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SPECIAL REPORT
DURABILITY AND DURABILITY
TESTS FOR PAVING ASPHALT
A State-of-the-Art Report

Dah-yinn Lee
Conducted by the Engineering
Research Institute
for the
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DURABILITY AND DURABILITY TESTS FOR PAVING ASPHALTS

INTRODUCTION

This special report is prepared to review durability and durability tests for paving asphalt, both in theory and in application. The report summarizes and evaluates factors related to asphalt durability, problems associated with durability study and development of durability tests, important work on durability and practical design implications concerning asphalt durability. It is a state-of-the-art report and a part of the study under HR-124, Development of Laboratory Durability Test for Asphalts.

For clarity, the following definitions are used in this report:

Bitumen: Mixtures of hydrocarbons which are completely soluble in carbon disulfide. Bituminous materials are substances in which the predominating constituents are bitumens.

Asphalt: Black to dark brown, solid to semi-solid substances, in which the predominating constituents are bitumens, all of which occur in nature (native asphalts) or are obtained as the residue of fractional distillation of petroleum crudes (petroleum, refined or manufactured asphalts, or, as used in this report, simply asphalts).

It is to be noted that, in Great Britain, this material is called asphaltic bitumen, and the term asphalt is used for the mixture, either natural or mechanical, of asphaltic bitumen and inert mineral aggregates.

Durability: All bituminous paving materials change with time and under the action of heat, weather, and traffic. Such changes are, in general, detrimental to their function as binders and as waterproofing agents. The degree of resistance of a bituminous binder to these changes is referred to as its durability.

BACKGROUND

Current practice in the design of asphaltic paving mixtures is to try to arrive at a balanced design among a number of desirable mix properties, including (a) stability, (b) durability, (c) flexibility, (d) fatigue resistance, (e) skid resistance, (f) imperviousness and (g) fracture or tensile strength.

Studies and experiences have shown that mixtures with higher asphalt content are usually associated with desirable durability which also give adequate flexibility, fatigue resistance, imperviousness and fracture strength properties. On the other hand, low asphalt content mixture yields high stability and good skid resistance. As a result, asphalt paving mixture design, i.e., selection of aggregate gradation and asphalt content, is a compromise between stability and durability of the mixture.

Durability of a paving mixture is usually defined as its resistance to weathering, aging and traffic loading or as the ability to resist change due to these destructive or deteriorative factors. The best way to examine the problems of asphalt durability is from the viewpoint of pavement failure.

Bituminous pavements, may fail due to (a) cracking, (b) disintegration and (c) instability, any of which may result from improper selection and use of types and amounts of asphalt. From thepractical point of view, the desired properties of any bituminous paving binder should include:

1. Proper consistency or rheological properties: the material can be made sufficiently fluid to permit complete mixing with the aggregate to retain a good bond between the binder and the road aggregate to resist stress and water action. The material should be relatively viscous at high pavement temperatures to insure pavement stability and not become brittle at low pavement temperatures to avoid cracking and disintegration.

2. Adequate durability properties: the material should resist deterioration resulting from chemical and physical changes during production, and weathering and traffic stresses and strains in service, especially changes in hardness or consistency. The increase in hardness may result in loss of ability to deform without fracture and loss of adhesion and fatigue resistance.

The consistency of asphalt can be reasonably determined, predicted and specified in terms of either empirical tests such as penetration and ductility, or more fundamental measurements such as viscosity, stiffness or complex (dynamic) modulus, over ranges of temperature and rate of loading or frequency. In a more recent paper, Dobson[1] suggested the use of complex modulus at 25°C to specify paving bitumen.

The determination, prediction and specification of durability of paving asphalts, however, is much more complex and much less readily obtainable, even though many efforts have been spent on the subject. The major reasons are:

1. Durability can not be easily and clearly defined. Mechanisms of asphalt deterioration and parameters to measure or indicate durability or deterioration are difficult to express in simple physical terms.

2. Durability depends on the chemical makeup of asphalts, yet chemical composition of asphalt is not very well understood.

3. Durability is time dependent. The real test of durability is on the road. Road testing is not only time consuming and expensive, but many local mixture variables greatly affect durability. These factors include: type and grade of asphalt, asphalt content, type of mixture, traffic conditions, temperature, moisture, air content, film thickness, aggregate surface effects, permeability, etc.

Nevertheless, great progress has been made since the 1950's. In the following sections, the author attempts to discuss and summarize some of the more important current knowledge and some recent studies concerning factors that affect asphalt durability. Also discussed are tests and methods to predict potential durability, and current practices and future trends in insuring the use of durable asphalt in paving mixtures.

FACTORS THAT AFFECT DURABILITY OF PAVING ASPHALT

It is generally agreed that asphalt hardening is the most important single factor that causes asphalt paving to crack and disintegrate. Therefore, the degree and rate of asphalt hardening can indicate relative durability of asphalt and many durability tests have been proposed centering around the evaluation of the resistance of asphalt to hardening.

Factors which may contribute to asphalt hardening under service conditions are[2,3]: (a) oxidation, (b) volatilization, (c) polymerization, (d) thixotropy (age hardening or hardening with time), (e) syneresis (exudation of oil), (f) separation (absorption and adsorption of asphaltic components by aggregate), (g) photo oxidation, (h) water, (i) microbiological deterioration, and (j) photochemical action.

There is considerable disagreement as to the exact mechanism and dominant factors influencing the hardening of asphalts. However, the first four of the ten above-listed factors represent the major important factors recognized by most asphalt paving technologists both in the United States and in Europe. The ten factors have been studied by various research workers. They are defined and discussed, as related to durability of paving asphalts, in more or less qualitative terms, as follows.

Oxidation

Since asphalts are largely hydrocarbon materials, they are subject to oxidation in the atmosphere. This reaction leads to an increase in viscosity and a change in the colloidal structure of the materials (increase in asphaltene content) from "sol" to "gel" types. It is influenced by time, temperature and sunlight. However, oxidation in the absence of sunlight is of prime importance as far as paving asphalt durability is concerned.

One of the earliest papers to point out that oxidation takes place when asphalt is exposed to weathering was that of Hubbard and Reeve[4]. They exposed asphalt cements, liquid asphalts and tars in a layer 1/8-in. thick outdoors for one year and found that oxidation contributes to an increase in hardness and the development of insoluble matter in the asphalt.

One of the earliest works in isolating and measuring oxygen absorption by asphalt was that of Thurston and Knowles[5]. They thus established oxidation rates for both asphalts and their constituents, then concluded that: (a) oxygen absorption increased with time, and (b) asphaltenes oxidized faster than the original asphalt, while the oil constituents were the most resistant to oxidation.

One important work in this area was carried out by van Oort[6] who investigated the absorption of oxygen by thin layers of bitumen in the order of 10μ at temperatures between 20° and 70°C. It was found that the rate of hardening due to oxidation, as measured by viscosity, decreases with time and depends markedly on temperature and the chemical type of the bitumen. These findings are in general agreement with those of many others.

Blokker and van Hoorn[7] extensively studied oxidation of road bitumen. They concluded that the rate of oxidation in terms of oxygen absorption is much higher in the presence than in the absence of light. Oxidation in the light is promoted mainly by the ultra-violet part of the spectrum and hardening is restricted to 4μ. In the dark, all reacting oxygen is bound in SO groups and CO groups. Hardening in this case may occur down to depths of 3 to 5 mm. Work done by the author[8] on the effect of film thickness in Thin Film Oven Test indicates that, based on evidences of changes in viscosity and asphaltene content due to oxidation, oxidation penetration ranged between 4 to 7 mm.

