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SOMMAIRE

Cet article étudie les conséquences des intempéries sur un mastic d'étanchéité à un seul composant, à base de silicone, à polymérisation chimique. Les propriétés des joints qui ont été exposés aux intempéries sont comparées au moyen d'essais de traction à celles des joints vieillis à la chaleur et controlés.

Weathering of Silicone Sealant on Strain-cycling Exposure Rack

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Investigation of the possibility of predicting performance of sealants from laboratory testing *(1)* indicated that cycling tests provide the closest laboratory reproduction of outdoor behavior. Because such tests are time consuming, a relation has been established between laboratory cycling and faster tensile tests at various strain-cycling rates *(2)*. At the low rates occurring in practice, however, laboratory methods are impractical and a strain-cycling outdoor exposure rack was designed *(3)*. This paper describes the changes a one-part chemically curing silicone sealant undergoes while exposed on the strain-cycling rack.

Specimens were cast on primed aluminum substrate and then exposed on a rack that produces a range of maximum yearly movements with superimposed daily movements. To separate the effects of outdoor exposure from those of strain-cycling, specimens were also exposed without movement and were stored in a constant-temperature room for the same three years of exposure. Comparison was made as well with heat-aged specimens, and the effect of fall versus spring cycling starts was examined. Periodically, the specimens were assessed visually and subjected to tensile tests. From these, the log strain versus log time at break was plotted, shown by previous work *(4)* to be the best method of characterizing the properties of the sealant.

Experimental

The sealant investigated was a one-part chemically curing silicone sealant conforming to Canadian General Standards Board (formerly Canadian Government Specifications Board) Standard 19-GP-9. Grade 6061T6 aluminum, primed according to the manufacturer's recommendation, was used as substrate. The sealant beads (12.70 x 12.70 x 50.80 mm; 0.500 x 0.500 x 2.000 in.) were cast in a constant-temperature room at 22°C (72°F) and 50 percent relative humidity. They were cured for about two months before being exposed to allow easy handling and scheduling of the work.

The specimens were bolted on a strain-cycling rack (Figures 1 and 2) *(3)*, and differential movement between the aluminum and the steel parts of the rack (induced by outdoor temperature changes) was transmitted to the sealant beads. The amount of movement the specimens underwent varied with their position on the rack and was measured weekly by maximum-minimum gauges *(5)*. Simultaneously, the surface temperature of the rack was recorded and related by statistical analysis to the movement. This enabled deduction of an accurate average value for the yearly movements that occurred during the three years of exposure. One quarter of the total rack area produced identical movements: the maximum of the average yearly width changes on the 12.70 mm (0.500 in.) wide specimens was ±15.8 percent. On the remaining area, movement increased from ±4.8 to ±30.4 percent in 36 increments. These values varied somewhat with the year, the season, and the load on the rack, causing a variation of between ±0.1 and ±1.1 percent in addition to the ±4.8 and ±30.4 percent values, respectively, with proportional changes between these extremes.

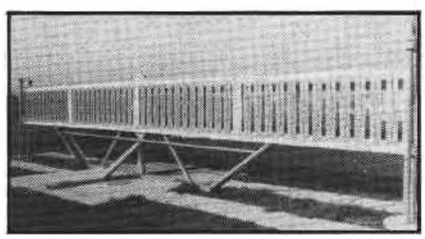


Figure 1. Strain-cycling exposure rack.

Figure 2. Specimens are bolted to rack.

The specimens were subjected to continuous movement on the rack for three years, starting in November 1974, and a few specimens for two and a half years, starting in May 1975. In both cases the air temperature was around the yearly average when the specimens were attached to the rack so that they underwent equal amounts of extension and compression during the yearly cycles. The samples faced south in a vertical position. Simultaneously, specimens were exposed, without movement, on a vertical rack facing south, using spacers and clamps to maintain the original width. After various exposure times specimens were taken for tensile tests from the different locations. Those not at the original width were put on spacers and clamps and conditioned for at least two weeks prior to testing to restore them to the original configuration. Testing and conditioning were done at 22°C (72°F) and 50 percent relative humidity.

