In Situ Measurement of Compression Set in Building Sealants During Outdoor Aging

ABSTRACT: The durability of sealants is dictated by many factors such as joint design, surface preparation, application, formulation, joint movement, and weather. Among these factors the link between formulation (material behavior) and weathering durability is difficult to assess in short term tests. We attempt to address this challenge by monitoring changes in apparent modulus during exposure to outdoor weathering and cyclic strain. This is accomplished via custom built systems that apply cyclic strain to 16 samples simultaneously via programs that simulate wood (cold compression) and concrete/metal (hot compression) construction materials. A key finding of the research presented here is that changes in apparent modulus are primarily driven by underlying changes in the compression set, a potentially critical contributor to stress in structures during rapid temperature changes. Detection of the compression set is made possible by the in situ material property assessments used in this research. Aging tests that rely on offline evaluation of property changes may miss or underestimate this effect on the sealant's stiffness due to time delay and/or losing track of the original zero stress–zero strain state.

KEYWORDS: Building sealant, compression set, durability, outdoor aging, strain cycling, apparent modulus

Introduction

Sealants are a critical part of a structure’s moisture and weather barrier envelope. Understanding and predicting the service life expectancy of sealants is necessary to prevent serious damage to the cosmetic and structural integrity of buildings and structures. Given this well recognized need, several challenges prohibit the direct assessment of such knowledge. One of the primary challenges is the difficulty in assessing the critical combinations of factors (environmental, displacement, fatigue, formulation, material property, etc.) that determine a sealant’s durability in a well characterized service condition. Secondly, pass–fail tests address lifetime prediction by exposing materials to conditions much more severe than the conditions of use. Severe conditions are necessary based on a factor of safety approach and the short testing times desired. The approach taken in this research program is to conduct simultaneous outdoor weathering and cyclic strain aging of sealants while recording environmental conditions and changes in modulus via in situ measurements. This data set is intended to provide the means for obtaining a dosage versus damage model that may facilitate better predictions of service life for a given application and environment.

The outdoor exposure takes place in Madison, Wisconsin via a custom built computer controlled instrument named the Badger IIIa [1]. The advantage of this approach is that it provides active feedback on incremental material property changes as a function of weathering and cyclic strain dosage. The disadvantage of cyclic testing is having sufficient sample throughput to provide statistically significant data. The instrument we designed addresses this issue by applying controlled displacement to 16 sealant specimens simultaneously as a function of temperature. The applied displacement is computer controlled such that custom functions can be developed to fit a service environment. The functions are principally...
temperature based. Consequently, the specimens experience instantaneous and daily diurnal displacement
cycles of a controlled magnitude and rate induced by temperature changes within set strain limits. Cycling
is stopped once per week to run a stress relaxation profile allowing the calculation of the sealant’s apparent
modulus [2].

Compression set is a well-known phenomenon defined as the fraction of applied compressive strain
remaining in the sealant after full compressive strain is removed. This, of course, is a time-dependent phe-
nomenon, since upon removal of the compressive strain the sealant moves toward the original dimensions
more slowly over time and may never recover its original shape. In most sealant testing, specimens are
allowed to relax between the compression application and the measurement of properties. This has a very
practical benefit, removing a time-dependent factor, making property measurements more repeatable. The
sacrifice, however, is that information about the contribution of transient compression set to sealant prop-
erties is lost.

Herein we describe the results from two sealants exposed to five months of hot compression cycling
and outdoor weathering on an instrument that was designed to enable the measurement of stress relaxation
behavior and the calculation of apparent modulus immediately after stopping cyclic strain exposure. This
test protocol more closely simulates exposure conditions on buildings with exteriors that respond quickly
to changes in temperature.

