Perspectives on Weatherability Testing Of Automotive Coatings

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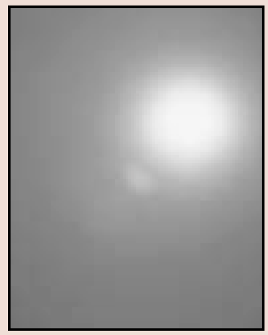

Automotive coatings continue to evolve rapidly in order to reduce the environmental impact of coating operations and to meet rising customer expectations for appearance and performance. The anticipated changes in chemistry, together with improved appearance retention and the need to respond rapidly to customer desires, have placed new challenges on weathering protocols. This paper discusses these issues and describes several methodologies that are likely to be part of the next generation of accelerated testing protocols. Of critical importance is the need to understand and minimize where possible sources of variability in testing that can lead to uncertain results and increased risk of failure in service.

INTRODUCTION

Weatherability evaluations occur during all stages of coating development and implementation.[1] Coating manufacturers and raw material suppliers screen new chemistries for potential weatherability. They also screen new additives to enhance performance. As coating systems are developed, suppliers establish "formulation windows" to ensure that a particular coating technology can be adjusted to meet specific customer needs without the risk of a decrease in weathering performance. End users are most concerned with verifying that a given coating meets all its customers' performance expectations. End users are also concerned with determining the effects of application on coating performance and in the case of automotive coatings insuring that the new colors that are introduced perform to expectations. Each of these needs poses different challenges to weatherability testing protocols. For example, screening new chemistries and additives requires that weathering information be generated as fast as possible (months). The information does not have to provide absolute measures of performance, but must give sound directional information (formulation or compound A is better than B). By contrast, end users generally can afford to wait a longer period (up to year for new colors and several years for new technologies); however, they do require a measure of absolute performance against all possible failure modes. In this paper, the improvements in weatherability test protocols necessary to insure long-term customer satisfaction are discussed.

Weatherability testing of automotive coatings generally involves a combination of outdoors and accelerated laboratory testing. When available, outdoor exposure results generally take precedence over accelerated test results because they are felt to be more indicative of in-service performance. Old style monocoat paint systems are generally weathered by slow, but steady, loss of gloss. In-service, gloss loss could be restored by waxing and polishing. The rate of gloss loss could be determined in one to two years of exposure in Florida allowing for the ready identification of problem resins and colors. Circumstances changed in the 1980s when use of basecoat/clearcoat systems began to be extensive. These coatings tend to lose gloss very slowly. Coating performance was also less sensitive to color change due to the protection of the color pigments by UV absorbers in the clearcoat. Short-term (one to two year) exposures in Florida rarely yielded useful information on long-term weatherability. Occasionally, very long exposures indicated the possibility of abrupt appearance changes resulting from either clearcoat cracking or peeling of one layer from another. New performance standards were implemented requiring up to five years of exposure in Florida. Of course, numerous formulation changes will likely be made to a technology from the time it is first developed and panels exposed outdoors to when it is finally implemented. The effects of these changes have to be

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evaluated using accelerated tests with confirmation from outdoor tests coming afterwards. This puts a more significant burden on accelerated testing protocols.

