Proposed Performance Criteria for Encapsulant Coatings for Lead-Based Paint:
I. Abrasion Resistance

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ABSTRACT

This study was conducted to develop preliminary performance criteria for encapsulants for lead-based paint. ASTM standards have been developed for encapsulants and data in the study may support revisions to these standards. Encapsulants are liquid-coating-based products installed over the surface of the lead-based paint to help to minimize the hazards of lead-based paint in housing. This report, the first in a series on the performance criteria, describes the cure time and abrasion resistance of 10 commercial encapsulants. Six nonreinforced and four reinforced encapsulants were selected. Six were acrylic-resin based; others were epoxy-, polyester-, and polyurethane-resin based, and one was inorganic-cement-based with an acrylic binder. Two household paints were included as controls. Before conducting the abrasion tests, it was necessary to characterize the cure of the samples; that is, the change of the liquid-applied coating from a wet film to a solid film. Characterization of the cure was accomplished by following the glass transition temperature over time using dynamic mechanical analysis (DMA). The results indicated that seven encapsulants and the two paint samples showed little or no change in glass transition temperatures within four weeks or less after application; i.e., they were essentially fully cured. In contrast, three encapsulants displayed glass transition temperatures which were still increasing after at least 13 weeks, although the dry films exhibited no indications that they should not be tested after fours weeks.

The abrasion tests were conducted according to ASTM test method D 4060 for 2000 abrasion cycles in increments of 500 cycles. Four of the six nonreinforced encapsulants and all four reinforced encapsulants had better abrasion resistance than the paints. Based on the test results, the following preliminary performance criteria for abrasion resistance were proposed: (a) for nonreinforced encapsulants, specimens shall not lose, on the average, more than 20 % of the film thicknesses when subjected to 1000 abrasion cycles, and (b) for reinforced encapsulants, no abrasion completely through the reinforcement shall occur, and the polymeric component shall totally fill all voids in the reinforcement after the specimen has been subjected to 1000 abrasion cycles; that is, the substrate shall be totally covered with polymeric component and reinforcement after abrasion. In both cases, the criterion is based on testing the encapsulant specimen at the minimum thickness recommended by the manufacturer.

Key words: abrasion resistance; building technology; coatings; curing; dynamic mechanical analysis; encapsulants; glass transition temperature; lead-based paint; performance criteria
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1. INTRODUCTION

1.1 Background

The U.S. Department of Housing and Urban Development (HUD) has estimated that about 57 million homes in the United States have lead-based paint (LBP) on at least one painted surface [1]. One way of helping to minimize the hazards of lead-based paint in housing is to apply an encapsulant over the lead-based paint [1]. Encapsulant is the term equally used to describe liquid-coating-based products that are either nonreinforced or reinforced with woven or nonwoven fabrics or mesh applied in the field. In either case, the liquid component is typically applied to the substrate like a paint; that is, by brush, roller, sponge applicator, or spray equipment. When the encapsulant contains fabric reinforcement, this component is generally embedded into a freshly applied layer of the liquid (polymeric) component. A subsequent layer of the liquid component then covers the reinforcement.

In some cases, a reinforcement component having an adhesive backing may be applied directly to the substrate and then covered with a layer of the liquid component. After application, the liquid component solidifies into a coherent film either by evaporation of a volatile component or by chemical reaction. As coating-based products, encapsulants rely on adhesion for attachment to the lead-based paint, as opposed to “enclosures” — a term used to describe preformed materials which depend on mechanical fasteners for securement over the paint.

As an abatement strategy, the application of an encapsulant coating may under some circumstances be more readily accomplished than use of other means such as the application of an enclosure or the removal of the lead-based paint. However, a major deterrent to the use of encapsulants is the scarcity of performance data. The Environmental Protection Agency (EPA) has issued a report describing a pilot program conducted on several commercial encapsulants [2]. The EPA study was conducted to provide data on properties being considered by ASTM Subcommittee E06.23 on Lead Paint Abatement for incorporation in standard specifications for encapsulants. Included in the EPA study were measurements of adhesion, scrub resistance, flexibility, impact resistance, abrasion resistance, tensile strength, elongation, blistering, chalking, and hardness.

Because of the need for performance criteria to assist in the selection and use of encapsulants, HUD requested that the National Institute of Standards and Technology (NIST) conduct a study to propose performance criteria for these products. The objective of the study was to develop preliminary performance criteria for encapsulants for lead-based paint in housing using short-term laboratory tests. A series of reports will be issued describing the results of the study. This is the first report in the series, and addresses proposed performance criteria for abrasion resistance. An overview of the performance criteria concept is given in Section 1.3.


1.2 Protection Against Exposure to Lead in Paint

To perform their intended function of reducing exposure to the lead in paint to low levels, encapsulants should be capable of surviving in their service environment over relatively long periods
of time — perhaps up to 20 years or more. Factors affecting the performance of encapsulants (or any building material) include [6]:

- use factors such as normal wear and tear, abuse, and maintenance procedures,
- stress factors such as sustained and periodic loads,
- weathering factors such as radiation, temperature, water, normal air constituents, air contaminants, freeze-thaw, and wind,
- biological factors such as fungi and bacteria, and
- incompatibility factors that may be either physical or chemical.

In developing the performance criteria, it is necessary to state the performance requirements and to know as much as possible about the intensities of the various factors and the modes by which the encapsulants fail. With such information, performance test methods may be selected to determine an encapsulant’s capability to perform as required. If the degradation factors and their intensities are not known, an empirical approach must be taken to evaluate whether an encapsulant is capable of performing satisfactorily under the more common degradation factors that they would likely encounter in service. That is, on the most general performance level, the encapsulant would, with an acceptable probability, ensure that the lead is kept in place and prevented from contaminating the environment for the desired time.

The use of encapsulants for containing lead-based paint is a rather recent development. As a consequence, field data are not available that describe common failure modes and quantify stresses to which encapsulants are exposed. Thus, in initiating the development of performance criteria for encapsulants, the question raised was: How might an encapsulant be adversely affected in service such that it would lose its intended function to reduce human exposure to the lead in lead-based paint? In answer, it was considered that the integrity of the in-place encapsulant could be compromised and, thus, expose building occupants to lead from the lead-based paint through the following:

- abrasion — the encapsulant might be mechanically worn away by friction forces due to actions such as cleaning, rubbing, or scouring.
- inadequate adhesion — the encapsulant might peel or otherwise delaminate from the paint surface due to lack of proper adhesion, or cause failure (e.g., peeling, flaking and chipping) of the underlying lead-based paint layer due to encapsulant application.
- diffusion — the encapsulant layer might have poor resistance to the transport of lead ions under circumstances where lead might be leached from the underlying paint.\(^1\)
- impact — the encapsulant might be fractured or otherwise damaged under rapidly applied loads.
- joint movement — the encapsulant might crack or split over moving cracks in the substrate due to cyclic expansion and contraction.
- chemical (fluid) damage — the encapsulant might excessively soften, swell, or be otherwise damaged when exposed to household fluids normally encountered in service for activities such as maintenance, cleaning, and cooking.

Preliminary performance criteria to be developed in the present study will address encapsulant attributes that consider the above phenomena which are related to the integrity of the in-place

\(^1\)Diffusion is a technical term describing the transport of a species through a medium due to random molecular motions. ASTM has defined the term, lead accessibility, as “the ability of an encapsulation product to resist or inhibit the transport of lead to its surface” [5]. Diffusion is one means by which lead may be transported to the surface of an encapsulant.
encapsulant. Other performance attributes such as those related to health and fire safety, installation, repairability, and aesthetics will not be considered.

Lead-based paint may be found on many room components including walls, ceiling, floors, doors, windows, and trim molding. It is reasonable to assume that encapsulants applied to certain components would be more prone to specific types of damage than if applied to others. For example, it would be expected that an encapsulant on a wall might be more susceptible to abrasion damage than if it were applied on a ceiling. This example illustrates that the requirements for satisfactory performance of encapsulants may vary depending on their end use. Performance criteria developed for encapsulants need to recognize this fact. Unfortunately, at the present state-of-knowledge wherein little field data are available on the encapsulant performance, it is not appropriate at this time to suggest criteria that deal with specific use conditions. Consequently, a limitation to the initially proposed performance criteria is that they may be more severe than needed for some situations. In the future, as the state-of-knowledge becomes more refined, this limitation may be overcome by refining the initially proposed criteria.