In view of the limited number of specimens exposed to the same movement cycle, all specimens (failed or not) were taken off the rack and tensile tested. A careful examination of the results and of the tested specimens showed that a cross-sectional failure area less than or equal to about 40 mm² (0.06 sq. in.) had developed on exposure, but it did not influence the tensile curves. Failure areas larger than these lowered the tensile load for the same extension and were, therefore, not included in the tensile results. Failures, either on exposure or in the tensile test, usually started at a corner on the side exposed to the south and

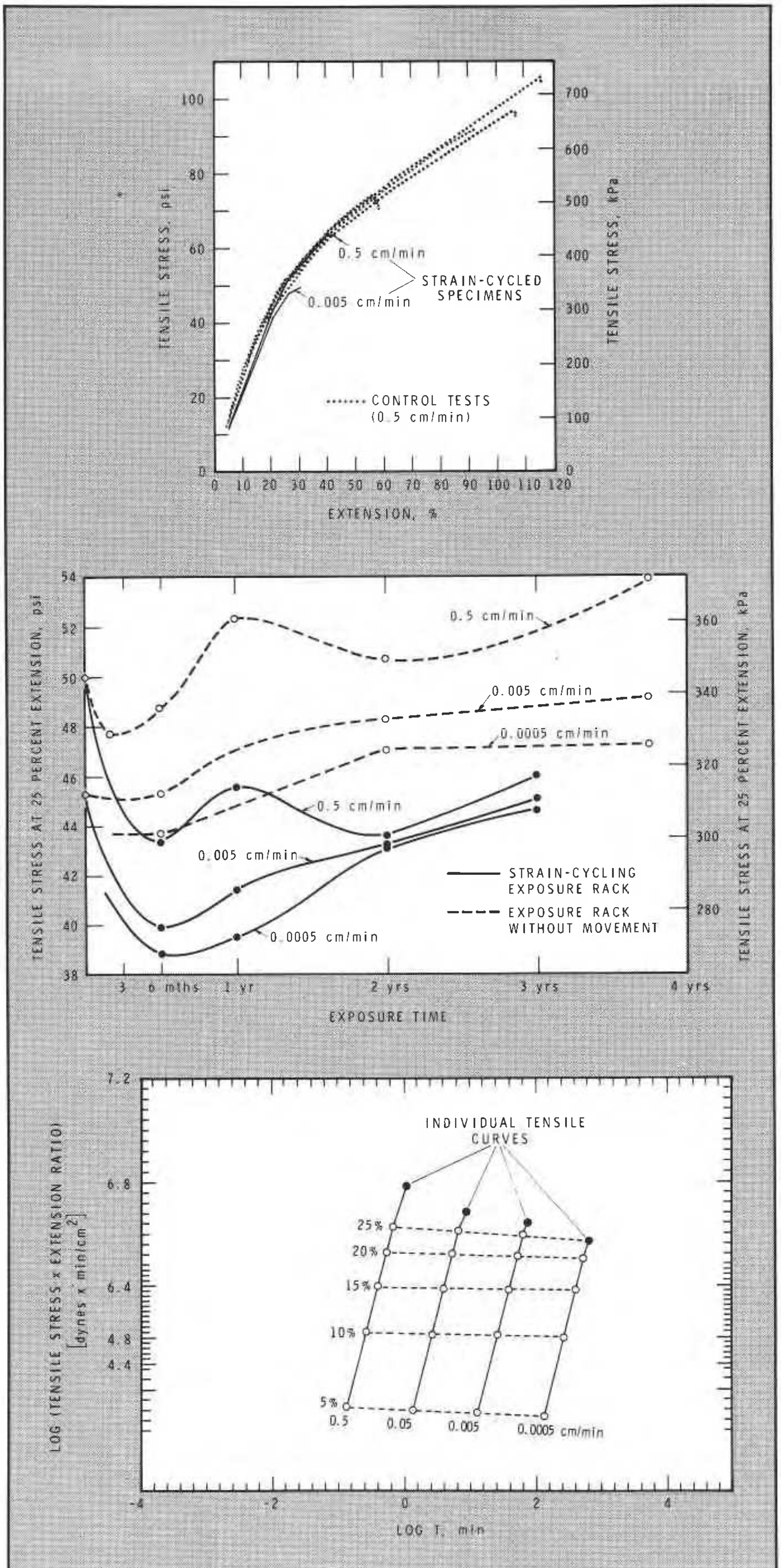


Figure 3 (top): Specimens exposed on strain-cycling rack and control tests. Figure 4 (center): Tensile stress at 25% extension as a function of exposure time. Figure 5 (bottom): Best fit lines (---) of various extensions at different rates.

propagated cohesively. The test either propagated a failure developed on exposure or started one at another corner, provided the original failure did not exceed the 40 mm² (0.06 sq. in.). Only one of the 66 specimens exposed on the cycling rack exhibited complete cohesive failure. Records comprising notes, sketches, and photographs were kept of the visual changes the specimens underwent during exposure.

One batch of specimens was heated at 70°C (158°F) for 30 days, then tensile tested at room temperature to examine the use of heat as an accelerator of the aging process.