A wide variety of accelerated tests for weatherability have been developed including those based on high intensity sunlight, fluorescent bulb exposures, carbon arc, and xenon arc exposures.[2] Until recently, automotive accelerated weathering specifications were based on either UVB-313 (or FS-40) fluorescent bulbs or xenon arc light filtered with a quartz/borosilicate filter package. Although widely different in test protocols, these two tests methods achieve high levels of acceleration by employing UV light having wavelength emissions much shorter than observed outdoors (i.e., < 295 nm). As a result of the use of short-wavelength UV light, the acceleration factors and failure mechanisms for these tests have been found to be very sensitive to material type and even additive package.[3,4] This behavior is particularly important in that during the same time period that basecoat clearcoat systems became dominant, numerous different coating chemistries were introduced driven by the need to reduce solvent content and improve acid etch resistance. The correlations that were established between outdoor exposures and accelerated tests for old-style monocoats turned out to be invalid for these newer chemistries. In some cases, the accelerated test did not even reproduce in-service failure modes. In response to these problems, some suppliers have changed the light source used in accelerated testing protocols. For example, UVA-340 fluorescent bulbs, which more accurately match sunlight UV wavelength distributions (out to 360 nm), have replaced UVB-313 bulbs in some tests. Similarly, boro/boro filtered xenon light, which is a better match to sunlight than quartz/boro filtered light, is now more widely used. Concentrated sunlight exposures (EMMAQUA for example) have also found increased use in evaluating coatings.[3] Questions that have arisen include whether the changes in light sources are sufficient and whether or not accelerated tests can be used to reliably predict service life. This paper addresses the points posed above and suggests modifications to testing protocols aimed at improving our ability to predict service life. Critical to successful service life prediction using exposure test results is the need to understand all sources of variability in coating performance and test results. First, issues with setting basic requirements are addressed.

ESTABLISHING WEATHERING REQUIREMENTS

A critical step in establishing coating specifications is to determine customer expectations. This is not as easy as it sounds. It is especially difficult to establish quantitative expectations that can be translated into quantitative test requirements. Customers vary widely in their expectations. For example, while a typical customer expects automotive coatings to retain reasonable appearance for about 10 years in service, some customers only care about the first few years of service while others may care about the total life of the vehicle (which can be greater than 15 years). What constitutes reasonable appearance also varies dramatically from customer to customer. While all may agree that catastrophic peeling is not acceptable, they may not agree on levels of gloss loss, color change, or other changes in appearance. Solid statistical analyses of both customer expectations and failure modes will be necessary to derive improved testing requirements.

One approach to such an analysis involves attempting to calculate the cost of coating failure for different technologies and to compare that cost to the cost of using those technologies. This approach has a number of potential advantages. Coatings with different attributes can be compared on a total cost basis. The cost of painting can also be included and the effects of application changes on failure rates and total cost can be estimated. Currently the cost of any paint appearance change will be borne by either the vehicle or paint manufacturer (in warranty) or by the customer (in loss of resale or out-of-pocket repair). Ultimately, the cost incurred by the customer will come back to the manufacturer either in need to discount a product due to its perceived lower resale value or loss of future sales due to a loss of customer loyalty.

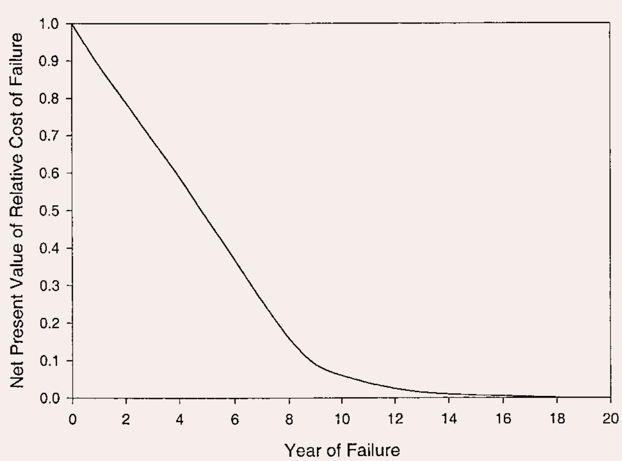

Figure 1—Hypothetical dependence of the net present value per unit of the relative cost of weathering related coating failures vs. the year in which the failure occurs.