Another limitation at the present state-of-knowledge is that encapsulants are recommended for application only on substrates that are in acceptable condition through proper surface preparation. If the substrate surface is not acceptable, then the encapsulant should not be applied. Consequently, the initial performance criteria proposed in the present study are based on the performance of products on properly prepared substrates. In the future, if encapsulant products become available which are considered to be suitable for application on substrates that are less than acceptable at today's state-of-knowledge, then future revisions to the criteria would also be needed.

1.3 Performance Format

The criteria to be developed in the study are to be presented in a performance criterion format. A performance specification defines a product exclusively in terms of criteria relevant to the functions it is to perform; in contrast, a prescriptive specification defines a product primarily in terms of its constituents and their relationships to each other [7]. A performance-criteria-based specification describes a product less narrowly than a prescriptive specification. The performance criterion format has four key elements: the Requirement, the Criterion, the Evaluation, and the Commentary, which are defined as follows:

- The Requirement is a qualitative statement which describes what the product is to accomplish.
- The Criterion is a quantitative expression of the level of performance which the product achieve to perform acceptably.
- The Evaluation sets forth the test or other method(s) to be used for determining whether the product conforms to the stated criterion.
- The Commentary provides for comment concerning the background or an explanation of the reason for, or intent of, the stated criterion. Commentary is presented for informational purposes and is non-mandatory.

Tables A-1 through A-6 given in Appendix A present an initial outline for the development of performance criteria for encapsulants. The encapsulant attributes for which performance criteria are considered in Tables A-1 through A-6 are those discussed above: abrasion resistance, adhesion, diffusion resistance, impact resistance, joint-movement capacity, and chemical resistance. With the exception of abrasion resistance which is addressed in the main text of this report, the criteria for the other attributes are not fully developed in the Appendix. These other attributes will be the subject of
future reports in which the test results from commercial encapsulants will be presented and criteria will be recommended. Note, in Tables A-1 through A-6, that the outlines of the suggested performance criteria do not divide the requirements along product lines varying by factors such as the type of encapsulant (e.g., synthetic-resin-based versus inorganic-cement-based) or whether or not it is reinforced. This is in keeping with the performance criterion concept which defines a product according to its functions and not its constituents. However, the practicality of developing evaluative procedures may preclude such an approach when criteria are recommended based on specific test methods.

1.4 Objectives and Scope of Report

One objective of this report is to describe the encapsulants in this performance criterion study including the time required for their cure after application and exposure at ambient laboratory conditions. A second objective is to present data on their abrasion resistance and recommend an abrasion resistance performance criterion.

Cure of a material or system has been defined as the process by which it attains its intended properties through evaporation, chemical reaction, heat, radiation, or a combination thereof [8]. Consistent with this definition, liquid-applied encapsulants cure in place to form solid films either through loss of the volatile component(s) (e.g., water) or through chemical reaction(s). Before conducting abrasion tests, it was desirable to confirm that the encapsulant was essentially fully cured so that abrasion tests would not be conducted on specimens undergoing large changes in properties. Thus, the experimental plan was, to the extent possible, to conduct tests on products only after the cure was essentially complete. However, as will be seen later in the report, some products were still experiencing changes more than three months after film application. In these cases, practical considerations associated with the necessity of conducting tests within a relatively short period (e.g., a month) after film application precluded waiting until it appeared that cure was mostly attained.

The property selected to characterize whether an encapsulant film was close to being fully cured after film application was the glass transition temperature ($T_g$); the $T_g$ values were determined using dynamic mechanical analysis (DMA). Hill [10] has noted that DMA has been applied frequently in recent years to follow the cure of coatings. In the present study, specimens of newly-formed encapsulant films were periodically subjected to DMA characterization from 1 week to 13 weeks. The encapsulant was considered to be essentially fully cured when the glass transition temperature displayed relatively little change over time.

1.5 The Glass Transition and the Glass Transition Temperature

The glass transition has been defined as "the reversible change in amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one" [11]. The process normally occurs over a narrow temperature range. The approximate midpoint of this temperature range is taken as $T_g$, the glass transition temperature. Not only do hardness and brittleness of the polymer change rapidly at the

---

\[\text{Not all definitions of cure include evaporation; for example, see reference [9]. However, evaporation is considered appropriate for encapsulants as some are water-borne liquid-applied coatings.}\]
glass transition temperature, but other properties such as modulus and other mechanical properties, thermal expansion, and heat capacity change rapidly as well [11]. The \( T_g \) is affected by factors such as molecular weight, crosslink density, length of side chains, intermolecular forces, rotation about molecular bonds, and plasticization [12,13]. Thus, as the freshly-applied liquid encapsulant solidifies into a film, the \( T_g \) may increase over time as parameters such as molecular motions decrease, and chain crosslinks and stiffness increase [10]. For example, in the case of a film formed by solvent evaporation, as the solvent evaporates from the coating film, its \( T_g \) increases [14]. Then, as the film-forming approaches completion, the \( T_g \) shows little change over time. It is noted that the value of the \( T_g \) is dependent upon the measurement technique and, thus, the reported value must be accompanied by reference to the measurement method and heating rate [10,13]. And, as a corollary, the same method should be used when comparing the \( T_g \)'s of a series of samples.

2. EXPERIMENTAL
2.1 Description of Encapsulants and Preparation of Films

Table 1 presents the ten commercial encapsulants included in the study. They were supplied by manufacturers. The descriptions of the type of encapsulant and reinforcements were taken from the product container or literature. The film thicknesses were determined as described later in this Section (2.1). Six (Sample Nos. 1 - 6) were nonreinforced and four (Sample Nos. 7 - 10) were reinforced — two with nonwoven and two with woven fabrics. Six of the ten encapsulants were based on water-borne acrylic resins. Three other encapsulants were based on epoxy, polyester, and polyurethane resins. One product (Sample No. 10) was inorganic-cement-based with an acrylic binder. Two commercial house paints, one acrylic (Sample No. 11) and the other alkyd (Sample No. 12), were also included as controls. For both paints, a note on the container stated that they conformed to the Federal Specification designation in Table 1.

After receipt, all encapsulants and paints were stored in their original sealed containers at ambient laboratory conditions. The temperature was 21 °C ± 2 °C (72 °F ± 3 °F). The relative humidity ranged from about 25 % to 60 % depending upon the time of year. Immediately before use, the contents of the opened containers were well stirred. To control the thickness of the film samples, they were prepared using a drawdown technique\(^3\). Free films, used initially to characterize the thickness of the samples, were cast on release paper, about 140 mm by 250 mm (5.5 in by 10 in) in area. Thickness measurements were made using a Mitutoyo Thickness Gage, Model No. 192-655\(^4\). Its calibration was checked using a set of standard gage blocks.

Table 1 includes the dry film thicknesses of the encapsulants. In the case of the nonreinforced products, the thicknesses were chosen to be in accordance with each manufacturer's recommendation. Some manufacturers' literature provided recommended wet film thicknesses; whereas other manufacturers' recommendations were for dry film thicknesses. In this latter instance, experimentation in applying varying wet films was conducted to determine the resulting dry film

\(^3\)This technique uses an adjustable knife blade (i.e., the drawdown blade), bar, or rod to control distribution of the adhesive on the substrate [15]. The adhesive thickness is controlled by the distance between the blade edge and the substrate surface.

