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NHTSA Tire Aging Test Development Project Phase 2 — Evaluation of Laboratory Tire Aging Methods

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Author(s)	Performing Organization Report No.
Larry R. Evans, Transportation Research Center, Inc., & James D. MacIsaac Jr., National Highway Traffic Safety Administration	

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Abstract

As a result of the TREAD Act of 2000, NHTSA initiated an effort to develop a laboratory-based accelerated service life test for light vehicle tires (often referred to as a "tire aging test"). It is believed that if such a test method was successful, then light vehicle tires could eventually be required to meet standards that would make them more resistant to operational degradation and possibly reduce their failure rate during normal highway service. The first phase of test development examined how six tire models changed during service by measuring their roadwheel performance levels and material properties after varying lengths of service and accumulated mileages in Phoenix, Arizona. This report documents the second phase of test development in which new tires of the models collected from service in Phoenix were subjected to one of three laboratory aging test methods and compared to the results of the service-aged tires. Two of the laboratory tire aging methods evaluated were combined roadwheel aging and durability tests that were fully developed when provided by tire manufacturers for evaluation. The third was a tire oven aging method undergoing development by a vehicle manufacturer that would significantly accelerate the degradation of the tire materials prior to a structural evaluation, such as a post-oven roadwheel durability test.

Canonical correlation showed that the properties of the tire components tended to change in the same direction with both increased time in service in Phoenix and increased time in each of the laboratory aging methods. All of these changes are consistent with the proposed mechanism of thermo-oxidative aging. In general, the longest roadwheel test times showed the same level of change in properties as tires with 1 to 3 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 3 to over 6 years of service in Phoenix. However, oven aging alone could not reproduce the modulus properties in the belt-packages of certain tire models without a pre-oven roadwheel-break-in of the tire. Also, after 6 weeks of oven aging, the oxygen-enriched tire inflation gas had decreased from an initial average of approximately 45% O_2 to approximately 35% O_2, resulting in the onset of oxygen-deprived aging (*i.e.*, not representative of service aging). Therefore, future oven aging test development will include periodic venting and re-inflation of the tires with fresh oxygen-enriched gas.

Key Words	Distribution Statement
Tire, tire aging, tire safety, Phoenix, FMVSS No. 139, TREAD Act, accelerated service life, tire durability, roadwheel oven-aging	Document is available to the public from the National Technical Information Service www.ntis.gov

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EXECUTIVE SUMMARY

During the September 2000 Congressional hearings regarding the Firestone tire failures on Ford light trucks and SUVs, members of Congress expressed concerns that the then-current Federal Motor Vehicle Safety Standards (FMVSS) did not evaluate how well tires perform after being subjected to environmental variables, such as heat, over time, which can accelerate structural degradation. The National Highway Traffic Safety Administration was asked to consider the feasibility of requiring a "tire aging test" (i.e. accelerated service life test for tires) that could evaluate the risk of failure at a period later in the life of a tire than the current regulation, which only evaluates new tires. 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. The TREAD Act contained provisions mandating NHTSA to "revise and update" the passenger car and light-truck tire safety standards; however, the legislation did not mandate specific requirements.

In response to the TREAD Act, the agency examined the effectiveness of the current tire safety standards and embarked on an ambitious tire test development program that culminated in the new Federal Motor Vehicle Safety Standard (FMVSS) No. 139, "*New pneumatic radial tires for light vehicles.*" Though three methods of evaluating long-term tire durability were evaluated during the development of FMVSS No. 139, data from agency as well as industry evaluations of the three methods demonstrated the need for additional test development. Of primary need was a better understanding of service-related tire degradation that could serve as the "real-world" baseline for the development of a laboratory-based accelerated service life test (herein referred to as "tire aging test"). These needs were the basis of the NHTSA Tire Aging Test Development Project, which started in late 2002.

The first goal of the NHTSA Tire Aging Test Development Project was to develop better understanding of service-related tire degradation over time. Phase 1 of project would study how tires change during actual service as measured by changes in their roadwheel performance levels and quantitative material properties when compared to new versions of each tire. Since the rate of degradation of tire rubber components increases with temperature, NHTSA expected that the "worst case" tires in service in the United States would be found in the relatively hotter southern States. It was thought that designing a tire-aging test to simulate service in a severe environment that has high relative tire failure rates would offer the best margin of safety nationwide. Per this approach, Phoenix, Arizona, was selected as the location for the collection of on-vehicle tires for analysis. The results of this research indicated that there are two mechanisms operating to produce changes in tire properties, particularly in the critical belt-edge region. First is degradation of the rubber compound and material interfaces due to the effects of heat and reaction with oxygen (thermo-oxidative aging). The second is the effect of cyclic fatigue during tire deformation, which can initiate and propagate cracks and separations.

Phase 2 of the project focused on developing an accelerated, laboratory-based tire test that simulates real world tire aging and evaluates the remaining structural durability of the aged tires. The six tire models that had previously been evaluated after long-term service (aging) on vehicles in Phoenix were evaluated. Three methods of laboratory aging these tires were evaluated. Two were combined tire aging and durability tests conducted on the 1.707-m indoor roadwheel: (1) The Long-Term Durability Endurance Test, proposed by Michelin. Tires were inflated with a mixture of 50% nitrogen and 50% oxygen and run on a 1.707m roadwheel for up to 500 hours, and (2) The Passenger Endurance Test proposed by Continental, in which the tire is run on the roadwheel under proprietary conditions for up to 240 hours. The third method was based on tire research by Ford, who recommended that the agency use a method in which the tire is inflated using the 50% nitrogen and 50% oxygen mixture and heated in an oven for a period of time. This oven-aging step was to accelerate the aging process by speeding up chemical reactions, and thus material property changes. The oven-aged tire may then be studied for material property changes, or physically evaluated, such as run on a roadwheel to determine any change in structural durability.

The material and chemical properties of the components of new tires, and tires subjected to one of the three candidate aging methods, were evaluated. 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 belts in the tire tread and belt area (shoulder area) were studied in the most detail. Canonical correlation of the properties showed that the properties of the components tended to change in the same direction versus increased time of service in Phoenix or increased time of laboratory aging on each candidate method. That is, the hardness, modulus, cross-link density, and oxygen content tended to increase; and the tensile, ultimate elongation, peel adhesion, and flex properties tended to decrease over time. All of these changes are consistent with the mechanism of thermo-oxidative aging, which is well documented in scientific literature.

The data was subsequently analyzed using one-way analysis of variance (ANOVA) and linear regression models to determine what variables had the most significant effect on the change in properties. Nearly all of the ANOVA models had a probability > F value of less than 0.05, indicating that overall changes in properties taking place during service in Phoenix, or during laboratory aging, were statistically significant. Analysis by linear regression was unable to provide predictive models with R^2 values above 0.95 in most cases because of the scatter in the data. Coefficient of variation for nearly all the test results ranged from 10 percent to over 25 percent.

The changes in properties of indentation modulus, ultimate elongation, modulus at 100 percent elongation, and peel adhesion in the wire skim-coat and wedge area of the tires that were laboratory aged were compared to changes in these properties for the tires taken from service in Phoenix. The changes in the properties 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 in properties as tires with 1 to 3 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 3 to over 6 years of service in Phoenix. Oven aging at 3 weeks produced very little change, and had the unexpected result that aging for 3 weeks at 60°C produced a greater change in properties than did aging 3 weeks at 70°C. Oven aging for 6 weeks at 60° to 70°C, or 12 weeks at 55°C, produced changes in properties similar to those found after roadwheel testing, which is approximately equivalent to 1 to 3 years of service in Phoenix. For the type E, H and L tires, the hardness tended to decrease during roadwheel testing or service in Phoenix, but to increase 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, but harden with increased temperature in the absence of flexing. The mechanical energy of a break-in cycle softened these compounds, thus replicating field-like characteristics, and will be incorporated into subsequent test development efforts.

The slope of the plot of log(ultimate elongation) versus log(modulus at 100% extension) has been shown to correspond to the mechanistic type of aging of rubber compounds. 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 of 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. The oxygen content of the fill gas in tires after 6 weeks of oven aging had decreased from an initial average of approximately 45% O_2 to approximately 35% O_2 at the end of the test. Based on these data, subsequent testing will include venting the fill gas and re-inflating with fresh 50%N/50%O gas.

Selected tires were tested after oven aging 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, at the elevated terminal loads, 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. Roadwheel testing for tire models E and H appears to be most sensitive to oven aging times, 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 the maximum rated load for the tires. 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 the maximum rated load for the tires.

The 24-hour roadwheel break-in prior to oven aging tended to decrease the roadwheel running time in the test after aging. 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 that failed 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, and material property data, the severity of the break-in cycle will be reduced from the 24 hours at 120 km/h (75 mph) used in these experiments.

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 do not evaluate how well tires perform when significantly underinflated or after being subjected to environmental variables, such as heat, which accelerate aging.[1] As a result of the committee's actions, the TREAD Act 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 (however, the legislation did not mandate specific test requirements). During the consideration and enactment of the TREAD Act, Members of Congress placed particular 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) of tires. With regards to aging, the agency was asked to consider the feasibility of requiring a "tire aging test" (i.e. accelerated service life test for tires) that could evaluate the risk of failure at a period later in the life of a tire than the current regulation, which only evaluates new tires.

In response to the TREAD Act the agency 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 vehicles 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."[2]

NHTSA conducted tire safety research in support of what would become the new FMVSS No. 139, "*New pneumatic radial tires for light vehicles.*"[a] During this effort, agency researchers conducted comprehensive literature reviews and had numerous consultations with industry regarding the long-term effects of service on radial tire durability. The agency concluded that while most tire manufacturers conduct some form of accelerated service life testing on their tires ("tire aging tests"), their approaches varied widely and a single industry-wide recommended practice did not exist. As part of FMVSS No. 139 development

[a] Previously, passenger tires were regulated by FMVSS No. 109 ("Passenger car tires") and light truck tires under the separate FMVSS No. 119 ("Tires for vehicles other than passenger car"). FMVSS No. 119 had less severe test conditions than FMVSS 109 and did not include a high speed or bead unseat test for tires. FMVSS No. 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.

research, the agency conducted an evaluation of multiple laboratory-based accelerated service life tests for tires that were based on either industry submissions or previous agency test experience. In the March 5, 2002, Notice of Proposed Rulemaking (NPRM) section related to tire aging for FMVSS No. 139 (67 FR 10050), the agency proposed three alternative tests that could be used to evaluate a tire's long term durability. These approaches can be characterized as: (1) 24-hours of roadwheel conditioning followed by an adhesion (peel strength) test between the belts; (2) an extended duration road-wheel 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, the agency decided to defer action on the proposal to add an aging test to the new FMVSS No. 139 until further research was conducted. To conduct this further research, the agency initiated its NHTSA Tire Aging Test Development Project in late 2002.

Phase 1 of the NHTSA Tire Aging Test Development Project consisted of the analysis of six different tire models collected from service on privately owned vehicles in the Phoenix metropolitan area during spring 2003. This study was conducted to provide a better understanding of service-related tire degradation and to serve as the "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, the performance of 109 tires retrieved from service in Phoenix of varying age and mileage were compared to 45 new tires of the same type and model in one of two laboratory roadwheel tests. Analysis of these data led the agency to conclude that peel adhesion decreases 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 and the intrinsic interfacial adhesion was unknown. Therefore the agency 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 that has 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 five of the six tire types, the hardness increased with service time. For the sixth 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 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]

Ahagon et al. have shown several mechanistic 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 (i.e., occurs with available oxygen) thermo-oxidative aging, as shown by the slope of the log(extension ratio at break) versus log(modulus at 100%, MPA) plots.[3]

The primary goal of the FMVSS for tires is to specify tire performance requirements for structural integrity and resistance to operational conditions that the government has determined minimally necessary for new tires to possess. What is unknown is whether or not these new tire performance requirements translate into adequate tire durability throughout the entire service life of the tire. NHTSA's Office of Defects Investigation maintains a searchable database of NHTSA safety-related defect and compliance campaigns since 1966 (including tire recalls[b]), which can be queried through a Web interface or downloaded in raw form.[9] Analysis of these records indicates that over 95 percent of the campaigns that involve full-size tires and list the component/region involved cite the "TREAD/BELT" area of the tire as the component. Therefore, tread and belt region durability is the primary consideration for long-term tire safety.

Table 1. NHTSA Safety-Related Tire Defect and Compliance Campaigns Since 1966

Stated Reason for Recall	Number of Tires Recalled	Percent of Recalled Tires
Tread/Belt	58,161,358	95.7%
Sidewall	1,170,380	1.9%
Bead	462,855	0.8%
Other	458,080	0.8%
Unspecified	476,632	0.8%

As tires last longer, concerns over long term safety-related durability increase. In 1973 the average tread life of a passenger car tire in the United States was approximately 24,000 miles. By 2004, this number had risen to approximately 44,700 miles.[10] Using average miles traveled by passenger vehicles of 9,992 miles in 1973 and 12,497 miles in 2004, the average tire service life was calculated to be around 2.4 years in 1973 and 3.6 years in 2004.[11,12] Assuming that approximately 4.5 percent of the average tire's life is spent in actual rolling operation,[c] this would trans-

[b] "*RMA* [Rubber Manufacturers Association] *estimates that there have been about 295 tire recalls in this country. Only four of these recalls have involved more than 1 million tires and only 51 recalls have involved over 10,000 tires. Furthermore, RMA estimates that 142 recalls have involved less than 1,000 tires."* Comments of the RMA, On Notice of Proposed Rulemaking: Motor Vehicle Safety; Disposition of Recalled Tires, February 19, 2002, p. 2, Docket Document ID: NHTSA 2001-10856-0004.

c. Research and Innovative Technology Administration, Bureau of Transportation Statistics,. (2001). *Highlights of the 2001 National Household Travel Survey*: 29.1 miles per day per person/55.1 minutes per day of travel per person = average speed of 31.7 mph. 12,497 miles per year vehicle use/31.7 mph = 394 hours per year of vehicle use. www.bts.gov/publications/highlights_of_the_2001_national_household_travel_survey/html/section_02.html late into approximately 1,400 hrs and 44,700 miles of total rolling operation. Assuming an average vehicle speed of 31.77 mph in the US,[d] an average 16-inch passenger tire would experience approximately 35 million deflection cycles during service.[e] To attempt to account for this, the performance tests in Federal safety standards subject new tires to conditions on the curved indoor roadwheel that are more severe than normal operating conditions on a flat road surface. However, unlike actual tire service, the straight-line indoor roadwheel tests in the safety standard do not input cornering and camber forces into the tire and do not evaluate the structural durability of a tire after it has experienced long-term material property degradation under cyclic fatigue.[13,14]

Statistically, instances where a tire fails during operation by a sudden, catastrophic failure such as complete or partial tread separation, sidewall failures (blow-outs and zipper failures), bead failures, etc. are rare. However, depending upon the vehicle and the driving conditions it may be difficult for the driver to maintain vehicle control when a sudden catastrophic tire failure occurs. These instances can result in a loss of control, which may in turn result in the vehicle leaving the roadway. It is logical therefore to ask whether or not 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. In other words, would one-year-old passenger vehicle tires have less chance of a structural failure during service than the seven percent of passenger vehicle tires that are still in operation after eight years?

Accordingly, the first goal of tire aging research was to develop a better understanding of service-related tire degradation over time. Once this goal had been achieved, the second goal was to develop an accelerated, laboratory-based tire test that simulates real world tire aging. NHTSA tire aging research to date has been divided into multiple phases: Phase 1 addressed the first of these goals -- better understanding how tires change over time. The research recognized that there are two mechanisms operating to produce changes in tire properties, particularly in the critical belt-edge region. First is degradation of the rubber compound and material interfaces due to the effects of heat and reaction with oxygen (thermo-oxidative aging), and second is the effect of cyclic fatigue during tire deformation.

This report documents Phase 2 of the project, which focuses on developing an accelerated, laboratory-based tire test that simulates real world tire aging and evaluates the remaining structural durability of the aged tires. Three methods of laboratory aging the tires were evaluated:

> **Long-Term Durability Endurance Test:** Michelin offered the 97-km/h (60-mph) Long-Term Durability Endurance (LTDE) test as a combined tire aging and durability test. In this test, the tire is inflated using an oxygen-enriched air mixture and run on an indoor roadwheel for up to 500 hours at elevated loads and pressures to fatigue the tire structure and induce heat, which in conjunction with the oxygen-enriched inflation mixture accelerated the aging process. The oxygen-enriched air mixture consisted of

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[d] Product of speed and percentage time across all fourteen cycles (31.77 mph), Final Technical Support Document Fuel Economy Labeling of Motor Vehicles: Revisions to Improve Calculation of Fuel Economy Estimates, Page 49, Environmental Protection Agency, EPA420-R-06-017, December 2006.

[e] Most popular replacement passenger tire size 2007 (source RMA Preliminary 2007): P225/60R16; (44,700 mi * 63,360 in/mi * 1 rev/2 Pi radians)/12.75 in Dynamic Radius = 35.4 million cycles

50%N/50%O, topped-off every 24 hours (regular compressed air contains only about 21% oxygen).

Passenger Endurance) Test: Continental submitted a version of its passenger endurance (P-END) test under the confidential submission process. As with the LTDE test, the P-END test is a combined aging and durability test in which the tire is inflated with air and then run on an indoor roadwheel for up to 240 hours. Although this test was designed to be used for passenger cars, the tire manufacturer provided separate conditions under which light truck tires could be tested.

Oven-Aging Method: Ford, in the course of its own tire aging research, had sufficient data to recommend that the agency use a method in which the tire is inflated using the 50% oxygen air mixture previously mentioned, 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.

Summary results for the tires retrieved from service in Phoenix are included in this report for comparison. The detailed results for the Phase 1, Phoenix tires have been reported separately.[3]

2.0 METHODOLOGY

The six tire models collected and tested in Phase 1 of the program are listed in Table 2. These tire models had been identified by the manufacturer as having no significant design changes during the period 1998 to 2003.[f] Accordingly, new tires of these six models were purchased and evaluated by one of the three candidate protocols for laboratory aging versus new tires and tires retrieved from service in Phoenix. A description of the tests employed in the study is shown in Appendix 1. The complete list of tires referred to in this study is shown in Table 5. All raw data used in the report are available in the public NHTSA Tire Aging Dataset.[15]

Table 2. Tire Models Selected for Testing

NHTSA Tire Type	OE Fitment?	Tire Brand	Tire Model	Tire Size	Load Range	Speed Rating
B	Yes	BFGoodrich	Touring T/A SR4	P195/65R15	89	S
C	Yes	Goodyear	Eagle GA	P205/65R15	92	V
D	Yes	Michelin	LTX M/S	P235/75R15XL	108	S
E	Yes	Firestone	Wilderness AT	P263/75R16	114	S
H	No	Pathfinder*	ATR A/S	LT245/75R16	120/116E	Q
L	Yes	General	Grabber ST	255/65R16	109	H

*Manufactured for the Discount Tire Company by the Kelly-Springfield Tire and Rubber Company, a subsidiary of the Goodyear Tire and Rubber Company

Tire aging (service-related degradation) was shown to occur due to changes in tire materials, particularly the tire rubber and its bonds to adjacent components. The two main mechanisms for this change are thermo-oxidative degradation of the rubber compounds and other materials and fatigue damage due to repeated stressing of the tire components, particularly at material interfaces. These mechanisms are extremely complex and do not operate independently. As was noted, the most critical region of the tire is the belt edge region of the tire shoulder. A simplified illustration of what would properly be called the mechano-thermo-oxidative degradation of tire components is shown in Figure 1. The major aspects of the factors that affect degradation are:

- Deformation of the tire provides mechanical energy that:
    - produces cyclic stress fatigue leading to crack initiation and propagation, and
    - generates heat in the tire components.
- Oxygen is supplied to the internal tire components, primarily by diffusion of O_2 from the inflation gas.
- Deformation energy and heat, along with heat from ambient conditions, forms chemical free radicals that:
    - Initiates reactions including:
        - oxidation of polymer;

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[f] Since multiple variants of each tire model can exist simultaneously, this confirmation of design consistency with the manufacturers for each tire model was only marginally effective. Three of the six tire models studied had what would be considered design changes or line-to-line build variations during the 1998-2003 time period.

- ▪ Polymer chain reactions, such as chain scission and formation of carbon-carbon crosslinks; and
- ▪ Reactions with sulfur, such as rearrangements of sulfur bonds and formation of sulfoxides.
  - Many reactions generate another radical, causing a self-sustaining (autocatalytic) cycle.
- Oxygen is replenished by diffusion into tire components.
- Reactions continue until oxygen is depleted in the tire component, or radicals are consumed in a reaction or trapped by antioxidant material.
- Changes in tire component properties change the response to deformation and resistance to fatigue.
- For the majority of tires the cycle is repeated until the tread is worn out and the tire is replaced.
  - o Under rare conditions, some tires may fail during service.

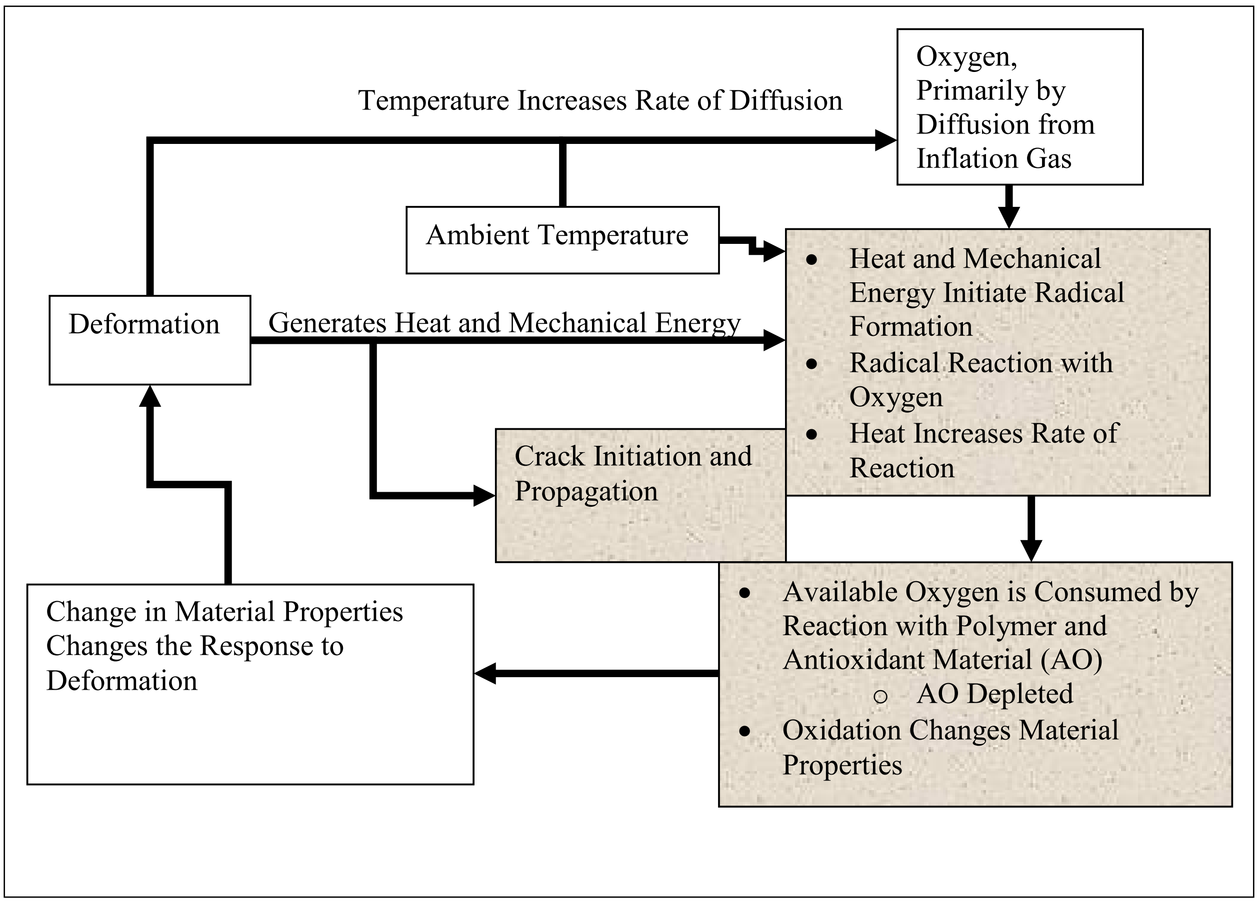

Figure 1. Simplified Illustration of Rubber Degradation by Mechanical Energy, Heat Energy, and Reaction With Oxygen

There are three primary considerations for a laboratory test to simulate the aging that takes place in a tire in normal service. It has been shown that the primary mechanism by which tire components degrade during service involve reaction with oxygen,[16] therefore a sufficient supply of oxygen into the internal components of the tire to provide available O_2 is necessary to avoid a condition of anaerobic reaction, known as diffusion limited oxidation (DLO).[4] Second, in order to complete the test in a reasonable length of time to allow evaluation of large numbers of tires, the temperature needs to be increased to speed up the reactions. In general, the effects of increasing the temperature on the rates of the competing reactions that take place in the rubber compound (including the diffusion of oxygen into the internal components of the tire) are described by the Arrhenius equation (Equation 1). The pre-exponential factor and energy of activation are specific to the materials participating in the reaction, thus the rate of increase is different for each of the competing reactions. If the temperature of the test is increased too far beyond the operating temperature of the tire in normal service, different reactions may become dominant.

$$k = Ae^{(-E_a/RT)}$$

Where:
k = rate of reaction
A = pre-exponential factor
E_a = energy of activation
R = ideal gas constant
T = absolute temperature

Equation 1. Arrhenius Equation[g]

Finally the tire components must be stressed, either during the test procedure itself, or by evaluating the change in resistance to stress after the properties of the tire components have experienced thermo-oxidative changes. The evaluation of the Firestone Wilderness tires involved in the recall of the tires on the Ford Explorer showed that the peel adhesion, a measure of the instantaneous maximum stress that the component could withstand, was reduced significantly after the tires had been in service.[1] Tires removed from service in Phoenix also showed reduced peel adhesion after having been in service. While the peel test did correlate well with material property changes, it was not deemed appropriate as a predictor of whole tire performance since absolute levels of adhesion did not correlate to measured levels of performance.[3] Thus, part of this study also evaluated the residual resistance of the tires to durability on a roadwheel test.

2.1 Michelin LTDE Test

The LTDE test procedure was supplied by Michelin for evaluation. The test tire is inflated with a 50%N/50%O gas mixture in an attempt to provide sufficient O_2 to the internal components of the tire to avoid diffusion limited oxidation. The tire is run on a 1.707 m (67.23 in.) roadwheel at 97 km/h (60 mph) for up to 500 hours. The test conditions are shown in Table 3. Tires were evaluated based on hours survived during the test, and for the material properties shown in Table 4 after completing approximately 100, 300 and 500 hours of testing. Both the durability and change in material properties were compared to the same model of new tires and tires collected from ser-

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[g] *"An equation in physical chemistry that relates the increase in the rate of a chemical reaction to a rise in temperature."* Source: Encarta World English Dictionary,North American Edition, 2007. Although the Arrhenius equation provides insight, the equation is based on the proportion of reacting species that have sufficient energy to react during random collisions in gas or dilute solutions. The limited mobility and non-random distribution of reactive sites and species in polymer matrices may produce significant departures from the idealized case.

vice in Phoenix. Though a balanced test matrix was designed for the LTDE test for all six tire models, of 137 valid roadwheel tests (i.e., proper test procedure conducted/no wheel or equipment failures), 28 tires (20.4%) failed before reaching the prescribed number of roadwheel hours. Though some failed tires were sufficiently intact to allow limited material properties analysis, data was only analyzed from tires that successfully completed the full amount of prescribed test hours without failure.

Table 3. Test Parameters -- Long-Term Durability Endurance Test

Designation	**P-metric/Metric**		**Light Truck**			
Load Range	Standard Load	Extra Load	B	C	D	E
Test Temp	38°C ±3°C (100°F ±5°F)					
Speed	60 mph (97 km/h)					
Filling Gas	50%N/50%O (hot pressure at 4 hour mark maintained and topped-off daily)					
Load (% max single)	111		142	112	98	92
Initial Pressure (psi)	40	46	57		65	80

Table 4. Material Properties Evaluated[h]

Material Property Test	Tire Component
Shore A Hardness	Tread
	Liner
	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
Indentation Modulus	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Liner
	Tread
	Fabric Coat Stock
	Overlay Stock
	Tread/Shoulder Wedge
	Shoulder
	Tread Base
Tensile and Elongation	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
180º Peel Adhesion	Skim Area of Belt
	Wedge Area of Belt
Micro-DeMattia Flex	Wedge Area of Belt
Cross-Link Density and Distribution	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Tread Compound
Two-Ply Laminate Fatigue	Cut Section of Tire Belt
Shearography	Whole Tire Test
Fixed Oxygen	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Tread Compound

2.2 Continental P-END Test

The P-END test procedure was supplied by Continental Tire for evaluation. This is also an endurance test using a 1.707 m (67.23 in.) roadwheel for up to 240 hours. The exact test conditions are confidential. Tires were evaluated based on hours survived during the test, and for the material properties shown in Table 4 after completing approximately 96, 168 and 240 hours of testing. Although this test was designed to be used for passenger cars, the tire manufacturer provided separate conditions under which light truck tires could be tested. Both the durability and change in material properties were compared to the same model of new tires and to tires collected from service in Phoenix. As with the LTDE test, though a balanced test matrix was designed for the P-END test for all six tire models, of 95 valid roadwheel tests (*i.e.,* proper test procedure conducted/no wheel or equipment failures), 44 tires (46.3%) failed before reaching the prescribed number of roadwheel hours. Though some failed tires were sufficiently intact to allow limited mate-

[h] Not every test was run on every tire, the complete list of tires and tests are shown in Reference 15.

rial properties analysis, data was only analyzed from tires that successfully completed the full amount of prescribed test hours without failure.

2.3 Oven Aging

Tires were inflated with a mixture of 50%N_2/50%O_2 to a specified pressure and exposed to a constant elevated temperature between 55ºC and 70ºC in a circulating air oven for varying lengths of time. A subset of tires was subjected to a break-in on a 1.707 m roadwheel for 24 hours at 120 km/h (75 mph) prior to oven exposure. For most of the tires the inflation pressure was capped, that is the pressure was set at the beginning of the test and no further inflation gas was added during the exposure period. However, a subset of tires was refilled weekly with the 50%N_2/50%O_2 gas. After oven exposure times of 3 to 12 weeks, the durability on a roadwheel test and the material properties were compared to the same model of new tires and tires collected from service in Phoenix. Of the 91 valid oven aging tests (i.e., proper test procedure conducted/no wheel or equipment failures), no tires failed before reaching the prescribed amount of oven aging.

2.4 Air Permeability

Diffusion of oxygen into the internal components of the tire, especially at the critical belt-edge region, is an important part of the aging phenomenon. Therefore air permeability was tested by the ASTM F1112-00 procedure. This method uses tires inflated with air and capped (that is no additional inflation gas added during the test) for 12 weeks at the standard temperature of 21ºC and at 70ºC. The material properties of the tires were measured for comparison to new tires and tires that were aged while inflated with 50%N_2/50%O_2.

2.5 Roadwheel Testing

After oven exposure, tires were tested on a roadwheel, using the stepped-up-load until catastrophic failure protocol that was used for the Phoenix tires in the Phase 1 testing. The tire is inflated, stabilized at the laboratory temperature, then run continuously and uninterrupted at 120 km/h (75 mph) for 4 hours at 85 percent maximum load, 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. If the tire passes inspection, the SUL test restarts and runs the tire through additional load steps that increase by 10 percent of max load every 4 hours until catastrophic structural failure. The initial load and incremental loads are proportional to the maximum load rating for each tire. Failure times and types were compared to new tires and tires retrieved from service in Phoenix. These results have been reported in detail.[17]

2.6 Tires Studied

The tires used in the Phase 2 work and aging conditions are shown in Table 5.

Table 5. Tires and Tests Used in Phase 2 Aging Study

Tire Type	Aging Type	Description	Barcode	DOT Number
B	Oven	12 Weeks @ 55°C, 50%N_2/50%O_2 Inflation Gas	1081	APC6BB113803
			1082	APC6BB113803
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1068	APC6BB113803
			1072	APC6BB113803
			1088	APC6BB113803
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1069	APC6BB113803
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1070	APC6BB113803
			1089	APC6BB113803
		6 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1071	APC6BB113803
			1090	APC6BB113803
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1076	APC6BB113803
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1077	APC6BB113803
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24 hr. Break-in @ 120 km/h	1086	APC6BB113803
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1087	APC6BB113803
	Roadwheel	LTDE Test, 100 hrs	1058	APC6BB113803
			1074	APC6BB113803
			1075	APC6BB113803
		LTDE Test, 292 hrs	1059	APC6BB113803
			1080	APC6BB113803
		LTDE Test, 508 hrs	1060	APC6BB113803
			1084	APC6BB113803
		P-END Test, 96 hrs	1043	APC6BB113803
			1044	APC6BB113803
			1047	APC6BB113803
		P-END Test, 168 hrs	1048	APC6BB113803
			1049	APC6BB113803
			1050	APC6BB113803
			1051	APC6BB113803
			1091	APC6BB113803
		P-END Test, 240 hrs	1053	APC6BB113803
			1054	APC6BB113803
			1055	APC6BB113803
			1056	APC6BB113803
C	Oven	12 Weeks @ 55°C, 50%N_2/50%O_2 Inflation Gas	1581	M6URFJ2R4802
			1582	M6URFJ2R4802
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1516	M6URFJ2R4802
			1561	M6URFJ2R4802
			1564	M6URFJ2R4802

Tire Type	Aging Type	Description	Barcode	DOT Number
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1568	M6URFJ2R4802
			1569	M6URFJ2R4802
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1565	M6URFJ2R4802
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1562	M6URFJ2R4802
			1566	M6URFJ2R4802
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1570	M6URFJ2R4802
		6 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1563	M6URFJ2R4802
			1567	M6URFJ2R4802
		6 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1571	M6URFJ2R4802
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1576	M6URFJ2R4802
			1577	M6URFJ2R4802
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1586	M6URFJ2R4802
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1587	M6URFJ2R4802
		12.9 Weeks @ 70°C, Air Inflation Gas	1521	M6URFJ2R4802
			1522	M6URFJ2R4802
			1596	M6URFJ2R4802
	Roadwheel	LTDE Test, 100 hrs	1558	M6URFJ2R4802
			1574	M6URFJ2R4802
			1575	M6URFJ2R4802
			1588	M6URFJ2R4802
			1591	M6URFJ2R4802
			1601	M6URFJ2R4504
			1602	M6URFJ2R4504
			1603	M6URFJ2R4504
			1604	M6URFJ2R4504
			1605	M6URFJ2R4504
			1606	M6URFJ2R4504
			1607	M6URFJ2R4504
			1608	M6URFJ2R4504
			1609	M6URFJ2R4504
			1610	M6URFJ2R4504
		LTDE Test, 292 hrs	1579	M6URFJ2R4802
			1580	M6URFJ2R4802
		LTDE Test, 300 hrs	1589	M6URFJ2R4802
			1592	M6URFJ2R4802
			1611	M6URFJ2R4504
			1613	M6URFJ2R4504
			1614	M6URFJ2R4504
			1615	M6URFJ2R4504
			1616	M6URFJ2R4504

Tire Type	Aging Type	Description	Barcode	DOT Number
			1617	M6URFJ2R4504
			1618	M6URFJ2R4504
			1619	M6URFJ2R4504
			1620	M6URFJ2R4504
		LTDE Test, 500 hrs	1590	M6URFJ2R4802
			1624	M6URFJ2R4504
			1625	M6URFJ2R4504
			1626	M6URFJ2R4504
			1627	M6URFJ2R4504
			1628	M6URFJ2R4504
		LTDE Test, 508 hrs	1630	M6URFJ2R4504
			1584	M6URFJ2R4802
		P-END Test, 96 hrs	1543	M6URFJ2R4802
			1544	M6URFJ2R4802
			1547	M6URFJ2R4802
		P-END Test, 168 hrs	1548	M6URFJ2R4802
			1550	M6URFJ2R4802
			1551	M6URFJ2R4802
			1597	M6URFJ2R4802
			1598	M6URFJ2R4802
		P-END Test, 240 hrs	1553	M6URFJ2R4802
			1554	M6URFJ2R4802
			1555	M6URFJ2R4802
			1556	M6URFJ2R4802
			1557	M6URFJ2R4802
D	Oven	12 Weeks @ 55°C, 50%N_2/50%O_2 Inflation Gas	1181	B3DD472X2003
			1182	B3DD472X2003
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1161	B3DD462X2903
			1165	B3DD462X2903
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1162	B3DD462X2903
			1166	B3DD462X2903
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1163	B3DD462X2903
			1167	B3DD462X2903
		6 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1164	B3DD462X2903
			1168	B3DD462X2903
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1176	B3DD472X2003
			1177	B3DD472X2003
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1186	B3DD472X2003
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1187	B3DD472X2003
	Roadwheel	LTDE Test, 100 hrs	1158	B3DD462X2903
			1174	B3DD462X2903
			1175	B3DD462X2903
			1198	B3DDBDUX2303

Tire Type	Aging Type	Description	Barcode	DOT Number
		LTDE Test, 292 hrs	1159	B3DD462X2903
			1179	B3DD472X2003
			1180	B3DD472X2003
		LTDE Test, 300 hrs	1199	B3DDBDUX2303
		LTDE Test, 500 hrs	1200	B3DDBDUX2303
		LTDE Test, 508 hrs	1184	B3DD472X2003
			1185	B3DD472X2003
		P-END Test, 96 hrs	1115	B3DD462X2903
			1143	B3DD462X2903
			1144	B3DD462X2903
		P-END Test, 168 hrs	1151	B3DD462X2903
		12.9 Weeks @ 70°C, Air Inflation Gas	1191	B3DDBDUX2303
			1192	B3DDBDUX2303
			1193	B3DDBDUX2303
			1194	B3DDBDUX2303
E	Oven	12 Weeks @ 55°C, 50% N_2/50% O_2 Inflation Gas	1383	VN73WMB0702
			1393	VN73WMB0902
		3 Weeks @ 60°C, 50% N_2/50% O_2 Inflation Gas	1305	VN73WMB4901
			1324	VN73WMB4002
		3 Weeks @ 60°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1365	VN73WMB4502
		3 Weeks @ 70°C, 50% N_2/50% O_2 Inflation Gas	1316	VN73WMB4002
			1329	VN73WMB4202
		3 Weeks @ 70°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1367	VN73WMB4502
		6 Weeks @ 60°C, 50% N_2/50% O_2 Inflation Gas	1322	VN73WMB4002
		6 Weeks @ 60°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1369	VN73WMB4502
		6 Weeks @ 70°C, 50% N_2/50% O_2 Inflation Gas	1323	VN73WMB4002
			1360	VN73WMB4002
		6 Weeks @ 70°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1391	VN73WMB4502
		8 Weeks @ 65°C, 50% N_2/50% O_2 Inflation Gas	1361	VN73WMB4002
		8 Weeks @ 65°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1394	VN73WMB1402
		8 Weeks @ 65°C, 50% N_2/50% O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1395	VN73WMB1702
		12.9 Weeks @ 70°C, Air Inflation Gas	1342	VN73WMB0402
			1374	VN73WMB0402
			1388	VN73WMB0402
			1396	VN73WMB0602
	Roadwheel	LTDE Test, 100 hrs	1325	VN73WMB0902
			1330	VN73WMB4002

Tire Type	Aging Type	Description	Barcode	DOT Number
			1353	VN73WMB0902
			1362	VN73WMB4002
			1392	VN73WMB0402
			1631	VN73WM03205
			1632	VN73WM03205
			1633	VN73WM03205
			1634	VN73WM03205
			1635	VN73WM03205
			1636	VN73WM03205
			1637	VN73WM03205
			1638	VN73WM03205
			1639	VN73WM03205
			1640	VN73WM03205
		LTDE Test, 292 hrs	1338	VN73WMB4002
			1366	VN73WMB1602
			1380	VN73WMB1702
		LTDE Test, 300 hrs	1643	VN73WM03205
			1645	VN73WM03205
			1646	VN73WM03205
		LTDE Test, 500 hrs	1387	VN73WMB0402
			1651	VN73WM03205
			1652	VN73WM03205
			1654	VN73WM03205
			1655	VN73WM03205
			1656	VN73WM03205
			1657	VN73WM03205
			1659	VN73WM03205
		LTDE Test, 508 hrs	1311	VN73WMB4002
			1354	VN73WMB4002
			1355	VN73WMB4002
		P-END Test, 96 hrs	1332	VN73WMB4502
			1339	VN73WMB4502
			1340	VN73WMB4502
			1346	VN73WMB4502
			1347	VN73WMB4502
		P-END Test, 168 hrs	1341	VN73WMB4002
			1343	VN73WMB4002
			1345	VN73WMB4002
			1351	VN73WMB0702
			1373	VN73WMB0502
		P-END Test, 240 hrs	1371	VN73WMB4502
			1377	VN73WMB4502
			1379	VN73WMB4502
H	Oven	12 Weeks @ 55°C, 50%N_2/50%O_2 Inflation Gas	1281	PJ11FKKV4403
			1282	PJ11FKKV4403
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation	1268	PJ11FKKV4403

Tire Type	Aging Type	Description	Barcode	DOT Number
		Gas	1272	PJ11FKKV4403
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1269	PJ11FKKV4403
			1296	PJ11FKKV4403
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1270	PJ11FKKV4403
			1297	PJ11FKKV4403
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1271	PJ11FKKV4403
			1298	PJ11FKKV4403
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1276	PJ11FKKV4403
			1277	PJ11FKKV4403
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1286	PJ11FKKV4403
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1287	PJ11FKKV4403
		12.9 Weeks @ 70°C, Air Inflation Gas	1221	PJ11FKKV4403
			1222	PJ11FKKV4403
			1223	PJ11FKKV4403
	Roadwheel	LTDE Test, 100 hrs	1263	PJ11FKKV4403
			1275	PJ11FKKV4403
		LTDE Test, 300 hrs	1300	PJ11FKKV4403
		P-END Test, 96 hrs	1247	PJ11FKKV4403
			1255	PJ11FKKV4403
			1293	PJ11FKKV4403
L	Oven	12 Weeks @ 55°C, 50%N_2/50%O_2 Inflation Gas	1481	A33X3HB3003
			1482	A33X3HB3003
		3 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1461	A33X3HB3003
			1465	A33X3HB3003
		3 Weeks @ 70°C, 50%N_2/50%O_2 Inflation Gas	1462	A33X3HB3003
			1466	A33X3HB3003
		6 Weeks @ 60°C, 50%N_2/50%O_2 Inflation Gas	1463	A33X3HB3003
			1467	A33X3HB3003
		6 Weeks @70°C, 50%N_2/50%O_2 Inflation Gas	1464	A33X3HB3003
			1468	A33X3HB3003
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas	1476	A33X3HB3003
			1477	A33X3HB3003
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h	1486	A33X3HB3003
		8 Weeks @ 65°C, 50%N_2/50%O_2 Inflation Gas, 24-hr. Break-in @ 120 km/h, Weekly Refill	1487	A33X3HB3003
		12.9 Weeks @ 70°C, Air Inflation Gas	1491	A33X3HB3003
			1492	A33X3HB3003
	Roadwheel	LTDE Test, 100 hrs	1416	A33X3HB3003
			1422	A33X3HB0204
			1471	A33X3HB3003
			1474	A33X3HB3003

Tire Type	Aging Type	Description	Barcode	DOT Number
			1475	A33X3HB3003
			1489	A33X3HB3003
		LTDE Test, 292 hrs	1459	A33X3HB3003
			1480	A33X3HB3003
		LTDE Test, 300 hrs	1472	A33X3HB3003
			1490	A33X3HB3003
		LTDE Test, 500 hrs	1421	A33X3HB0204
		P-END Test, 96 hrs	1443	A33X3HB3003
			1444	A33X3HB3003
			1445	A33X3HB3003
			1446	A33X3HB3003
			1447	A33X3HB3003
		P-END Test, 168 hrs	1450	A33X3HB3003

3.0 RESULTS

3.1 Pearson R, Product-Moment Correlations

The first stage was to investigate if there are systematic changes in any measured property correlated to the conditions of aging. The Pearson R value ranges between -1 for perfect inverse correlation to +1 for direct correlation, with zero representing no correlation. In this work, the terms in Table 6 will be used as general descriptions of the level of correlation:

Table 6. Terms for Strength of Correlation Used

Strength of Correlation	Pearson R range
Insignificant	-0.39 to 0.39 (Not Reported)
Weak	-0.59 to -0.40 | 0.40 to 0.59
Moderate	-0.79 to -0.60 | 0.60 to 0.79
Strong	-1.00 to -0.80 | 0.80 to 1.00

Correlations of each measured property were calculated to duration of test condition. This correlation only determines if there is a systematic change and the direction of that change, and provides no information about the magnitude of the change. In other words a systematic change of 1% per week with only 6 data points will provide a good correlation while an average change of 10% per week with wide variation and a large number of data points will provide a lower correlation coefficient. Therefore, one-way ANOVA and linear regression analysis of those properties that showed significant correlations were used to determine if the general correlations could be refined based on additional terms; for example, temperature in the oven, or type of roadwheel test. In the following tables properties with insignificant correlations are left blank.

3.1.1 Shore A Hardness

Durometer readings on the Shore A scale were completed for some tires.[i] The tread compound, the liner compound, the skim-coat compound and the wedge compound were measured per the ASTM D 2240-02b standard test method. The results of the correlations are shown in Table 7.[j] For the liner, the only significant correlations were negative for the Phoenix tires and positive for the oven tires. The skim-coat generally showed moderate positive correlations except for the type H tires, which showed a moderate negative correlation. The wedge coat compound generally showed moderate negative correlations. The tread compound generally hardened over time.

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[i] Not all tires or components were measured for each property. The limitations of this will be discussed later. The complete dataset is available at Reference 15.

[j] In all tables of correlation results; insignificant correlations are left blank, and NM = Not Measured.

Table 7. Correlations of Shore A Hardness to Time

Tire Type / Component Measured	Test Type	B	C	D	E	H	L
Liner	Phoenix			-0.54		-0.63	
	Roadwheel						
	Oven	0.75	0.61		0.56		0.65
Skim	Phoenix	0.46			0.76	-0.61	-0.42
	Roadwheel	0.54			0.42	-0.69	
	Oven	0.80	0.43	0.63	0.81		0.50
Wedge	Phoenix			-0.54		-0.63	
	Roadwheel						
	Oven	0.75	0.61		0.56		0.65
Tread	Phoenix		NM	0.43	-0.52	0.85	0.78
	Roadwheel	0.66		0.78		0.40	
	Oven	NM	0.44	NM	NM		0.53

3.1.2 Indentation Modulus

The indentation modulus of rubber components of selected tires was measured using the method shown in Appendix 1. Details of the test method have been reported separately.[18] The results of the correlations are shown in Table 8. Nearly all compounds showed a strong tendency to harden over time; in service, on a roadwheel test, or in the oven.

Table 8. Correlations of Indentation Modulus to Time

Tire Type Component Measured	Test Type	B	C	D	E	H	L
Liner	Phoenix	0.98	0.99		0.90	0.58	0.99
	Roadwheel	0.72	0.75		0.92	0.89	0.57
	Oven	0.82	0.69	0.79	0.91	0.76	0.88
Skim	Phoenix	0.84	0.60	0.99	0.56		
	Roadwheel	0.83	0.58	0.41			
	Oven	0.75	0.88	0.45	0.91	0.70	0.61
Wedge	Phoenix	0.83	-0.93	0.83	0.69	-0.57	0.47
	Roadwheel	0.89	0.46			-0.63	-0.84
	Oven	0.86	0.92	0.76	0.99		0.61
Tread	Phoenix	0.75	0.74	0.99	0.62	0.84	0.96
	Roadwheel	0.65	0.62	0.83	0.72	0.99	0.43
	Oven	0.81	0.73	0.58	0.78	0.65	0.74
Shoulder	Phoenix	0.93		0.96	0.95	0.96	-0.84
	Roadwheel	0.84	0.55	0.96		0.68	-0.40
	Oven	0.80	0.87	0.70	0.60	0.69	0.78
Fabric Coat Stock	Phoenix	0.54	0.58		0.87	0.71	0.89
	Roadwheel	0.80	0.75	0.65	0.94	0.64	0.53
	Oven	0.88	0.52	0.70	0.69	0.75	0.84
Tread Base	Phoenix	0.94	0.66	NM	NM		
	Roadwheel	0.69	-0.79				
	Oven	0.80	-0.83			0.69	
Overlay	Phoenix	NM	0.88	NM	NM	0.95	0.70
	Roadwheel						-0.82
	Oven		0.85			0.55	0.87
Overlay Gumstrip	Phoenix	NM	NM	NM		NM	
	Roadwheel				-0.41		-0.92
	Oven				0.78		0.99

3.1.3 __Tensile and Elongation of Skim-Coat Compound__

Tensile and elongation of the rubber compound were measured according to the method in ASTM D 412. Correlations of the tensile and elongation properties for the wire skim-coat compound are shown in Table 9. The breaking strength and ultimate elongation to break both tended to moderately decrease over time, whether taken from service, tested on a roadwheel, or aged in an oven. Correspondingly, the modulus of the compounds showed a moderate increase over time. For the tires which were aged in the laboratory, particularly those aged in the oven, the ultimate elongation decreased below 300 percent, so those correlations are based on a limited number of values.

Table 9. Correlations of Tensile and Elongation for Skim-Coat Compound

Tire Type Measurement	Test Type	B	C	D	E	H	L
Ultimate Breaking Strength	Phoenix	-0.40	-0.44			-0.73	-0.49
	Roadwheel			-0.57			
	Oven	-0.52	-0.60		-0.67	-0.79	-0.67
Ultimate Elongation	Phoenix	-0.77	-0.71	-0.66	-0.62	-0.92	-0.77
	Roadwheel	-0.42		-0.74	-0.48		-0.45
	Oven	-0.72	-0.52		-0.69	-0.84	-0.69
Modulus @50% elongation	Phoenix	0.62	0.73	0.68	0.84	0.65	0.70
	Roadwheel	0.60	0.40	0.71	0.59		0.50
	Oven	0.65			0.77	0.65	0.59
Modulus @100% elongation	Phoenix	0.75	0.77	0.75	0.88	0.79	0.74
	Roadwheel	0.69	0.42	0.67	0.60		
	Oven	0.63			0.60	0.69	
Modulus @200% elongation	Phoenix	0.79	0.81	0.67	0.86	0.61	
	Roadwheel	0.60		0.59	0.45		
	Oven	0.73		-0.77	0.44		0.71
Modulus @300% elongation	Phoenix	0.67	0.74	0.70	0.52		
	Roadwheel	0.52					
	Oven	0.66		-0.80			0.65

3.1.4 Tensile and Elongation of Wedge Compound

The wedge compound is the compound between the wire belts in the shoulder area. The shoulder area is part of the tread/belt region which has been shown to be where 95 percent of tire failures have been reported to NHTSA in the published defects reports.[9] This compound may be of 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.

Correlations of the tensile and elongation properties for the wedge compound are shown in Table 10. The breaking strength and ultimate elongation to break both tended to show moderate to strong decreases over time, whether taken from service, tested on a roadwheel, or aged in an oven. Correspondingly, the modulus of the compounds increased over time. For the tires which were aged in the laboratory, particularly those aged in the oven, the ultimate elongation decreased below 300 percent, so those correlations are based on a limited number of values. For tire type L, all samples for tires removed from service in Phoenix failed before reaching 300 percent extension.

Table 10. Correlations of Tensile and Elongation for Wedge Compound

Tire Type / Measurement	Test Type	B	C	D	E	H	L
Ultimate Breaking Strength	Phoenix		-0.62		-0.62	-0.46	-0.63
	Roadwheel			-0.60	-0.61	-0.58	-0.46
	Oven	-0.54	-0.53		-0.74	-0.75	-0.66
Ultimate Elongation	Phoenix	-0.80	-0.73	-0.58	-0.84	-0.82	-0.79
	Roadwheel	-0.65		-0.72	-0.73	-0.65	-0.67
	Oven	-0.59	-0.66		-0.74	-0.80	-0.71
Modulus @50% elongation	Phoenix		0.60		0.82		0.78
	Roadwheel	0.66		0.75			-0.56
	Oven	0.50			0.77	0.59	0.67
Modulus @100% elongation	Phoenix	0.82	0.64	0.66	0.88	0.67	0.78
	Roadwheel	0.75		0.75	0.61		
	Oven	0.48			0.72	0.51	0.55
Modulus @200% elongation	Phoenix		0.62	0.67	0.84	0.77	0.57
	Roadwheel	0.71		0.59	0.41		
	Oven				0.47		
Modulus @300% elongation	Phoenix	0.74	0.55	0.62	0.46	0.48	NM
	Roadwheel	0.76		0.45			0.40
	Oven	0.42		-0.67			0.53

3.1.5 Micro-DeMattia Flex Test

The micro-DeMattia flex test is an adaptation of the ASTM D813 method as described in Appendix 1. A crack is initiated in the sample, which is repeatedly flexed, and the growth of the crack monitored. The reported value is the calculated inches of growth per million cycles of flexing. Several of the samples failed within a few cycles – for those samples a value of 1,000 was inserted. The crack growth rate in the critical wedge compound of the tires tended to show a moderate increase over time, whether in service in Phoenix or in laboratory aging.

Table 11. Correlations of Micro-DeMattia Flex Fatigue for Wedge Compound

Tire Type / Measurement	Test Type	B	C	D	E	H	L
Crack Growth Rate	Phoenix	0.76		0.42	0.92		0.74
	Roadwheel	0.49	0.87		0.72	0.75	
	Oven	0.56	0.68			0.75	

3.1.6 Crosslink Density and Distribution

The crosslink density of the tread, skim-coat and wedge compounds were measured for a limited number of tires retrieved from Phoenix service or tested by the laboratory roadwheel aging methods. Tires from oven aging, which at the time had no developed test procedure, were not tested in this phase of work. The total number of crosslinks per cm^3 of rubber compound, and the percentage of strong, moderate, and weak crosslinks were measured by cleavage and subsequent swelling of the rubber compound. These crosslinks are sometimes referred to as S1, S2, and Sx or S8 sulfur crosslinks; but may contain other types of crosslinks, particularly carbon-carbon and sulfoxide linkages. Complete details of the test procedure and interpretation of the results, including oven-aged tires from the next phase of testing (Phase 3) of the work, have been reported separately.[6] The major conclusion from these data is that the total crosslink density has a strong tendency to increase with increasing time in service in Phoenix or accumulated mileage on a roadwheel test.

Table 12. Correlation of Crosslink Density and Distribution

Tire Component	Tire Type / Measurement	Test Type	B	C	D	E	H	L
Tread	Total Crosslink Density	Phoenix	0.94	NM	NM	NM	0.85	0.98
		Roadwheel	0.42	0.54	0.81	0.73	0.98	
	Strong Crosslinks, %	Phoenix	0.43	NM	NM	NM	0.90	0.48
		Roadwheel				-0.45		
	Moderate Crosslinks, %	Phoenix	-0.58	NM	NM	NM		-0.48
		Roadwheel						-0.48
	Weak Crosslinks, %	Phoenix		NM	NM	NM		
		Roadwheel						
Skim-Coat	Total Crosslink Density	Phoenix	0.70	NM	NM	NM	0.92	0.78
		Roadwheel	0.60	0.81	0.92	0.78	0.95	0.69
	Strong Crosslinks, %	Phoenix	-0.81	NM	NM	NM		0.87
		Roadwheel	0.47	0.75	0.61	0.71	0.94	0.80
	Moderate Crosslinks, %	Phoenix	-0.76	NM	NM	NM		
		Roadwheel		-0.76				
	Weak Crosslinks, %	Phoenix	0.90	NM	NM	NM		-0.75
		Roadwheel			-0.49	-0.74	-0.74	-0.75
Wedge Compound	Total Crosslink Density	Phoenix	0.78	NM	NM	NM	0.66	0.99
		Roadwheel	0.86	0.84	0.65	0.48	0.88	0.59
	Strong Crosslinks, %	Phoenix	-0.91	NM	NM	NM		
		Roadwheel	0.68		0.52		0.93	
	Moderate Crosslinks, %	Phoenix		NM	NM	NM		
		Roadwheel	-0.42	-0.41			-0.70	
	Weak Crosslinks, %	Phoenix	0.93	NM	NM	NM		0.45
		Roadwheel	-0.51	0.44	-0.51		-0.67	-0.44

3.1.7 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 six tire types recovered from service in Phoenix has been reported.[3] While peel adhesion decreased with time in service, it was concluded that peel adhesion could not be used as a predictive measure because: (1) most samples did not fail at the wire/rubber interface, but failed cohesively in the rubber compound and (2) due to the physics of the peel sample, the actual force measured is a function of rubber properties such as thickness and modulus. Peel adhesion decreased as the tires accumulated service in the field, mileage on a roadwheel, or hours of exposure in an oven.

Table 13. Correlation of Peel Adhesion for Skim and Wedge Compounds

Tire Type / Measurement	Test Type	B	C	D	E	H	L
Peel Adhesion, Skim Compound	Phoenix	-0.71	-0.68	-0.81	-0.81	-0.69	-0.74
	Roadwheel	-0.60	-0.63	-0.91		-0.64	-0.73
	Oven	-0.87	-0.68		-0.82	-0.74	-0.83
Peel Adhesion, Wedge Compound	Phoenix	-0.48	-0.76	-0.96	-0.86	-0.73	-0.74
	Roadwheel	-0.76	-0.56	-0.81	-0.63	-0.79	-0.71
	Oven	-0.80	-0.68		-0.79	-0.82	-0.81

3.1.8 Two-Ply Laminate Fatigue Test

Two-Ply composite fatigue testing has been used to evaluate the effect potential construction changes (compound formulation, cord construction, cord angle, skim gauge, etc.) may have on tire durability. Unlike most traditional testing of rubber compounds and tire composites, which are subjected to extension forces (tensile strength and peel force) or compression forces (indentation hardness), the two-ply fatigue configuration stresses the rubber layer between the steel belts of the tire by subjecting it to shear forces. Shear is the primary type of force seen by the tire in service, especially in the belt region.

