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Publisher’s version / Version de l'éditeur:

Durability of Building Materials, 5, 1, pp. 35-51, 1987

National Research Council Canada
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Reprinted from
Durability of Building Materials
Vol. 5, No. 1, 1987, p. 35 - 51
(IRC Paper No. 1483)

Price $4.00

NRCC 28443

Canada

RÉSUMÉ

On a soumis à des mouvements cycliques, en laboratoire, des éprouvettes de produits d'étanchéité en utilisant des dispositifs de serrage réglables à la main. On a comparé les déformations permanentes à celles que l'on avait observées à l'occasion de travaux de recherche antérieurs visant des éprouvettes exposées sur une clôture d'essai et soumises à des cycles de déformation. En comparant les trois cas de déformation permanente observés, on a pu établir une relation entre le comportement à l'extérieur et les essais en laboratoire.

Durability of Building Materials, 5 (1987) 35–51
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

Laboratory Fatigue Test of a Two-Part Polysulfide Sealant Correlated to Outdoor Performance

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(Received October 1986; accepted February 19, 1987)

Keywords

Sealants	Polymers	Properties	Mechanical Physical
		Testing and performance	Laboratory

ABSTRACT

Karpati, K.K., 1987. Laboratory fatigue test of a two-part polysulfide sealant correlated to outdoor performance. *Durability of Building Materials*, 5: 35–51.

Movement cycles have been imposed on sealant specimens in the laboratory using manually adjusted vises, and permanent deformations compared with those found in previous investigations of similarly-cycled specimens exposed on a test fence and others exposed on a strain-cycling rack. On the basis of the three observed cases of permanent deformation, the connection between outdoor performance and laboratory testing has been established.

INTRODUCTION

When sealants fail in use, the failure mechanism can vary, depending upon the type of material and the service conditions. With some there may be adhesive failure at the interface with the substrate. Others may not have sufficient extensibility and crack in the body of the sealant bead. Conversely, sealants that are quite flexible may, after being considerably extended, suffer permanent deformation and not return to their original shape when the joint width becomes small again. Permanent deformation can be separated into three stages:
(a) the smallest movement at which it is just observable;
(b) formation of lengthwise bulges with part of the sealant bead folded under;

0167-3890/87/$03.50

(c) the smallest amount of movement at and above which holes develop, i.e., perforation occurs.

A previous investigation of a two-part polysulfide sealant (Karpati, 1984) indicated that cycling is the most important service factor leading to deformation and eventual perforation. Indoor aging or outdoor weathering without cycling have comparatively little effect on performance. In that study cycling was produced on an outdoor rack that reproduced the cycling movement of building joints by imposing on the specimens daily and yearly joint width changes caused by air temperature changes. In three years of exposure the first two stages of permanent deformation could be observed on a two-part polysulfide sealant, but the third stage, perforation, did not occur.

All three stages of permanent deformation were observed in another work (Karpati, 1985) in which cycling was produced on manually operated vises with simultaneous exposure. Although only the yearly movement of natural cycling was simulated, after three years of exposure the greater movement range (up to $\pm 90\%$) produced the third stage.

The objective of the present work was to determine whether failures produced by cycling during natural weathering would result solely from cycling in the laboratory, i.e., whether a fatigue test could be developed. The sealant specimens mounted on the vises were extended and compressed to different amplitudes at three cycling rates. The conditions necessary to produce the three stages of failure are reported and one cycling rate is recommended for laboratory testing.

EXPERIMENTAL

The same commercial brand of light grey, two-part polysulfide sealant as in the previous studies (Karpati, 1984, 1985) was applied without primer on aluminum substrates. In both cases the substrate was first cleaned manually, then with trichloroethylene vapour. The sealant bead was $12.7 \times 12.7 \times 50.8$ mm ($0.50 \times 0.50 \times 2.00$ in), and the aluminum bars were $12.7 \times 25.4 \times 76.2$ mm ($0.50 \times 1.00 \times 3.00$ in).

In accordance with the manufacturer's instructions the substrate was not primed before application of the sealant. The specimens were cast using silicone release paper underneath the bead and on the end spacers that limited the length of the bead to two inches. The specimens were cured to the same degree as the specimens exposed on the strain-cycling rack. When this was checked by tensile tests it was found that four days' curing at 30°C (86°F) resulted in the same load and extension as that found for those specimens which had been cured for two months in the laboratory.

Following curing, the specimens were attached to hand-operated vises (Karpati, 1979) that imposed various extensions and compressions. The total movement ranged from $\pm 10\%$ ($+10\%$ extension; -10% compression) to

±80% of the joint width, in steps of 5%, and is referred to as amplitude of movement, shown in some figures as strain of the cyclic movement. Duplicates were used at each amplitude.

Earlier work (Karpati, 1973) indicated that the mechanical properties of two-part polysulfide sealants are rate dependent. It would, therefore, be desirable in a laboratory test to stay as close as possible to the rates of movement occurring in practice. Under practical conditions the daily as well as the yearly rate of cycling are involved in producing failure. For example, a 12.7 mm (0.5 in) wide sealant bead cycling between +25% and −25% of the width has, as the result of temperature changes, a mean yearly rate of movement of about 0.0002 mm/min. In the same joint the daily temperature changes cause joint width changes of about ±4% per day, but because of the short time the rate of movement is faster, about 0.001 mm/min. The latter rate was chosen for laboratory testing, resulting in one cycle/week for ±25% amplitude of movement. To increase the severity of the test two higher rates were also used: $2\frac{1}{2}$ and $7\frac{1}{2}$ cycles/week. These are shown in Figs. 1a–c and in Table 1 where rates for both ±25% and ±80% are shown to indicate the increase in rate with amplitude. (In calculating rates the movement and the testing periods are averaged.) The cycles consisted of extensions to full amplitude in one step, holding the extension, then changing to complete compression, holding that, then extending again, and so on. During weekends the specimens were held in either tension or compression, alternating with the weeks, but never kept at the original width.