Data on chemical changes during oxidation of asphalts are not conclusive. Studies by Greenfeld and others [5,9,10] indicated the following: (a) the oil fraction produced asphaltenes, water-soluble products, and volatile products; (b) the asphaltene content increases due to oxidation of oils rather than polymerization of the oils; (c) formation of higher molecular weight or oxygen-rich asphaltenes; (d) the sulfur and nitrogen compounds in oils form sulfur and nitrogen compounds which were pentane insoluble; (e) formation of water-soluble degradation products, water, and carbon dioxide.

Volatilization

Volatilization is the evaporation of the volatiles or the lighter constituents from asphalt and is primarily a function of temperature. Volatilization may or may not be important as a factor causing hardening of asphalt depending upon the type of application (especially temperature) and nature (chemical composition and viscosity) of asphalt.

Volatile evaporation from asphalt cements produced by a modern refining process at road service temperatures is unlikely or else is insignificant. However, when hot asphalt is mixed with hot aggregate and spread in thin films, as in the mixing process of an asphaltic concrete, the conditions are very favorable to loss of volatiles. Many investigators[11,12,13] believe that during the mixing process volatilization is one of the major causes for hardening of asphalt.

Qualitative detection of existence of volatiles in asphalt can be determined by the Flash and Fire Point Test (ASTM D92). To quantitatively assess how much hardening during hot mixing of asphalt is due exclusively to volatilization, or the relative importance of volatilization in asphalt hardening is difficult, if not impossible. This is due to the closely associated mechanisms of volatilization, oxidation and polymerization. One way to evaluate the hardening of asphalt due to volatilization is by heating asphalt in inert atmosphere. In this approach, while oxidation is eliminated, only a part of the asphalt hardening may be attributed to the loss of volatiles since heat is involved. Another approach to the problem has been various forms of oven heating or volatilization tests, especially Loss on Heating (ASTM D6) and Thin Film Oven Test (ASTM D1754).

Polymerization

Polymerization refers to the process of combining like molecules to form larger molecules. For the asphalt paving technologists this term is used to explain or designate the hardening of asphalt by heat alone. The rate of hardening due to polymerization depends mainly on temperature, time and the composition of the bitumen. Hardening due to polymerization

increases with temperature and time. However, there was evidence[3] that when the temperature of heating is very high, some asphalts may "depolymerize," resulting in a decrease of viscosity.

Thixotropy

This is also called age hardening or aging. It is a progressive hardening due to the formation of an internal structure in the asphalt over a period of time and at temperatures well below the softening point. This is considered a thixotropic effect because much of the hardening can be destroyed by reheating and mechanical working of the material. Traxler and Schweyer[14] demonstrated that asphalts stored in a viscometer at 25°C hardened with time. An aging index was proposed which was the slope of the log viscosity vs log time curve for an asphalt. The index was expressed as AAI = m where m is the slope at 100 hr. The degree of age hardening and amounts of reversible and permanent hardening depend upon the composition of the asphalt. Gel-type bitumen is more susceptible to age hardening than the sol-type bitumens[14].

Thixotropic hardening is most evident in pavements which have little or no traffic.

Syneresis

Syneresis is the exudation of paraffinic oils from the asphalt. With the elimination of these oily constituents, the asphalt becomes harder. The amount of syneresis is directly related to the colloidal nature of the asphalt.

Studies[15,16] have shown that a sol-type, in which the asphaltenes are well-dispersed by the presence of adequate aromatic resins, generally shows very little oil exudation. On the other hand, gel asphalts, in which the asphaltenes are not well dispersed, show marked syneresis. Asphalts from various crudes and processed in different ways show widely varying degrees of exudation.

This phenomenon is related to loss of oil from a paving asphalt to a porous stone through absorption.

Separation

Separation is the removal of the oily constituents, resins or asphaltenes from the asphalt as caused by selective absorption or adsorption of some aggregates. Absorption of oil by aggregate has been known to occur in pavement containing porous or absorptive aggregates[3,17,18]. The amount and nature of absorption depends on time, porosity, and pore size distribution of aggregate, composition of the aggregate, and viscosity and composition of asphalt[18]. Selective absorption may result in harder and thinner films in a mixture, thus affecting durability of the pavement.

Adsorption of asphaltenes and resins by soils containing clay was reported by Traxler[19]. However, the effect of this reaction on durability of asphalt is not well documented.

Photooxidation and Photochemical Actions

These effects are the hardening of asphalt due to the action of actinic light in the presence and absence of oxygen. Both are effective only to a short distance into an asphalt film (about 3 to 10μ)[3,6,20].

The mechanisms of these reactions are not well established. However, work by Strieter and Snoke[21] indicated that some water-soluble products including acids and ketones were formed. The formation of these compounds depended on the combined action of light, heat and oxygen. A study by Dickinson, et al.[20] showed that light in the wavelength range of 3000 to 5000Å accelerated the reaction of oxygen with bituminous binders.

Extensive, excellent research work on effects of light on durability of roofing asphalts has been done at the U. S. National Bureau of Standards. For a general discussion, the reader is referred to Ref. 22.

Water

The usefulness of asphalt in a majority of its applications lies in its low water permeability and low water absorption. The permeability constants for asphalts are 2 to 5 X 10^{-9} g - cm/sq cm - mm Hg - hr[22]. Asphalt also has a limited solubility for water which is the range of 0.01%[23].

Studies have shown that water absorption by asphalt is a surface phenomenon. Percent absorption decreases with increasing film thickness. Slotboom[23] exposed 5 mm asphalt films to tap water for 10 yr. He found 6.0% water absorption for the outer layer and 1.5% for the inner layer. Note that this condition of extended exposure to water rarely exists for paving asphalts.

The major detrimental effects of water on paving asphalt are in: (a) loss of water-soluble degradation products due to oxidation and photo-oxidation; and (b) since water is an effective catalyst for a number of chemical reactions, oxidation and photooxidation may be accelerated by its presence[3]. However, qualitative data are needed in this area to substantiate these.

It must not be overlooked that water plays an important role in the durability of asphalt paving mixtures through its influence on adhesion. Deterioration of the bond at the aggregate-binder interface and permeability may cause disintegration and failure of the pavement structure.

Microbiological Deterioration

Under environments favorable for bacterial or fungal growth, pipe coatings, paving cements and other asphalts in contact with soil can be degraded by soil flora. Burgess[24] presented an interesting discussion of bacterial action on roads. Harris, et al.[25] have studied the effect of bacteria on asphalt-aggregate systems and found that, depending on the nature of the bacteria present, either softening or hardening of asphalt may result from microbial action.

Traxler[26] reported some exploratory studies on the bacterial degradation of asphalts. The most important factors that control the rate of degradation associated with the microbial attack were enumerated as follows: (a) type of microorganism, (b) temperature of growth, (c) effect of pH, (d) effect of oxygen tension, and (e) composition of the asphalt.

Mixture Properties

Since asphalt is used in conjunction with aggregate in paving mixtures and represents only 5% by weight of the mixture, other variables associated with the mixture and the aggregate in the mixture should not be overlooked when evaluating the durability of the asphalt pavement. The more important of these factors include: type and gradation of aggregate, aggregate degradation, aggregate-water adhesion (particularly in the presence of water),

and air voids and film thickness in the mixture. Degradation of aggregate in an asphalt paving mixture depends on the kind of aggregate, gradation, compactive effort and particle shape, and can be evaluated by the Los Angeles Abrasion Test[27]. With respect to adhesion of asphalt to aggregate, the following general statements can be made:

1. Adhesion of asphalt to aggregate, especially in the presence of water, is a surface phenomenon involving physico-chemical forces acting in the system. The problem, if one occurs, is more a result of the chemical or mineral composition of the aggregate than the asphalt binder.