\(^4\)Certain trade names or company products are mentioned in the text to specify adequately the experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment is the best available for the purpose.
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Polymeric Component</th>
<th>Reinforcement Fabric</th>
<th>Dry Film Total Thickness (in)</th>
<th>Dry Film Thickness Applied Over Reinforcement (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Acrylic</td>
<td>None</td>
<td>0.18 (0.007)</td>
<td>NA</td>
</tr>
<tr>
<td>2</td>
<td>Acrylic</td>
<td>None</td>
<td>0.20 (0.008)</td>
<td>NA</td>
</tr>
<tr>
<td>3</td>
<td>Acrylic</td>
<td>None</td>
<td>0.15 (0.006)</td>
<td>NA</td>
</tr>
<tr>
<td>4</td>
<td>Acrylic</td>
<td>None</td>
<td>0.15 (0.006)</td>
<td>NA</td>
</tr>
<tr>
<td>5</td>
<td>Epoxy</td>
<td>None</td>
<td>0.07 (0.003)</td>
<td>NA</td>
</tr>
<tr>
<td>6</td>
<td>Polyester</td>
<td>None</td>
<td>0.29 (0.011)</td>
<td>NA</td>
</tr>
<tr>
<td>7</td>
<td>Acrylic</td>
<td>Nonwoven Synthetic</td>
<td>0.51 (0.020)</td>
<td>0.18 (0.007)</td>
</tr>
<tr>
<td>8</td>
<td>Acrylic</td>
<td>Nonwoven Glass</td>
<td>0.41 (0.016)</td>
<td>0.13 (0.005)</td>
</tr>
<tr>
<td>9</td>
<td>Polyurethane</td>
<td>Woven Glass</td>
<td>1.2 (0.047)</td>
<td>0.10 (0.004)</td>
</tr>
<tr>
<td>10</td>
<td>Inorganic-Cement-Based With an Acrylic Binder</td>
<td>Woven Glass</td>
<td>1.3 (0.050)</td>
<td>0.69 (0.027)</td>
</tr>
<tr>
<td>11</td>
<td>Latex (Fed. Spec. TTE 2784)</td>
<td>None</td>
<td>0.06 (0.002)</td>
<td>NA</td>
</tr>
<tr>
<td>12</td>
<td>Alkyd (Fed. Spec. TTE 489)</td>
<td>None</td>
<td>0.08 (0.003)</td>
<td>NA</td>
</tr>
</tbody>
</table>

*The descriptions of the type of encapsulant and reinforcements were taken from the product container or literature. The film thicknesses were determined as described in Section 2.1 of this report.

*NA indicates not applicable.*
thickness. In the case of the reinforced products, the thickness of each sample was selected to be consistent with the manufacturers’ recommendations for the thickness of the film to be applied over the reinforcement. If that information was not available, then the selected film thickness was approximately the total thickness of the reinforced product (as suggested in the manufacturer’s literature) minus the thickness of the reinforcement.

2.1.1 Specimens for DMA Analysis. As in the case of the original thickness measurements, encapsulant and paint samples for DMA analysis were prepared on release paper as free films using the drawdown blade technique. These films were kept on the release paper at ambient laboratory conditions until analyzed. Because the intent was to measure the glass transition temperature of the base polymeric component as it cured over time after film application, the reinforcements for Sample Nos. 7 through 10 were not incorporated in the films used for DMA measurements. Rectangular test specimens having dimensions of 5 mm to 10 mm by 23 mm (0.2 in to 0.4 in by 0.9 in) were cut from the drawdown sections using a razor-edge knife. Each specimen was used for only one DMA analysis; that is, replicate analyses of all samples were conducted on different rectangular test specimens. In all cases, the release paper was removed immediately before analysis.

2.1.2 Specimens for Abrasion Resistance Tests. Samples for abrasion resistance tests consisted of reinforced and nonreinforced encapsulant and paint films applied on 6 mm (0.25 in) thick polymethyl methacrylate (PMMA)\textsuperscript{5} sheet using a drawdown technique to provide dry film thicknesses as listed in Table 1. The dimensions of the PMMA sheet to which a wet film was applied were 610 mm by 203 mm (24 in by 8 in). The width of the wet film which was drawn parallel to, and in the middle of, the long dimension of the PMMA sheet was approximately 140 mm (5.5 in). As a consequence, a strip approximately 35 mm (1.4 in) wide was left uncoated along each long edge of the PMMA sheet. After the wet film dried, the average thickness of the dry film on the PMMA sheet was measured for use in calculating the percent film loss during abrasion testing. Twenty pairs of measurements were made along the long dimension of each PMMA sheet (fig. 1). Each pair consisted of a determination of the thickness of the coated sheet (Thc) and a determination of the thickness of the uncoated sheet (Thu) at an adjacent location. A dry film thickness (Thn) was calculated as the difference of two determinations:

\[
Th_n = Th_c - Th_u
\]

The average of the twenty pairs of measurements was then calculated and taken as the average initial thickness of the dry film (Thn). This value was used in the calculation of the percent film lost during abrasion (Section 2.3). For the 12 samples in the study, the coefficients of variation of the initial thickness averages ranged from 4 % to 22 %, which may reflect, in some cases, some unevenness of the encapsulant and paint films.

After determining the average initial film thickness, abrasion resistance test specimens with dimensions of 100 mm by 100 mm (4 in by 4 in) were cut from the larger PMMA sheet. A hole with a diameter of approximately 6 mm (0.25 in) was drilled in the center of the specimen to allow its placement in the abrasion test device.

\textsuperscript{5}Note that the ASTM standard specifications [4,5] specify the use of cold rolled steel panels that may be primed. The use of PMMA was selected for this study prior to issue of the ASTM standards. Because abrasion resistance is a surface property, little difference due to substrate would be expected.
Figure 1. Schematic of the measurement pattern used in the determination of the average dry film of an encapsulant or paint sample.

2.2 Dynamic Mechanical Analysis Procedure

In conducting a DMA analysis, the specimen is subjected to a sinusoidal mechanical strain of a selected frequency and amplitude while the temperature is raised at a programmed rate. The DMA equipment used was a Rheometrics RSA II analyzer. Each encapsulant was analyzed at least twice at each age; i.e., time elapsed after film application, which was generally between 1 week and 13 weeks. In a limited number of cases where the cure of the encapsulant appeared to be continuing beyond the 13-week period, additional DMA tests were performed at longer times.
The following experimental parameters were used in the DMA analyses:

- specimen loading: tension/compression
- specimen length: 23 mm
- specimen width: 5 mm to 10 mm
- specimen thickness: free film applied thickness (Table 1)
- temperature range: -70 °C to +80 °C
- temperature sweep: dynamic temperature steps (temperature increments of 2 °C in Tg range; 10 °C otherwise)
- time per measurement: soak time of 30 s
- frequency: 10 Hz (62.8 rad./s)
- strain: 0.05 %
- delay before test: 1 min
- auto tension applied: yes
- auto strain applied: yes

2.3 Abrasion Resistance Procedure

Abrasion resistance was determined according to ASTM D 4060, “Abrasion Resistance of Organic Coatings by Taber Abraser” [16]. This method was considered appropriate for the initially proposed performance criteria because it simulates a rubbing action that an encapsulant could be subjected to in service. The abrasion device was a Taber Instrument Corporation Model 503 Abraser Tester equipped with a CS-17 abrasion wheel. For each encapsulant and paint sample, four replicate specimens having a minimum age of 28 days (see pages 17 and 18) were tested. The abrasion tests were conducted at ambient conditions in increments of 500 cycles until 2000 cycles were reached or until the coating was abraded to the point of exposing the PMMA sheet. A cycle consisted of one complete revolution of the test specimen in the abrasion device. After each 500 cycles, the specimen was visually examined to observe its condition, and the mass of the specimen and the thickness of the coating in the abraded area were measured.

Mass was measured using an analytical balance sensitive to 0.01 mg. Its calibration was checked using a set of standard weights. Mass loss due to abrasion (Mb) was calculated by subtracting the mass of the abraded specimen (Ma) from that of the unabraded specimen (Mw):

\[ M_b = M_w - M_a \]

The thickness of the film lost during each 500-cycle increment of abrasion was determined using the following procedure. Near each of the four corners of the 100 mm by 100 mm (4 in by 4 in) test specimen, measurements of the film thickness in the abrasion track (Thb) and just outside both edges of the abrasion track (Th01 and Th02) were made (fig. 2). The film thickness lost (Thb) at each of these four locations was taken as:

\[ Th_b = [(Th_{01} + Th_{02})/2] - Th_r \]

---

Soak time “specifies the time interval between temperature equilibrium at the selected zone temperature and the start of the measurement cycle.” (See Rheometrics Solids Analyzer Manual Section 2-25.) In this procedure, the temperature is automatically incremented from selected upper and lower limits (i.e., the zone).
Figure 2. Schematic of an abrasion specimen indicating the abrasion track and thickness measurement locations.

Then, the average film thickness lost ($\text{Th}_{pl}$) near the four corners was calculated. About three-quarters of the coefficients of variation of these averages ranged from 10% to 60% with the majority of the remainder being greater than 60%. This variation appears to indicate that the abrasion is nonuniform at the four corners of the specimen, and supports performing more than one measurement of thickness loss. The percent film thickness lost ($\text{Th}_{pl}$), which is the parameter used in the presentation of the abrasion resistance data, was calculated as:

$$
\text{Th}_{pl} = \left( \frac{\text{Th}_{al}}{\text{Th}_{at}} \right) \cdot 100
$$

(Note: $\text{Th}_{at}$ is defined in Section 2.1.2.)