The specimens for the $7\frac{1}{2}$ cycles per week were prepared at a time different from that of the other cycles. To eliminate possible differences arising from this variation, the $2\frac{1}{2}$ cycles per week test was repeated for ±50% to ±80% movements using the same batch as the $7\frac{1}{2}$ cycles per week test. This study showed that the results are repeatable within acceptable error. The appearance of the specimens was recorded photographically at the end of each rest period just before a new width change was made.

RESULTS AND DISCUSSION

Stages of permanent deformation

By using a range of amplitudes, three stages of permanent deformation could be observed on the laboratory-cycled specimens, as established previously on specimens cycled while exposed outdoors. Photographs of specimens in various stages of deformation are shown in Figs. 2 to 5. (These were defined previously (Karpati, 1985) and are quoted in the Introduction.)

The permanent deformations occur over a range of amplitudes rather than at a specific amplitude, as shown in Fig. 6 where stages 1, 2 and 3 are illustrated by □, ○ and ●, respectively, and the boundaries for stages 1 and 3 are outlined. Where the duplicate results were the same, they were arbitrarily sepa-

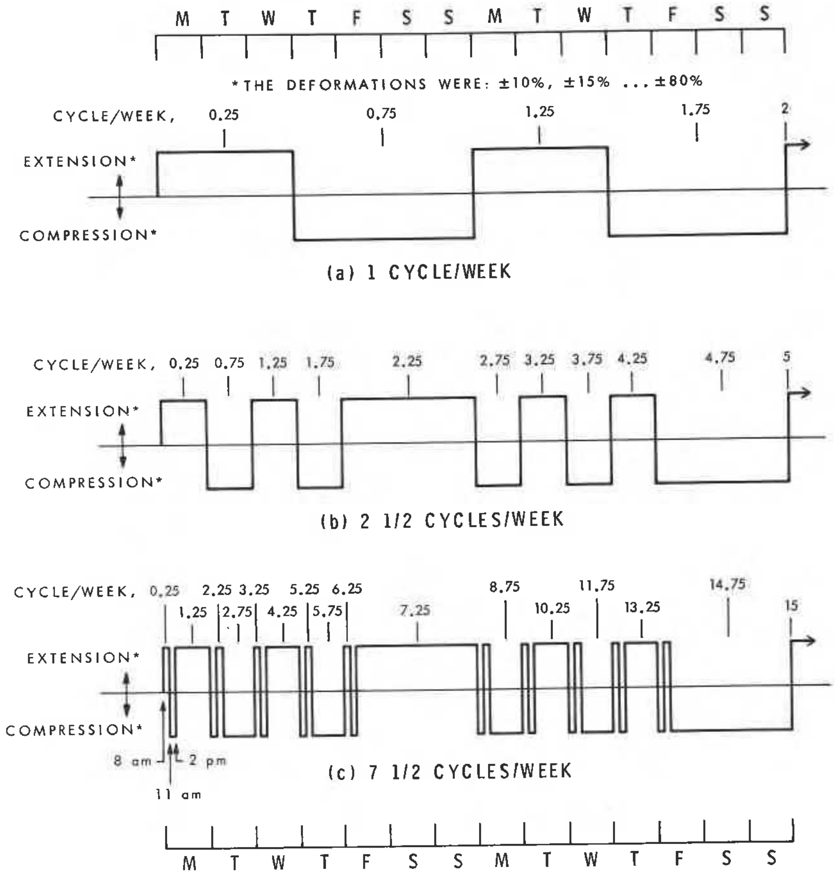
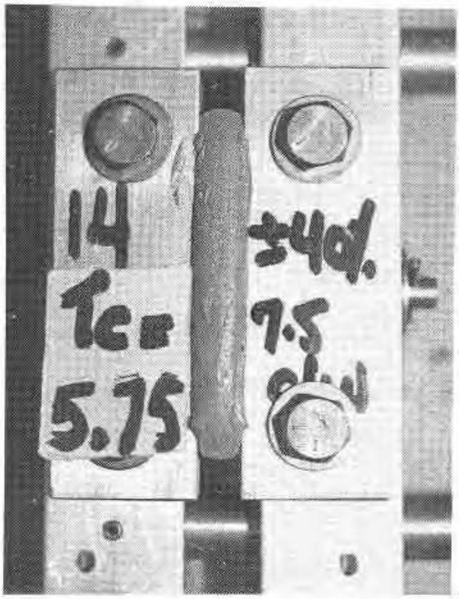


Fig. 1. Movement cycles used in laboratory testing. (a) 1 cycle/week; (b) 2.5 cycles/week; (c) 7.5 cycles/week.

TABLE 1

Mean movement rate of 12.7 mm (0.5 in) wide sealant specimens

	Rate (mm/min)	
Amplitude	±25%	±80%
Exposed on strain-cycling rack, yearly rate	0.00002	—
Exposed on strain-cycling rack, daily rate	0.001	—
Exposed on vises, 1 cycle/year	0.00002	0.00007
Laboratory test, 1 cycle/week	0.001	0.004
Laboratory test, $2\frac{1}{2}$ cycles/week	0.003	0.01
Laboratory test, $7\frac{1}{2}$ cycles/week	0.01	0.03



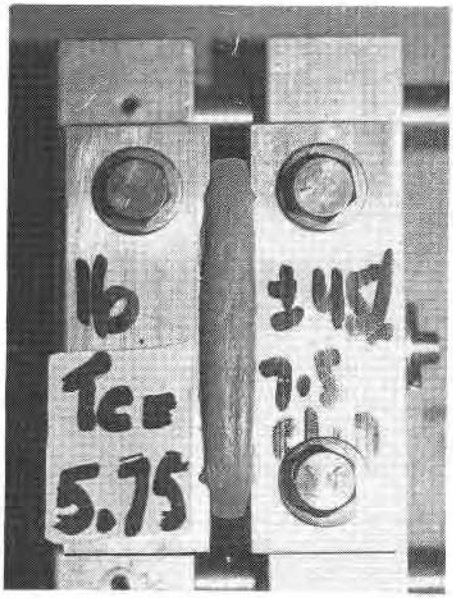
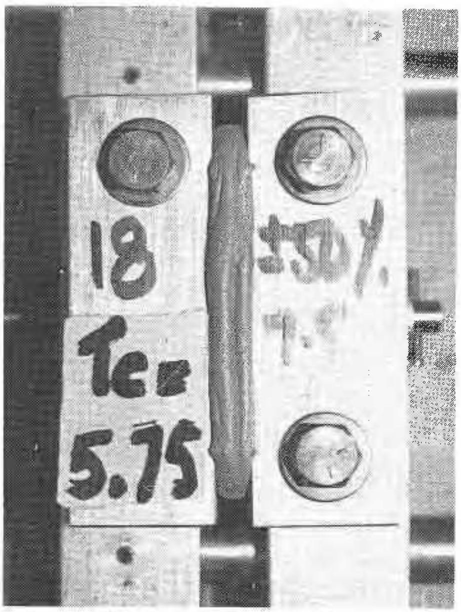