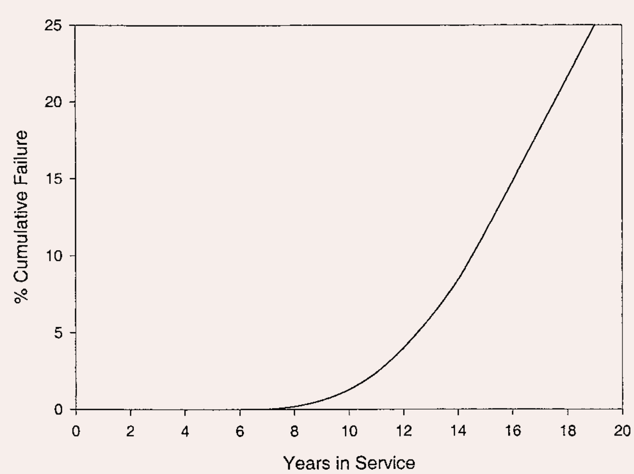

Figure 2—Cumulative failure vs. years in service for a hypothetical automotive coating technology in the United States.

While warranty costs are useful for evaluating early failure costs, they only provide information for a fraction of the service life of a typical vehicle (~15 years). It is by no means trivial to estimate the cost of any particular failure that might occur at a given time. Several factors need to be considered. First is the cost of repair. Catastrophic failures such as peeling or cracking usually require repainting to restore the finish. Gloss loss may simply be restored by polishing. Repainting ceases to be a reasonable option when the cost of repainting is significantly greater than the loss in resale caused by the coating failure. In this case, the cost of failure is the difference between the book value of the vehicle and its current resale value. The cost of the coating is an up front cost while the cost of failure is not incurred until after the coating fails so that its cost must be discounted. The longer the time-to-failure, the larger is the discount. Also, vehicles which are removed from service prior to coating failure do not contribute to the cost of coating failure. Thus, the net present value per unit produced of the cost of fixing a particular failure is expected to drop dramatically as the in-service failure time increases. Clearly, determination of the cost of failure and its variation with time requires detailed study. For the purposes of discussing improvements in weathering protocols necessary to improve service life prediction, a hypothetical relative cost-of-failure curve has been generated and is shown in *Figure* 1. The cost per vehicle of early failures is much higher than failures at long times in service.

Another factor in setting requirements is that the distribution of times-to-failure in service is very broad. For a particular coating technology, the distribution of times-to-failure in a given market region depends on a number of factors. The most important is the variation in harshness over the region. For most weathering related failures this translates into the variation in photooxidation, which in turn is primarily determined by variations in UV load, temperature, and humidity. The distribution in photooxidation rate for different markets has been calculated based on a cumulative damage model.[5] Another factor is customer use. Parking habits strongly influence UV dose. The variation in photooxidation rate with coating color (due to differences in part temperature caused by color as well as other factors) results in further broadening of the distribution of the rate of photooxidation for a particular coating technology. It has been estimated that dark colors photooxidize one and a half to two times faster than light colors.[5] Finally, variations in film thickness (and possibly cure) can affect the time-to-failure for some failure modes. For example, typical variations in clearcoat film thickness lead to ±20% variation in time to clearcoat delamination.[6] By combining all these distributions, it is possible to make reasonable estimates of the shape of the distribution function of time-to-failure for a given technology. A reasonable cumulative distribution function for weathering failures due to photooxidation in the United States is shown in *Figure* 2 for a hypothetical but representative coating.

If one had distributions such as those in *Figure* 2, they could be combined with the cost of failure in *Figure* 1 to determine the total cost of failure of a given technology. This cost could be added to the cost of the paint and paint application to determine the true cost of painting the vehicle. The whole purpose of outdoor and accelerated testing is ultimately to be able to estimate the failure distribution prior to implementation. This leads to the necessity of developing a relationship between the performance in a particular test and the actual in-service failure distribution. Since performance after so many years in Florida is the traditional measure of paint performance, one approach would be to relate the time-to-failure in Florida with in-service failure rates in other regions. Accelerated test requirements are then generated by estimating an acceleration factor and converting the years of Florida to hours of accelerated testing.

