Effects of Prestrain, Outdoor Aging, and Simultaneous Strain Cycling on the Durability of Building Sealants

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Introduction

Elastomeric sealants are vital to the construction of energy efficient and water tight buildings. Sealants are a small fraction of the construction budget. Upon failure their removal and reinstallation, and repair of moisture/mold damage can run in to the 10's of millions with the added risk of illness based litigation. In this research program we are seeking to discern the significance of environmental and strain effects on sealant durability as well as the initial state of stress (aka installation/cure conditions). Sealant bonded specimens are fixtured on automated test instruments and static specimen holders outdoors with three prestrains to simulate winter, spring/fall, and summer installation. The automated test frame imposes hot compression/cold tension strain cycling in accordance with the temperature of exterior building components simulated here by a section of poly(vinyl chloride) (PVC) pipe. Herein we report the results of 45 months of continuous outdoor exposure.

Experimental

Specimens: The sealant samples were provided by Dow Corning and an unnamed company referred to here as Company X. Dow Corning provided a one part neutral condensation cure room temperature vulcanizing (RTV) silicone. Company X provided a RTV polyurethane sealant. The adherends consist of anodized 6063 aluminum blocks (76.2×12.7×12.7mm) bonded together with sealant in the form of a 50.8×12.7×12.7mm bond line cured in conformance with ASTM C719.[1]

Strain Cycling: Sealant specimens were mounted in an instrument consisting of a fixed upper beam and a moveable lower beam. The instrument, shown in Figure 1, was custom built at the Forest Products Laboratory with further details available in prior literature.[2]

Strain: The instrument displacements were updated every 5 minutes in accordance with equation 1 where Δ is the displacement in centimeters and T is the measured temperature in degrees Celsius of a section of gray PVC pipe exposed to the weather and solar irradiation. Parameters were selected such that the specimens would experience strains between +25% and -25% corresponding to temperatures of between -18°C and 38°C with zero strain at 10°C.

$$\Delta = (10-T)/8.75 \qquad (1)$$

One third of the specimens were mounted so that they were unstrained when the instrument was at its neutral position (e=0 @ T=10°C), 1/3 of the specimens were mounted with +20% prestrain (e=0 @ T=32°C), and another 1/3 were mounted with -20% prestrain (e=0 @ T=-12°C). These specimens would experience-25 to +25% strain, -5 to +45% strain and -45 to +5% strain over the seasonal temperature range, respectively. These prestrains simulated situations where sealants were installed in concrete/steel/polymer constructed building joints that were smaller or larger than their average gap because of the thermal state of the building at the time of sealant installation (20% =summer, 0%=spring or fall, -20%=winter installation). The range of -25 - +25% strain was selected as a typical value of strain rating for building sealants. Another set of specimens was installed next to the strain cycling instruments with the same preset strains and inclination to the sun yet remained fixed in static position throughout the testing period.

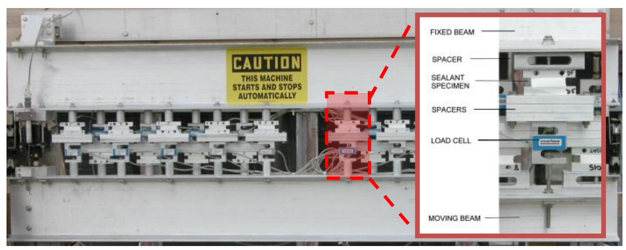

Figure 1: Photograph of mounted sealant specimens. The inset image is a close up of one assembly.

Modulus: Twice a week environmental strain cycling was stopped and an apparent modulus (Ea) cycle was run to check for changes in Ea resulting from weather and cyclic displacement aging. The cycle consisted of a 25 minute zero strain hold followed by compressive and tension segments consisting of two 15% strain peaks that act to remove Mullins effects from the sealants and a 10% strain stress relaxation period. The de-Mullins peaks are intended to remove any effects of filler bonds and secondary bonds that contribute to non-reversible load-displacement behavior.[3] Thus, the stress relaxation period occurs at lower strains than the first two peaks and is free of these effects.

Ea is determined using 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.[3]

$$E_a(t,\lambda) = \frac{3L(t)}{WB(\lambda - \lambda^{-2})} \quad (2) \qquad \lambda = 1 + \Delta/h \quad (3)$$

Weather: Temperatures were measured using T type (copper-constantan) thermocouples. Both ambient air temperature and the temperature of the PVC pipe were recorded. Relative humidity and solar irradiance was also

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measured and recorded. Additional weather data was obtained from the National Weather Service as recorded at the Madison Airport located approximately 20 km from the test site. Sealant exposure started in November 2011 and ended in October, 2016.