Experiment

Specimens: The sealant specimens are provided by the National Institute for Standards and Technology
(NIST) in Gaithersburg, MD via their consortium of sealant companies who fabricate the specimens [3].
All chemical information (formulations, base chemistry, fillers, etc.) about the samples is hidden from the
Forest Products Laboratory and NIST due to the blind nature of this study. Additionally, chemical analysis
of the samples is not permitted. The sealants consist of Consortia C and ASTM round robin B (ASTM B).
The Consortia C sealant is specially formulated to fail earlier than commercially available sealants. Per
the limitations of this study, it is not known what formulation attribute was changed in order to achieve
this intent. The ASTM B sealant is commercially available with a $625\%$ movement rating. The speci-
mens consist of a pair of anodized 6063 aluminum blocks ($12.7 \times 12.7 \times 76.2$ mm) bonded together with
sealant in the form of a $12.7 \times 12.7 \times 50.8$ mm bond line cured in conformance with ASTM C719-93 [4].

Strain Cycling: The custom built test machine shown in Fig. 1 consists of two parallel aluminum
I-beams with up to 18 sealant specimens fixtured between them. The I-beams are driven by two captive
stepper linear actuators (size 34, Hayden Kerk, Waterbury, CT) whose position is monitored by two linear
variable differential transformers (model HSD 750 250-010, Macro Sensors, Pennsauken, NJ). The pro-
grammed displacement follows the temperature profile of polyvinylchloride (PVC) based durability
engines in operation at NIST. The displacement ($\Delta$, cm) versus temperature ($T$, °C) equation for such
engines is given in Eq 1. Strain gauges were not applied to individual specimens. Thus, the values reported
as strain are based upon the linear variable differential transformer position of the I-beam and are
approximate

$$\Delta = -(T - 4.5)/105$$

(1)

FIG. 1—Photograph of Badger IIIa with 18 sealant specimens.
The temperature is recorded from a thermocouple embedded in a piece of PVC pipe exposed to solar irradiation. The hot compression cycling displacement boundaries were set in this experiment such that +25 % strain occurred at −29°C and −25 % strain at 38°C, corresponding to climate norms for the Wisconsin test site. The load response to the applied displacement is independently measured for each specimen by S-type load cells (model SSM-AJ-250, Interface, Scottsdale, AZ).

Motion control, load cell conditioning, and data acquisition during testing was accomplished via a National Instruments (Austin, TX) Compact RIO (cRIO)-9073 integrated 266 MHz real time controller. Once per week, displacement cycling was stopped and an apparent modulus cycle was run to check for changes in modulus as a result of weather and displacement aging; see Fig. 2. The cycle consisted of two peaks of approximately 15 % strain that act to remove the Mullins effects from the sealants followed by a 10 % estimated strain stress relaxation period [5]. The 15 % peaks remove any effects of filler bonds and secondary bonds that contribute to non-reversible stress-strain behavior. Thus, the stress relaxation period occurs at a lower strain than the first two peaks and is free of these effects.

The apparent modulus (Ea) is determined using a stress relaxation test proposed by NIST as a new ASTM International sealant test method [2]. The Ea is calculated via Eq 2, where t is time, \( \lambda \) is the extension ratio, L is the load, W is the specimen width, and B is the specimen thickness. Here, \( \lambda \) is calculated using Eq 3 where \( \Delta \) is the displacement and h is the specimen height. This methodology is taken from the statistical theory of rubber elasticity [6,7]

\[
E_a (t, \lambda) = \frac{3L(t)}{(WB(\lambda - \lambda^{-2}))}
\]

\[
\lambda = 1 + \Delta/h
\]

Weather: Solar irradiance, temperature, and relative humidity were recorded during outdoor exposure. Spectral irradiance is recorded via a Smithsonian SERC 18 scanning radiometer model, SR-18. This model records the UVB spectral irradiance in mW/(m²* nm) and the Smithsonian uses a radiative transfer model to calculate the UVA and visible bands. Air temperature and relative humidity are recorded via a weather station. The test started in March and concluded in August of 2010. A lighting strike in April damaged the equipment resulting in the loss of approximately one month of weather data.