Fig. 2. Specimen before stage 1, $7\frac{1}{2}$ cycles per week, $\pm 40\%$, after $5\frac{3}{4}$ cycles.

Fig. 3. Specimen at lower edge of the area defining stage 1, $7\frac{1}{2}$ cycles/week, $\pm 45\%$, after $5\frac{3}{4}$ cycles.



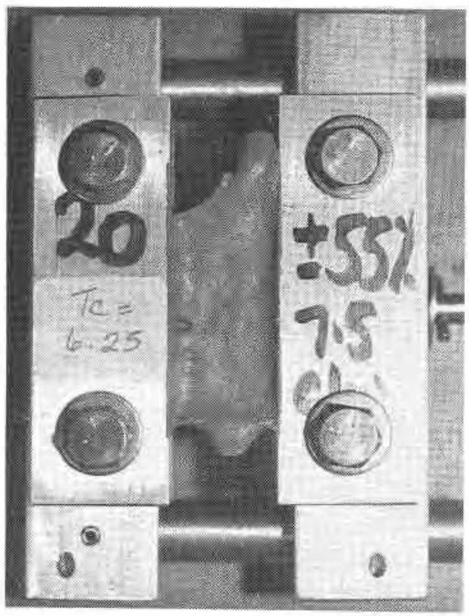


Fig. 4. Specimen at lower edge of area defining stage 2, $7\frac{1}{2}$ cycles/week, $\pm 50\%$, after $5\frac{3}{4}$ cycles.

Fig. 5. Specimen at lower edge of range defining stage 3, $7\frac{1}{2}$ cycles/week, $\pm 55\%$, after $6\frac{1}{4}$ cycles.

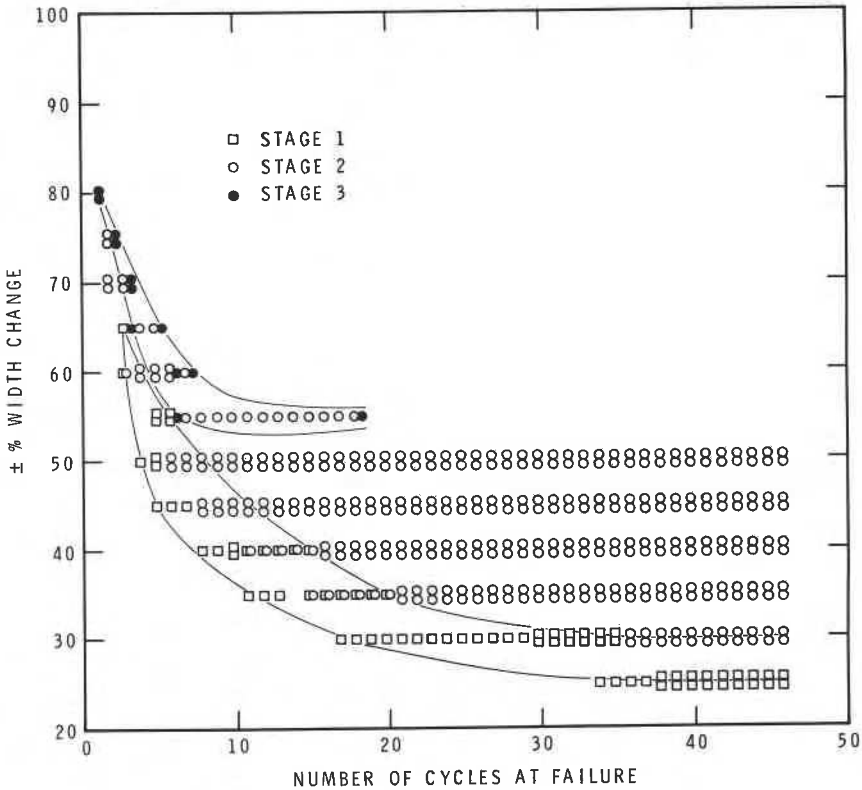


Fig. 6. The three stages occurring at $7\frac{1}{2}$ cycles/week.

rated by 1% in the plot to allow the total number of observations in the experiment to be shown.

In the subsequent plots the areas rather than the individual observations are shown. Figures 7, 8 and 9 present stages 1, 2 and 3, respectively, for the three cycling rates. Part of the $2\frac{1}{2}$ cycles/week test was repeated (see Experimental), but the results are not shown because they agreed with the first results.

As may be seen from Figs 6 to 9, the three stages could be observed at all three cycling rates. Generally, for a given number of cycles, increasing the cycling rate causes a particular deformation stage to occur at higher amplitudes. As the number of cycles increases, however, deformations occur at decreasing amplitudes but the curves taper off at higher cycles. The reason why deformation at lower amplitudes occurs with the fewest cycles per week is, probably, that the sealant is extended for longer periods. Stages 1 and 2 cover fairly large areas but the conditions for stage 3, perforation, are more well defined.