For the two-ply laminate fatigue test, nine circumferential strips were removed from the crown region of each tire: three each from approximately 0 degrees, 120 degrees, and 240 degrees, where the angular measurements reference the "DOT" in the Tire Identification Number on the sidewall. Each sample was approximately 1-inch wide by 12 inches long and 0.275-inches thick. There were three tires in this study which were manufactured with a cap ply (also known as an overlay) and/or belt edge strips: Type C - Goodyear (all), Type H - Pathfinder (some), and Type L - General (all). In each case, the cap ply/strip was removed prior to testing; otherwise the vast majority of the load would have been carried by this ply. A picture of a failed two-ply test sample is shown in Figure 2.

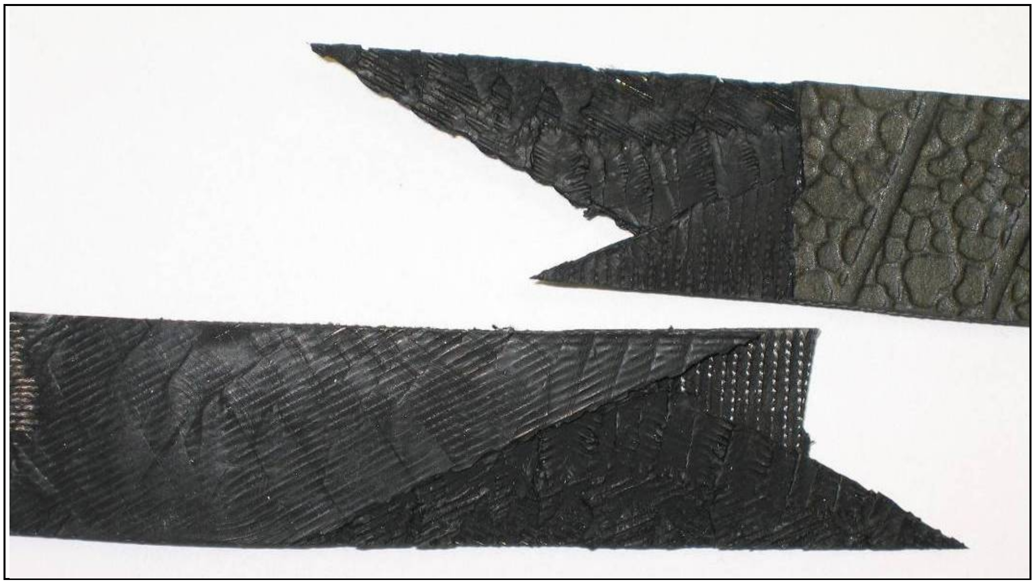

Figure 2. Picture of Two-Ply Test Sample Post-Test

The samples were then mounted in a load frame using specially made wedge grips to insure there was no slippage. An aluminum bar connected the servo-hydraulic actuator to a rod and bearing mounted on the load frame. There was no practical way to compare the test output of cycles to failure and average energy input to the sample since the critical property of shear strain could not be determined. A procedure was developed to determine the cycles to failure at 500 N-m of energy; a variable by which samples could be directly compared. Complete details and analysis has been reported separately.[19] Table 14 shows the moderate to strong trend for the cycles to failure at 500 N-m to decrease with increasing time in Phoenix or miles on a roadwheel test.

Table 14. Correlation of Two-Ply Fatigue Parameters

Tire Type / Measurement	Test Type	B	C	D	E	H	L
Cycles to Failure at 500 N-m	Phoenix	-0.81		NM	-0.71	-0.59	-0.86
	Roadwheel		-0.93	-0.64	-0.69	-0.79	-0.99

3.1.9 Shearography

Tires were examined by optical shearography for separations between components. The interior of the tire is illuminated by lasers and an image of the surface is obtained. The tire chamber is then exposed to a vacuum and a second image is obtained. If there are internal separations in the tire components the movement of the interior surface is obtained by the difference between the images. The test output is total area of separation (if any) in mm^2 from the procedure. Table 15 shows the correlation of increased separation as the tires accumulated mileage in Phoenix or time on a roadwheel test. A detailed analysis of the procedure and results has been reported.[17]

Table 15. Correlation of Shearography Separation Area

Measurement \ Tire Type	Test Type	B	C	D	E	H	L
Shearography Separation, mm^2	Phoenix	0.44	0.50		0.87	0.47	
	Roadwheel	0.59	0.47	0.70	0.55	0.85	

3.1.10 Fixed Oxygen Level

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). The agency examined the level of fixed oxygen in the skim, wedge, and innermost tread of six tire models collected from on-vehicle use in Phoenix after various amounts of service. These results confirmed the aerobic chemical reactions in the shoulder region of light vehicle tires during service. The level of fixed oxygen for tires tested on the roadwheel and in the oven was measured, and the correlation with time is shown in Table 16. As noted, the level of fixed oxygen increases in all components for tires in service. There was no correlation of fixed oxygen with increased time for tires tested on the roadwheel tests, while the skim and wedge compounds for some tires showed increased oxygen levels. The fixed oxygen level was used to model the extent of diffusion-limited oxidation in a number of tire models and has been reported separately.[4]

Table 16. Correlations of Fixed Oxygen Level

Component Measured \ Tire Type	Test Type	B	C	D	E	H	L
Skim Compound	Phoenix	0.85	0.66	0.69	0.54	0.68	0.51
	Roadwheel						
	Oven		0.52			0.70	0.75
Wedge Compound	Phoenix	0.75	0.90	0.46	0.58	0.72	
	Roadwheel						
	Oven		0.65	-0.44		0.60	0.65
Innermost Tread Compound	Phoenix	0.55	0.58	0.55		0.58	0.44
	Roadwheel						
	Oven			-0.46			

3.2 Analysis of Variance and Linear Regression Analysis

The Pearson R correlations indicated whether the property measured showed a general tendency to increase or decrease over time, whether in-service or in laboratory testing. The data was then analyzed using one-way analysis of variance testing to identify specific variables that influenced changes in each property using the variables in Table 17. Variables which had significant effects and the direction of the effect were noted. Subsequent SAS GLM and STEPWISE procedures were used to produce linear regression models when possible. Each tire type was analyzed separately and those models with R^2 values above 0.80 from the initial GLM procedure will be commented on and/or analyzed further. Note that the Phoenix tires were modeled only versus time in service for comparison. As reported previously,[17] the best models for rubber properties of the Phoenix tires involved both time in service and mileage on the tire.

Table 17. Terms Used in Linear Regression Model

Test	Model Terms Used for Regression	Notes
Phoenix-retrieved Tires	Time in Service, weeks	-
Roadwheel Tires	Time on Roadwheel, hours	-
	Test	LTDE = $50\%N_2/50\%O_2$ Fill Gas PEND = Air Fill Gas
Oven Tires	[k]Time in Oven, hours	8 weeks, 12 weeks or 180 days
	Break-in	None or 24 hours @ 75 mph filled with $50\%N_2/50\%O_2$
	Temperature	12 weeks at 55ºC, 8 weeks at 65ºC; permeability tires 180 days at 70ºC
	Fill Gas	Permeability tires filled with Air, all others with $50\%N_2/50\%O_2$

The ANOVA models were considered significant if the Probability > F was less than 0.05 and individual terms were considered significant if the Probability > |t| was less than 0.05. In the following tables numbers were reported only if the terms were considered significant. For terms that were not significant, only the sign of the term was reported for comparison between types.

3.2.1 Shore A Hardness

The models for the liner, tread, and wedge compound were not statistically significant for any tire type on the roadwheel tests or from oven exposure. The skim compound for tire types B and E gave statistically significant models for oven exposure as shown in Table 18. The hardness of the type B tire increased most significantly with temperature, while the type E tire hardness had a significant increase over time in the oven. For all tires except the type H, the effect of the use of $50\%N_2/50\%O_2$ fill gas versus air, and the effect of the 24-hour break-in, was to increase the hardness of the skim compound. For the Phoenix tires, the hardness increased with increasing time in service, except for the type H tire, although the models were not statistically significant.

[k] Covariant with temperature and fill gas

For tire type H recovered from Phoenix the hardness decreased as time in service increased. Further analysis indicated that this phenomenon was likely due to the use of a phenol-formaldehyde resin in tire type H compounds. A detailed report has been issued separately.[5]

Table 18. ANOVA and Linear Regression Terms for Shore A Hardness

Tire Type			B	C	D	E	H	L
Component Measured	**Test**	**Variable**						
Innerliner	Roadwheel	[l]Pr > F	0.010	< 0.001	0.045	0.410	0.137	NM
		[m]Model R^2	0.84	0.82	0.67	0.08	0.46	NM
		[n]Hours	+ 0.023	+ 0.016	+ 0.023	+	-	NM
		Test						NM
	Oven	Pr > F	< 0.001	<0.001	<0.001	<0.001	<0.001	<0.001
		Model R^2	0.65	0.77	0.23	0.63	0.63	0.80
		Temperature	+	-	+	-	-	-
		Hours	+	-	-	+	-	+
		Fill Gas	AIR > 50/50	50/50 > AIR	AIR > 50/50	50/50 > AIR	50/50 > AIR	50/50 > AIR
		Break-in	BI > no BI	BI > no BI	no BI > BI	BI > no BI	BI > no BI	BI > no BI
Tread	Roadwheel	Pr > F	0.002	0.084	< 0.001	0.423	0.048	0.186
		Model R^2	0.43	0.09	0.61	< 0.01	0.16	0.07
		Hours	+	+	-	+	+	+
	Oven	Pr > F	NM	0.388	NM	NM	0.423	0.549
		Model R^2	NM	0.13	NM	NM	0.33	0.20
		Hours	NM	+	NM	NM	-	+
Skim	Roadwheel	Pr > F	0.053	0.272	0.448	0.147	0.158	0.205
		Model R^2	0.28	0.10	0.12	0.14	0.43	0.19
		Hours	+	+	-	+	-	+
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.96	0.29	0.54	0.92	0.34	0.80
		Temperature	+0.327	-	-	+	+	-
		Hours	-	+	+	+ 0.003	+	-
		Fill Gas	50/50 > Air	50/50 > Air	50/50 > Air	50/50 > Air		50/50 > Air
		Break-in	BI > no BI	BI > no BI	BI > no BI	BI > no BI		BI > no BI

3.2.2 <u>Indentation Modulus</u>

Indentation modulus measurements were taken from cut tire sections for selected tires to map the changes in modulus of the tire over time. The method used a nano-indenter in accordance with the methodology contained in RC&T, 74, No.3, pp. 428ff (2001) 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). The nano-indenter and an example tire section are shown in Figure 3. The measurements were carried out on the locations of the tire indicated in

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[l] ANOVA models were considered significant if Probability > F values were less than 0.05.
[m] Regression models were considered significant if R^2 values were greater than 0.85, and very significant if R^2 values were greater than 0.95.
[n] Significant terms with Probability > |t| values of less than 0.05 are reported with values. Only the sign of terms with larger Probability > |t| values are reported.

Table 19. Since each tire type had different components (e.g., some had nylon overlay strips, while others did not), the ANOVA and linear regression models were only calculated for the wire coat compound in the critical area of the belt-edge. These are shown in Table 20. All types showed a tendency to harden during oven aging, although only the model for the type E tires was statistically significant. Parallel to the results for Shore A hardness, the type H and type L tires showed a tendency to soften during roadwheel testing and for tires retrieved from Phoenix service, while all other types tended to harden.

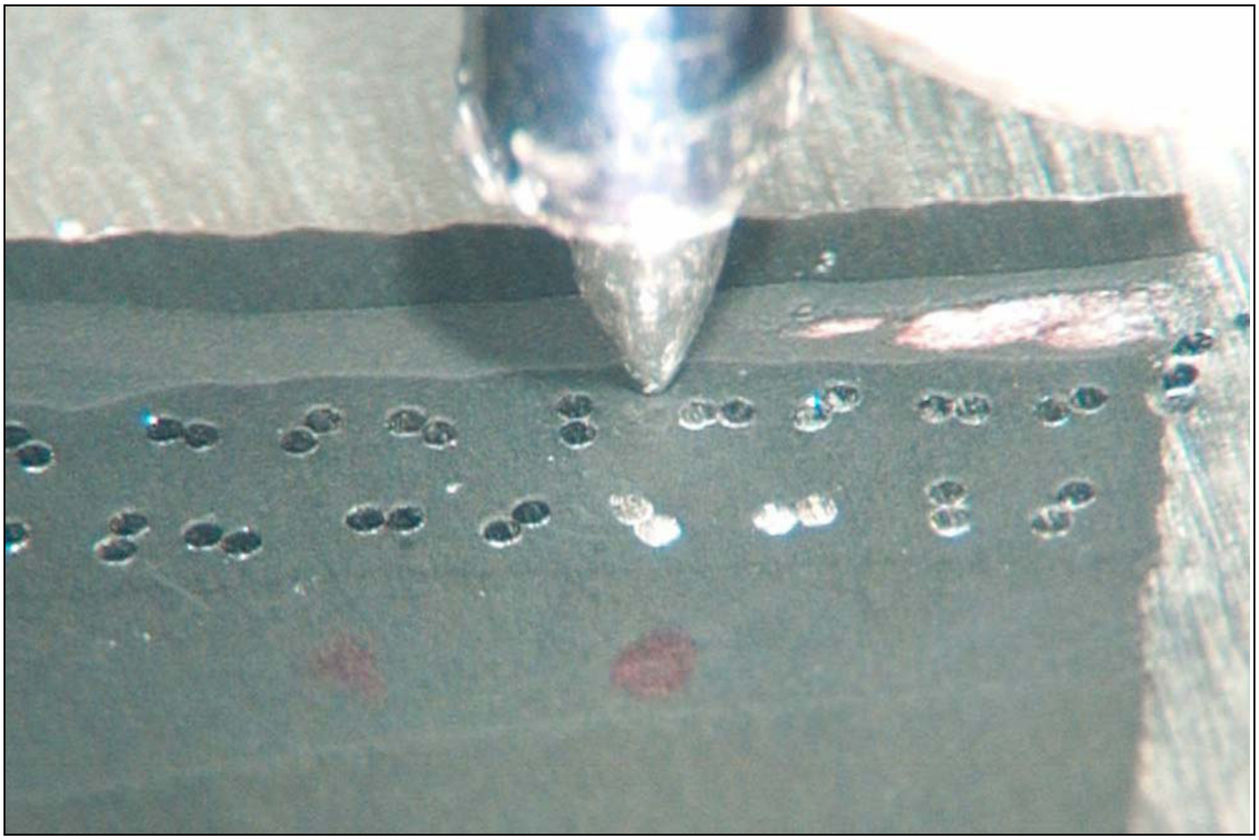


Figure 3. Example Micro-Indentation Modulus

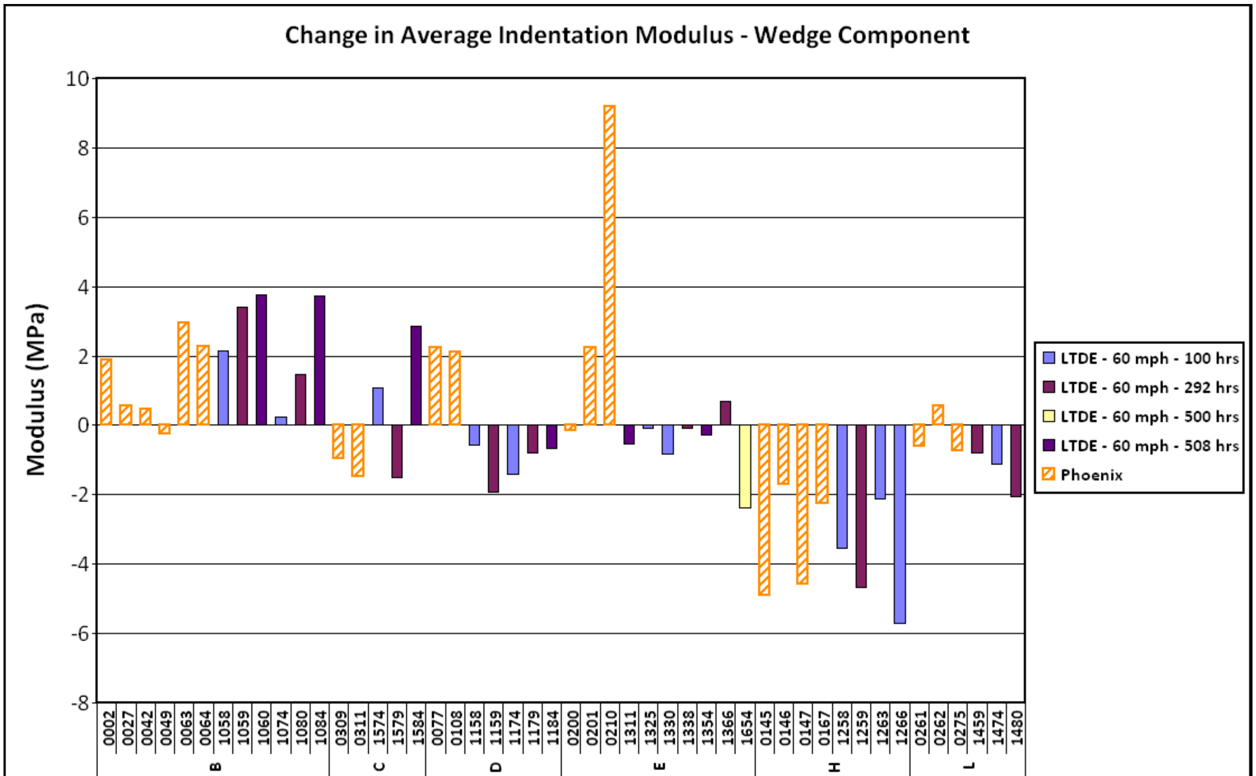
Table 19. Components Measured by Indentation Modulus

Component
Innerliner
Squeegee
Plycoat
Fabric Gumstrip
Wedge
Skim
Nylon Overlay Compound
Overlay Gumstrip
Tread Base
Tread Compound
Fabric Toeguard
Apex
Chafer
Sidewall Compound

Table 20. ANOVA and Linear Regression Terms for Indentation Modulus of Belt Coat Compound in Belt-Edge Area

Tire Type		B	C	D	E	H	L
Test	**Variable**						
Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.72	0.37	0.19	0.08	0.05	0.12
	Hours	+ 0.006	+ 0.003	+	+	-	-
	Test	LTDE > PEND		LTDE > PEND			
Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.002
	Model R^2	0.81	0.82	0.30	0.92	0.75	0.48
	Temperature	+	+	+	+0.219	+	-
	Hours	+	+	+	-	+	+
	Fill Gas						
	Break-in	BI > no BI				no BI > BI	

The profiles of the average indentation modulus change of the wedge compounds for the roadwheel-tested and Phoenix service tires are shown in Figure 4. The results are shown as MPa change from the value for a new tire of the same model. The results for tire types B, and H were directionally similar to those of the Phoenix tires. That is the type B tires showed consistent increases in modulus, while the type H tires showed decreases in modulus with increasing time in service in Phoenix or on the LTDE or P-END roadwheel test. There were mixed results for the type C tires and L tires which showed little change in service in Phoenix. For the type D and E tires the Phoenix tires increased in modulus while the roadwheel tested tires decreased in modulus.



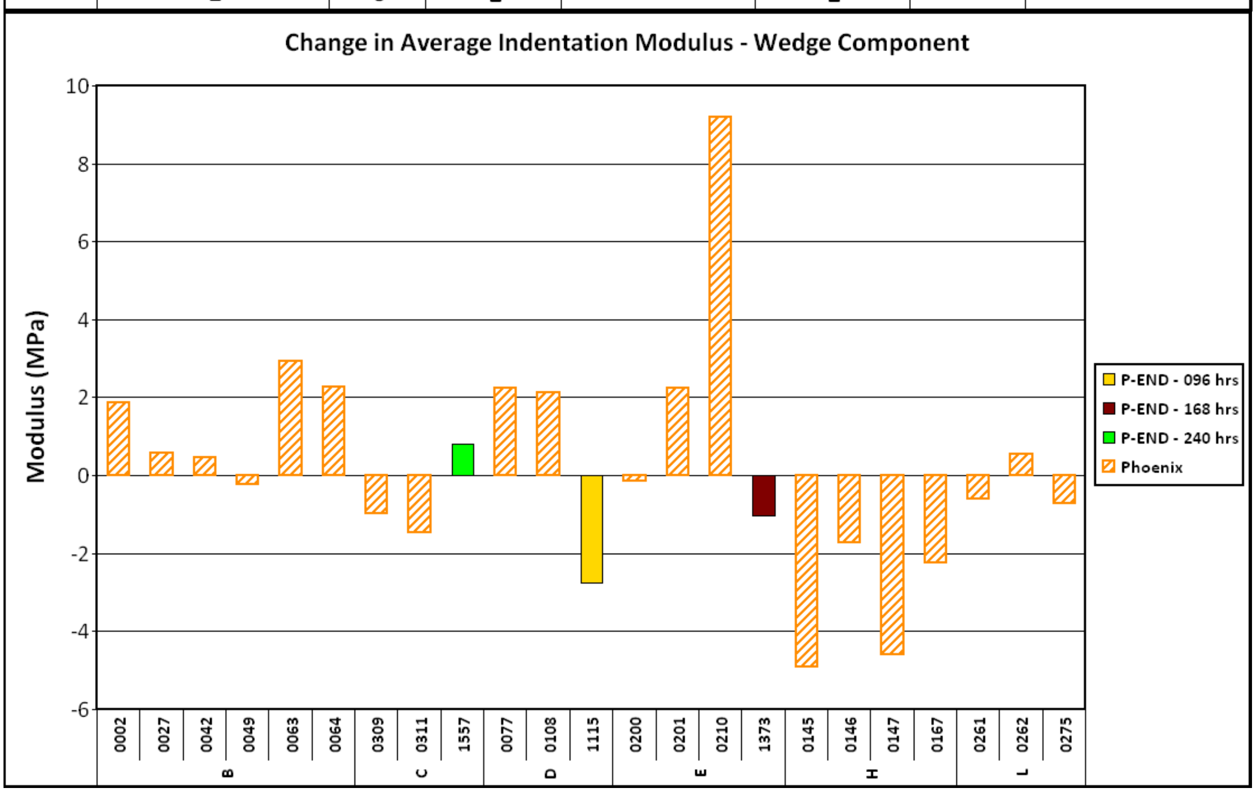


Figure 4. Changes in Indentation Modulus for Roadwheel Tests and Phoenix Service Tires

The profiles of the average indentation modulus change of the wedge compounds for the oven-aged and Phoenix service tires are shown in Figure 5. The results for tire types B, D, and E were directionally similar to those of the Phoenix tires, showing consistent increases in modulus. The type C tires decreased slightly in Phoenix service and the type L tires showed no change in modulus, but both types increased significantly during oven aging. The type H tires showed large decreases in modulus with increasing time in service in Phoenix, but hardened significantly during oven aging. The softening in service was found to be due to the breakdown of the 3-dimensional structure of a reinforcing resin used in the type H tire.[5] A 24-hour break-in of the tire at 75 mph on a roadwheel caused a similar breakdown of the resin structure, and the modulus decreased even after oven aging.

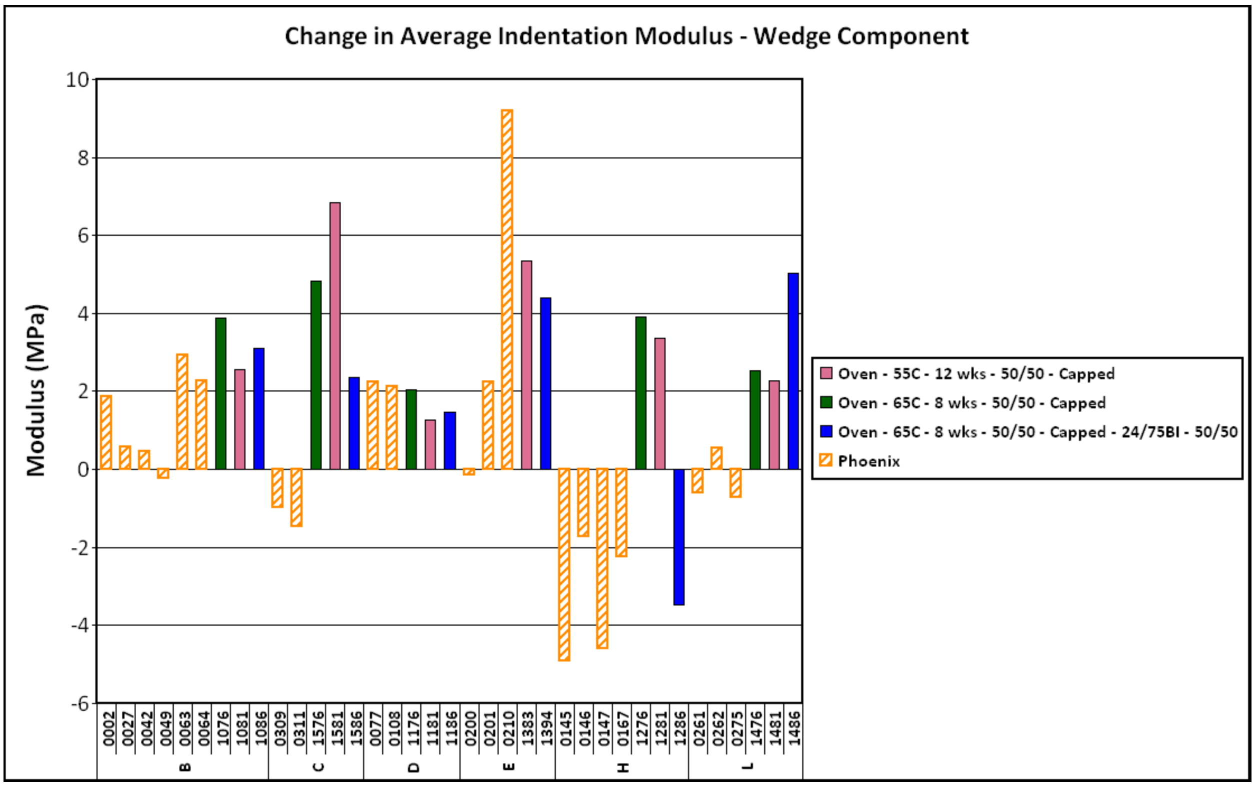


Figure 5. Changes in Indentation Modulus for Oven-Aged and Phoenix Service Tires

The profiles of the indentation modulus for each tire type and service condition are shown in Appendix 3. An example is shown in Figure 6 for the Phoenix-retrieved type B tires. The inner surface of the tire cross-section is assigned a zero position and the end of each component is indicated. The modulus of all components for this tire type tended to increase with increased time in service in Phoenix. Note particularly the values for the wedge compound between 4 and approximately 5 mm from the inner surface.

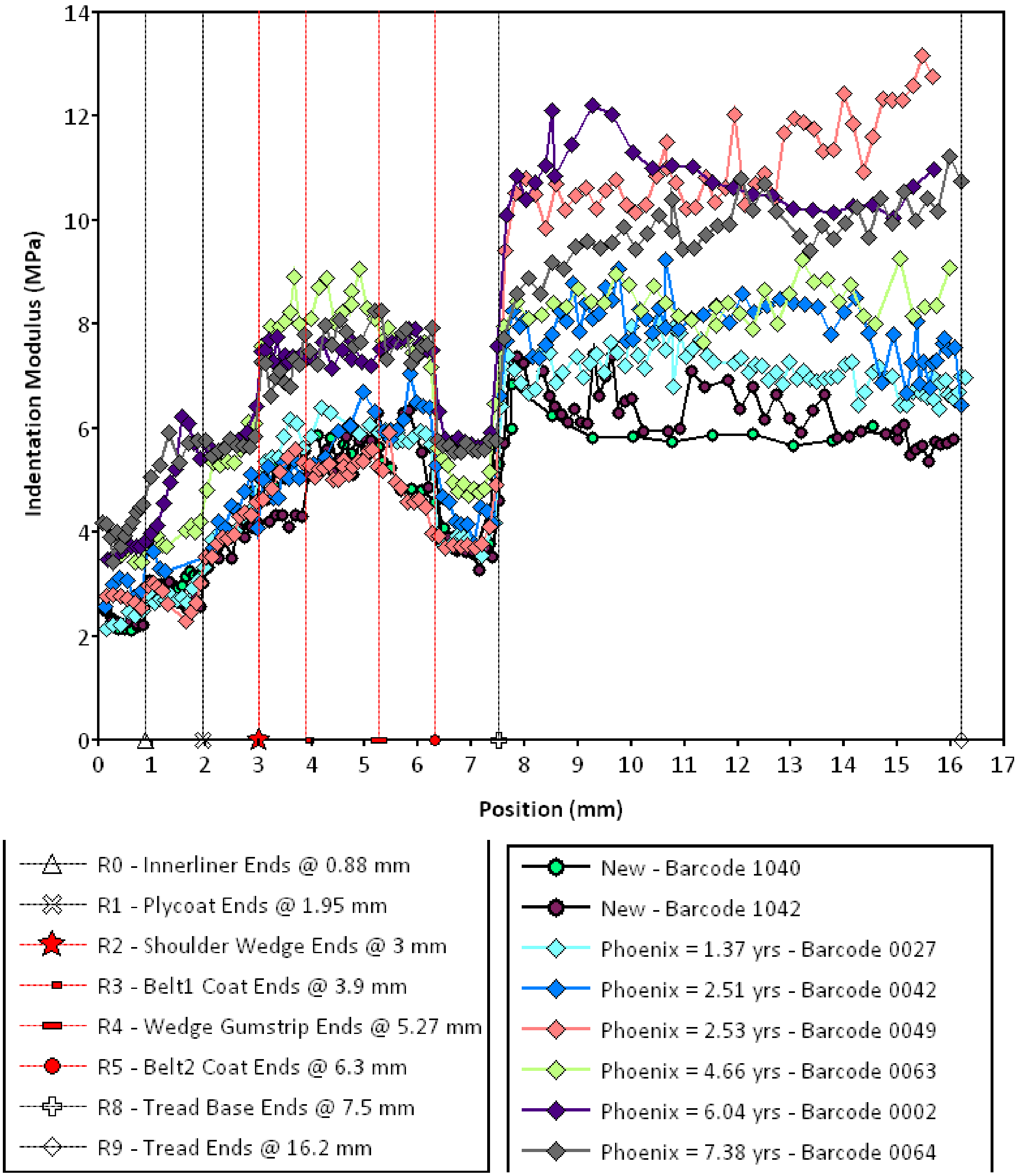


Figure 6. Modulus Profile of Type B Phoenix-Retrieved Tires

Complete profiles of each tire type are shown in Appendix 3, and are generally described in the following sections.

3.2.2.1 LTDE Test

At 100 hours, the modulus of most components of the type B tire increased to approximately the modulus of tires with 1 to 2 years of Phoenix service. At 292 hours most components approximated the modulus of tires with 4 to 6 years of service in Phoenix. At 508 hours the modulus of the wedge region had increased beyond that seen in tires with 6 to 7 years of service in Phoenix while the outermost tread showed decreasing modulus values indicating reversion of the compound.

For the type C tire, at 100 hours the wedge and tread modulus increased to approximately the level of a tire with 1.8 years of service in Phoenix. At 292 hours the modulus of the wedge and tread compound increased to approximately the level of a tire with 5.5 years of service. At 508 hours the modulus of the belt-coat, wedge, and tread compounds increased beyond that of tires from Phoenix service. The tread modulus was still much lower than that of a spare tire from service in Phoenix.

For the type D tire, at 100 hours, the modulus of the belt-coat and wedge compounds increased to approximately the level of a tire with 1.5 years of Phoenix service. At 508 hours the modulus increased to approximately the level of a tire the 4 years of service in Phoenix.

For the type E tire with the VN plant (Joliette, Quebec) production code, at 100 hours, the modulus of the belt-coat and wedge compounds approximately matched that of a tire with 0.5 years of Phoenix service. Even at 508 hours the modulus of the wedge and belt-coat compound was significantly less than that of a tire with 3 years of Phoenix service. However it was approximately that same as a type E tire with a W2 plant (Wilson, NC) production code which was retrieved from Phoenix with 3.3 years of service.

For the type H tire, at 292 hours, the modulus of the belt-coat and wedge compound decreased to approximately the level of a tire with 2 to 3 years of Phoenix service. The modulus of the tread only increased slightly during roadwheel testing. At 292 hours the modulus was still below the level of a tire with 1.4 years of Phoenix service.

For the type L tire, the directional changes in both the belt-coat and tread area were opposite that seen during Phoenix service. The modulus of the belt-coat and wedge compound increased significantly after 292 hours of roadwheel testing while there was little change in the modulus of the belt coat and wedge compounds for these tires during Phoenix service. The hardness of the tread compound did not increase significantly during roadwheel testing but did increase significantly during Phoenix service.

3.2.2.2 P-END Test

Due to the high rate of premature tire failures on the roadwheel, indentation modulus test data was only available for the type C and E tires. At 100 hours, the wedge and tread compounds of the type C tires had modulus values approximately equal to a tire with 5.5 years of service in Phoenix. For the type E tire (produced at Joliette, Quebec), after 168 hours of testing the modulus of the belt-coat and wedge compounds were decreased from that of the new tire, while the modulus of the tires in Phoenix service increased during service.

3.2.2.3 Oven Aged Tires

The modulus of the wedge and tread compounds of the type B tires increased significantly during oven aging for 8 weeks at 65ºC or 12 weeks at 55ºC. The modulus after oven aging was higher than that seen in tires with 6 to 7 years of service in Phoenix.

For the type C tires, the modulus of the compounds, particularly the belt coat compound, increased to a level significantly higher than that of a tire with 5.5 years of service in Phoenix by aging 8 weeks at 65ºC or 12 weeks at 55ºC. The modulus of the belt-coat compound was decreased by the break-in cycle on a roadwheel prior to oven aging. After 8 weeks at 65ºC, the hardness of the tread compound increased to approximately the level of the 10.7-year-old spare tire from Phoenix.

For the type D tires, after 12 weeks at 55ºC, the modulus increased to approximately that of a tire with 1.5 years of Phoenix service. After 8 weeks at 65ºC, the modulus was approximately that of a tire with 4 years of Phoenix service.

For the type E tires, with VN production plant code, oven aging for 8 weeks at 65ºC or 12 weeks at 55ºC increased the modulus of the belt-coat and wedge compound to approximately the level of a tire with 3 years of Phoenix service. The tread compound was significantly higher than that of a tire with 3 years of Phoenix service.

Unlike the other tire types, the type H tires tended to show decreases in modulus of the belt-coat and wedge compounds during service in Phoenix. Without a break-in cycle prior to oven aging, the modulus of the belt-coat and wedge compound increased significantly. A tire aged 8 weeks at 65ºC after a 24-hour break in at 75 mph showed only slight increases in modulus values. Conversely, the hardness of the tread compound increased significantly during Phoenix service, but only increased to approximately the level of a tire with 1.4 years of Phoenix service during oven aging.

The modulus of the belt-coat and wedge compounds of the type L tires increased significantly during oven aging, while the modulus of these components in Phoenix service were relatively unchanged. A break in period prior to oven aging did not decrease the modulus of the aged type L tires. Oven aging for 12 weeks at 55ºC increased the modulus of the tread compound to approximately that of a tire with 1 year of Phoenix service. Oven aging for 8 weeks at 65ºC increased the modulus of the tread compound to approximately that of a tire with 2.6 years of Phoenix service.

3.2.3 Tensile and Elongation of Skim-Coat Compound

The analysis of variance and regression of the modulus, tensile and elongation of the skim-coat compound is shown in Table 21. For all tire types, the elongation to break decreases and the modulus at high strain increases. This is parallel to the data obtained from tires in service in Phoenix.

Table 21. ANOVA and Linear Regression Terms for Modulus, Tensile, and Elongation of Skim-Coat Compound

Tire Type			B	C	D	E	H	L
Measurement	**Test**	**Variable**						
Modulus @ 50% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.41	0.26	0.49	0.36	0.01	0.28
		Hours	+ 0.0013	+ 0.0015	+ 0.0014	+ 0.0023	-	+ 0.0027
		Test						
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.70	0.49	0.56	0.77	0.61	0.73
		Temperature	-	+ 0.0155	+	+ 0.0282	+ 0.0210	+ 0.0343
		Hours	+ 0.009	- 0.0002	+ 0.0002	+ 0.0005	+ 0.0002	-
		Fill Gas	[o]50/50 > AIR		50/50 > AIR			
		Break-in	BI > no BI		BI > no BI	BI > no BI		BI > no BI
Modulus @ 100% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.58	0.35	0.43	0.42	0.06	0.17
		Hours	+ 0.0029	+ 0.0029	+ 0.0022	+ 0.0035	-	+ 0.0034
		Test		P-END > LTDE				P-END > LTDE
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	<0.001	< 0.001	< 0.001
		Model R^2	0.69	0.62	0.47	0.61	0.75	0.39
		Temperature	+ 0.0137	+ 0.295	+	-	+ 0.0369	+ 0.0537
		Hours	+ 0.0010	- 0.0002	+	+	+ 0.0003	-
		Fill Gas	50/50 > AIR		50/50 > AIR			
		Break-in	BI > no BI		BI > no BI	BI > no BI	BI > no BI	no BI > BI
Modulus @ 200% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.55	0.38	0.29	0.38	0.01	0.08
		Hours	+ 0.0062	+ 0.0045	+ 0.0033	+ 0.0060	+	+
		Test	P-END > LTDE	P-END > LTDE		P-END > LTDE		P-END > LTDE
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.82	0.57	0.46	0.61	0.67	0.07
		Temperature	+ 0.0443	+ 0.0604	+ 0.0311	+ 0.0761	+ 0.0573	+
		Hours	+ 0.0011	- 0.0006	- 0.0008	+	+	+
		Fill Gas	50/50 > AIR		50/50 > AIR			
		Break-in	BI > no BI					
Modulus @ 300% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.53	0.39	0.01	0.17	0.02	0.20
		Hours	+ 0.0085	+ 0.0055	+	+ 0.0076	-	-
		Test	P-END > LTDE	P-END > LTDE		P-END > LTDE		

[o] For all measures, compares the predicted mean value of the condition noted (e.g. “BI > no BI” denotes that a break-in period produces a higher modulus value than tires with no break-in)

	Oven	Pr > F	< 0.001	< 0.001	<0.001	NM	NM	NM
		Model R^2	0.53	0.55	075	NM	NM	NM
		Temperature	+ 0.0495	+ 0.0893	+ 0.0996	NM	NM	NM
		Hours	+	- 0.0015	- 0.0045	NM	NM	NM
		Fill Gas				NM	NM	NM
		Break-in				NM	NM	NM
Tensile	Roadwheel	Pr > F	< 0.001	< 0.1	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.01	0.15	0.35	0.18	0.04	0.16
		Hours	-	- 0.0092	- 0.0105	- 0.0112	-	- 0.0153
		Test						
	Oven	Pr > F	< 0.001	<0.001	<0.001	<0.001	<0.001	<0.001
		Model R^2	0.31	0.36	0.32	0.55	0.65	0.54
		Temperature	-	-	+	- 0.0778	- 0.0330	-
		Hours	- 0.0316	- 0.0033	-	- 0.0019	- 0.0035	- 0.0029
		Fill Gas			AIR > 50/50			
		Break-in			no BI > BI			no BI > BI
Elongation at Break	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.29	0.33	0.55	0.23	013	0.23
		Hours	- 0.205	- 0.274	- 0.261	- 0.263	- 0.303	- 0.281
		Test		LTDE > P-END				
	Oven	Pr > F	< 0.001	<0.001	<0.001	<0.001	<0.001	<0.001
		Model R^2	0.71	0.49	0.41	0.62	0.85	0.67
		Temperature	- 2.002	- 2.575	- 1.533	- 0.033	- 0.060	- 0.036
		Hours	- 0.050	-	+	- 0.274	- 1.820	- 2.053
		Fill Gas			AIR > 50/50			
		Break-in	no BI > BI		no BI > BI			no BI > BI

Ahagon et al. have shown that a slope near –0.75 for the plot of log(elongation at break, %) versus log(modulus at 100% strain, MPa) is indicative of thermo-oxidative aging of rubber compounds.[7,8] Figure 7 to Figure 12 show the modulus at 100 percent strain and percent elongation at break versus time for those properties obtained for tires aged in the laboratory and retrieved from service in Phoenix. Note that the mean value of the log(elongation) is plotted along with the 95-percent confidence level for the log(elongation) as a reference to the amount of variability in the data. The actual 95-percent confidence intervals include the variation in the log(modulus) as well and therefore are elliptical regions around the mean values.

For the type B tires, the average modulus increase and elongation decrease for either 6 weeks at 65ºC or 12 weeks at 55ºC are approximately equal to that for tires with 4 to 6 years of service in Phoenix. The average levels of modulus and elongation for the roadwheel-tested tires approximate that of tires with 1 to 2 years of service in Phoenix.

For the type C tires, oven aging for 6 to 8 weeks at 65ºC did not increase the modulus beyond that of tires with 2 years of service in Phoenix but did decrease the elongation to approximately the levels of tires with 4 to 6 years of service in Phoenix. Roadwheel aging produced only nominal changes in modulus or elongation which were less than that found in a tire with 2 years of service in Phoenix.

For the type D tire, roadwheel testing or oven aging for 3 weeks at 65ºC approximated the levels of modulus and elongation for the Phoenix tire with 2 years of service available for comparison.

The type E tires showed no significant changes in 100 percent modulus up to 3 years of service in Phoenix, while both roadwheel aging and oven aging produced significant increases in modulus for the tires. Elongation was reduced by the roadwheel tests or by 3 weeks of oven aging at 65ºC to approximately the level found in Phoenix tires with 3 years of service.

For the type H tires, oven aging for 6 to 8 weeks at 65ºC or 12 weeks at 55ºC increased the modulus and decreased the elongation to approximately that found in a tire with 3 years of service in Phoenix. The changes were still significantly less than that found in a tire with 5 years of Phoenix service.

For the type L tires oven aging between 3 and 6 weeks at 65ºC or 12 weeks at 55ºC produced modulus and elongation values approximately equal to those found in a tire with 5 years of service in Phoenix. Roadwheel testing produced changes approximately equal to those of tires with 2 years of service in Phoenix.

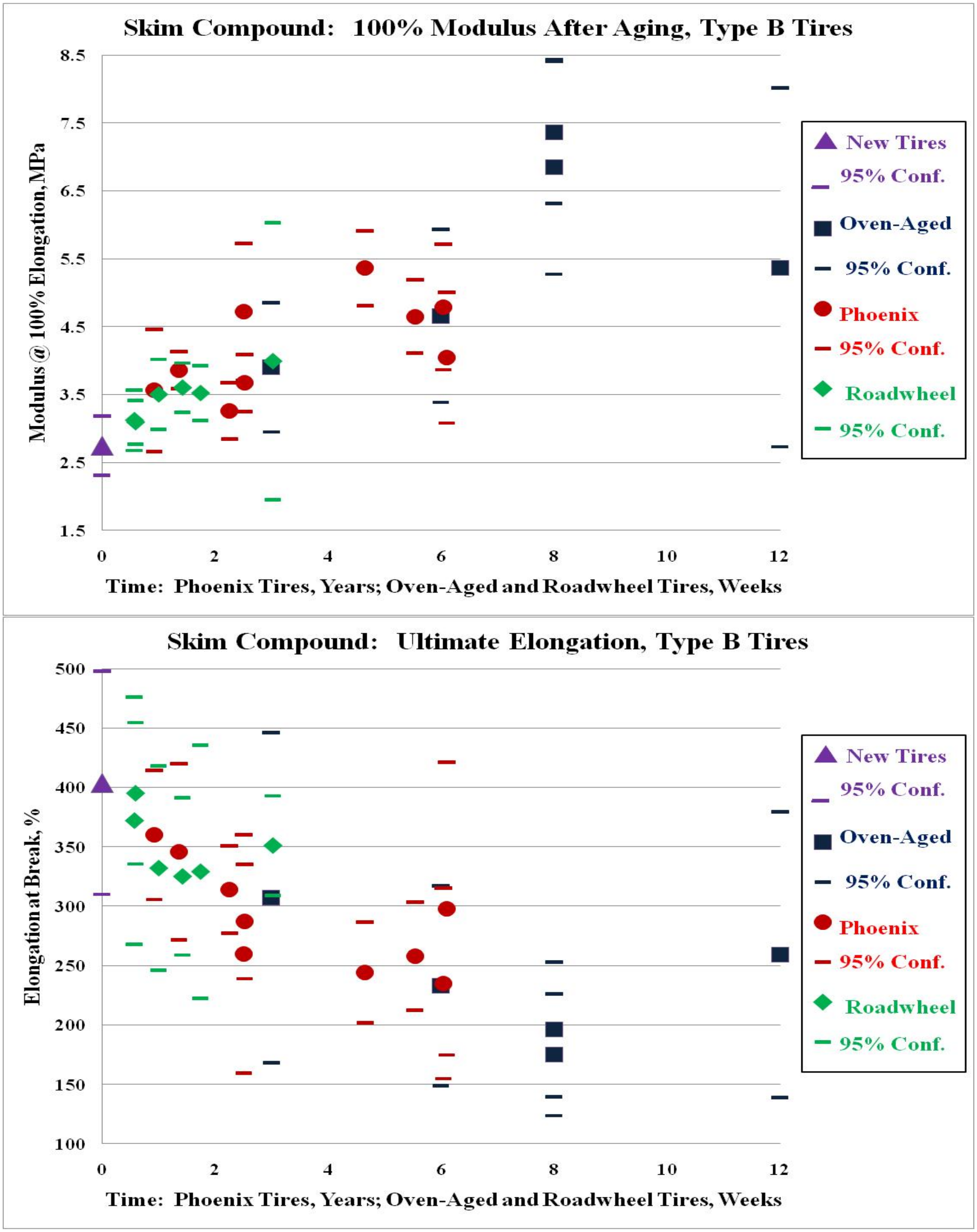


Figure 7. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type B Tires

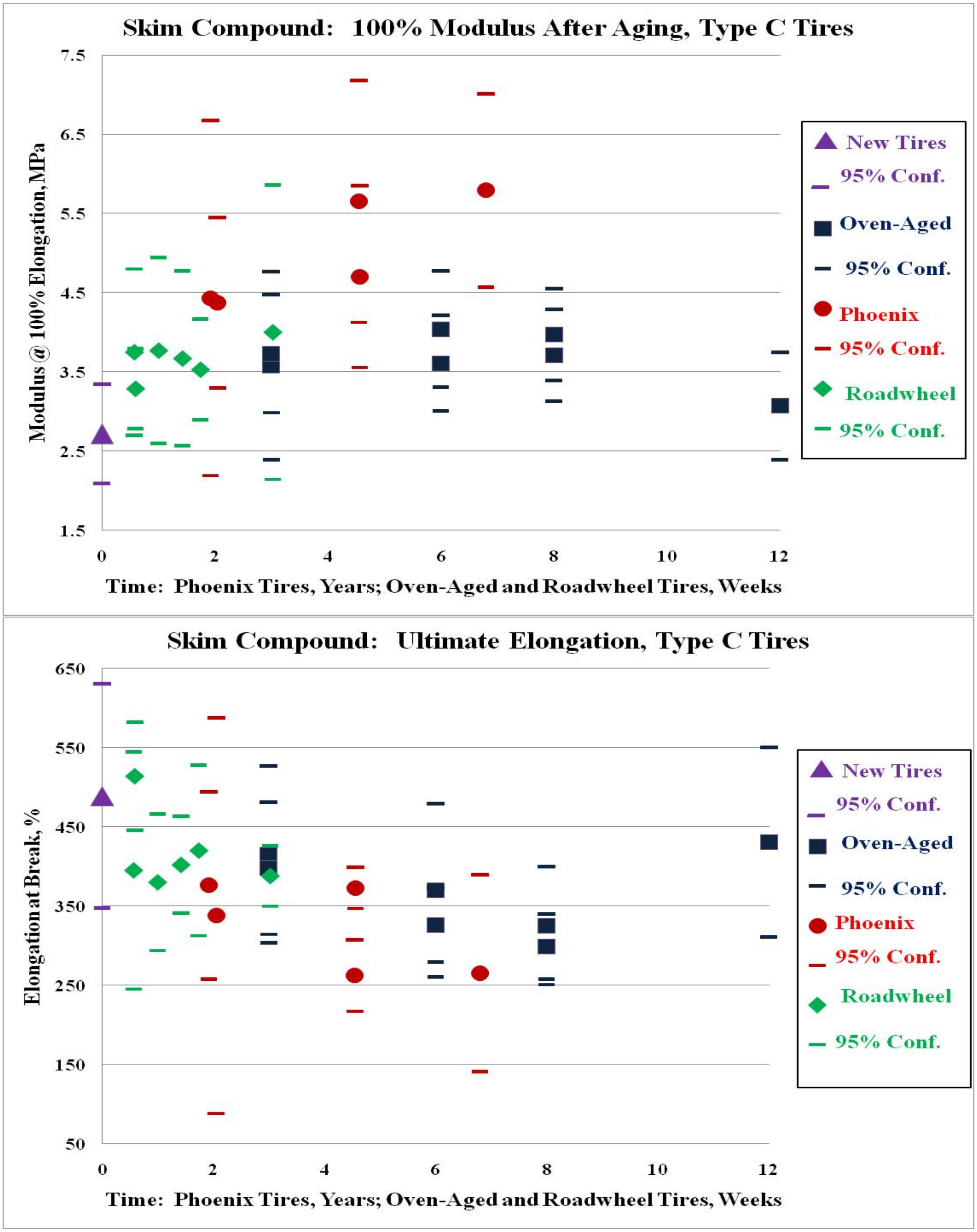
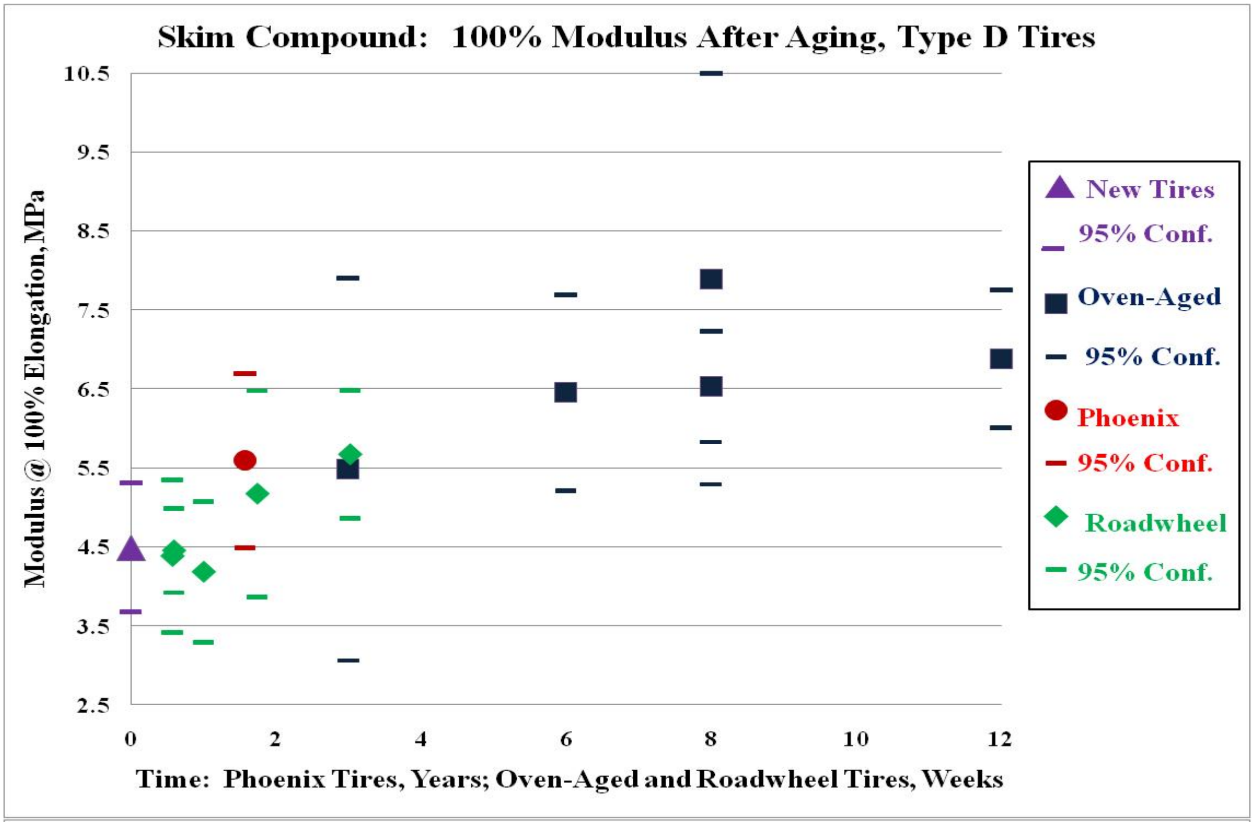


Figure 8. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type C Tires



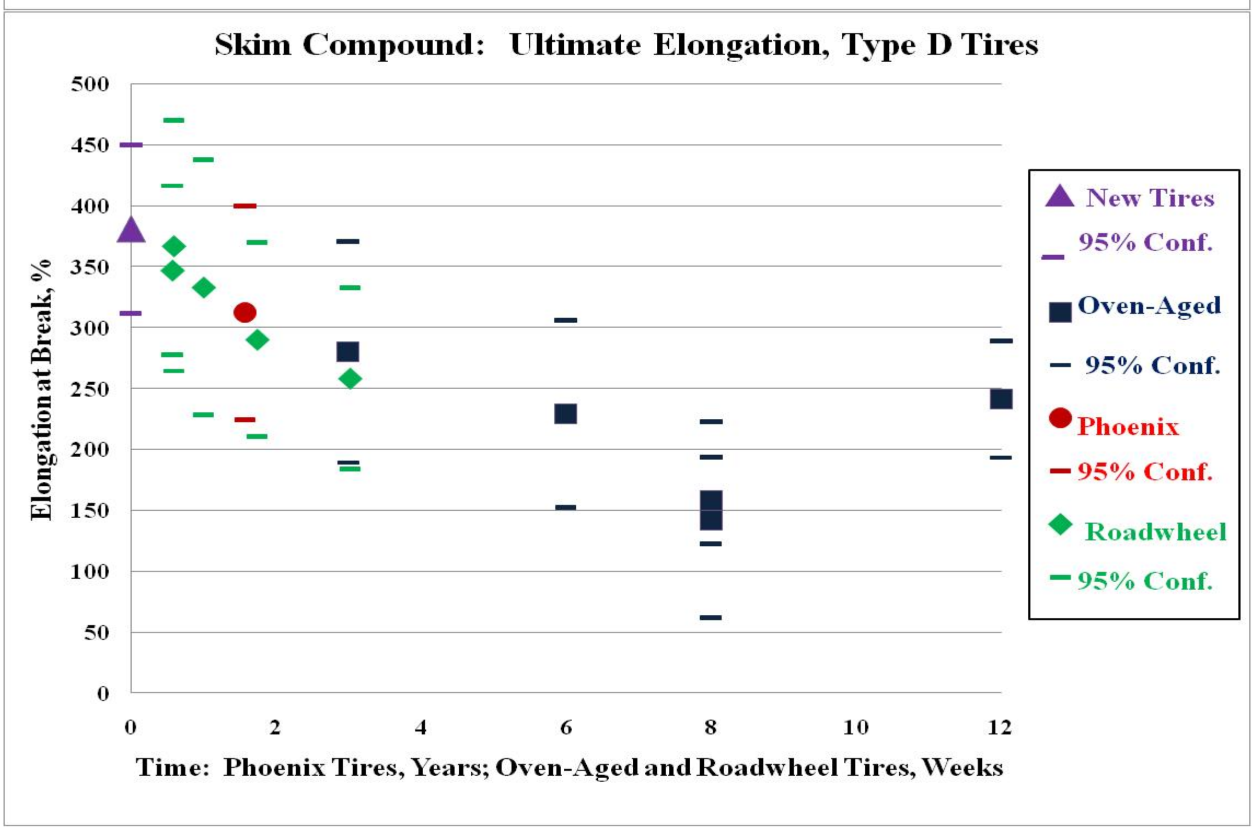


Figure 9. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type D Tires

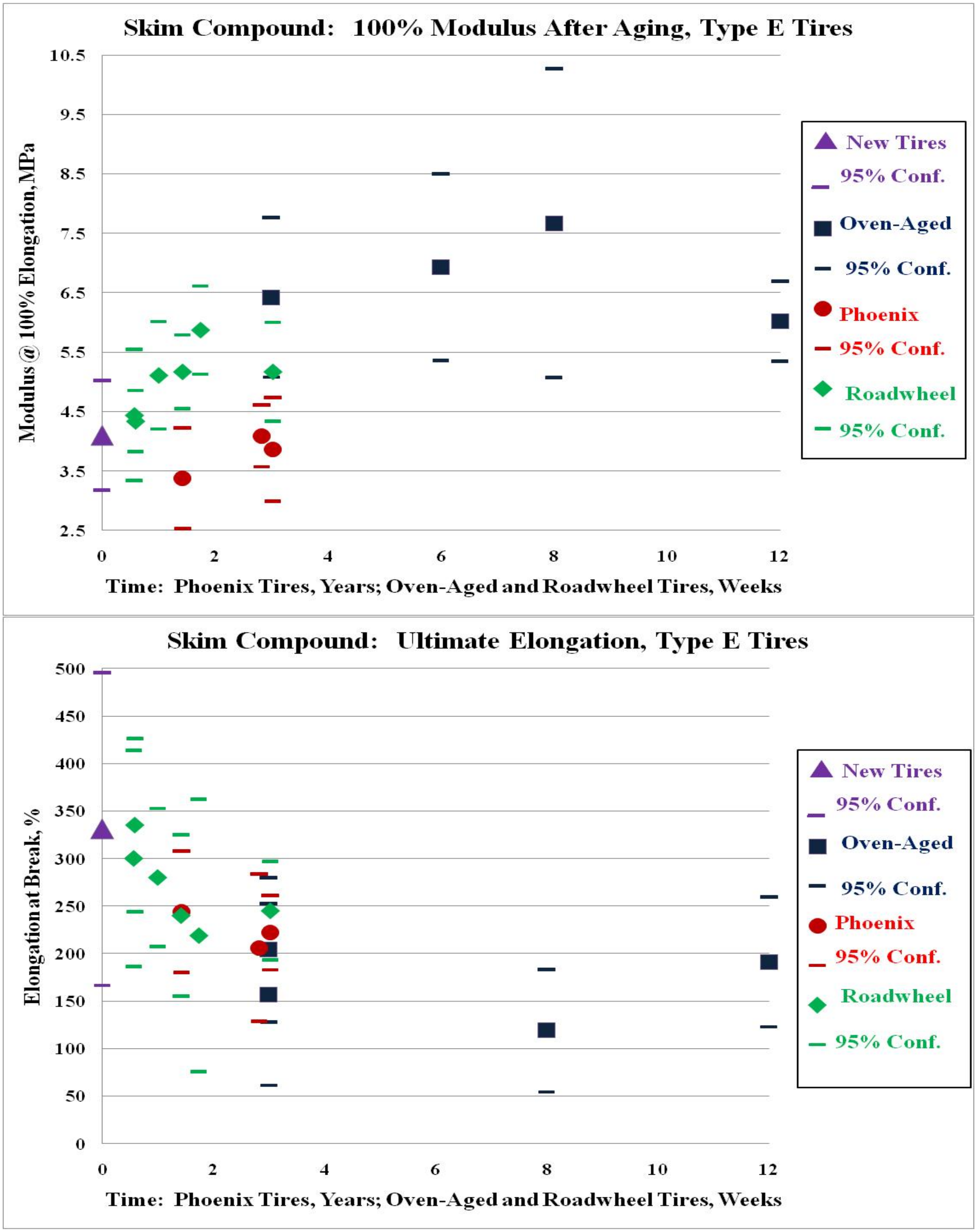
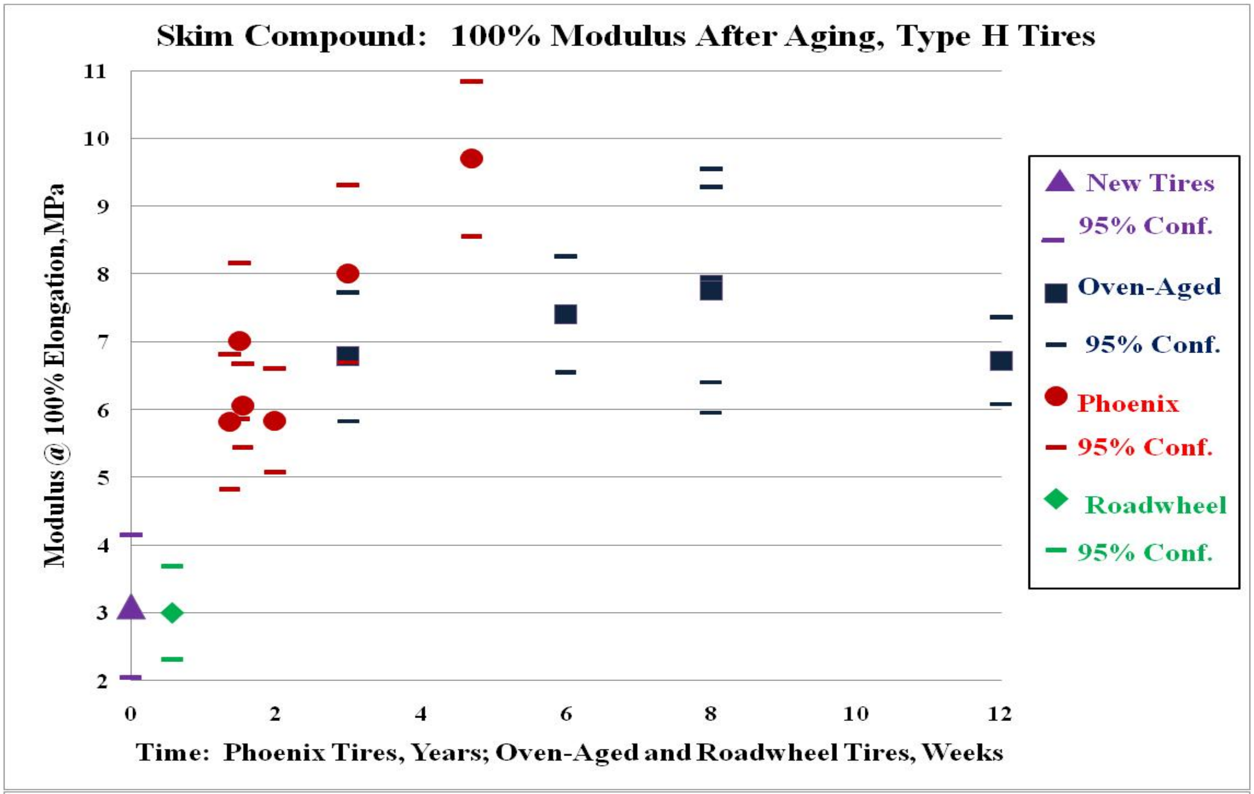