2. Adhesion can be improved and stripping can be eliminated or reduced by certain fillers (such as hydrated lime and Portland cement) and other anit-stripping additives. The most effective of these show cationic surface activity.

3. Most available laboratory tests to predict or evaluate aggregate-asphalt stripping or adhesion are arbitrary and have, at best, only qualitative value. A quantitative, objective and definitive method is needed.

Air voids can be considered as a measure of permeability and pore surface area. Film thickness can be expressed either in terms of calculated average thickness of the asphalt film or in terms of bitumen index which is the ratio between asphalt content and specific area of the aggregate (sq ft/lb of aggregate)[28]. Both of these variables are affected by asphalt content and aggregate gradation. Asphalt or binder content is considered the most important factor in deciding durability of a paving mixture. The effects of binder content (as reflected in terms of air voids and film

thickness) on hardening of asphalt as indicated by retained penetration (percent original penetration) are shown in Figs. $1 - 5$[10,11,23,28]. The effects of initial void content on the change of penetration of asphalt in a test section at the Shell Oil Company's Wood River refinery (Illinois) are shown in Fig. 6[29]. These data emphasize the importance of obtaining low void content in the in-place asphalt concrete surfacing. The general conclusions of all these findings[28] can be stated as:

1.   Increasing air content results in a reduction of the retained penetration as a function of aggregate gradation.

2.   Increasing film thickness results in an increase in the retained penetration as a function of the aggregate gradation.

It is the consensus of opinion among asphalt paving technologists, both in the U. S. and in Europe, that a longer life is associated with higher asphalt content and increased density, and that it is a sound practice in bituminous paving mixture design to use as high a binder content as possible without losing stability. It is both significant and interesting to note that opinions differ as to the grade or hardness of binder to be used in the mixture. The American practice is to use the softest grade binder possible on the premise it would take longer for such a system to reach the critical level of hardness often indicated as a penetration of 20 or viscosity of 10^8 poises at $77^\circ F$[30]. The United Kingdom emphasis, on the other hand, is to use the hardest possible grade of binder to get the highest possible binder content to maintain desirable level of stability.

The American practice of using the highest possible penetration grade binder was recently questioned[31] based on four major considerations:

1. Using a high penetration asphalt initially does not always assure a high penetration after mixing and in service.

2. Higher asphalt consistency may be needed to obtain increased film thickness or asphalt content.

3. Based on limited fatigue test data[32,33,34], asphalts of low penetration may provide better fatigue resistance, especially when used in thick asphaltic surfacings.

4. Resistance to the effects of water may be increased by using lower penetration asphalts[35].

DURABILITY TESTS FOR ASPHALTS

Durability of asphalts has been studied by many investigators for many years to find:

1. Mechanisms or causes of asphalt deterioration.

2. Methods for controlling or preventing undue hardening of asphalts or improving of the durability of asphalts.

3. Tests to predict the behavior and durability of an asphalt during mixing, laying, and in pavement service.

Studies in the first group can be considered as basic research. Studies directed to the second and third objects can be classified as applicational research. It is obvious that they are interrelated: without clear understanding of the chemical makeup of the asphalt and the mechanism of failure of asphalt by disintegration and cracking, it is difficult to devise durability improvements and to choose the properties of the asphalt for investigation in durability tests and as indices for durability. On the other hand, a reliable durability test is needed when

evaluating the effectiveness of methods for durability improvement in the second category.

The importance of heat stability or resistance of paving asphalt was recognized early in the history of asphalt paving technology. It was reported that, in 1897, Allen Dow suggested two methods for determining asphalt hardening when heated to high temperatures[36]. One of the methods involved heating 20g of asphalt in a 2-oz glass retort at 400°F for 30 Hr and determining weight and penetration loss. Specifications based on this test required a maximum loss on heating of 8% and minimum retained penetration of 75%. This was probably the original loss on heating test. Prior to 1911, there were a number of other heat tests proposed; all were used to determine weight loss and penetration drop. At that time, a test was made on practically all bitumens except tars and was also occasionally made at 400°F for 5 hr. In 1911, ASTM issued a method for the determination of the loss on heating of oil and asphaltic compounds (ASTM D6-11). A 20g sample was placed in a flat bottom tin, 6 cm in diameter, and heated for 5 hr at 325°F. In 1916, the loss on heating test (ASTM D6-11) was revised. The size of the sample was increased to 50g and the 3-oz tin in use today was specified, making a film of 13/16 in. The present ASTM designation for this test is D6-67. The basic conditions of the test ASTM D6-16 are still the same.

Many attempts were made to explain the field behavior of asphalts in terms of results obtained from the standard loss on heating tests. Anderson et al.[37] developed a "resistance to hardening" value based on the penetration of residues of the standard loss on heating test after 5 and 10 hr. The "resistance to hardening" value equals

$$5 \ \frac{Y_1 - 1}{Y_1 - Y_2} + 5$$

in which Y_1 = log penetration after 5 hr at $325^{\circ}F$, and

Y_2 = log penetration after 10 hr at $325^{\circ}F$.

They set a resistance to hardening value of 55 as the dividing line between satisfactory and unsatisfactory materials. Resistance value could also be interpreted as the hours it would take to harden the asphalt in the standard loss on heating test at $325^{\circ}F$ to a penetration of 10.

Clark studied the volatility of asphalts by the standard loss on heating test[12,13]. He concluded that the hardening of asphalt during mixing, as measured by loss in penetration, was proportional to the volatility of the asphalts as measured by the standard loss on heating.

Nevertheless, the value of the standard loss on heating test in predicting the hardening of asphalt was questioned by many technologists. It is now generally agreed that, because of the depth of the sample in a standard loss on heating test, the conditions are not sufficiently severe to evaluate and differentiate the hardening properties of asphalts. With the advent of vacuum distilled asphalts, the value of the test in characterizing the early steam-refined petroleum asphalts was also lost.

Realizing the inadequacy of the standard loss on heating test in providing more information on the quality of asphalts, the U. S. Bureau of Public Roads undertook a series of investigations on oven heating tests of asphalt in films thinner than the 7/8-in. layer of the old loss test. This work was first reported by Lewis and Welborn[36]. This report represented the results of tests on eighty 50-60 and 85-100 pen. asphalts made on the residues from the standard loss on heating tests as well as residues from the Thin Film Oven Tests (TFOT) of 1/8 in. films. The properties of eight residues from the TFOT were compared to those of the same asphalts recovered from Ottawa sand and sheet asphalt mixtures used in the Shattuck mixing

tests[38]. In addition, test sections of sheet asphalt were constructed

using one of the 50-60 pen. asphalts. Samples were taken immediately after

the hot mix was laid and compacted. Penetration, ductility, and softening

point of the recovered asphalt were compared with those of the residue

from the TFOT on the original asphalt. The most significant conclusions

from this study were:

1. The standard loss on heating test does not furnish adequate

information concerning the probable behavior of asphalts for

use in hot-mix paving.

2. The changes that occur during the TFOT for 5 hr at $325^{\circ}F$ with

1/8-in. films in asphalts of the 50-60 grade are comparable to

the changes that may be expected in bitumen recovered from

mixtures prepared in paving plants.

3. The ability of asphalts to retain their original characteristics

after the 5-hr, 1/8-in. film oven tests offers a means of

evaluating their relative durability.

