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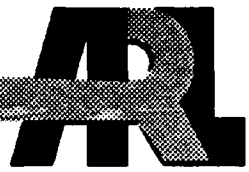

Simulant Breakthrough Time With Laminates of Unlike Rubbers

Anthony F. Wilde and Stephen C. Bodnar

ARL-TR-510

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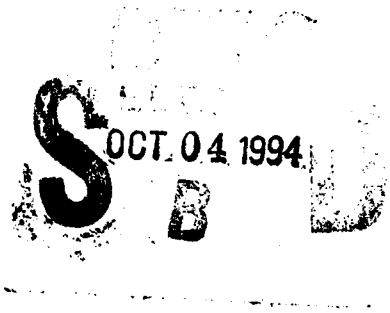

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For chemical protective application, laminates of unlike rubbers may constitute structures with properties that are unavailable from either material alone. Laminates of Butyl/Silicone and of Nitrile/Butyl were obtained by contract from a commercial firm. With 2-chloroethyl ethyl sulfide (CEES) as the permeant, the U.S. Army Research Laboratory, Materials Directorate (ARL-MD) determined the breakthrough times (t_b) of these laminates and of the individual rubber materials by means of the MIL-STD-282, Method 204, permeation test. To interpret the t_b of the laminates in terms of t_b of the single rubbers, a lag time model published in the literature was adopted. The resultant predicted values of t_b for the laminates agreed fairly well with the experimental results obtained for these layered structures.

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Introduction

Chemical agent barriers that are incorporated into, or serve as components of, clothing items must be lightweight and flexible. The so-called impermeable types of these barriers are, therefore, usually based upon rubber materials. However, additional properties are also desirable. For example, silicone rubber, being very flexible, provides good comfort and fit as a face piece material for a gas mask. However, silicone has poor barrier properties. Lamination of a less permeable rubber to silicone might provide a structure with the necessary combination of impermeability and flexibility. Another example is given by butyl rubber which is a good barrier material. However, it tends to sorb and to be swelled by hydrocarbon based liquids such as lubricants, fuels, and solvents thereby losing mechanical and barrier integrity. Lamination of an oil-resistant rubber to butyl might provide a structure with a useful combination of impermeability and oil resistance.

The purpose of this preliminary effort has been to investigate the simulant permeation breakthrough times (t_b) of some experimental laminates of unlike rubbers. However, the permeation t_b of a laminate is not a simple additive function of t_b of the individual layers. For example, in the simplest case of a given material in several thicknesses or layers, Fickian diffusion implies that t_b should scale as something like the square of the total thickness. Hence, t_b is not simply the sum of t_b of the individual layers. This nonadditivity is a result of the fact that the operative concentration gradients of the permeant in the barrier become progressively smaller as the barrier thickness increases. Obviously, this nonadditivity behavior would also be manifested in the case of permeation of a liquid through layers of unlike materials.

This paper will describe some introductory experiments involving simulant permeation through laminates of unlike rubbers. In addition, a scheme will be presented for rationalization of the laminate t_b results in terms of t_b values for the individual rubber materials.

Materials

By contract with the U.S. Army Research Laboratory, Materials Directorate (ARL-MD), Smithers Scientific Services, Inc. of Akron, OH fabricated two types of laminates of unlike rubbers. A number of specimens of each laminate was produced in a size of 6" x 6".

Butyl/Silicone

Semi-finished rubber sheets dressed with Chemlok 950 adhesive, pressed together and molded at 10,000 lbs for five minutes at 320°F, and has a typical thickness of 0.044 inch (44 mils).

Nitrile/Butyl

Semi-finished rubber sheets dressed with Chemlok 252 adhesive, pressed together and molded at 50,000 lbs for seven minutes at 320°F, and has a typical thickness of 43 mils.

To provide control specimens of the individual rubbers, Smithers also fabricated laminates of like rubbers. These were similarly furnished in a size of 6" x 6".

Butyl/Butyl

Semi-finished rubber sheets pressed together and molded at 50,000 lbs for 12 minutes at 320°F, and has a typical thickness of 33 mils.