Results and Discussion

Evaluation by Tensile Tests

A representative sample of the tensile curves obtained with specimens of various exposures is shown in Figure 3. The continuous lines refer to specimens subjected for three years on the strain-cycling rack to ±15.8 percent maximum yearly movement and tested at 22°C (72°F) at 0.5 and 0.005 cm/min extension rates. The dotted lines present results of tensile tests on unexposed specimens cured in the constant temperature room for two months and tested at a rate of 0.5 cm/min. The difference in shape of the continuous and dotted curves is very small, although the exposure conditions varied greatly. There is considerable variation in stress and extension at failure, producing a scatter in the results. It may also be seen that the extension at failure diminishes with exposure and with decreasing rate of extension.

Because the number of specimens was limited for a given exposure condition, failure points alone could not serve as a reliable basis for the analysis of results. The curves were compared first, therefore, at 25 percent extension because that was the maximum extensibility claimed for the sealant when used in joints.

The stress readings at 25 percent extension are plotted in Figure 4 for three different rates of extension. Three of the points are means of two to four readings; all others were calculated using the type of plot shown in Figure 5 where the logarithm of stress versus logarithm of time is plotted *(4)*. The continuous lines show the recalculated tensile curves obtained at different rates, and the dashed lines are the best fitting lines across the tensile curves connecting points at the same extension. The values plotted in Figure 4 were obtained from the best fitting line for the 25 percent extensions, thus increasing the number of points taken into consideration and the reliability of the results.

In Figure 4 the continuous lines show the results obtained with specimens exposed on the strain-cycling rack, and the dashed lines indicate results for those subjected to exposure without movement. The points at zero exposure are the results of tests carried out after two months of curing in a constant-temperature room at 22°C (72°F) and 50 percent RH, the approximate time of laboratory curing the specimens underwent before exposure.

The sealant appears to be softer when strain-cycled, stress being lower in the strain-cycled specimens. Generally, there is a decrease in stress during the first six months in both cases. After that, the stress increases and stays above zero exposure value without cycling, but it does not regain the original value if cycled. The curves obtained at different rates of extension show similar behavior, with lower stress at lower rates. After two years the strain-cycled specimens had very little rate dependence, as shown by the parallel lines.

As a further analysis of the effect of strain-cycling, the tensile stress at 25 percent extension was plotted as a function of maximum yearly strain-cycle (Figure 6). Each plotted point represents a single tensile test and each curve a different exposure time. Although the number of test points available is limited, it can be deduced that a marked decline of stress occurs with increasing yearly strain.

To compare stress versus exposure time (as in Figure 4) at different strain-cycles, the readings were divided into two groups, one less than or equal to and one greater than ±21 percent yearly strain-cycles. This division provided enough points for plotting the curves shown in Figure 7, which correspond to the uppermost continuous line in Figure 4 at 0.5 cm/min rate of extension. The separation between the two curves of Figure 7 further confirms that strain-cycling had a stress-reducing effect. (There were not sufficient readings at rates other than 0.5 cm/min for similar analyses.)

Having analyzed tensile stress at 25 percent extension in Figure 4, the investigator plotted it as a function of exposure time in Figure 8; and the two plots were compared. The curves have similar shapes, but the changes are attenuated in Figure 8. Without the sup-