A second paper in 1946 by Lewis and Halstead reported the results of

TFOT on asphalts of 60-70, 100-120, and 120-150 penetration grades[39].

The data in this report generally supported the earlier findings for the

50-60 and 85-100 grades. Requirements of percent weight loss, retained

ductility and penetration on the Thin Film Oven Test (1/8-in. film

heated 5 hr at $325^{\circ}F$) for all grades of asphalts were suggested.

The TFO test has been adopted as an ASTM Tentative Standard (D 1764)

since 1960 and is being considered in Committee D-4 of the ASTM to advance

as standard. This test has replaced the Loss on Heating Test in the

specifications of the majority of agencies in the U. S. and is also required

in the German Standard DIN 1995 for road bitumens. While the value of
the TFO test in predicting long-term service durability of asphalt in
pavements is inconclusive, the ability of the test in duplicating the
hardening in asphalt during hot mixing and in qualitatively indicating
the ability of an asphalt to retain its original consistency and ductility
has made the TFO test one of a few tests that exists in present day
asphalt specifications that may be considered quality test or durability
test.

Way et al.[40] correlated the TFOT with the limiting penetration. The
limiting penetrations were calculated from field data, considering the
hardening of bitumen in the field is a hyperbolic function of time and
the penetration at infinite time as the limiting penetration. Figure 7
is a plot of limiting penetration against penetration of the TFOT residue
where the percentages of the original penetration are used for both ordinates.

The Asphalt Institute is studying the use of viscosity ratio at 140^OF
on asphalt sample after the TFO test and before TFO test as durability
requirements in its proposed specifications for asphalt cement. The
ratio is set at the maximum of 5.

Many investigators believe that oxidation is a major factor causing
asphalt hardening. Procedures for evaluating the susceptibility of asphalt
to oxidation were developed by Thurston and Knowles[41], Anderson, Stross,
and Ellings[42], and Ebberts[43].

Thurston and Knowles, in a series of pioneering works, subjected
asphalts and asphalt-sand mixtures to oxygen in a closed system at tem-
peratures from 170^OF to 400^OF and the oxygen absorbed was measured.
Their data showed that: (a) all of the constituents normally present
in an asphalt were susceptible to photo-oxidation, (b) there were

differences among asphalts of different sources with respect to the resistance to the action, and (c) an asphalt surface should be protected from exposure to sunlight to increase durability.

Anderson et al. developed two procedures to measure the oxidation stability of asphalts and to allow forecast of their road behavior. The first method, a rapid one for routine testing, uses data obtained by the standard loss on heating test, except penetration of the asphalt is determined both after 5 hr and after 10 hr in a standard oven. The logarithms of the penetrations are plotted against time in hours in the oven. A straight line is drawn through the points and extrapolated to 10 penetration. The time required for the penetration to drop to 10 is termed the resistance to hardening. They reported a fair correlation between resistance to hardening and service ratings according to road performance for 15 asphalts. In their second method, the asphalt to be tested is dissolved in benzene and then oxidized in an oxygen bomb at 108 psig at 50°C for 40 hr. The pressure in the bomb is recorded. At the end of the run the asphalt is recovered from the solution and penetration determined. The penetration and pressure drop data and "deterioration index" are calculated. Correlation between service rating and deterioration index showed that while an index lower than 15 indicated fully satisfactory performance, poor durability can definitely be expected with an index above 20.

A simple procedure was proposed by Ebberts for measuring oxidation of asphalts in thin films. In his method a thin film of asphalt of known weight is heated to 140°F with a sulfuric acid solution of 0.1 N potassium permangenate. The milliliters of permangenate consumed in

oxidizing the asphalt are determined and plotted against the time. A high permangenate consumption indicated a high oxidation susceptibility and thus lack of durability.

One of the most significant achievements of progress in asphalt technology, especially in asphalt durability, was the development of a microviscometer by van Oort[6]. He observed that changes in asphalts due to atmospheric exposure were restricted to a surface layer a few microns thick. It was recommended that very thin films of asphalt (5 to 10μ thick) be used in evaluating hardening effects.

Since the introduction of the microviscometer, many investigators have used so-called microfilm durability techniques in which asphalt is aged in films of only 5 to 15μ. Hardening is measured by the viscosity ratio or aging index after the film is exposed to heat and air.

The first of these was proposed by Griffin et al.[11]. In this method a 5μ film of asphalt is aged on glass in an oven for 2 hr at 225°F. The hardening which occurs is determined by measuring the viscosity of the asphalt before and after aging both at the same temperature of 77°F and the same rate of shear of $5 \times 10^{-2} \text{sec}^{-1}$. The durability is reported as an aging index which is the ratio of the viscosity of the aged sample to that of the original. A low aging index signifies a more durable asphalt. Heithaus and Johnson[32] have shown that the results by this microfilm durability test correlated well with relative performance of the asphalts in test roads (Figure 8). Similar correlations were also revealed from data collected in the important Zaca-Wigmore Test Road in California[44]. This test has been published by the ASTM as a proposed method to predict asphalt durability.

Essentially the same test was used by Traxler[45] to study the hardening of asphalts used by the Texas Highway Department. Time of aging was 2 hr and the temperature used was $225^{\circ}F$. However, a film of 15μ was used in aging instead of the 5μ films proposed by ASTM. This was done because the thicker film more nearly approximated the geometry of the films present in most bituminous pavements. As a result of this study, the Texas Highway Department has included an aging index (on 15μ films) requirment of less than 4-6 in its asphalt cement specifications.

Hveem et al.[46] and Skog[47] presented results of an extensive study on the durability of asphalt by the Shot-Abrasion test and the microviscometer technique. Weathering was achieved by subjecting 2% asphalt Ottawa sand mixtures to infrared radiation in a weathering oven at $140^{\circ}F$. A correlation study showed that exposure of 1000 hr in their weathering machine was about equal to 5 yr of pavement service time for California conditions. For routine control testing purposes, a new Rolling Thin Film Oven (RFTO) test (film thickness of 5 to 10μ, exposed in oven at $325^{\circ}F$ for 75 min) was developed to predict change in asphalt during mixing operation. To simulate weathering during service life, a durability test was developed. In this test the 20μ films of residue from the RTFO tests are weathered at $210^{\circ}F$ for 24 hr and viscosity is determined by a microviscometer. Exposing asphalts first to an RTF exposure ($325^{\circ}F$, 75 min), followed by thin film durability exposure (20μ, $210^{\circ}F$, 2 hr), is intended to simulate 5 yr of field service hardening under California weathering conditions. These conditions produced hardening equivalent to that of 1000 hr in the weathering machine at $140^{\circ}F$ or 5 yr of service life.

Correlation between the California RFTO test and Thin Film Oven (TFO) test was found good[44,46-48] and the RFTO test is under consideration in the ASTM Committee D-4 for adoption as ASTM standard. The durability test requirements in terms of viscosity and ductility at 77°F on residue after durability test are included in California's newly proposed specification for 85-100 pen. paving asphalts. The maximum viscosity at 77°F and at a shear rate of 0.05 sec^{-1} was set at 25 megapoises and the minimum microductility (0.5 cm/min) was set at 10 mm[47].