Silicone/Silicone

Semi-finished rubber sheets dressed with Chemlok 607 adhesive, pressed together and molded at 50,000 lbs for eight minutes at 320°F, and has a typical thickness of 45 mils.

Nitrile/Nitrile

Semi-finished rubber sheets dressed with Chemlok 252 adhesive, pressed together and molded at 50,000 lbs for five minutes at 300°F, and has a typical thickness of 42 mils.

Consequently, even with the single materials there was a laminate construction usually including an adhesive. The control specimens thus contained an interface to correspond to that of the laminates of unlike rubbers so that a better determination could be made of the effect of unlike material combination upon the barrier behavior.

Permeation: Methods and Background

For this preliminary investigation, the permeation testing was confined to the relatively simple MIL-STD-282, Method 204 test. The arrangement is shown schematically in Figure 1. Filter paper (previously soaked in congo red dye and then dried) served as the permeation indicator. Before the test, a tetrachloroethane solution of indicator dye S238 was swabbed on the top surface of the paper; next was placed the rubber specimen to be tested. A drop of approximately 15 microliters of 2-chloroethyl ethyl sulfide (CEES) simulant was deposited upon the rubber specimen. This was followed by placement of an O-ring and glass cover to contain the CEES liquid/vapor challenge.

This sandwich assembly was located on a glass shelf (along with other test assemblies) in a clear plastic box serving as a controlled temperature diffusion chamber, as seen in Figure 2. Underneath the shelf, a 45° mirror permitted direct observation of the bottom surface of the test assemblies. Chamber temperature was maintained at 37°C.

At the first arrival of CEES at the bottom surface of the rubber specimen, corresponding to breakthrough, a reaction took place with the S238 to form an acid product. This immediately caused a localized color change from red to blue in the adjacent indicator paper, appearing as a blue spot that was visible by means of the mirror. The elapsed time (between deposition of the CEES droplet and appearance of the blue spot) constituted the value of t_b.

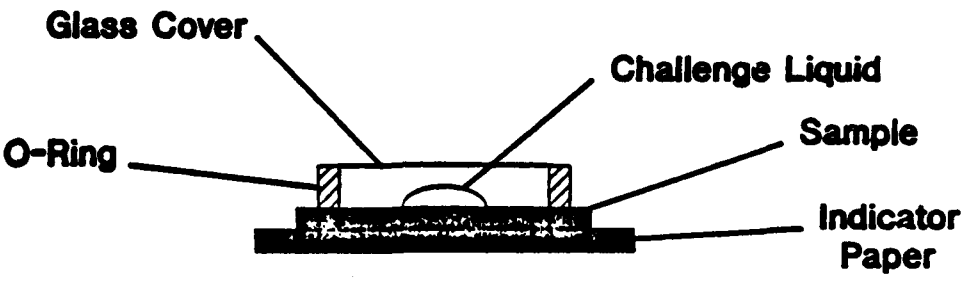

Figure 1. MIL-STD-282, Method 204, droplet permeation test.

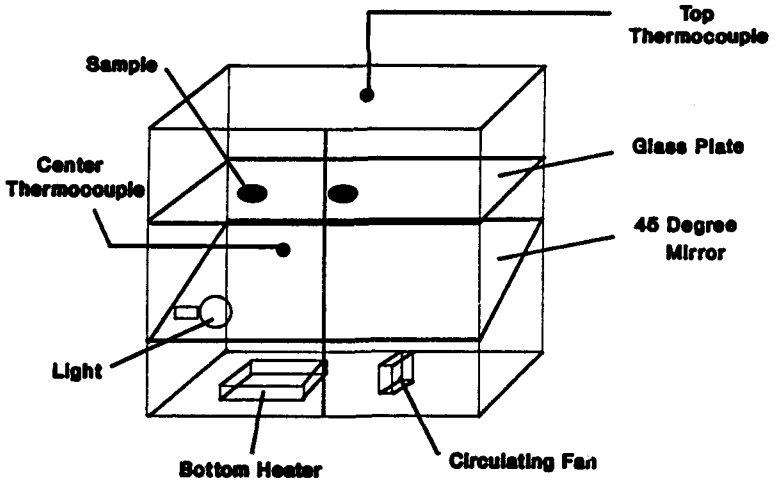

Figure 2. MIL-STD-282, Method 204, static diffusion chamber.

