DURABILITY OF SEALANTS EXPOSED TO OUTDOOR WEATHERING AND HOT COMPRESSION CYCLES

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Introduction

Sealants play an important role in weatherproofing structures by filling gaps and preventing air and water intrusion. When incorrectly selected or improperly applied, they may fail quickly, compromising durability of the structure. To ensure reliability and prevent the need for costly repairs to structures, it is necessary to measure durability and predict life expectancy of sealants. Our approach to these challenges is to conduct accelerated testing per designed parameters to populate a predictive model and test this model via outdoor aging with prescribed cyclic displacements and *in situ* modulus measurements. Accelerated weathering is conducted in environmental chambers connected to an integrating sphere irradiation source at the National Institute of Standards and Technology (NIST). Outdoor durability testing is conducted at the Forest Products Laboratory (FPL) in Madison, WI, with a custom-built computer-controlled instrument.[1] This instrument applies controlled displacement to 16 sealant specimens simultaneously as a function of temperature. Consequently, specimens experience a daily displacement cycle induced by the diurnal temperature change within set strain limits. Cycling is stopped once a week to run a stress relaxation profile allowing measurement of the sealants' apparent modulus.[2] Weather is recorded during exposure such that a full set of dosage factors (solar irradiance, temperature, % RH, displacement) is captured and compared to changes in modulus.

Here we describe results from two sealants exposed to five consecutive months of hot compression cycling and outdoor weathering.

Experimental

Specimens: Sealant specimens were provided by an NIST consortium of sealant companies that fabricated the specimens. The two sealants tested in this study are consortia C and ASTM round robin B. The consortia C sealant is specially formulated to fail earlier than commercially available sealants. The ASTM B sealant is commercially available with a ±25% strain rating. All chemical information (formulations, base chemistry, fillers etc) about the samples was hidden from FPL and NIST because of the blind nature of this study. Additionally, chemical analysis of samples is not permitted. The adherends consist of anodized 6063 aluminum blocks (12.7×12.7×76.2 mm) bonded together with sealant in the form of a 12.7×12.7×50.8 mm bond line cured in conformance with ASTM C719.[3]

Strain Cycling: The test machine, Badger IIIa (Figure 1), consists of two parallel aluminum I-beams with up to 16 sealant specimens fixtured between them. The I-beams are driven by two Hayden Kerk size 34 captive stepper linear actuators whose position is monitored by two linear variable differential transformers (LVDT) from Macro Sensors model HSD 750 250-010. The programmed displacement follows the temperature profile of polyvinylchloride-(PVC-) based durability engines in operation at NIST and imposes displacement on all sealant specimens simultaneously. The displacement (Δ, cm) versus temperature (T, °C) equation for such engines is given in Equation (1).

$$\Delta = -((9/5)T - 8)/1219 \qquad (1)$$

Temperature was taken from a thermocouple embedded in a piece of PVC pipe exposed to solar irradiation. Hot compression cycling displacement boundaries were set in this experiment such that +25% strain occurred at –29°F and –25% strain at 38°C, corresponding to climate norms for the Wisconsin test site. Load response to the applied displacement is measured for each specimen by S-type load cells (Interface model SSM-AJ-250).

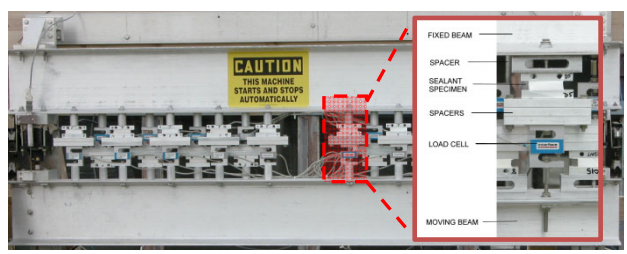

Figure 1: Photograph of Badger IIIa with sealant specimens. The inset image is a close up of one assembly.

Motion control, load cell conditioning, and data acquisition during testing were accomplished with a National Instruments Compact RIO (cRIO)-9073 integrated 266 MHz real-time controller.