Results and Discussion

The cyclic loading applied to the sealant specimens due to a temperature change during the third week of July 2010 is shown in Fig. 3 for the ASTM B samples. Offsets between the three ASTM B replicates result from differences in the specimens as confirmed by offline tension and compression tests. The temperature change is the mirror image of the stress response due to outdoor exposure being run in hot compression mode. This mode simulates the displacement–temperature behavior of concrete or metal structures. This
plot is typical of the exposure for all specimens with the magnitude and rate of change dependent on the weather. The variation in stress and temperature at the peak values is caused by a local variation in cloud cover inducing a rapid change in the temperature of the PVC pipe section acting to drive the computer controlled displacement.

The total recorded ultraviolet A band (UVA, 315–399 nm) dosage from March 1 to Aug. 7, 2010 was 130 MJ/m². This value does not represent the total dosage due to a lightning strike in April where data was lost. It is estimated that an additional 30–40 MJ/m² could be added to the total dosage. The UVA band was tracked as opposed to the total UV band due to the detected ultraviolet B (UVB) dosage being of low intensity by comparison to the UVA values. The daily UVA dosage during aging varied considerably due to cloud cover. Overall, the accumulated UVA dosage increased linearly with time. The daily average air temperature and relative humidity values recorded at the Wisconsin test site are shown in Fig. 4. The spring temperature and humidity values varied widely. The average temperature started at below 0°C in early March and rose to 20°C by the end of the month with several cold periods in between. The average humidity varied considerably from 50 to 100% during this time frame. Data loss due to the April lightning

FIG. 3—Stress (left axis) and bulk temperature (right axis) versus time cycles that were recorded.

FIG. 4—Air temperatures (left axis) and RH (right axis) recorded at the Madison, WI exposure site from March through September of 2010.
strike is responsible for the gap in the data from April 6 to April 21. The transition from spring to summer took place in May via a relatively uniform rise in average temperatures from 5 to 25°C. The daily average relative humidity (RH) during May continued to vary widely from 50 to 100 %. Starting in early June and continuing to August average temperatures achieved and maintained a consistent value of approximately 25°C. The RH during this time similarly became more consistent, ranging from 70–100 % with values clustered around an average of approximately 90 %. Comparing recorded weather data during the testing period with historical averages revealed that the high and low temperatures were above normal average high and low temperatures from March through August [8]. High temperatures recorded in May were near record highs [8]. The precipitation was double the normal amount during June and July 2010 for the south central region of Wisconsin that includes the Madison test site [8].

The mean strain ($e_m$) and daily strain ratio ($e_r$) resulting from the PVC temperature change (Eq (1)) are plotted in Figs. 5 and 6. The strain ratio and mean strain were calculated via Eqs. (4) and (5), respectively, where $e_{\text{min}}$ is the day’s minimum strain and $e_{\text{max}}$ is the day’s maximum compressive strain

$$e_r = e_{\text{min}} / e_{\text{max}}$$

$$e_m = (e_{\text{max}} + e_{\text{min}}) / 2$$

The daily values of $e_{\text{min}}$ and $e_{\text{max}}$ are also plotted in Fig. 5 where they are indicated as bars radiating from the daily mean strain value. Not surprisingly, the mean strain values correspond to the average air temperature pattern shown in Fig. 3. The effect of solar heating variation due to cloud cover changes is reflected in the difference and variation in the maximum strain values shown in Fig. 5. The minimum strain typically occurred at night and hence is less variable on a day to day basis and is more in correspondence to the air temperature. The mean strain and max/min values vary considerably from April through mid-May. During this time, mean strain, minimum, and maximum values varied from 0 to $-11 \%$, 5 to $-6 \%$, and $-3$ to $-20 \%$, respectively. Thus, there are day to day transitions from compression to tension strain exposure. As with the air temperature, the mean strain dropped during May from $-5$ to $-20 \%$ with a recovery to $-13 \%$ at the end of the month. The mean strain and its daily variability decreased as the temperature rose towards a relatively uniform value of $-15 \%$ strain starting in June and gradually decreased though August. The minimum strain made a transition during this time to values less than $-5 \%$ strain. Thus, the sealants experienced a constant state of compressive stress during the summer. The applied strain ratio increased during the seasonal transition from spring to summer and its variability