In summary, MIL-STD-282, Method 204 is simple to perform, is restricted to certain sulfur-containing permeants, and provides a breakthrough time, only. It does not indicate the amount of permeation, nor does it provide any information about permeation as a function of time after the occurrence of breakthrough.

For comparison, a more general description of a permeation test is shown in Figure 3. Here is plotted the total or cumulative permeation as a function of time for a well-behaved "generic" permeation test. A certain time is required for diffusion of enough permeant across the barrier to be observed by the detector; a time that is defined as t_b. After that event, both the total permeation and the rate of permeation increase with time. The permeation rate eventually reaches a steady-state value, as in the linear portion of the curve in Figure 3. Backward extrapolation of this portion to the time axis defines a characteristic time called the lag time, L.

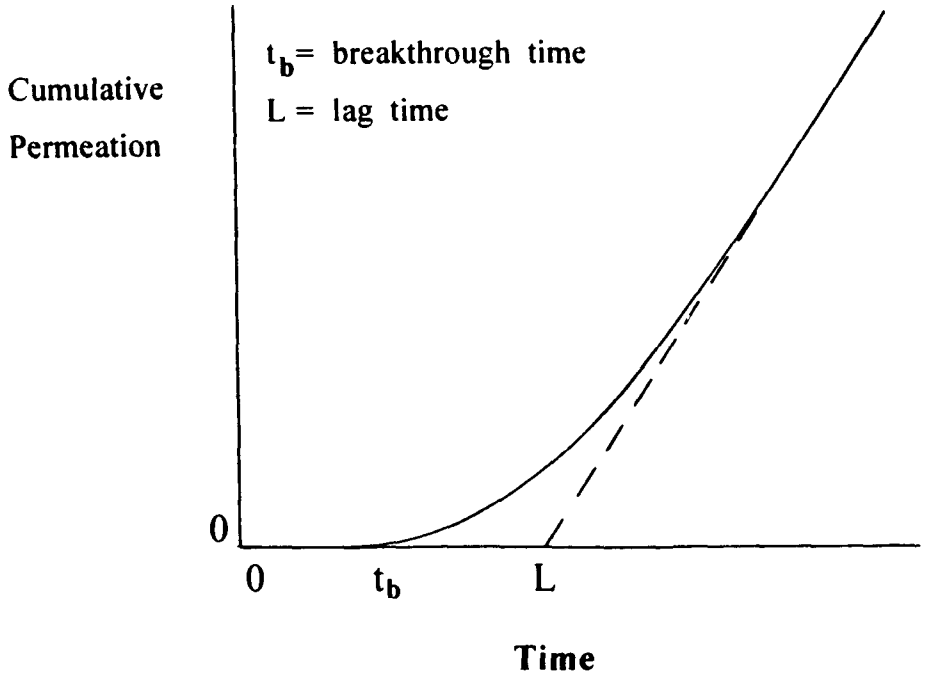

Figure 3. General description of permeation.

It is evident that the experimental breakthrough time, t_b, is difficult to define because the curve rises from the time axis in an asymptotic fashion. A very sensitive detector would lead to a lesser estimate of t_b than would a relatively insensitive detector. The experimental value of t_b is thus peculiar to the experimental procedure and to the sensitivity of the detector and, hence, is a somewhat ambiguous quantity.

Conversely, the lag time, L, is not obtained from an ambiguous quantity but is, instead, derived from a well-defined treatment of Fickian diffusion processes. L has little dependence upon the sensitivity of the detection method.

Theoretical treatments of laminate permeation in terms of t_b would be awkward and complex because the value of t_b would depend upon the definition of breakthrough. Theoretical treatments of laminate permeation in terms of L would be relatively straightforward because L, itself, is defined by well known mathematical descriptions of Fickian diffusion behavior.