Once a week displacement cycling was stopped and an apparent modulus cycle was run to check for changes in modulus resulting from weather and cyclic displacement aging. The cycle consisted of two peaks of approximately 15% strain that act to remove Mullins effects from the sealants and a 10% estimated strain stress relaxation period.[2,5,6] The de-Mullins peaks are intended to remove any effects of filler bonds and secondary bonds that contribute to non-reversible load-displacement 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

sealant test method.[2,4] Ea is calculated with Equation (2), where t is time, λ is extension ratio, L is the load, W is initial specimen width, and B is specimen thickness. λ is calculated using Equation (3) where h is the initial specimen height. This methodology is taken from the statistical theory of rubber elasticity.[5,6]

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

$$\lambda = 1 + {}^{\Delta}/_{h} \tag{3}$$

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

Results and Discussion

Figure 2 shows cyclic loading applied to sealant specimens during the third week of July 2010 for the ASTM B samples. The plot shows cyclic changes in stress and the bulk temperature during a week of exposure. Offset can be seen between the stress responses of B-6/B-7 and B-5. This results primarily from differences in the specimens as confirmed by offline Instron tests. Note that the trend in diurnal temperature change is opposite that of load response as the instrument was run in hot compression mode. This plot is typical of the exposure for all specimens with magnitude and rate of change dependent on the whims of Wisconsin weather. As discovered previously by Williams, Wisconsin weather patterns are quite varied: during a 3-year period, no day had the same temperature pattern.[1]

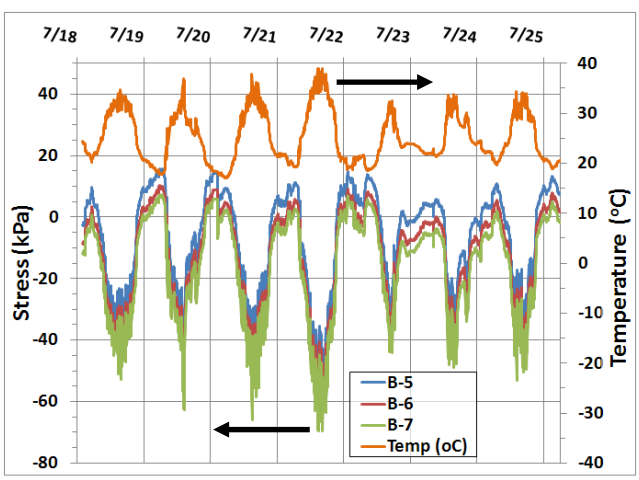

Figure 2. Load (or stress) and bulk temperature versus time cycles that were recorded during the third week of July.

The total UVA (315–399 nm) dosage the samples received during exposure was 138 MJ/m^2 not including the irradiance data lost in April from a lightning strike, which would add another 30-40 MJ/m^2. Comparing recorded weather data during the testing period with historical averages revealed that the high and low temperatures were above normal average temperatures from March through August.[7] Temperatures recorded in May were near record highs.[7] Precipitation was double the normal amount during June and July 2010 for the south central region of Wisconsin that includes the Madison test site.[7]

The applied daily strain ratio (e_r) and mean strain (e_m) resulting from the diurnal PVC temperature change are plotted together in Figure 3. The strain ratio and mean strain were calculated with Equations (4) and (5), respectively, where e_{min} is the day's minimum strain and e_{max} was the day's maximum strain. The applied strain ratio increased during the seasonal transition from spring to summer and its variability between days decreased. The mean strain and its daily variability decreased as temperature rose. Both trends are indicative of decreased diurnal temperature swings and hence more stable strain patterns applied to the sealants during summer. Additionally, the rising strain ratio reveals that during summer, sealants are carrying more strain and are more susceptible to compression set damage.

$$e_r = e_{min}/e_{max} \tag{4}$$

$$e_m = (e_{max}+e_{min})/2 \tag{5}$$

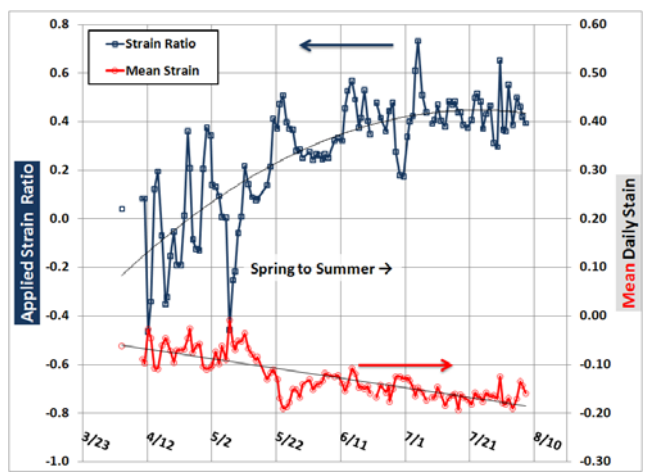

Figure 3. Daily strain ratio (upper curve, □) and mean strain (lower curve, ○) resulting from the diurnal PVC temperature change.