The highest number of cycles at which perforation occurred are 18, 18 and 7

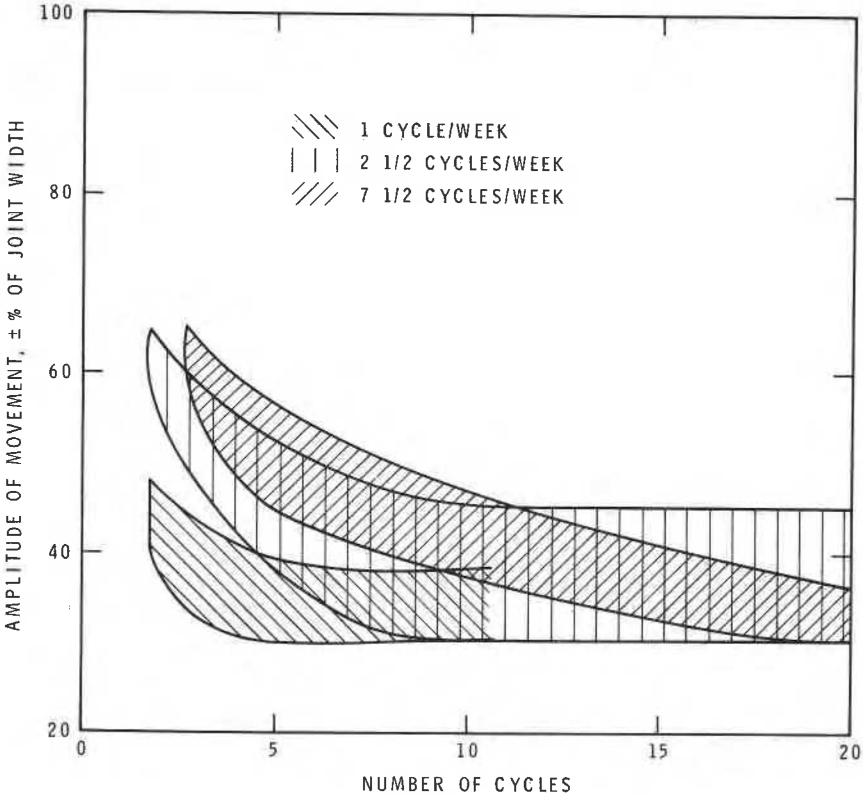


Fig. 7. Stage 1: permanent deformation starts to occur.

for $7\frac{1}{2}$, $2\frac{1}{2}$ and 1 cycle/week, respectively. Above these values there were no more perforations, although the total number of cycles was 46, 28, and 11, taking 43, 78 and 74 days, respectively, for the three rates.

Deformation as a function of time

When stage 3 observations are plotted as a function of the time required to reach that deformation, straight lines are obtained on a plot of log strain versus log time, as shown in Figs. 10, 11 and 12 for $7\frac{1}{2}$, $2\frac{1}{2}$ and 1 cycle/week, respectively. The time is obtained by multiplying the number of minutes to complete a cycle by the number of cycles. Whether a time reading taken at the beginning or end of a rest period (when the photographic recording of the deformation was made) would change the results was also examined. It was found that the very tedious process of calculating start and end readings was not justified because the differences were insignificant and the conclusions would be the same.

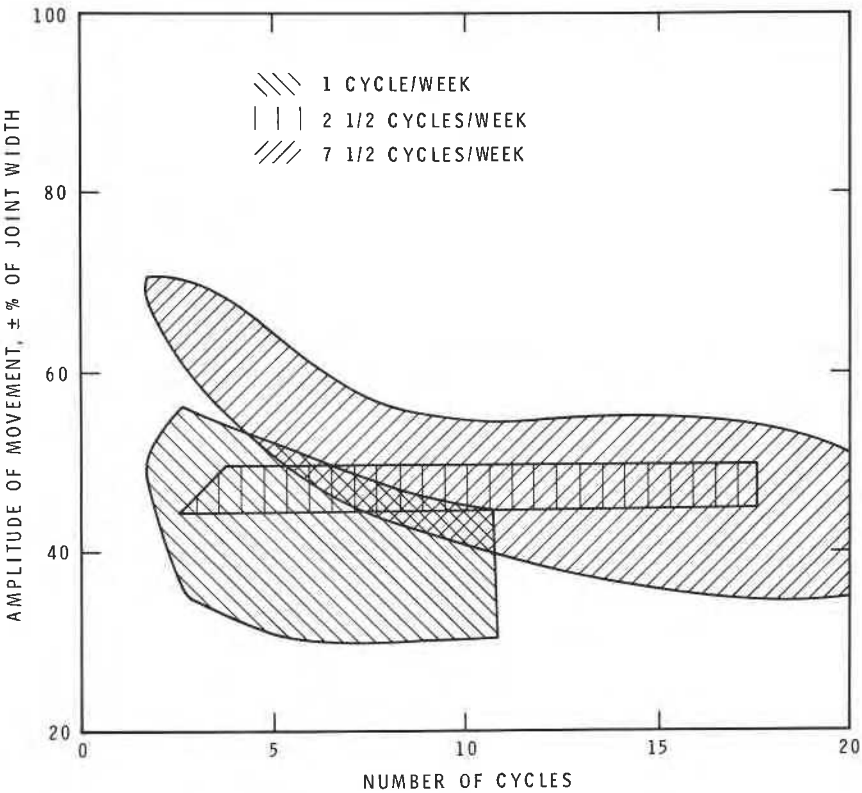


Fig. 8. Stage 2: lengthwise bulges and folds occur.

Points marked by index 2 in the plots mean that the duplicate tests gave the same result. Continuous lines are the best-fit lines; dashed lines are the 90% confidence limits of the best-fit lines. Comparison of the plots shows that the best-fit lines are very close to each other, shifting slightly towards longer times at lower cycling rates. With faster cycles, failures occur at lower amplitudes and sooner at the same amplitude. The fastest cycle used, $7\frac{1}{2}$ cycles/week, can therefore be recommended for routine testing.