A theoretical treatment of permeation through laminates of unlike materials is, therefore, more tractable when developed in terms of L. Selected examples have been summarized by Barrer[1], giving expressions for time lags for diffusion through laminates of various shapes (slabs, hollow cylinders, and spherical shells). For the current investigation, slabs are the appropriate shape. Expressions for these time lags are given in Figure 4 for both a single slab (A), as shown in Equation 1 and for two unlike slabs (AB), as shown in Equation 2.

Slab	Expression for L	
A	$\ell_A^2 \,/\, 6 D_A$	Eq. 1
AB	$\dfrac{\frac{\ell_A^2}{D_A}\left(\frac{\ell_A}{6 D_A} + \frac{k_1 \ell_B}{2 D_B}\right) + \frac{\ell_B^2}{D_B}\left(\frac{k_1 \ell_B}{6 D_B} + \frac{\ell_A}{2 D_A}\right)}{\frac{\ell_A}{D_A} + \frac{k_1 \ell_B}{D_B}}$	Eq. 2

where ℓ_A and ℓ_B are slab thicknesses

D_A and D_B are diffusion coefficients of permeant in each material

$k_1 = C_A \,/\, C_B$ is ratio of solubilities of permeant in each material

Figure 4. Time lags for diffusion through slabs.

Equation 1, for example, is well known for its use in estimating diffusion coefficients from lag time data of permeation experiments. Equation 2 is expressed in terms of five variables; i.e., the two slab thicknesses, the two diffusion coefficients, and k_l (the ratio of solubilities of the permeant in the two materials). In this model, it is assumed that the permeant (as it reaches the interface) will partition itself in the ratio of k_l in the the two materials at the interface, and maintain this ratio as the concentrations in each material increase to the steady-state permeation value. In the present investigation with CEES, k_l is not known because of the difficulties of determining the solubilities of this toxic simulant in materials. A means of dealing with this missing information will be addressed in a later section.

Empirical Approach

Evidently the theoretical expressions for laminate diffusion have been worked out in terms of L, whereas the ARL-MD permeation experimental results are in terms of t_b. Due to the lack of available simple expressions involving t_b, ARL-MD has simply adopted Equations 1 and 2 as a means of relating the various breakthrough times to each other. There is some justification for the approach based on considerations of consistency. First, all of these permeation experiments with the single rubber laminates and the unlike rubber laminates have been performed in the same way with the same permeant and the same detection method, thus providing a set of t_b values that should be systematically relatable to each other. Second, according to Figure 3, t_b should always be less than L. ARL-MD used the experimental t_b results from the single materials to estimate D_A and D_B by means of Equation 1, leading to an overestimate for these diffusion coefficients. Substitution of these exaggerated values of D_A and D_B into Equation 2 will lead to an underestimated time for L and, therefore (see Figure 3), will lead to a shift in direction toward the value of t_b for the laminate of unlike rubbers, thus tending to compensate for the initial interchange of t_b with L. In other words, the assumption has been made that the various values of t_b are related to each other in the same fashion as the various values of L are linked to each other. (In this treatment, ARL-MD will neglect the effects of this very thin (<1 mil) adhesive interlayer in order to reduce the computational difficulties associated with the addition of this third layer and its unknown values of thickness, diffusion coefficient, and interlayer solubility ratios.)

To restate the goal of this effort, it is desired to estimate t_b of laminates of unlike rubbers from experimental t_b values obtained with the single rubbers and then to compare the estimate to actual experimental t_b data for these laminates.

Results: Experimental and Predicted

Experimental results for the Butyl/Silicone system are shown in Table 1. For each of the single rubber laminates, values are listed for the average laminate thickness (ℓ), - average breakthrough time (t_b), number of replicate permeation tests, and the diffusion coefficient (D) estimated for CEES in each rubber by use of Equation 1. Despite its greater thickness, note the extremely short t_b characteristic of silicone, leading to its relatively large estimated value of D. Next, the experimental data for the Butyl/Silicone laminates are given. Values are listed for the average thickness of the individual layers and complete laminates, average breakthrough time (34.1 min), and number of replicate permeation tests.