3. RESULTS AND DISCUSSION
3.1 Dynamic Mechanical Analysis

In a DMA analysis, three properties are typically determined for each temperature. The first is the storage modulus ($E'$), which is a measure of the energy stored during each deformation cycle. This parameter is related to the stiffness of the specimen. The second property is the loss modulus ($E''$),
which is related to the energy dissipated. The third is \( \tan \delta \), the ratio of the loss modulus to the storage modulus.

Figures 3 through 6 illustrate plots obtained in DMA analyses of encapsulant films when the specimens were approximately 13 weeks old. For purposes of these illustrations, the measurements were made in 2 °C (3.6 °F) increments across the entire temperature range. The figures show the results of the analyses of an acrylic (Sample No. 4), the epoxy (Sample No. 5), the polyester (Sample No. 6), and the polyurethane (Sample No. 9). The data plots in each of the four figures are generally similar, although key features such as temperatures for the peak maxima are different for the different materials. Three curves are present in each plot: \( E' \), \( E'' \), and \( \tan \delta \). The temperature at which the maxima occur in both \( E'' \) and \( \tan \delta \) curves have commonly been used as a measure of the glass transition temperature [10]. Note that these two maxima in each of the four figures are not the same and, for this reason, the property (\( E'' \) or \( \tan \delta \)) taken as a measure of the glass transition must be specified along with the details of the experimental run such as frequency, amplitude, and rate of temperature rise.

In the present study, the change in glass transition temperature of the encapsulants with time elapsed after film application was generally followed using the peak of the \( E'' \) curve. This was in accordance with the recommendation of ASTM Standard D 4092 [11] wherein it is stated that “the most reliable estimates [of glass transition temperature] are normally obtained from the loss peak observed in dynamic mechanical tests ...” Thus, using the \( E'' \) curves, the glass transition temperatures of the acrylic (Sample No. 4), the epoxy (Sample No. 5), the polyester (Sample No. 6), and the polyurethane (Sample No. 9) specimens shown in Figures 3 through 6 were -15 °C, +31 °C, +17 °C, and +45 °C (5 °F, 88 °F, 63 °F, and 113 °F), respectively.

Note in Figures 3, 4, and 6 that the \( E'' \) and \( \tan \delta \) curves contain only one peak. In contrast, the \( \tan \delta \) curve in Figure 5 contains a pronounced shoulder at about 25 °C (77 °F). Additionally, the peak in the \( E'' \) curve of Figure 5 is relatively broad. A reason for these features may be the polyester-based encapsulant is not a homogeneous material, but contains more than one species or phase, each of which undergoes a transition at a different temperature. Investigation of this possibility was beyond the scope of the current study. A practical consequence of the broad peak in the \( E'' \) curve of Figure 5 is that it may be difficult to determine the temperature at which the peak maximum occurs and considerable scatter between replicate measurements of the glass transition temperature may result.

Figure 7 presents plots of the glass transition temperature measurements versus time elapsed after film application for the acrylic-resin based encapsulant specimens. Figure 8 contains similar plots for the other four encapsulants and the latex and alkyd paints. To demonstrate the differences among the 12 coatings, Table 2 gives the average glass transition temperature of the samples at 13 weeks. In Figures 7 and 8, the open circle indicates the average measurement, and the closed diamond represents the result of an individual measurement. Note that overstrikes in plotting data points are not distinguished. Material variability between the DMA test specimens may account for some of the scatter in the data shown in Figures 7 and 8, although an investigation of this possibility was not conducted. For the majority of the measurements, the coefficients of variation for the average values were 15 % or less, although a few values range from 25 % to 30 %. Sample No. 2 showed higher coefficients of variation (discussed below) that, with the exception of the average value at 13 weeks, ranged from 20 % to 80 %.
Figure 3. DMA results for an acrylic resin-based encapsulant film (Sample No. 4). The frequency and strain were 10 Hz and 0.05 %, respectively. Stepwise dynamic temperature programming was used with 2 °C temperature rise increments and the soak time was 30 s.

Figure 4. DMA results for the epoxy resin-based encapsulant film (Sample No. 5). The frequency and strain were 10 Hz and 0.05 %, respectively. Stepwise dynamic temperature programming was used with 2 °C temperature rise increments and the soak time was 30 s.
Figure 5. DMA results for the polyester resin-based encapsulant film (Sample No. 6). The frequency and strain were 10 Hz and 0.05 %, respectively. Stepwise dynamic temperature programming was used with 2 °C temperature rise increments and the soak time was 30 s.

Figure 6. DMA results for the polyurethane resin-based encapsulant film (Sample No. 9). The frequency and strain were 10 Hz and 0.05 %, respectively. Stepwise dynamic temperature programming was used with 2 °C temperature rise increments and the soak time was 30 s.
Figure 7. Plots of glass transition temperature versus time elapsed after film application for the acrylic resin-based encapsulant films. Overstrikes are not distinguished.
Figure 8. Plots of glass transition temperature versus time elapsed after film application for the non-acrylic resin-based encapsulant films and paint films. Overstrikes are not distinguished.
Table 2. Average glass transition temperature of the samples at 13 weeks

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Glass Transition Temperature</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average*</td>
<td>Standard Deviation</td>
<td>Average*</td>
</tr>
<tr>
<td></td>
<td>°C</td>
<td>°F</td>
<td>°C</td>
</tr>
<tr>
<td>1</td>
<td>12.3</td>
<td>54.1</td>
<td>0.5</td>
</tr>
<tr>
<td>2</td>
<td>-17.8</td>
<td>0.0</td>
<td>2.0</td>
</tr>
<tr>
<td>3</td>
<td>21.1</td>
<td>69.9</td>
<td>2.3</td>
</tr>
<tr>
<td>4</td>
<td>-16.9</td>
<td>1.5</td>
<td>1.1</td>
</tr>
<tr>
<td>5</td>
<td>40.5</td>
<td>104.8</td>
<td>5.0</td>
</tr>
<tr>
<td>6</td>
<td>54.2</td>
<td>129.6</td>
<td>2.2</td>
</tr>
<tr>
<td>7</td>
<td>8.5</td>
<td>47.3</td>
<td>0.1</td>
</tr>
<tr>
<td>8</td>
<td>-19.6</td>
<td>-3.2</td>
<td>0.1</td>
</tr>
<tr>
<td>9</td>
<td>19.3</td>
<td>66.7</td>
<td>1.2</td>
</tr>
<tr>
<td>10</td>
<td>18.5</td>
<td>65.2</td>
<td>0</td>
</tr>
<tr>
<td>11</td>
<td>20.6</td>
<td>69.1</td>
<td>1.5</td>
</tr>
<tr>
<td>12</td>
<td>19.5</td>
<td>67.2</td>
<td>0.1</td>
</tr>
</tbody>
</table>

*Average of a minimum of two measurements.

In examining the plots in Figure 7, it is evident that the results of the DMA analyses of the acrylic-based encapsulants varied among the products. First, the glass transition temperatures were not the same for all these materials. For example, the 13-week old specimens had glass transition temperatures bracketing a range from about -20 °C to +20 °C (-4 °F to 68 °F) (Table 2). Samples Nos. 2, 4, and 8 were near the lower end of this range; whereas Sample Nos. 1, 3, and 5 were near the upper end of the range. Second, the change in glass transition temperature over time also varied between the acrylic-based encapsulants. Sample Nos. 1, 4, 7, and 8 showed little change in glass transition temperature after about 2 to 4 weeks after application.

In contrast, the glass transition temperature of Sample No. 3 was still showing an increase up to 13 weeks after application, although the percent increase was greatest over the first 4 weeks. An additional DMA test of Sample No. 3 after about 9 months gave a glass transition temperature that was about 4 °C (7 °F) higher than that measured after 13 weeks, suggesting that the sample had not completely cured within the 13 week period. Finally, in the case of Sample No. 2, the glass transition temperature over time was widely variant showing apparently higher values at 3 to 6 weeks than at 1 to 2 weeks, or at 13 weeks. This behavior was attributed to the broadness of the E'' peak which spanned about 15 °C (27 °F), and which rendered it difficult to assign the temperature of the peak maximum. The peaks of the tan δ curves for Sample No. 2 tended to be sharper than those of the E'' curves. Thus, the temperatures of the tan δ peaks were examined and found to show little change after 2 weeks. The interpretation of this finding was that Sample No. 2 had essentially cured within 2 weeks of application.
In examining Figure 8, it can be seen that results of the DMA analyses of the non-acrylic-based encapsulant films differed among the products. When measured at 13 weeks, the glass transition temperatures for the epoxy (Sample No. 5), the polyester (Sample No. 6), the polyurethane (Sample No. 9), and the inorganic-cement-based product (Sample No. 10) were about 40 °C, 54 °C, 19 °C, and 18 °C (104 °F, 129 °F, 66 °F, and 64 °F), respectively (Table 2). Only Sample No. 10 displayed a glass transition temperature that showed little change after 13 weeks time, although there was some scatter between the averages measured at the different time periods. Sample No. 10 was difficult to handle (i.e., prepare specimens and mount them in the DMA analyzer) as a free film without its reinforcement. Some of scatter in the glass transition temperature data might have been due to this difficulty.