Figure 10. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type E Tires



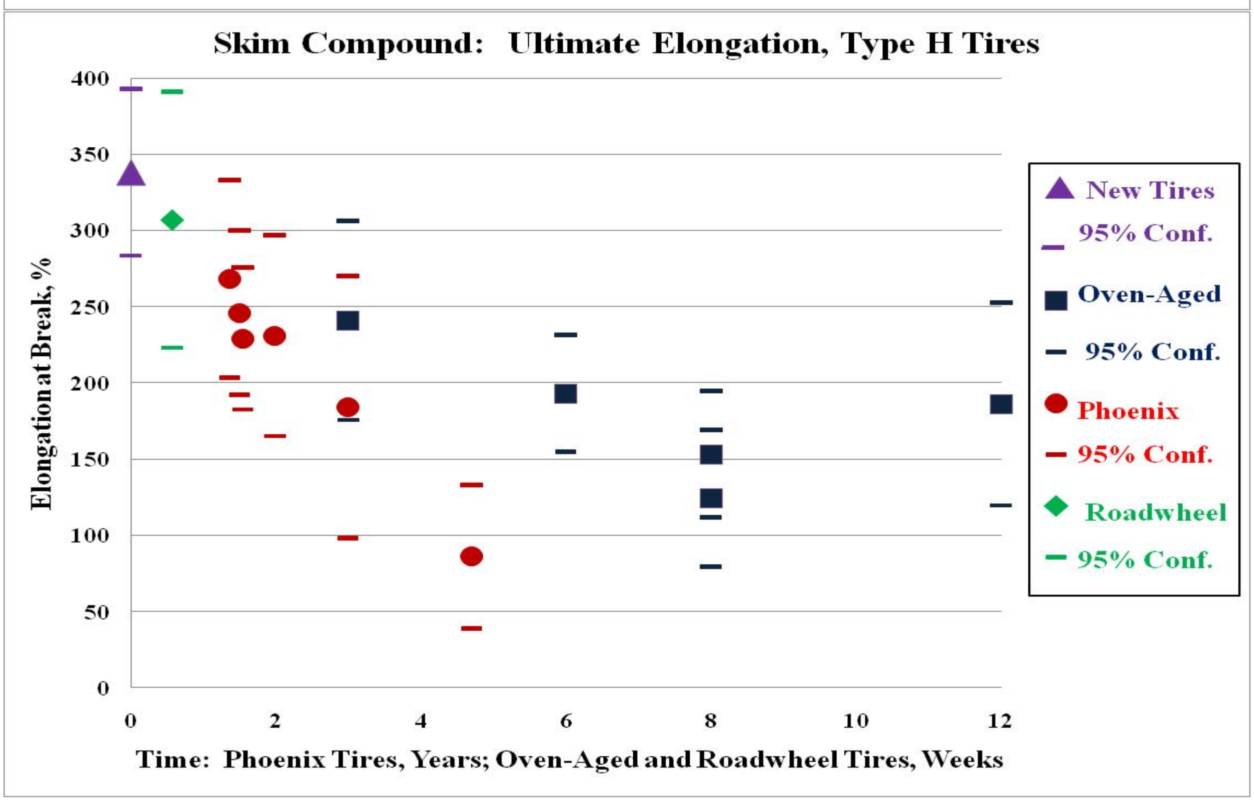
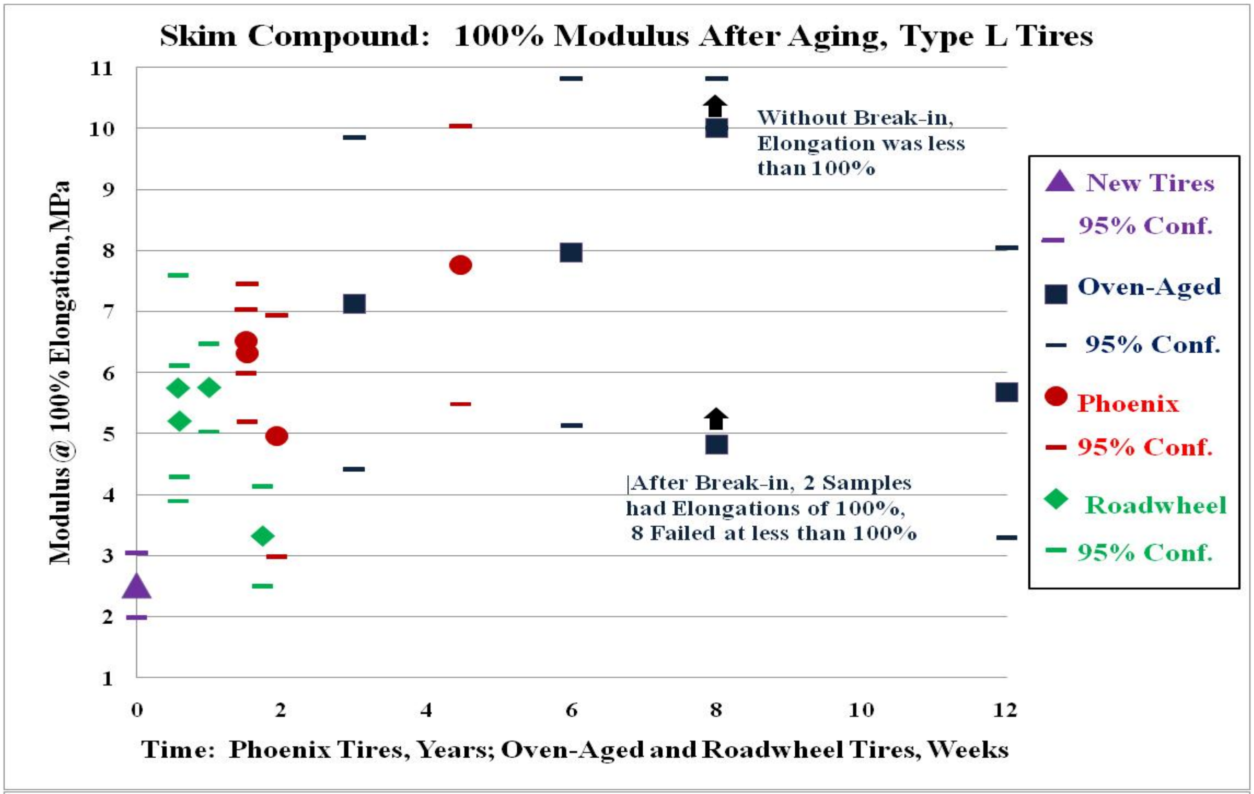


Figure 11. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type H Tires



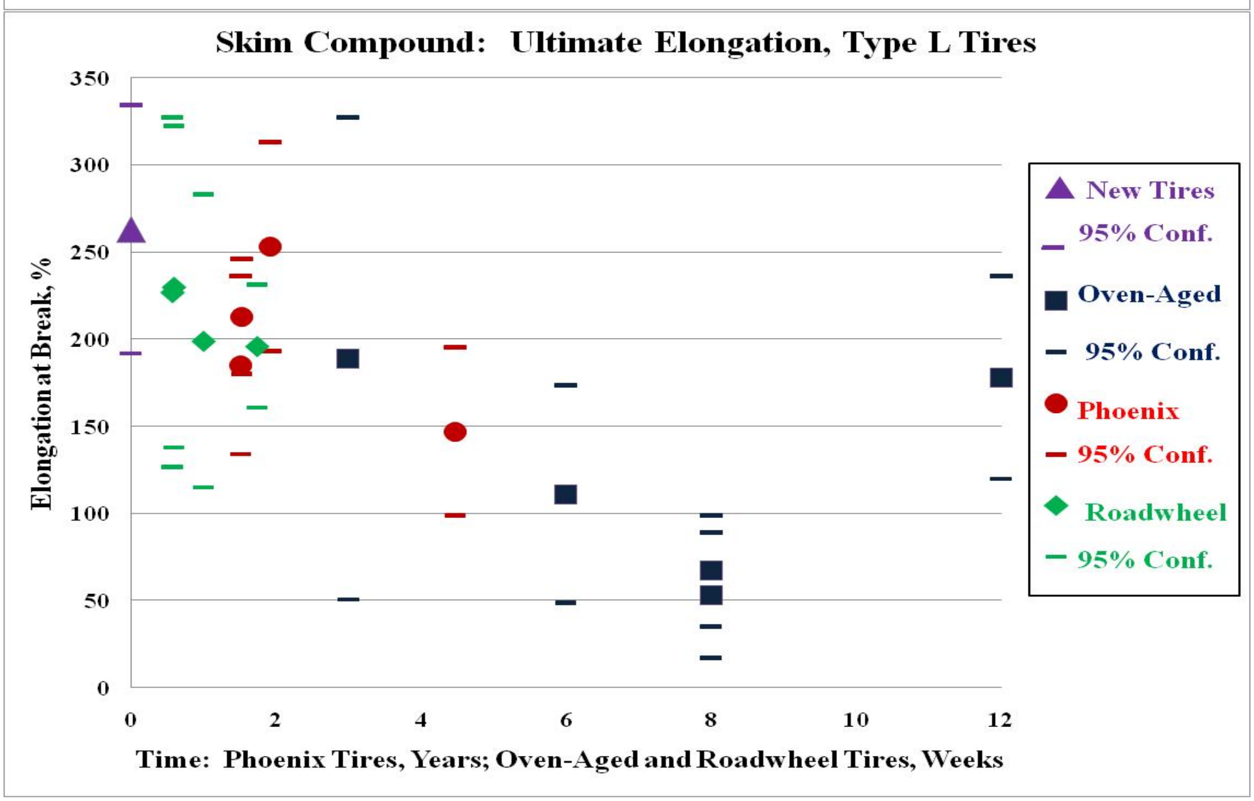


Figure 12. Modulus @ 100% Strain and Ultimate Elongation for Skim-Coat Compound of Type L Tires

The plots of log (Ultimate Elongation at Break, %) versus log(Modulus at 100% Strain, MPa), often referred to as Ahagon plots are shown in Figure 13 to Figure 18. As noted previously, a slope near -0.75 for these plots has been shown to correlate to oxidative (Type I) aging of rubber compounds. Reductions in elongation with less change in modulus are associated with anaerobic (Type II) aging, which would be indicated by a 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 22. All of the tire types from Phoenix service have slopes near 0.75, therefore during service in Phoenix the skim-coat compound of tires tends to age oxidatively. Note that tire type H has reduced modulus and elongation compared to the new tire for tires in Phoenix service which could indicate type II, anaerobic aging. However this tire was found to have high levels of resin in the wire coat compound and the breakdown of the 3-dimensional structure of the resin during service was thought to account for this. The slope of -0.86 for the tires during Phoenix service indicates oxidative aging in service. The slopes for the oven-aged tires are similar to those of the tires in Phoenix service for tire types B, C, H and L. Tire types D and E have slopes of -1.5 to -1.6, indicating a greater rate of reduction in elongation relative to the change in modulus which indicates that some type II anaerobic aging may be taking place. Similarly the slopes of the type D and E tires during roadwheel aging are -1.1 to -1.2. The elongation of the type L tire showed very little change during roadwheel testing.

Table 22. Ahagon Slopes for Skim-Coat Compound

Tire Type	**Ahagon Slope, from Log(Elongation) Vs. Log(100% Modulus)**		
	Phoenix Tires	**Oven-Aged Tires**	**Roadwheel-aged Tires**
B	- 0.76	-0.80	- 0.53
C	- 0.81	- 1.09	- 0.77
D	- 0.91[p]	- 1.65	- 1.11
E	-0.86[q]	- 1.52	- 1.25
H	- 1.00	- 0.84	Not Measured
L	- 0.44	- 0.47	- 0.14

[p] Based on a single tire in Phoenix service

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

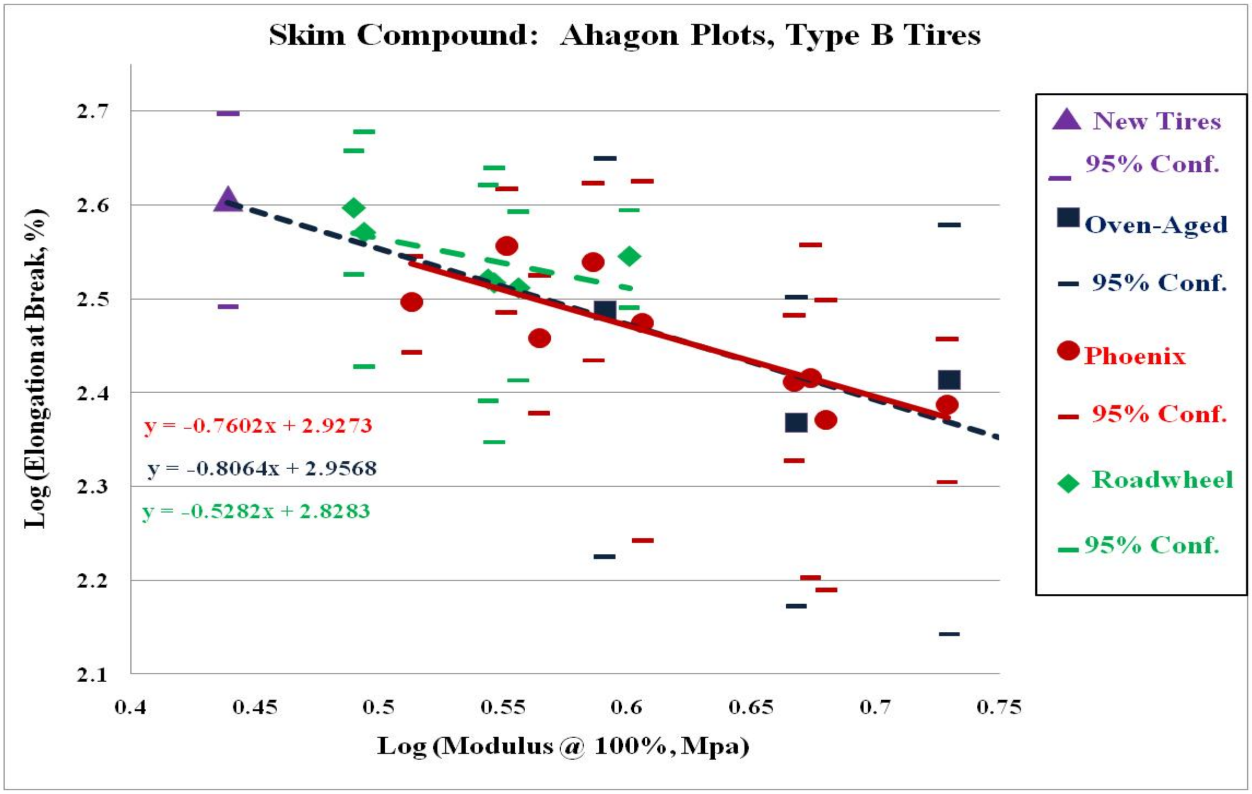


Figure 13. Ahagon Plot of Skim-Coat Compound for Type B Tires

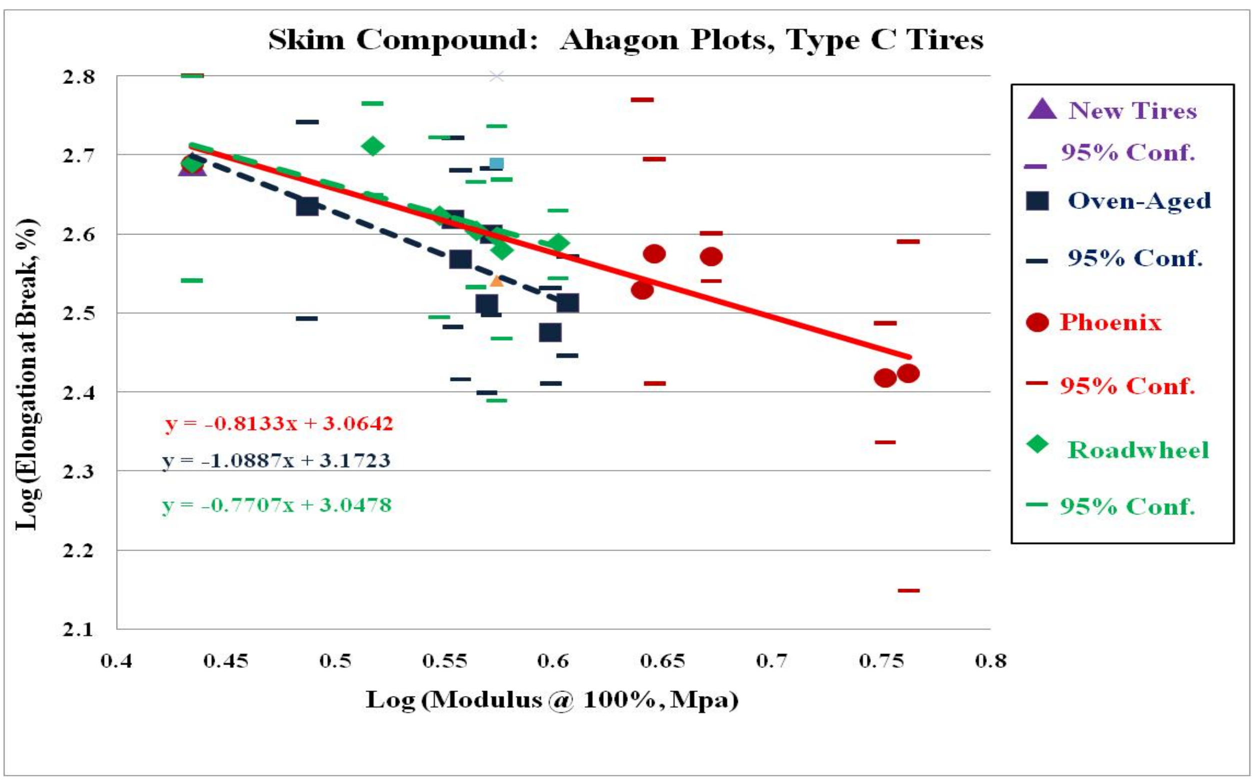


Figure 14. Ahagon Plot of Skim-Coat Compound for Type C Tires

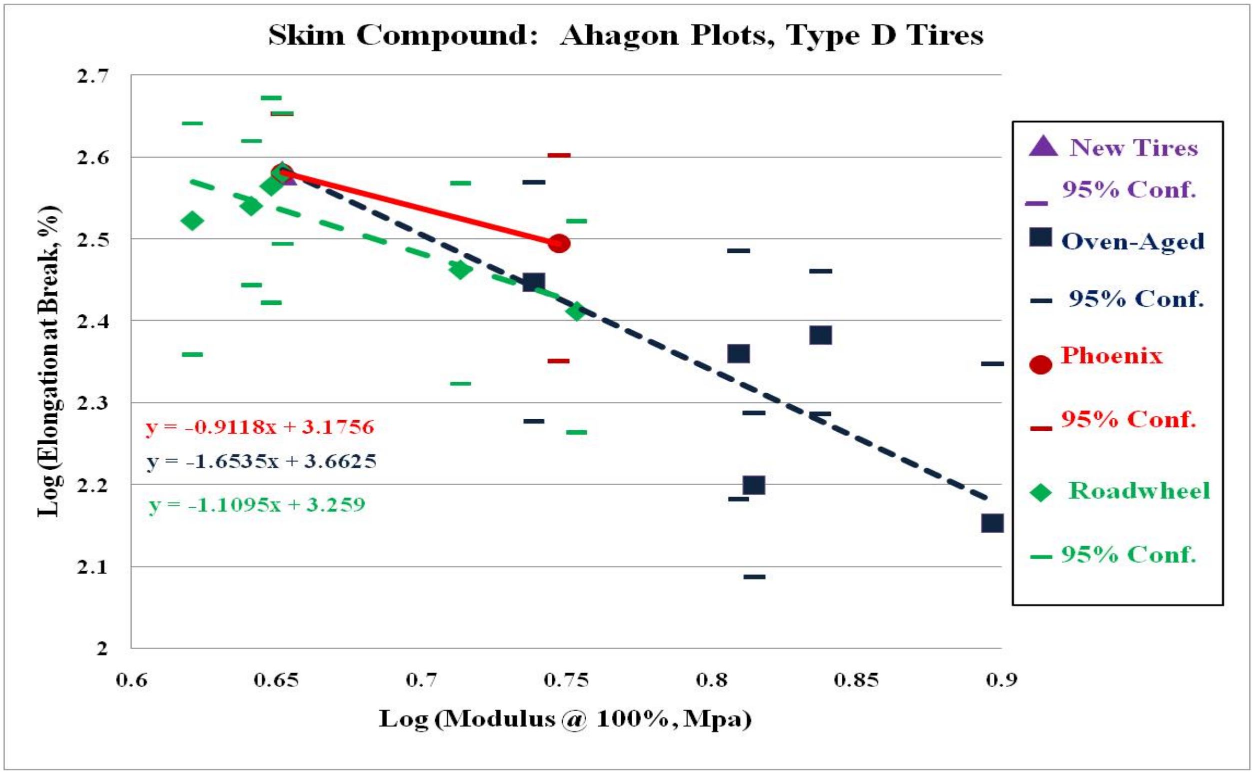


Figure 15. Ahagon Plot of Skim-Coat Compound for Type D Tires

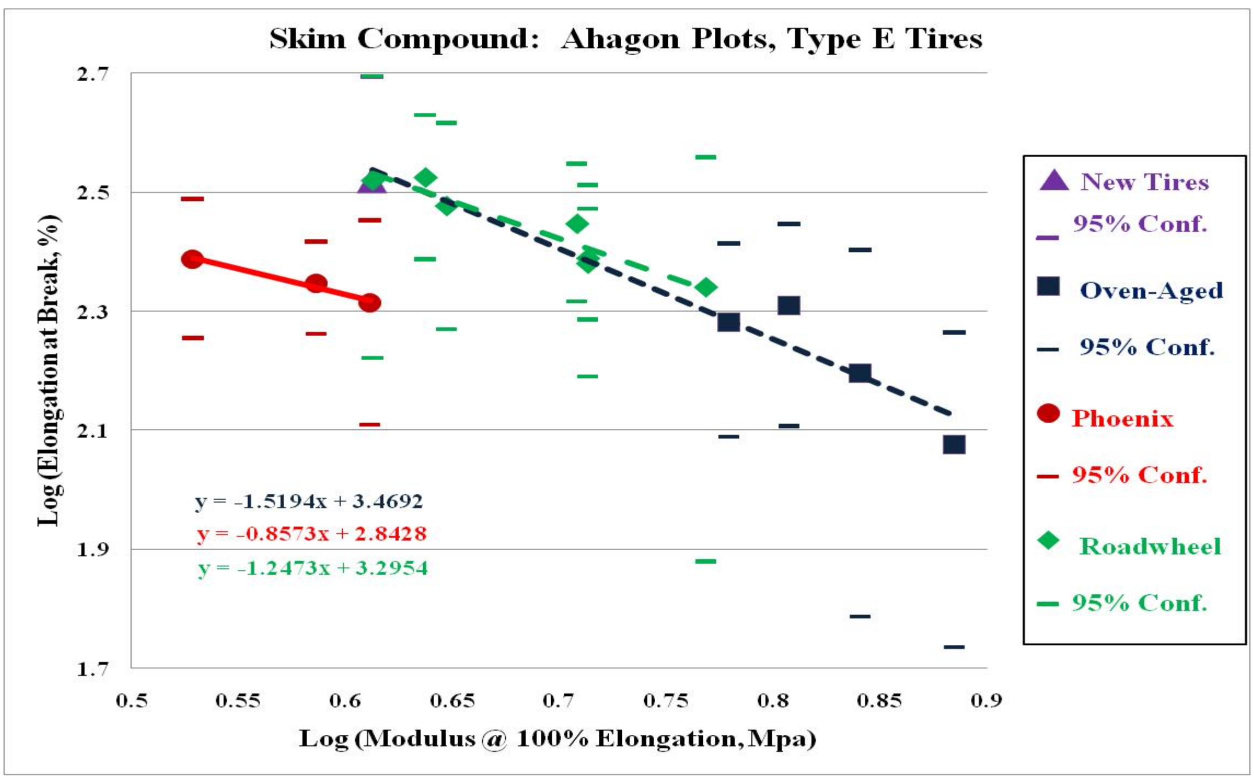


Figure 16. Ahagon Plot of Skim-Coat Compound for Type E Tires

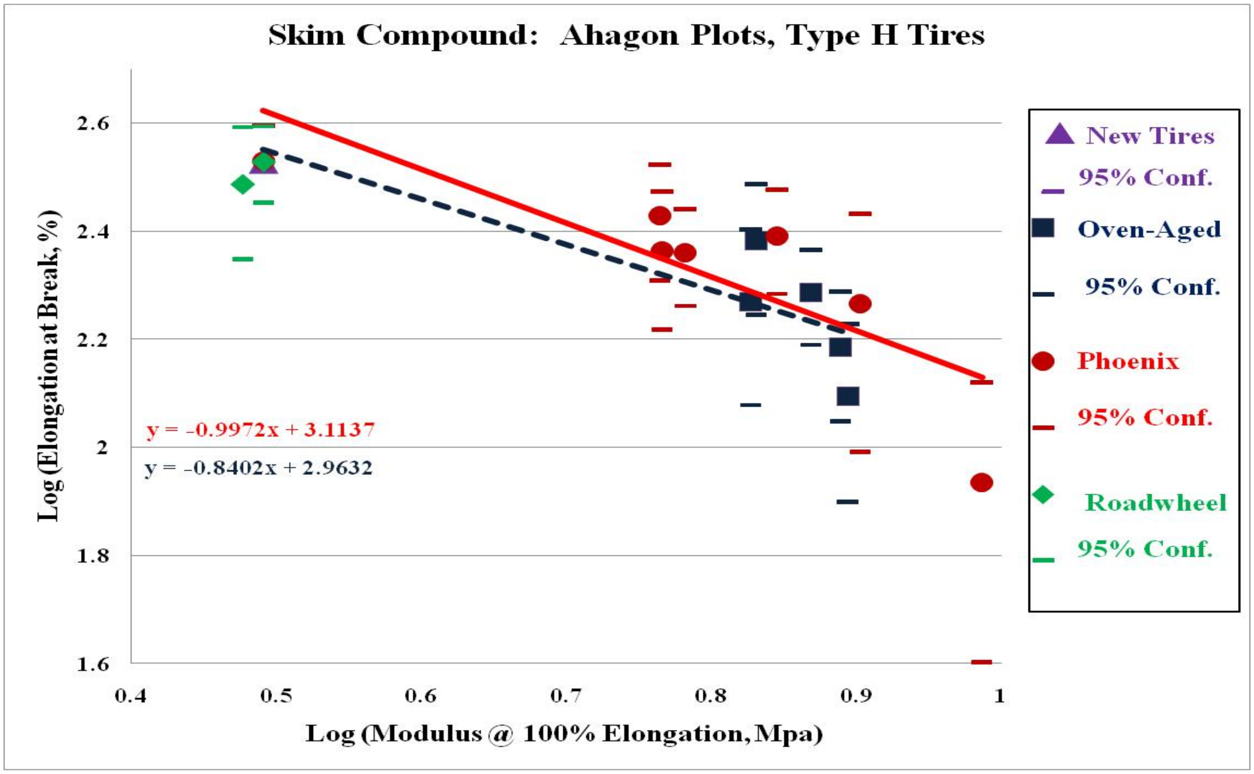


Figure 17. Ahagon Plot of Skim-Coat Compound for Type H Tires

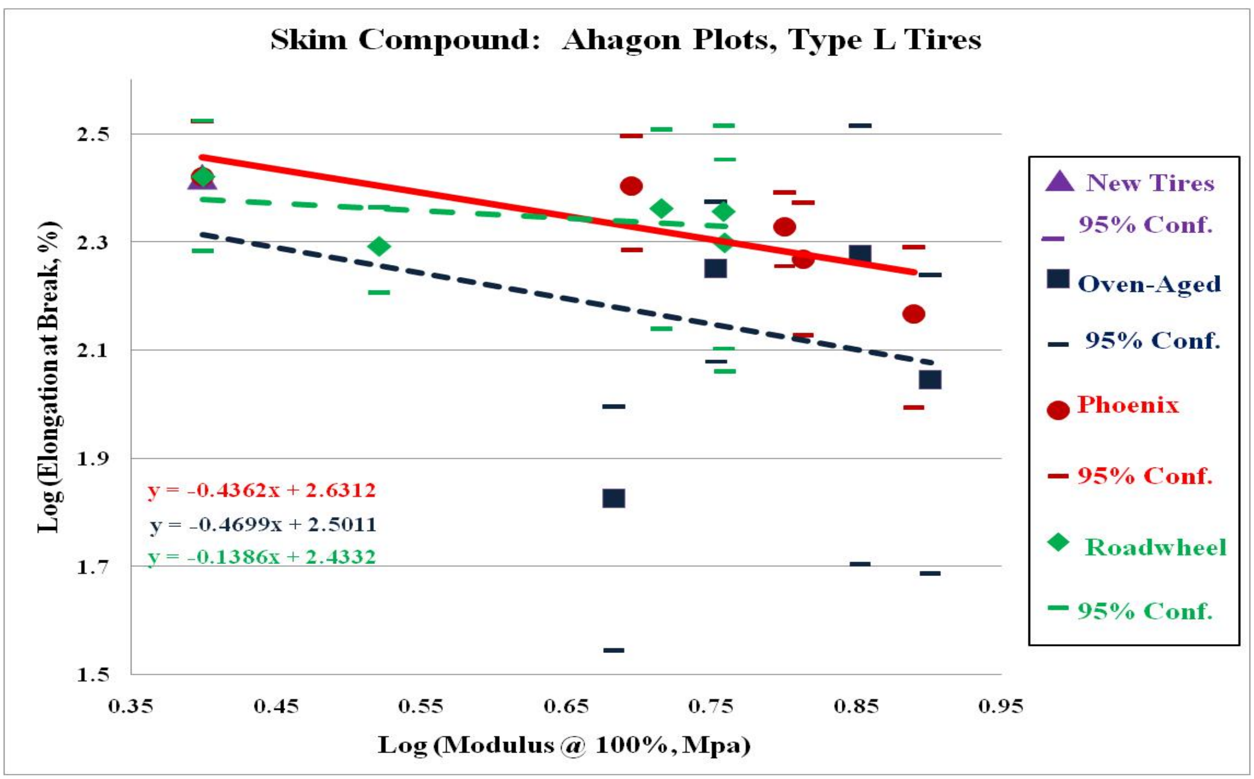


Figure 18. Ahagon Plot of Skim-Coat Compound for Type L Tires

3.2.4 Tensile and Elongation of Wedge Compound

The wedge compound is the rubber compound between the wire belts in the critical shoulder region of the tire. Over 95 percent of the structural tire failures (excluding those described as early wear-out of the tread or road hazard punctures) in the tire complaint data submitted to NHTSA by consumers are failures in this tread/belt region, within which the shoulder in a critical area. The wedge compound may simply be the adjoined layers of the skim-coat, it may be a thicker layer of the same compound as the skim-coat, or it may be an additional layer of rubber between the skim-coat compounds specifically engineered for this area. The analysis of variance and regression of the modulus, tensile and elongation of the wedge compound is shown in Table 23. For all tire types, the elongation to break decreases and the modulus at high strain increases. This is parallel to the data obtained from tires in service in Phoenix. The large amount of scatter in the data does not allow firm interpretation of the models. For the roadwheel-tested tires there is a consistent trend for increasing time to increase the modulus and decrease the tensile and elongation. Neither of the roadwheel tests have a consistent trend to elicit a greater change in properties. For the oven aged tires, the modulus increases and the tensile and elongation decrease with increasing temperature and/or time in the oven, consistent with general expectations for organic chemical reactions. In general, inflation with 50%N_2/50%O_2 causes a greater change in properties than inflation with air, which contains ~21% O_2. Breaking in the tires for 24 hours at 120 km/h (75 mph) also increases the amount of change in rubber properties.

Table 23. ANOVA and Linear Regression Terms for Modulus, Tensile, and Elongation of Wedge Compound

Tire Type			B	C	D	E	H	L
Measure-ment	**Test**	**Variable**						
Modulus @ 50% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.47	0.63	0.17	0.27	0.31	0.22
		Hours	+ 0.0013	+ 0.0032	+ 0.0007	+ 0.0014	+	+ 0.0030
		Test		LTDE > P-END	LTDE > P-END	LTDE > P-END		
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.78	0.41	0.46	0.73	0.53	0.65
		Temperature	- 0.0209	+	- 0.267	+ 0.0223	-	+
		Hours	+ 0.0008	- 0.0005	+ 0.0005	+ 0.0005	+	+ 0.0004
		Fill Gas	50/50 > AIR	50/50 > AIR	50/50 > AIR	AIR > 50/50	50/50 > AIR	
		Break-in	BI > no BI		BI > no BI			
Modulus @ 100% Elongation	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.57	0.57	0.43	0.23	0.06	0.17
		Hours	+ 0.0032	+ 0.0044	+ 0.0023	+ 0.0020	+	+ 0.0041
		Test		P-END > LTDE	LTDE > P-END	LTDE > P-END		
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.80	0.40	0.77	0.74	0.66	0.57
		Temperature	- 0.0204	+	- 0.0334	+ 0.0303	+	-
		Hours	+ 0.0010	-	+ 0.0005	+ 0.0005	+	-
		Fill Gas	50/50 > AIR	50/50 > AIR	50/50 > AIR		50/50 > AIR	50/50 > AIR
		Break-in	BI > no BI		BI > no BI			
Modulus @ 200% Elonga-tion	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.66	0.50	0.25	0.05	0.04	0.19
		Hours	+ 0.0073	+ 0.0058	+ 0.0035	+	+	+
		Test	P-END > LTDE	P-END > LTDE		LTDE > P-END		P-END > LTDE
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.79	0.40	0.74	0.53	0.64	0.14
		Temperature	-	-	- 0.0414	+ 0.0326	-	-
		Hours	+ 0.0009	+ 0.0283	+	+	- 0.0013	-
		Fill Gas	50/50 > AIR	50/50 > AIR	50/50 > AIR	50/50 > AIR	50/50 > AIR	
		Break-in	BI > no BI		BI > no BI	BI > no BI		
Modulus @ 300% Elonga-tion	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.74	0.47	0.03	0.04	0.16	0.02
		Hours	+ 0.0127	+ 0.0049	-	+	+	+
		Test	P-END > LTDE	P-END > LTDE		LTDE > P-END		
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.74	0.39	0.75	0.08	0.03	0.19
		Temperature	-	+	-	+	+	+
		Hours	+ 0.0006	- 0.0202	- 0.0617	+	-	-

		Fill Gas	50/50 > AIR	50/50 > AIR	50/50 > AIR			
		Break-in						
Tensile	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.12	0.37	0.41	0.38	0.21	0.42
		Hours	- 0.0072	- 0.0163	- 0.0126	- 0.0191	+	- 0.0239
		Test				LTDE > P-END		
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.38	0.56	0.21	0.69	0.63	0.57
		Temperature	-	- 0.2029	+	- 0.1076	-	-
		Hours	- 0.0018	- 0.0026	- 0.0020	- 0.0023	- 0.0035	- 0.0039
		Fill Gas		50/50 > AIR	50/50 > AIR			50/50 > AIR
		Break-in	no BI > BI		no BI > BI	no BI > BI	no BI > BI	no BI > BI
Elongation at Break	Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.63	0.59	0.59	0.45	0.44	0.33
		Hours	- 0.331	- 0.391	- 0.321	- 0.358	+	- 0.379
		Test	LTDE > P-END	LTDE > P-END		LTDE > P-END		LTDE > P-END
	Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		Model R^2	0.72	0.64	0.51	0.82	0.81	0.69
		Temperature	-	- 5.038	+ 2.070	- 2.838	-	-
		Hours	- 0.047	-	-	- 0.035	- 0.050	- 0.049
		Fill Gas	AIR > 50_50	50_50 > AIR	AIR > 50_50		AIR > 50_50	AIR > 50_50
		Break-in	no BI > BI		no BI > BI	no BI > BI	no BI > BI	no BI > BI

The plots of the Modulus at 100 percent strain and percent elongation at break versus time for the wedge compounds for tires aged in the laboratory and retrieved from service in Phoenix are shown in Figure 19 to Figure 24.

For the type B tires, the average modulus increase and elongation decrease for either 6 weeks at 60ºC to 70ºC or 12 weeks at 55ºC are approximately equal to that for tires with 4 to 6 years of service in Phoenix. The average levels of modulus and elongation change for the roadwheel-tested tires or tires with 3 weeks of oven aging approximate that of tires with 1 to 2 years of service in Phoenix.

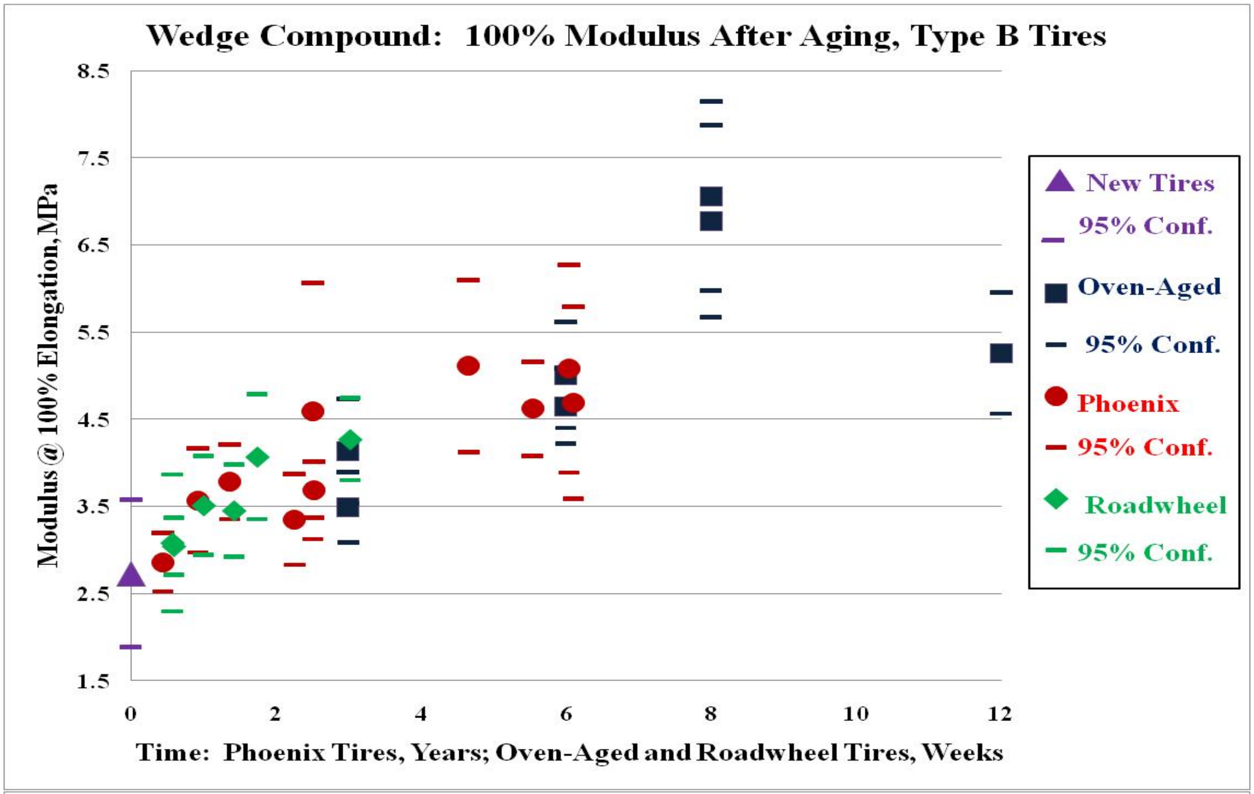
For the type C tires, the modulus increased to approximately the levels of tires retrieved having 2 years of service in Phoenix with 3 to 6 weeks of oven aging at 60ºC to 70ºC. Further increases in modulus were not observed with Phoenix service or increased duration of oven aging. Roadwheel testing for the maximum period tested (500 hours) achieved this plateau level of 100 percent modulus as well. Similarly, elongation to break reached a steady state level after 2 years of Phoenix service, 3 to 6 weeks of oven aging, or 500 hours of roadwheel testing.

For the type D tire, roadwheel testing for 300 to 500 hours, or oven aging for 3 weeks at 60ºC to 70ºC approximated the levels of modulus and elongation for the Phoenix tire with 2 years of service available for comparison.

Oven aging for 3 to 6 weeks at 60ºC to 70ºC, or roadwheel testing for 500 hours produced 100% modulus and elongation values approximately equal to type E tires with 2 to 3 years of service in Phoenix.

For the type H tires, oven aging for 3 weeks at 60ºC to 70ºC produced 100% modulus and elongation values approximately the same as tires with 2 to 3 years of service in Phoenix.

For the type L tires oven aging between 3 and 6 weeks at 60ºC to 70ºC, or 12 weeks at 55ºC, produced modulus and elongation values approximately equal to those found in a tire with 5 years of service in Phoenix. Roadwheel testing produced maximum changes approximately equal to those of tires with 2 years of service in Phoenix.



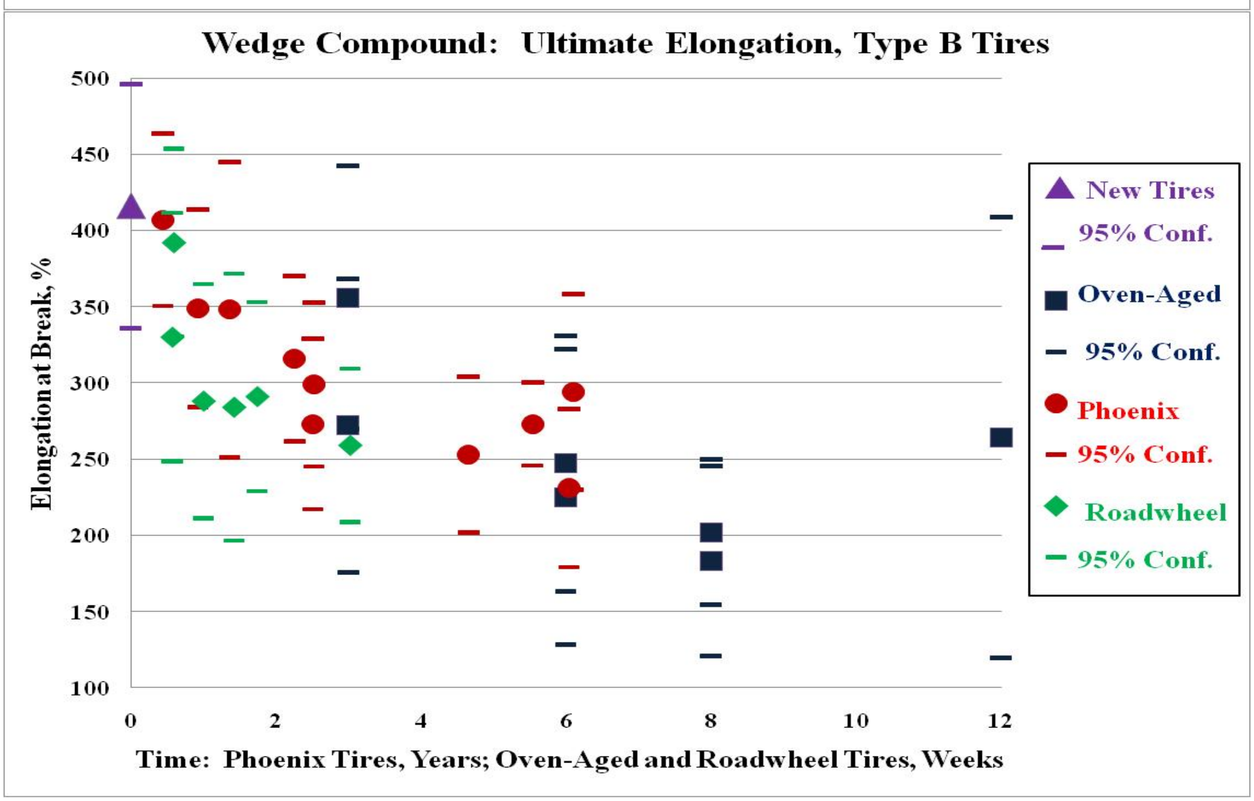
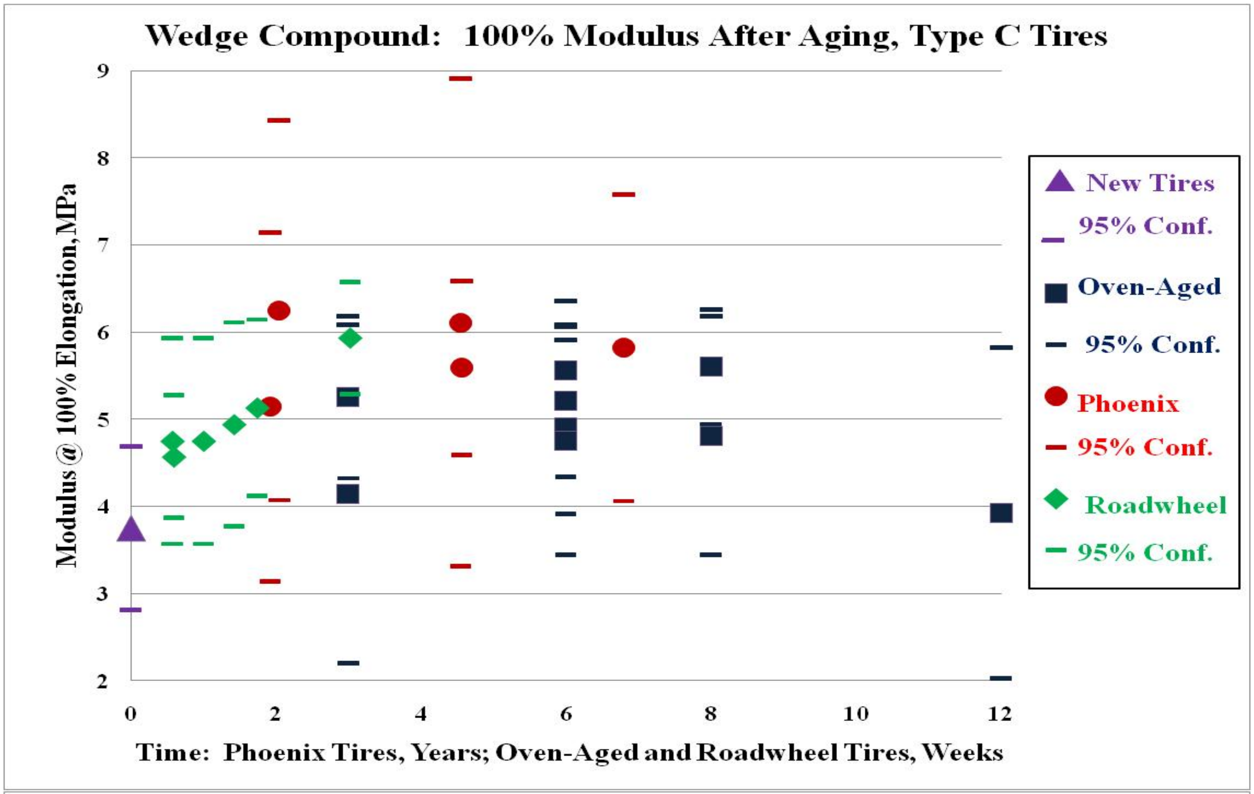