Also based on microfilm viscometer technique, Santucci and Schmidt[49] and Schmidt and Santucci[50] recently proposed two durability tests using mixture fatigue resistance as the criterion and viscosity as the parameter: the Rolling Microfilm (RMF) and the Rolling Microfilm on Original Asphalt (RMFO) tests. In the RMF proceudre, ½-g of residue from RTF test is dissolved in 10 ml of benzene and filtered into a regular RTF bottle. After the bottle rotation is started and benzene is evaporated, a 20μ film of asphalt is deposited in the walls of the bottle. Microviscosity is determined on the residue after 24 hr exposure at 210°F. The RMFO procedure is identical to the RMF procedure except that the original asphalt is used instead of the residue from the RTF test. All viscosity determinations are made at 77°F and at a constant stress of 167g/sq cm. Their data indicated that both tests correlated equally well with the pavement life of the Zaca-Wigmore test project. Since the RMFO test is the most convenient, economical and repeatable of all microfilm durability tests, they recommended that the RMFO constant stress viscosity be used to predict paving asphalt durability and mixture service life. Mixture service life in the Zaca-Wigmore test road as predicted by the California

durability test (also termed Thin Film Plate Durability Test), RMF test, is shown in Figures 9, 10, and 11[49].

An accelerated test procedure for assessing the aging characteristics of bitumens was investigated by Blokker and van Hoorn[7]. The procedure involved treating bitumen in thin films (5 to 200μ) in oxygen of 20 atm at 50°C (122°F) and measuring relative viscosity. The same approach was used by Martin[51]. The British Road Research Laboratory[38] has developed a pressure-oxidation test for road tars by exposing tar films 7 mm thick to oxygen at 300 psi and 65°C (149°F) for 64 hr and measuring the change in either Fraass brittle point or equiviscous temperature (e.v.t.).

At Iowa State University, the author[52] has developed a durability test to simulate realistically the two-stage hardening of paving asphalt during mixing and subsequent pavement service life. The test uses the well-established BPR Thin Film Oven Test and consists of first subjecting the asphalt to TFOT and then treating the residue from the TFOT in oxygen at high pressures. The TFOT at 325°F is used to simulate the changes that may occur in asphalt during mixing and the pressure oxidation process at 150°F to simulate the changes that may occur in asphalt during pavement service life.

The more important premises and concepts behind this durability test are:

1. The most important single factor that causes asphalt pavement to crack and disintegrate to the failure of the asphalt as a cementing and waterproofing agent in an asphalt mixture due to asphalt hardening. Therefore the extent and rate of asphalt hardening is considered indicative of the relative durability of asphalt.

2. Hardening and other pertinent changes that may occur in asphalt in an asphaltic concrete mix occur in two stages under two entirely different environments or conditions: hardening during short periods of time in the mixer at higher temperatures and higher rates, and hardening during longer periods of time of road service in pavement at relatively lower temperatures and lower rates. The hardening mechanisms and effects in these two stages are believed to be quite different.

3. Any realistic durability test for asphalt should include consideration of the two stages of hardening processes of asphalt in their logical order and their differences in mechanisms and effects.

4. In evaluating the quality or durability of asphalts, one is concerned with not only the characteristics of the original asphalt, but, even more important, the binder characteristics — the viscosity, the tendency to harden, the susceptibility to oxidation, the colloidal stability, etc. — of the asphalt in the finished pavement. This would include a study of the asphalt after the first stage hardening to assure that the properties of the binder following construction are satisfactory for future pavement performance.

5. Hardening during the mixing process may be simulated and predicted in the laboratory by the TFOT. Additional hardening and other changes in the asphalt in service may be simulated by laboratory pressure-oxidation tests at road service temperature on residue of the TFOT.

This program is in its second year of field correlation. It is expected that, once correlation is established, this test will provide a more realistic basis for quality control and durability prediction. Viscosity changes of two of the asphalts during the test are shown in Figure 12.

In recent years considerable efforts have been made to study asphalt on the basis of its chemical composition. This is a more fundamental approach to understanding asphalt and its rheological and weathering properties because it is believed that durability, adhesion and rheological properties should all be definable by its chemical makeup.

Rostler and White[53] have published information showing the relationship between asphalt durability and chemical composition as determined by methods which they developed. In their investigations durability was measured using a pellet abrasion test which was designed to correlate with California's shot abrasion test. This test evaluates the ability of asphalt to cement together coated particles of Ottawa sand. Chemical composition is expressed as the ratio of the reactive (nitrogen bases, N and first acidaffins, A_1) to the less reactive chemical compounds (second acidaffins, A_2 and paraffins, P) in the asphalt, i.e., $(N + A_1)/(A_2 + P)$. It was shown that asphalt durability varied inversely with this ratio. Using this ratio, they divided 85-100 pen. asphalts syntheses through blending of fractions, asphalt durability is independent of crude origin.

Halstead et al.[54] presented additional information relative to chemical composition and durability with a wide range of asphalts. Their efforts suggest the use of (a) durability grouping of asphalts by chemical composition and (b) asphalt viscosity as an additional parameter to assist in predicting the cementing properties of asphalt.

Based on extensive studies at the U. S. Bureau of Standards, Greenfeld and Wright[55] suggested the following four rapid methods to predict accelerated weathering durability for roofing asphalts: (a) asphaltene content — durability increases as asphaltene content decreases, (b) rate of filtration — durability increases as filtering time increases, (c) solubility parameter — durability increases as solubility decreases, and (d) carbonyl index — durability decreases as carbonyl index increases. The carbonyl index is determined by using infrared spectroscopy to measure changes in carbonyl-band absorption of asphalt.

Though somewhat crude and inconclusive, the Oliensis spot test has been suggested by some as a chemical approach for predicting asphalt durability[56,57,58].

EVALUATION OF DURABILITY TESTS

The research on durability tests for asphalt described in the preceding section is by no means a complete review of literature on durability. It represents, in the author's opinion, a cross section of the more important work on the subject. While it is difficult and too early to draw conclusions as to which of these durability tests is superior in its ability to identify poor quality nondurable asphalts and to predict the useful life of asphalts in pavements, it is believed that the following three criteria should be considered in developing or evaluating a durability test for paving asphalts: (a) realistic aging or weathering conditions, (b) relevant parameters in describing durability and performance, and (c) field tests.

Aging Conditions

For a durability test to be functional, that is, to be able to identify nondurable paving asphalts and to predict reliably the influence of asphalt properties on pavement life or the useful life of wide variety of asphalts in pavements, the asphalt must first be exposed to conditions which quickly simulate the changes taking place in asphalt in a typical pavement over a longer period of time.

The selection of realistic conditions of aging for paving asphalts presents little problem as far as simulation of the hot-mix operation is concerned. Both TFO and RTF tests have proved to result in hardening and other changes near those occuring in actual normal application. Simulation of long-term aging in a procedure short enough in duration for investigation or quality control purposes poses more serious problems. Increase of temperatures has been widely used. However, whether the mechanisms of chemical changes at different temperatures are the same is still debatable. Nevertheless, it seems to the author that California's microfilm plate (MFP) test on RTF test residue and the pressure-oxygen treatment on TFO test residue[52] are by far the most realistic and logical.

Durability Parameters

The selection and measurement of an appropriate property of asphalt before and after aging is a key factor in durability studies. In selecting this parameter(s), consideration should be given to how is durability defined, what are the criteria for pavement performance, what are the mechanisms of pavement failure, how do various factors contribute to pavement deterioration, and what are the critical properties that will define failure? The durability test can be of true value only when the

tests or properties measured can indicate the actual changes that cause asphalt deterioration. Most investigations have generally attempted to relate some type of consistency results to performance, on the premise that (a) the characteristic deficiencies of nondurable asphalt are disintegration and cracking (or fracture), and (b) the most important single factor that affects both disintegration and cracking is the asphalt's resistance to hardening. It is agreed that consistency properties of asphalt govern the durability of asphalt; yet, there is still considerable argument as to which measure of consistency correlates best with road performance. Ideally, since asphalt mechanical properties depend both on loading time and temperature, it is desired to obtain a complete picture of rheological behavior of asphalt over the entire range of loading time and temperature to which the pavement is exposed. While this is not possible, usually only a few critical measurements at critical conditions are made. The most used parameters have been penetration, ductility, viscosity and softening. Less used, but possibly more important and relevant parameters are: brittle point[59,60,61], stiffness[62], and complex or dynamic modulus[1].