The three-year exposure on the strain-cycling rack (1) did not produce stage 3 (complete) failure; only stages 1 and 2 could be observed. Consequently, it is important to examine these two stages in the laboratory tests in order to compare exposure and laboratory results. This will be discussed later. Figures 13 and 14 show all three stages for $7\frac{1}{2}$ and 1 cycle/week tests, respectively. The areas encompassing stage 2 are the most widespread; the others, especially those for stage 3, are fairly narrow. It is therefore preferable to use stages 1 and 3 for routine testing; they are also easier to observe in most cases. The $2\frac{1}{2}$ cycles/week test gave results between the two other rates and is not shown.

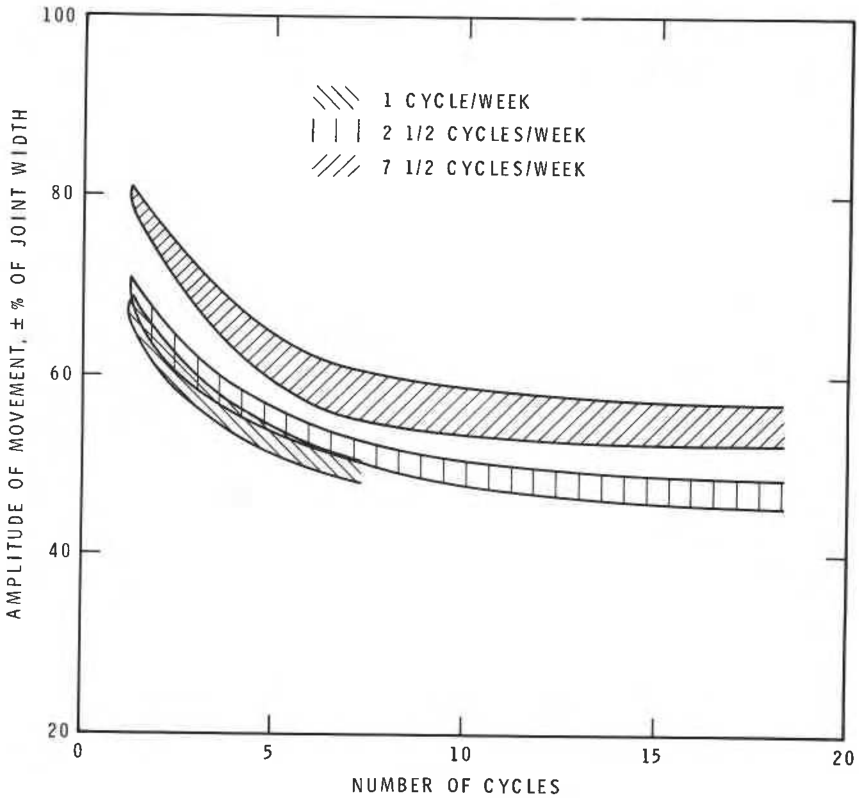


Fig. 9. Stage 3: perforation.

The best-fit lines can be extrapolated to longer times but other aspects of the mechanical properties of the sealant have first to be examined.

Failure envelope and the fatigue test

Previous work by Karpati (1973) showed that the basic mechanical properties of the same two-part polysulfide sealant can be characterized by the four primary variables: stress, strain, time, and temperature. By using time–temperature superposition, temperature can be eliminated and the remaining three variables used to describe the sealant by defining its failure envelope in a three-dimensional system. The envelope can be projected on various planes. From the point of view of sealant performance, the plane of strain-at-failure versus time-to-reach-failure is the most important since sealants, in general (and this one in particular), are not designed to have structural strength.

The failure envelope can be obtained by various methods, for example, by tensile tests (Karpati, 1973). The cycling test discussed here is a fatigue test

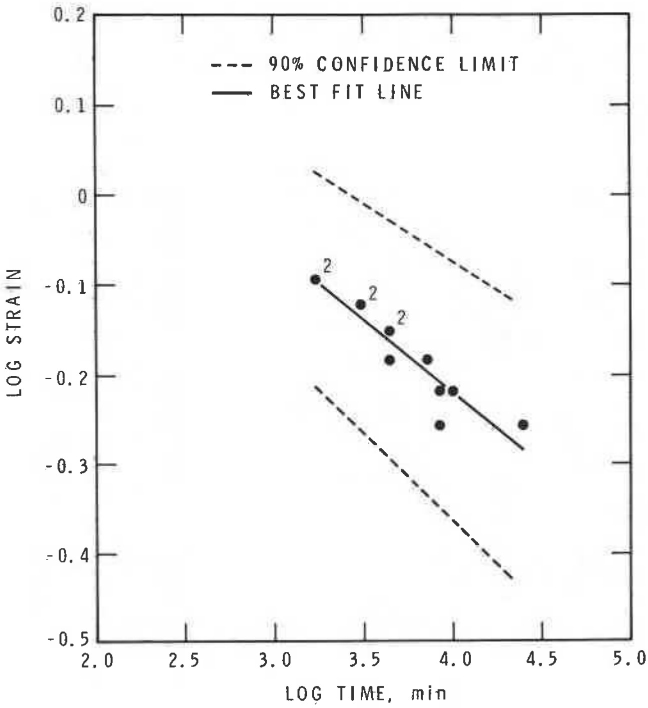


Fig. 10. Stage 3 results from $7\frac{1}{2}$ cycles/week test, log strain vs. log time.

that appears in the strain versus time plane as a sawtooth waveform that ends at the failure envelope (Landel and Fedors, 1964). To examine how the present results fit the failure envelope, the values from an earlier paper (Karpati, 1973, Fig. 15) are replotted in Fig. 15 (Log strain-at-break versus log time-to-failure, with the time scale adjusted by log a_T, the shift factor of the WLF (Williams, Landel, Ferry) equation (Karpati, 1973)). The figure in the earlier paper was for the reference temperature $T_0 = -34.5°C$ ($-30°F$), but the present one is for $T_0 = 23°C$ (73.5°F) since the laboratory fatigue test was carried out at this temperature.