Figure 19. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type B Tires



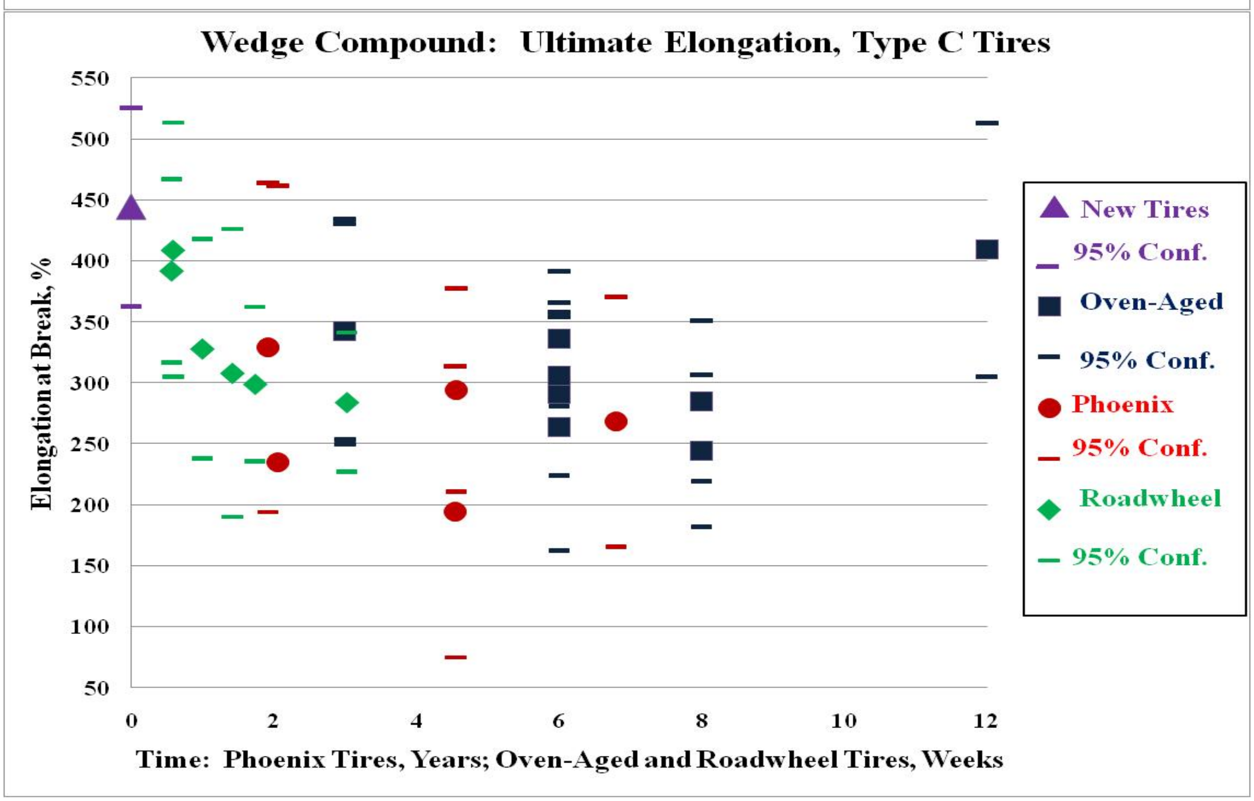


Figure 20. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type C Tires

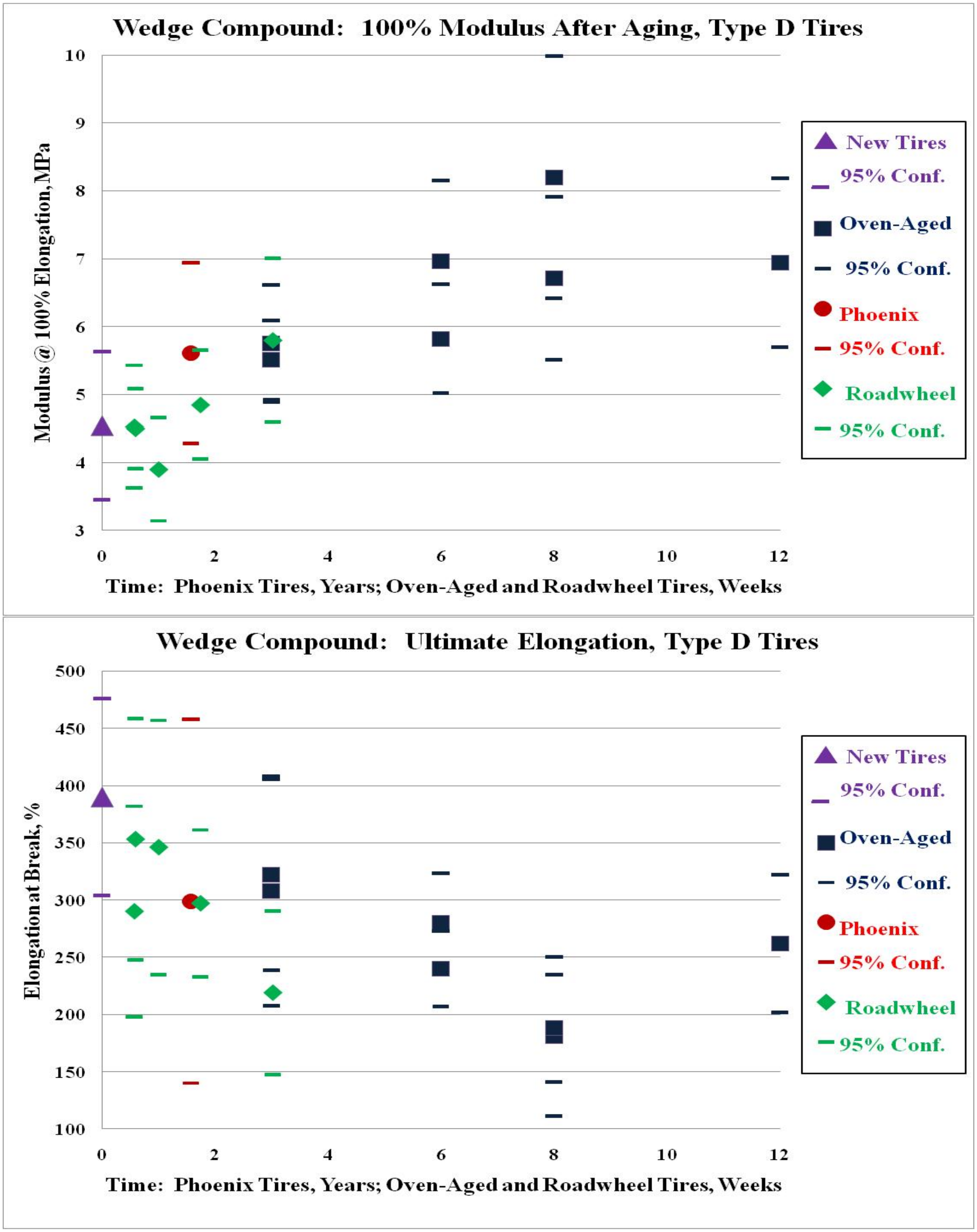


Figure 21. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type D Tires

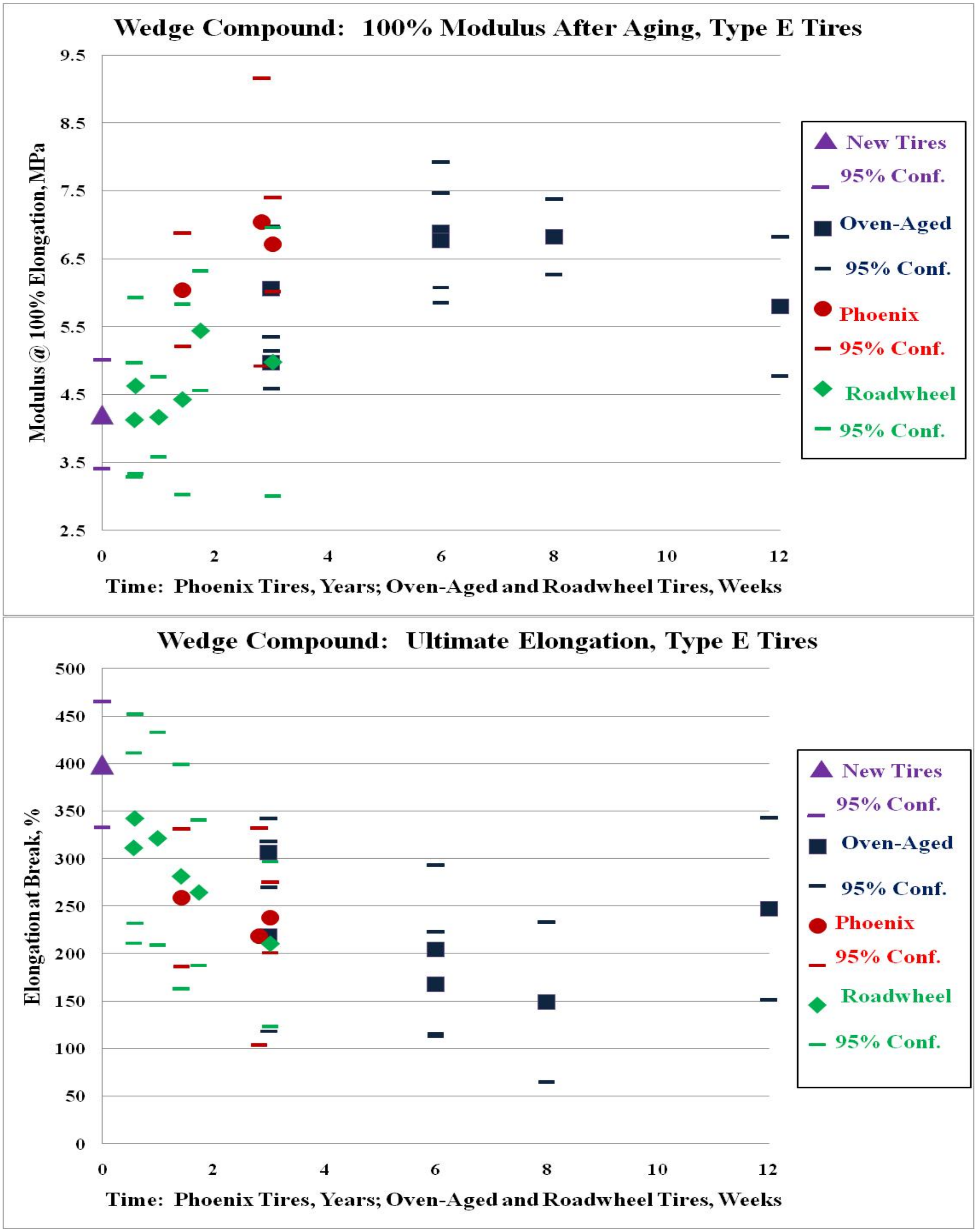
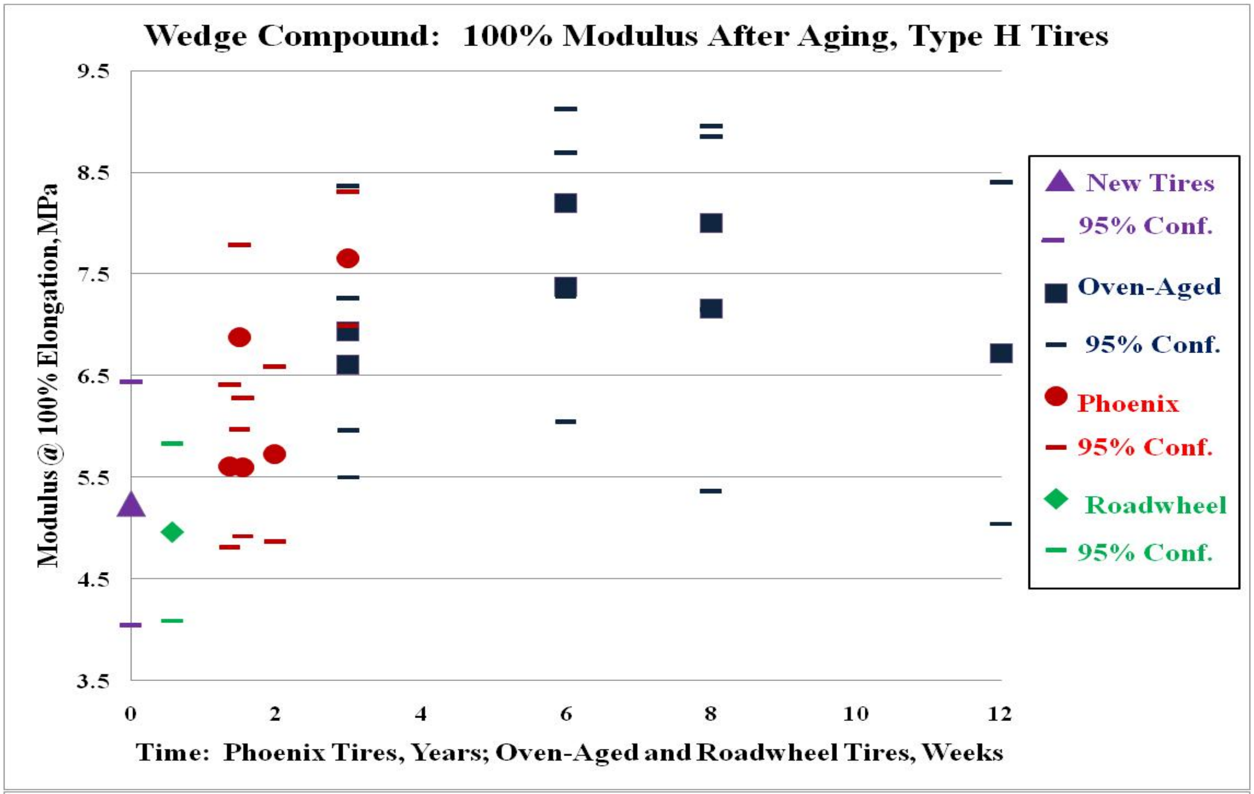


Figure 22. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type E Tires



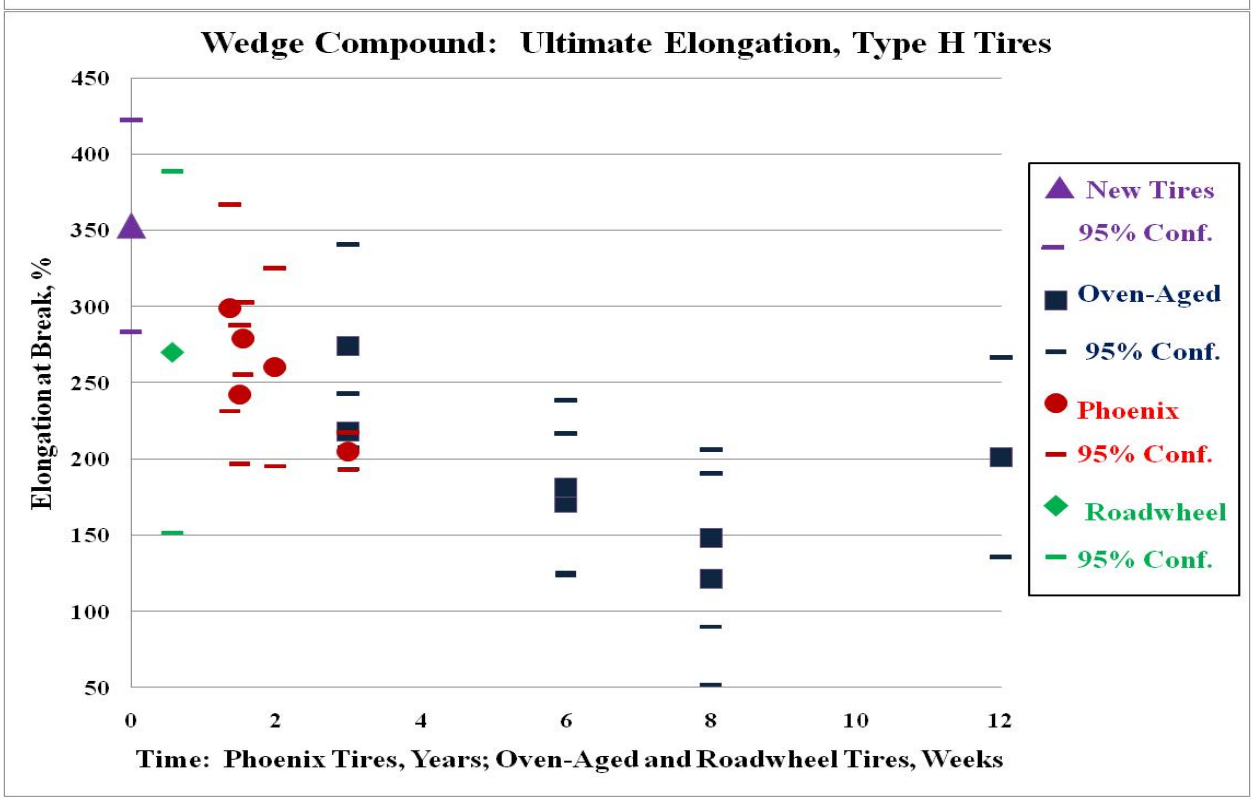
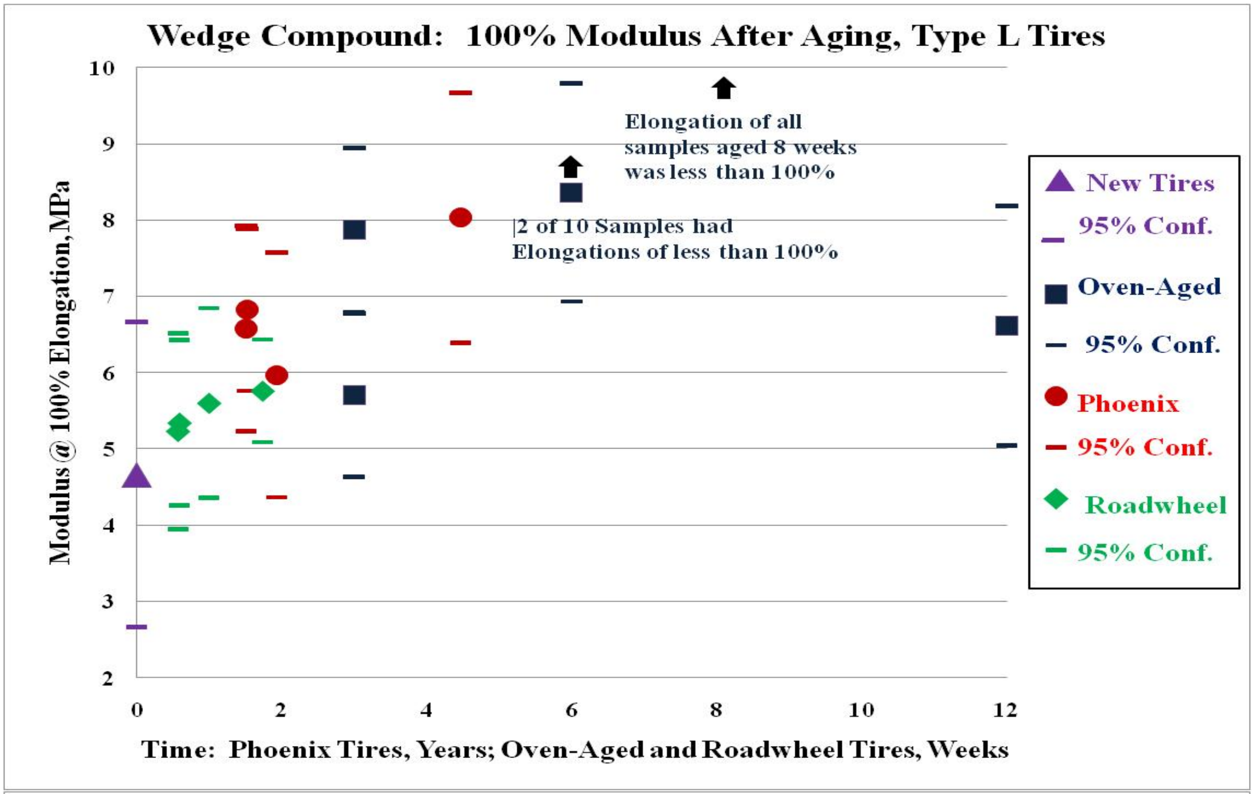


Figure 23. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type H Tires



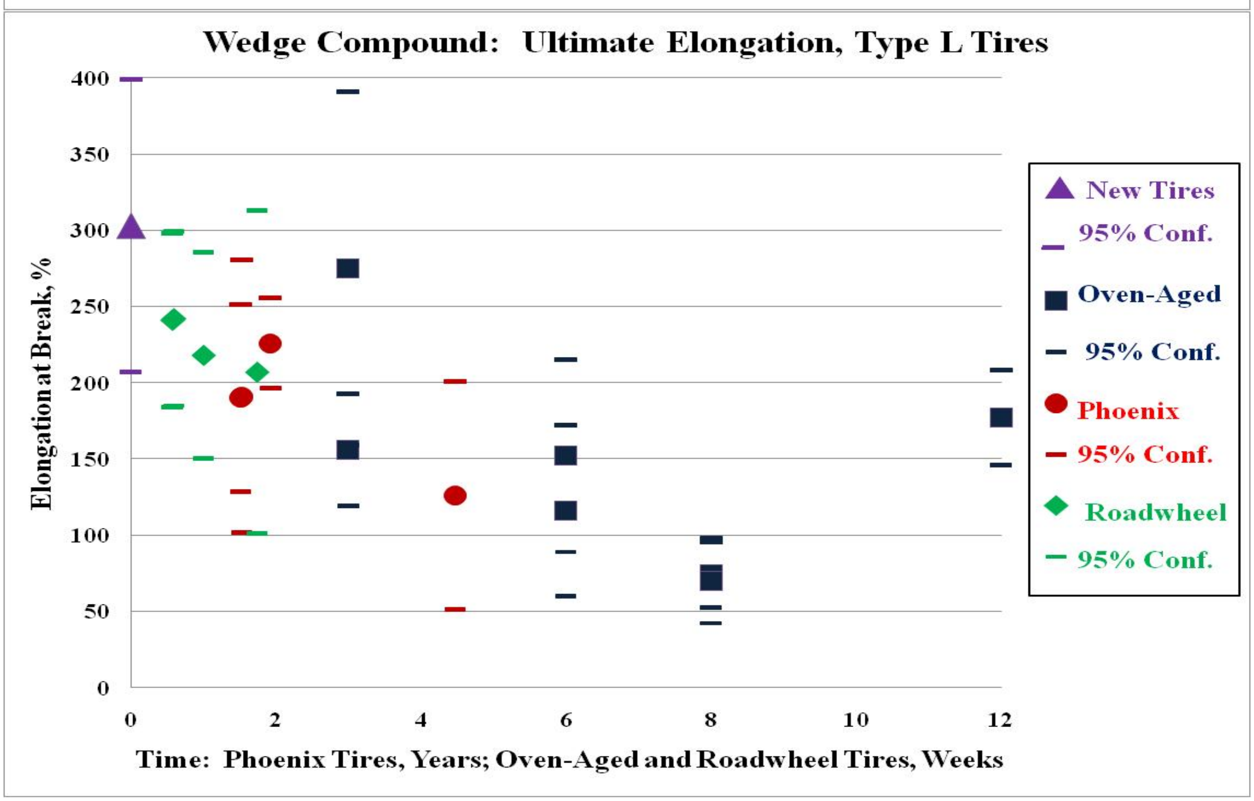


Figure 24. Modulus @ 100% Strain and Ultimate Elongation for Wedge Compound of Type L Tires

The plots of log(Ultimate Elongation at Break, %) versus log(Modulus at 100% Strain, MPa), often referred to as Ahagon plots are shown in Figure 25 to Figure 30. As noted previously, a slope near -0.75 for these plots has been shown to correlate to oxidative (Type I) aging of rubber compounds. Reductions in elongation with less change in modulus are associated with anaerobic (Type II) aging, which would be indicated by a very 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 24.

Tire type B has a slope near -0.75 for tires in service in Phoenix or for either oven aging or roadwheel testing, indicating oxidative aging of the wedge compound. All of the other tire types have slopes ranging from approximately -1 to -2, indicating some degree of anaerobic aging. For tire types C, D and L the slopes for oven aging are similar to the slope of the wedge compound from Phoenix service, while tire types E and H showed much steeper slopes for oven aging. As noted, these tires contained a reinforcing resin which may have influenced the modulus and elongation results. The slope of the change during roadwheel testing is similar to the slope for tires from Phoenix service.

Table 24. Ahagon Slopes for Wedge Compound

Tire Type	Ahagon Slope, from Log(Elongation) Versus Log(100% Modulus)		
	Phoenix Tires	Oven-Aged Tires	Roadwheel-aged Tires
B	- 0.77	- 0.84	- 0.99
C	- 1.39	- 1.20	- 1.10
D	- 0.80[r]	- 0.93	- 1.26
E	- 1.15	- 1.74	- 1.30
H	- 1.17	- 2.12	Not Measured
L	- 1.56	- 1.53	- 1.78

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

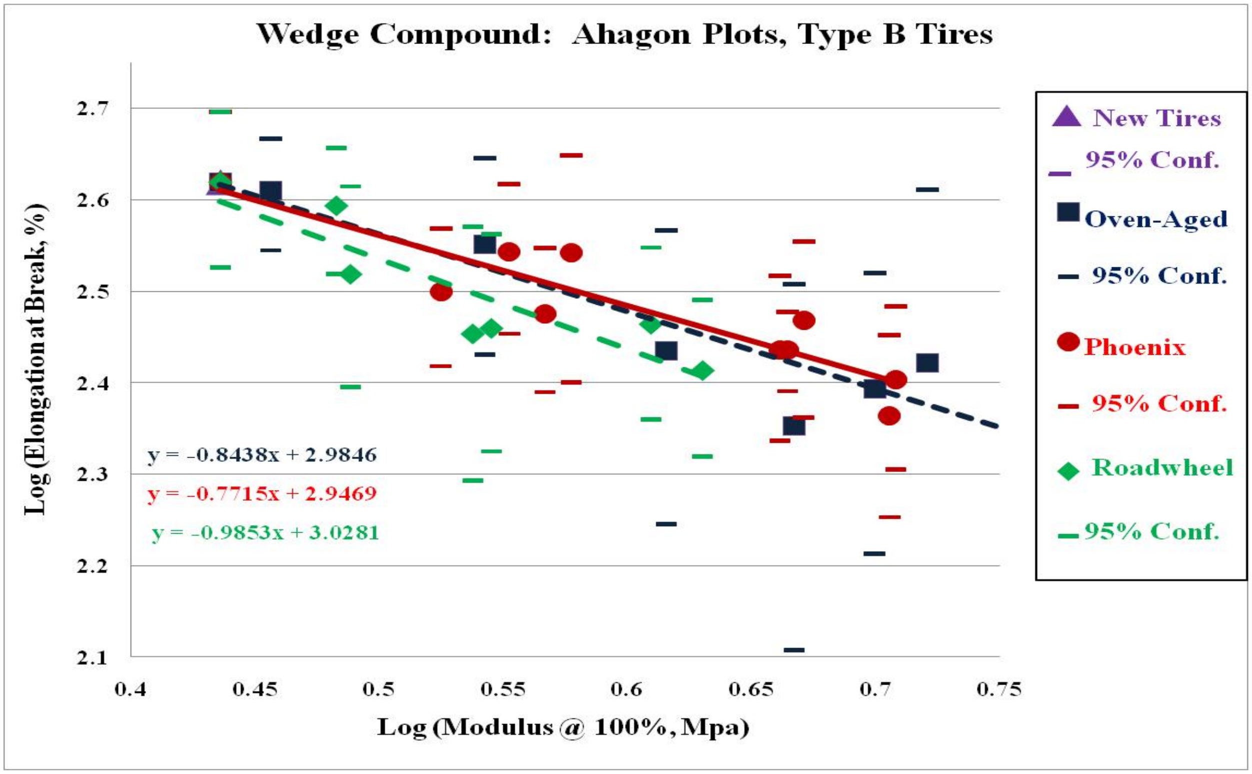


Figure 25. Ahagon Plot of Skim-Coat Compound for Type B Tires

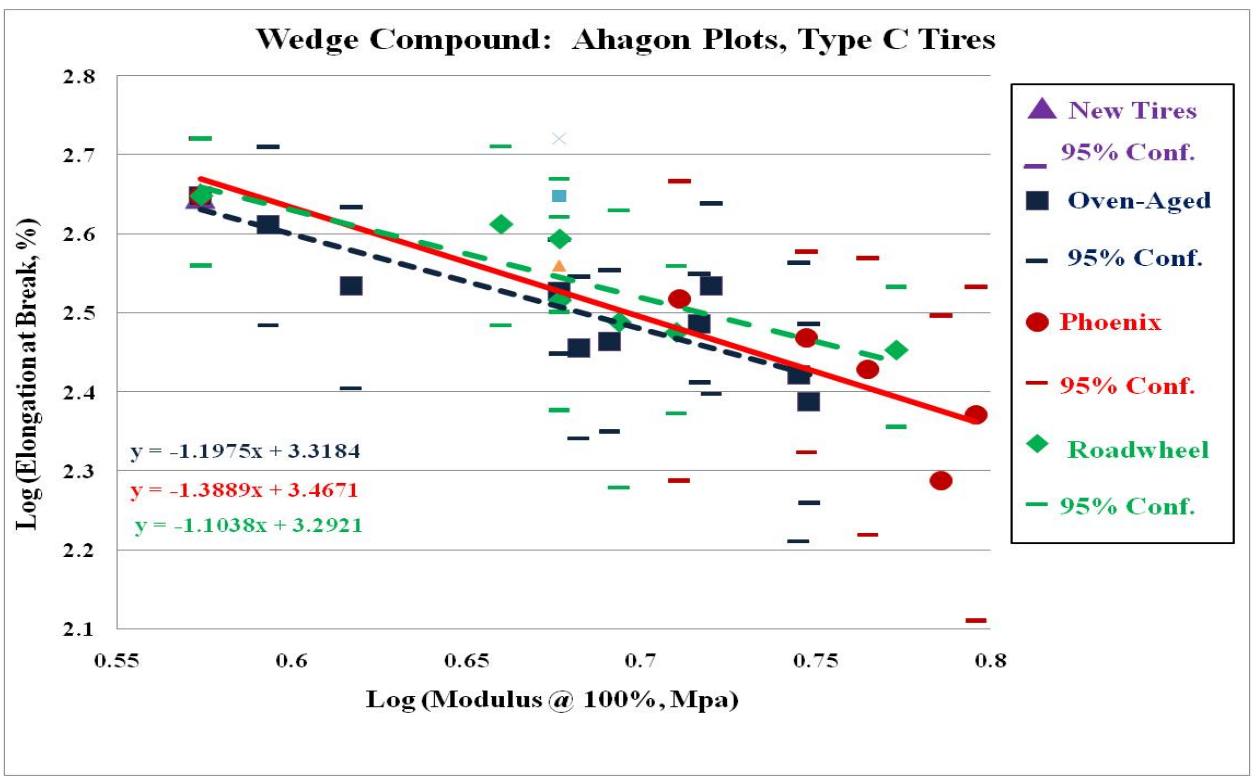


Figure 26. Ahagon Plot of Skim-Coat Compound for Type C Tires

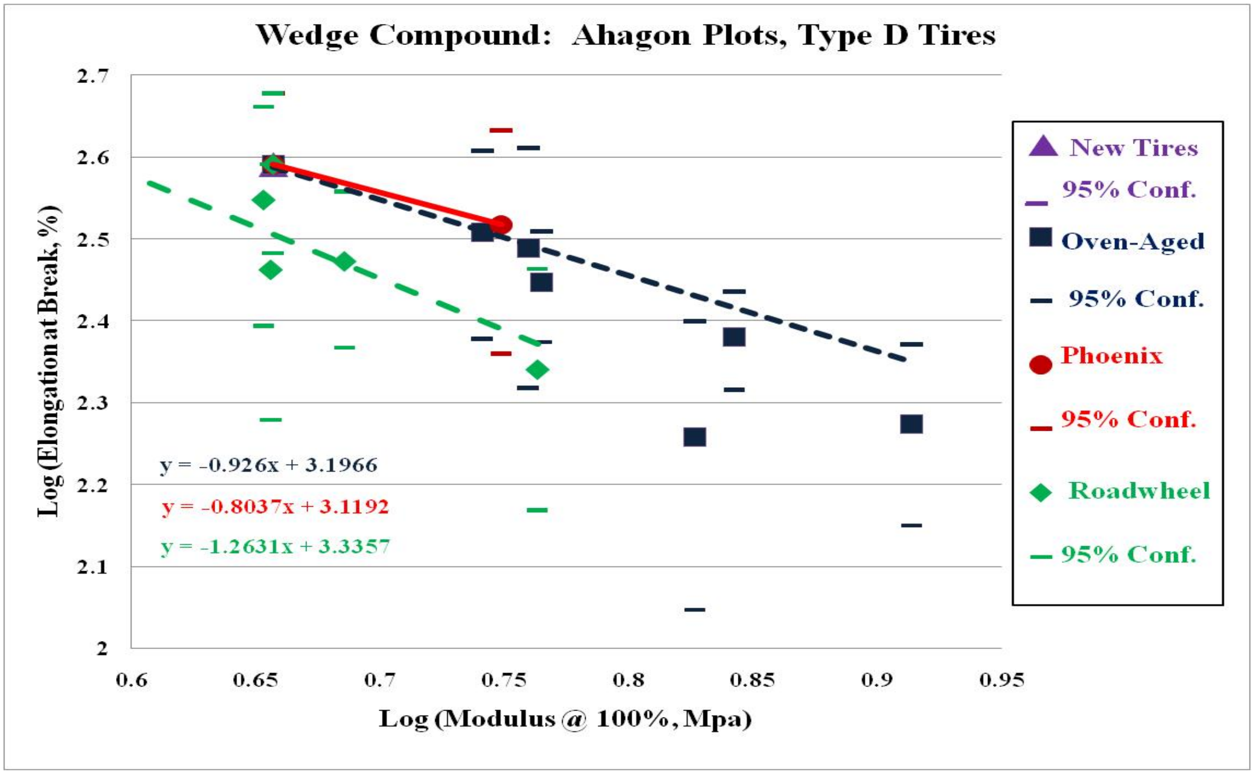


Figure 27. Ahagon Plot of Skim-Coat Compound for Type D Tires

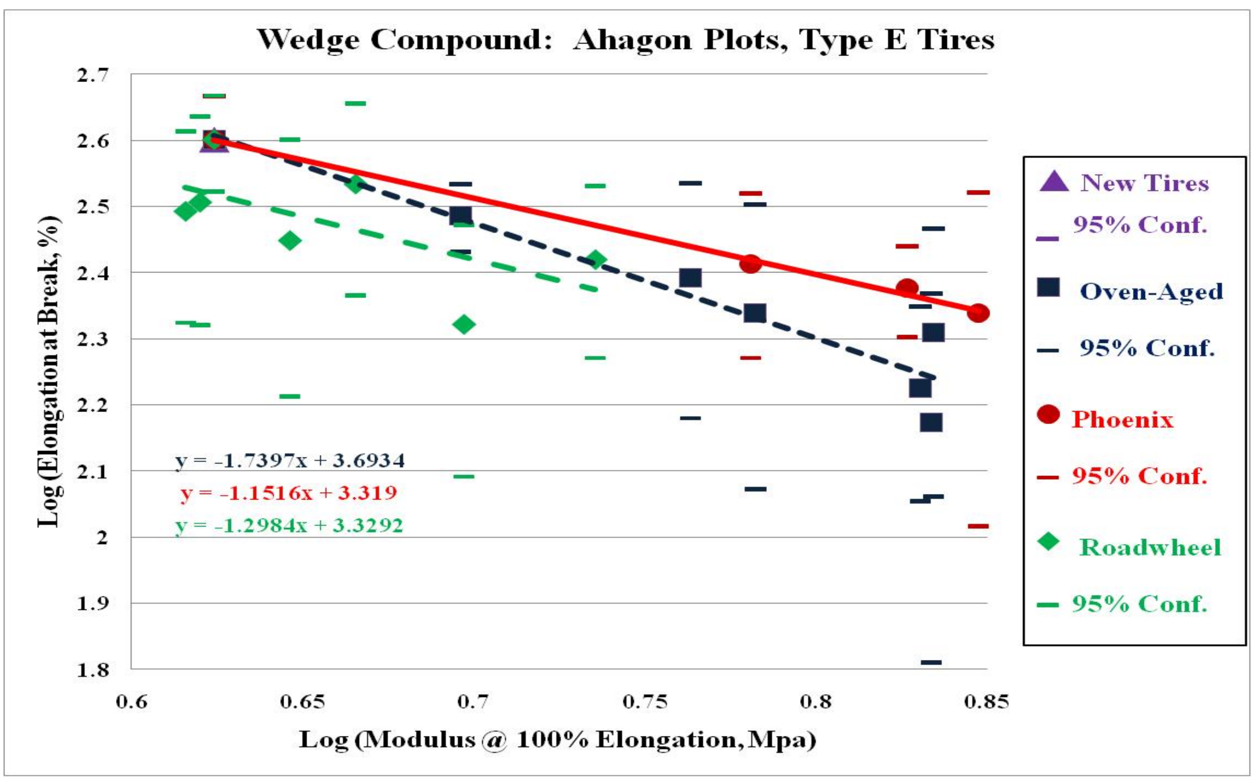


Figure 28. Ahagon Plot of Skim-Coat Compound for Type E Tires

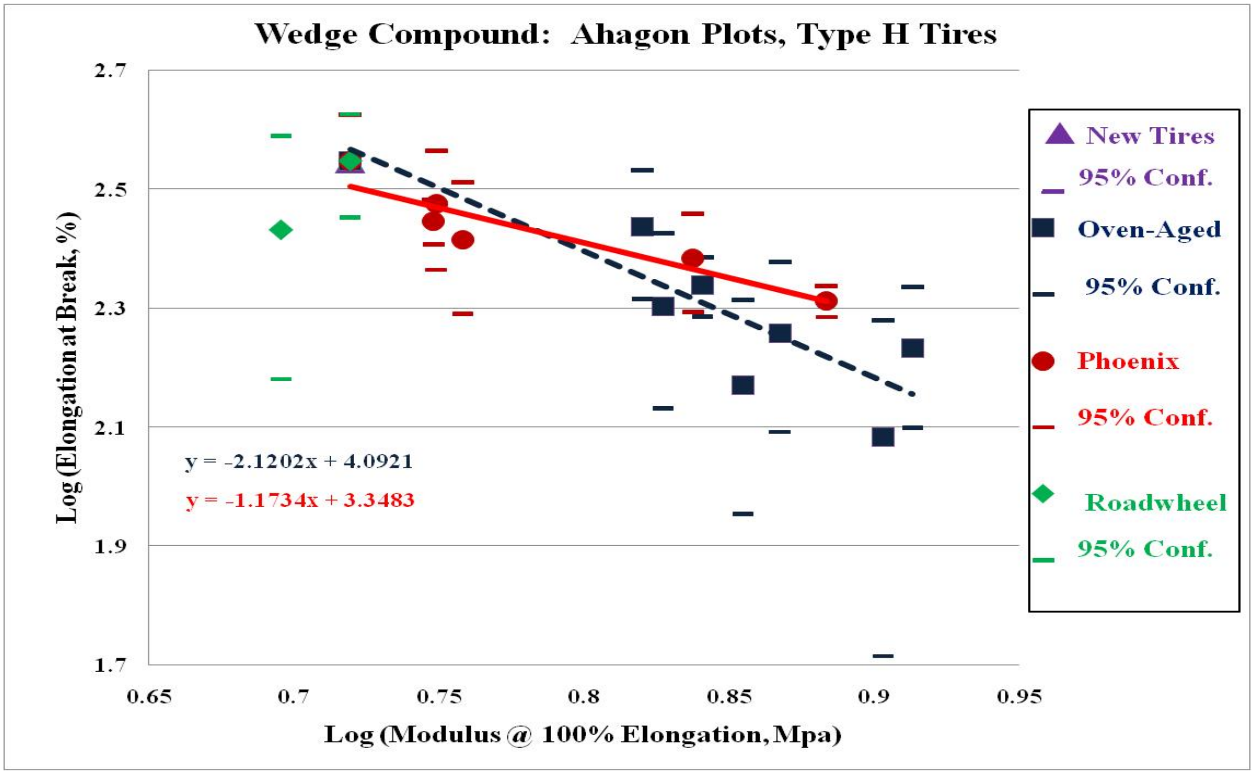


Figure 29. Ahagon Plot of Skim-Coat Compound for Type H Tires

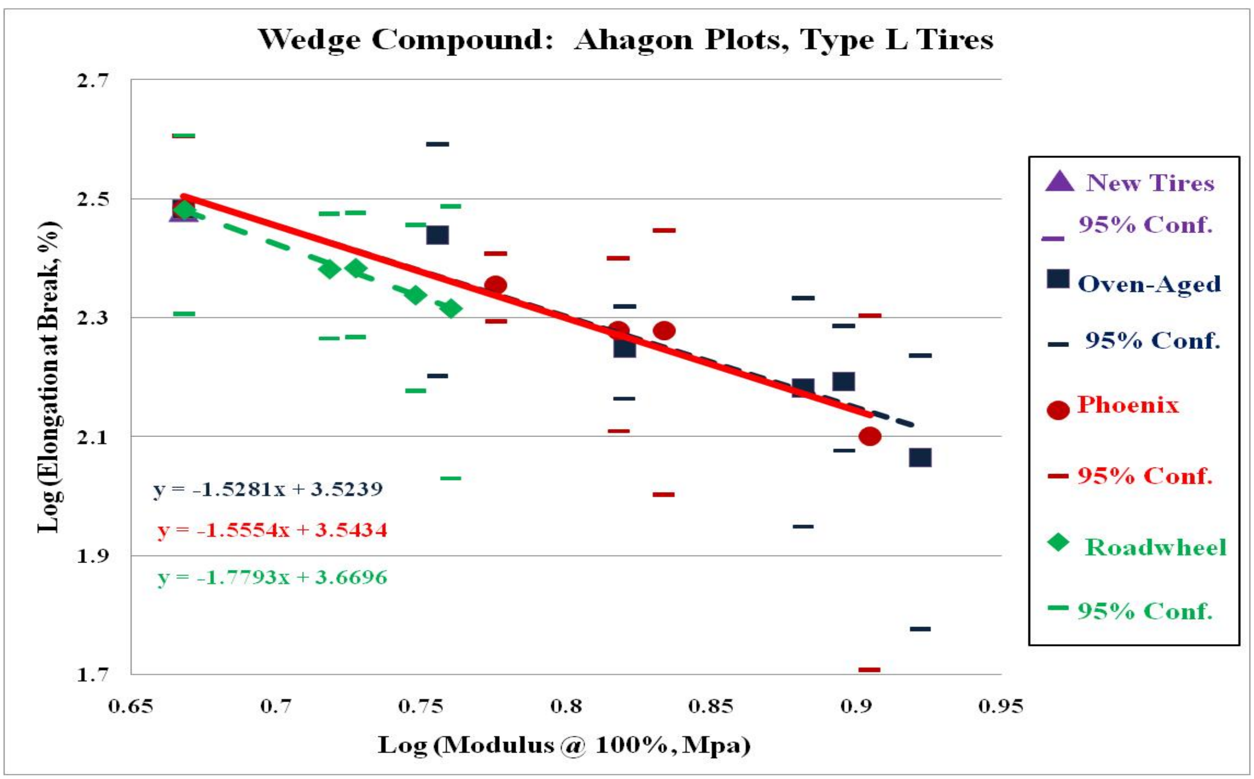


Figure 30. Ahagon Plot of Skim-Coat Compound for Type L Tires

3.2.5 Micro-DeMattia Flex Test

The crack growth rate measured by the Micro-DeMattia flex test showed a tendency to increase over time, whether in service in Phoenix or during laboratory aging. Tire types E and L showed the greatest tendency for increased crack growth rate in Phoenix service or during oven aging, while tire type D showed very little increase in crack growth rate for any aging condition. During oven aging there was no consistent trend for either temperature or time in the oven to dominate the increase. The data was too limited to determine if different tire types are more sensitive to increased temperature, or if the actual variable that determines the increase is some product of temperature and time. The roadwheel test produced smaller changes in crack growth rate than oven aging. The LTDE test tended to produce greater changes in the crack growth rate than the P-END test.

Table 25. ANOVA and Regression of Micro-DeMattia Flex Fatigue for Wedge Compound

Tire Type		B	C	D	E	H	L
Test	**Variable**						
Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.13	0.86	0.10	0.52	0.69	0.40
	Hours	+ 0.0002	+ 0.0014	-	+ 0.0223	+ 0.0051	+
	Test		LTDE > P-END	LTDE > P-END			LTDE > P-END
Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.79	0.81	0.72	0.53	0.80	0.77
	Temperature	+ 0.0245	+ 0.0092	-	+ 10.00	-	+ 13.47
	Hours	-	+	+ 0.0020	-	+ 0.5008	-
Phoenix	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	0.0401	< 0.001
	Model R^2	0.58	0.10	0.18	0.84	0.14	0.55
	Weeks	+ 0.0027	+	+	+ 0.0206	+	+ 0.0458

3.2.6 Crosslink Density and Distribution

A summary of the findings for crosslink density is provided below:[6]

The belt-coat, wedge, and innermost tread experienced an increase in total crosslink density during normal on-vehicle service. The primary cause, which is well documented in literature and textbooks, is oxidation. Gent and Hamed (2001) state:

> "In general, the reaction of oxygen with elastomers causes both chain scission and crosslinking. If chain scission dominates during aging, the elastomer softens and eventually may become tacky. …most technical elastomers eventually harden and embrittle during oxidation, a consequence of the dominant crosslinking reactions."[20]

The total crosslink density in the belt-coat, wedge, and innermost tread regions were affected by tire age and tire mileage.

The heavier gauge tires, in general, had higher total crosslink formation rates than lighter gauge tires during normal service in the belt-coat.

The spare tire (tire type C) had slightly slower formation rates of total, weak, and strong-type crosslinks than in-service tires. The change in the total crosslink density in the wedge of the spare tire (type C) studied was more than 60 percent slower than the comparable in-service tire, the belt-coat region 40 percent slower, and the innermost tread was not significantly different.

The Michelin Long-Term Durability and Endurance (LTDE) test was not able to reach total crosslink density levels of a four-year-old tire in a roadwheel time of 500 hours. Four of the six tire types aged faster in the wedge than in the belt-coat, which was different from field tires (which aged about the same rate in wedge and belt-coat). The weak-type crosslinks were formed very slowly or decreased during LTDE testing. Most of the crosslinks formed during LTDE testing were strong-type crosslinks, which may relate to higher temperatures of the belt-edge region than in-service belt edge temperatures.

The Continental Passenger Tire Endurance Test (P-END) was not able to reach total crosslink density levels in a four-year-old field tire. The distribution of crosslink types formed during P-END was different from in-service tires. The weak-type crosslinks were formed very slowly or decreased during P-END testing. Most of the crosslinks formed during P-END were strong-type crosslinks. The crosslink type formation indicated that the tire running temperatures during the P-END test were higher than the LTDE test.

When the six tire models studied were subjected to 2 to 9 weeks at 60°C - 70°C in the “Capped-Inflation Oven Aging” procedure (no inflation gas refill) from the initial phase of the project, they could reach the total crosslink density levels of the four-year-old field (in-service) tires in the belt-coat, wedge, and innermost tread regions. However, at 55°C the aging times could be longer than 9 weeks to reach the level of a 4-year-old tire in service. Due to the ongoing development of the oven aging test procedure, the more expensive crosslink distribution testing was not conducted on the main body of oven-aged tires.

ANOVA and Stepwise ANOVA analyses using SAS/JMP software screening effect analysis showed that model fit parameters for total crosslink density in the belt-coat, wedge, and innermost tread regions during oven aging were aging time, aging temperature, and tire type. Unlike the belt-coat and innermost tread, roadwheel break-in was a significant factor in the wedge. Presumably, the wedge experienced higher temperatures and strains during the roadwheel break-in than the belt-coat or innermost tread, which accelerated the net aging rate in the oven-aging test for the wedge.

3.2.7 Peel Adhesion

3.2.7.1 Peel Adhesion – Skim Compound

The peel adhesion of the skim compound has been shown to consistently decrease as tires experienced service in Phoenix.[3] The results of the ANOVA and regression are shown in Table 26. On the roadwheel tests, all tire types also consistently decreased in adhesion over time. Only the type E tire showed any significant sensitivity to the type of roadwheel test, with the P-END test producing a greater adhesion loss per hour of testing than the LTDE test. In the oven testing the peel strength tended to be very sensitive to increases in temperature and consistently showed a reduction in peel adhesion with increased time of aging. Unfortunately, no samples were saved from the peel testing and analysis of the peel patterns was not possible.

Table 26. ANOVA and Regression for Peel Adhesion of Skim-Coat Compound

Tire Type		B	C	D	E	H	L
Test	**Variable**						
Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.44	0.45	0.80	0.34	0.37	0.61
	Hours	- 8.48	- 6.23	- 9.56	- 4.82	- 18.30	- 9.68
	Test				LTDE > P-END		
Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.91	0.67	0.54	0.92	0.63	0.85
	Temperature	- 100.44	- 79.79	- 68.79	- 82.53	- 53.43	- 76.77
	Hours	- 2.44	- 0.79	- 0.49	- 0.76	- 0.88	- 1.96
	Fill Gas			Air > 50/50			
	Break-in						
Phoenix	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	0.0401	< 0.001
	Model R^2	0.50	0.46	0.66	0.66	0.47	0.54
	Weeks	- 15.97	- 15.07	- 48.82	- 23.23	- 19.00	- 19.08

The average and 95-percent confidence levels of the peel adhesion for the skim compounds are shown in Figure 31 to Figure 36. Testing by the LTDE or P-END test gradually reduced the peel adhesion values for all tire types. At the longest testing time of 500 hours, or about 3 weeks, the peel adhesion was approximately the same as that observed for tires with 1 to 3 years of service in Phoenix. Oven aging at 60°C to 70°C for 3 weeks produced approximately the same reduction in peel adhesion. Oven aging for 6 weeks at 60°C to 70°C or 12 weeks at 55°C reduced the peel adhesion to approximately that observed in tires with 4 to 6 years of service in Phoenix, while oven aging for 8 weeks at 65°C further reduced the peel adhesion values.

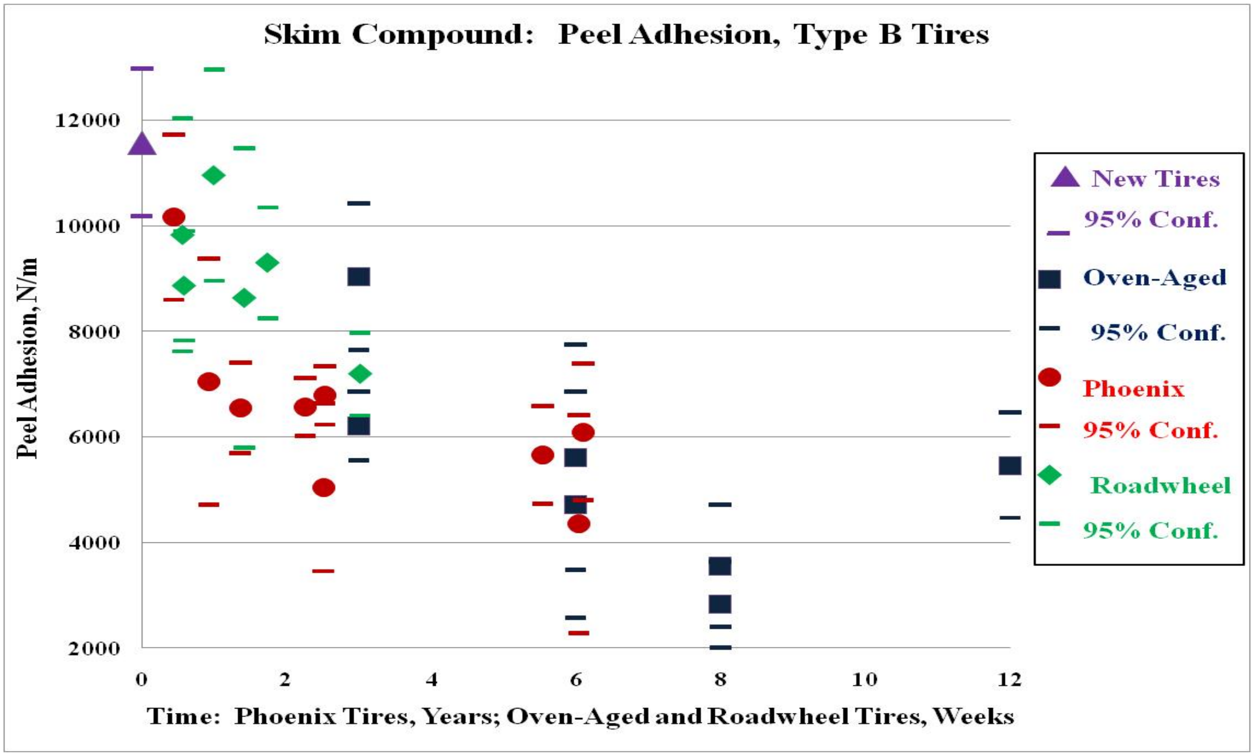


Figure 31. Peel Adhesion Type B Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

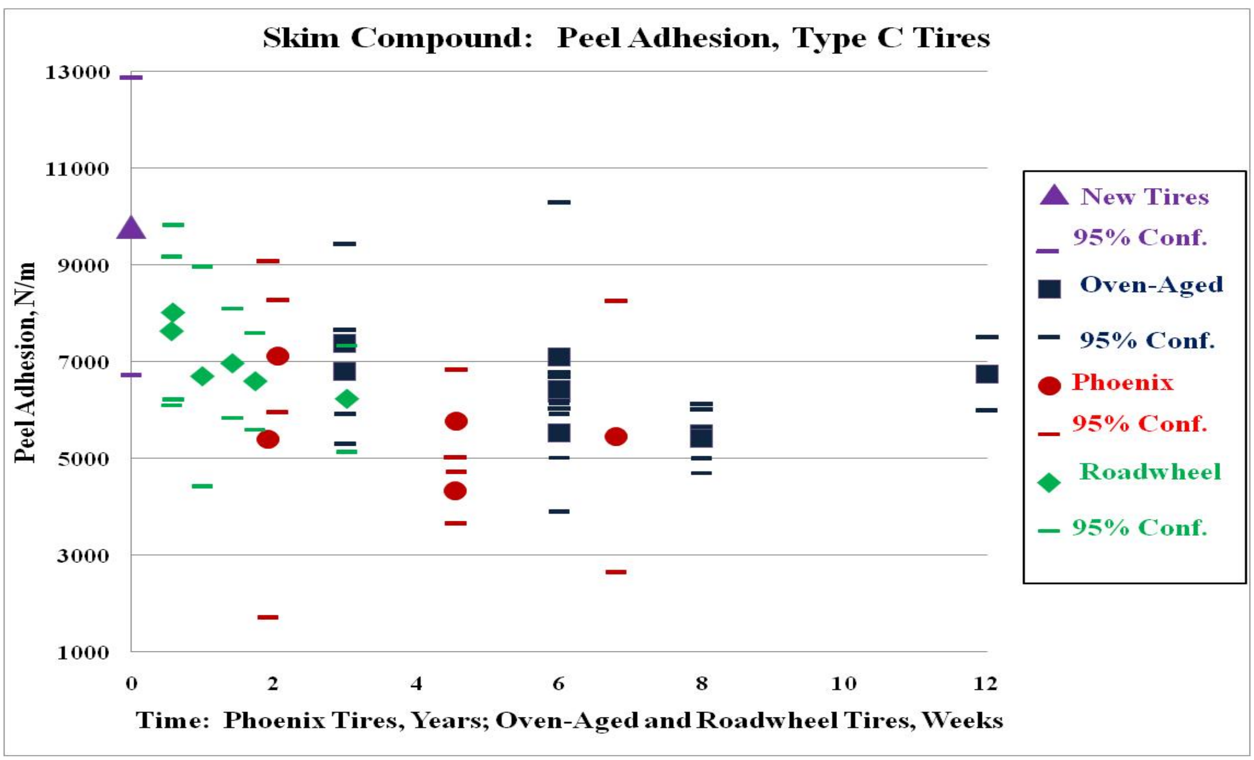


Figure 32. Peel Adhesion Type C Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

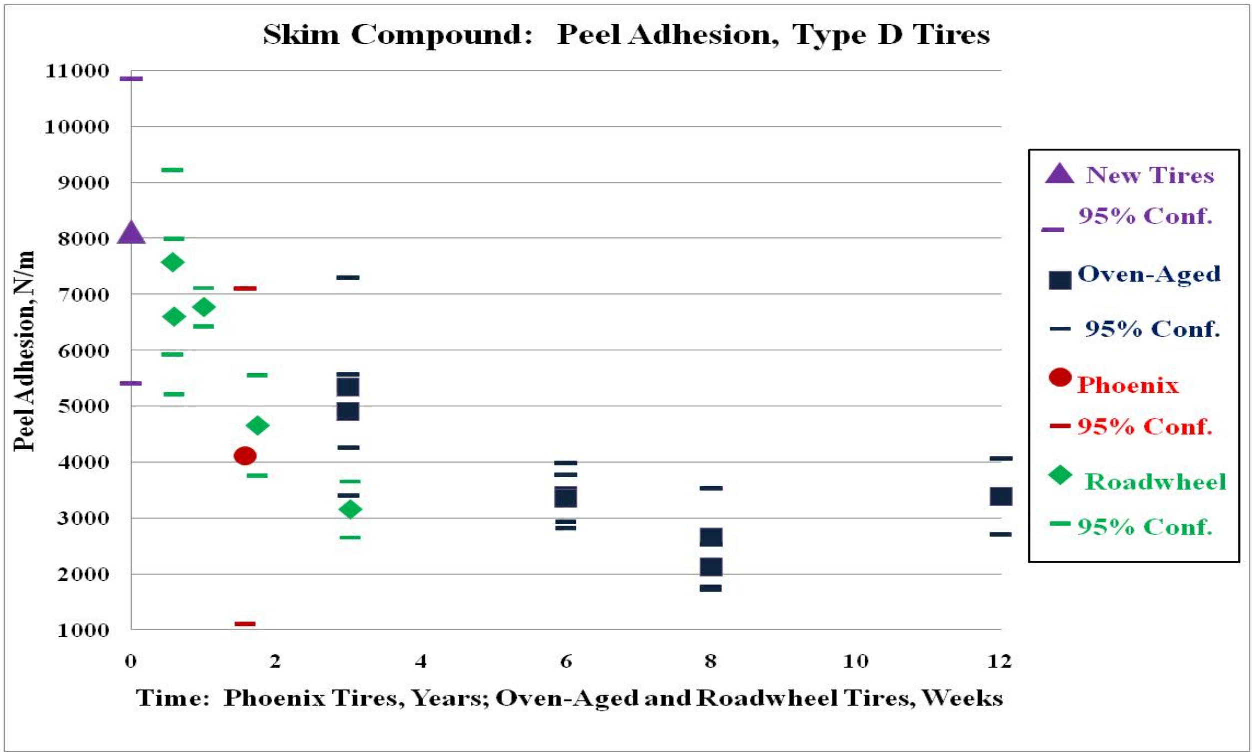


Figure 33. Peel Adhesion Type D Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

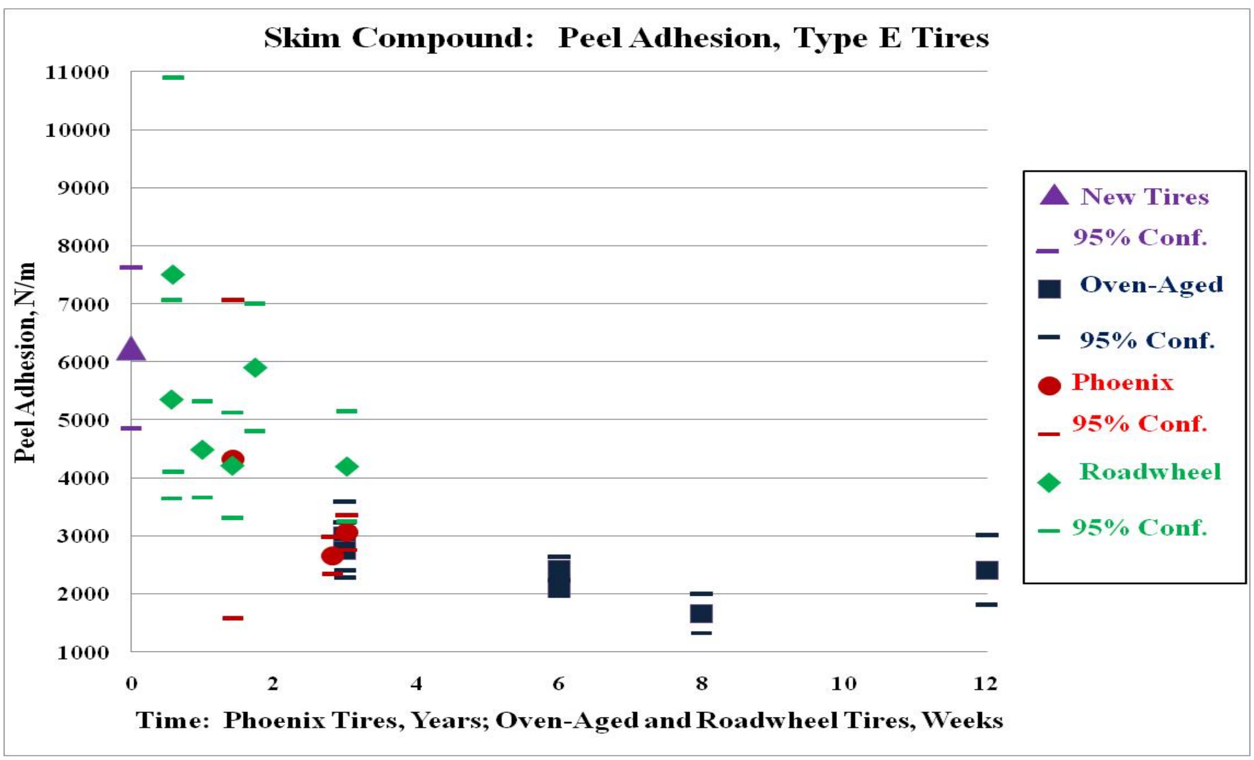


Figure 34. Peel Adhesion Type E Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

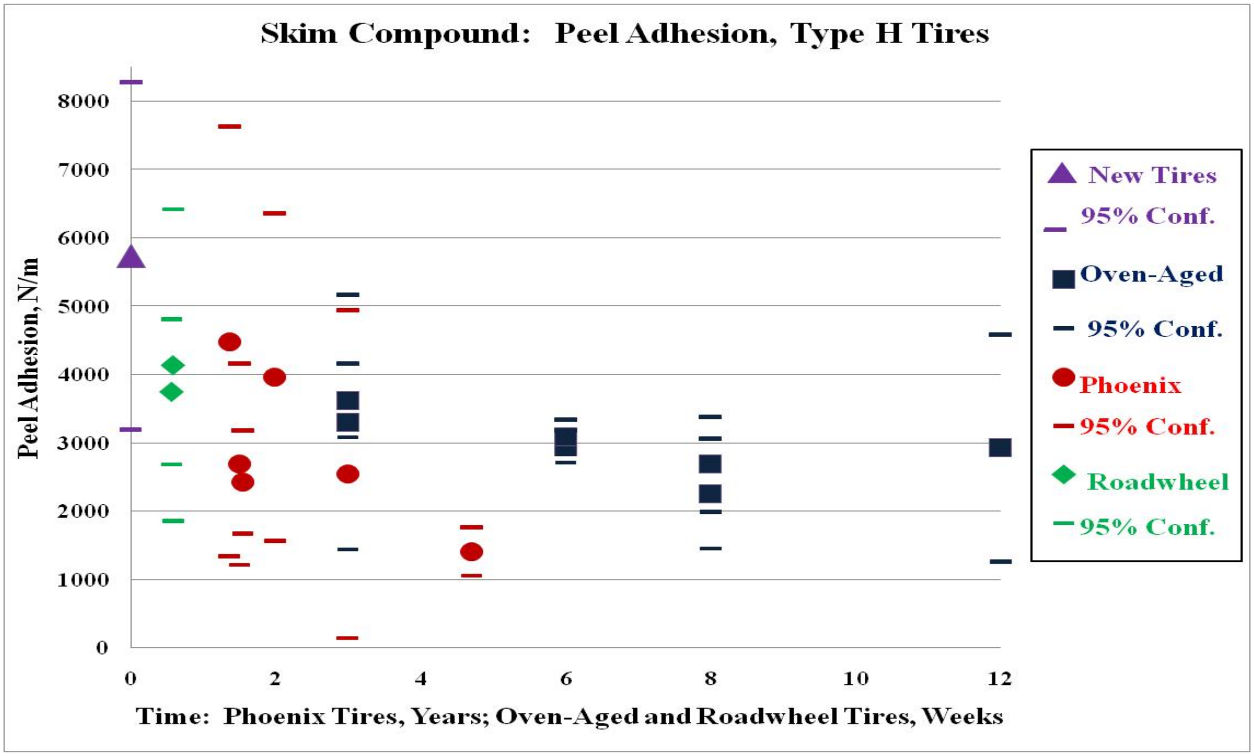


Figure 35. Peel Adhesion Type H Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

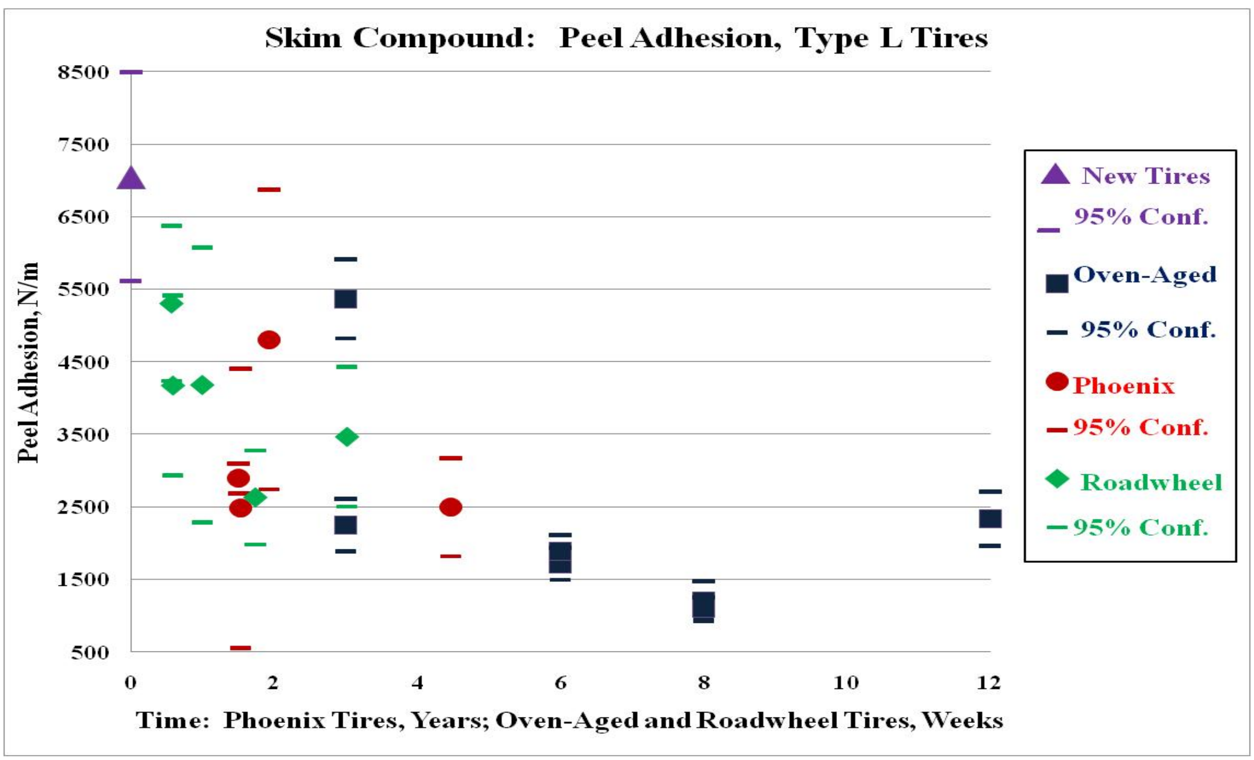


Figure 36. Peel Adhesion Type L Tire Skim Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

3.2.7.2 Peel Adhesion – Wedge Compound

The peel adhesion of the wedge compound parallels the behavior of the skim-coat compound. The results of the ANOVA and regression are shown in Table 27. On the roadwheel tests, all tire types consistently decreased in adhesion over time. Only the type E tire showed any significant sensitivity to the type of roadwheel test, with the P-END test producing a greater adhesion loss per hour of testing than the LTDE test. In the oven testing the peel strength tended to be very sensitive to increases in temperature and consistently showed a reduction in peel adhesion with increased time of aging. Tire type D had a significantly greater adhesion loss 50%N_2/50%O_2 than for inflation with air. No samples were saved from the peel testing and analysis of the peel patterns was not possible.