Not to be ignored is the more basic approach using chemical composition. This may be either in terms of conventional asphaltene, resin and oil content, or $(N + A_1)/(A_2 + P)$, suggested by Rostler and White[53] or C/H ratio concept as suggested by Barth[63]. It is the author's opinion that both the rheological parameter and chemical parameter should be included in durability tests and durability studies.

Field Correlations

An additional requirement for a good asphalt durability test is
that the aged material should be tested by a method giving test values
related to field performance and that laboratory durability test results
can be correlated with field service behavior. Only when such correlations
exist can the durability test be used to predict field performance of
asphalt, to identify nondurable asphalts, and to specify asphalts
according to quality and durability.

CONCLUSIONS

Based on the discussions presented in the preceding sections, the
following general conclusions appear warranted:

1. All bituminous paving materials are affected by heat, weather,
   traffic and time. The combined effects result in a hardening
   of the asphalt and less desirable adhesion and rheological
   characteristics. The long-term ability of an asphalt to resist
   these changes due to the heat oxidation and other environmental
   factors is usually defined as its durability.

2. It is generally agreed that the most important single factor
   causing asphalt to crack and disintegrate is asphalt hardening.
   Therefore, the degree and rate of hardening of asphalt under
   specific weathering and traffic conditions indicate the
   relative durability of the asphalt.

3. Investigations have shown that asphalt produced from different
   crude sources or produced by different methods of production
   can have various degrees of durability under equivalent conditions

of construction, traffic and climate. Investigations have also
demonstrated that the present specifications for paving asphalts
do not exclude the possibility of the use of poor durability asphalts.

4. In spite of the tremendous research work made to date on asphalt
durability, the paving industry still needs test methods and
specifications to assure the exclusion of nondurable asphalts
and a reasonable prediction of the behavior of asphalt in
pavement and the useful life of asphalts used. An adequate
durability test is especially needed when an entirely new source
of asphalt is to be used and evaluated.

5. Special efforts should be made in the following areas as
related to the durability of paving asphalts: (a) chemical and
colloidal structure of asphalts and how they affect rheological,
adhesion and durability properties of asphalt; (b) chemical
mechanisms of aging at all phases of asphalt application;
(c) specific effects of environments such as film thickness,
temperature, oxygen exposure, pavement structure, etc.;
(d) improved techniques to define durability pertinent parameters
such as stiffness, complex modulus; and (e) guidelines for
evaluating the performance of asphalt in in-service pavements.

6. Selection and use of established durability tests should include
consideration of differences in local factors; these factors
include climatic and environmental conditions and design and
construction practices. For example, the California durability
test temperature, time and film thickness were selected to produce
aging equivalent to 5 yr of pavement service life for California

conditions. This aging equivalency factor may or may not be true for Iowa pavement conditions.

7. From the practical point of view, the following considerations should be given relative to durability[31]:

    a. Mixes should be designed to provide for a maximum asphalt content without instability.

    b. Mix designs should include minimum film thickness requirements.

    c. Mixes should be designed to have low permeability and low voids.

    d. Consideration should be given to the use of harder asphalts consistent with mixing and compaction requirements.

ACKNOWLEDGMENTS

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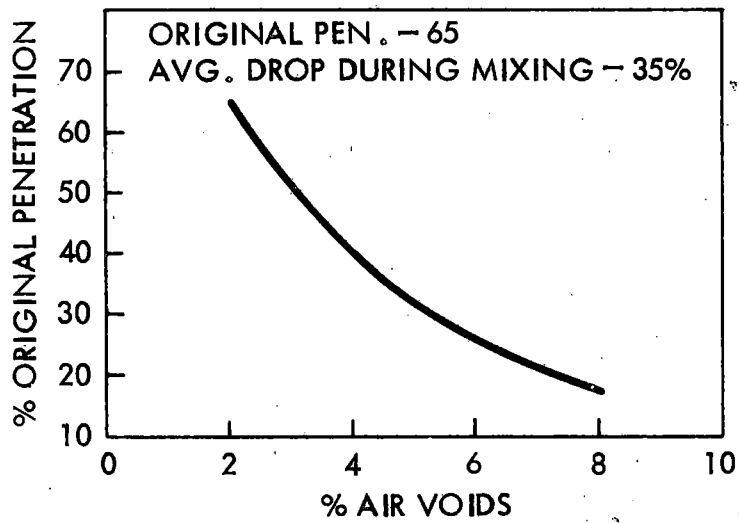
Fig. 1.   Voids vs Rate of Hardening for a Mexican Bitumen after 15 Yr of Service[23]
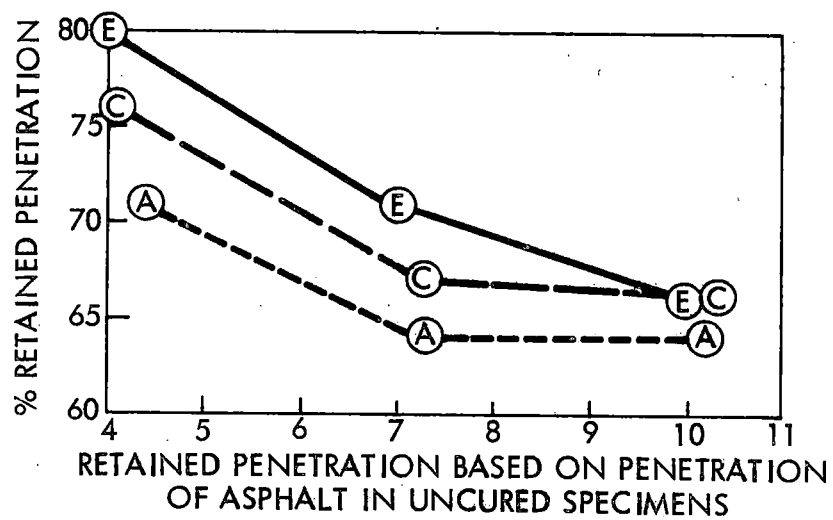
Fig. 2.   Effect of Air Voids on Degree of Asphalt Hardening after 12-Day Oven
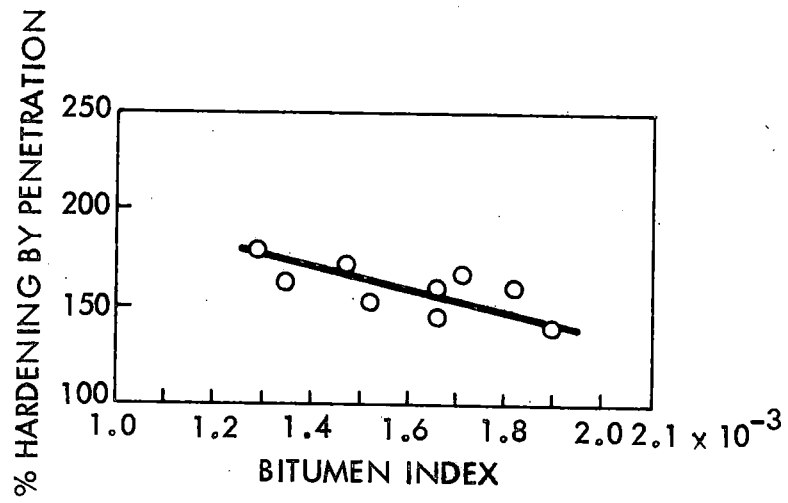
Curing at $140°F^{31}$