Table 1. Butyl/Silicone system

Experimental Results

Single Rubber Laminate	Avg ℓ mil	Avg t_b min	Replicates	Est D cm^2/sec
Butyl	33.4	76.6	5	2.61×10^{-7}
Silicone	45.6	6.6	4	5.64×10^{-6}

Butyl / Silicone Laminate

Layer	Rubber				
A	Butyl	21			
B	Silicone	23			
Avg total ℓ		44	34.1	7	

Predicted Result for Butyl / Silicone Laminate

$$t_b = 92.5 \left(\frac{7.5 + k_1}{19.7 + k_1} \right) \text{ min}$$

Hypothetical value of k_1	0.2	0.5	1.0	2	5
Predicted t_b, min	35.8	36.6	38.0	40.5	46.8

The predicted value of t_b for this laminate is shown in Table 1, as calculated from Equation 2. Although k_1 is not known, it seems likely that the solubility of CEES in one rubber is not more than five times that in the other rubber (or, inversely, not less than 0.2 of that in the other rubber). Incorporation of a series of possible k_1 values into the expression provides a list of predicted t_b values. Although t_b does vary somewhat with hypothetical values of k_1, all of the t_b values (35.8 to 46.8) are reasonably close to the experimentally determined t_b. This level of agreement is gratifying.

Another example of laminate behavior is illustrated by the Nitrile/Butyl system, as summarized in Table 2. For each of the single rubber laminates, values are listed for average ℓ, t_b, number of replicates, and D as estimated by Equation 1. Also given are the experimental results for the Nitrile/Butyl laminates. Out of the 10 laminate tests, three gave very much longer t_b, for reasons not known. Discarding these anomalous results left seven tests with an average t_b of 88.8 minutes.

Table 2. Nitrile/Butyl system

Experimental Results

Single Rubber Laminate	Avg ℓ mil	Avg t_b min	Replicates	Est D cm^2/sec
Nitrile	41.9	48.5	6	6.48×10^{-7}
Butyl	33.4	76.6	5	2.61×10^{-7}

Nitrile / Butyl Laminate

Layer	Rubber				
A	Nitrile	26			
B	Butyl	17			
Avg total ℓ		43	106.7	10	(3 of these are much higher)
			88.8	7	(Remaining 7)

Predicted Result for Nitrile / Butyl Laminate

$$t_b = 75.9 \left(\frac{0.634 + k_1}{0.615 + k_1} \right) \text{ min}$$

Hypothetical value of k_1	0.2	0.5	1.0	2	5
Predicted t_b, min	77.7	77.2	76.8	76.5	76.2

The predicted t_b for this laminate is shown in Table 2, as calculated from Equation 2. Again, because k_1 is not known, a series of hypothetical values of k_1 was used to furnish estimates of t_b. Here, t_b is very insensitive to k_1, and all values (76.2 to 77.7) fall fairly close to the experimentally determined t_b. Again, this agreement is pleasing.

In summary, this mathematical expression of breakthrough results applied to the Butyl/Silicone laminates and the Nitrile/Butyl laminates suggests that use of Equations 1 and 2 may provide a viable method of predicting t_b values for laminates of unlike rubbers from t_b of the individual rubber materials.

Conclusions

In spite of the approximations inherent in this approach, the calculated or predicted breakthrough times of CEES through the laminates of unlike rubbers agree fairly well with experiment. Three kinds of approximations are involved here: First. Equations 1 and 2 implicitly assume that the diffusion processes are Fickian and that the diffusion coefficients can be considered as constant for each case, not significantly dependent upon concentration of the permeant. Second, despite the fact that Equations 1 and 2 were developed in terms of the lag time, the present treatment has adopted them to express the laminate behavior in terms of the breakthrough time. Third, the effects of the very thin adhesive interlayer are neglected.

Another conclusion is that both the experimental data and the predicted results confirm the fact that breakthrough times of laminates are not a simple additive function of the individual layers.

Suggested Future Efforts

Future efforts that should be considered are:

- By indirect methods, estimate solubility of CEES in these rubbers to specify the values of k_1.
- Test this laminate prediction method with other permeants and other laminate systems.
- Perform fully-wetted surface permeation experiments by quantitative instrumental methods. This will permit direct comparison of the use of Equations 1 and 2 in terms of both the lag time and the breakthrough time.

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