![FIG. 5—Daily mean strain resulting from the diurnal PVC temperature change with error bars indicating the strain differential for each day.](image-url)
between days decreased. Both trends are indicative of decreased daily temperature swings and hence, more stable strain patterns applied to the sealants during the summer. Additionally, the rising strain ratio reveals that during the summer the sealants are carrying more strain.

The strain ratio shown in Fig. 6, is commonly prescribed in uniform continuous cyclic fatigue testing as a means to set the predominate mode of exposure. Specifically, high values indicate that the samples will carry a large amount of strain during the fatigue exposure. This results in a combined fatigue and stress relaxation exposure in controlled strain testing, as used here, or fatigue and creep exposure in controlled load testing. Low strain ratio values induce a large delta strain in the samples during each cycle. Such cycles can result in temperature build up (not expected in this study), and an acceleration of any hysteresis based damage mechanisms such as filler particle debonding, the Mullins effect, crazing, delamination, crack formation and growth, etc. The daily extremes represented in Fig. 6 are quite variable from March to mid-May indicating no consistent trend towards stress relaxation or hysteresis based exposure. The mean strain in Fig. 5 reveals a period of relatively low compression strain with variation from 0 to 11% during this same period. This changed dramatically in mid-May to high positive strain ratios due to both extremes being compressive. This continued through August with a significantly lower variation.

The Ea cycle described in the experimental section is run on a weekly basis in order to track changes in apparent modulus during aging. The Badger’s computer controlled stepper motors allow such measurements to take place without having to remove the specimens. The stress versus time responses from an Ea cycle run on Apr. 27, 2010 are shown in Figs. 7 and 8 for ASTM B and Consortia C sealants, respectively. During this time frame the Badger did not meet the programmed strain level of 15% for the Mullins peaks, yet the peaks did sufficiently exceed the 10% strain for the stress relaxation period used to calculate Ea. The error bars in these plots are the standard deviations among the sealant replicates. As previously stated, offline testing confirmed that the variations in ASTM B are due to sample variations. The offline tests on the Consortia C replicates revealed them to be relatively uniform. Therefore, the variation seen here is due to variations in the sample shape or shimming of the specimens during installation on the Badger resulting in slightly different applied strain values. The difference in error bar density is due to a difference in the sampling rate during the Ea cycle. The time gaps after each Mullins peak are prescribed to be zero strain, allowing time for the samples to relax to zero stress, thereby alleviating any dynamic effects. Here, both sealants do not quite make it to zero stress at zero strain before the stress relaxation period begins.

The Ea versus stress relaxation time curves from April 13 to Aug. 3, 2010 are shown in Figs. 9 and 10 for the ASTM B and Consortia C sealants, respectively. A power law fit to the data resulted in high

![Graph](image-url)
correlation coefficients and two parameters. The first parameter, the intercept at the Ea axis, acts as a good indication of the change in the overall stiffness of the sealants. The second parameter, an exponent, represents the time dependency of the sealant and how it changes during aging. Figure 9 shows that the Ea value at 5.8 ks increases by 62 % over the course of the experiment. The time dependent exponent decreased by 39 % indicating that the sealant exhibited less viscous behavior after aging.