In the upper section of Fig. 15 the tensile break points obtained in the previous work appear between arbitrarily drawn upper and lower failure limits. In the lower right section of the graph, parts of Figs. 10, 13 and 14 are reproduced. The larger vertically-shaded area at the bottom is stage 1 of the $7\frac{1}{2}$ cycles/week test, while the three smaller areas above it are, from left to right, stage 3 of the $7\frac{1}{2}$, $2\frac{1}{2}$ and 1 cycle/week tests. All stage 3 readings, i.e., failures obtained in the fatigue tests, are in the approximate region where the lower failure limit of the tensile tests would lie if extended below 100% movement. This descending part of the curve is generally expected for polymers as the time to reach failure increases.

The tensile break points could be made to occur in the area where the fatigue

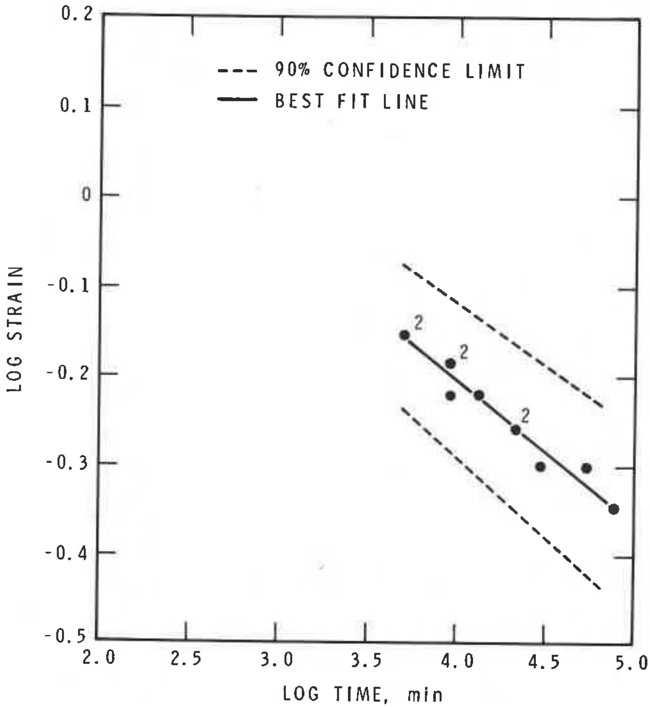


Fig. 11. Stage 3 results from $2\frac{1}{2}$ cycles/week test, log strain vs. log time.

test results are located by using a much higher test temperature than 38°C (100°F) or the same temperature with much lower strain rates than the lowest (0.1 mm/min) used in the earlier study (Karpati, 1973). The tensile tests appear to approach more closely the upper limit of the failure band while the fatigue test results are in the neighbourhood of the lower limit. Thus the fatigue test should be a more conservative method for evaluating sealants.

The individual readings shown as inverted triangles above the start of the stage 3 failure areas in Fig. 15 are for specimens that failed before the end of the first cycle. They are farther into the failure band because observations are normally taken at the end of cycles or at regular intervals so that early failures at higher amplitudes were observed after the event. These points were not included in the calculation of the best-fit lines because they would have biased it. Such failures did not occur at $7\frac{1}{2}$ cycles/week, for which the best-fit line is shown in Figs. 13 and 15.

Fatigue tests on exposure

To compare the results of the exposure tests to those from the laboratory tests, the right part of Fig. 15 is shown in Fig. 16 in light lines while the expo-

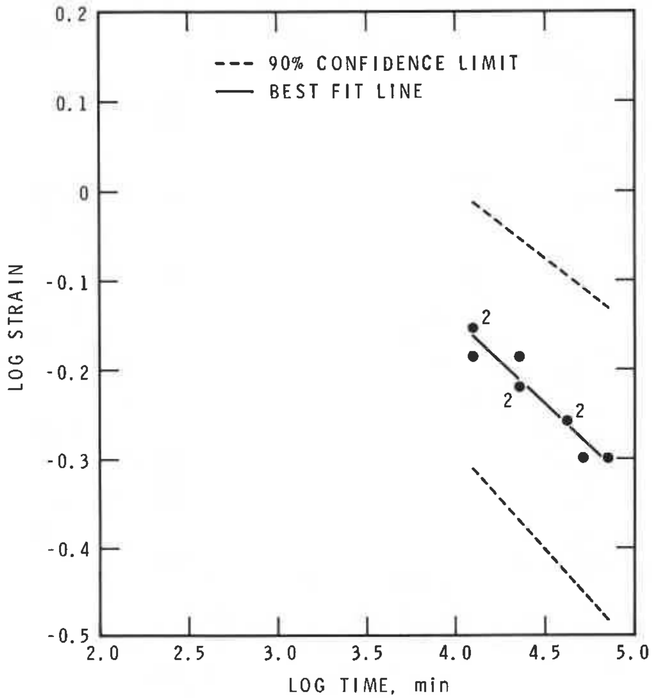


Fig. 12. Stage 3 results from 1 cycle/week test, log strain vs. log time.

sure results are in dark lines. For the field tests, sealant specimens were mounted on vises and subjected to a yearly movement cycle imposed by monthly manual adjustments. The two loops of stages 1 and 2 are indicated by × and +, respectively, and the single stage 3 result, which occurred after three years, is marked by an asterisk. Although these readings have been published (Karpati, 1985), they are presented here on the logarithmic scale, shifted along the time scale in accordance with the time–temperature superposition principle used in the previous paper. Because the specimens were exposed to outdoor temperature changes, the results cannot be plotted directly on the $T_0 = 23\,°C$ (73 °F) graph. Permanent deformation is accelerated at high temperatures and retarded by lower ones so that the mean temperature is considered to be a reasonable basis for determining the shift factor, log a_T of the WLF equation (Karpati, 1973). It was found to be 1 °C (34 °F) for the natural exposure in this study, resulting in the factor of 1.57 for shifting the values toward the lower readings of the time scale. The areas for stage 1 fall within the same general area of amplitude and time for specimens cycled on vises in the laboratory and outdoors. Stage 2 occurs at higher amplitudes for the exterior exposed specimens than for those in the laboratory since the area covered is approximately the same as that for