Table 27. ANOVA and Regression for Peel Adhesion of Wedge Compound

Tire Type		B	C	D	E	H	L
Test	**Variable**						
Roadwheel	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.63	0.40	0.62	0.51	0.61	0.54
	Hours	- 16.27	- 6.26	- 8.75	- 9.41	-28.14	- 14.60
	Test				LTDE > P-END		
Oven	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Model R^2	0.80	0.81	0.47	0.86	0.75	0.76
	Temperature	- 104.4	- 99.22	- 62.35	- 92.67	- 55.57	- 87.16
	Hours	- 1.73	- 0.36	- 0.34	- 1.19	- 1.48	- 2.25
	Fill Gas			Air > 50/50			
	Break-in						
Phoenix	Pr > F	< 0.001	< 0.001	< 0.001	< 0.001	0.0401	< 0.001
	Model R^2	0.23	0.58	0.92	0.74	0.53	0.55
	Weeks	- 18.49	- 27.95	- 62.87	- 58.34	- 34.79	- 35.66

The average and 95-percent confidence levels of the peel adhesion for the wedge compounds are shown in Figure 37 to Figure 42. Testing by the LTDE or P-END test gradually reduced the peel adhesion values for all tire types. At the longest testing time of 500 hours, or about 3 weeks, the peel adhesion was approximately the same as that observed for tires with 1 to 3 years of service in Phoenix. Oven aging at 60°C to 70°C for 3 weeks also produced approximately the same reduction in peel adhesion as tires with 1 to 3 years of service in Phoenix. Oven aging for 6 weeks at 60°C to 70°C or 12 weeks at 55°C reduced the peel adhesion to approximately that observed in tires with 4 to 6 years of service in Phoenix, while oven aging for 8 weeks at 65°C further reduced the peel adhesion values.

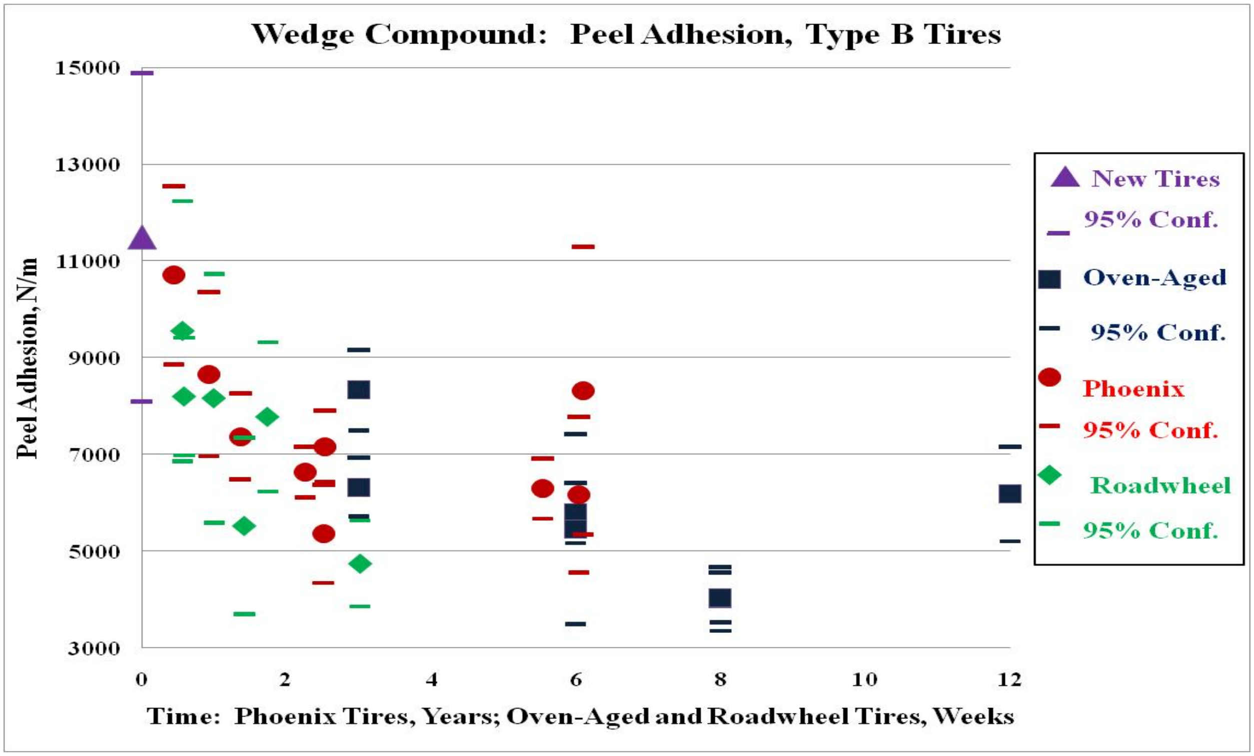


Figure 37. Peel Adhesion Type B Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

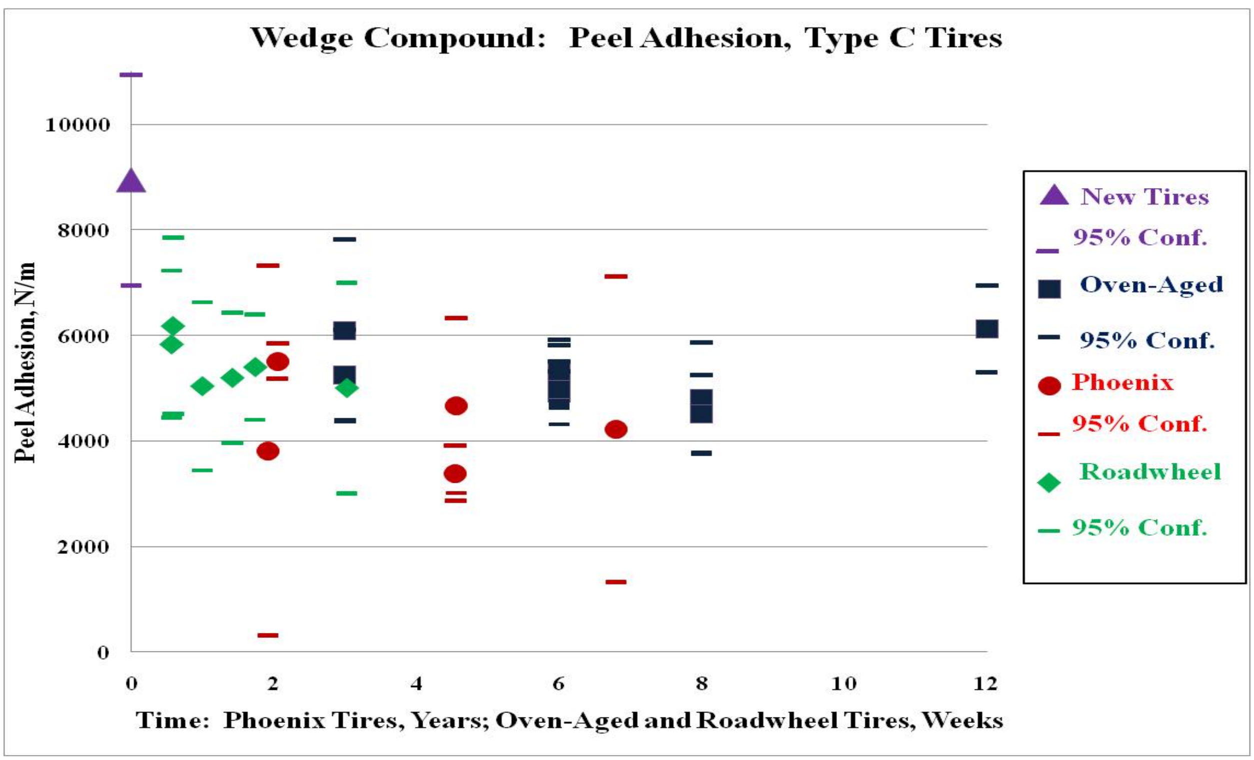


Figure 38. Peel Adhesion Type C Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

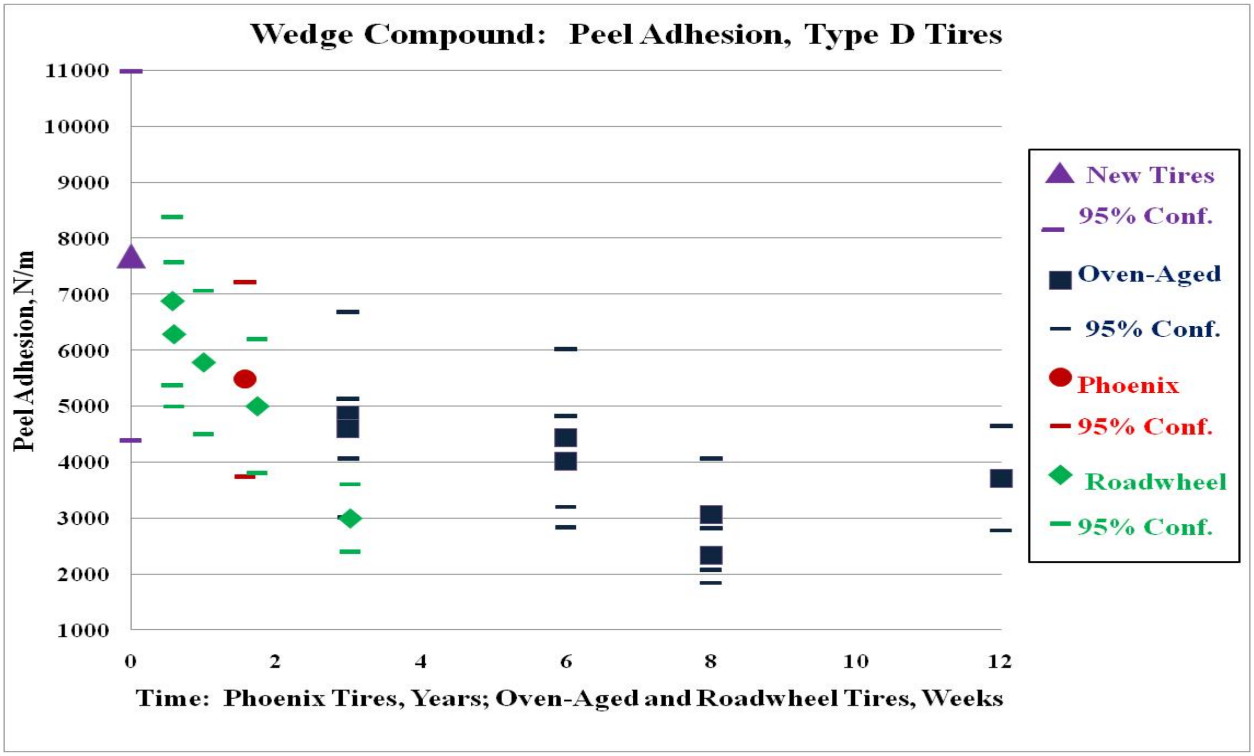


Figure 39. Peel Adhesion Type D Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

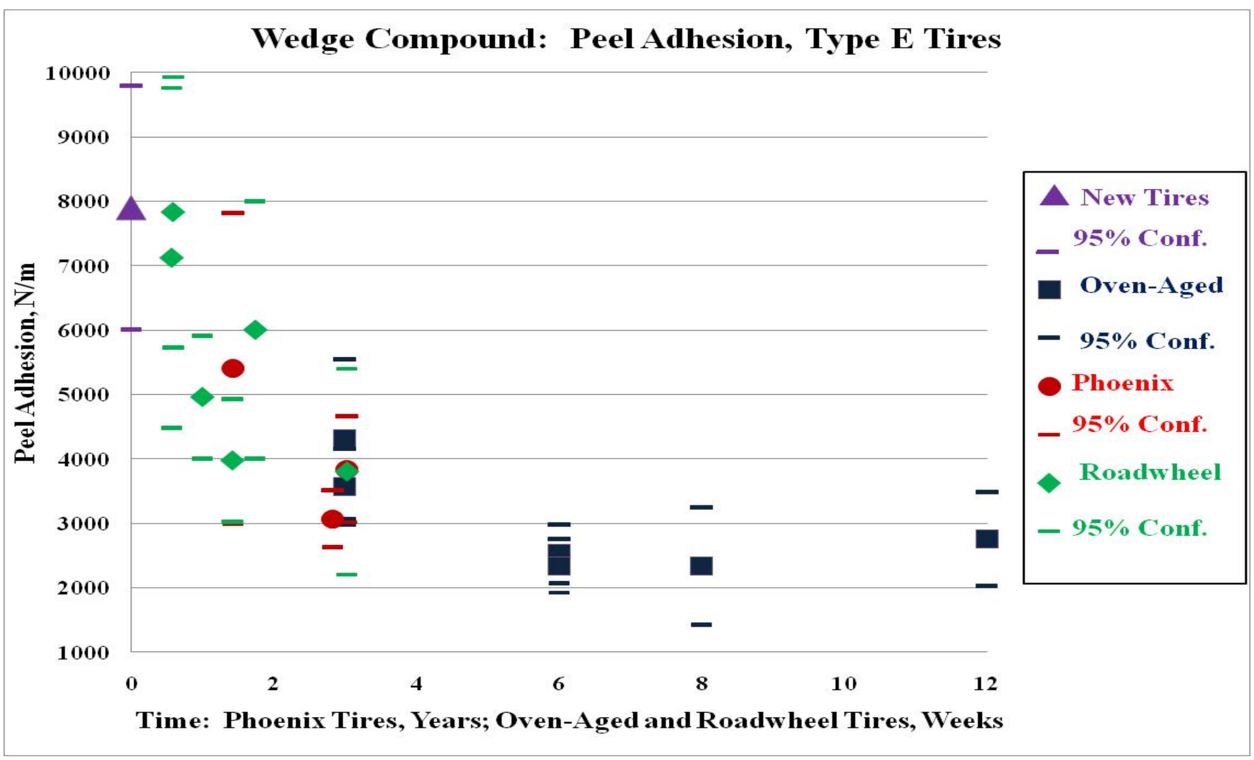


Figure 40. Peel Adhesion Type E Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

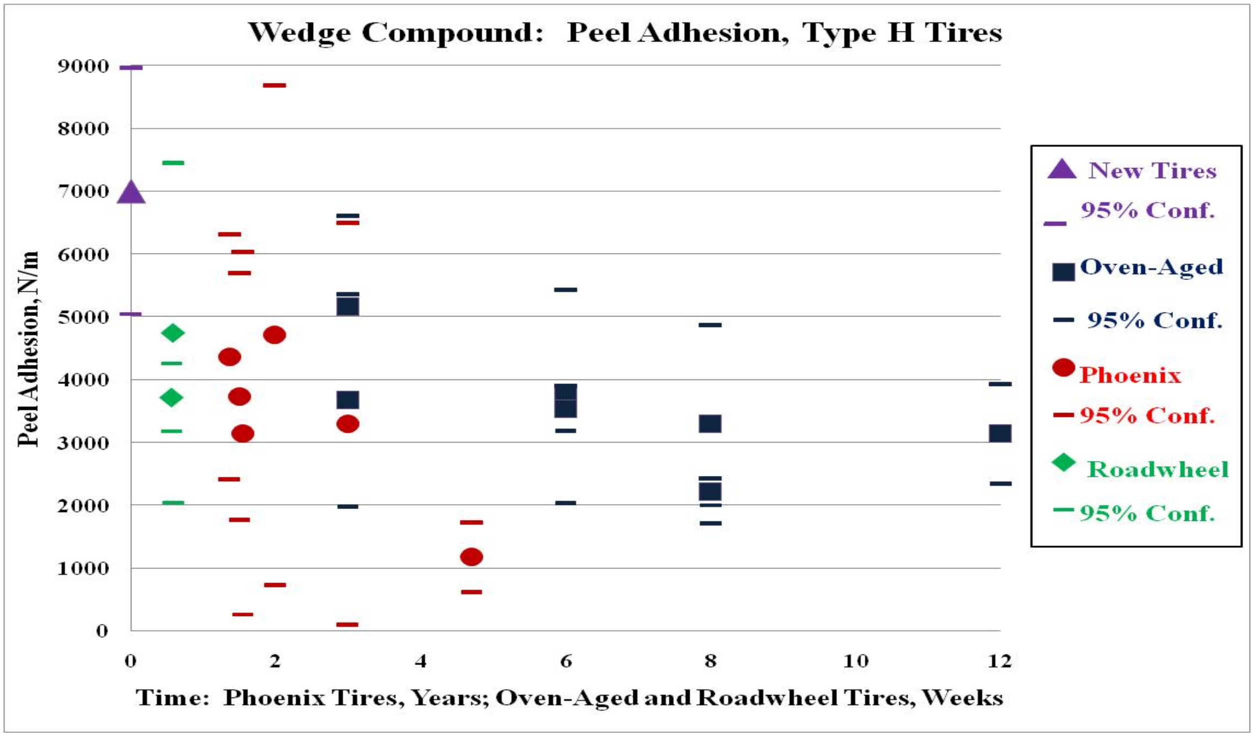


Figure 41. Peel Adhesion Type H Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

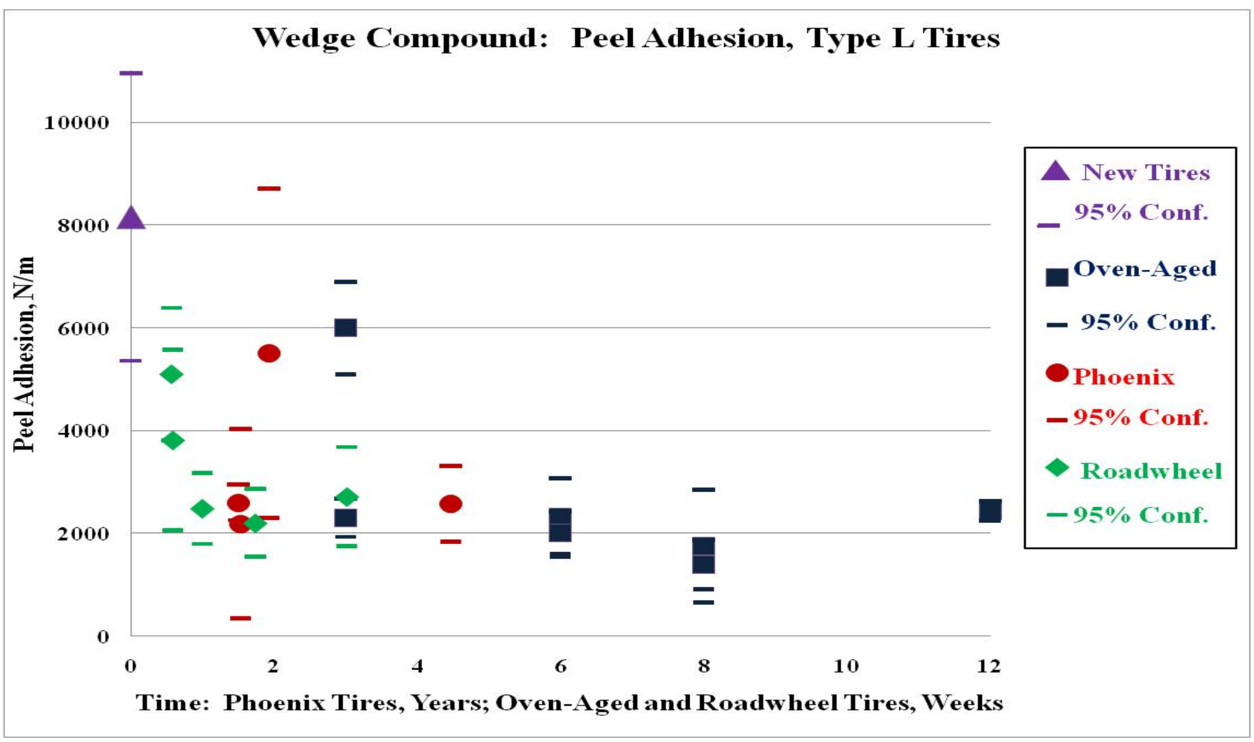


Figure 42. Peel Adhesion Type L Tire Wedge Compound After Oven-Aging, Roadwheel Testing or Service in Phoenix

3.2.8 Two-Ply Laminate Fatigue Test

Results of the two-ply laminate fatigue test have been reported separately.[19] Preliminary data analysis of the variables showed that cycles to failure or log of cycles to failure had no statistically significant correlation to the age of the tire or to the mileage accumulated in Phoenix service. As expected from laminar theory, the new tires showed a relatively good fit to a linear decrease in log of cycles to failure as the strain increased. The modeled difference between the expected log of cycles to failure at the input strain and the actual log of cycles to failure found in the testing showed a general trend for the log of cycles to failure to decrease more than expected as the tire age and mileage increased. In all cases, the scatter in the data was too great to support anything more than general conclusions about trends in the data.

Analysis showed that cycles to failure or log of cycles to failure at any of these strains had no statistically significant correlation to tire type, age, mileage, or duration of accelerated roadwheel aging. However, since the test machine runs in "load control," small differences in the amount of strain experienced by each sample per unit load can result in large differences in input energy (load x displacement) over the many tens or hundreds of thousands of cycles to failure. As a result, the test laboratory ARDL developed a new analytical technique to calculate the cycles to failure at an average energy input of 500 N-mm. This would allow for comparisons of test results between samples from the same tire and samples from different tires. This new method uses a power law fit to the average input energy versus the number of cycles to failure in the steady state crack growth phase of the test to calculate an average number of cycles to failure at a total input energy of 500 N-mm (termed "Cycles to Failure at 500 N-mm").

Three of the five models collected from service in Phoenix showed a relatively strong correlation of the Cycles to Failure at 500 N-mm variable to both the mileage and age variables, with a general reduction in the cycles to failure with increasing age and mileage. Correlations improved only slightly when results were plotted against an estimated mileage per year rather than age or mileage separately. Excessive scatter resulted in no correlation for the other two tire models.

New versions of the six tire models collected in Phoenix were subjected to the two-ply analysis following one of two laboratory roadwheel tests supplied by the tire industry for evaluation. Five of the six tire models showed a relatively strong correlation to time on the roadwheel test for the Cycles to Failure at 500 N-mm calculation, with cycles to failure decreasing with increasing test duration. For these five tire models, the duration of the test was statistically significant and yielded very similar results regardless of the roadwheel test method used. In sharp contrast, the "Type B" BFGoodrich tire model, the smallest and lightest tire of the group, was almost completely insensitive to durations of 100, 292, and 508 hours of the Michelin Long-Term Durability Endurance (LTDE) test and 240 hours of the Continental P-END test. No explanation for this anomalous behavior was apparent.

3.2.9 Shearography

Detailed analysis of the internal separations measured by shearography has been reported.[17] Figure 43 displays the results of full bead-to-bead shearography for the new and Phoenix-collected on-road tires that were subjected to roadwheel testing. The total level of separation was plotted against the age of the tires at collection. New tires of each model had essentially no measurable separation when tested (i.e., no detectable manufacturing anomalies). For some tire models, the total level of internal separation increased significantly in older tires, for other models it did not. With the exception of cases of probable damage to the tread or sidewall, the separations in all six tire models were almost exclusively located at the edges of the two steel belts. This makes the region at the two belt-edges the region of focus when attempting to artificially simulate the state of in-service tires.

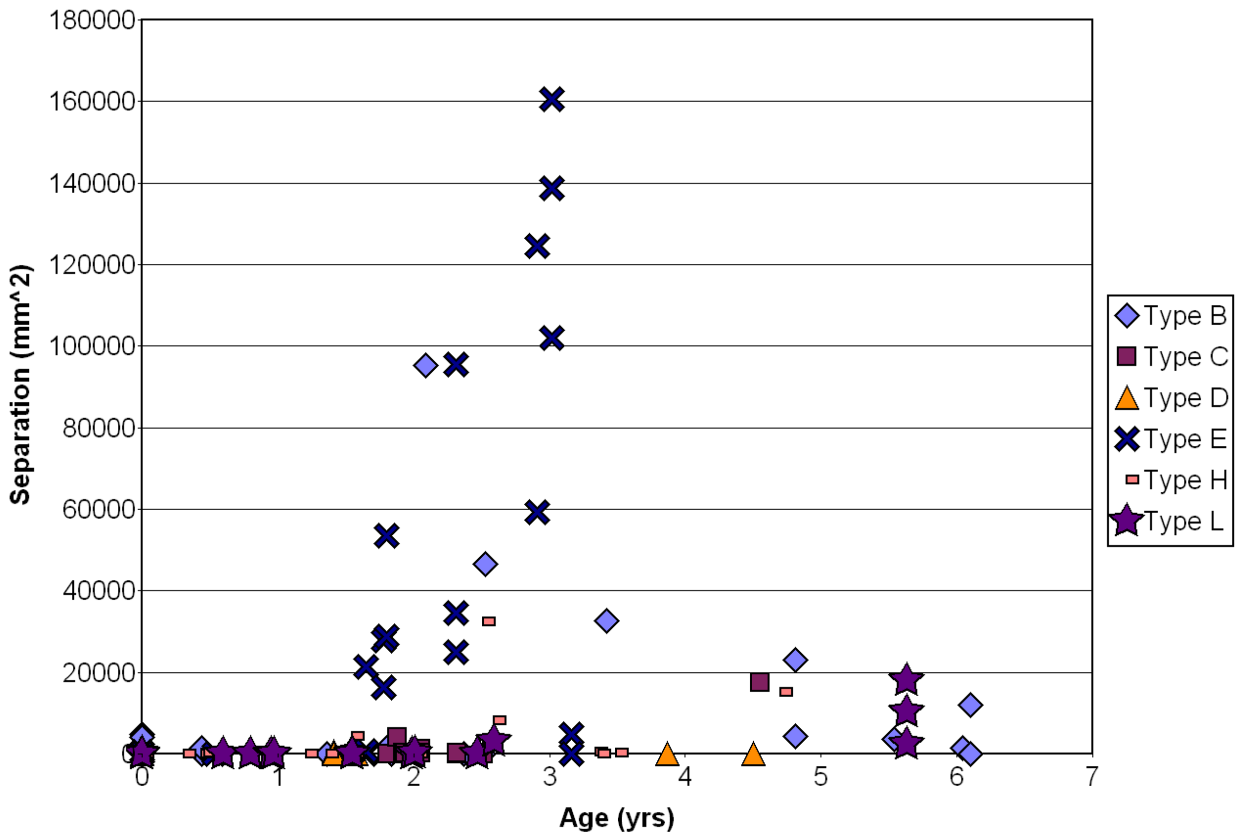


Figure 43. Bead-to-Bead Shearography Separation @ 50 mbar Vacuum Versus Age, New, and Phoenix On-Road Tires (45 New Tires, 106 Phoenix Tires)

Figure 44 plots the measured level of internal separation versus the mileage of the tires. (The mileage of replacement tires has been estimated from tire age and annual vehicle mileage.) Again, for some tire models the total level of internal separation increased significantly in higher mileage tires, for other models it did not.

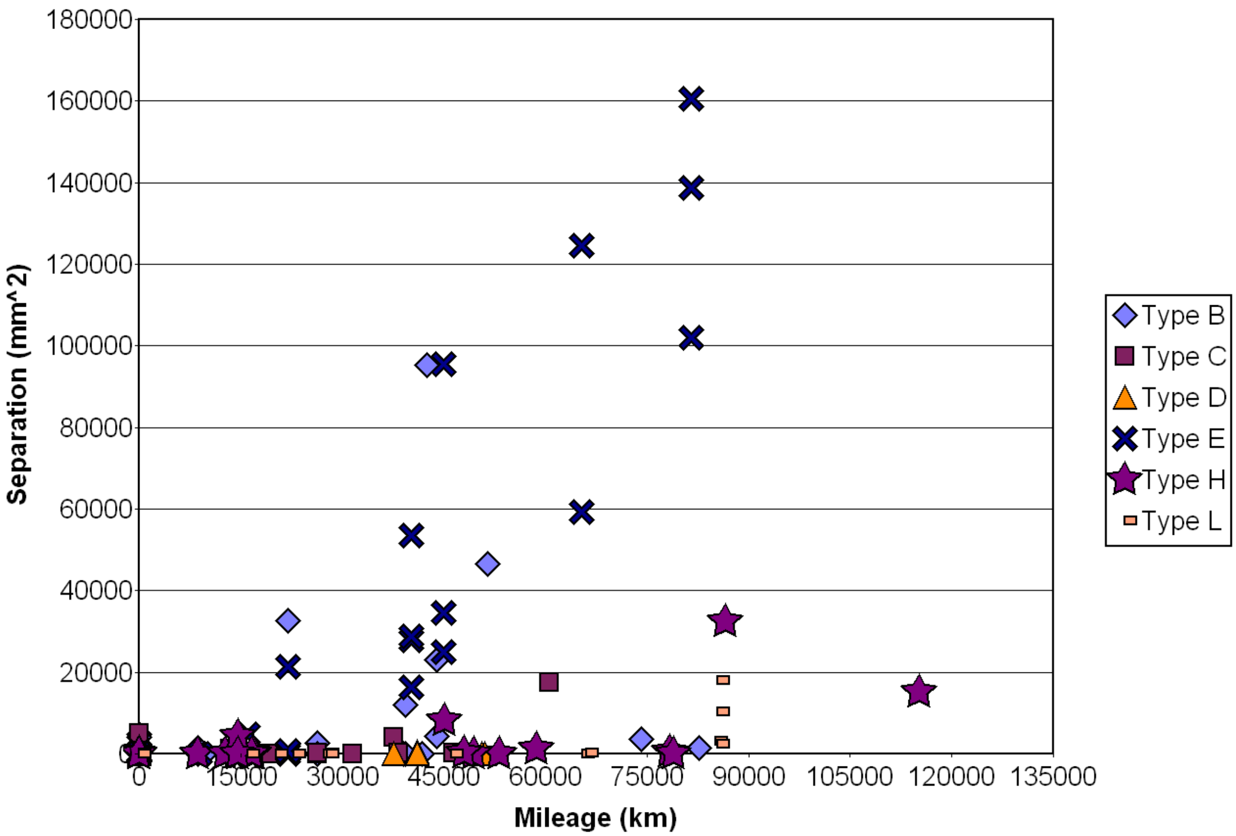


Figure 44. Bead-to-Bead Shearography Separation @ 50 mbar Vacuum Versus Mileage, New, and Phoenix On-Road Tires (45 New Tires, 106 Phoenix Tires)

Though the mileage is estimated for the replacement tires, the results shown in Figure 44 suggest that total level of internal separation appears to correlate to mileage better than to the age of the tire. This is an intuitive observation, since the initiation and growth of internal cracks and separations are driven primarily by the cyclical deformation (fatigue) of the tire during the 5 percent of the time they are operated rather than the 95 percent of the time the vehicle is parked.

The ANOVA analysis of the roadwheel tested tires showed no statistically significant differences among the rates of generating separations in the tires. Table 28 shows the coefficients of the linear regression separation in mm^2 versus hours of running time for the roadwheel-tested tires. The low R^2 values do not allow more than a general comparison, but it appears that the road-wheel tests do not correlate with the results found from the Phoenix tires. Note particularly the low rate of separation for the type E tires, which had the highest amount of separation in Phoenix service and the relatively high rate for type D tires which showed almost no separation during Phoenix service.

Table 28. Linear Regression Coefficients: Shearography mm2 Separation per Hour of Roadwheel Time

Tire Type	B	C	D	E	H	L
R^2	0.55	0.15	0.35	0.24	0.87	0.04
mm^2/hour	+ 174	+ 74	+ 189	+ 68	+ 337	+ 76

3.2.10 Fixed Oxygen Level

As was shown in a previous report,[4] the level of fixed oxygen in the skim-coat compound, the wedge compound, and the innermost tread compound increased during service in Phoenix. This is consistent with the anticipated mechanism of aerobic oxidation of these compounds during normal service. The coefficients from linear regression are shown in Table 29. Although there is a great deal of scatter in the data as evidenced by the low R^2 values, the oxygen content of the compounds all increased at rates from 6×10^{-4} percent to 43×10^{-4} (average of $18 \times 10^{-4} \pm 5 \times 10^{-4}$) percent per week. The roadwheel tests produced little or no significant change in the fixed oxygen level of the compounds for any tire types.

Table 29. ANOVA and Linear Regression of Percent Fixed Oxygen Level for Skim-Coat, Wedge and Innermost Tread Compounds

Test	Component	Tire Type / Property	B	C	D	E	H	L
Phoenix	Skim-Coat	R^2	0.72	0.39	0.47	0.29	0.46	0.21
		O_2/week	+ 0.0015	+ 0.0012	+ 0.0021	+ 0.0014	+ 0.0043	+ 0.0009
	Wedge	R^2	0.56	0.82	0.22	0.33	0.52	0.08
		O_2/week	+ 0.0015	+ 0.0026	+ 0.0016	+ 0.0017	+ 0.0040	+ 0.0006
	Tread	R^2	0.30	0.33	0.30	0.01	0.34	0.19
		O_2/week	+ 0.0019	+ 0.0017	+ 0.0025	-	+ 0.0024	+ 0.0015
Roadwheel	Skim-Coat	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.18	0.16	0.04	0.08	0.04	0.05
		O_2/hour	+ 0.0004	+ 0.0004	+ 0.0002	+	-	+
		Test						
	Wedge	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.11	0.16	0.09	0.23	0.12	0.00
		O_2/week	+	+ 0.0006	-	-	-	-
		Test						
	Tread	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.02	0.42	0.06	0.36	0.05	0.10
		O_2/hour	+	-	-	-	-	+
		Test		P-END > LTDE		P-END > LTDE		
Oven	Skim-Coat	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.59	0.37	0.63	0.68	0.57	0.61
		O_2/hour	+ 0.0002	+	+ 0.0004	+ 0.0004	+ 0.0001	+ 0.0002
		O_2/degree	+ 0.0017	+ 0.0042	-	+	+ 0.0045	+ 0.0025
		Fill Gas	50/50 > Air	Not Measured	50/50 > Air	50/50 > Air	Not Measured	Not Measured
	Wedge	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.34	0.50	0.51	0.75	0.46	0.41
		O_2/hour	+ 0.0001	+ 0.0002	+ 0.0002	+ 0.0003	+	+ 0.0004
		O_2/degree	+	+ 0.0018	-	-	+ 0.0035	-
		Fill Gas	50/50 > Air	Not Measured	50/50 > Air	50/50 > Air	Not Measured	Not Measured
	Tread	F Value	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		R^2	0.03	0.04	0.38	0.20	0.15	0.40
		O_2/hour	+	-	+ 0.0002	+ 0.0002	+	-
		O_2/degre	+	+	-	-	-	+ 0.0078

		e						
		Fill Gas	50/50 > Air	Not Meas-ured	50/50 > Air	50/50 > Air	Not Meas-ured	Not Meas-ured

3.2.11 Oxygen Depletion During Oven Aging

The oxygen content of the fill gas of the tires was measured at the beginning of the oven aging period and at the end of 3 and 6 weeks of aging at 60°C or 70°C. The oxygen content of the fill gas decreased from an average of 45% during the initial fill to approximately 35% after 6 weeks of oven aging, as shown in Figure 45. Based on the regression, both conditions will have depleted the oxygen to normal atmospheric oxygen content of 21% by 9 weeks and theoretically would deplete all available oxygen by 12 weeks at 70°C or 14 weeks at 60ºC. This may have contributed to the evidence of anaerobic aging noted in the Ahagon plots and minimal levels of increased fixed oxygen in the wedge compound. Based on these data, replenishment of the 50/50 nitrogen/oxygen fill gas was recommended for the next phase of test development.

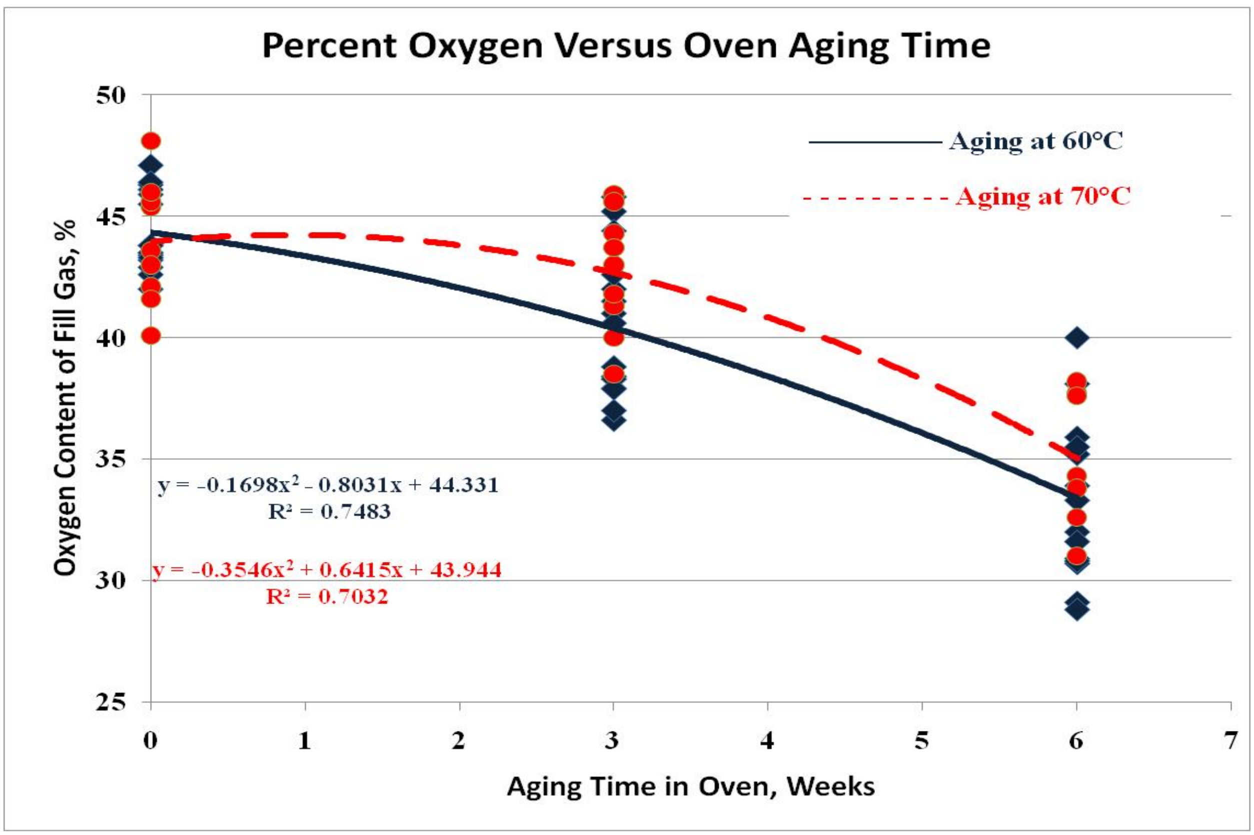


Figure 45. Percent Oxygen Content of the Fill Gas Versus Oven-Aging Time

3.2.12 Roadwheel Testing: Stepped-up Load to Structural Failure After Oven Aging

Selected tires were subjected to testing on a 1.7-meter roadwheel after they had been oven aged for varying periods of time. The conditions of the roadwheel test are shown in Table 30. The first 34 hours of the test are the same as those of the FMVSS 139 Endurance Test.[21] After the first 34 hours, the load is increased every 4 hours by increments of 10 percent of the maximum rated load. The results were compared to the average predicted data for tires removed from service in Phoenix. The results and discussion of the Phoenix service tires have been reported separately.[17]

Table 30. Stepped-Up Load Roadwheel Test Conditions

Test Stage	Duration (hours)	Load as a percentage of tire maximum load rating	
Optional Break-in	24	100%	
1	4	85%	FMVSS 139
2	6	90%	FMVSS 139
3	24	100%	FMVSS 139
Inspection	1		
4	4	110%	Until Failure
5	4	120%	Until Failure
Etc.	4	Increment load by +10% every 4 hours until failure	Until Failure

The stepped-up load test was intended to compare the structural integrity of the tire after aging to that of a new tire, stepping the tires through their normal range of operational deflections and then beyond until ultimate failure occurs. Consequently, many tires failed at loads much higher than their maximum rated load capacity. Since this is undoubtedly higher than their designed load capacity by some factor, tires that failed past the 34-hour/100 percent load step are not necessarily expected to show correlation to failures that may happen in normal tire service. The results are shown in Figure 46 to Figure 51 compared to the predicted values from linear regression of the tires taken from Phoenix service at varying ages. The mean predicted failure time for the Phoenix tires is indicated by the solid black line, and the 95-percent confidence limits above and below the mean are indicated by the dashed light blue lines. The green line at 34 hours represents 100 percent of rated load and testing at loads above this level stress parts of the tires, such as the sidewall and bead, beyond that which is expected during normal operation, often causing failures to occur in these components.

Noting that the tests shown in the bar charts below usually represent a single test and have significant uncertainty, there are general conclusions that can be made.

- Aging tires for 3 weeks does not cause a significant decrease in structural integrity compared to a new tire of the same model.

- Tires aged for 70°C for 3 weeks tended to have longer running times than tires aged at 60°C for 3 weeks.

- The 24-hour break-in prior to oven aging tended to decrease the running time in the test after aging. If the 24 hours are added to the test time (light pink bars in the following charts), the total running time tends to be longer than the tires without break-in. Note that since the break-in was done at 100 percent of maximum load the only direct comparisons possible are for tires that failed at less than 34 hours on the test after aging (note the results for tire models D, E and H aged 8 weeks at 65°C).

- Tire models C and L had no failures from roadwheel testing at loads below 100 percent of maximum load. Both of these models also had predicted failure times above 100 percent load even after 6 years of service in Phoenix.

- Tire models B and D showed failures below 100 percent load at aging times of 8 weeks at 65°C and predicted failure times below 100 percent load after 5 or more years of service in Phoenix.

- Roadwheel testing results for tire models E and H appear to be sensitive to oven aging times. They also appear to be the most sensitive to service time 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 the maximum rated load for the tires. 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 the maximum rated load for the tires.

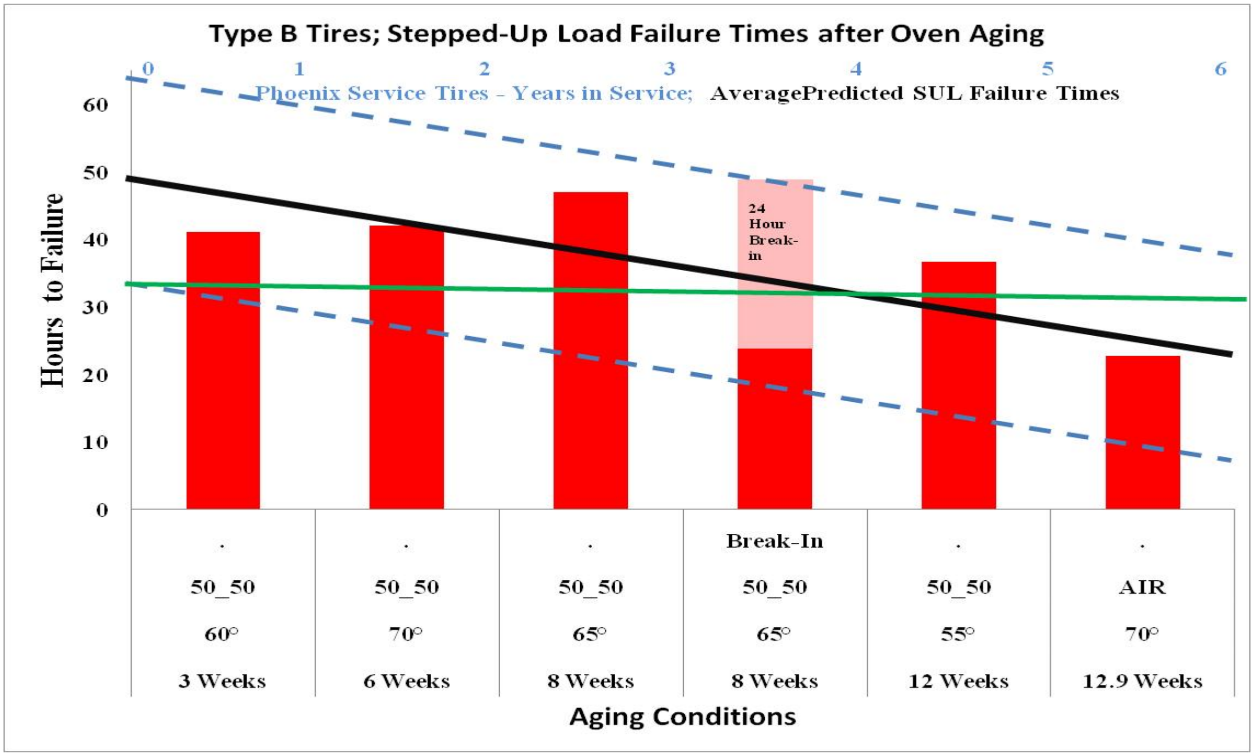


Figure 46. Stepped-up Load Test Failure Times: Oven-Aged Type B Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

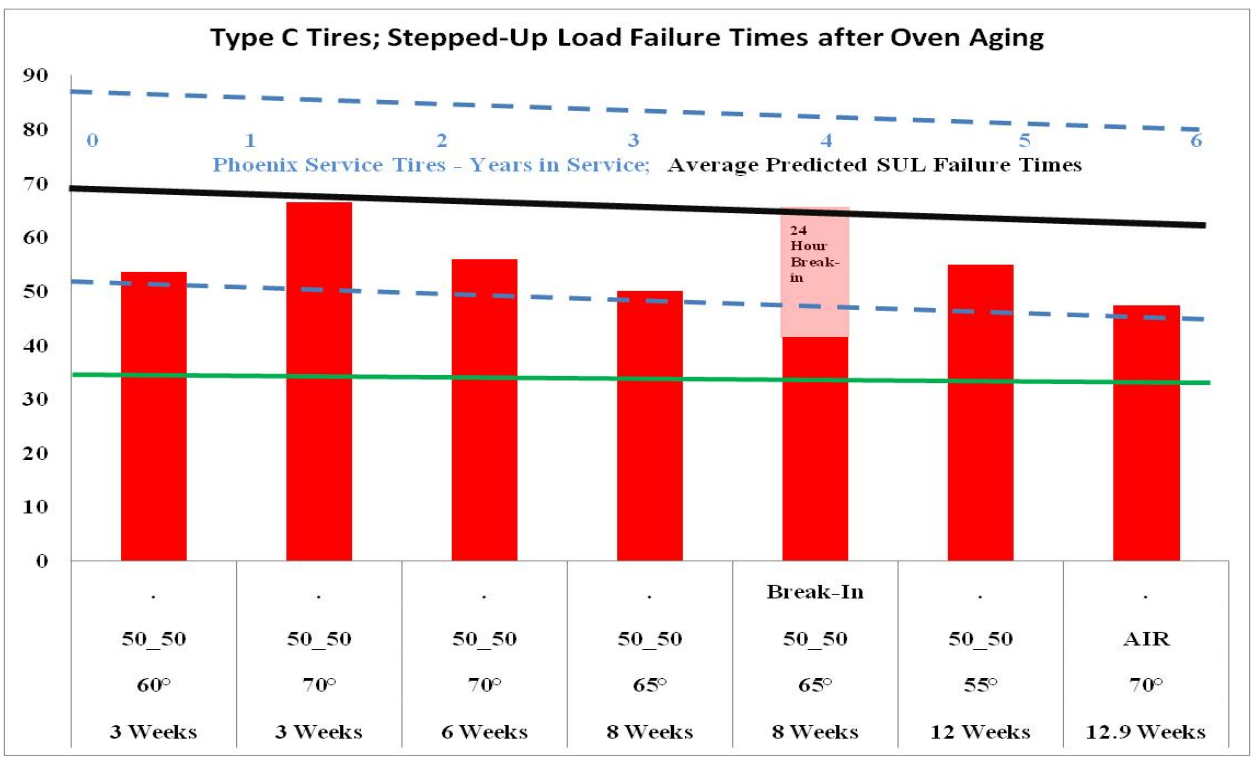


Figure 47. Stepped-up Load Test Failure Times: Oven-Aged Type C Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

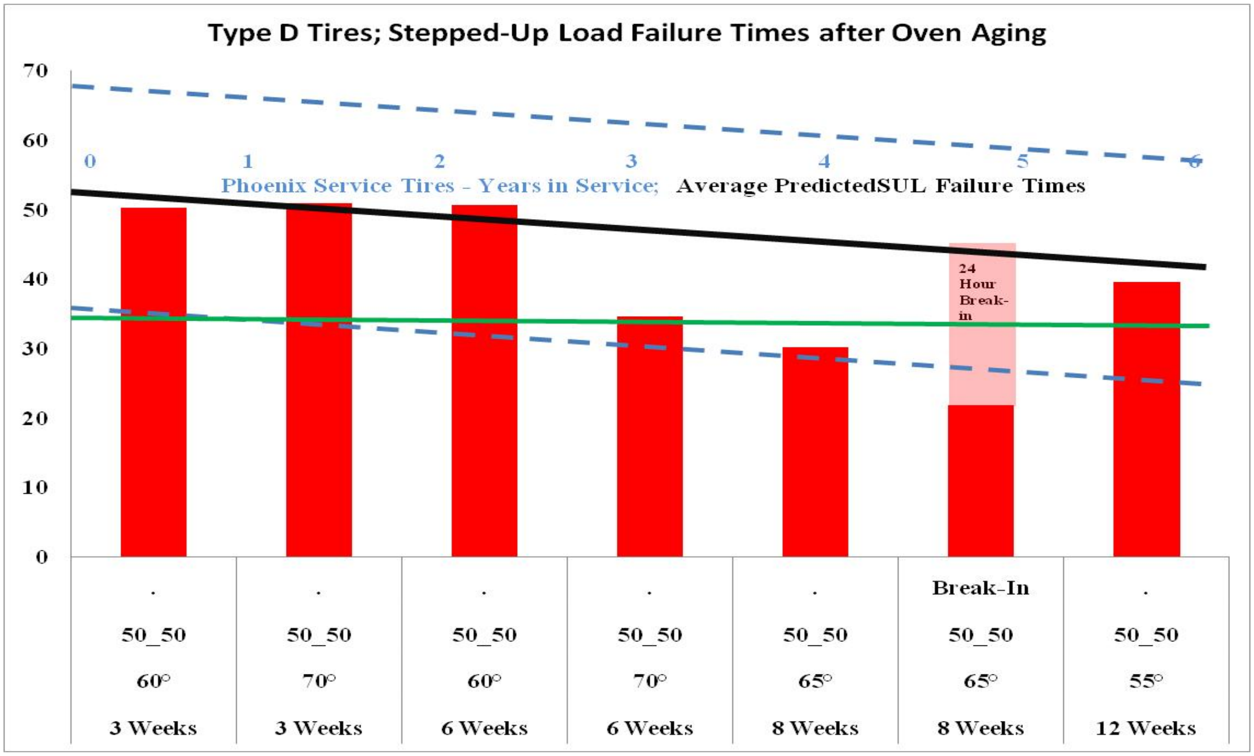


Figure 48. Stepped-up Load Test Failure Times: Oven-Aged Type D Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

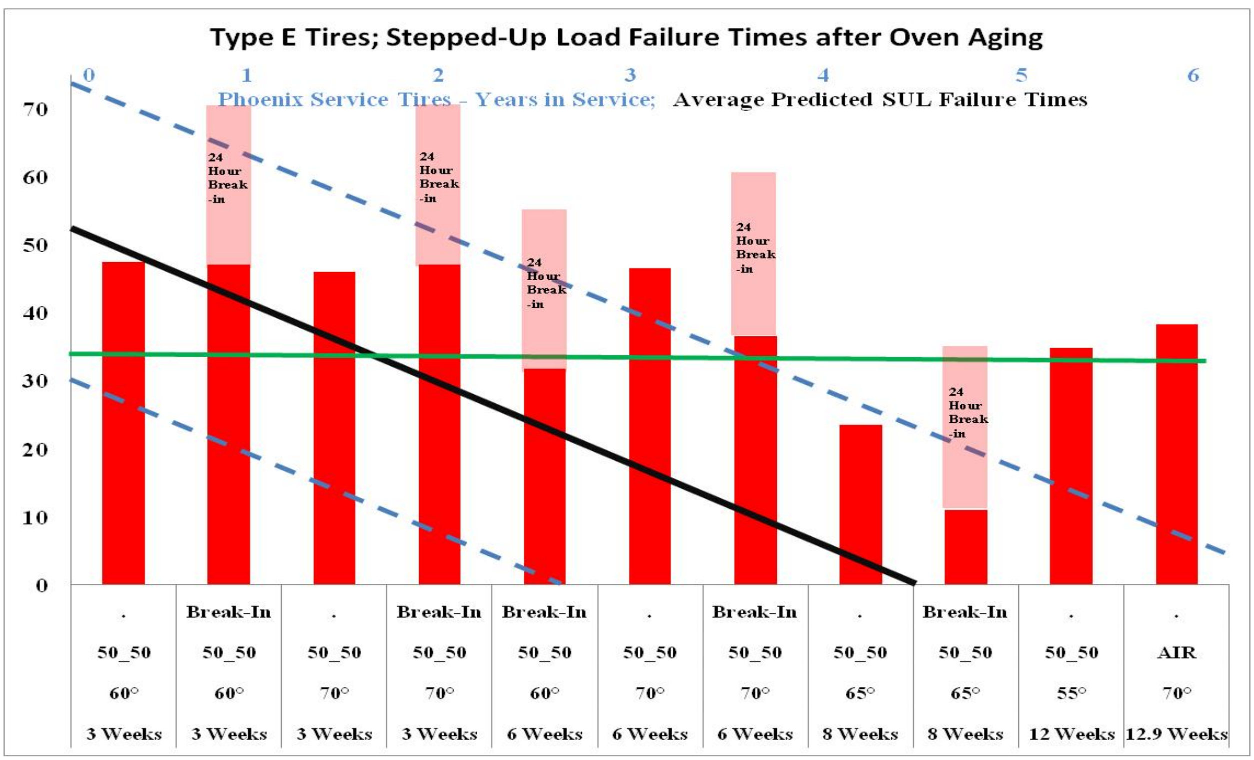


Figure 49. Stepped-up Load Test Failure Times: Oven-Aged Type E Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

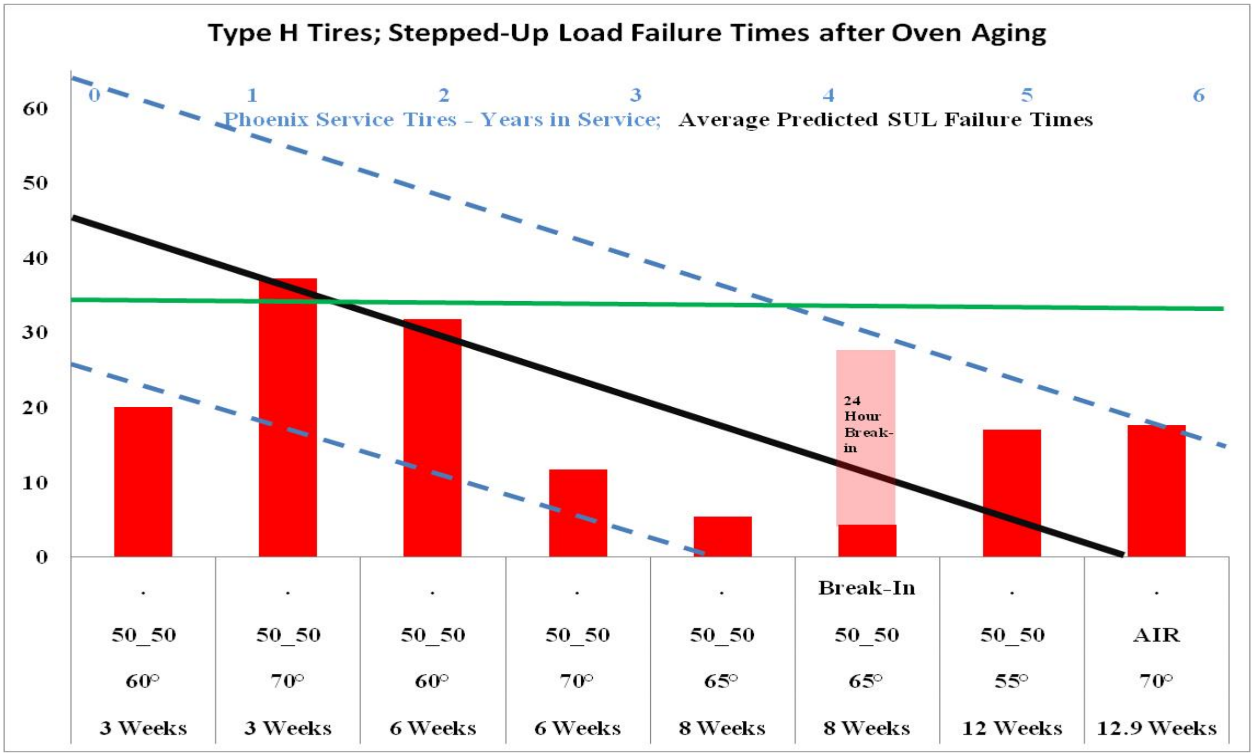


Figure 50. Stepped-up Load Test Failure Times: Oven-Aged Type H Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

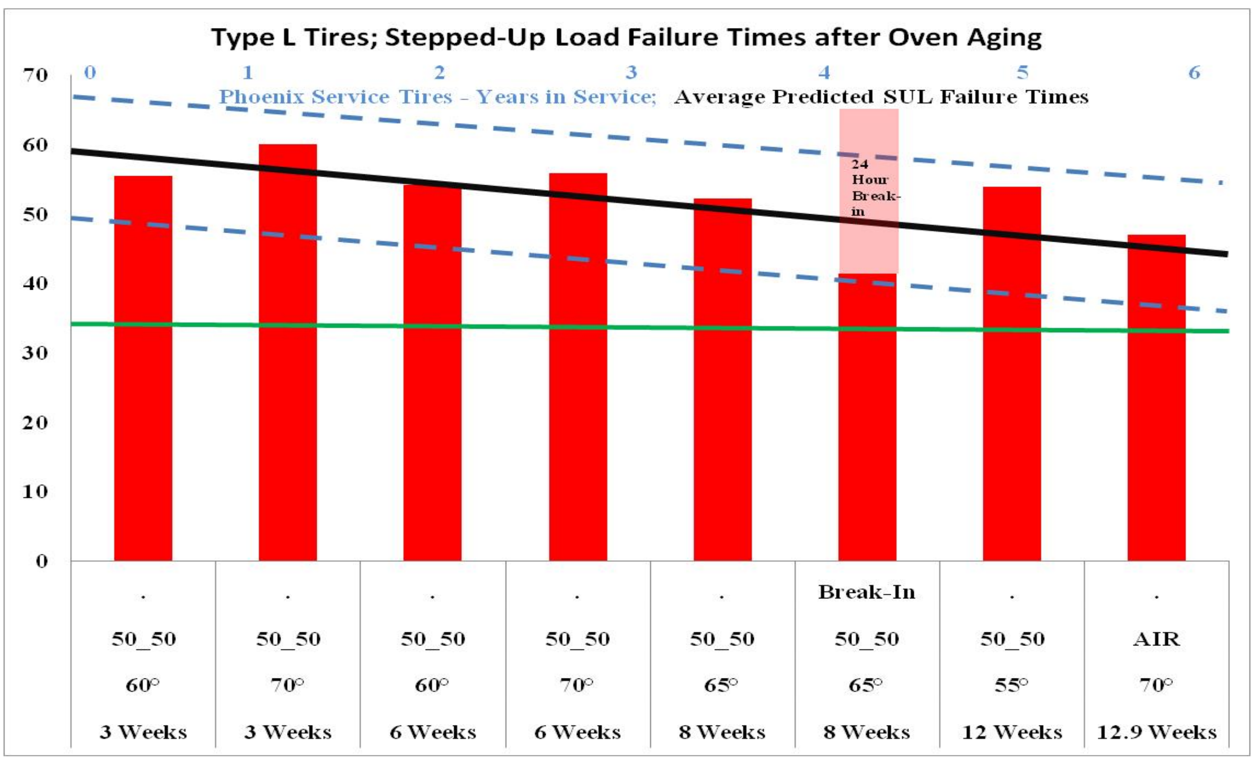


Figure 51. Stepped-up Load Test Failure Times: Oven-Aged Type L Tires Compared to Predicted Values for Tires With 1 to 6 Years of Service in Phoenix

4.0 SUMMARY AND CONCLUSIONS

This work focuses on developing an accelerated, laboratory-based tire test that simulates real world tire aging and evaluates the remaining structural durability of the aged tires. Six tire models that had previously been evaluated after long-term service (aging) on vehicles in Phoenix were evaluated in Phase 1 of the project. In Phase 2 of the project, three methods of laboratory aging were evaluated using new tires of the six models collected from service in Phoenix. Two were combined tire aging and durability tests conducted on the 1.707 m diameter roadwheel: (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 for up to 500 hours. (2) The Passenger Endurance Test proposed by Continental in which the tire is run for up to 240 hours. The third method was based on tire research by Ford, who recommended that the agency use a method in which the tire is inflated using the $50\%N_2/50\%O_2$ 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 components shown in Table 31 were measured for new tires and for tires after each of the candidate aging methods. Over 95 percent of tire failures recorded by NHTSA in its recall and complaint database involve the tire tread and belt area. Therefore, 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 belts in the tire tread and belt area (shoulder area) were studied in the most detail. Canonical correlation of the properties showed that the properties of the components tended to change in the same direction with increased time in service in Phoenix or increased time of laboratory aging on each candidate method. Particularly, the hardness, modulus, cross-link density, and oxygen content tended to increase; and the tensile, 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.