The weekly stress relaxation test yielded Ea values from Equation (2) over 4.6 orders of magnitude in relaxation time. Change in Ea during aging is summarized by plotting the Ea value at each magnitude of relaxation time over the length of the aging period, as shown in Figure 4 and 5 for sealants ASTM B and consortia C, respectively. Error bars in these figures for the Ea(5.8ks) data are the standard deviation among the sample replicates.

Average Ea data indicate a consistent increase in modulus during aging from April to August for both sea-

lants. Linear fits to sealant B data are shown in Figure 4 for Ea(10s) and Ea(5.8ks) data, allowing the total change in Ea and the rate of change in Ea to be compared. Similarity in the slopes of these lines indicates that relaxation behavior at short and long relaxation times and the overall rate of modulus increase did not markedly change during aging in March.

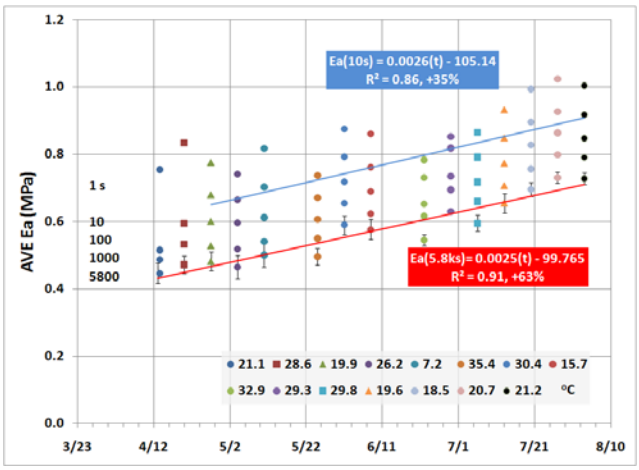

Figure 4. Plot of average Ea over 4.6 orders of stress relaxation time for sealant B, legend shows temperature.

Overall change in modulus is calculated from the change in Ea at the end of aging. The Ea(10s) data show a 35% increase in modulus, primarily because this data point was not sampled earlier in the study. The Ea(5.8ks) data show a 63% increase in modulus. The change in Ea at aging times earlier than 4/13/2010 are not shown, as the instrument fell short by approximately 2% of the prescribed estimated strain levels. Secondly, stress relaxation behavior was not measured on the sealants prior to the start of aging in March.

Applying the same analysis to the consortium C sealant reveals that the Ea(10s) and Ea(5.8ks) slopes are not similar and that the overall change in modulus is significantly less than sealant B. Hence, the stress relaxation behavior of consortia C may have changed during aging. Secondly, its modulus may be more stable than sealant B, indicative of higher durability under these conditions. The significance of these observations is balanced against the larger error bars for the Ea(5.8ks) data, indicating more variability within the consortium C sealant replicants.

Conclusions

Methods employed here allow real time instrumented outdoor aging with *in situ* tracking of durability as a function of dosage. A short period of this type of aging was sufficient to induce significant modulus rise. This trend may lead to sealants with decreased ability to maintain a seal under displacement, thereby compromising durability of the affected structure. The dose-damage data gathered in this study and going forward will be used as a data set for validating a building sealant durability model.

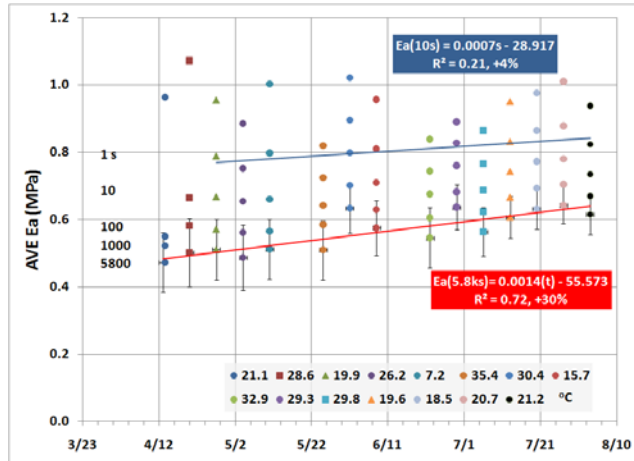

Figure 5. Plot of average Ea over 4.6 orders of stress relaxation time for sealant C, legend shows temperature.

Acknowledgements

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