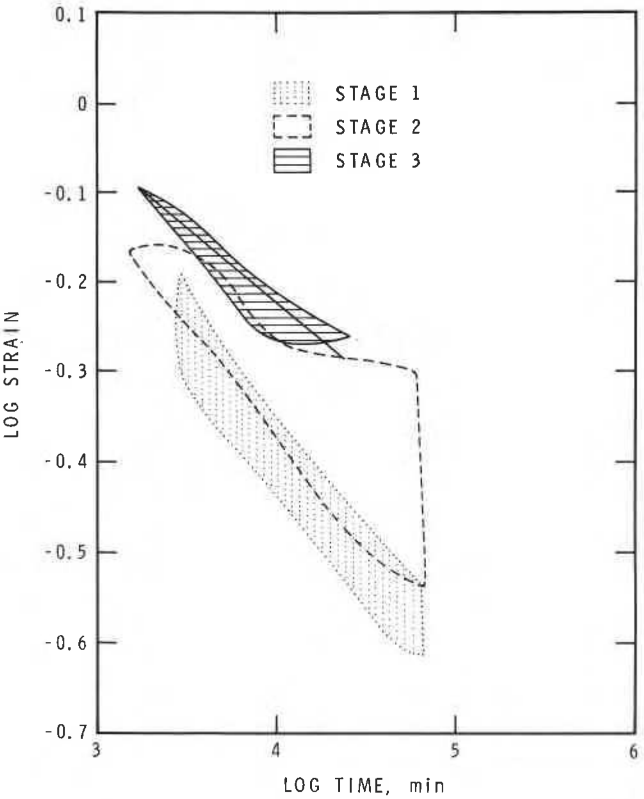


Fig. 13. Stages 1, 2 and 3 from $7\frac{1}{2}$ cycles/week test.

stage 3 of the 1 cycle/week test (see 1 cycle/week, stages 2 and 3 in Fig. 14, and stage 3 in Figs. 15 and 16).

The same sealant was also exposed on the strain-cycling rack (Karpati, 1984) where movement is continuous and follows the air temperature fluctuations. The values were shifted on the time scale in the same way as for the specimens exposed on vises. Stage 1 and 2 failures occurred as shown by the two squares in the lower right quarter of Figure 16. Because daily cyclic movement combined with exposure increases deformation, stages 1 and 2 occur at lower amplitudes than on specimens exposed on the vises where movement took place at monthly intervals only. Stage 3 could not be observed because the amplitude of movement was limited to $\pm 30\%$ on the strain-cycling rack and the observation time was limited to 3 years. Comparison with the vise-exposure results indicates that it should appear somewhere between $\pm 30\%$ and $\pm 45\%$. Since stage 2 on the strain-cycling rack falls on the upper edge of the area describing stage 1 permanent deformation of the sealant on the exposed vise and in the

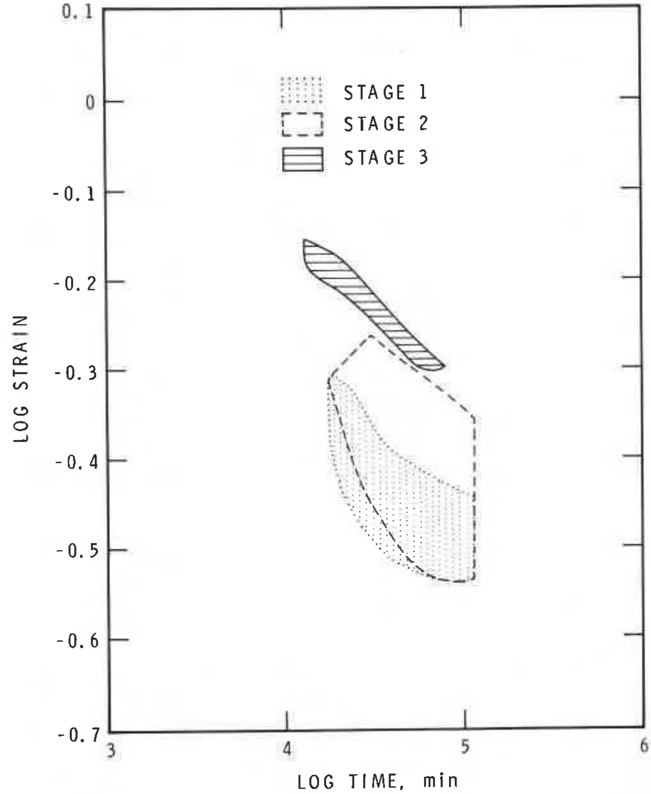


Fig. 14. Stages 1, 2 and 3 from 1 cycle/week test.

laboratory vise tests, the latter can safely be used as the limit below which the sealant does not fail in a joint in three years: i.e., $\pm 30\%$ movement.

Extrapolation for long periods

Although the results do not provide information on exposures for longer than three years, it is possible to predict long-term performance. The best-fit lines of stages 3 of the laboratory fatigue tests (Figs. 10 to 12) give movements of $\pm 19\%$, $\pm 21\%$ and $\pm 19\%$ for $7\frac{1}{2}$, $2\frac{1}{2}$ and 1 cycle/week, respectively, when extrapolated to 20 years. Values for stage 1 from $7\frac{1}{2}$ cycles/week fall in a relatively narrow band that decreases with increasing time. The amplitude reaches $\pm 7\%$ to 9% when extended to 20 years. Stage 2 for the $7\frac{1}{2}$ cycle rate and both stages 1 and 2 for the other two test rates cannot be extrapolated, however, because these areas widen on the logarithmic scale (on a non-logarithmic scale they flatten out, as may be seen in Figs. 7 to 9). The two extrapolations suggest that failure would be reached between $\pm 5\%$ and $\pm 20\%$ in 20 years. The pre-