Table 31. Material and Chemical Tests and Tire Components Studied

Material Property Test	Tire Component
Shore A Hardness	Tread
	Liner
	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
Indentation Modulus	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Liner
	Tread
	Fabric Coat Stock
	Overlay Stock
	Tread/Shoulder Wedge
	Shoulder
	Tread Base
Tensile and Elongation	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
180º Peel Adhesion	Skim Area of Belt
	Wedge Area of Belt
Micro-DeMattia Flex	Wedge Area of Belt
Cross-Link Density and Distribution	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Tread Compound
Two-Ply Laminate Fatigue	Cut Section of Tire Belt
Shearography	Whole Tire Test
Fixed Oxygen	Wire-Coat Skim Compound
	Wire-Coat Wedge Compound
	Tread Compound

The data was analyzed using one-way ANOVA and linear regression models to determine what variables had the most significant effect on the change in properties. Nearly all of the ANOVA models had a probability > F value of less than 0.05, indicating that the overall changes in properties taking place during service in Phoenix, or during laboratory aging, were statistically significant. Analysis by linear regression was unable to provide predictive models with R^2 values above 0.95 in most cases because of the scatter in the data. Coefficients of variation for nearly all the test results ranged from 10 percent to over 25 percent.

A brief summary of the effects from each test follows:

Shore Hardness The models for the liner, tread, and wedge compound were not statistically significant for any tire type on the roadwheel tests or from oven exposure. The skim compound gave statistically significant increases in hardness during oven exposure for the type B and E tires. For all tires except the type H, the effect of the use of $50\%N_2/50\%O_2$ fill gas versus air, and the effect of the 24-hour break-in, was to increase the hardness of the skim compound. For the Phoenix tires, the hardness increased with increasing time in service, except for the type H tire, which decreased as time in service increased. Further analysis indicated that this phenomenon was likely due to the use of a phenol-formaldehyde resin in tire type H compounds.

Indentation Modulus Indentation modulus measurements were taken in 0.1-mm increments from interior to exterior surfaces (innerliner to outer tread surface) of cut tire sections to map the changes in modulus of the tire over time. Since each tire type had different components (e.g. some had nylon overlay strips while others did not) the ANOVA and linear regression models were only calculated for the wire coat compound in the critical area of the belt-edge. All types showed a tendency to harden during oven aging, although only the model for the type E tires was statistically significant. Parallel to the results for Shore A hardness, the type H and type L tires showed a tendency to soften during roadwheel testing and for tires retrieved from Phoenix service, while all other types tended to harden. The indentation modulus of the belt coat compounds for tires aged at the most severe conditions on each test were compared to tires of the same model removed from service in Phoenix. The approximate equivalent results are shown in Table 32.

Table 32. Approximate Equivalent Average Indentation Modulus for Laboratory-Aged Tires and Tires in Phoenix Service

Tire Model	Test	Age of Approximately Equivalent Phoenix Service Tire, Years	Notes
B	LTDE, 500 hours	6 to 7	
	P-END, 292 hours	Not Tested	
	Oven, 8 weeks @ 65ºC	6 to 7	
C	LTDE, 500 hours	> 6	
	P-END, 292 hours	5.5	
	Oven, 8 weeks @ 65ºC	> 6	
D	LTDE, 500 hours	4	
	P-END, 292 hours	Not Tested	
	Oven, 8 weeks @ 65ºC	4	
E	LTDE, 500 hours	3	
	P-END, 292 hours	Modulus decreased during laboratory test, but increased in Phoenix	
	Oven, 8 weeks @ 65ºC	3	
H	LTDE, 292 hours	2 to 3[s]	
	P-END, 292 hours	Not Tested	
	Oven, 8 weeks @ 65ºC	Modulus decreased during Phoenix service, but increased during oven aging	
L	LTDE, 500 hours	Modulus decreased during Phoenix service, but increased during roadwheel testing	
	P-END, 292 hours	Not Tested	
	Oven, 8 weeks @ 65ºC	Modulus decreased during Phoenix service, but increased during oven aging	

Tensile and Elongation For all tire types, tensile strength and the elongation to break of the wire coat compounds decreased with aging in the laboratory, or during Phoenix service. Correspondingly, the modulus increased with aging. Based on work done by Ahagon et al.,[8] the ultimate elongation and the modulus at 100 percent strain were studied in detail. A slope of log(modulus@ 100 percent) to log(ultimate elongation) near to -0.75 was found in their work to correspond to thermo-oxidative aging of rubber. Table 33 shows the approximate equivalent age of the tire skim-coat and wedge compounds, based on a comparison to tires in Phoenix service for at the most severe aging conditions on either roadwheel test or in the oven. There was no significant difference between the results for the two roadwheel tests. It also shows the slope of the Ahagon plot for each component.

[s] Only tested to 292 hours

Roadwheel aging tended to produce changes roughly equivalent to tires with 1 to 2 years of service in Phoenix. Oven aging for 8 weeks at 65ºC produced modulus and elongation values equivalent to tires with at least 4 to 6 years of service in Phoenix. The slope of the Ahagon plots were significantly different from 0.75, indicating that anaerobic aging took place in many tests, especially for the thicker area of the tire in the wedge. Measurements after capped-inflation aging showed significant depletion of the oxygen in the fill gas at the longest aging times. Consequently, replenishment of the oxygen by periodic vent and refill of the inflation gas was recommended for subsequent work.

Table 33. Approximate Equivalent Average Elongation and Modulus for Laboratory-Aged Tires and Tires in Phoenix Service

Tire Model	Test	Age of Approximately Equivalent Phoenix Service Tire, Years				Slope of Ahagon Plot	
		Skim-Coat		Wedge		Skim-Coat	Wedge
		100% Modulus	Elongation @ Break	100% Modulus	Elongation @ Break		
B	Roadwheel	1 to 2	1 to 2	1 to 2	1 to 2	- 0.53	- 0.99
	Oven, 8 wks @ 65ºC	4 to 6	4 to 6	> 6	4 to 6	- 0.80	- 0.84
C	Roadwheel	< 2	< 2	< 2	2	- 0.77	- 1.10
	Oven, 8 wks @ 65ºC	2	4 to 6	4 to 6	4 to 6	- 1.09	- 1.20
D	Roadwheel	2	2	2	> 2	- 1.11	- 1.26
	Oven, 8 wks @ 65ºC	> 2	>> 2	> 2	>> 2	- 1.65	- 0.93
E	Roadwheel	> 3	2 to 3	2	2 to 3	- 1.25	- 1.30
	Oven, 8 wks @ 65ºC	>> 3	> 3	3	> 3	- 1.52	- 1.74
H	Roadwheel	No significant change				Not Measured	
	Oven, 8 wks @ 65ºC	2	4	3 to 4	>> 4	- 0.84	- 2.12
L	Roadwheel	2	2	2	2	- 0.14	- 1.78
	Oven, 8 wks @ 65ºC	>> 5	>> 5	>> 5	> 5	- 0.47	- 1.53

Micro-DeMattia Flex Fatigue The crack growth rate measured by the Micro-DeMattia flex test showed a tendency to increase over time, whether in service in Phoenix or in laboratory aging. Tire types E and L showed the greatest tendency for increased crack growth rate in Phoenix service or during oven aging, while tire type D showed very little increase in crack growth rate for any aging condition. The roadwheel test produced smaller changes in crack growth rate than oven aging. However, the LTDE test tended to produce greater changes in the crack growth rate than the P-END test.

Peel Adhesion, Wire-Coat Skim Compound, and Wedge Compound The peel adhesion of the skim compound has been shown to consistently decrease as tires experienced service in Phoenix. On the roadwheel tests, all tire types also consistently decreased in adhesion over time. Only the type E tire showed any significant sensitivity to the type of roadwheel test, with the P-END test producing a greater adhesion loss per hour of testing than the LTDE test. In the oven testing the peel strength tended to be very sensitive to increases in temperature and consistently showed a reduction in peel adhesion with increased time of aging. The approximate equivalent average level of adhesion of the laboratory-aged tires compared to tires from Phoenix service is shown in Table 34.

Table 34. Approximate Equivalent Average Peel Adhesion Values for Laboratory-Aged Tires and Tires in Phoenix Service

Tire Model	Test	Age of Approximately Equivalent Phoenix Service Tire, years	
		Skim-Coat Compound	Wedge Compound
B	Roadwheel	1 to 2	2 to 4
	Oven, 8 wks @ 65ºC	6	> 4
C	Roadwheel	2 to 6	2 to 6
	Oven, 8 wks @ 65ºC	2 to 6	2 to 6
D	Roadwheel	2	> 2
	Oven, 8 wks @ 65ºC	> 2	>> 2
E	Roadwheel	2 to 3	3
	Oven, 8 wks @ 65ºC	> 3	> 3
H	Roadwheel	1 to 2	2 to 4
	Oven, 8 wks @ 65ºC	2 to 4	3 to 5
L	Roadwheel	2 to 4	2 to 4
	Oven, 8 wks @ 65ºC	> 4	> 4

Two-Ply Laminate Fatigue Test Three of the five models collected from service in Phoenix showed a relatively strong correlation of the Cycles to Failure at 500 N-mm to both mileage and age, with a general reduction in the cycles to failure with increasing age and mileage. Excessive scatter resulted in no discernible correlation for the other two tire models. New versions of the six tire models collected in Phoenix were subjected to the two-ply analysis following laboratory roadwheel tests. Five of the six tire models showed a relatively strong correlation to time on the roadwheel test to reduced Cycles to Failure at 500 N-mm. For these five tire models, the duration of the test was statistically significant and yielded very similar results regardless of the roadwheel test method used. In sharp contrast, the type B tire model was almost completely insensitive to durations of up to 508 hours of the LTDE test and 240 hours of P-END test.

Shearography Tires in Phoenix service tended to show increases in separation, particularly for the type E tires. Although the roadwheel tests did generate increased area of separation, particularly in the belt edge area of the tires, there was no statistically significant difference between the roadwheel tests or tire models.

Fixed Oxygen Level The level of fixed oxygen in the skim-coat compound, the wedge compound, and the innermost tread compound increased during service in Phoenix, consistent with the anticipated mechanism of aerobic oxidation of these compounds during normal service. The roadwheel tests produced little or no significant change in the fixed oxygen level of the compounds for any tire types. Although there is a great deal of scatter in the data as evidenced by the low R^2 values, the oxygen content of the same compounds all increased at rates from $6x10^{-4}$ percent to $43x10^{-4}$ (average of $18x10^{-4} \pm 5x10^{-4}$) percent per week during oven aging. The oxygen content of the fill gas of the tires was measured at the beginning of the oven-aging period and at the end of 3 and 6 weeks of aging at 60°C or 70°C. The oxygen content of the fill gas decreased from an average of 45% during the initial fill to approximately 35% after 6 weeks of oven aging. Based on the regression, both conditions will have depleted the oxygen to normal atmospheric oxygen content of 21% by 9 weeks. This may have contributed to the evidence of anaerobic aging noted in the Ahagon plots and minimal levels of increased fixed oxygen in the wedge compound. Based on these data, replenishment of the 50/50 nitrogen/oxygen fill gas was recommended for the next phase of test development.

Stepped-Up Load After Oven Aging The stepped-up load test was intended to compare the structural integrity of the tire after aging to that of a new tire and many 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. General conclusions are:

- Aging tires for 3 weeks does not cause a significant decrease in structural integrity compared to a new tire of the same model.

- Tires aged for 70°C for 3 weeks tended to have longer running times than tires aged at 60°C for 3 weeks.

- The 24-hour break-in prior to oven aging tended to decrease the running time in the test after aging. If the 24 hours are added to the test time, the total running time tends to be longer than the tires without break-in. Note that since the break-in was done at 100 percent of maximum load the only direct comparisons possible are for tires that failed at less than 34 hours on the test after aging (tire models D, E and H aged 8 weeks at 65°C).

- Tire models C and L had no failures from roadwheel testing at loads below 100 percent of maximum load. Both of these models also had predicted failure times above 100 percent load even after 6 years of service in Phoenix.

- Tire models B and D showed failures below 100 percent load at aging times of 8 weeks at 65°C and predicted failure times below 100 percent load after 5 or more years of service in Phoenix.

- Roadwheel testing results for tire models E and H appear to be sensitive to oven aging times. They also appear to be the most sensitive to service time 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 the maximum rated load for the tires. 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 the maximum rated load for the tires.

Appendix 1. Test Descriptions

Microscopy
Replicates: Four. Sample Regions: Radial cross sections at 0, 90, 180, and 270 degrees. 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 **Data Units:** mm
Tire Air Permeability per ASTM F 1112-00, 21°C (70°F)
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

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

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

Peel Strength per ASTM D 413-98(2002)∈1 23°C (73.4°F)

Replicates: Four repeated measurements per tire sample region

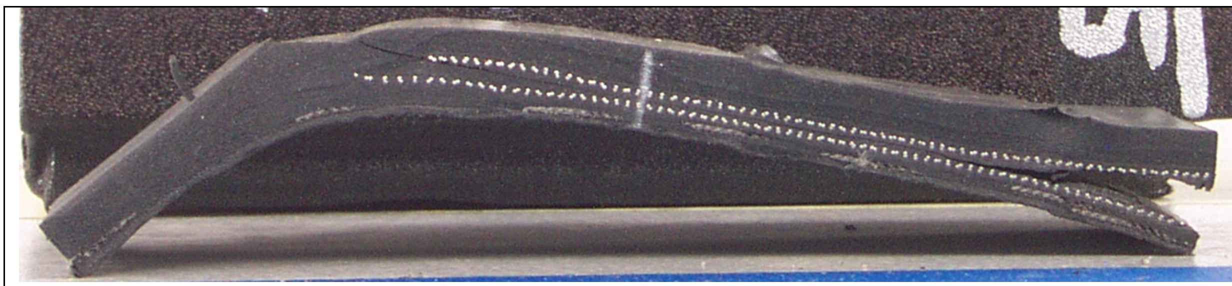
Sample Regions: Skim and wedge rubber.

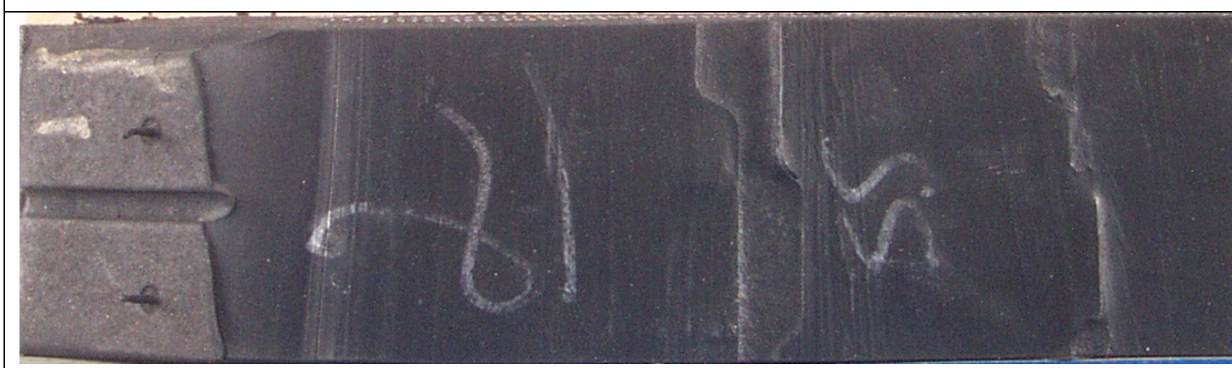
Specifications: General - Section tire into appropriate test specimens. Measure peel strength of the tire skim rubber between the steel belts as well as the wedge rubber (belt edge) per ASTM D 413-98(2002)$^{\epsilon 1}$ *Standard Test Methods for Rubber Property—Adhesion to Flexible Substrate*.

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 1st (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, %

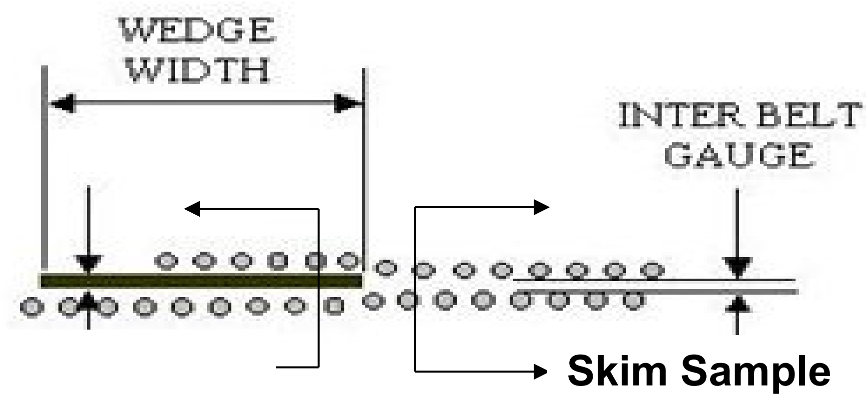
Total Crosslink Density

Replicates: Five repeated measurements in each sample region.

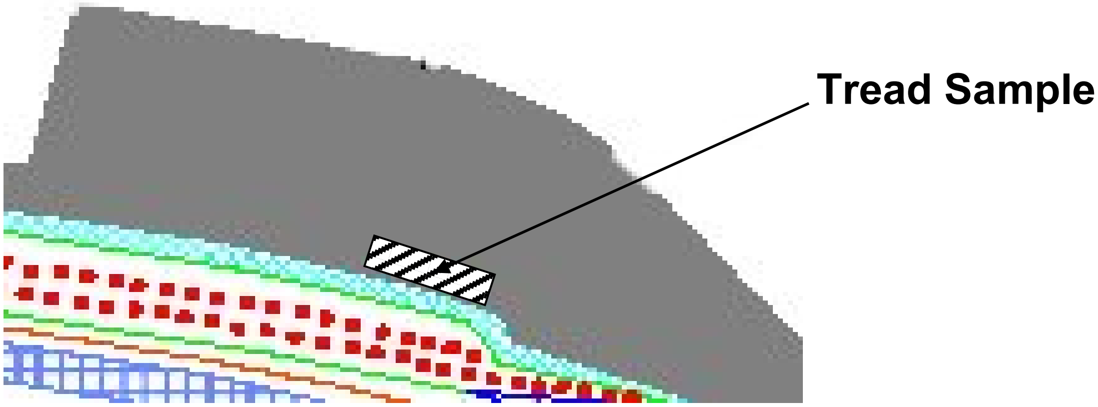
Sample Regions: Skim Rubber 0° SS, Skim Rubber 180° OSS, Wedge Rubber 0° SS, Wedge Rubber 180° OSS, Tread Rubber 0° SS, Tread Rubber 180° OSS.

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.



- 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, undertread, 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

Crosslink Density Distribution (S1-S8)

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 ***Total Crosslink Density*** 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-sulphidics based on the numbers obtained from 4.1 through 4.3

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.

Data Units: mol/cc^3, %

Fixed Oxygen by Weight

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 ***Total Crosslink Density*** 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

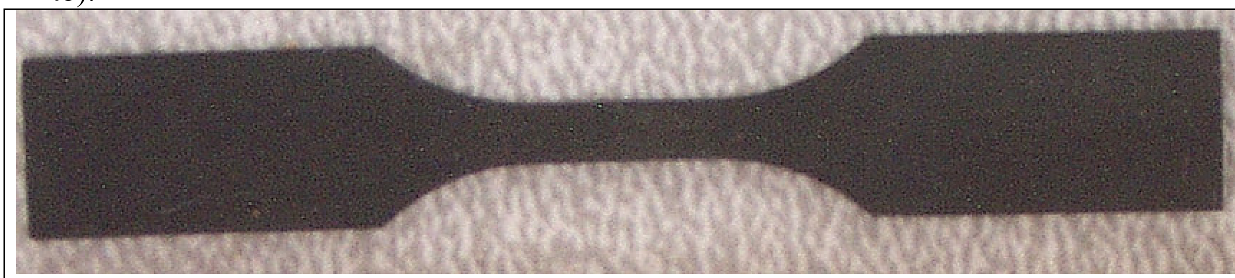
Tensile Properties per ASTM D 412-98a(2002)∈1

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 and 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 ***Total Crosslink Density*** 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, °C, %

Shore A Hardness per ASTM D2240-02b

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 ***Total Crosslink Density*** sample descriptions. Measure inner liner 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 (Five 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 #.

Indentation Modulus Profiling

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 utilized 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 be 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 an MS Excel format.

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

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:

- 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.

Oven Aging, Contractor's Ovens

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.
- 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.

Analysis of Innerliner Compound by FTIR

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.

Inflation Gas Oxygen Concentration

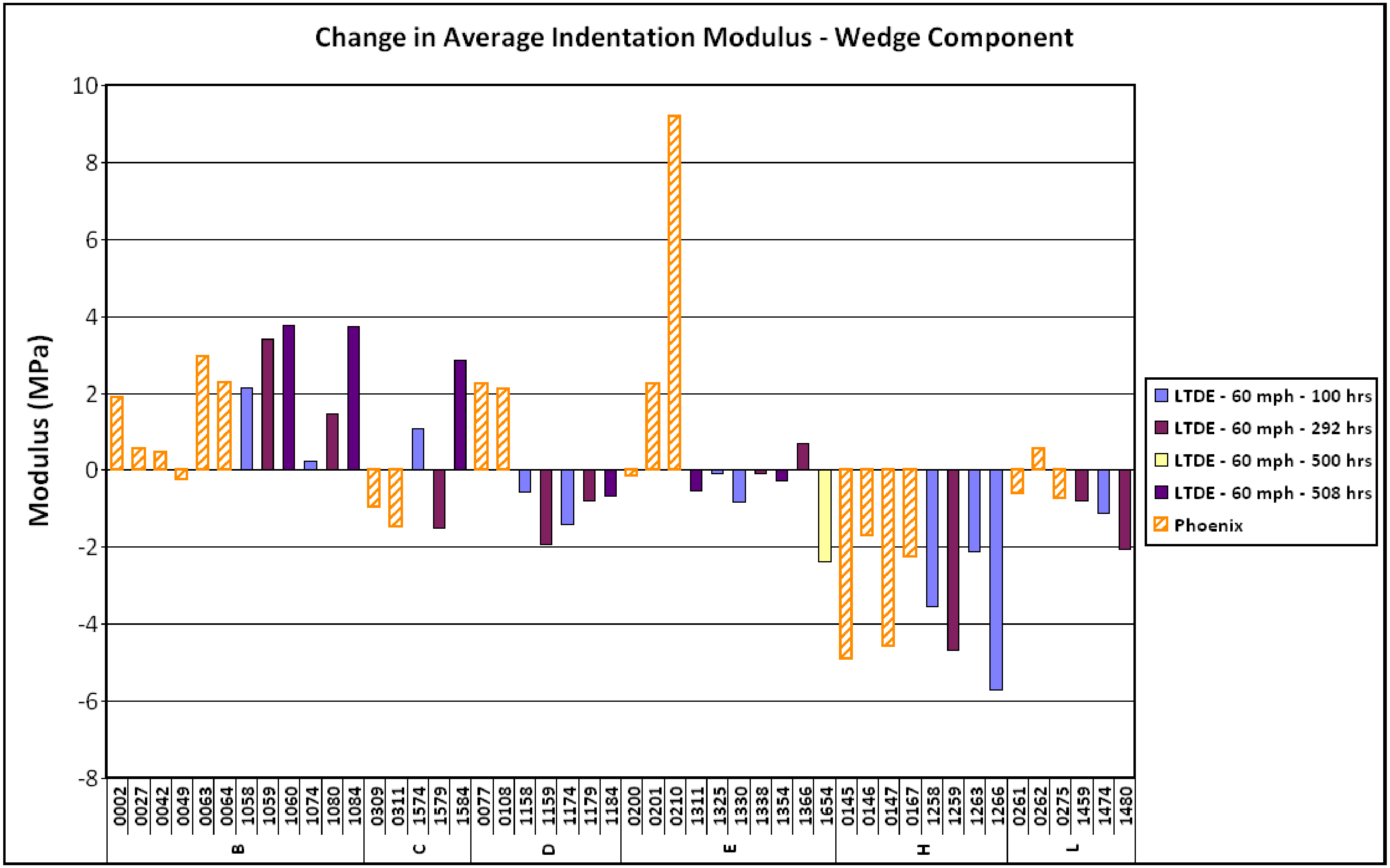
Replicates: One measurement per tire.

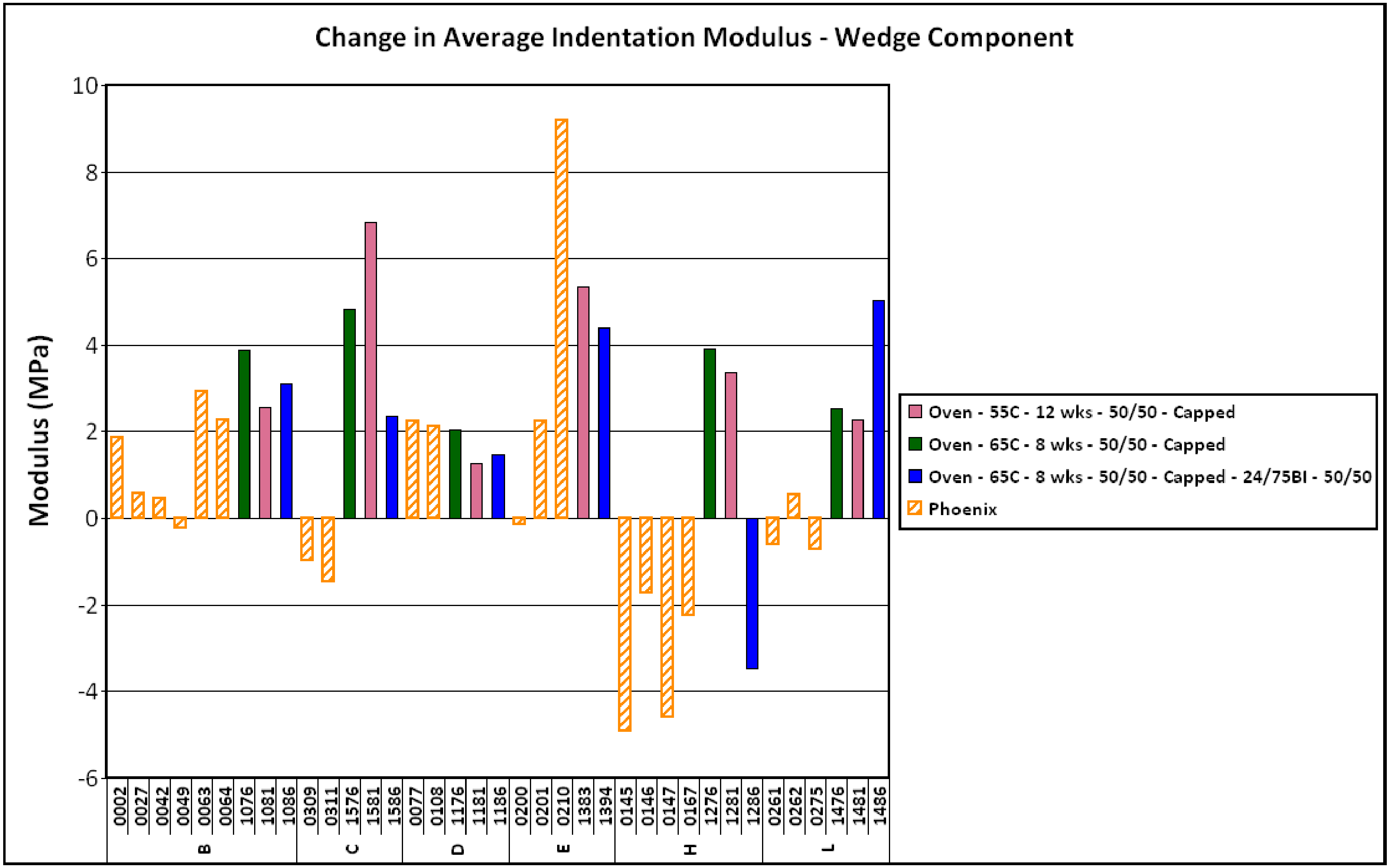
Sample Regions: Inflation gas

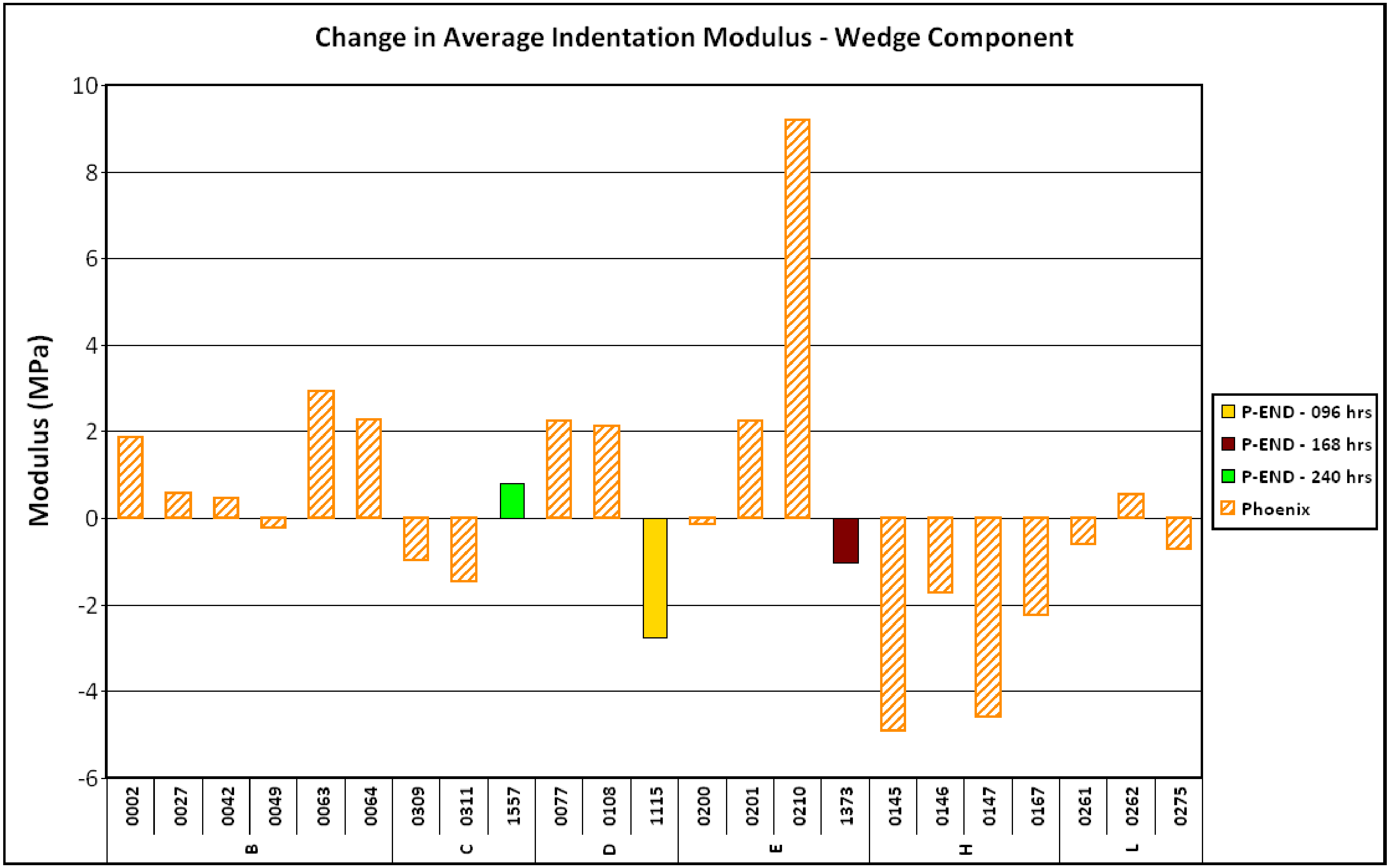
Specifications: Non-dynamic, pre- or post-test; measure the percent oxygen concentration in the tire's inflation gas mixture with a gas analyzer accurate to within ±0.1%.

Data Units: %

Appendix 2. Indentation Modulus Profiles for the Wedge Compounds

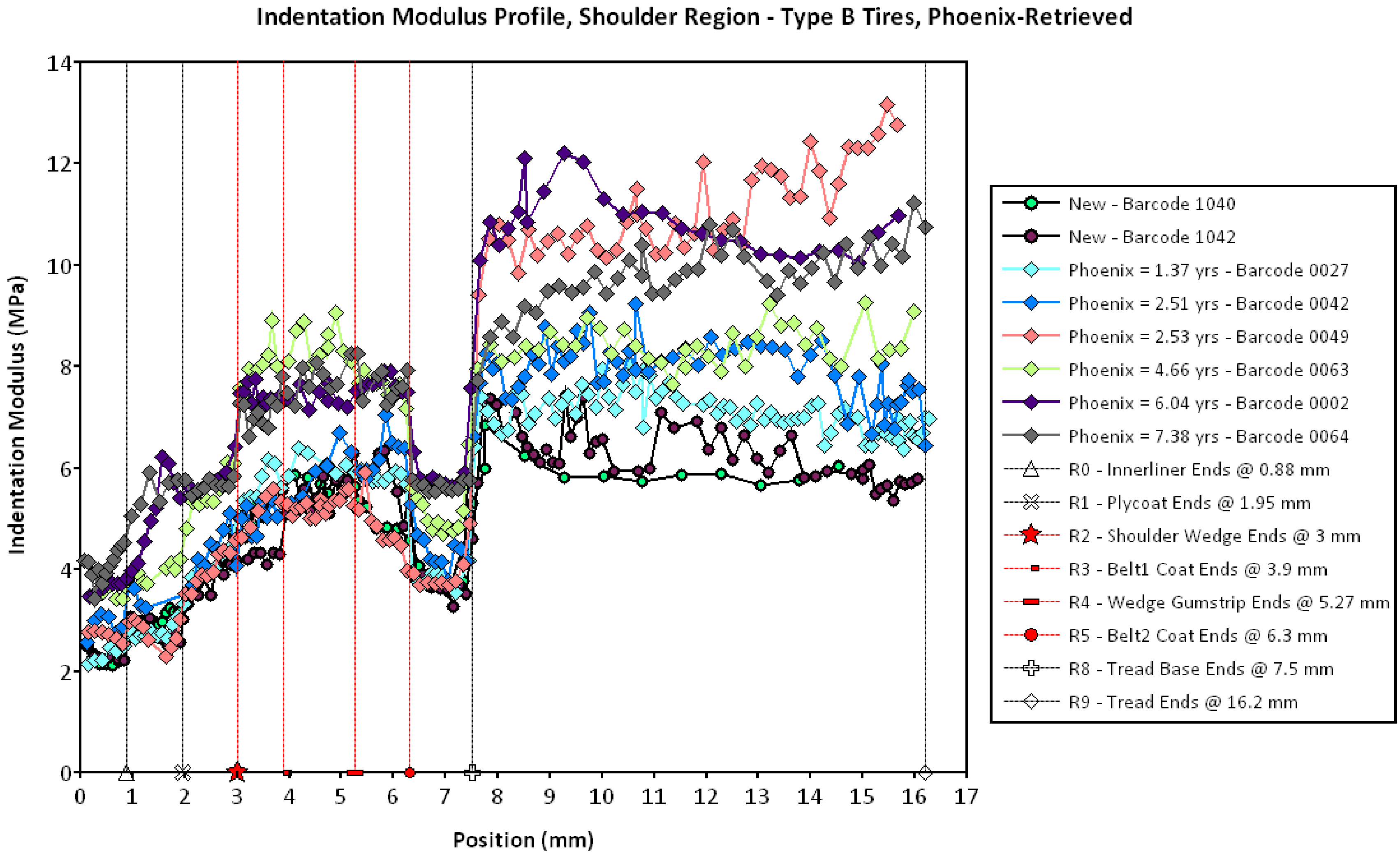



Change in Average Indentation Modulus - Wedge Component
Modulus (MPa)
10
8
6
4
2
0
-2
-4
-6
0002
0027
0042
0049
0063
0064
1076
1081
1086
0309
0311
1576
1581
1586
0077
0108
1176
1181
1186
0200
0201
0210
1383
1394
0145
0146
0147
0167
1276
1281
1286
0261
0262
0275
1476
1481
1486
B
C
D
E
H
L
Oven - 55C - 12 wks - 50/50 - Capped
Oven - 65C - 8 wks - 50/50 - Capped
Oven - 65C - 8 wks - 50/50 - Capped - 24/75BI - 50/50
Phoenix

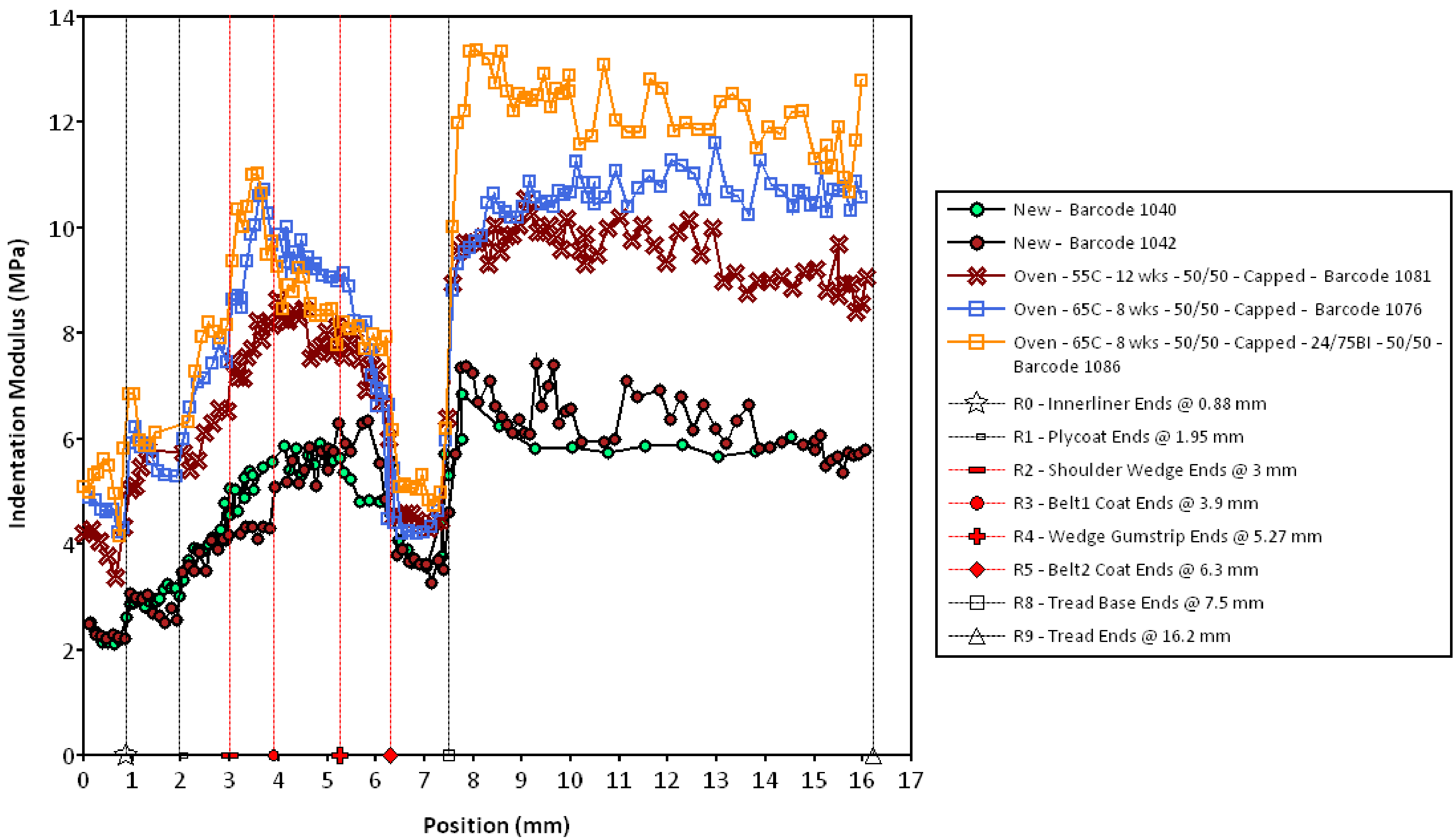


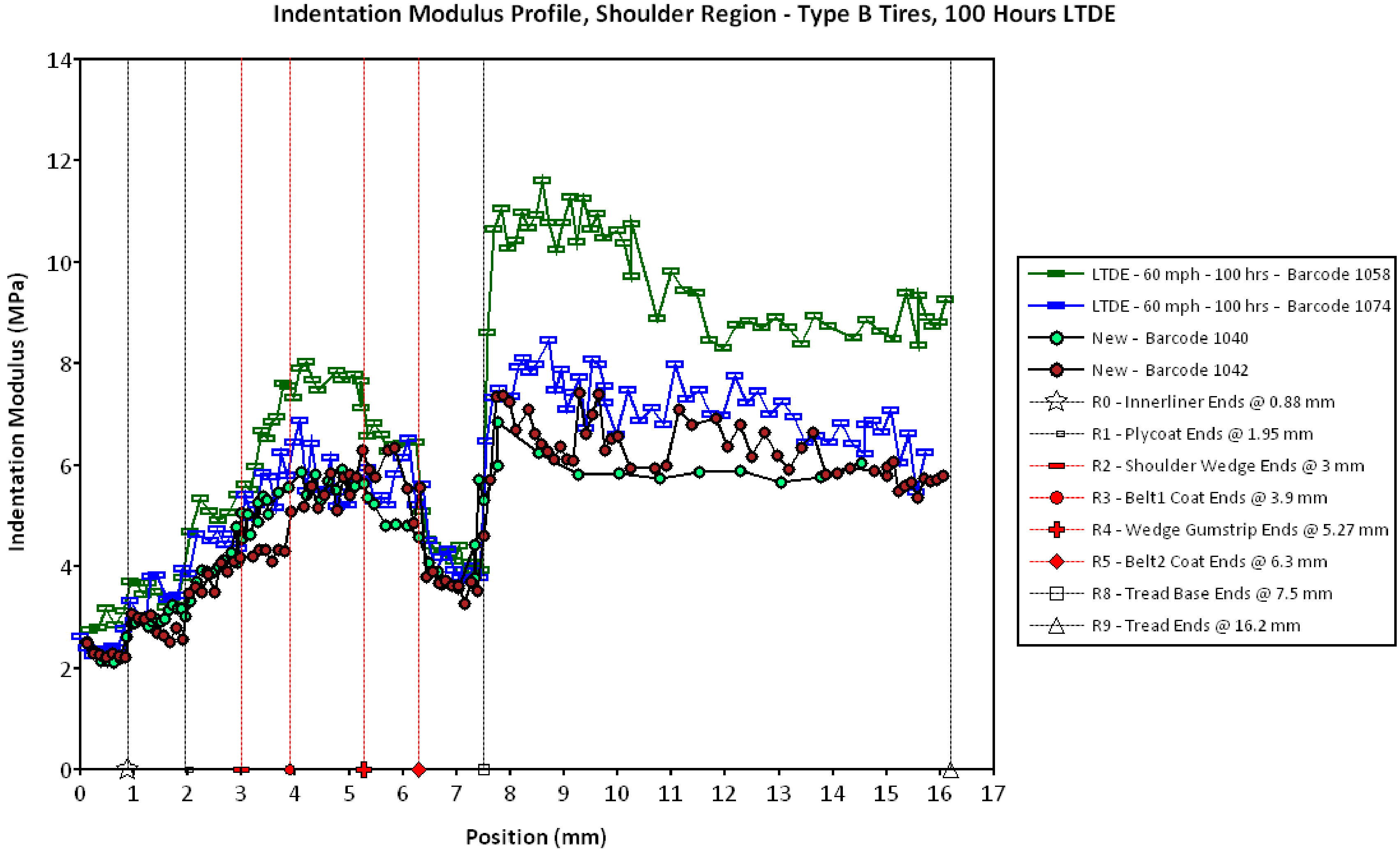
Change in Average Indentation Modulus - Wedge Component
Modulus (MPa)
10
8
6
4
2
0
-2
-4
-6
0002
0027
0042
0049
0063
0064
0309
0311
1557
0077
0108
1115
0200
0201
0210
1373
0145
0146
0147
0167
0261
0262
0275
B
C
D
E
H
L
P-END - 096 hrs
P-END - 168 hrs
P-END - 240 hrs
Phoenix