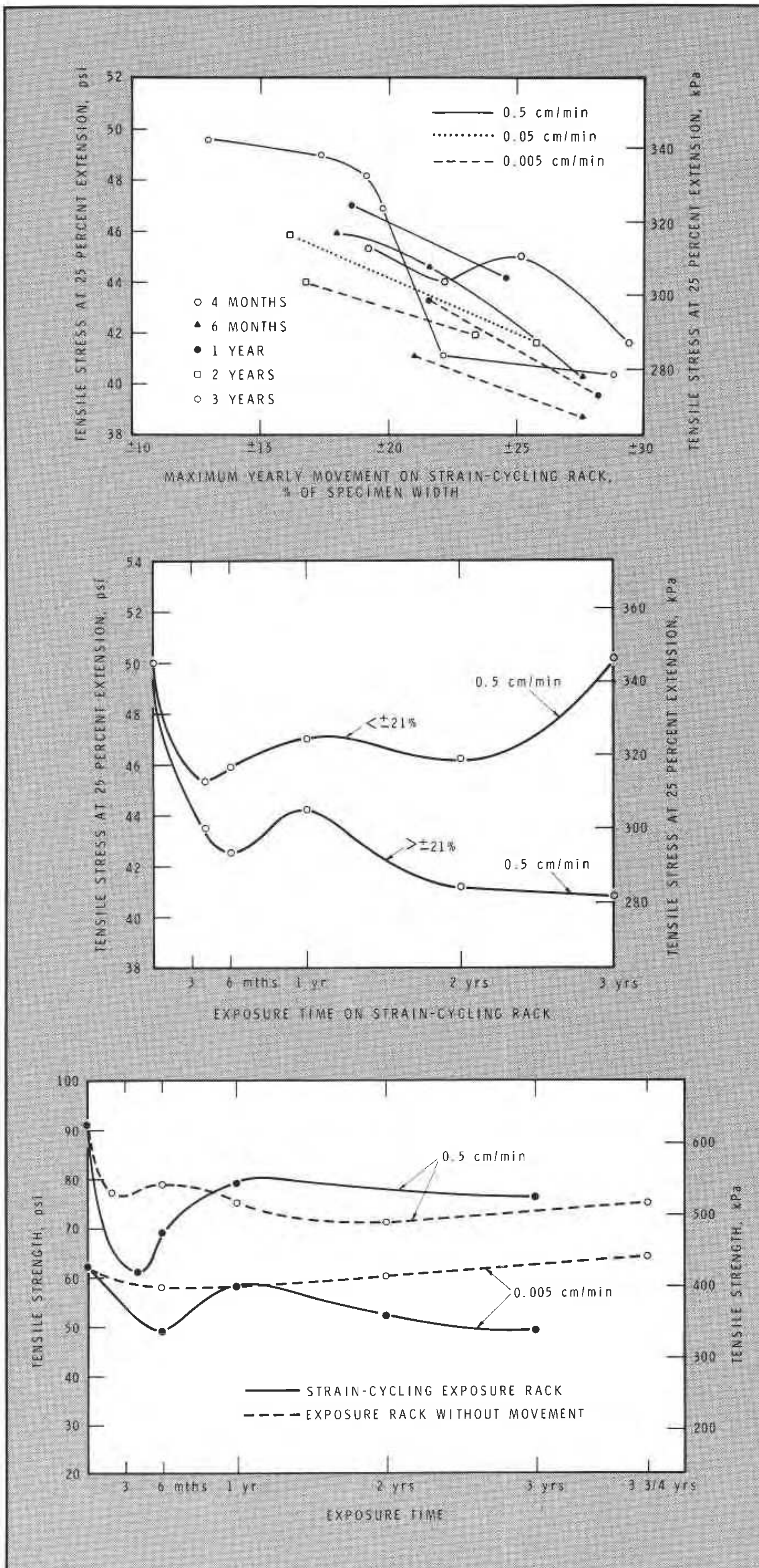


Figure 6 (top): Tensile stress at 25% extension as a function of yearly strain. Figure 7 (center): Tensile stress at 25% extension as a function of exposure time and strain-cycle. Figure 8 (bottom): Tensile strength as a function of exposure time.

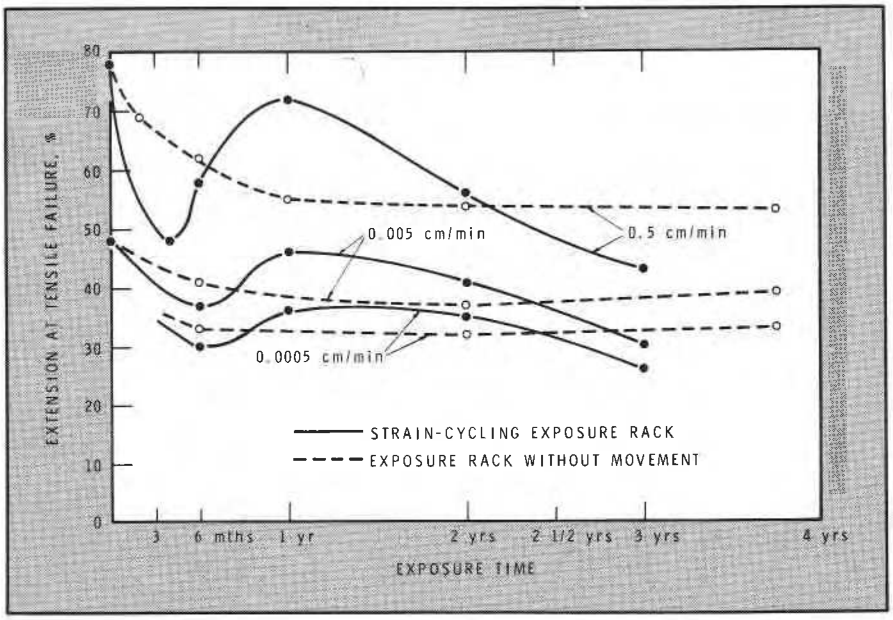


Figure 9. Extensibility as a function of exposure time.

Figure 11. After two months' exposure.

porting evidence of Figure 4 showing the same changes, the variations of the stress at break would be considered mainly due to scatter of the readings.