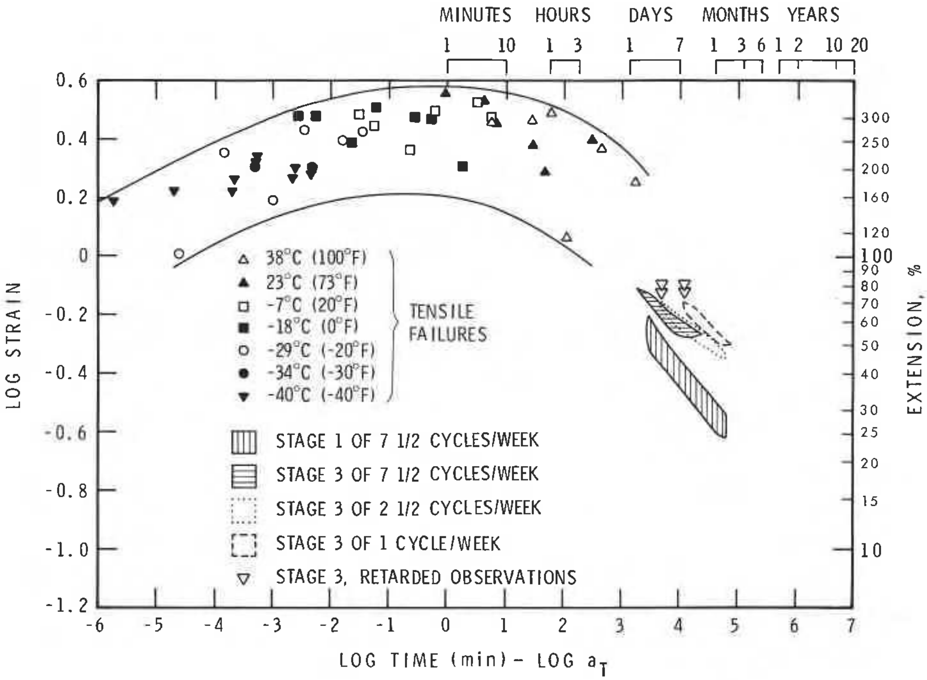


Fig. 15. Tensile strain at break and laboratory fatigue test. Tensile results (from Karpati (1973), Fig. 15) shifted to T_0=23 °C (73 °F).

dicted values could be lowered by dimensional irregularities in a real joint and could probably be increased by increased depth of the sealant.

CONCLUSION

All three stages of permanent deformation previously observed on specimens mounted on vises and exposed outdoors have been produced in the laboratory. They provide the relationship between laboratory test results and those obtained from 3 years of exposure on the strain-cycling rack. From the laboratory test it is possible to estimate the amplitude of movement that would cause stage 3, which did not occur in the rack owing to limitations in available amplitude and exposure time.

The results could be correlated on the basis of amplitude alone but it is theoretically more appropriate to evaluate sealant behaviour in a three-dimensional co-ordinate system and project the results onto the log strain versus log time plane. In this way it has been shown that the fatigue test results are consistent with the failure envelope delineated earlier by tensile tests. As the failures produced in the laboratory with vises occur in less than a month, there is a considerable saving in time and in the cost of using tensile testing machines.

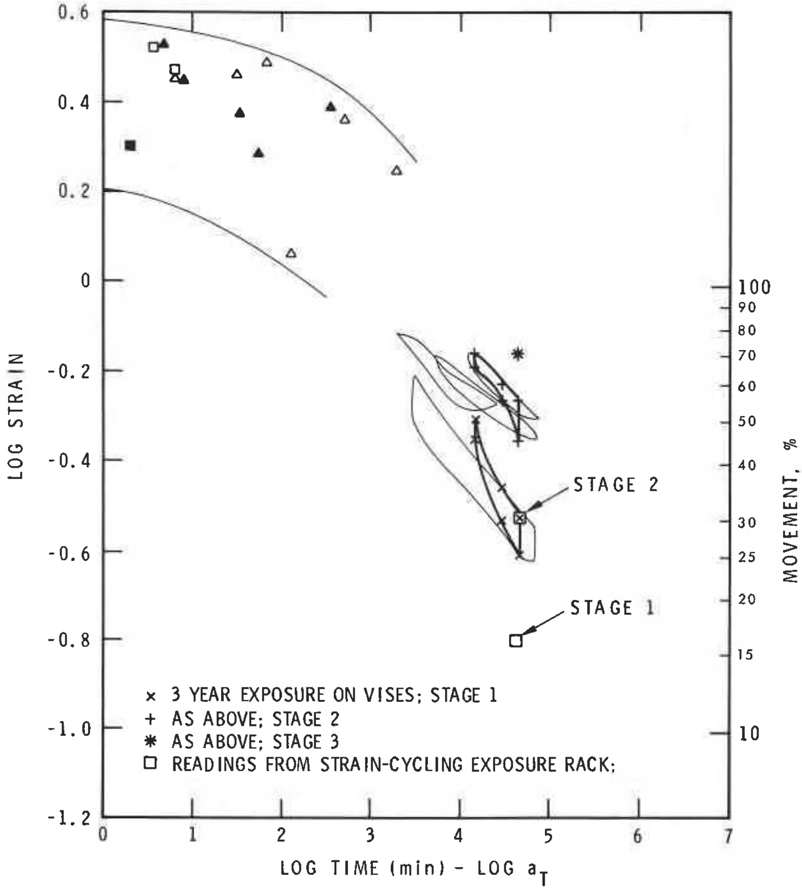


Fig. 16. Tensile and laboratory fatigue test results (light lines) versus exposure results shifted to $T_0 = 23\,^\circ C$ (73 °F) (heavy lines).

It is proposed that the $7\frac{1}{2}$ cycles/week fatigue test be used to rate polysulfide sealants that fail by permanent deformation and that the stage 1 deformation be used as the criterion in predicting behaviour in building joints after 3 years of exposure.

The test method could be used to predict the performance of other types of sealant that fail by the same mechanisms of progressive permanent deformation caused by cyclic movement. Long control tests involving outside exposure on the strain-cycling rack are needed, however, to determine the validity of the test.

Because the fatigue test approach is not specific it should be applicable to substrates other than the one used in this investigations. It could also be used to test sealants immersed in water without the complications that carrying out such a test on a tensile machine would entail.

ACKNOWLEDGMENT

The author wishes to thank Colette Niessen for experimental assistance and H.E. Ashton for assistance in preparing the paper. This paper is a contribution from the Institute for Research in Construction, National Research Council of Canada.

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