Appendix 3. Indentation Modulus Profiles of Tires by Type

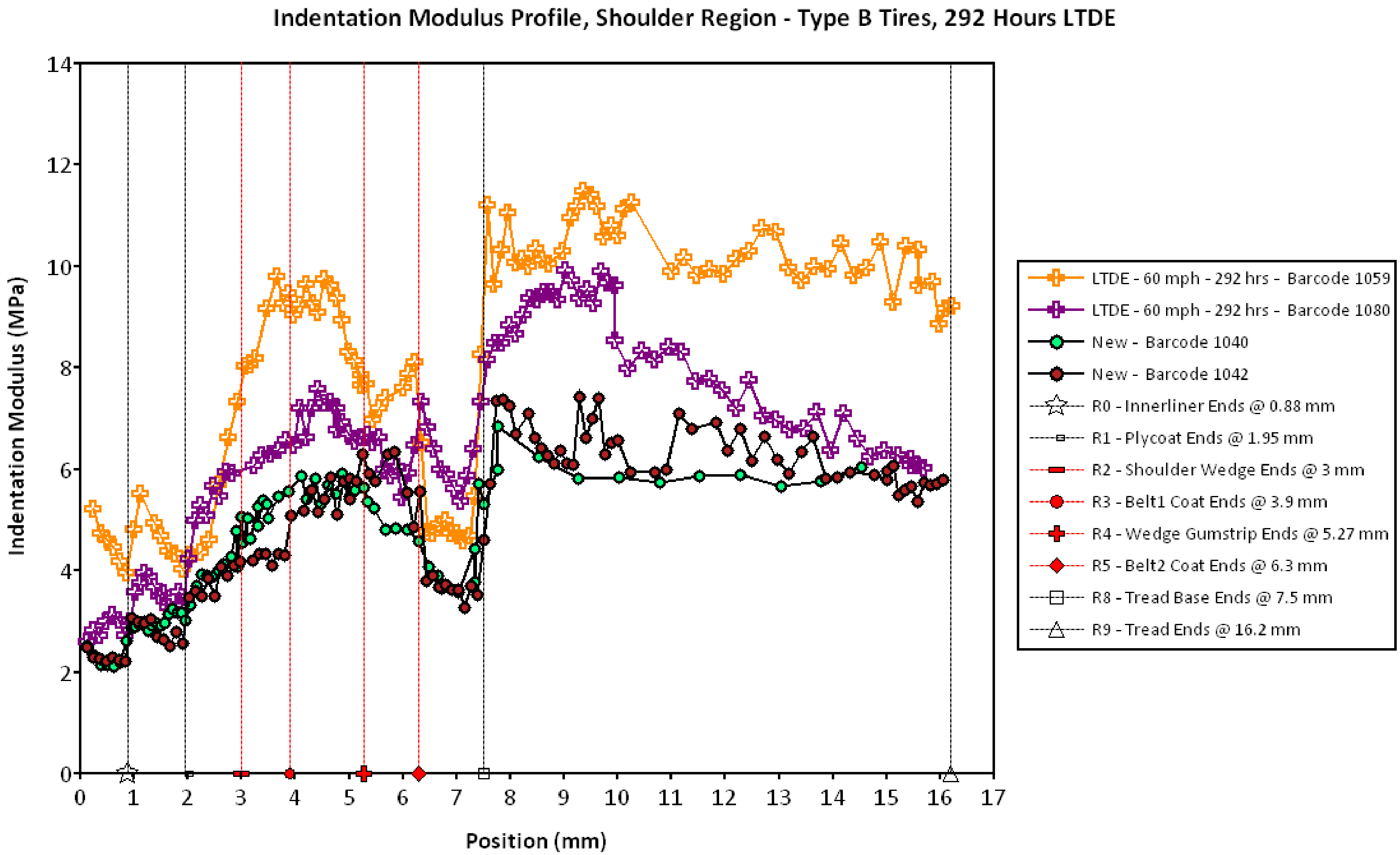


Indentation Modulus Profile, Shoulder Region - Type B Tires, Oven Aged

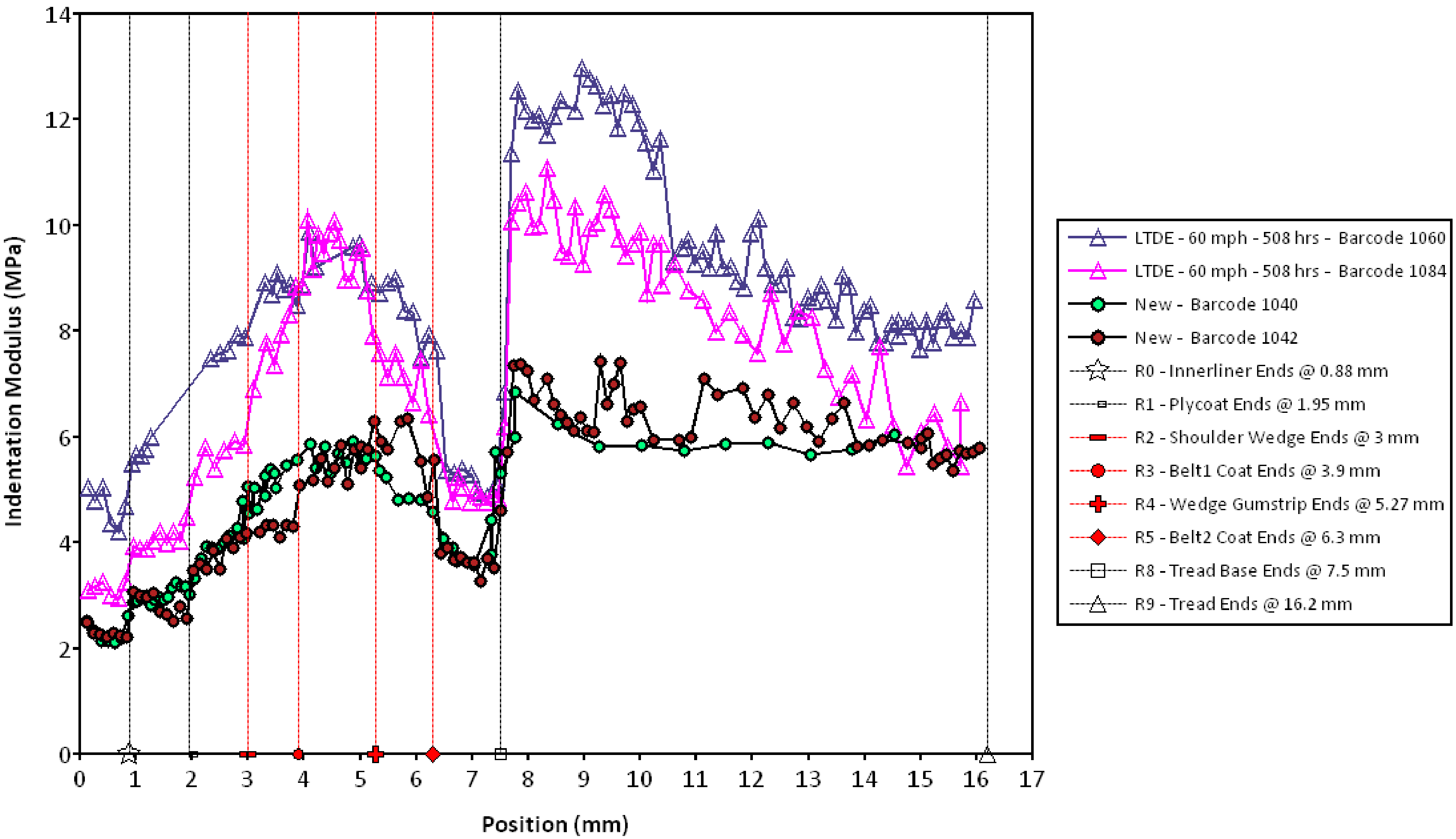


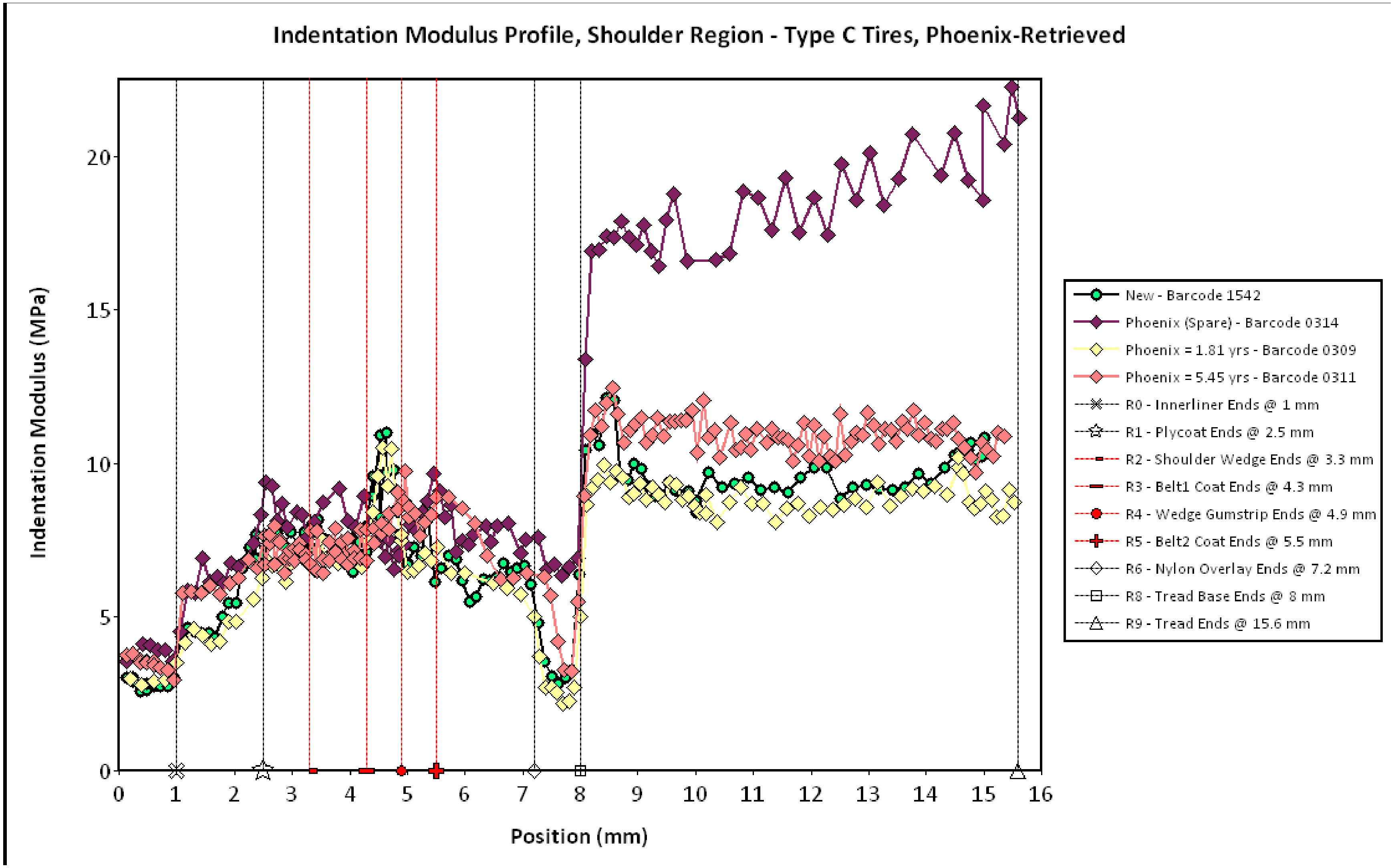


Indentation Modulus Profile, Shoulder Region - Type B Tires, 100 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
LTDE - 60 mph - 100 hrs - Barcode 1058
LTDE - 60 mph - 100 hrs - Barcode 1074
New - Barcode 1040
New - Barcode 1042
R0 - Innerliner Ends @ 0.88 mm
R1 - Plycoat Ends @ 1.95 mm
R2 - Shoulder Wedge Ends @ 3 mm
R3 - Belt1 Coat Ends @ 3.9 mm
R4 - Wedge Gumstrip Ends @ 5.27 mm
R5 - Belt2 Coat Ends @ 6.3 mm
R8 - Tread Base Ends @ 7.5 mm
R9 - Tread Ends @ 16.2 mm

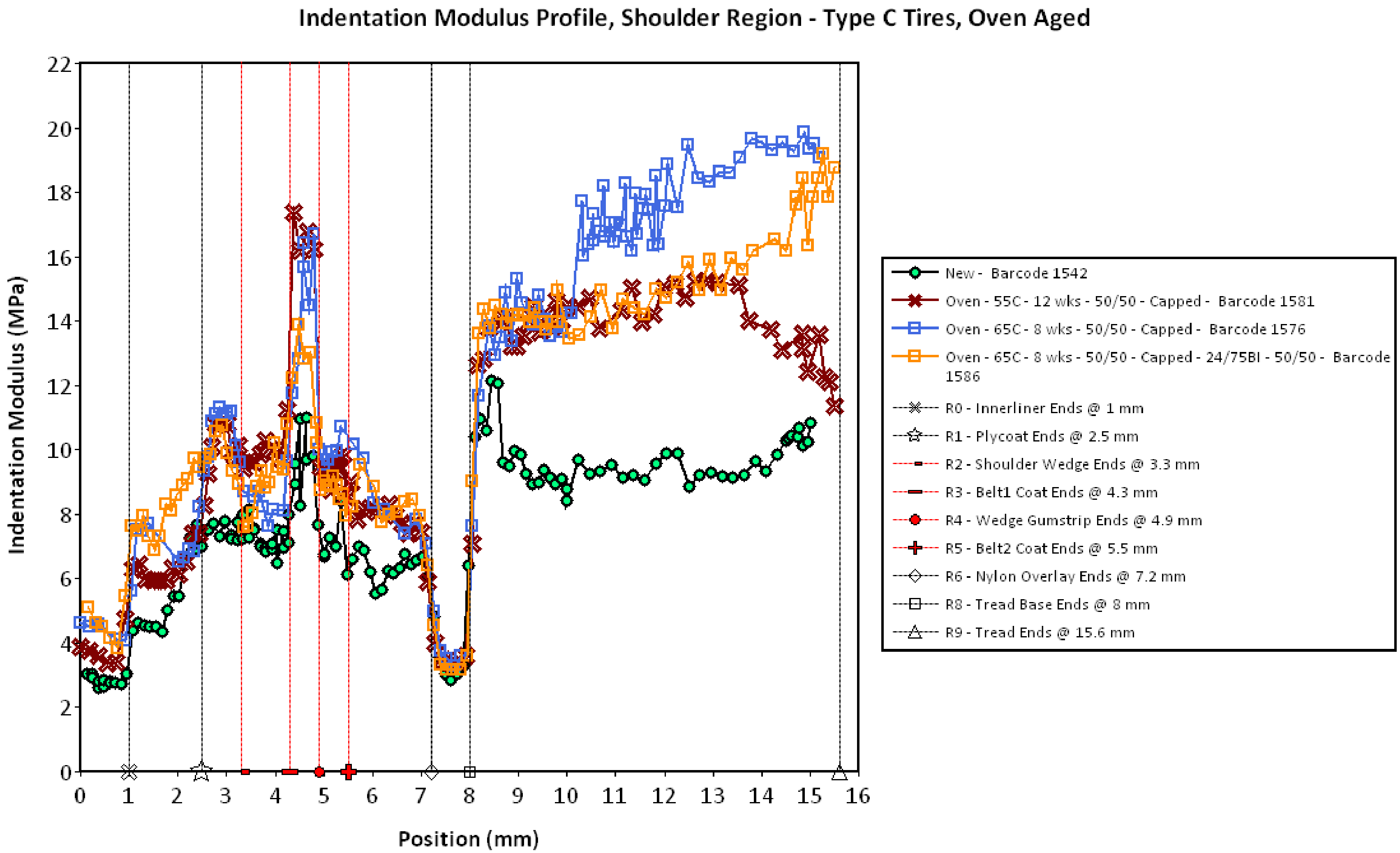

Indentation Modulus Profile, Shoulder Region - Type B Tires, 292 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
LTDE - 60 mph - 292 hrs - Barcode 1059
LTDE - 60 mph - 292 hrs - Barcode 1080
New - Barcode 1040
New - Barcode 1042
R0 - Innerliner Ends @ 0.88 mm
R1 - Plycoat Ends @ 1.95 mm
R2 - Shoulder Wedge Ends @ 3 mm
R3 - Belt1 Coat Ends @ 3.9 mm
R4 - Wedge Gumstrip Ends @ 5.27 mm
R5 - Belt2 Coat Ends @ 6.3 mm
R8 - Tread Base Ends @ 7.5 mm
R9 - Tread Ends @ 16.2 mm

Indentation Modulus Profile, Shoulder Region - Type B Tires, 508 Hours LTDE

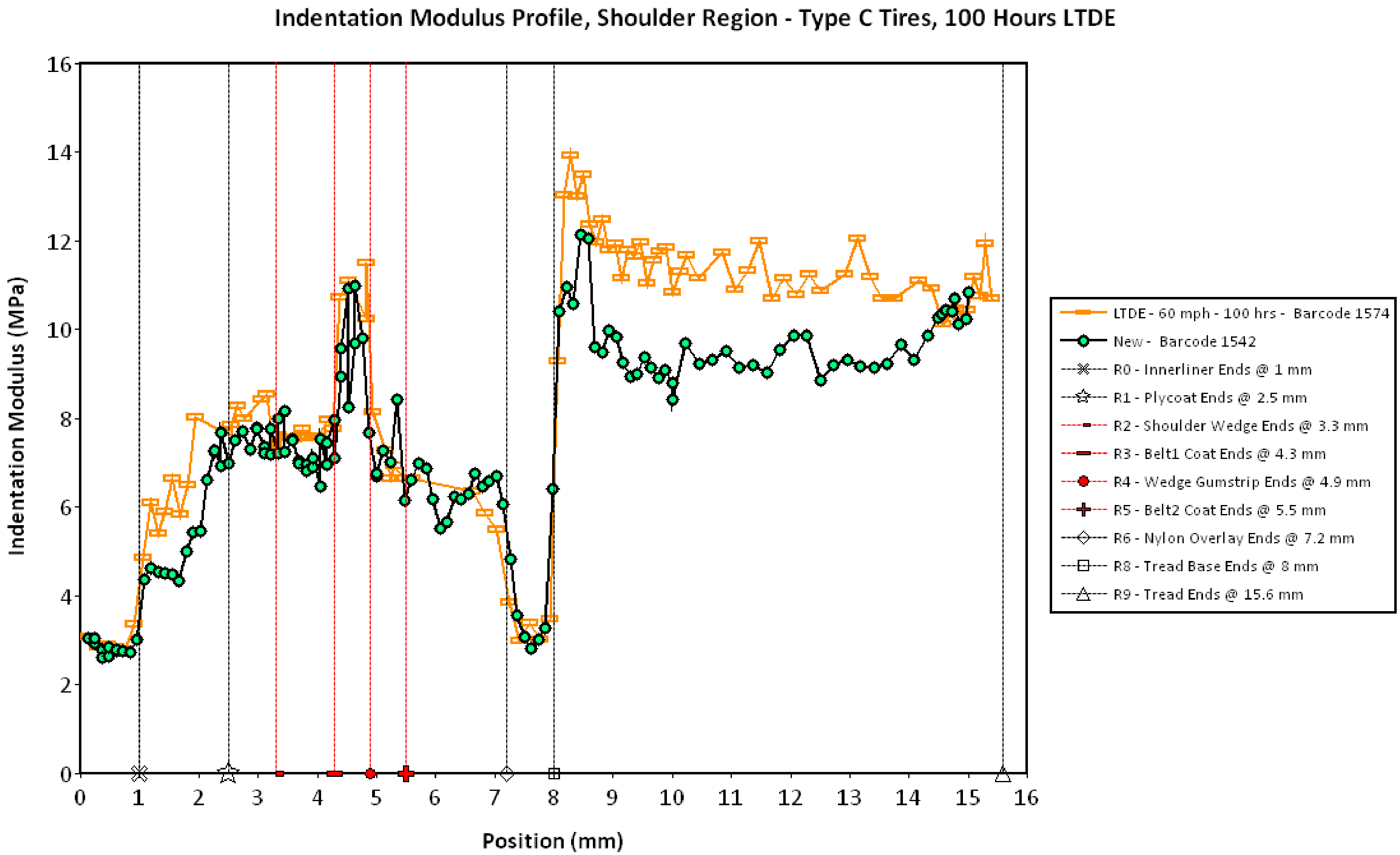




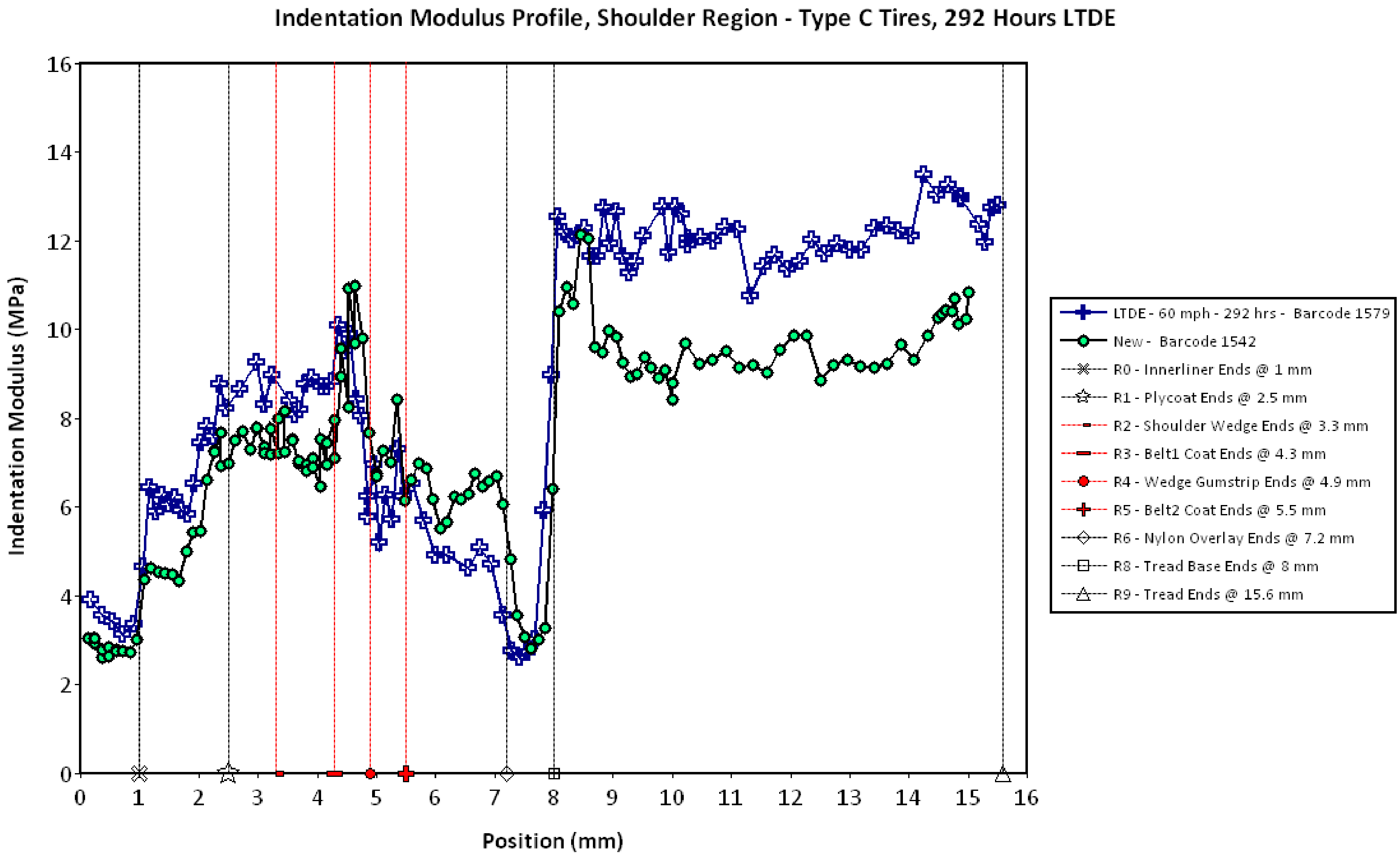
Indentation Modulus Profile, Shoulder Region - Type C Tires, Phoenix-Retrieved
Indentation Modulus (MPa)
Position (mm)
0
5
10
15
20
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
New - Barcode 1542
Phoenix (Spare) - Barcode 0314
Phoenix = 1.81 yrs - Barcode 0309
Phoenix = 5.45 yrs - Barcode 0311
R0 - Innerliner Ends @ 1 mm
R1 - Plycoat Ends @ 2.5 mm
R2 - Shoulder Wedge Ends @ 3.3 mm
R3 - Belt1 Coat Ends @ 4.3 mm
R4 - Wedge Gumstrip Ends @ 4.9 mm
R5 - Belt2 Coat Ends @ 5.5 mm
R6 - Nylon Overlay Ends @ 7.2 mm
R8 - Tread Base Ends @ 8 mm
R9 - Tread Ends @ 15.6 mm

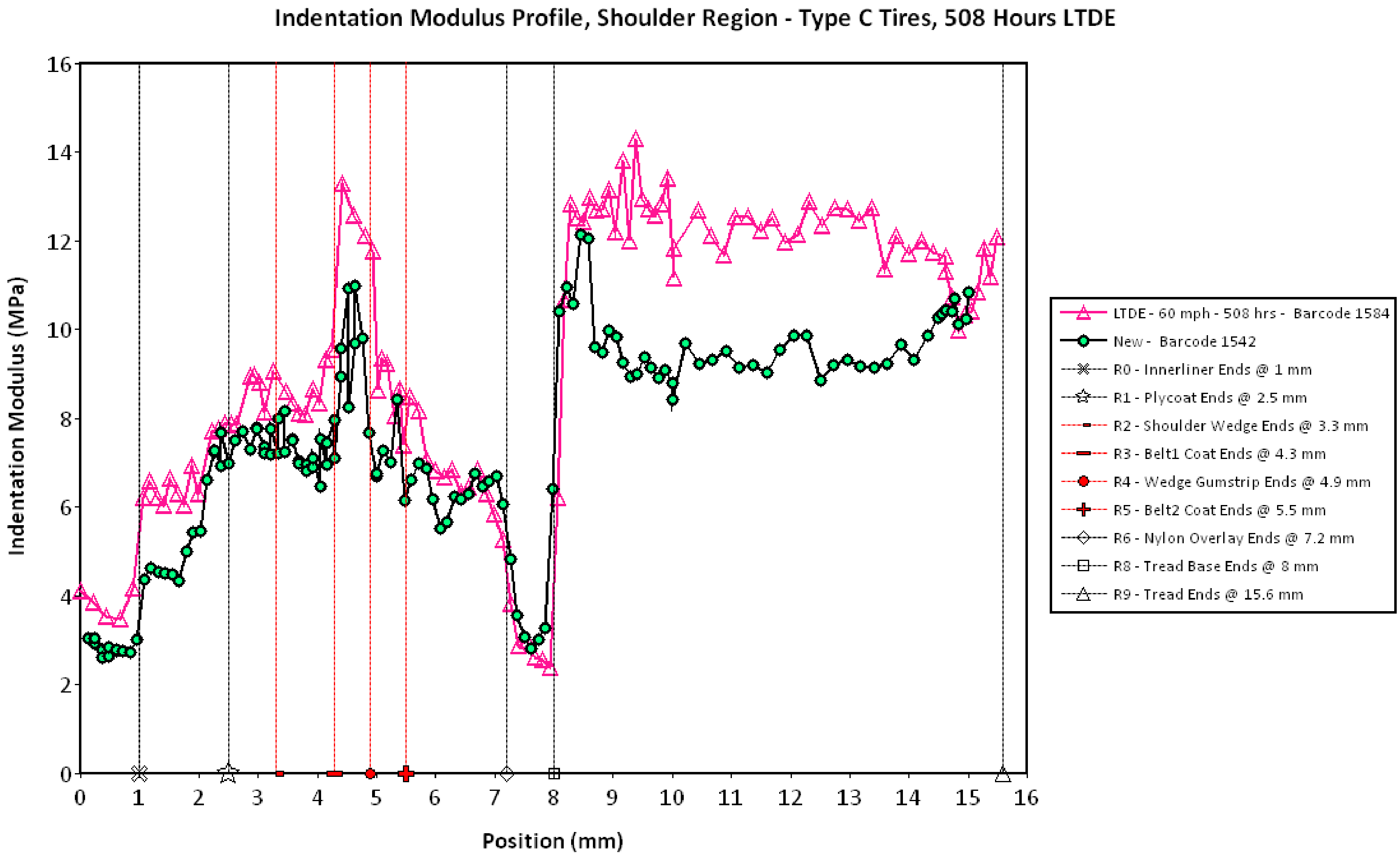


Note: Observe how the 24-hour break-in at 75 mph for tire 1586 produced less belt package hardening than 1576 at the same oven conditions with no break-in.

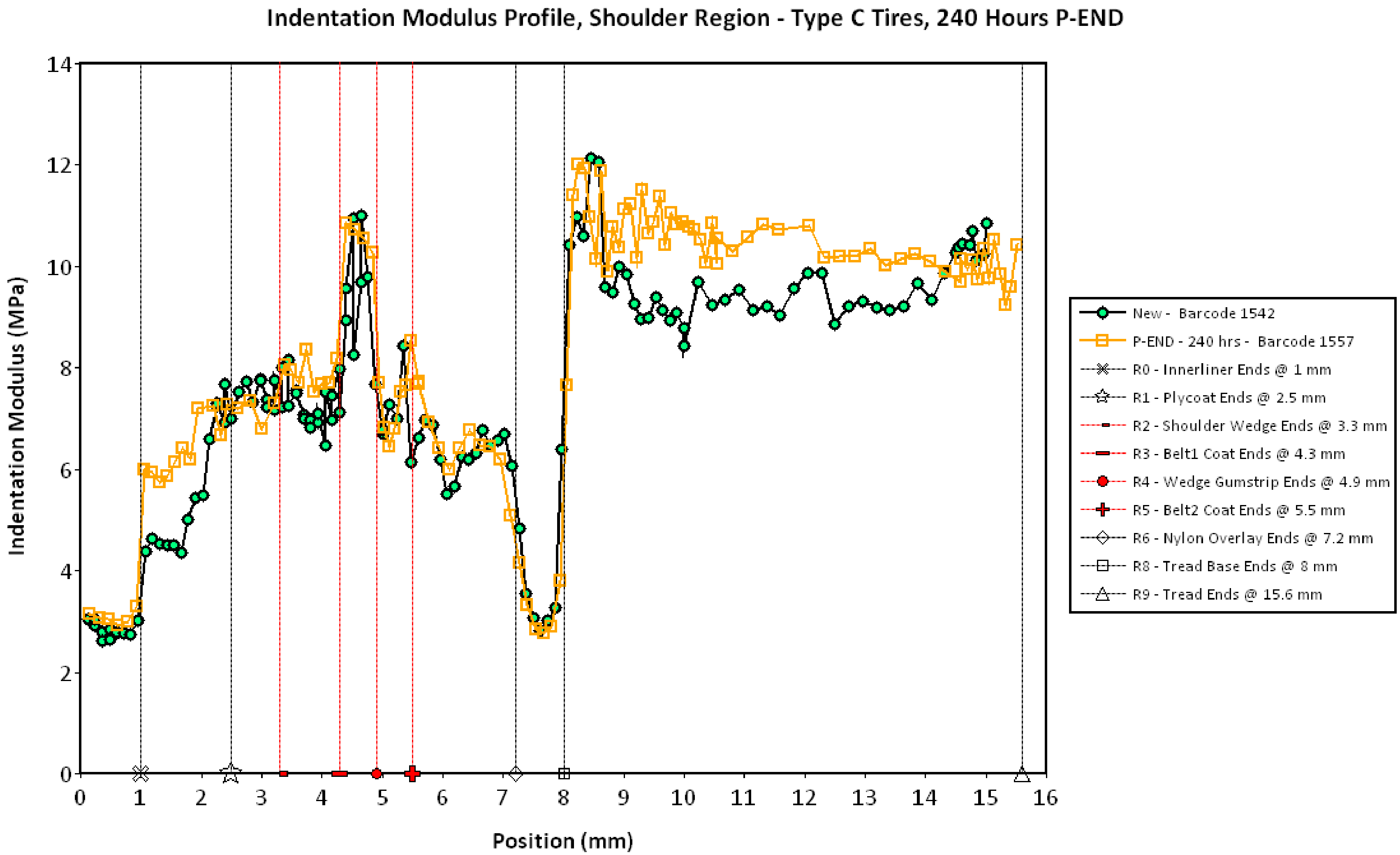


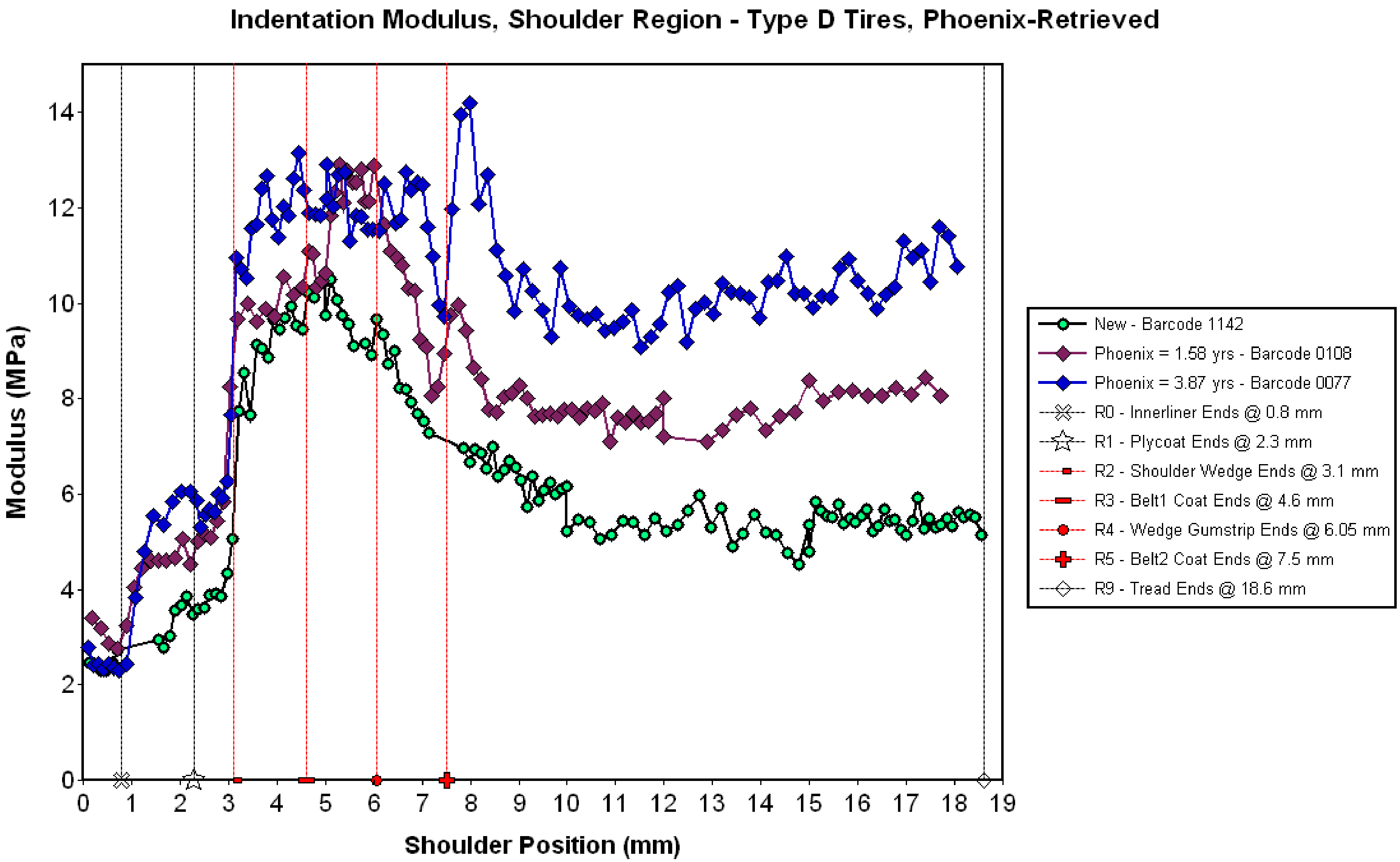
Indentation Modulus Profile, Shoulder Region - Type C Tires, 100 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
LTDE - 60 mph - 100 hrs - Barcode 1574
New - Barcode 1542
R0 - Innerliner Ends @ 1 mm
R1 - Plycoat Ends @ 2.5 mm
R2 - Shoulder Wedge Ends @ 3.3 mm
R3 - Belt1 Coat Ends @ 4.3 mm
R4 - Wedge Gumstrip Ends @ 4.9 mm
R5 - Belt2 Coat Ends @ 5.5 mm
R6 - Nylon Overlay Ends @ 7.2 mm
R8 - Tread Base Ends @ 8 mm
R9 - Tread Ends @ 15.6 mm

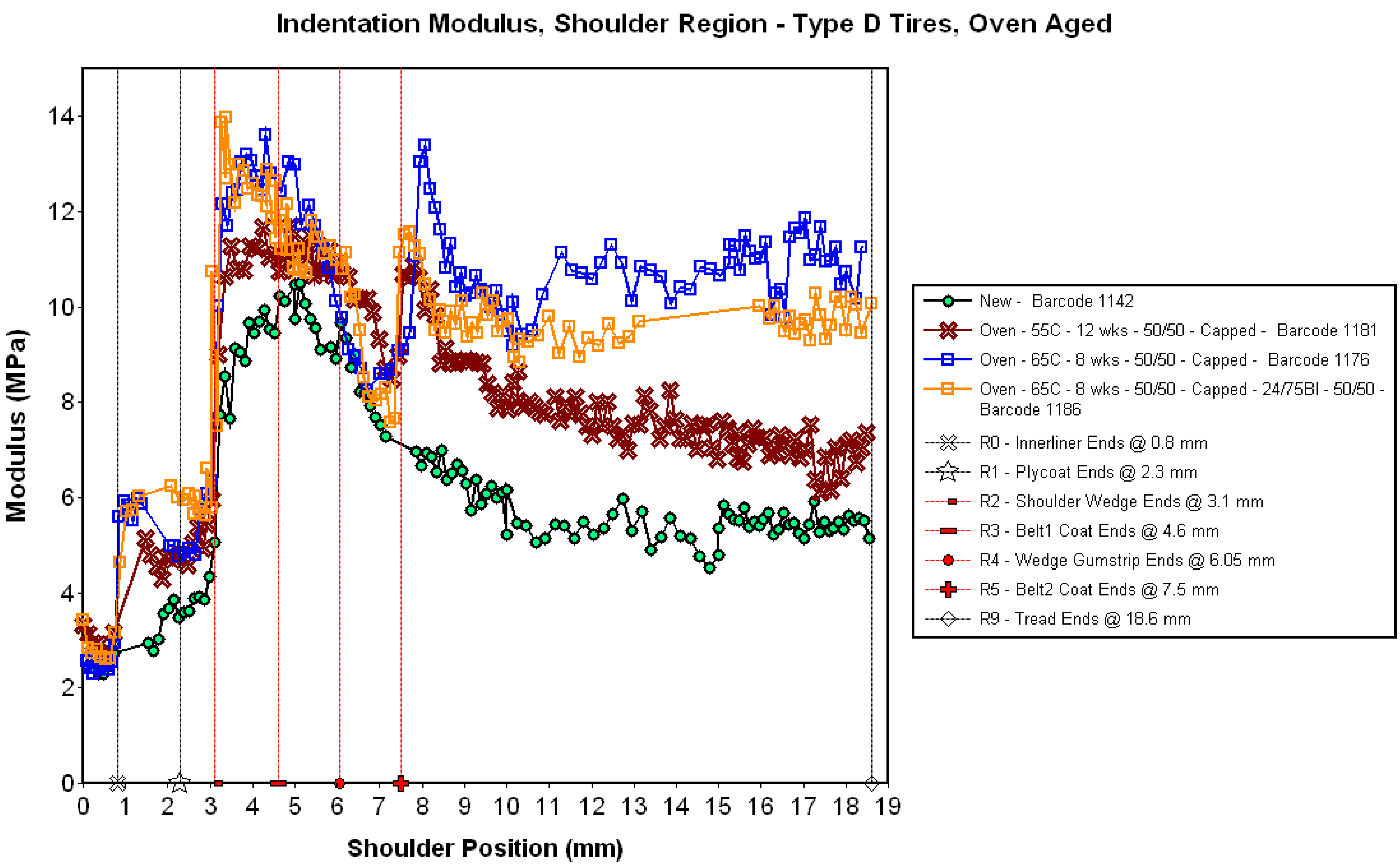

Indentation Modulus Profile, Shoulder Region - Type C Tires, 292 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
LTDE - 60 mph - 292 hrs - Barcode 1579
New - Barcode 1542
R0 - Innerliner Ends @ 1 mm
R1 - Plycoat Ends @ 2.5 mm
R2 - Shoulder Wedge Ends @ 3.3 mm
R3 - Belt1 Coat Ends @ 4.3 mm
R4 - Wedge Gumstrip Ends @ 4.9 mm
R5 - Belt2 Coat Ends @ 5.5 mm
R6 - Nylon Overlay Ends @ 7.2 mm
R8 - Tread Base Ends @ 8 mm
R9 - Tread Ends @ 15.6 mm

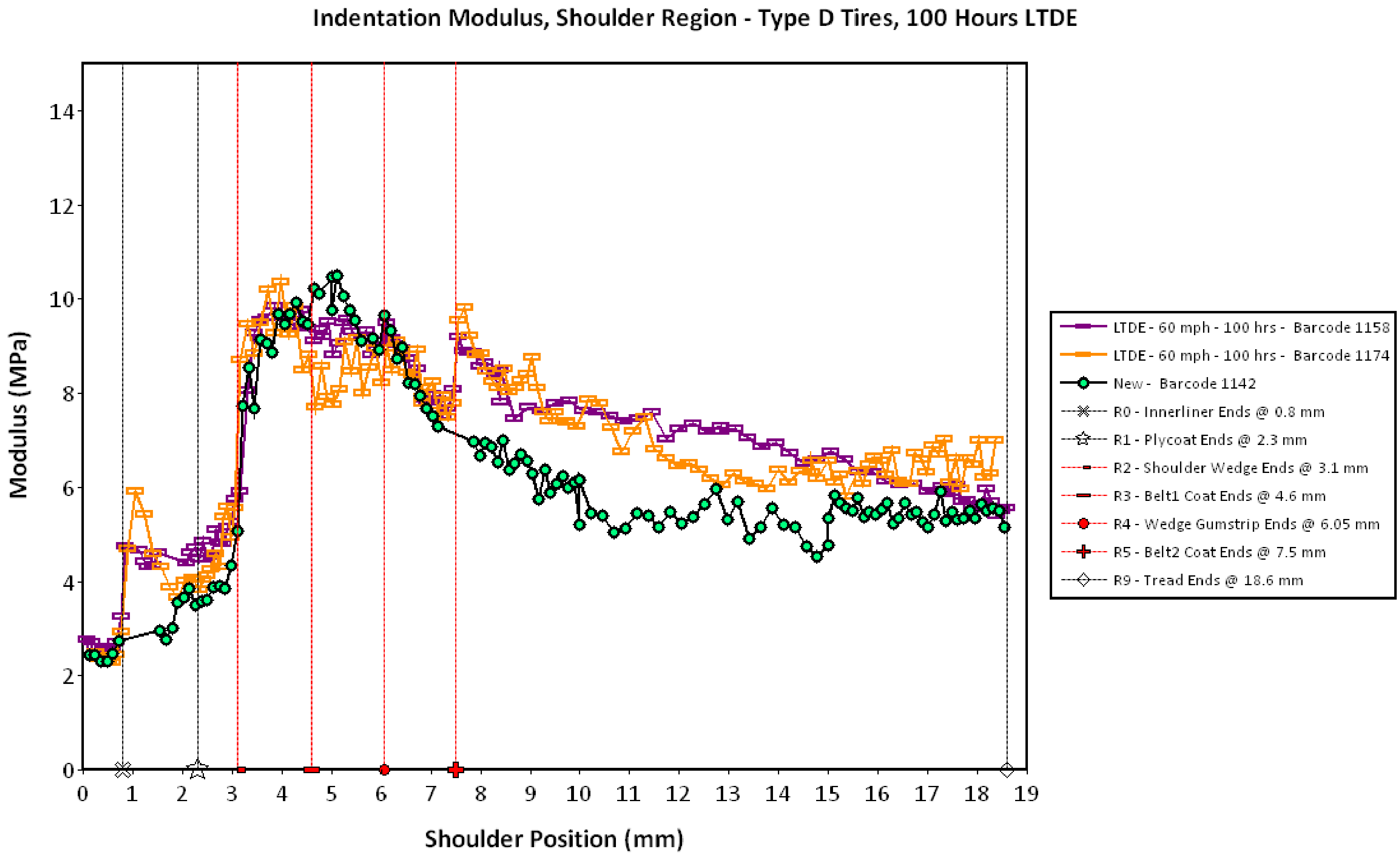


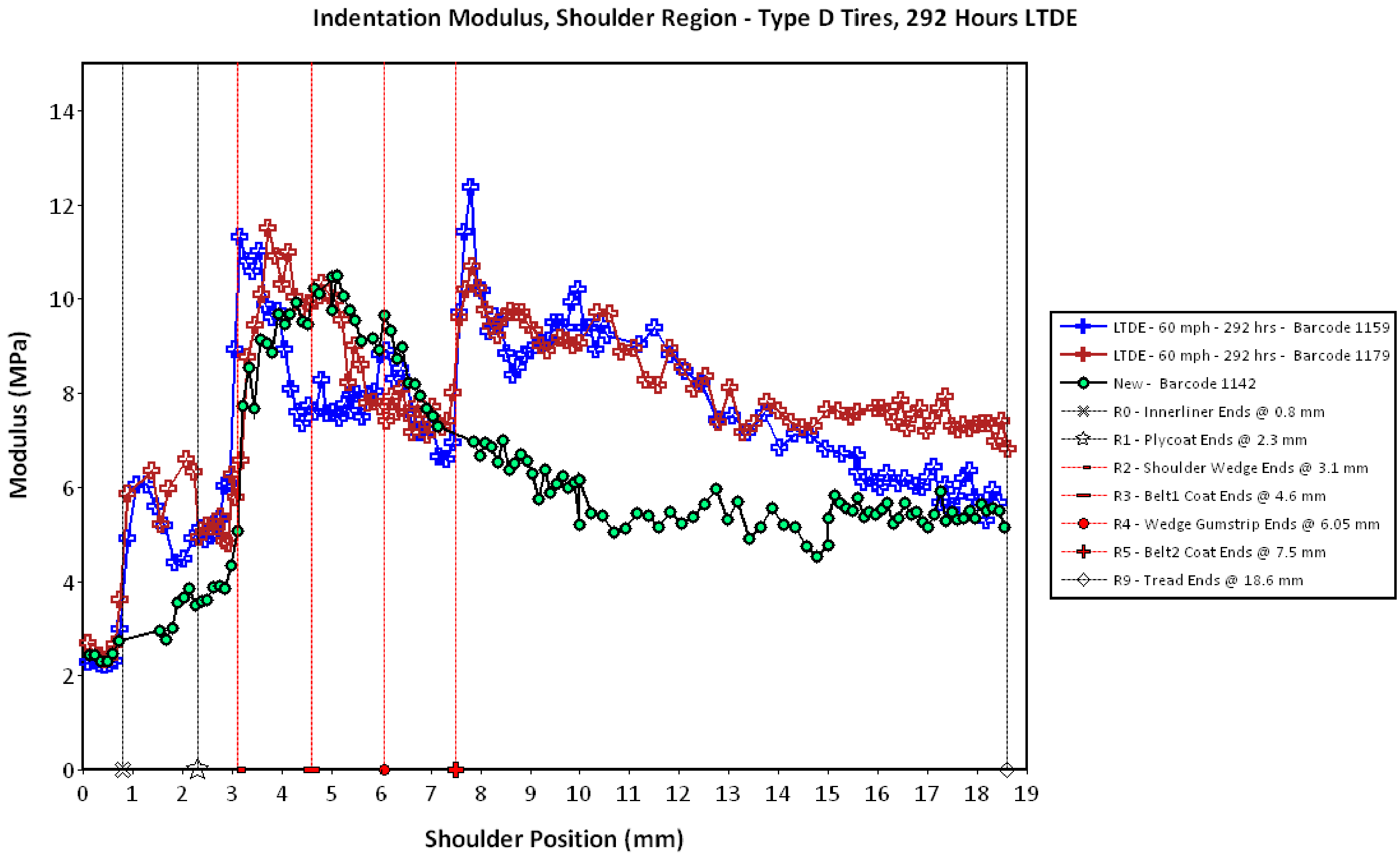
Indentation Modulus Profile, Shoulder Region - Type C Tires, 508 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
LTDE - 60 mph - 508 hrs - Barcode 1584
New - Barcode 1542
R0 - Innerliner Ends @ 1 mm
R1 - Plycoat Ends @ 2.5 mm
R2 - Shoulder Wedge Ends @ 3.3 mm
R3 - Belt1 Coat Ends @ 4.3 mm
R4 - Wedge Gumstrip Ends @ 4.9 mm
R5 - Belt2 Coat Ends @ 5.5 mm
R6 - Nylon Overlay Ends @ 7.2 mm
R8 - Tread Base Ends @ 8 mm
R9 - Tread Ends @ 15.6 mm


Indentation Modulus Profile, Shoulder Region - Type C Tires, 240 Hours P-END
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
New - Barcode 1542
P-END - 240 hrs - Barcode 1557
R0 - Innerliner Ends @ 1 mm
R1 - Plycoat Ends @ 2.5 mm
R2 - Shoulder Wedge Ends @ 3.3 mm
R3 - Belt1 Coat Ends @ 4.3 mm
R4 - Wedge Gumstrip Ends @ 4.9 mm
R5 - Belt2 Coat Ends @ 5.5 mm
R6 - Nylon Overlay Ends @ 7.2 mm
R8 - Tread Base Ends @ 8 mm
R9 - Tread Ends @ 15.6 mm

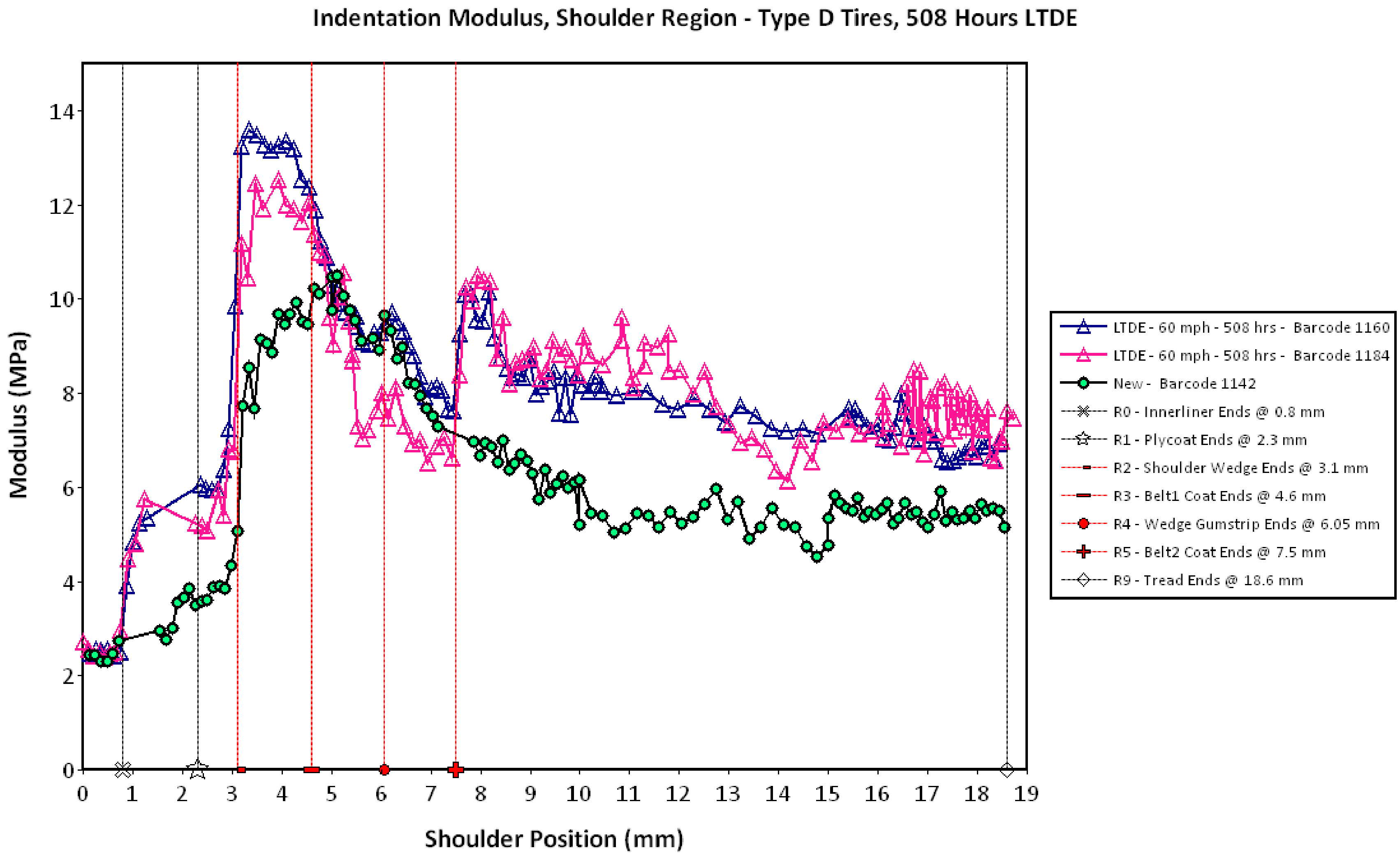

Indentation Modulus, Shoulder Region - Type D Tires, Phoenix-Retrieved
Modulus (MPa)
0
2
4
6
8
10
12
14
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19
Shoulder Position (mm)
New - Barcode 1142
Phoenix = 1.58 yrs - Barcode 0108
Phoenix = 3.87 yrs - Barcode 0077
R0 - Innerliner Ends @ 0.8 mm
R1 - Plycoat Ends @ 2.3 mm
R2 - Shoulder Wedge Ends @ 3.1 mm
R3 - Belt1 Coat Ends @ 4.6 mm
R4 - Wedge Gumstrip Ends @ 6.05 mm
R5 - Belt2 Coat Ends @ 7.5 mm
R9 - Tread Ends @ 18.6 mm


Indentation Modulus, Shoulder Region - Type D Tires, Oven Aged
Modulus (MPa)
Shoulder Position (mm)
0
2
4
6
8
10
12
14
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19
New - Barcode 1142
Oven - 55C - 12 wks - 50/50 - Capped - Barcode 1181
Oven - 65C - 8 wks - 50/50 - Capped - Barcode 1176
Oven - 65C - 8 wks - 50/50 - Capped - 24/75Bl - 50/50 - Barcode 1186
R0 - Innerliner Ends @ 0.8 mm
R1 - Plycoat Ends @ 2.3 mm
R2 - Shoulder Wedge Ends @ 3.1 mm
R3 - Belt1 Coat Ends @ 4.6 mm
R4 - Wedge Gumstrip Ends @ 6.05 mm
R5 - Belt2 Coat Ends @ 7.5 mm
R9 - Tread Ends @ 18.6 mm

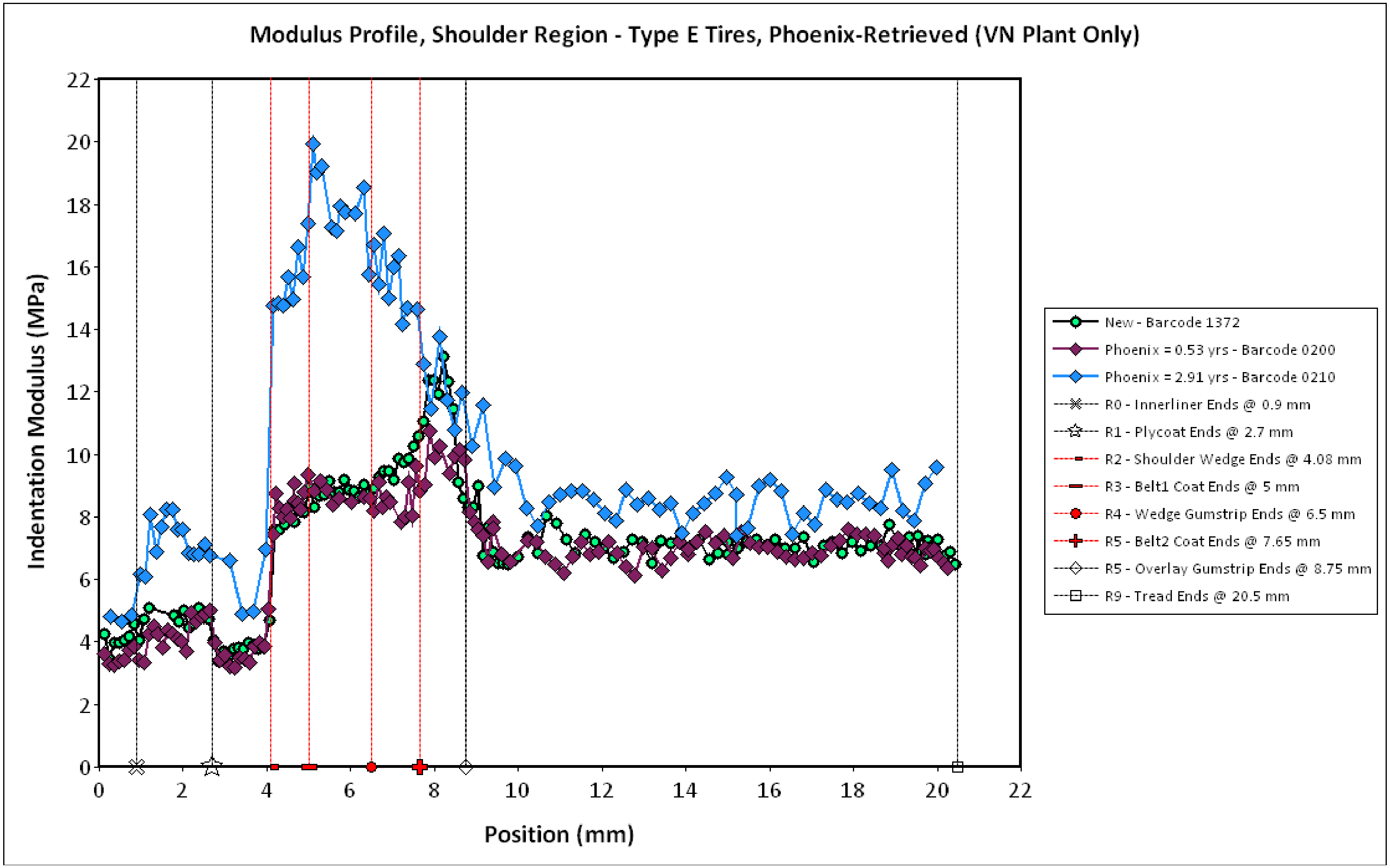

Indentation Modulus, Shoulder Region - Type D Tires, 100 Hours LTDE
Modulus (MPa)
Shoulder Position (mm)
LTDE - 60 mph - 100 hrs - Barcode 1158
LTDE - 60 mph - 100 hrs - Barcode 1174
New - Barcode 1142
R0 - Innerliner Ends @ 0.8 mm
R1 - Plycoat Ends @ 2.3 mm
R2 - Shoulder Wedge Ends @ 3.1 mm
R3 - Belt1 Coat Ends @ 4.6 mm
R4 - Wedge Gumstrip Ends @ 6.05 mm
R5 - Belt2 Coat Ends @ 7.5 mm
R9 - Tread Ends @ 18.6 mm



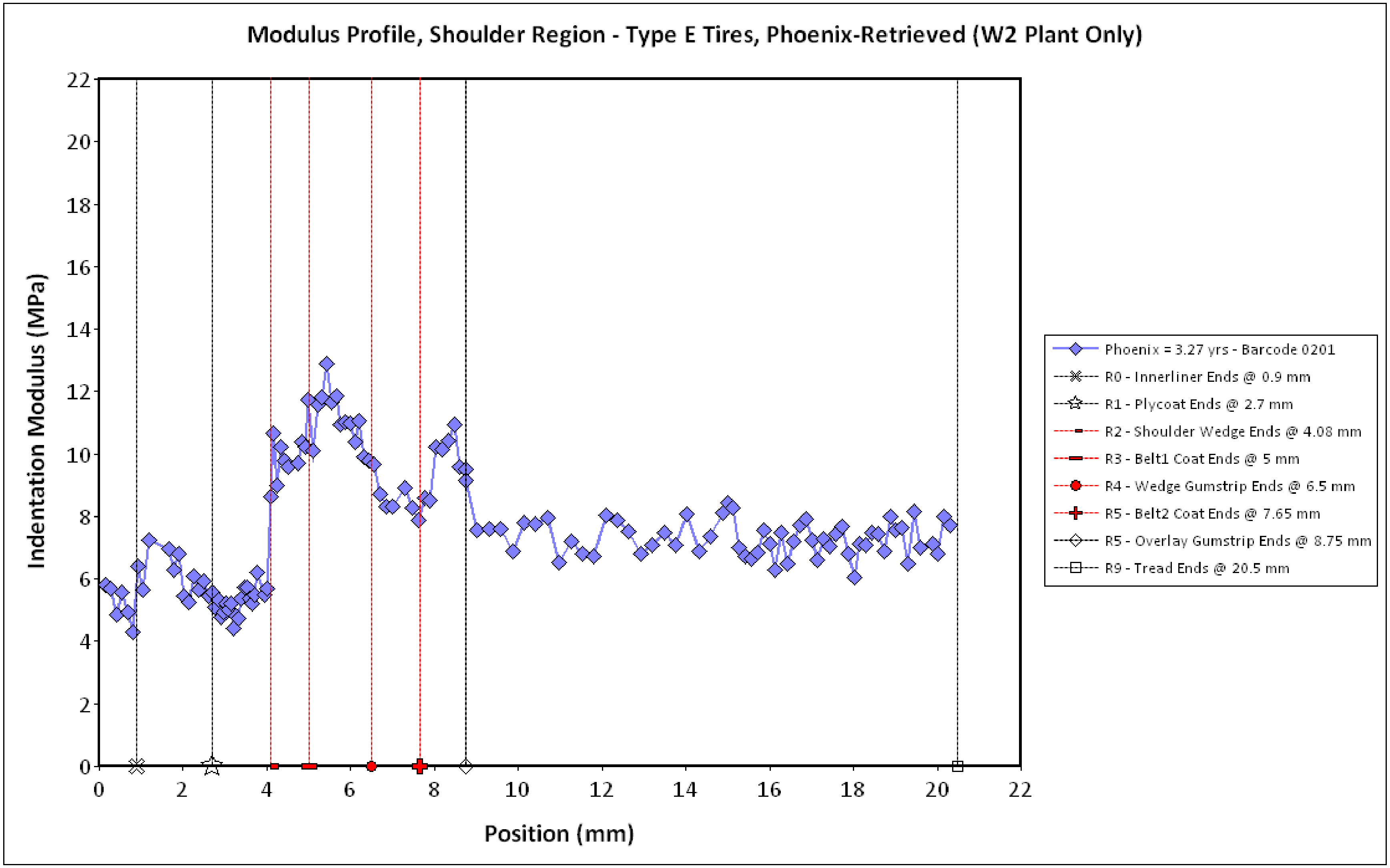
Indentation Modulus, Shoulder Region - Type D Tires, 292 Hours LTDE
Modulus (MPa)
Shoulder Position (mm)
0
2
4
6
8
10
12
14
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19
LTDE - 60 mph - 292 hrs - Barcode 1159
LTDE - 60 mph - 292 hrs - Barcode 1179
New - Barcode 1142
R0 - Innerliner Ends @ 0.8 mm
R1 - Plycoat Ends @ 2.3 mm
R2 - Shoulder Wedge Ends @ 3.1 mm
R3 - Belt1 Coat Ends @ 4.6 mm
R4 - Wedge Gumstrip Ends @ 6.05 mm
R5 - Belt2 Coat Ends @ 7.5 mm
R9 - Tread Ends @ 18.6 mm