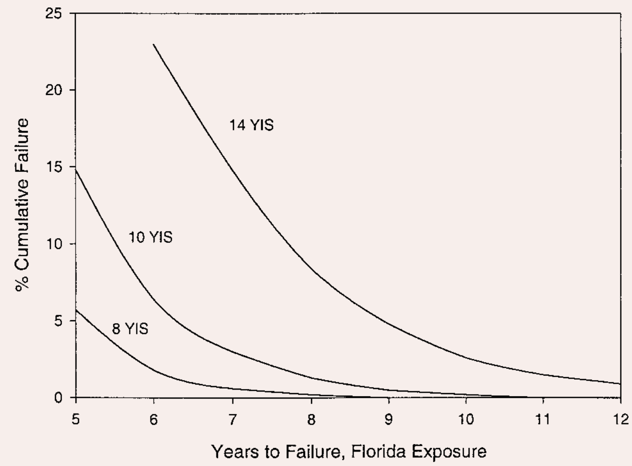

Figure 3—Cumulative failure at different years in service in the United States as a function of Florida weathering performance. Florida failure time is determined by the weakest color in the color palette.

Using the same model that was used to estimate the shape of the in-service distribution of photooxidation in *Figure* 2, it is possible to show that if all vehicles in the United States were painted with a coating system identical to a panel that fails in eight years of exposure to "average" Florida conditions then the extent of in-service failure after 10 years would be ~5%.[5] Europe, having a milder distribution of weathering conditions than does the United States , requires only five years of Florida durability to achieve the same level of in-service performance. As noted above, different colors in a given technology will have different levels of performance in Florida. If we require that the weakest color meet a particular standard (say eight years of Florida exposure) before failure, then the failure rate at 10 years in service for that technology would be 1-2% depending on color distribution. In fact, the failure rate distribution shown in *Figure* 2 was generated based on a coating system that meets this eight-year requirement for all colors. The predicted rates of failure at different times in service versus performance in Florida are shown in *Figure* 3. This model predicts that the in-service failure rate at any particular time increases dramatically as the time-to-failure in Florida decreases.

Combining *Figures* 1-3, it is possible to estimate the cost of paint failure as a function of performance in Florida, as shown in *Figure* 4. A given technology can have a fairly wide range of weathering performance through the use of different additive packages and other modifications. As a general rule, increased performance for a given technology implies increased cost. A typical dependence is also shown in *Figure* 4. By adding the failure cost to the initial

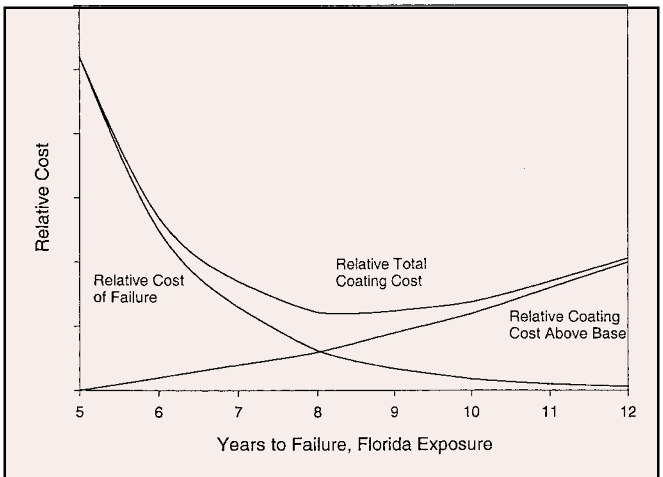

Figure 4—Relative cost of failure, relative coating cost, and total cost vs. Florida weathering performance for a hypothetical coating technology.

paint cost we obtain the total cost of the paint as a function of outdoor exposure performance (*Figure* 4). For this hypothetical coating, the optimum level of performance is eight to nine years Florida. The cost rises rapidly as performance drops below seven years due to both the higher cost of early failures and to the larger number of failures at any given time.