The pattern of variation of the tensile strength having been confirmed as inherent behavior of the material, it may be assumed that variation of the extension at break as a function of exposure is also inherent behavior and not a result of scatter in the readings. The extension at failure for the various exposures is tabulated in Table I and plotted in Figure 9. The curves show a variation with exposure time that is very similar to that of stress behavior with less than two years of exposure: both stress and strain decrease in the first six months and go through a maximum at one year if the specimens also undergo strain-cycling. Without strain-cycling the maximum does not occur and the curves flatten out. Either type of exposure produces a drop of extensibility after two years or more, and the drop is larger if the specimens are cycled. With decreasing strain rates in the tensile tests, the curves flatten and show the same shape; but the values are lower.

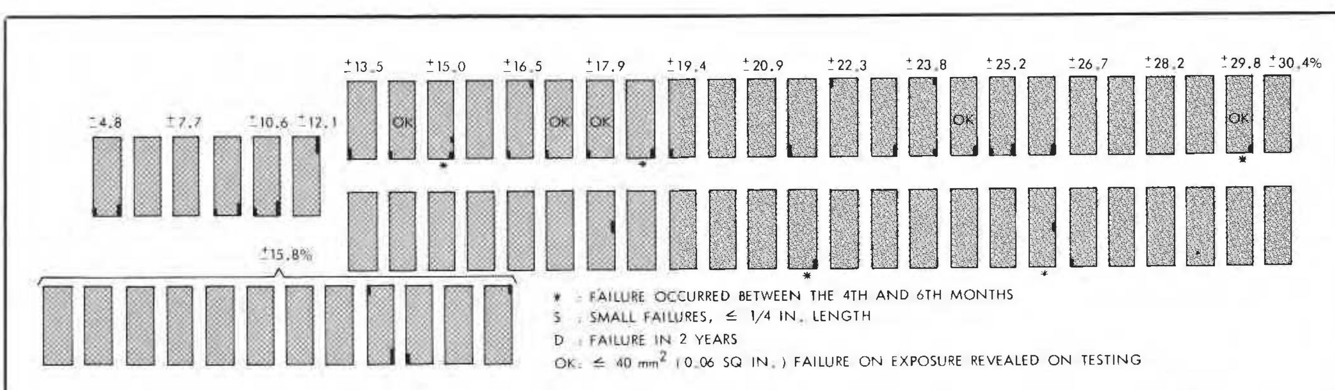


Figure 10a. Adhesive failures occurring on the specimens within six months on strain-cycling rack.

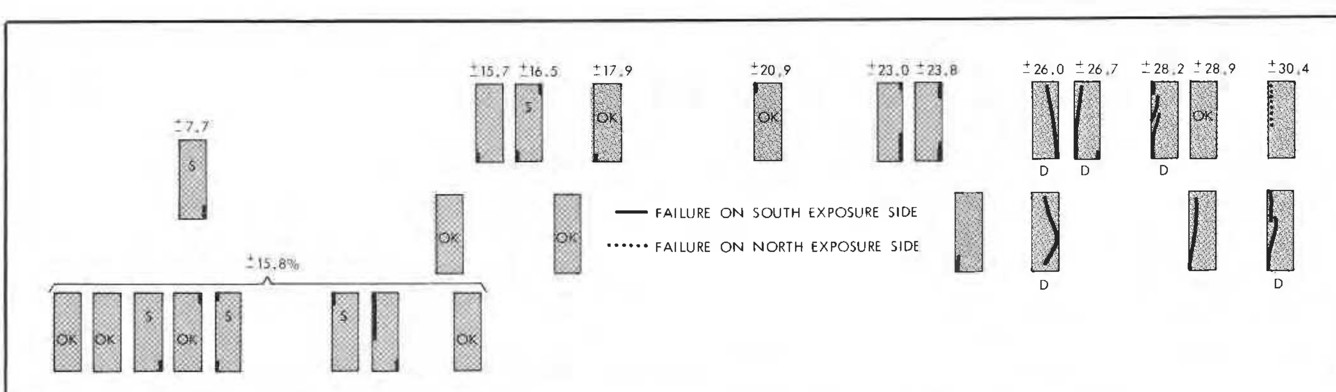


Figure 10b. Specimens after three years of exposure on strain-cycling rack.

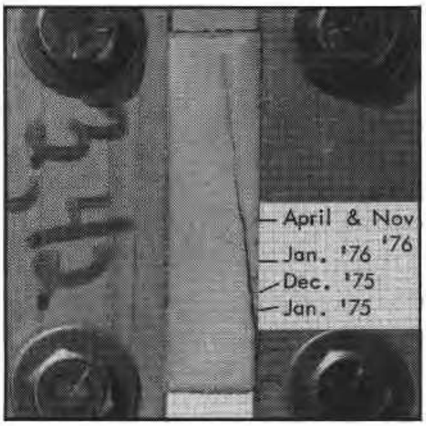


Figure 12. Failure advancing.