Results and Discussion

The daily strain extremes (delta strain) and average strains imposed on the sealant specimens for are shown in Figure 2 along with the average PVC drive temperature for the day. Here the temperature scale is inverted for ease of comparison with the strain values. Delta strain maintained a relatively constant variation of 10 to 25% per day regardless of season. The average strain follows the inverted temperature plot due to the hot compression operating mode. Values ranged from 25 to -25% not taking prestrain in to account. With prestrain the values range from 5 to -45% (-20%) and 45 to -5% (+20%). Unusual variations are seen 4 times due to thermocouple abnormalities that caused the testing machine to pause then finally to fail the samples due to over strain in the fall of 2016.

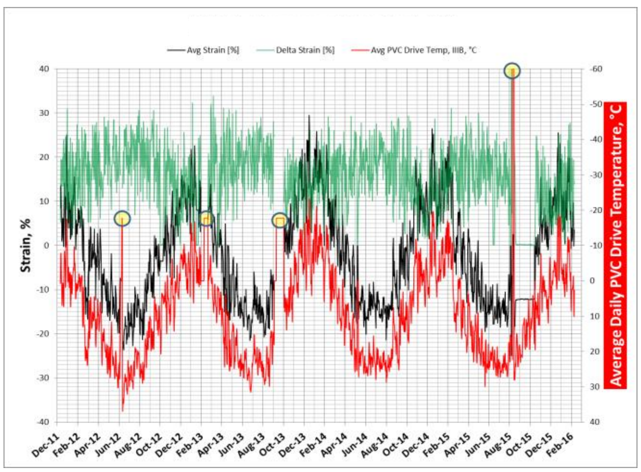

Figure 2: Daily delta and average strain, and average PVC drive temperature (scale inverted).

Figure 3 shows a week of strain cycling for the urethane (top) and silicone (bottom) specimens. This week is taken from the spring of 2013 where temperatures are near freezing at night and moderate to summer like during the day. Note that the temperature scale is inverted here to coincide with the stress values. The loads imposed on the sealants are highest for the -20% prestrain and lowest for the +20% prestrain. The silicone has a higher stress response than the urethane for all conditions as a result of its higher modulus. The multitude of sharp peaks at the midpoint of each day is due to solar heating of the PVC pipe driving the strain cycling being interrupted by intermittent cloud cover. Thus the strain exposure is a composite of a 24h tension and compression cycle with high frequency compression cycles at the midpoint.

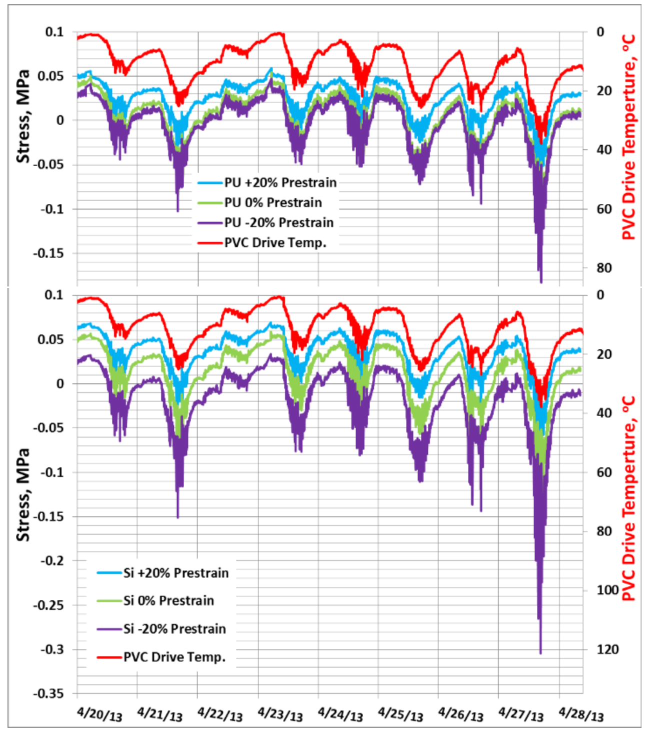

Figure 3: Week of daily strain cycles for urethane (top) and silicone (bottom) sealant specimens during spring of 2013.