One critical factor that has to be recalled at this point is that the relationships used to derive *Figures* 2 and 3 were based on exposures to "long-term average" Florida conditions. In fact real exposures never reproduce those average conditions. The advantage of using the cost approach of *Figure* 4 is that it is possible to make reasonable risk assessments resulting from the uncertainty of weathering results. One important consequence of the asymmetry of *Figure* 4 is that relatively small errors in overestimating in-service performance can lead to large increases in total cost. For example, a 25% error in evaluating a coating (a reduction in performance of eight to six years) leads to a four-fold increase in failure cost. In this case, to minimize the possibility of higher cost due to an unexpected, premature failure, it is necessary to increase the performance requirement (build in a safety factor) from eight years Florida to 10 years Florida. This also adds to the initial coating cost. It is critical to understand and minimize the sources of uncertainty in weatherability testing.

Improving the Relationship Between Outdoor Testing and In-Service Performance

The key factors that lead to uncertainty in the relationship between outdoor test results and actual in-service performance are changes in material and application from test to in-service, variability in the outdoor exposure from one test period to another, failure to test a wide enough range of exposure conditions, and variations in in-service harshness. Typically, outdoor testing precedes in-service vehicle use by several years. During this time numerous changes are made to the formulation and the color palette. One of the functions of formulation development is the establishment of formulation windows and the evaluation of the effects of formulation changes on weathering. As noted earlier, accelerated tests are usually employed to determine the formulation window due to time considerations. The lack of correlation between accelerated tests and in-service performance can lead to poor formulation decisions. Improving accelerated test reliability (see below) lessens this risk. The use of analytical characterization tools to monitor subtle changes in performance resulting from these changes will also greatly improve formulation reliability.[1,7] Processing variations in film thickness and bake condition can also lead to significant variations in performance. Traditional long-term outdoor exposure testing utilized samples prepared under nominal conditions. Most failures occur first on samples at the extremes of process (peeling on thin films, cracking on thick films). While it is not always possible to anticipate the actual application processes used, it is important to evaluate the dependence of the performance of a given coating technology on relevant application variables. Such data could be used in evaluating tradeoffs between the costs of changes in a process that reduce variability and the decrease in failure costs resulting from the reduced variability.

The time-to-failure in Florida testing is often used as an absolute measure of coating weathering performance. Yet, it is well known that there is substantial variation in performance in Florida for identical coating systems.[8] Short-term variations in gloss loss rate of nominally identical coatings after approximately one year exposure in Florida are >30%.[9] While longer time exposures are expected to show less variation, they would appear to be large enough (>10%) to provoke concern about using exposure time in Florida as the absolute measure of performance. Traditionally, Florida exposure results have been compared to results for control panels of "known" durability. Use of control panels to compensate for variations in the weather loses some relevance during periods of rapidly changing technology and uncertainty in control performance just adds to the overall uncertainty of the estimate of performance. Another, more analytic, way to approach this problem is to keep track of all relevant outdoor variables and to measure exposure in terms of cumulative damage rather than years.[10] It is critical that the cumulative damage model that is used accurately reflects coating behavior. It is important to note that the variability in outdoor testing results is not solely a result of variations in average conditions. It is important to consider the effects of day-to-day fluctuations in temperature, humidity, and sun load on accumulated damage. Monte Carlo simulations have shown that there is substantial variation in harshness from run to run for nominally identical exposures.[11] For this reason a critical recommendation for outdoor testing is that sufficient and frequent measures of test condition be made. These include not just sun load, ambient temperature, humidity and rainfall, but also UV distributions, and actual part temperatures.[12]

Another source of variation of outdoor test results with in-service results is that a single site may not represent the harshest condition for all coatings or failure modes (note, this is equivalent to having multiple cumulative damage models). It is well known that different test sites are required to test different failure processes. For example, a number of sites are used to evaluate acid etch resistance and these sites are different from those used to evaluate weathering. Of particular concern for weathering is the dependence of wetness and humidity on degradation of different coatings. While Florida is the harshest North American environment for most coatings failures, Arizona has a higher solar and heat load than Florida and may be harsher for some systems.[5] By exposing samples in both Arizona and Florida, it is possible to minimize the uncertainty associated with the dependence of different coating systems and failure modes on moisture. Thus, the second recommendation for outdoor testing is that unless the sensitivity to moisture is known, samples should be exposed in both Florida and Arizona (or their equivalent).