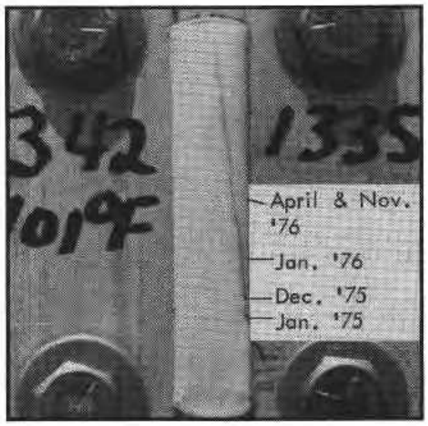


Figure 13a. After 2.5 years' exposure.

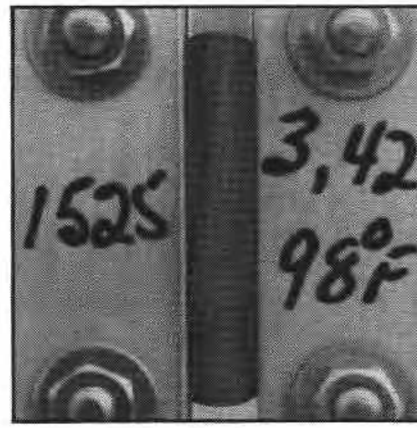


Figure 13b. After 3 years' exposure.

These results have been obtained with specimens that did not fail on exposure. They will have to be compared with qualitative assessment of exposed specimens where both failed and nonfailed specimens are assessed to obtain a complete picture of material behavior.

Visual Assessment of Exposed Specimens

The specimens exposed on the rack without cycling movement showed no failure even after three years; neither did the specimens stored in the constant-temperature room. Strain-cycling, however, produced various degrees of failure on 38 of the 66 exposed specimens.

Figure 10a shows the layout of the specimens on the strain-cycling rack. The rectangles represent the side of the sealant bead exposed to the south. The average maximum yearly movement is shown on top. The failures that occurred within the first six months are indicated schematically. All are adhesive failures of shallow depth (less than the length shown). Very few occurred on the back or ends of the sealant bead and these are not shown in the schematic presentation.

Failures started within six months on 27 specimens. Those marked by asterisks occurred after March 1975, leaving 22 specimens that showed some failure during the first winter. Specimens with and without failure were removed from the rack from time to time and tensile tested. This left 41 specimens exposed after six months, and only two showed any progress in failure during the subsequent summer (about 0.2 cm). These specimens are shown in Figures 11 to 16, where progress of failure may be followed through the entire three-year period. They illustrate failure characteristic of all the specimens.

Figure 11 shows the start of failure within the first two months of exposure for a specimen exposed where the average maximum yearly movement was ±26.0 percent. Figure 12 indicates the progress of failure on the same specimen. It may be seen that the second half of the failure occurred during the third winter. Figures 13a and 13b show the sides of the same specimen exposed to the south and the north when compressed on the rack in the last summer at air temperatures of about 38°C (100°F). The adhesive failure is barely visible under compression on this specimen (mostly undetectable in others). The front and back views of the specimen following removal from the rack after three years of exposure show that there was no further change after the third winter (Figures 14a and 14b).

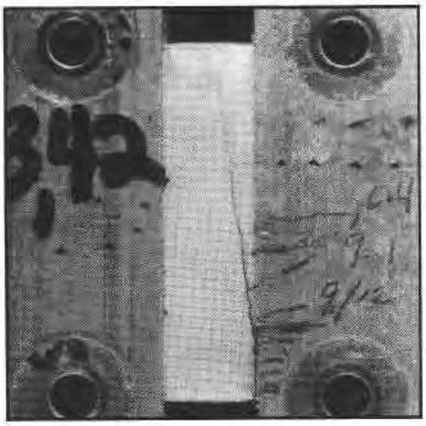
The specimen shown in Figures 15 and 16 was exposed to ±15.8 percent average maximum yearly movement. Two corners show adhesive failure after two months; one remained unchanged while the other advanced with time of exposure (Figure 16a). Figure 16b shows the back of the specimen where there is hardly any failure; this specimen was held in a slightly bent position while being photographed to make the failure more visible.

