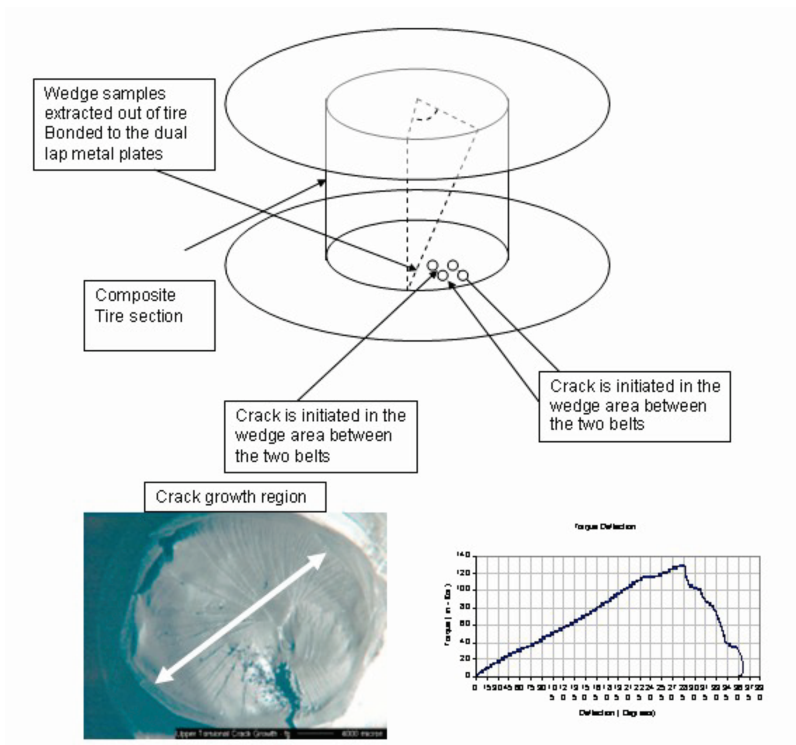
Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Three repeated measurements.

Sample Regions: Wedge region - 0°, 120°, 240°.

Specifications: Prepare the necessary test samples to conduct a torsional belt ply test to failure. The torsional test is conducted on the entire tire composite structure by bonding the 1-inch diameter section, cut through the wedge region, and obtaining the torque versus the deflection curve till the precut crack introduced circumferentially grows to fail the sample through the wedge after propagating through the wedge. In this method the actual torque and its effect on the crack growth in the wedge transmitted though the composite for different tires can be evaluated.



Data Units: N-m, mm.

Deliverables: Three raw and one average set of torsion and deflection curves.

Item 39 – DMA: Dynamic Testing, Micro Dumbbell Samples per ASTM D5992-96(2001)e1

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Three repeated measurements.

Sample Regions: Specified by COTR in task order.

Specifications: Dynamic material properties are evaluated at fixed strains % of 5, 10, 15, and 20% in tension for 4 frequencies of 5, 10, 15, 20 Hz and 4 temperatures of 70, 80, 90, and 100°C.

Data Units: Tan delta, MPa

Deliverables: Three raw and one average tan delta and dynamic modulus at 5, 10, 15, and 20% in tension for 4 frequencies of 5, 10, 15, 20 Hz and 4 temperatures of 70, 80, 90, and 100°C for a sample region.

Item 40 – Two-ply Laminate Fatigue Test

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Predecessors: Inspection and shearography for used tires.

Replicates: Three repeated measurements per region.

Sample Regions: Specified by COTR in task order.

Specifications: Section tire into appropriate test specimens. Measure fatigue life in number of cycles to failure by cycling under load control at specified minimum and maximum load at a specified temperature. For used tires, the contractor shall use previously conducted tire shearography images to locate two regions approximately 120 degrees from one another around the circumference of the tire that are not damaged or separated. For new tires, the first region shall be located at the DOT number (0°), with the second region located 120 degrees around the circumference of the tire (120°). The third region located 240 degrees around the circumference of the tire (240º). Prepare the sample according to the following guidelines:

- Cut one (10") wide circumferential section from each location.
- Cut (2½") wide circumferential section from the sample region.
- Buff tread flat on each section.
- Buff edge of sample until smooth, minimizing heat generation.
- Score the sides of each specimen at a point midway between the belts to a depth of 3.175 mm (1/8"). The scoring will extend from the end of the gripping surface to the other end of the gripping surface.
- Place in test equipment with 6" separation between jaws.
- Testing is conducted by fatigue to failure under cyclic deformation MTS servo-hydraulic instrument. Three loading conditions will be used, designed to reflect tire unload and load conditions (see Interlaminar Shear Test)
- Test shall be conducted under the following environmental conditions: 23 ± 2°C (73.4 ± 3.6°F) and a relative humidity of 50 ±5 %, with the specimens being conditioned at these conditions for a minimum of 24 hrs. The temperature and relative humidity shall be recorded for each test.