The final source of uncertainty in predicting in-service coating performance from outdoor exposures is actual variability in service conditions. Of most concern is the variation in harsher regions of specific markets since this is where failures would first occur. Fortunately, the long-term variation in weather over such regions in a large market like North America or Europe is smaller than variations in specific test sites. In addition to variations about a mean, it is necessary to consider long-term trends in the mean values of critical variables. Two issues that should be considered are the effects of global warming and ozone depletion on the rate of weathering in different environments. For typical coatings, 1°C increase in temperature results in a 3-5% increase in photooxidation rate. Similarly, a 10% loss in ozone would result in a 5% increase in the photooxidation rate of typical coatings.[5] Trends in temperature and ground level UV should continue to be evaluated for their effect on weathering. Uncertainty in forecasting future weather conditions is unavoidable. A safety factor to account for the possibility that the next 10 years will be harsher than the last 10 is advisable.

Improving the Relationship Between Accelerated Testing and In-Service Performance

Accelerated test harshness can be characterized by the acceleration factor between exposure results in the test and a representative outdoor site.* Accelerated tests suffer from uncertainty in acceleration factor. As is the case for outdoor exposures, any significant uncertainty in acceleration factor (> ± 10%) leads to the necessity of adding increasingly large safety factors to the accelerated test requirement. The two basic causes of uncertainty in accelerated test results are variability resulting from differences in exposure conditions over time or from chamber-to-chamber and the combination of variations in material sensitivity and differences in the accelerated exposure conditions relative to outdoors.[13] Round robin testing of accelerated test chambers reveals chamber-to-chamber variations >25% though significant efforts have been made to improve reproducibility.[14] The chamber-to-chamber variability is at least as large as the variability associated with outdoor test results. It is clearly important to understand where this variability comes from and to minimize it. It seems likely that a significant fraction of the variability is due to variations in the light intensity on different samples and the temperatures of different samples. Sample temperatures are not generally measured in accelerated testing. Rather, temperature control is accomplished through a combination of air temperature and black panel temperature. The actual sample temperature is determined not only by these temperatures, but also by the color of the sample, the nature of the substrate, the light intensity at the sample (higher than average light intensities likely will lead to higher than average sample temperatures), by the colors of nearby samples and loading of the chamber, and by variations in air flow around the sample. A five percent variation in light intensity could lead to a 1-2°C difference in sample temperature. Together these factors could by themselves account for a 10-15% variation in photooxidation rate from sample-to-sample. It is clearly important to monitor all exposure variables at the samples. This data can be used to redesign test chambers to improve uniformity. It can also be used to correct for unavoidable residual variability using a cumulative damage model in a manner similar to that proposed for outdoor exposure. Another source of variability is variation in wavelength distribution. Variation in filters and aging of filters can lead to different intensity distributions. This can have a substantial impact on the rate of photooxidation.