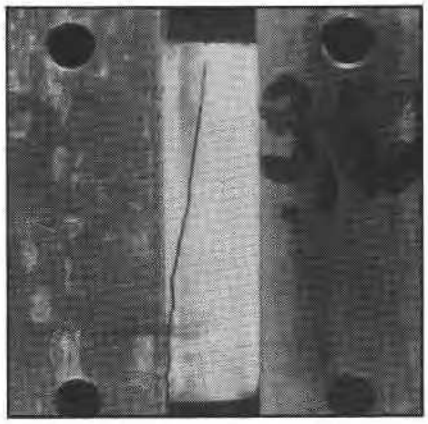
The specimens — failed and unchanged — were progressively removed for tensile testing; the state of those left exposed until the end of the third year is shown in Figure 10b. The marks indicate failures of 13 mm (½ in.) or longer through the entire depth of the bead, with failure surfaces perpendicular to the sealant surface

Table I — Extension at Tensile Failure

	No. of readings	Rate of extension (cm/min) 0.5	0.005	0.0005
		%	%	%
Stored in constant-temperature room				
2 months	4	78*	—	—
2 months	2	—	48*	—
3 years	9	65	47	40
Exposure without movement				
2 months	2	69*	—	—
6 months	4	62	41	33
1 year	4	55*	—	—
2 years	8	54	37	32
3¾ years	11	53	39	33
Exposure with strain-cycle				
4 months	4	48*	—	—
6 months	6	58	37	30
1 year	4	72	46	36
2 years	5	56	41	35
3 years	9	43	30	26

*Average reading





Figures 14a and 14b: After three years' exposure.

shown. Except for these specimens, all were tested and those that revealed less than or equal to 40 mm² (0.06 sq. in.) of failure surface (developed before testing) are marked "OK" in Figure 10. The presence or absence of dust on the failure surface made possible the distinction between failure on exposure and failure on testing. Sample specimens were taken at intervals from evenly distributed areas of the rack so that the proportion of specimens at various yearly movements was approximately maintained. There were relatively more specimens left on the rack at the larger movements because larger failures occurred there, making tensile tests impossible. Those with failures of ½ to 1 in., marked "D," were left on the rack so that the progress of further failure could be followed. These large failures developed mainly during the second winter at or above ±26 percent movement. They usually showed no advance during the summer but continued to advance during the third winter. Two failures developed to medium size during the final summer (at ±23.8 and ±24.5 percent).

The total number of specimens in Figure 10b is 26, of which five (marked "D") failed at two years of exposure, leaving 21 specimens that reached three years of exposure. Following tensile testing of all of them, nine proved to be good (≤0.06 sq. in. failure area on exposure) and five had minor failures (marked "S") of ¼ in. long or less separation at one corner and the same depth (>0.06 sq. in.). Approximately half of the specimens exposed for three years failed. When equal numbers of specimens cycled below and above ±22 percent maximum yearly movement were compared, there were fewer failures among those cycled at less than ±22 percent. The larger difference is in the appearance of the failure, which is often complete at larger movements but much less extensive at smaller ones.

Spring versus Fall Start of Sealant Installation

To investigate spring versus fall starts for sealant exposure, twelve specimens exposed on the rack without movement in November 1974 were transferred to the cycling rack in May 1975. These specimens, therefore, did not undergo movement during the first winter; and their behavior was different from that of all other specimens, although they were prepared at the same time from the same batch. All reached the end of the exposure period without failure, having been subjected to strain-cycling exposure for two and a half years. The strain imposed on them varied, as it did on the other specimens: four were cycled at ±12.2, four at ±15.8, and four at different values between ±19.4 and ±27.4 percent. All specimens were tensile tested after exposure, but the tensile results did not reveal significant differences when compared with results obtained at other conditions because of the scatter in test results.

Effect of Heat Aging

Correlation of the tensile properties of heat-aged and exposed specimens was sought. The shape of the tensile curves did not change with heat aging, but failure occurred at higher extensions and stresses. Changes in properties are best illustrated by plotting the logarithm of extensibility against the logarithm of time to reach failure, as in Figure 17, where the best fit line of the points is indicated but the actual points are omitted for the sake of clarity. The continuous line representing the heat-aged specimens is at higher extensibility values than any of the other lines.

The slope of the best fit lines decreases with increasing exposure time, and at two years, and after it is parallel to that of the heat-aged specimens. The extensibility increase with

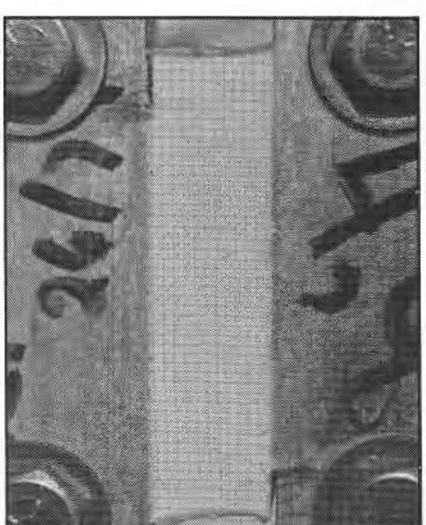


Figure 15. After two months (±15.8%).