Data Units: Number of cycles to failure as a function of load expressed in Mpa.

Deliverables: From the measurements, provide the following data for a sample region in an MS Excel format:
- Number of cycles to failure as a function of loading conditions expressed in Mpa.
- Temperature and relative humidity reading per test.

Item 41 – Bulk Modulus of Belt Edge: Hopkinson Bar Testing

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Five repeated measurements per region.

Sample Regions: Specified by COTR in task order.

Specifications: Measure bulk modulus versus strain rate with the Hopkinson Bar test of 2-3 mm thick and ½" diameter buttons excised from the specified sample region.

Data Units: GPa

Deliverables: Table of bulk modulus and strain rates for a sample region.

Item 42 – Pure Shear Crack Growth Test

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Three repeated measurements per region.

Sample Regions: Specified by COTR in task order.

Specifications: Skim region extracted and buffed on both sides to give parallel and uniform surface. Dynamic cycling is performed and the coordinates of the tip of the crack continuously monitored.

Data Units: Rate of crack growth, SI units.

Deliverables: Three raw and one average rate of dynamic crack growth for a sample region.

Item 43 – Thermo-Gravimetric Analysis (TGA) for % Carbon Black & % Ash

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Three repeated measurements per sample region.

Sample Region(s): Specified by COTR in task order.

Specifications: Excise test samples and perform a Thermo-Gravimetric Analysis (TGA) to determine % carbon black and % ash content per ASTM E1131-98 *Standard Test Method for Compositional Analysis by Thermogravimetry*.

Data Units: %

Deliverables: Three raw and one average % carbon black & % ash numbers for a sample region.

Item 44 – Interlaminar Shear Test

Pricing: Price is per sample region. Price includes all costs associated with the service, including test sample preparation, repeated measurements, data report, and sample & data storage.

Labor Costs: Included in the price.

GFE: New and used tires.

Replicates: Four repeated measurements per sample region.

Specifications:
- Tire is notched at four locations to expose the rubber between bets at the edge of the belt #2.
- A vertical (radial position) mark is made.
- The tire is inflated. The positions of the marks are measured.
- The tire is loaded and the position of the notch is measured under static (unloaded) and loaded conditions.
- Calculate strain energy density at unload and strain energy density at maximum rated load. Calculate R-ratio, ratio of minimum strain (unloaded conditions) to maximum strain (at maximum rated load). To calculate strain energy density and R-ratio, produce a stress/strain curve (generated by tensile test).
- Note – same testing can be used for an underinflated tire, if specified.

Data Units: MPa

Deliverables: From the measurements, provide the following data points for each sample region in an MS Excel format:
- Interlaminar shear strain, strain energy density, and R-ratio as a function of load expressed in Mpa
- Temperature and relative humidity reading per test

Item 45 – Labor, Program Manager

Pricing: Price is per hour. Provide both regular and overtime rates.

Specifications: Loaded hourly regular and overtime labor rates for a program manager.

Deliverables: One hour of program manager labor.

Item 46 – Labor, Project Engineer

Pricing: Price is per hour. Provide both regular and overtime rates.

Specifications: Loaded hourly regular and overtime labor rates for a project engineer.

Deliverables: One hour of project engineer labor.

Item 47 – Labor, Instrumentation Engineer / Technician

Pricing: Price is per hour. Provide both regular and overtime rates.

Specifications: Loaded hourly regular and overtime labor rates for an instrumentation engineer/technician.

Deliverables: One hour of instrumentation engineer/technician labor.

Item 48 - Labor, Test Equipment Operator

Pricing: Price is per hour. Provide both regular and overtime rates.

Specifications: Loaded hourly regular and overtime labor rates for a test equipment operator.

Deliverables: One hour of test equipment operator labor.

Item 49 - Labor, Administrative Assistant

Pricing: Price is per hour. Provide both regular and overtime rates.

Specifications: Loaded hourly regular and overtime labor rates for an administrative assistant.

Deliverables: One hour of administrative assistant labor.

Item 50 – Metal Valve Stem Surcharge

Item 51 – High Speed Surcharge on Item 30

Item 52 – Bundle of Items 14, 20, 23, 25, 26, 27 and 28

Item 53 – Bundle of Items 17 and 18

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