Sample No. 6 displayed considerable scatter, particularly for the measurements made between 2 and 6 weeks. Thus, it was difficult to judge whether cure had occurred. The other two non-acrylic-based encapsulants (Sample Nos. 5 and 9) displayed increasing glass transition temperatures over the 13-week period, although a large percentage of the increases occurred within the first 4 to 6 weeks. Because their glass transition temperatures appeared to be still increasing after 13 weeks, an additional DMA analysis was conducted on Sample Nos. 5, 6, and 9 when they were about 9 months old. In the case of Sample No. 6, the glass transition temperature was 44 °C (111 °F), which was comparable to the values measured after 3, 4, and 6 weeks. This sample was considered to have essentially attained cure. Variability between DMA test specimens may have contributed to the relatively high E" peak temperature measured at 13 weeks.

In the case of Sample Nos. 5 and 9, the glass transition temperatures at about 9 months were 50 °C and 24 °C (50 °F and 75 °F), respectively, which were 10 °C and 4 °C (18 °F and 7 °F) higher than those measured at 13 weeks. It was considered that these two samples had not completely cured after 13 weeks.

Also included in Figure 8 are the DMA results for the latex (Sample No. 11) and alkyd (Sample No. 12) paints. As is evident, both paints had comparable glass transition temperatures in the range of 13 °C to 17 °C (55 °F to 63 °F). These values displayed little variation over time after film application, although the alkyd paint (Sample No. 12) showed slightly greater scatter among the average measurements than the latex.

Reasons for differences in cure times among the encapsulant and paint samples were not investigated. However, in using DMA measurements over time to characterize the cure of the liquid-applied encapsulant and paint films, a determination was made as to whether or not the glass transition temperature was showing little or no change within 4 weeks after film application. Selection of 4 weeks was based on the practicality of a short wait between preparing test specimens and conducting tests. In practice, the use of encapsulants having long cure times may also be impractical as the application site may need to be kept vacant, for example, until cure is sufficiently advanced to prevent damage.

A 4-week cure is longer than that used between preparation and testing of paints. For example, typical cure times for high-performance interior coatings are normally between three and 21 days [17]. However, as just presented above, the DMA data on the encapsulants indicated that a shorter time, say on the order of a week or two, was not sufficient for some encapsulants in the study, as discussed below.
As discussed, the DMA analyses indicated that seven (Sample Nos. 1, 2, 4, 6, 7, 8, and 10) of the 10 encapsulants showed little or no change in glass transition temperature within 4 weeks or less after film application; that is, little additional curing took place after 4 weeks. Additionally, the latex and alkyd paints (Sample Nos. 11 and 12) displayed little change in glass transition temperature within 4 weeks. In fact, as just mentioned above, these paints underwent little change in glass transition temperature over the 1-week to 13-week period of measurement. Thus, for these seven encapsulants and two paints, a 4-week minimum cure time after film application was sufficient, if not conservative, before conducting tests.

The three remaining encapsulants (Sample Nos. 3, 5, and 9) had glass transition temperatures that were still increasing not only after 4 weeks, but also apparently after 13 weeks. Investigations of the mechanisms and kinetics of the curing of the individual encapsulants were beyond the scope of the study, and studies to determine reasons for not achieving constant glass transition temperatures were not performed. In spite of the DMA findings that Sample Nos. 3, 5, and 9 had not attained complete cure after 4 weeks, these samples had apparently cured to an extent that the dry films physically exhibited no indications that they should not be tested (or not considered suitable for use in occupied residences). Moreover, the practicality of conducting tests in a relatively short period of time after their preparation precluded waiting longer to begin testing. The decision was made to conduct tests on these samples after a minimum waiting period of 4 weeks.

Note that a minimum 4-week cure time is in contrast to the provisions of ASTM standard E 1795 and E 1797, in which a 7-day cure is specified. Data are not available relating abrasion resistance after 7 and 28 days cure time. Based on the DMA findings, the task groups responsible for the ASTM standard specifications should review the cure time requirement.

3.2 Abrasion Resistance

The abrasion resistance tests of the 10 encapsulant and two paint films were conducted for 2000 cycles unless a specimen abraded to the PMMA substrate before then. With the exception of Sample Nos. 9 and 10, after each 500-cycle increment, measurements of film mass loss and thickness loss were conducted. For Sample Nos. 9 and 10, only mass loss measurements were made, as discussed below. Figures 9, 10, and 11 present the results for the paints, nonreinforced encapsulants, and reinforced encapsulants, respectively, versus the number of abrasion cycles. Four replicate specimens were tested for each sample. In the figures, the open circle indicates the average measurement, and the closed diamond represents the result of an individual measurement. Overstrikes are not distinguished, and the lines in the plots are for convenience and only represent connecting of the points. In most cases where the plots are lacking data over the full 2000 cycle range, abrasion to the substrate occurred during the next 500 cycles after the last data point given on the plot. For example, Sample No. 2 (fig. 10) abraded to the substrate between 1000 and 1500 cycles, as evidenced by the last data point appearing at 1000 cycles.

For the average data for a given encapsulant film (figs. 9 - 11), there was generally less variation in the mass loss measurements than in the thickness measurements. The majority of the average mass loss measurements had coefficients of variation of less than 20%; whereas for the average thickness loss measurements, the coefficients of variation were generally less than 40%.
Figure 9. Mass loss and thickness loss versus abrasion cycles for paint films.

Abrasion resistance varied considerably between the test specimens which, it is important to note again, were applied at normally recommended film thicknesses. In the case of the paints, neither the latex (Sample No. 11) nor the alkyd (Sample No. 12) survived 2000 cycles without abrading to the PMMA substrate (fig. 9). In the case of the nonreinforced encapsulants, Sample Nos. 1, 3, 4, and 6 resisted 2000 cycles without abrading to the PMMA substrate; whereas Sample Nos. 2 and 5 abraded to the PMMA substrate after 1000 and 500 cycles, respectively (fig. 10). All four reinforced encapsulants (Sample Nos. 7-10) withstood 2000 cycles without exposing the PMMA substrate (fig. 11). In fact, at worst, these four samples only abraded to the top of the reinforcement layer.

7In the case of Sample No. 9, the mass losses at 1500 cycles and 2000 cycles were inadvertently not recorded. Thus, Figure 11 only shows mass loss data up to 1000 cycles.
Figure 10. Mass loss and thickness loss versus abrasion cycles for the nonreinforced encapsulant films.
Figure 10. Mass loss and thickness loss versus abrasion cycles for the nonreinforced encapsulant films (continued).
Figure 11. Mass loss and thickness loss versus abrasion cycles for the reinforced encapsulant films. (Note: In the case of Sample No. 9, the mass losses at 1500 cycles and 2000 cycles were inadvertently not recorded. Thus, this figure only shows mass loss data up to 1000 cycles.)
In developing a criterion for the abrasion resistance of encapsulant coatings, it was considered that encapsulants should be more resistant than typical household paints. The rationale is based on experience with paint performance and expectations for encapsulant performance. In the case of paints, experience has shown that those having good quality can provide acceptable service for 5 to 10 years. However, such a time period would represent a relatively short service life for an encapsulant which may be expected to provide service for perhaps 20 years or more. For this reason, to provide a point of reference with the encapsulants, the two paints conforming to Federal Specifications (Table 1) were included as controls. As “specification” paints, they presumably have good quality and, consequently, can provide a benchmark against which the abrasion resistance of the encapsulants may be judged. In this regard, as just noted, the basic performance of the two paints was that they did not withstand 2000 abrasion cycles without exposing the substrate. In comparison, eight of the 10 encapsulants, applied at their normally recommended thicknesses, resisted 2000 cycles without abrasion to the substrate. That is, qualitatively, when applied at normally recommended thicknesses, eight encapsulants were more abrasion resistant than the Federal Specification paints. In a similar finding, EPA reported that some, but not all, encapsulants in its study were more abrasion resistant than typical household paints [2].