The sealants were checked for compression or tension set (CTS) during the biweekly modulus measurements by monitoring the load value at zero strain before the tensile stress relaxation period. CTS is a known issue for most all elastomers and generally most polymers exposed to prolonged strain especially at high temperatures. CTS is reversible and permanent change in shape to match the imposed displacement. It effectively resets the zero strain value. Sealants exposed to long hot summers will develop compression set that may be reversed during the winter to neutral or tensile set. Rapid transitions in temperature at the end of a season may result in unexpectedly high levels of stress imposed on the sealants. The CTS stress values at zero strain are plotted in Figure 4 for representative urethane (top) and silicone (bottom) specimens at the three prestrains along with the average daily PVC drive temperature. The CTS behaves similarly for both sealants. Starting the test in cold tension maintained the +20% state of tension on the samples at the start of testing and the hot compression was insufficient to modify the prestrain throughout the testing. The overstrain event in 2016 was insufficient to damage these samples. The winter start of the testing was at or near the -20% prestrain zero strain point thereby maintaining the compressive present with a transition to summer hot compression acting to convert it to compression set that moderately recovers during the following winters. The 0% prestrain specimen starts the test in tension due to winter temperatures. The first summer of hot compression exposure changes this to compression set where it remains for rest of the testing with slight recovery during winter.

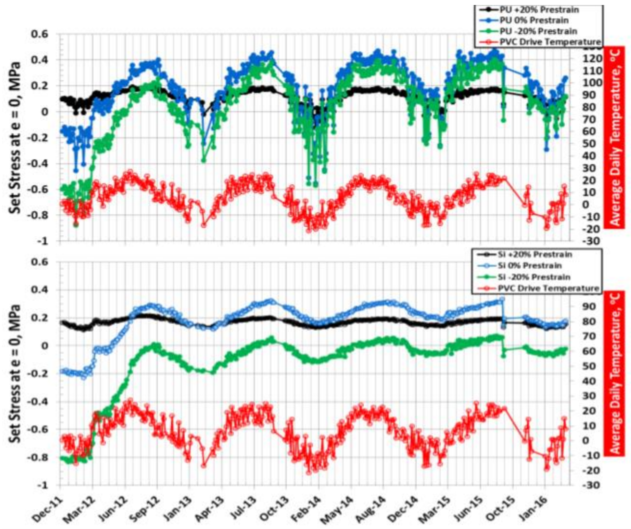

Figure 4: CTS stress and PVC drive temperature (right hand axis) versus exposure time for silicone (top) and urethane (bottom) sealants.

The 0%/-20% silicone sealant specimens softened rapidly (~-27% Ea) during the first 4 months of exposure followed by a gradual drop (~-9% Ea) in apparent modulus throughout the testing period. This transformation was not complete when the experiment abruptly ended due to equipment failure. The -20% prestrain specimens started with an Ea similar to the 0% prestrain yet softened at a greater rate over time. The 20% prestrain underwent slight softening during the first four months then was relatively stable for the remainder of the test period. This is likely due to the low compressive loads it experienced.

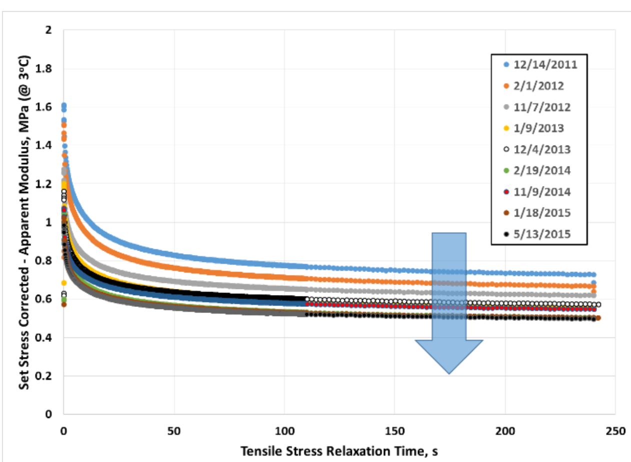

Figure 5: Tensile stress relaxation of silicone 0% prestrain specimen at times throughout the exposure period where the temperature during the measurement was ~3ºC.