Figure 10 shows that the Consortia C sealants underwent a 30 % increase in Ea at 5.8 ks as a result of outdoor aging. The increase in stiffness does not appear to be linear with aging time since the Ea values calculated on August 3 are lower than the values in June and late July. Such a phenomenon does not agree with the general concept of irreversible degradation or consumption of the chemical bond or other formulation additive. Since the formulations are not known to us and we are prohibited from chemical investigations, we cannot specifically comment on the chemical mechanisms that may be at work here. The power law fit exponent for Consortia C decreases by 42 % due to outdoor aging representing less time dependency and less viscous behavior.

![FIG. 7—Average stress for the three ASTM B sealant specimens resulting from the Ea cycle completed on Apr. 27, 2010.](image1)

![FIG. 8—Average stress for the three Consortia C sealant specimens resulting from the Ea cycle completed on Apr. 27, 2010.](image2)
The % change in \(E_a\) at 5.8 ks is plotted in Fig. 11 as a function of aging time for both sealants. Overall, there is a significant increase in \(E_a\) with time with occasional periods of reduction or recovery in \(E_a\). Both sealants have increases and recovery in \(E_a\) that appear to be coincident in time indicating that the aging conditions applied to the sealants are inducing a similar type of change in the sealants. Also plotted in Fig. 11 is the strain ratio applied to the sealants during the aging time. The rise in \(E_a\) coincides with the transition in strain ratio to values greater than 0.2 where the samples begin experiencing compression 24 h a day. Comparing the change in \(E_a\) versus the applied strain ratio indicates a generally increasing \(E_a\) with strain ratio values at 0.2 and higher. Notable periods of decreasing \(E_a\) occur in contrast to this observation.

These observations led us to search for other factors that may affect \(E_a\). This revealed that the non-zero stress level at zero strain observed in Figs. 7 and 8 as a fraction of the stress relaxation stress grows with aging time to become a significant fraction of the stress relaxation stress. This acts to unintentionally raise the starting stress for the stress relaxation period resulting in higher and higher \(E_a\) values. Since the non-zero stress at zero strain is positive, it is the result of a residual compressive strain in the sealants remaining from hot compression aging exposure. This effect is commonly referred to as the compression

**FIG. 9**—Stress relaxation behavior of the ASTM B sealant from April 13 to Aug. 3, 2010.

**FIG. 10**—Stress relaxation behavior of the Consortia C sealant from April 13 to Aug. 3, 2010.
set. Figures 7 and 8 provide some evidence to support the hypothesis that the tensile load resulting from the compression set does relax with time. Unfortunately, the Mullins portion of the $E_a$ cycle used during this research did not hold at zero strain long enough to allow an accurate measurement of compression set relaxation time. The compression set value prior to the start of the stress relaxation period can instead be used to correct the $E_a$ values calculated soon after the stress relaxation period has started. Figures 12 and 13 plot for sealants ASTM B and Consortia C, respectively, the compression set stress, the average stress relaxation stress at 100 s, and the corrected average stress relaxation stress at 100 s. The average stress relaxation stress are the values that would be used to calculate the apparent modulus at 100 s, $E_a(100)$. Subtracting the compression set stress from this value results in the corrected average stress relaxation stress at 100 s. Comparing the change in compression set stress with the stress relaxation stress over aging time makes it clear that the principal driving force behind the change in stress relaxation stress and the $E_a$ values calculated from them is the compression set stress. It is not clear whether the compression set is temporary or permanent from these results. The contribution of the compression set stress is subtracted to yield a corrected stress relaxation at 100 s value. Figure 12 plots the corrected value for ASTM B versus

FIG. 12—Compression set stress, average stress relaxation stress at 100 s, and corrected average stress relaxation stress for ASTM B.
aging time. This plot shows only minor deviations through Aug. 3 from the value measured on April 13. Applying the same analysis to the Consortia C sealant, Fig. 13 reveals a gradual decrease in corrected stress relaxation stress from April 13 to Aug. 3 to a value that is approximately 14.6% lower. Given the variation among the Consortia C replicates, this result does not appear to translate to a significant change in stiffness calculated from this value. In short, after removing compression set effects from ASTM B and Consortia C sealants, we could detect no significant change in stiffness during aging.