To quantify abrasion resistance of the nonreinforced encapsulants, the abrasion resistance requirement specified in ASTM Standard Specification E 1795 [4] was considered. This abrasion resistance requirement is that, when applied at its normally recommended (by the manufacturer) minimum thickness, the encapsulant specimen shall not lose more than 20% of its thickness after 1000 abrasion cycles performed according to ASTM D 4060.

Table 3 gives the average percent thickness loss at 1000 cycles for the 10 encapsulants and 2 paints. The uncertainty in Table 3 is a 95% confidence interval for the mean percent thickness loss. The uncertainty considers the scatter in the data used to develop the mean value. ASTM D 4060 provides no information on the expected uncertainty of thickness loss measurements [16]. However, uncertainty estimates (95% confidence interval) for mass loss measurements are included and are about ± 50% of the average value. Assuming that the uncertainty in thickness loss measurements is comparable to that for mass loss, the uncertainties in Table 3 are within the expected range given by D 4060.

In examining Table 3, it is evident that the two paints displayed relatively poor abrasion resistance relative to the ASTM encapsulant requirement. The latex paint lost 56% of its film thickness, while the alkyd paint lost 37%. For the latex and alkyd paints, these values were about thrice and twice, respectively, more than that allowed by ASTM E 1795. This finding was not surprising as the requirement in E 1795 was intended to be stringent for encapsulants and not intended for paints. That the latex lost a greater percentage of the film thickness than the alkyd may be attributed to the differences in the two film thicknesses (i.e., 0.06 vs 0.08 mm or 2 vs 3 mil). Note in Figure 9 that both paints lost about the same amount of mass — 0.13 g for the latex and 0.14 g for the alkyd — after 1000 cycles.

In the case of the six nonreinforced encapsulants, three samples (Nos. 1, 4, and 6) met the ASTM E 1795 abrasion requirement, showing thickness losses of less than 20%. The three other samples (Nos. 2, 3, and 5) did not meet the ASTM E 1795 abrasion requirement with two of them (Nos. 2
Table 3. Average thickness loss after 1000 abrasion cycles

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Type of Coating</th>
<th>Average Thickness Loss* After 1000 Abrasion Cycles, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Nonreinforced Encapsulant</td>
<td>19 ± 10</td>
</tr>
<tr>
<td>2</td>
<td>Nonreinforced Encapsulant</td>
<td>53 ± 4</td>
</tr>
<tr>
<td>3</td>
<td>Nonreinforced Encapsulant</td>
<td>22 ± 3</td>
</tr>
<tr>
<td>4</td>
<td>Nonreinforced Encapsulant</td>
<td>11 ± 2</td>
</tr>
<tr>
<td>5</td>
<td>Nonreinforced Encapsulant</td>
<td>Abraded to Substrate</td>
</tr>
<tr>
<td>6</td>
<td>Nonreinforced Encapsulant</td>
<td>15 ± 2</td>
</tr>
<tr>
<td>7</td>
<td>Nonwoven Fabric Reinforced Encapsulant</td>
<td>13 ± 3</td>
</tr>
<tr>
<td>8</td>
<td>Nonwoven Fabric Reinforced Encapsulant</td>
<td>6 ± 3</td>
</tr>
<tr>
<td>9</td>
<td>Woven Fabric Reinforced Encapsulant</td>
<td>Not Determined</td>
</tr>
<tr>
<td>10</td>
<td>Woven Fabric Reinforced Encapsulant</td>
<td>Not Determined</td>
</tr>
<tr>
<td>11</td>
<td>Latex Paint</td>
<td>56 ± 7</td>
</tr>
<tr>
<td>12</td>
<td>Alkyd Paint</td>
<td>37 ± 14</td>
</tr>
</tbody>
</table>

* The uncertainty is the 95% confidence interval for the mean percent thickness loss.

and 5) losing considerably more than the 20% loss requirement. Sample No. 2 lost 53% of its thickness after 1000 cycles, which was comparable to the performance of the latex paint (Sample No 11). Sample No. 2 was the thickest of the nonreinforced encapsulants, and was about 2.5 to 4 times thicker than the paint samples. Sample No. 5 did not survive 1000 cycles without abrading to the substrate, which was worse than either paint. This sample was a relatively thin encapsulant film, applied at 0.08 mm (3 mil), which was also the thickness of the alkyd paint film.

Abrasion is a phenomenon which occurs at a surface and, consequently, it would be expected that, in the case of encapsulants and other coatings, the absolute abrasion loss at the specimen surface would be independent of the specimen thickness (provided that abrasiion to the substrate does not occur). In this regard, Sample No. 3 was borderline vis-a-vis the ASTM E 1795 requirement, displaying a 22% average loss after 1000 cycles. Sample No. 3 may have conformed to the requirement if it had been slightly thicker. Similarly, Sample No. 1 was also borderline, but conformed to the ASTM E 1795 requirement in that it lost 19% of its thickness after 1000 cycles. Perhaps, if this sample had been slightly thinner, it may not have conformed. The lesson learned is that, in considering conformance to a specification requirement based on a reduction of surface thickness, it is important to control the specimen thickness properly. For a test conducted on specimens having thickness

8 Any of the encapsulant products would conform to the ASTM E 1795 requirement if they were applied at sufficiently thick films. However, unless performance in the ASTM abrasion resistance test is borderline to the requirement, increasing the thickness of the product to the point of test conformance may be precluded by practical considerations such as cost or ease of application.
recommended by suppliers, those prepared somewhat thin may not show conformance whereas, at
normally applied thicknesses, they perhaps would conform.

The results for the nonreinforced encapsulant films indicate the ASTM E 1795 requirement for
abrasion resistance was able to differentiate relative differences in performance between the
nonreinforced encapsulant films themselves, and also between typical household paints and the
encapsulants. On this basis, a proposal for an abrasion resistance performance criterion for the
nonreinforced encapsulants is that the ASTM E 1795 requirement be adopted.

With regard to the reinforced samples, as noted above, it was only possible to measure the percent
thickness loss on Sample Nos. 7 and 8. These two products were reinforced with nonwoven fabrics
which had relatively uniform thickness. When the liquid component of the encapsulant was applied
on these fabrics, the resultant test specimens had, in turn, relatively uniform thickness. When
subjected to abrasion testing, the average thickness losses for Sample Nos. 7 and 8 at 1000 cycles
were 13 % and 6 %, respectively. Neither specimen abraded through the reinforcement; when
Sample Nos. 9 and 10 were tested, they also did not abrade through the reinforcement.

It was not possible to measure the percent thickness loss on Sample Nos. 9 and 10 because the
specimen surfaces were not uniformly planar. The reinforcements for these two samples are woven
fabrics. The texture of the weave was reflected in the surface of the specimens with resulting high
spots and low spots; that is; the initial thickness of the specimens was not uniform. As a
consequence, in conducting the ASTM D 4060 test method, it was found that thickness
measurements were not appropriate for these samples. The individual data points would vary
considerably depending on the location on the specimen surface where the foot of the thickness gage
was placed.

The finding that making thickness loss measurements on non-uniform specimens were inappropriate
was not unexpected as ASTM D 4060 states that the method is for specimens having uniform
thickness [16]. ASTM D 4060 is a common method in the coatings industry for evaluating the
abrasion resistance and, for this reason, the non-uniform reinforced encapsulants were subjected to
the test to determine whether it might be applicable to these types of products. Having an abrasion
test criterion for reinforced encapsulants that is based on a common test method would be beneficial
in expediting routine evaluation of commercial products.

When the reinforced encapsulants were tested according to D 4060, it was found that the method
was readily conducted. It was also found through visual examination that Sample Nos. 9 and 10
having the woven reinforcements abraded non-uniformly, more on the high spots of the surface than
on the low spots. Particularly with Sample No. 9, in some limited surface locations, the encapsulant
coating abraded completely to the reinforcement. However, more important to these observations,
the results of the tests showed the reinforced encapsulants to be relatively abrasion resistant. As
already indicated, none abraded through the reinforcement after 2000 cycles; that is, polymeric
component and reinforcement remained in place covering the entire area of the test specimen
substrate after the 2000 cycles. This finding was similar to that observed by EPA who reported that
the reinforced encapsulants were quite abrasion resistant, resisting 5000 cycles without abrading to
the substrate [2].