Another source of uncertainty in accelerated test results has to do with the fact that the accelerated exposure conditions are different from the weighted average outdoor conditions at a particular site and that different materials have different dependencies on those conditions.[13] This leads to different apparent acceleration factors for different materials. The most obvious difference in exposure between outdoors and accelerated tests is the wavelength distribution of the light exposure. With the exception of EMMAQUA exposure which uses focused sunlight, no commonly used accelerated test light source accurately matches the spectral distribution of the solar spectrum. Most light sources have emissions at wavelengths shorter than that for sunlight (<295nm). Some sources ignore the long wavelength UV and visible part of the spectrum. Short wavelength UV light can induce chemical changes in some coating systems that do not occur at higher wavelength.[3] Even in cases where the apparent degradation chemistry is similar, the presence of short wavelength UV light often causes changes in acceleration factors for very similar coating materials.[4] This is presumably due to the fact that quantum efficiencies are highest at short wavelength and vary significantly with coating composition. It is not practical to measure quantum efficiencies for all samples. To minimize this source of error, it is critical that the accelerated test light source not have significant emissions below 295 nm. The question then arises as to how accurately the accelerated test light source has to match the solar spectrum over the entire wavelength range. This is not a trivial question since the UV wavelength distribution varies with season, cloud cover, latitude, and stratospheric ozone. The most reasonable suggestion would seem to be to match a "worst-case" UV distribution such as found in a noon Miami summer exposure.[15] It should also be noted that some failure modes are sensitive to light into the visible part of the spectrum. For these failure modes, it is important to match the outdoor distribution over both the UV and visible regions. It does appear to be possible to accelerate degradation reliably by increasing the intensity of light over ambient conditions. Use of high light intensities may make temperature control more difficult, however.

*Typically, Florida is used as the benchmark. Given the variability in outdoor conditions, an accurate measure of acceleration factor requires use of long-term average Florida exposure conditions.

Another source of error caused by material sensitivity results from temperature differences between the accelerated test and a weighted average outdoor temperature. Different materials will have different activation energies for photooxidation. If the accelerated test temperature is only 10°C greater than the "weighted average" temperature of the outdoor exposure, a difference in activation energy between 5 and 10 kcal/mole (a typical range) leads to a 24% difference in the acceleration factor. Since accelerated test conditions normally maximize acceleration by attempting to match peak conditions, the accelerated test temperature will likely always be significantly above a "weighted-average" value (which depends not only on outdoor conditions but also on the activation energy). For this reason it is necessary to measure the activation energy for photooxidation for all coating systems. Ambient humidity conditions vary widely (Florida versus Arizona). Laboratory conditions are often much harsher than that observed outdoors (50°C condensing humidity for example). If the dependence of weathering degradation on humidity or moisture is not known, then it is necessary to measure the rate of weathering under conditions that reflect the harshest wet and dry conditions that the system will see. Another important consideration is to match the change in temperature and humidity typically encountered in a daily cycle. This is necessary to provide reasonable stresses on samples in order to induce the correct failures.[16] It may be possible to weather the coating under constant conditions and periodically remove the coating from exposure and perform a cyclic stress test that mimics outdoor stresses.

One final consideration for accelerated testing is the role of acid deposition on overall weathering performance. Until recently, acid-etch and weathering were treated as separate phenomena that could be evaluated in separate exposures. It has been suggested that more reliable results can be obtained by adding acid deposition to accelerated test protocols.[17] Further studies are needed to determine whether acid attack and photooxidation are so intimately related that it is necessary to combine the exposures or whether it is possible to continue to evaluate materials in separate tests.

CONCLUSION

While accelerated test chambers have significantly advanced over the years, further improvements are necessary because the challenge of predicting long-term weathering performance has become increasingly difficult as expectations increase and technologies proliferate. Improved methodologies to estimate the cost of failure need to be developed in order to optimize the value of coating systems. Increased use of analytical methods to assess small changes in weathering associated with formulation changes and process variations will improve the comparison of results on different coatings.

A critical factor in improving both outdoor and accelerated test protocols is reducing the uncertainty associated with variability in test results. Current test uncertainty requires adding a substantial safety margin into the weathering requirement to minimize the risk of premature failure. This leads not only to longer test times and higher test costs, but also to higher material costs due to the need to "over-engineer" the material. Measurements of actual sample exposure conditions in-service, in outdoor exposures, and in accelerated tests are critical to move from time exposures to exposures based on cumulative damage. Such measurements will assist in exposure variability reduction in accelerated test chambers. The measurements will also facilitate the development of robust cumulative damage models. Exposures at multiple sites and multiple test conditions are necessary to insure that the cumulative failure models capture all failure modes and material sensitivities for new technologies. Although these new test protocols will be more complex than traditional tests, the potential for cost savings due to risk reduction is significant.

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