The change in the time dependent exponent of stress relaxation observed over the course of the experiment could also be explained by the compression set. Since some of the stress applied to the specimens originates with the compression set, the stress relaxation rate would be a combination of the stress relaxation of the applied load and the relaxation of the compression set. Even the small compression set apparent following the Mullins peaks in Figs. 7 and 8 suggest that the compression set relaxation is slower than the applied load relaxation that follows. In other words, the time dependent exponent for the relaxation of the compression set is smaller than the value for the applied load, and so the combined (measured) value decreases as the contribution of the compression set increases over the course of the experiment. There is also the possibility that a fraction of the compression set is permanent such that its overall relaxation halts prematurely.

While the specimens did not undergo a definitive change in stiffness during aging, the compression set has real implications on actual performance. Because of compression set, the effective stiffness of the sealants did increase and would act to restrict the movement of attached substrates to a proportional degree as if the sealant’s modulus had indeed increased. Thus, similar sealants bonded to metal or cement substrates would impart greater stresses to the bond area after hot compression induced set than would be predicted by the original properties of the sealant. Such high stresses could precipitate failure well in advance of what short term accelerated testing may have predicted. Outdoor exposure tests that do not determine apparent modulus in situ such as the Badgers are more likely to miss this effect due to relaxation after sample recovery and before offline testing is completed and/or if reference to the original zero strain/stress point is not tracked.

The measurement rate of the compression set and the severity of its contribution should be matched to the rate of movement of the building envelope. If a building moves very slowly, the finding presented here may not be important because the compression set may have time to relax during building movement, provided it is not permanent. Metal exterior cladding systems, however, have the potential to move very quickly. It is conceivable that during the summer, the sealants joining the metal skin of a building experience long periods of compression at high temperature, causing compression set. The arrival of a thunderstorm, and the wetting of the surface from rain, however, can cause a precipitous drop in surface temperature and hence, a fast opening of the joint. It is this kind of situation where the increased apparent stiffness from compression set will most likely exceed the bond strength of the sealant and result in failure.

FIG. 13—Compression set stress, average stress relaxation stress at 100 s, and corrected average stress relaxation stress for Consortia C.
Clearly, sealants on highly absorbing (dark), thin cladding systems with high thermal transfer and expansion coefficients, such as metal skins, are most vulnerable to this kind of problem. Sealants intended for this kind of application should be tested to determine the extent and rate of recovery from compression set. When choosing a sealant, these values should be compared to the potential rate of the temperature change of the skin and hence the rate of movement of the joint.

Conclusions

The methodology and instrumentation developed and implemented in this experiment allowed the successful tracking of environmental exposure and strain dosage factors and the progressive change in sealant stiffness with reference to the original zero stress/strain state. The stiffness increases observed here are predominately, if not completely, due to a progressive compression set inducement. This gradual process creates the appearance of increasing stiffness in our strain controlled modulus measurements via a non-zero stress at the programmed or original zero strain–zero stress state. This process can be seen as an artifact that interferes with attempts to track real time changes in sealant properties during aging. An alternative view is that this is a real phenomenon that takes place in buildings and structures during prolonged high temperatures. A rapid transition to cooler weather after prolonged compression may result in exceeding the failure stress of the sealants due to unexpectedly high strain from compression set. If the sealant or its bond is already compromised due to aging or other factors, failure could result from such a combined effect. Therefore, the compression set may be a key factor in a sealant’s durability and warrants further study. The methods used in this study are ideally suited to its detection and the monitoring of its effects. Short term accelerated weathering or outdoor aging approaches that are not long enough in duration to induce compression set or do not allow for in situ measurement of compression set and its effect on apparent modulus may miss or underestimate this important factor in service life prediction.

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