Considering the practicality of conducting the test, it is considered that the ASTM D 4060 test
procedure should be used for abrasion testing of the reinforced encapsulants. A proposed criterion
for abrasion resistance of reinforced encapsulants is that, after 1000 abrasion cycles, the polymeric component and reinforcement should remain in place over all areas of the test specimen substrate; that is, no abrasion completely through the reinforcement should occur and the polymeric component should totally fill all voids in the reinforcement. Such a criterion would eliminate the need to quantify the surface loss of material that is not uniform due to the non-uniformity of the surface. For these types of encapsulants, if abrasion is limited to the encapsulant coating above the reinforcement or does not proceed completely through the reinforcement, then the encapsulant remains capable of providing protection for the lead-based paint.

The criterion suggested for reinforced encapsulants that, after 1000 abrasion cycles, the polymeric component and reinforcement should remain in place over all areas of the test specimen substrate differs from that given in ASTM E 1797. The criterion in ASTM E 1797 [5], which is the same as that in ASTM E 1795 for nonreinforced encapsulants, is that the encapsulant specimen shall not lose more than 20 percent of its thickness after 1000 abrasion cycles performed according to ASTM D 4060. Based on the finding of the present study that some nonuniformly thick reinforcements preclude the measurement of thickness loss during abrasion testing, it is considered that the task group responsible for the ASTM standard specifications should review the specified failure requirement for reinforced encapsulants.

As a final comment regarding the uniformity of the surface loss during abrasion, note in Figure 9 that the mass loss per 500 cycles was relatively constant for each of the nonreinforced samples. A major exception was Sample No. 4 which lost little mass in general, and that loss occurred mostly in the first 500 cycles. For the nonreinforced encapsulants and paints, assuming that the density of the coating is constant across the specimen surface, then mass loss should be linearly related to the thickness loss if the abrasion is relatively uniform (e.g., large particles are not lost or the abrasion is not concentrated on high spots on the specimen surface). With the exception of Sample No. 5 (where the data were insufficient), a linear relation between thickness and mass loss was found. An analysis of the linear relationships between mass loss and thickness loss showed $r^2$-values of 0.95 or better. In contrast, for reinforced Sample Nos. 7 and 8, whose surface uniformity may have been influenced by the reinforcement, the relationships between mass loss and thickness loss were less well correlated, having $r^2$-values of 0.88 and 0.58, respectively. This observation supports the suggestion that the performance criterion for the reinforced encapsulants should be selected such that it does not depend on the specimen surface which may be quite non-uniform, particularly if the reinforcement is a woven fabric.
4. PRELIMINARY PERFORMANCE CRITERIA FOR ABRASION RESISTANCE

Based on the results of the testing conducted, the following is proposed as the preliminary performance criteria for abrasion resistance of encapsulants for lead-based paint.

Table 4. Proposed preliminary performance criteria for abrasion resistance

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall be capable of withstanding, without exposure of the underlying paint surface, normally encountered abrasion forces due to actions such as cleaning, rubbing, or scouring.</th>
</tr>
</thead>
</table>
| Criteria    | a) for nonreinforced encapsulants, specimens shall not lose, on the average, more than 20 % of the film thicknesses when subjected to 1000 abrasion cycles.  
             b) for reinforced encapsulants, no abrasion completely through the reinforcement shall occur, and the polymeric component shall totally fill all voids in the reinforcement after the specimen has been subjected to 1000 abrasion cycles; that is, the substrate shall be totally covered with polymeric component and reinforcement after abrasion. |
| Evaluation  | ASTM Method of Test D 4060 using the CS-17 abrasion wheel; the test shall be conducted on four replicate specimens after a minimum cure time of 28 days.  
             a) For the nonreinforced encapsulants, the results shall be averaged.  
             b) For the reinforced encapsulants, all four specimens shall conform to the criterion. |
| Commentary  | ASTM D 4060 is commonly used in the coatings industry for evaluating the abrasion resistance of household paints and coatings. The method was considered appropriate for the initially proposed performance criteria because it simulates a rubbing action that an encapsulant could be subjected to in service. Encapsulants, which are expected to provide acceptable service for periods longer than common paints, should have abrasion resistance superior to that of common paints. Data available to date suggest that many encapsulant products out perform typical paints in the ASTM D 4060 test method.  
             The criterion for nonreinforced encapsulants is taken from the abrasion resistance requirement given in ASTM E 1795 [4].  
             It is proposed that the abrasion testing be conducted after a minimum cure time of 28 days to allow sufficient time for the encapsulant to attain essentially complete cure. Data in the study indicated that shorter times of the order of a week or two are insufficient for some encapsulants. Also, in some cases, data indicated that some encapsulants did not reach complete cure even after 28 days. In these cases, the encapsulants had apparently cured to an extent that the dry films exhibited physically no indications that they should not be tested after a 28-day cure. The practicality of conducting tests in a relatively short period of time after their preparation was considered in proposing the 28-day minimum cure time.  
             For situations where the user expects that the encapsulant would be subjected to only mild abrasive actions, less severe criteria would suffice. If the user requires an encapsulant that has increased abrasion resistance, then a reinforced encapsulant should be considered. In the future, as the state-of-knowledge of encapsulant performance becomes more refined, the suggested criteria should be revised to deal with situations where the encapsulant may be subjected varying abrasive conditions.  
             It is expected that product evaluation vis-a-vis the proposed criteria would only be conducted periodically. In this regard, it must be recognized that encapsulant composition (or formulation) may change over time, but the trade or brand name may be kept the same. It is important that product manufacturers provide assurance to encapsulant users that the composition has not changed since the last product evaluation was performed. |
5. SUMMARY AND CONCLUSIONS

This report is the first in a series that describes the results of a study to develop preliminary performance criteria for encapsulants for lead-based paint. Encapsulants are liquid-coating products installed on the lead-based paint to help to minimize the hazards of lead-based paint in housing. Encapsulants rely on adhesion for attachment to the surface of the leaded paint. Application of encapsulants to protect building occupants and users against lead-based paint is a relatively recent development, and performance criteria for encapsulant materials and systems are not available. The U.S. Department of Housing and Urban Development requested the National Institute of Standards and Technology to conduct a study to develop the needed criteria. Data developed in the study may support revisions to ASTM standards for these products.

The report described the encapsulants used in the test program and characterized their cure after film application. In addition, the report described the results of abrasion tests conducted on the encapsulant samples and proposed preliminary performance criteria for abrasion resistance. Ten encapsulants were included in this study; six were nonreinforced and four were reinforced with either a nonwoven or woven fabric. Six of the encapsulants were acrylic-resin based; others were epoxy-, polyester-, and polyurethane-resin based, and one was inorganic-cement-based with an acrylic binder. Two household paints were also included as controls.

Before conducting the abrasion tests, it was necessary to determine whether the liquid-applied samples that cure over time to coherent films had attained cure. The property selected for this determination was the glass transition temperature which was measured using dynamic mechanical analysis (DMA). The results of the DMA analyses showed that seven encapsulants and the two paints showed little or no change in glass transition temperatures within 4 weeks or less after application; that is, little additional curing occurred after 4 weeks. In contrast, three encapsulants had increasing glass transition temperatures after at least 13 weeks indicating that they had not attained complete cure after 4 weeks or longer. Taking into consideration the impracticality of a long waiting period between preparation of test specimens and conducting the abrasion tests, a minimum waiting period of 4 weeks was selected. In those cases where the DMA data indicated that complete cure was not reached, the dry films showed no physical indications that they should not be tested at 4 weeks.

The abrasion tests were conducted using the procedure described in ASTM test method D 4060. Tests were conducted for 2000 abrasion cycles in increments of 500 cycles. Mass loss and thickness loss measurements were performed. The results of the abrasion tests indicated that four of the six nonreinforced encapsulants and all four reinforced encapsulants had better abrasion resistance than the paints. None of the reinforced encapsulants abraded through the reinforcement. Based on the results of the abrasion tests, the following preliminary performance criteria using ASTM test method D 4060 were suggested:

a) for nonreinforced encapsulants, the specimen shall not lose more than 20 % of the film thickness when subjected to 1000 abrasion cycles.

b) for reinforced encapsulants, no abrasion completely through the reinforcement shall occur, and the polymeric component shall totally fill all voids in the reinforcement after the specimen has been subjected to 1000 abrasion cycles; that is, the substrate shall be totally covered with polymeric component and reinforcement after abrasion.