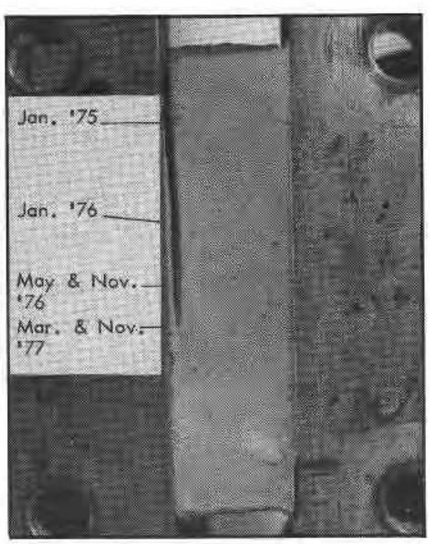


Figure 16a. South exposure.

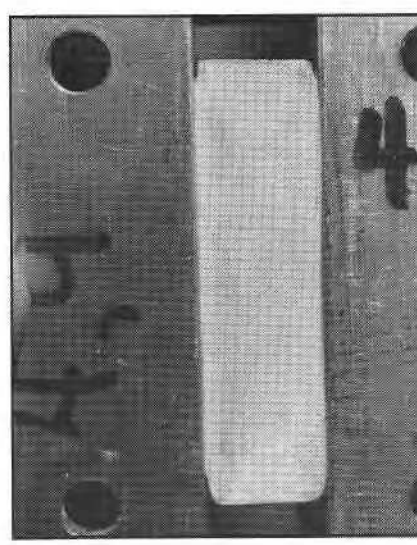
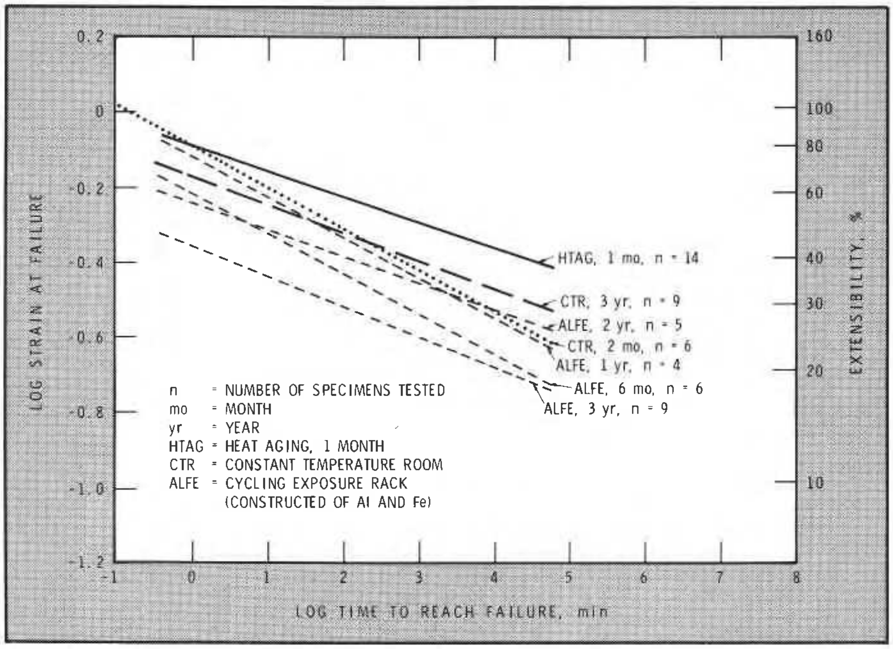


Figure 16b. North exposure.



Figue 17. Best fit lines of extensibility data as function of time to reach failure.

heat aging as opposed to the decrease found with strain-cycling indicates that some of the changes taking place on heat aging are different from those of natural aging. The most likely cause is an improvement in adhesion with heat aging, requiring higher strength to break the sample and resulting in higher extensibility. It may be concluded that heat aging is not suitable as an accelerated aging process for silicone sealants.

Specimens exposed without movement showed little change, and the best fit lines of the periodic tests (not shown) were in the region of the lines representing fresh specimens and specimens cycled for two years. The line of the laboratory-stored specimens stayed in the region of the line of fresh specimens, with the slope approaching that for heat-aged ones.

Conclusions

Strain-cycling movement is the predominant factor that causes failure during weathering of a one-part chemically curing silicone sealant; outdoor weathering alone is negligible.

Failure is also influenced by extent of movement, becoming more extensive at higher yearly strain-cycles. Tensile tests reveal a notable decrease in strength beyond about ±22 percent movement.

For this particular silicone sealant heat aging cannot be used to accelerate weathering since the properties improve instead of deteriorate, as on outdoor cycling. This is probably due to the establishment of a better bond between the sealant and the substrate or primer.

The silicone sealant installed in spring did not show failure at the end of three years of exposure because the adhesive bond was firmly established before being exposed to strain-cycling.

Acknowledgments

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