Fig. 3.   Percent Hardening by Penetration during Mixing vs Bitumen Index[10]
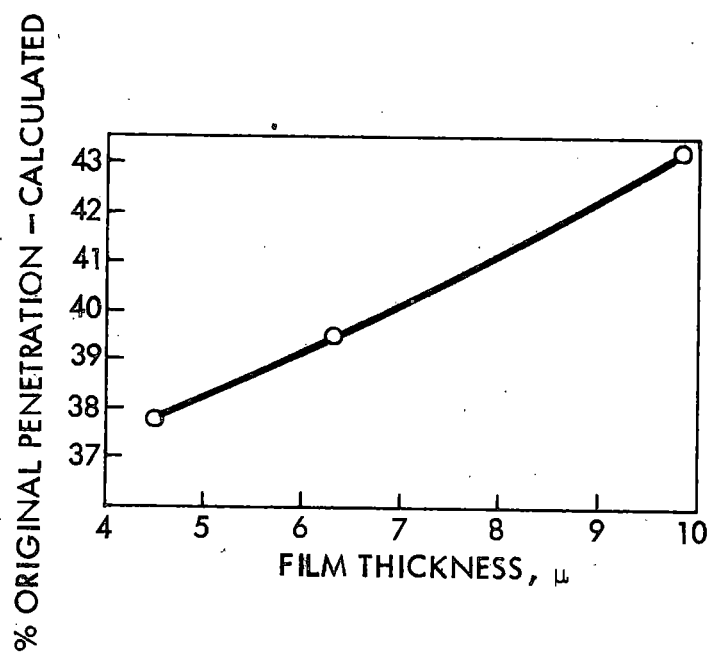
Fig. 4.   Consistency Change vs Film Thickness[11]
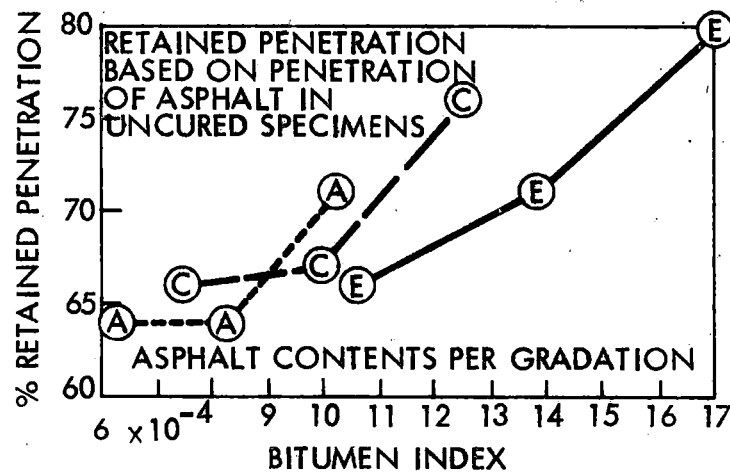
Fig. 5.   Effect of Bitumen Index on Degree of Asphalt Hardening after 12-Day
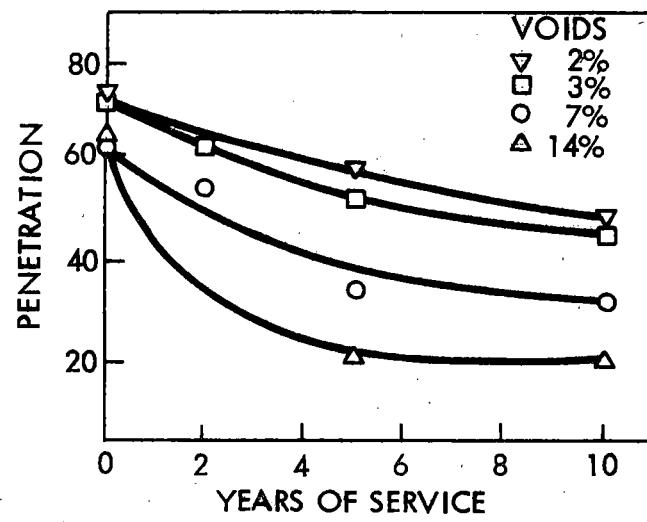
Oven Curing at 140°F[31]
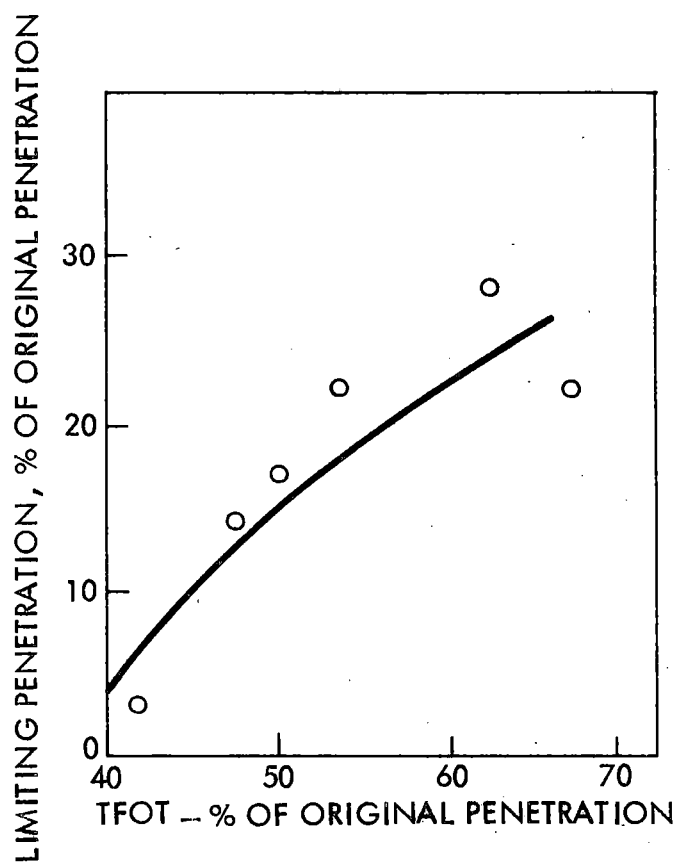
Fig. 6.   Asphalt Hardening in Several Midwestern Pavements[29]