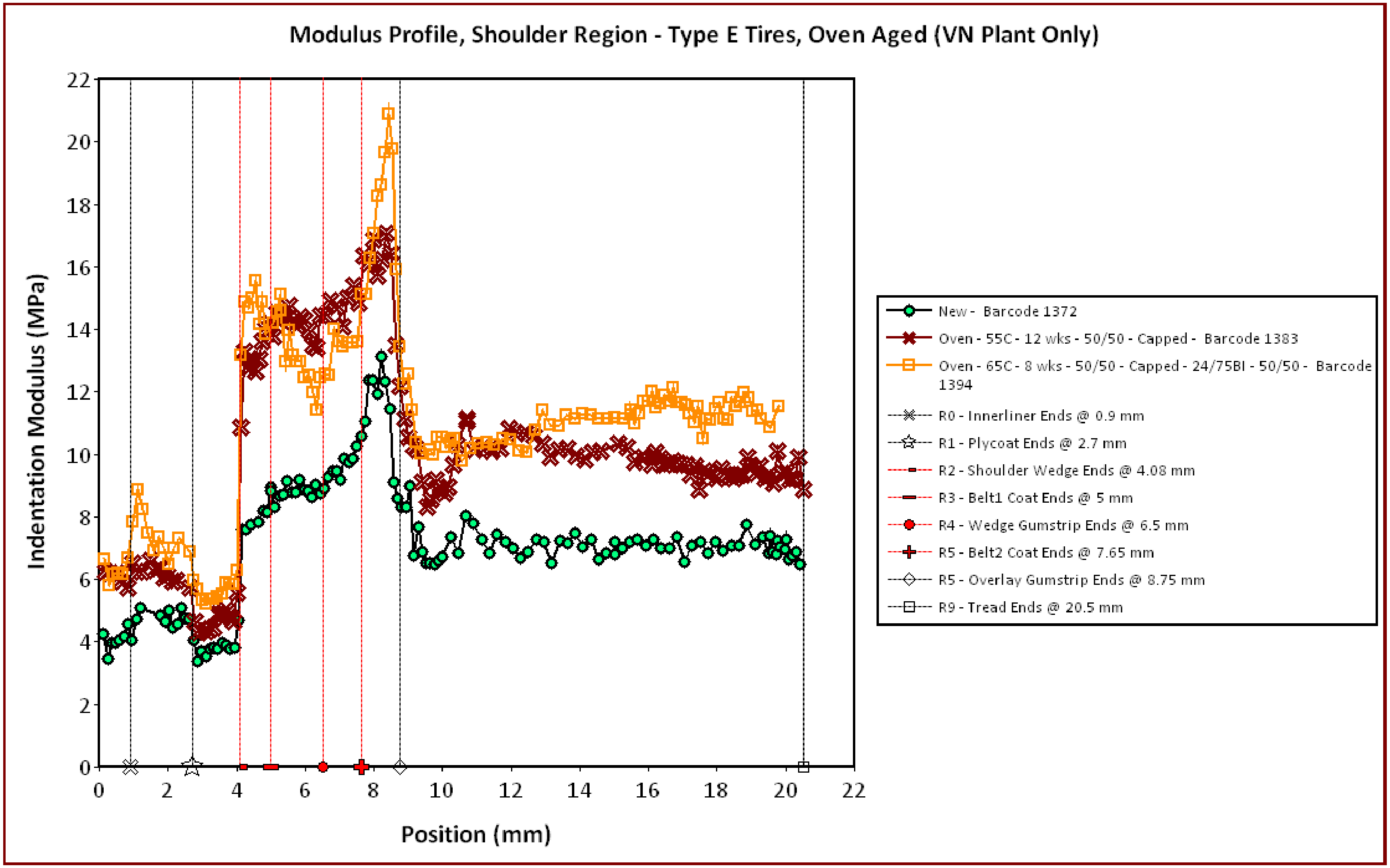
Indentation Modulus, Shoulder Region - Type D Tires, 508 Hours LTDE
Modulus (MPa)
Shoulder Position (mm)
0
2
4
6
8
10
12
14
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19
LTDE - 60 mph - 508 hrs - Barcode 1160
LTDE - 60 mph - 508 hrs - Barcode 1184
New - Barcode 1142
R0 - Innerliner Ends @ 0.8 mm
R1 - Plycoat Ends @ 2.3 mm
R2 - Shoulder Wedge Ends @ 3.1 mm
R3 - Belt1 Coat Ends @ 4.6 mm
R4 - Wedge Gumstrip Ends @ 6.05 mm
R5 - Belt2 Coat Ends @ 7.5 mm
R9 - Tread Ends @ 18.6 mm

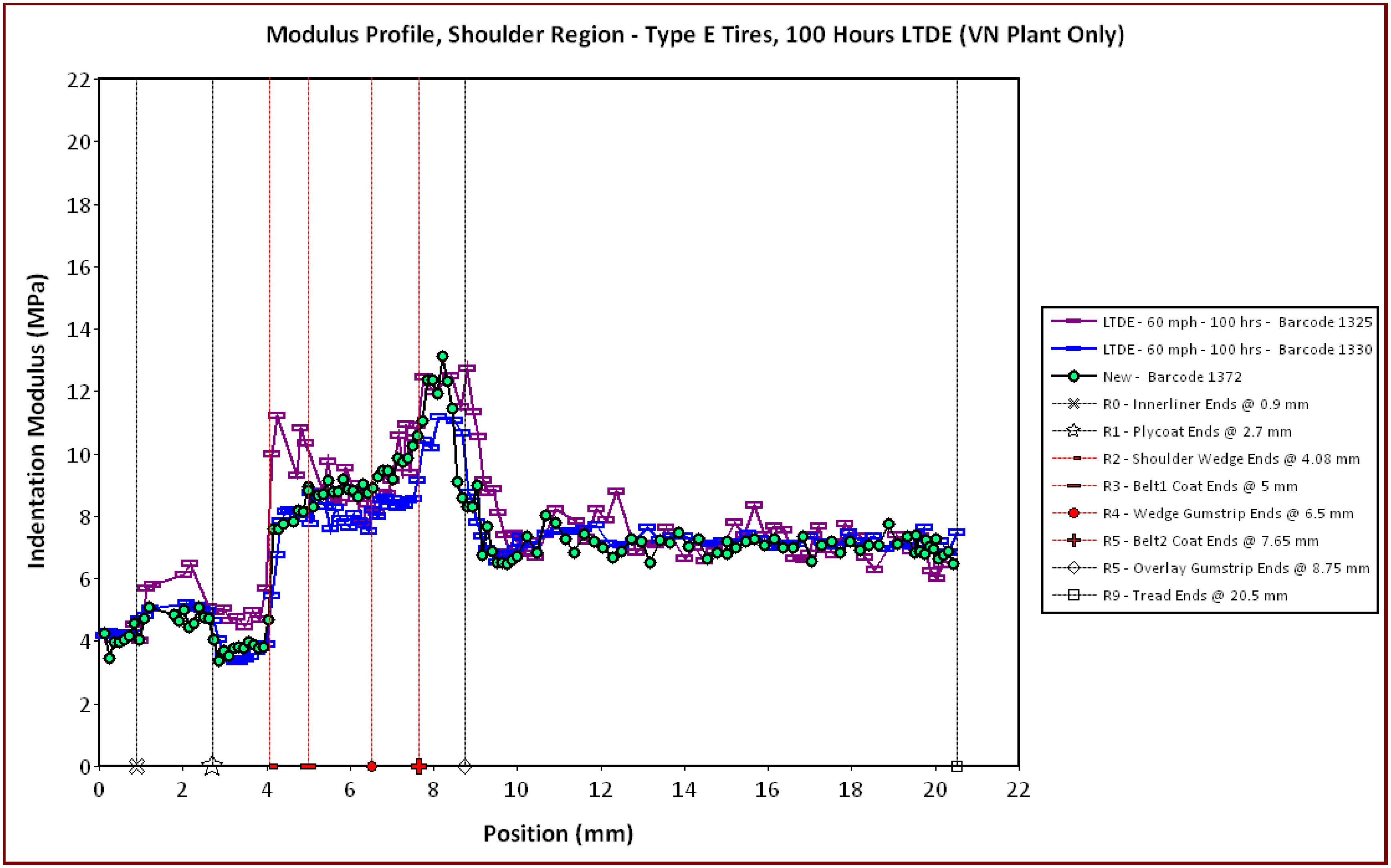


Modulus Profile, Shoulder Region - Type E Tires, Phoenix-Retrieved (VN Plant Only)
Indentation Modulus (MPa)
Position (mm)
New - Barcode 1372
Phoenix = 0.53 yrs - Barcode 0200
Phoenix = 2.91 yrs - Barcode 0210
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.7 mm
R2 - Shoulder Wedge Ends @ 4.08 mm
R3 - Belt1 Coat Ends @ 5 mm
R4 - Wedge Gumstrip Ends @ 6.5 mm
R5 - Belt2 Coat Ends @ 7.65 mm
R5 - Overlay Gumstrip Ends @ 8.75 mm
R9 - Tread Ends @ 20.5 mm

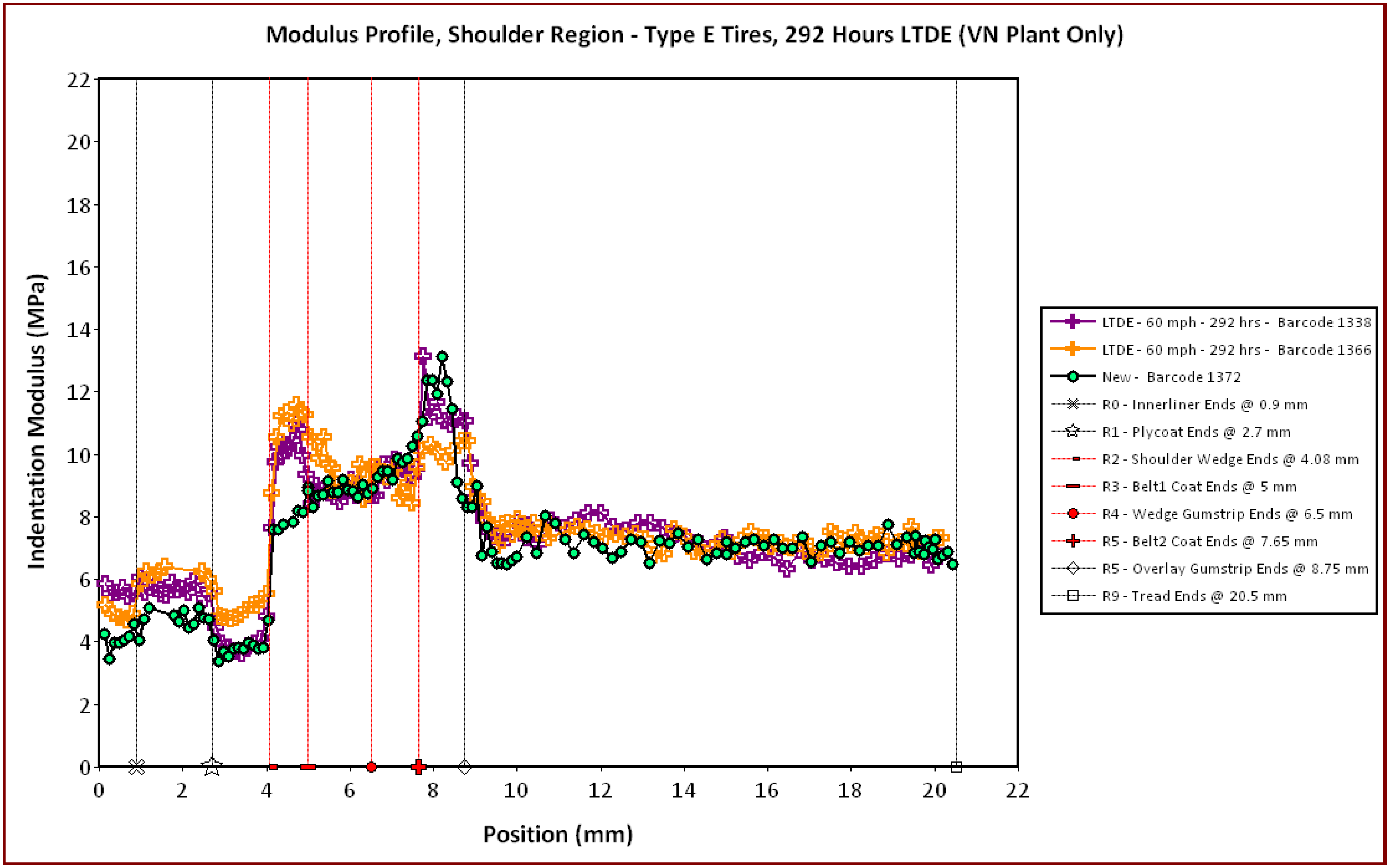


Note: Different constructions between VN & W2 plants.

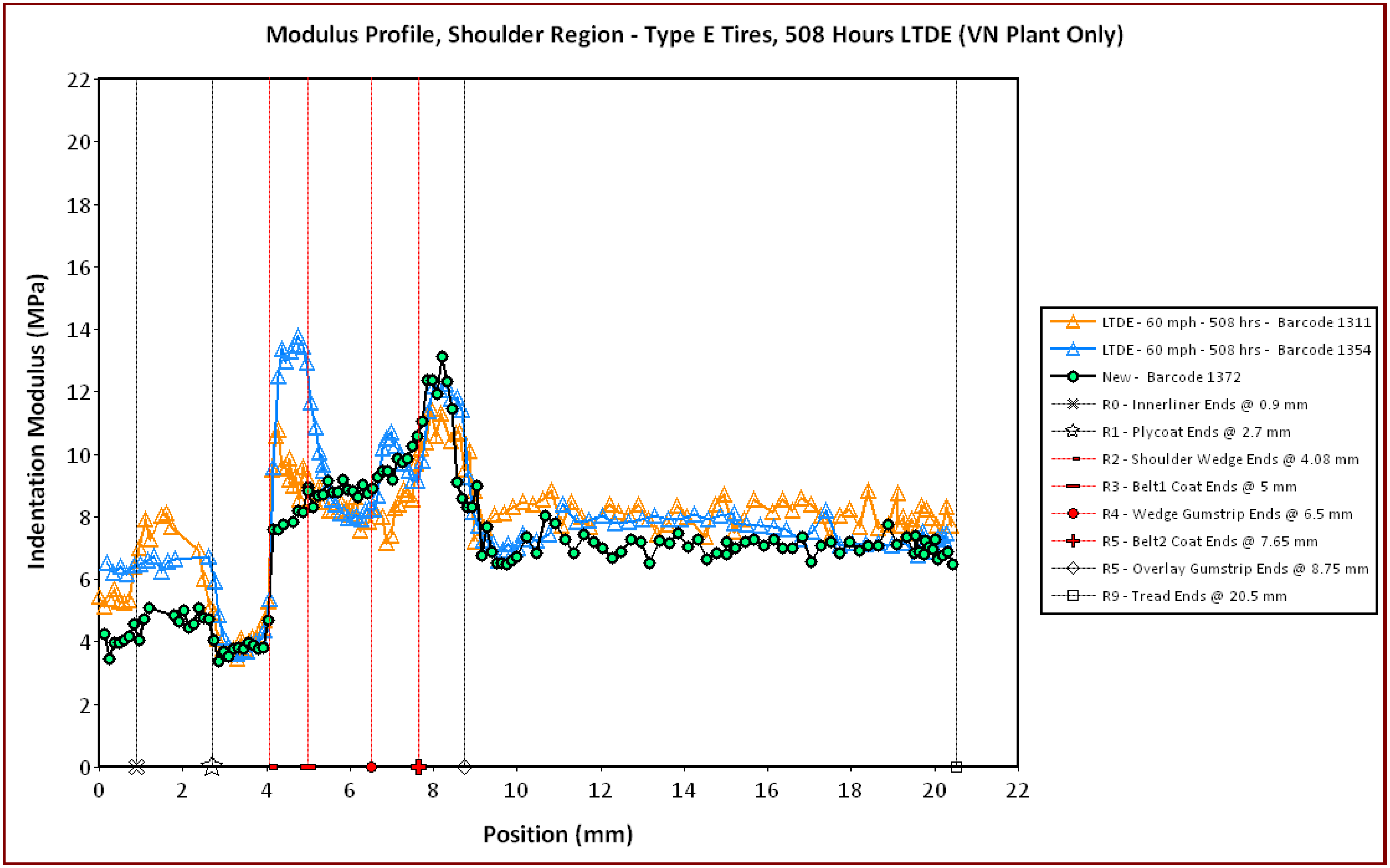

Modulus Profile, Shoulder Region - Type E Tires, Oven Aged (VN Plant Only)
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
22
New - Barcode 1372
Oven - 55C - 12 wks - 50/50 - Capped - Barcode 1383
Oven - 65C - 8 wks - 50/50 - Capped - 24/75BI - 50/50 - Barcode 1394
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.7 mm
R2 - Shoulder Wedge Ends @ 4.08 mm
R3 - Belt1 Coat Ends @ 5 mm
R4 - Wedge Gumstrip Ends @ 6.5 mm
R5 - Belt2 Coat Ends @ 7.65 mm
R5 - Overlay Gumstrip Ends @ 8.75 mm
R9 - Tread Ends @ 20.5 mm



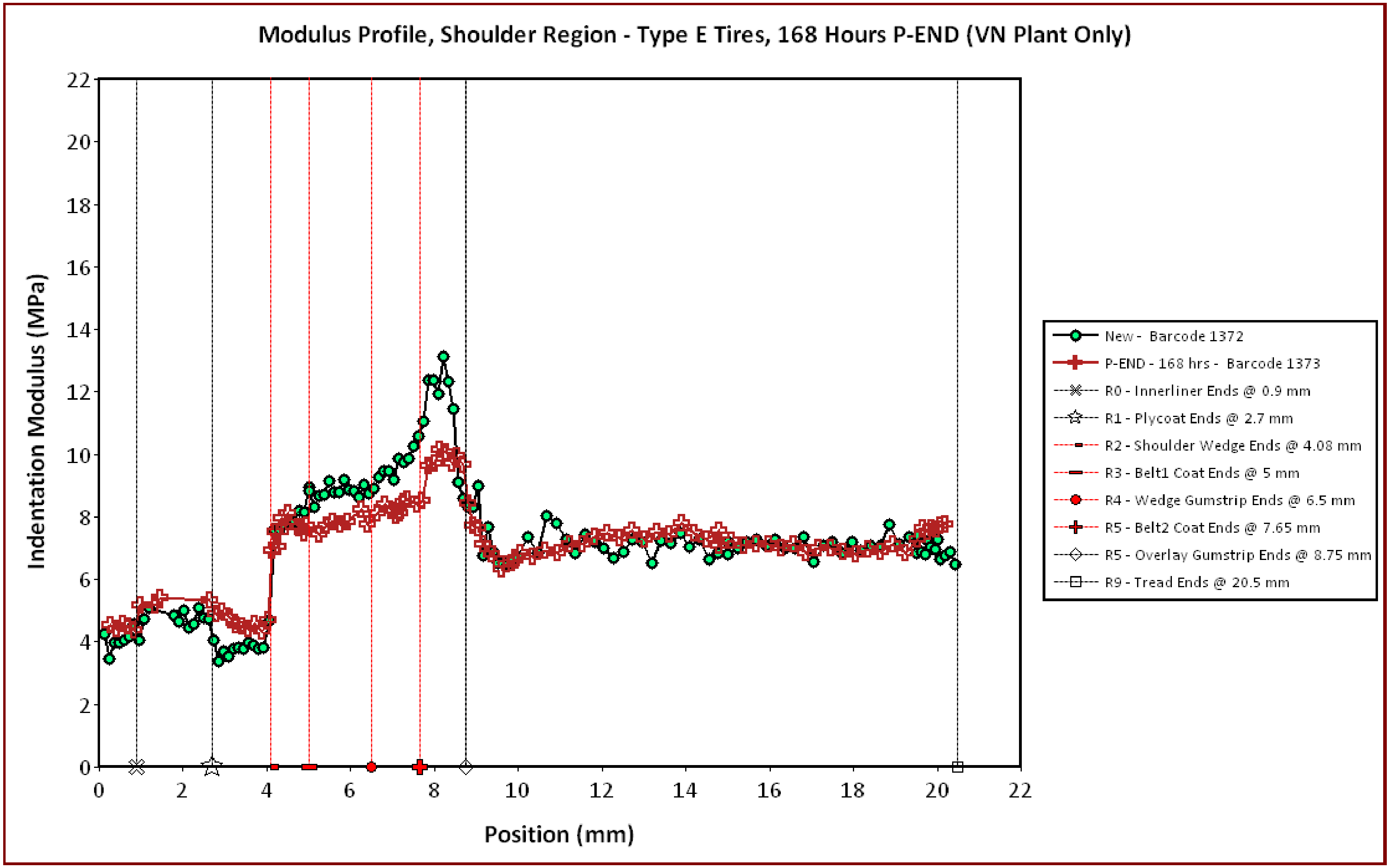
Note how LTDE targets the belt1 coat and wedge in this and subsequent figures.

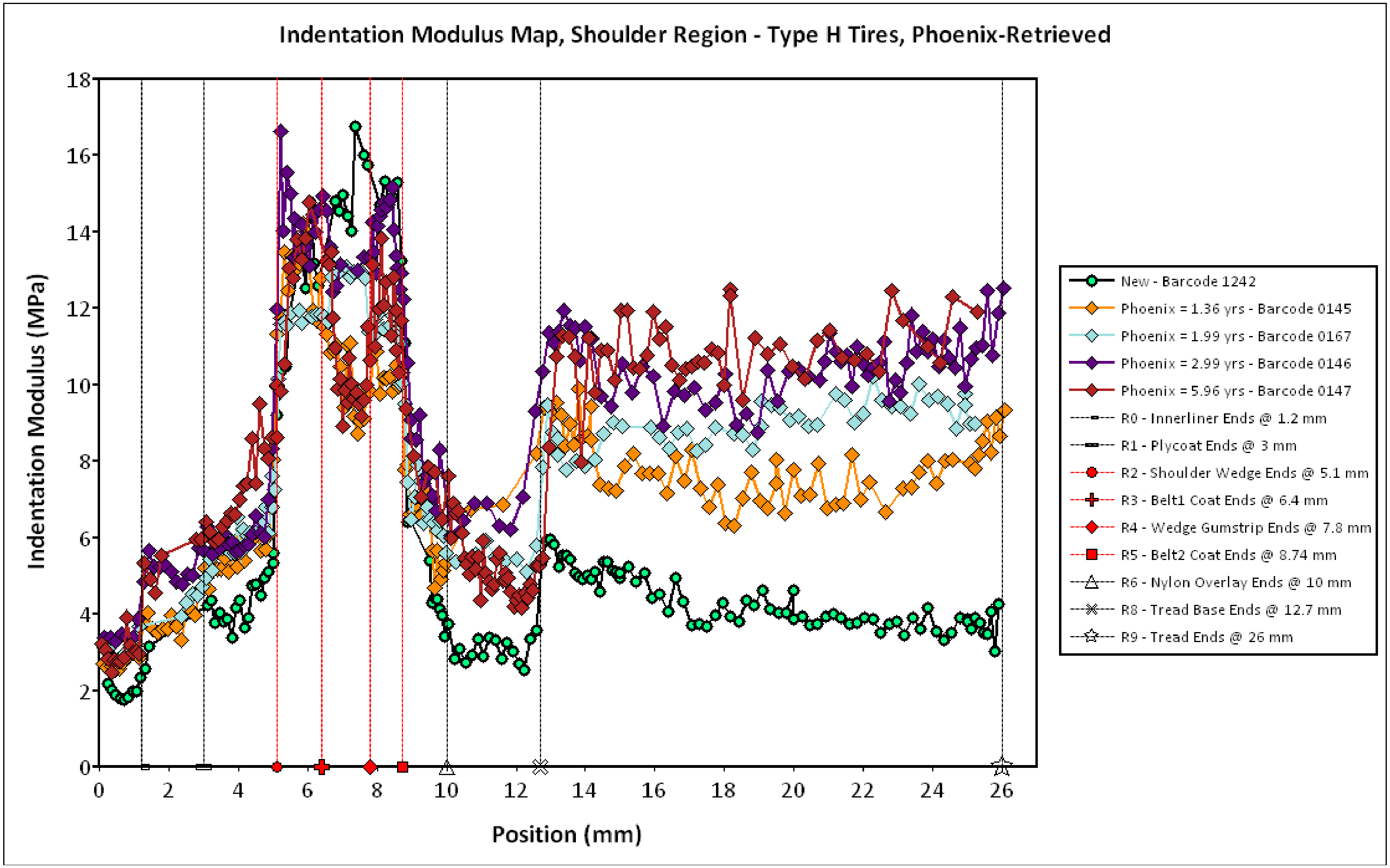


Modulus Profile, Shoulder Region - Type E Tires, 292 Hours LTDE (VN Plant Only)
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
22
LTDE - 60 mph - 292 hrs - Barcode 1338
LTDE - 60 mph - 292 hrs - Barcode 1366
New - Barcode 1372
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.7 mm
R2 - Shoulder Wedge Ends @ 4.08 mm
R3 - Belt1 Coat Ends @ 5 mm
R4 - Wedge Gumstrip Ends @ 6.5 mm
R5 - Belt2 Coat Ends @ 7.65 mm
R5 - Overlay Gumstrip Ends @ 8.75 mm
R9 - Tread Ends @ 20.5 mm

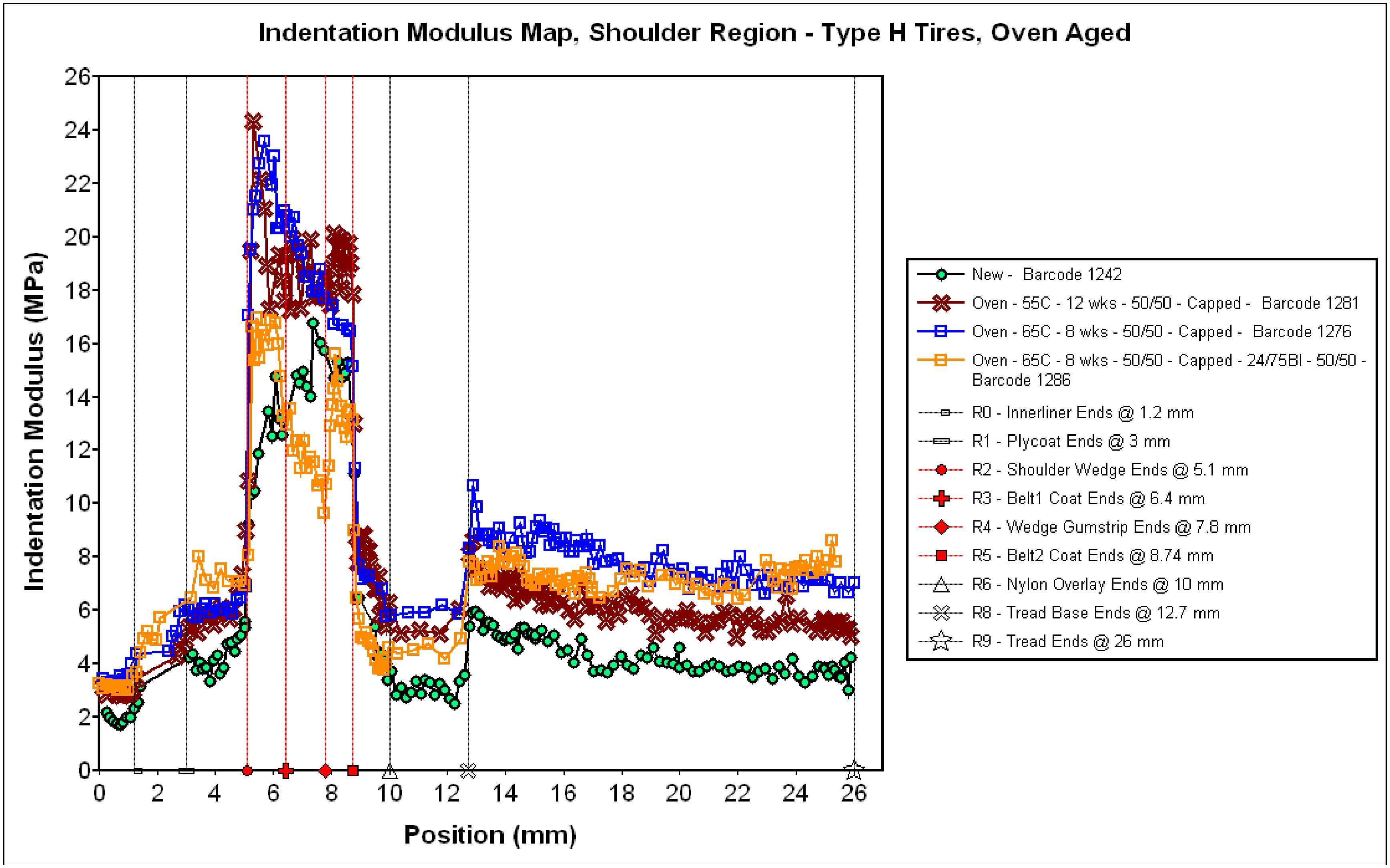


Modulus Profile, Shoulder Region - Type E Tires, 508 Hours LTDE (VN Plant Only)
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
22
LTDE - 60 mph - 508 hrs - Barcode 1311
LTDE - 60 mph - 508 hrs - Barcode 1354
New - Barcode 1372
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.7 mm
R2 - Shoulder Wedge Ends @ 4.08 mm
R3 - Belt1 Coat Ends @ 5 mm
R4 - Wedge Gumstrip Ends @ 6.5 mm
R5 - Belt2 Coat Ends @ 7.65 mm
R5 - Overlay Gumstrip Ends @ 8.75 mm
R9 - Tread Ends @ 20.5 mm

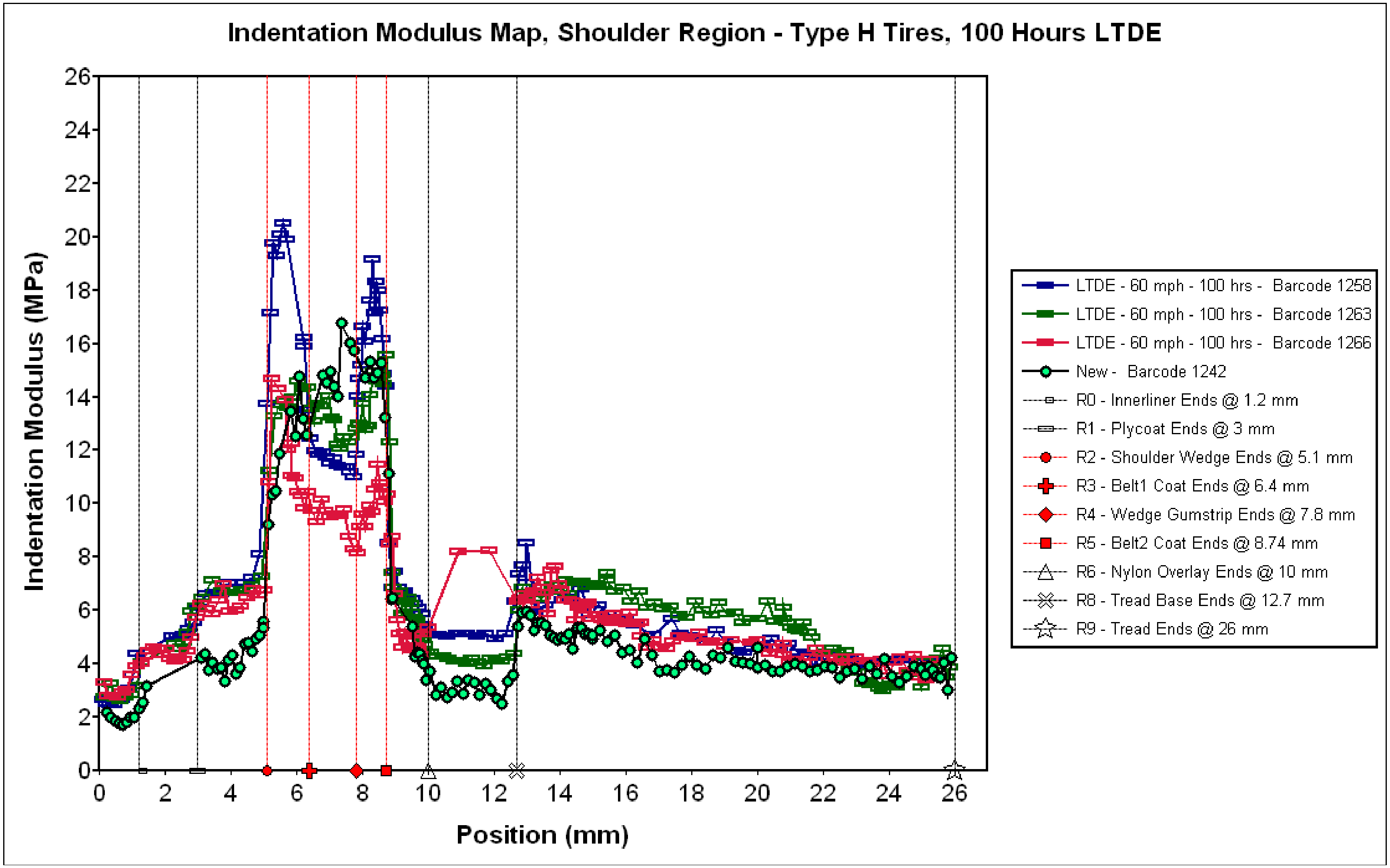

Modulus Profile, Shoulder Region - Type E Tires, 168 Hours P-END (VN Plant Only)
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
22
New - Barcode 1372
P-END - 168 hrs - Barcode 1373
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.7 mm
R2 - Shoulder Wedge Ends @ 4.08 mm
R3 - Belt1 Coat Ends @ 5 mm
R4 - Wedge Gumstrip Ends @ 6.5 mm
R5 - Belt2 Coat Ends @ 7.65 mm
R5 - Overlay Gumstrip Ends @ 8.75 mm
R9 - Tread Ends @ 20.5 mm



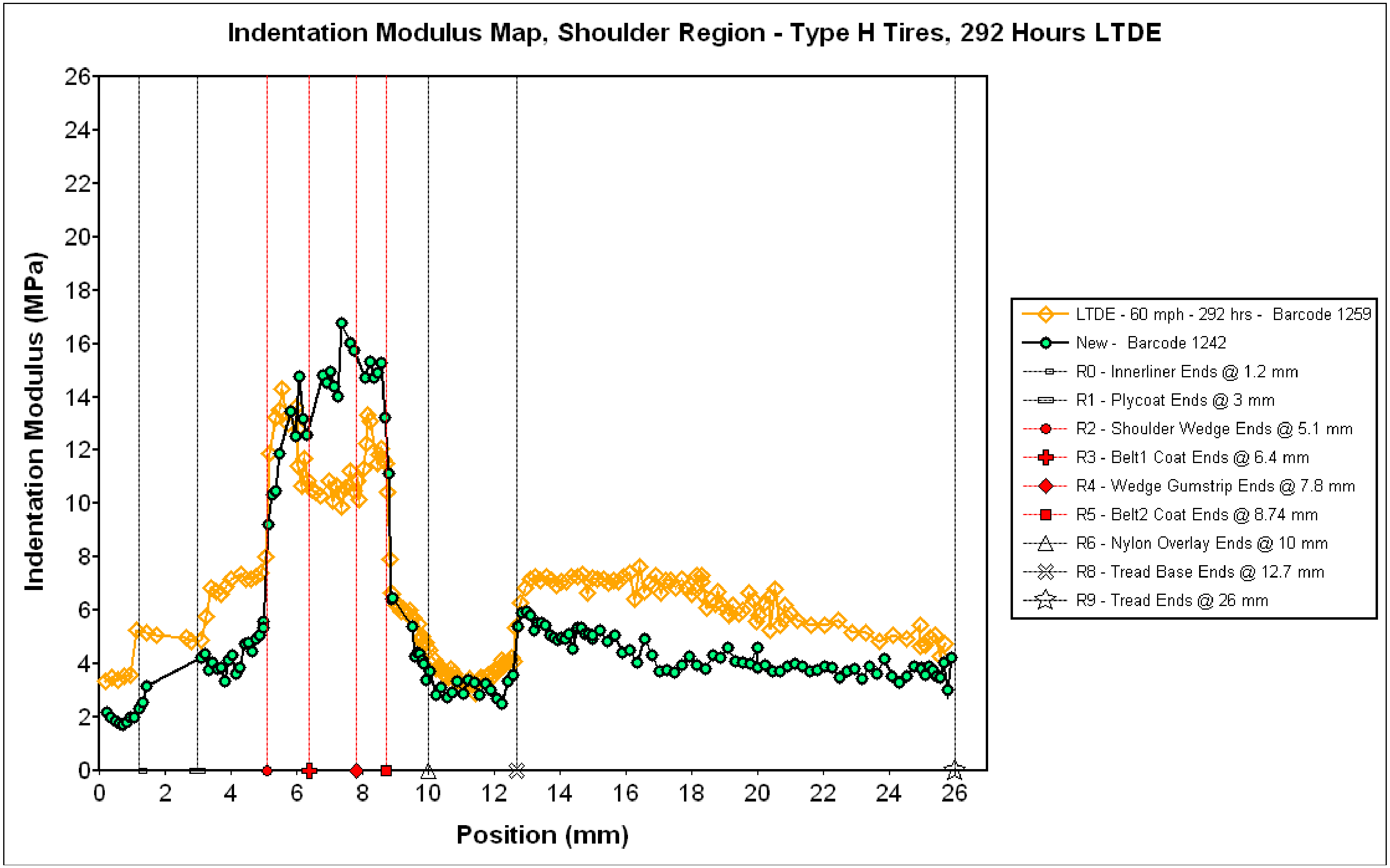
Note: Observe how the belt packages (5.1 mm to 8.74 mm) of the four type H tires retrieved from service in Phoenix soften relative to the new tire (1242).



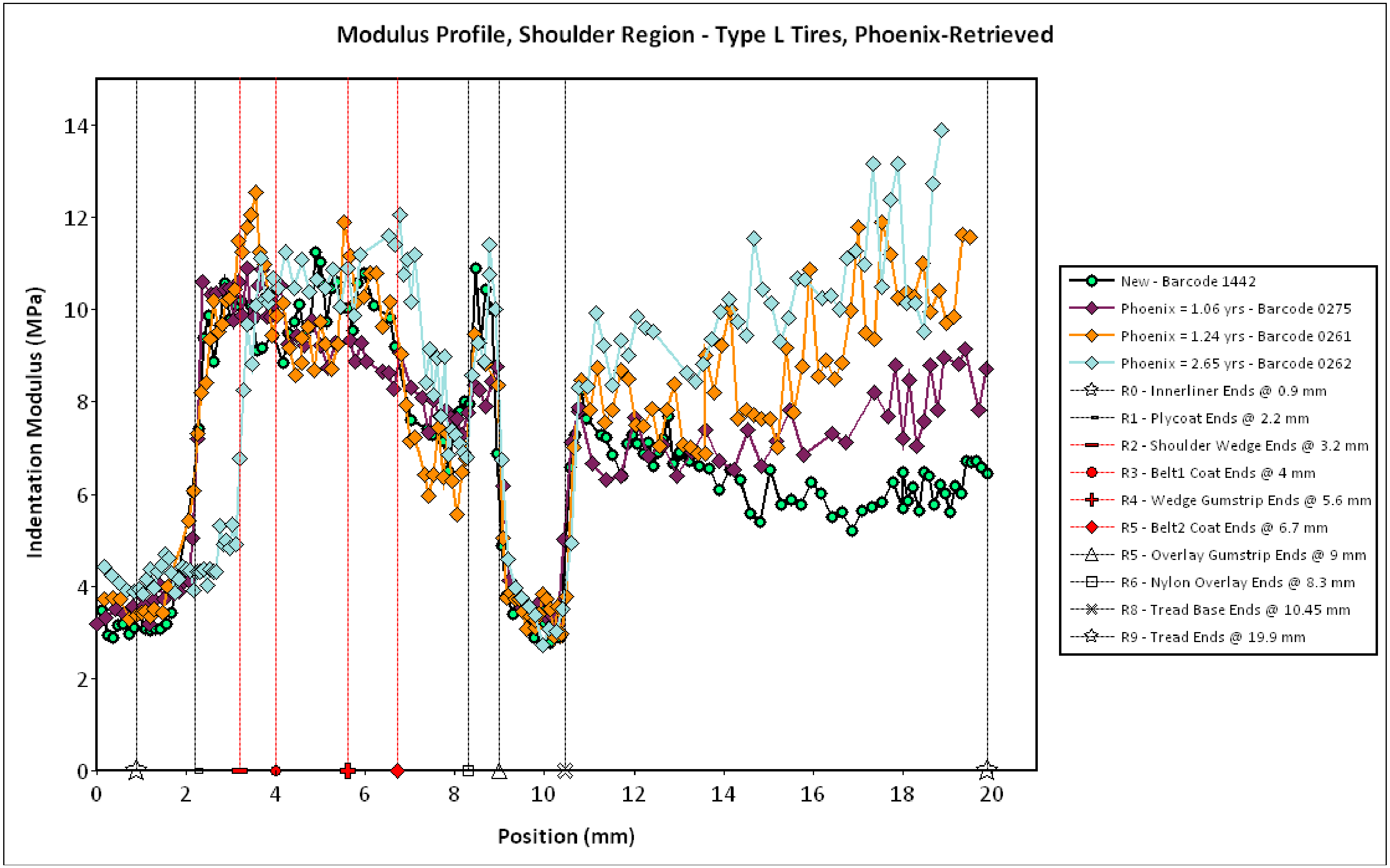
Note: Observe how the belt packages (5.1 mm to 8.74 mm) of the two type H tires that are oven aged without a roadwheel break-in harden relative to the new tire (1242), which is opposite of the trend observed in Phoenix-retrieved type H tires. Then observe how the 24-hour break-in at 75 mph prior to oven aging for tire 1286 produced results similar to the belt-package softening observed in Phoenix-retrieved tires.

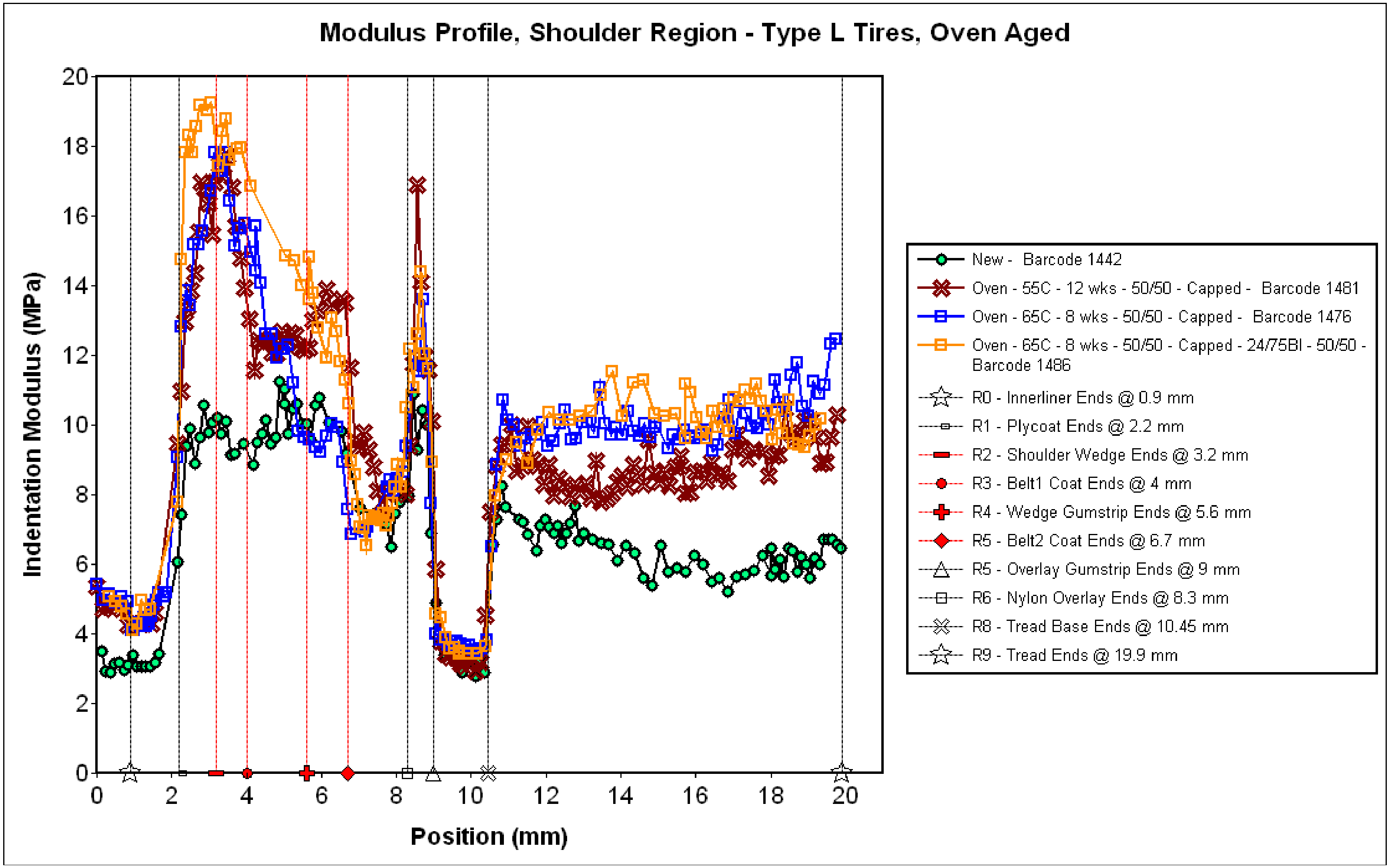


Note: Roadwheel testing of new H tires again produced belt-package softening similar to that observed in Phoenix-retrieved type H tires.

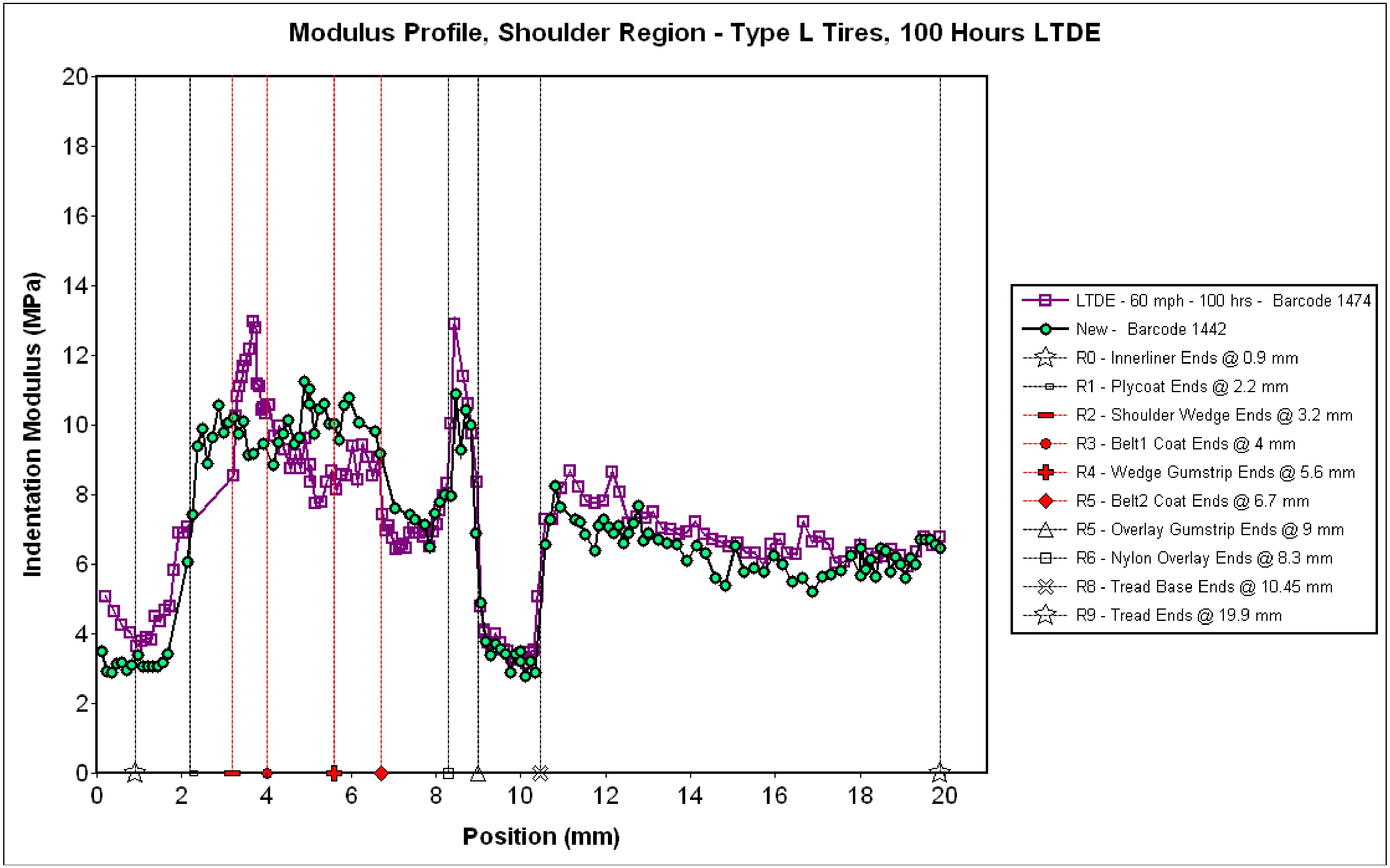


Note: Roadwheel testing of new H tires again produced belt-package softening similar to that observed in Phoenix-retrieved type H tires.

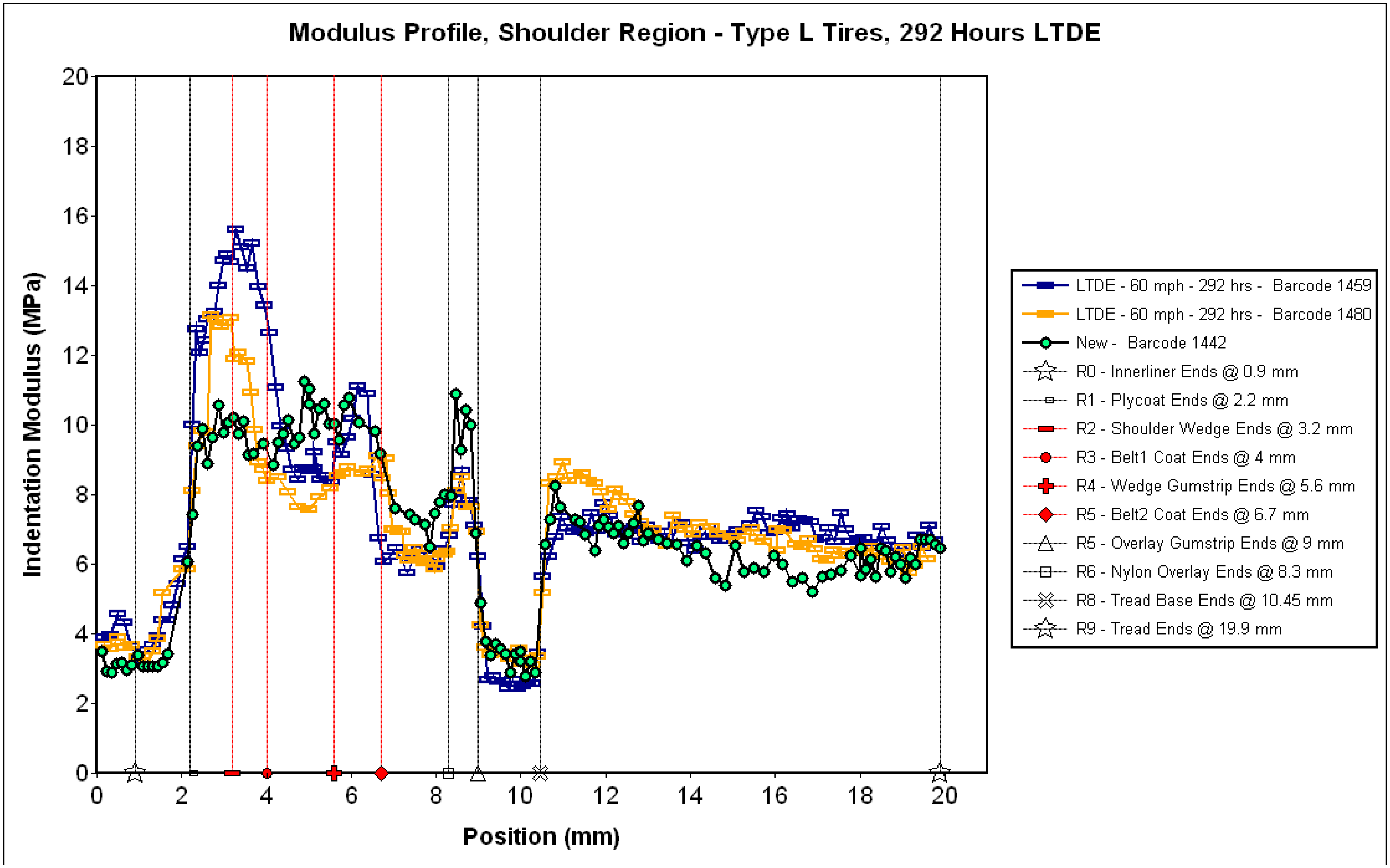

Modulus Profile, Shoulder Region - Type L Tires, Phoenix-Retrieved
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
New - Barcode 1442
Phoenix = 1.06 yrs - Barcode 0275
Phoenix = 1.24 yrs - Barcode 0261
Phoenix = 2.65 yrs - Barcode 0262
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.2 mm
R2 - Shoulder Wedge Ends @ 3.2 mm
R3 - Belt1 Coat Ends @ 4 mm
R4 - Wedge Gumstrip Ends @ 5.6 mm
R5 - Belt2 Coat Ends @ 6.7 mm
R5 - Overlay Gumstrip Ends @ 9 mm
R6 - Nylon Overlay Ends @ 8.3 mm
R8 - Tread Base Ends @ 10.45 mm
R9 - Tread Ends @ 19.9 mm



Modulus Profile, Shoulder Region - Type L Tires, Oven Aged
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
New - Barcode 1442
Oven - 55C - 12 wks - 50/50 - Capped - Barcode 1481
Oven - 65C - 8 wks - 50/50 - Capped - Barcode 1476
Oven - 65C - 8 wks - 50/50 - Capped - 24/75BI - 50/50 - Barcode 1486
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.2 mm
R2 - Shoulder Wedge Ends @ 3.2 mm
R3 - Belt1 Coat Ends @ 4 mm
R4 - Wedge Gumstrip Ends @ 5.6 mm
R5 - Belt2 Coat Ends @ 6.7 mm
R5 - Overlay Gumstrip Ends @ 9 mm
R6 - Nylon Overlay Ends @ 8.3 mm
R8 - Tread Base Ends @ 10.45 mm
R9 - Tread Ends @ 19.9 mm



Modulus Profile, Shoulder Region - Type L Tires, 100 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
LTDE - 60 mph - 100 hrs - Barcode 1474
New - Barcode 1442
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.2 mm
R2 - Shoulder Wedge Ends @ 3.2 mm
R3 - Belt1 Coat Ends @ 4 mm
R4 - Wedge Gumstrip Ends @ 5.6 mm
R5 - Belt2 Coat Ends @ 6.7 mm
R5 - Overlay Gumstrip Ends @ 9 mm
R6 - Nylon Overlay Ends @ 8.3 mm
R8 - Tread Base Ends @ 10.45 mm
R9 - Tread Ends @ 19.9 mm



Modulus Profile, Shoulder Region - Type L Tires, 292 Hours LTDE
Indentation Modulus (MPa)
Position (mm)
0
2
4
6
8
10
12
14
16
18
20
LTDE - 60 mph - 292 hrs - Barcode 1459
LTDE - 60 mph - 292 hrs - Barcode 1480
New - Barcode 1442
R0 - Innerliner Ends @ 0.9 mm
R1 - Plycoat Ends @ 2.2 mm
R2 - Shoulder Wedge Ends @ 3.2 mm
R3 - Belt1 Coat Ends @ 4 mm
R4 - Wedge Gumstrip Ends @ 5.6 mm
R5 - Belt2 Coat Ends @ 6.7 mm
R5 - Overlay Gumstrip Ends @ 9 mm
R6 - Nylon Overlay Ends @ 8.3 mm
R8 - Tread Base Ends @ 10.45 mm
R9 - Tread Ends @ 19.9 mm

5.0 REFERENCES

1. National Highway Traffic Safety Administration. (2001). *Engineering Analysis Report and Initial Decision Regarding EA00-023: Firestone Wilderness AT Tires;* Washington, DC: Author. 2001. Available at www.nhtsa.gov/nhtsa/announce/press/Firestone/firestonereport
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