In both cases, the encapsulant specimen shall be prepared at the manufacturer recommended minimum thickness.
6. ACKNOWLEDGMENTS

This study was sponsored by the U. S. Department of Housing and Urban Development (HUD). The authors acknowledge with thanks the support and encouragement of Ronald Moroney and William Wisner, HUD Office of Lead Hazard Control. The assistance provided through discussions with members of the encapsulant task group of ASTM Subcommittee E06.23 is also acknowledged. Thanks are expressed to the authors' NIST colleagues, Chiara Ferraris, Geoffrey Frohnsdorff, Lawrence Knab, Jonathan Martin, and Tinh Nguyen, who provided many noteworthy comments in reviewing this report. The authors appreciated the special assistance of Mark Vangel, NIST Statistical Engineering Division, in analyzing the thickness loss data.

7. REFERENCES


APPENDIX A. OUTLINE OF PRELIMINARY PERFORMANCE CRITERIA FOR ENCAPSULANTS

This appendix provides initial outlines for preliminary performance criteria for encapsulants for lead-based paint. The attributes considered are associated with the ability of the encapsulant to provide protection against exposure to lead from lead-based paint. The criteria are outlined in tabular form for abrasion resistance, adhesion, lead-ion diffusion resistance, impact resistance, joint-movement capacity, and chemical resistance. Criteria for abrasion resistance (which were suggested in Table A-1) were developed in the main body of this report. None of the other tables (A-2 through A-6) has been completed, but they will be the subject of future reports.

The outlines in Tables A-1 through A-6 follow the typical performance criteria format which has four elements: the Requirement, the Criterion, the Evaluation, and the Commentary, which are defined as follows:

- The Requirement is a qualitative statement which describes what the product is to accomplish.
- The Criterion is a quantitative expression of the level of performance which the product achieve to perform acceptably.
- The Evaluation sets forth the test or other method(s) to be used for determining whether the product conforms to the stated criterion.
- The Commentary provides for comment concerning the background or an explanation of the reason for, or intent of, the stated criterion. Commentary is presented for informational purposes and is non-mandatory.
Table A-1. Initial outline of performance statement for abrasion resistance

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall be capable of withstanding, without exposure of the underlying paint surface, normally encountered abrasion forces due to rubbing-type actions such as cleaning, rubbing, or scraping.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>To be developed</td>
</tr>
<tr>
<td>Evaluation</td>
<td>ASTM Method of Test D 4060</td>
</tr>
<tr>
<td>Commentary</td>
<td>ASTM D 4060 is commonly used in the coatings industry for evaluating the abrasion resistance of household paints and coatings. Encapsulants, which are expected to provide acceptable service for periods longer than common paints, should have abrasion resistance superior to that of common paints.</td>
</tr>
</tbody>
</table>

Table A-2. Initial outline of performance statement for adhesion

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall remain in place without peeling or otherwise delaminating from the surface of the lead-based paint over its intended service life.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>To be developed</td>
</tr>
<tr>
<td>Evaluation</td>
<td>To be developed</td>
</tr>
<tr>
<td>Commentary</td>
<td>The test method must be applicable to a wide variety of products that may vary in flexibility and thickness.</td>
</tr>
</tbody>
</table>

Table A-3. Initial outline of performance statement for lead-ion diffusion resistance

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall prevent the migration of lead ions under normally encountered service conditions.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>To be developed</td>
</tr>
<tr>
<td>Evaluation</td>
<td>To be developed</td>
</tr>
<tr>
<td>Commentary</td>
<td>This attribute can have significance in circumstances where the encapsulant is expected to be exposed to aqueous solutions capable of dissolving and/or transporting lead ions. The method is sensitive to detecting pinholes in the applied encapsulant; some products may only conform to the criterion if they are applied in two layers.</td>
</tr>
</tbody>
</table>
Table A-4. Initial outline of performance statement for impact resistance

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall be capable of withstanding, without loss of integrity, the normally encountered impact loads due to use and environmental exposure.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>To be developed</td>
</tr>
<tr>
<td>Evaluation</td>
<td>ASTM Method of Test D 2794</td>
</tr>
<tr>
<td>Commentary</td>
<td>ASTM D 2794 has become the industry-accepted method for determining the impact resistance of paints and coatings on steel substrates. It is considered appropriate for the initially proposed performance criterion on impact because it simulates a type of impact that an encapsulant may be subjected to in service. The head of the impact device is rounded. If impact with sharp objects is of concern, then another method of evaluation may be needed.</td>
</tr>
</tbody>
</table>

Table A-5. Initial outline of performance statement for joint movement

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall resist the movement of the encapsulant substrate encountered during normal service conditions without loss of integrity, or undue deformation.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>When tested, no splits, cracks, tears, or buckling shall occur in the encapsulant. In addition, total loss of adhesion of the encapsulant from the substrate shall not occur.</td>
</tr>
<tr>
<td>Evaluation</td>
<td>To be developed</td>
</tr>
<tr>
<td>Commentary</td>
<td>The ability of an encapsulant to withstand expansion and contraction generated by cyclic movement of the substrate, whether due to temperature or relative humidity changes or to vibration, is an important performance property. Factors affecting cyclic movement resistance include the type of encapsulant and its reinforcement, as well as the amplitude and frequency of the cycle.</td>
</tr>
</tbody>
</table>

Table A-6. Initial outline of performance statement for chemical resistance

<table>
<thead>
<tr>
<th>Requirement</th>
<th>The encapsulant shall be capable of withstanding normally encountered household fluids, such as those used for maintenance, cleaning and cooking, without damage.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Criterion</td>
<td>To be developed</td>
</tr>
<tr>
<td>Evaluation</td>
<td>To be developed</td>
</tr>
<tr>
<td>Commentary</td>
<td>To be developed</td>
</tr>
</tbody>
</table>
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Proposed Performance Criteria for Encapsulant Coatings for Lead-Based Paints I. Abrasion Resistance

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SUPPLEMENTARY NOTES

This study was conducted to develop preliminary performance criteria for encapsulants for lead-based paint. ASTM standards have been developed for encapsulants and data in the study may support revisions to these standards. Encapsulants are liquid-coating-based products installed over the surface of the lead-based paint to help minimize the hazards of lead-based paint in housing. This report, the first in a series on the performance criteria, describes the cure time and abrasion resistance of 10 commercial encapsulants. Six nonreinforced and four reinforced encapsulants were selected. Six were acrylic-resin based; others were epoxy-, polyester-, and polyurethane-resin based, and one was inorganic-cement-based with an acrylic binder. Two household paints were included as controls. Before conducting the abrasion tests, it was necessary to characterize the cure of the samples; that is, the change of the liquid-applied coating from a wet film to a solid film. Characterization of the cure was accomplished by following the glass transition temperature over time using dynamic mechanical analysis (DMA). The results indicated that seven encapsulants and the two paint samples showed little or no change in glass transition temperatures within four weeks or less after application; i.e., they were essentially fully cured. In contrast, three encapsulants displayed glass transition temperatures which were still increasing after at least 13 weeks, although the dry films exhibited no indications that they should not be tested after four weeks. The abrasion tests were conducted according to ASTM test method D 4060 for 2000 abrasion cycles in increments of 500 cycles. Four of the six nonreinforced encapsulants and all four reinforced encapsulants had better abrasion resistance than the paints. Based on the test results, the following preliminary performance criteria for abrasion resistance were proposed: (a) for nonreinforced encapsulants, specimens shall not lose, on the average, more than 20% of the film thicknesses when subjected to 1000 abrasion cycles, and (b) for reinforced encapsulants, no abrasion completely through the reinforcement shall occur, and the polymeric component shall totally fill all voids in the reinforcement after the specimen has been subjected to 1000 abrasion cycles; that is, the substrate shall be totally covered with polymeric component and reinforcement after abrasion. In both cases, the criterion is based on testing the encapsulant specimen at the minimum thickness recommended by the manufacturer.

KEY WORDS (MAXIMUM OF 9; 28 CHARACTERS AND SPACES EACH; SEPARATE WITH SEMICOLONS; ALPHABETIC ORDER; CAPITALIZE ONLY PROPER NAMES)

abrasion resistance; building technology; coatings; curing; dynamic mechanical analysis; encapsulants; glass transition temperature; lead-based paint; performance criteria

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