Fig. 7.   Limiting Penetration vs Hardening during Thin Film Oven Test[40]
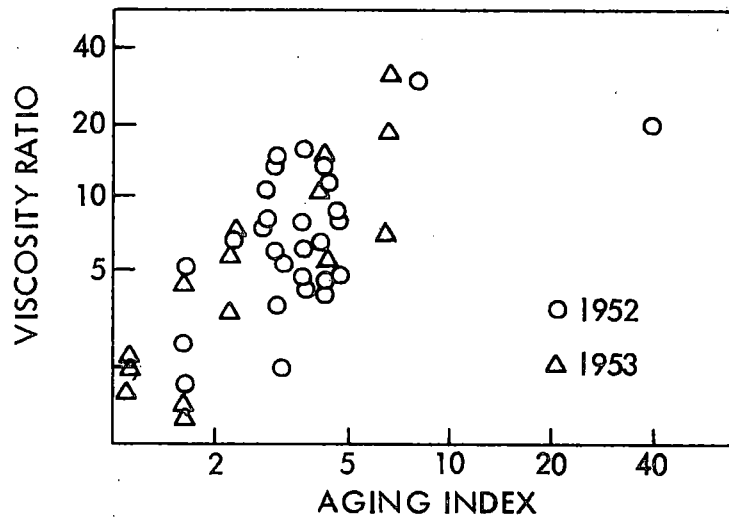
Fig. 8.   Hardening during 36 months' Service (Viscosity Ratio) vs Aging
           Index[29]
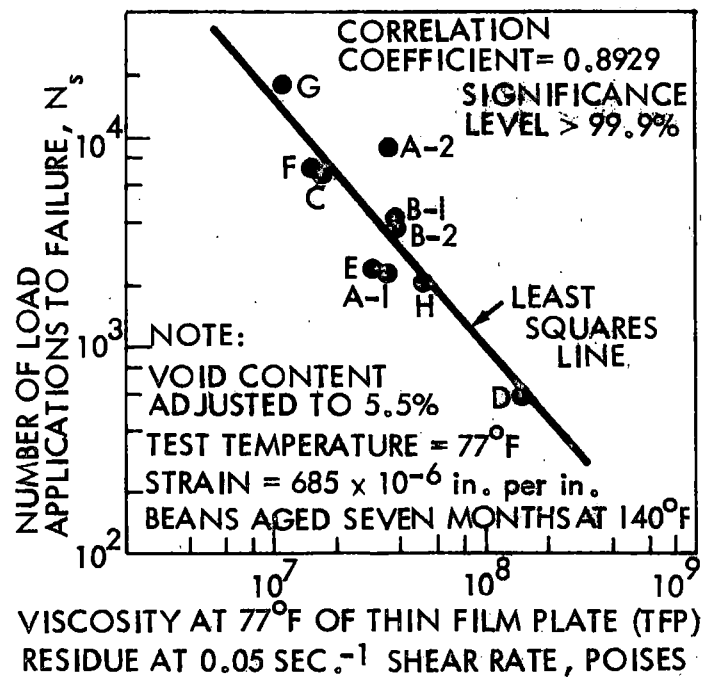
Fig. 9.   Mixture Service Life as Predicted by Constant Shear Rate Viscosity of
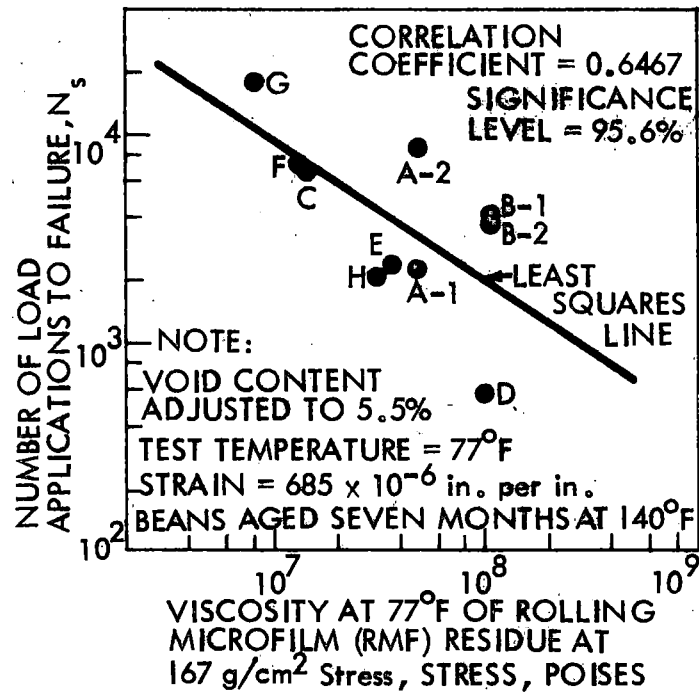
Thin Film Plate (TFP) Residue[49]

Fig. 10.   Mixture Service Life as Predicted by Constant Stress Viscosity

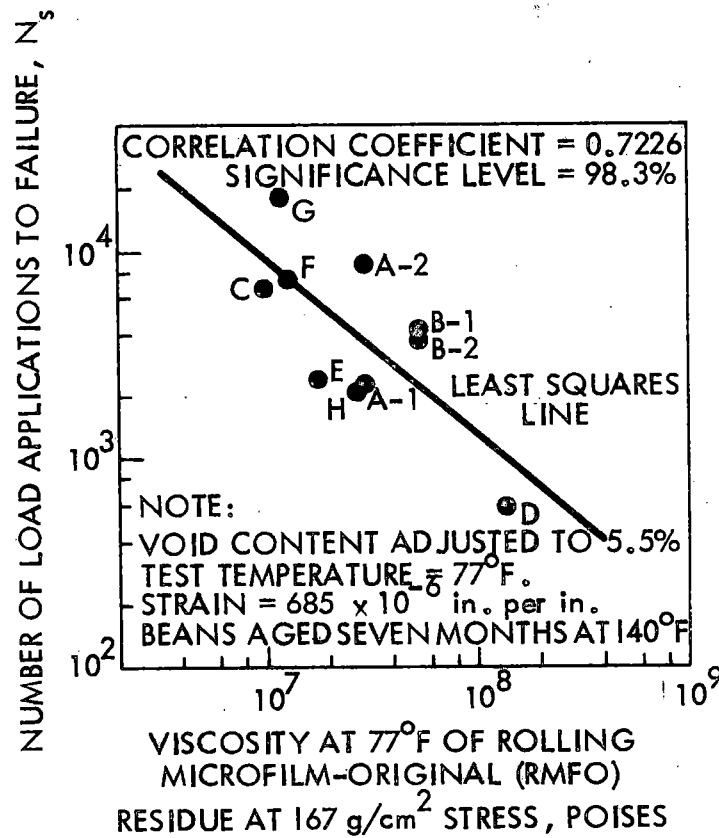of Rolling Microfilm (RMF) Residue[49]

Fig. 11.   Mixture Service Life as Predicted by Constant Stress Viscosity of
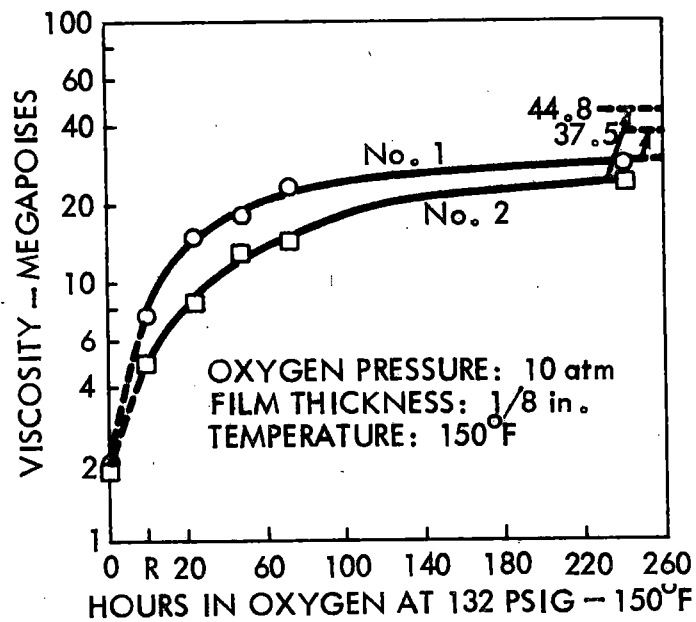
Rolling Microfilm-Original (RMFO) Residue[49]

Fig. 12.   Viscosity vs Time of Aging in Pressure Bomb[52]