The softening of the silicone occurred with no evidence of delamination or cracking. The surface of the specimens became soiled during exposure with evidence of mold growth, otherwise there was no obvious indication of damage. Thus, there was concern of dramatic changes in the polymer structure, plasticizer, filler, or interactions between these. The viscoelastic nature of the sealant provides insight into these properties and can be gauged by examining the stress relaxation behavior. Figure 5 shows the tensile stress relaxation profiles for a 0% prestrain specimen throughout its exposure where dates were selected where all tests were conducted approximately at 3ºC. The drop in modulus is apparent as a drop in magnitude of the stress relaxation profile. This is accompanied by a change in shape of the profile. As the silicone sealant ages the rate of stress relaxation to the plateau value increases. Effectively the transition from high instantaneous load to the plateau values becomes more of a right angle rather than a gradual transition. This is indicative of a significant loss in polymer network structure via breakage of polymer-filler or polymer-polymer bonds. Overall this leads to a loss in reinforcement of the sealant. In the absence of cracking or delamination this phenomena did not disrupt the function of the sealant. If continued the sealant may deform to a state where the seal is compromised. The effect slowed significantly after the first 4 months of exposure yet did not reach a stable plateau at the end of testing.

The stress relaxation profile for the urethane sealant is shown in Figure 6. No change in modulus was observed when comparing Ea values from similar temperatures at the start versus the end of testing. The stress relaxation profile appears to fluctuate without any particular trend. This is likely caused by variations in temperature about the target of 3ºC for selected stress relaxation profiles. Overall no change in modulus or shape of the stress relaxation profile indicating the absence of a significant change in the sealant during the test period. This is surprising as the surface of the urethane sealant underwent significant erosion with extensive surface cracking while no discoloration or soiling was observed. The erosion was sufficient to remove all residual flash on the surfaces of the adherends which was significant for some specimens yet did not appear to affect the thickness of the specimens. The surface cracking did not appear to affect the bond integrity or create a path for liquid or air to penetrate through the sealant.

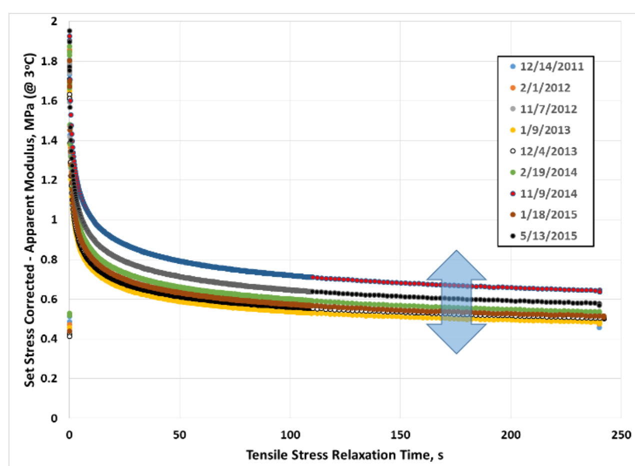

Figure 6: Tensile stress relaxation of polyurethane 0% prestrain specimen at times throughout the exposure period where the temperature during the measurement was ~3ºC.

The static specimens were removed from the test location, and allowed to sit in a laboratory drawer until they returned to their starting height if possible. The specimens were then run through the Ea strain profile via a screw driven test frame in the lab, and their Ea values compared at t=100s during the tensile stress relaxation period. None of the specimens showed a significant change in Ea compared to unexposed specimens even though their surface characteristics were the same as the cyclically strained specimens. This clearly indicates that the cyclic strain was the key stimulus that induced the silicone's loss in modulus.

Conclusions

After 45 months of hot compression strain cycling outdoors the silicone sealant exhibited modulus decrease, degradation of its network structure, and its surface became stained. The urethane maintained modulus over this time period with significant temperature dependence and surface erosion and cracking. The application of a 20% tensile prestrain to the sealants effectively changed the installation temperature to 32^{o}C a simulation of summer construction. This prestrain resulted in significantly lower compressive loads and lower loads overall. The -20% prestrain simulated a winter installation at -12^{o}C. These specimens exhibited the highest compression loading with the silicone softening more quickly than the 0% prestrain. Static specimens exposed alongside the cyclic specimens exhibited no significant change in modulus compared to unexposed specimens, illustrating the dominant role cyclic strain plays in ageing of silicone building sealants over other environmental stressors.

This study also illustrates the importance of designing for and attention to adverse installation conditions. The decision of when sealants can be installed is perhaps often a construction schedule issue as sealant manufacturers make the sealants curable in a wide range of weather conditions. This research indicates that the joint design and cladding materials of construction will need to be considered for appropriate installations conditions. Summer installation is preferred to minimize or avoid compressive loading that imposes the highest stress and out of plane deformation that leads to more rapid material transformation or bond damage. Winter installation should be considered only if gaps are sufficiently large to avoid high compression strain. If not, winter installation should be avoided.

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