Mechanical properties of sealants: II. Behavior of a silicone sealant as a function of rate of movement

Karpati, K. K.

Publisher's version / Version de l'éditeur:

Journal of Paint Technology, 44, 569, pp. 58-66, 1972-06

NATIONAL RESEARCH COUNCIL OF CANADA
CONSEIL NATIONAL DE RECHERCHES DU CANADA

Mechanical Properties of Sealants: II. Behavior of a Silicone Sealant As a Function of Rate of Movement

by
K. K. Karpati

Reprinted from
JOURNAL OF PAINT TECHNOLOGY
Vol. 44, No. 569, June 1972
p. 58-66

Research Paper No. 524
of the
Division of Building Research

OTTAWA
June 1972

Price 25 cents

NRCC 12652

PROPRIETES MECANIQUES DES PRODUITS DE SCELLEMENT. [illegible] LE COMPORTEMENT D'UN PRODUIT DE SCELLEMENT DE SILICONE COMME FONCTION DU TAUX DE MOUVEMENT

Résumé

On a examiné le comportement mécanique des produits de scellement de silicone à six différents taux d'extension (temps) et à trois températures. On a choisi des taux d'extension couvrant les taux ordinaires d'essai en laboratoire et les taux les plus élevés de mouvement qui se produisent dans les joints de dilatation des édifices, lesquels ont été évalués et déterminés de façon expérimentale. Les échantillons utilisés étaient des modèles des pièces de scellement employées dans les joints d'édifices. On a étudié la possibilité d'appliquer des renseignements théoriques à l'échantillon modèle, dont le champ de contrainte était compliqué. On pourrait construire une seule courbe à partir des courbes de tension individuelles obtenues à des taux différents pour chaque température.

Mechanical Properties of Sealants

II. Behavior of a Silicone Sealant As a Function of Rate of Movement

K. K. KARPATI
National Research Council of Canada*

The mechanical behavior of silicone sealants has been examined at six different rates of extension and at three temperatures. The rates of extension used were chosen to cover the usual laboratory testing rates and the highest rates of movement occurring in expansion joints of buildings. The latter were estimated and experimentally determined. The samples used were model representations of sealant beads as used in the joints of buildings. The possibility of applying theoretical information to the practical model sample, which had a complicated stress field, was investigated. A single curve could be constructed from the individual tensile curves obtained at varying rates at each temperature.

KEY WORDS: Sealants; Building sealants; Silicone sealants; Mechanical properties of sealants; Rate of movement dependence of mechanical properties of sealants; Rate of movement in building joints; Joint movement; Building joint movement.

INTRODUCTION

THE design of meaningful test methods for sealants to provide results that correlate with the performance of the materials in service can be approached in two ways.

(1) If the performance of a sealant is known over a long period of time, preferably the lifetime of a building, test methods that will distinguish between good and bad sealants can be designed through a purely empirical approach by comparison with the known good performer. There are pitfalls to this method, however, in that the passing of one set of test methods by a new material does not mean that all aspects of its behavior have been covered. The new material may have characteristics that were not present in the reference material; test methods designed on the basis of the reference material, therefore, would not reveal these new characteristics. It usually turns out that the new material has to prove itself in practice for a considerable length of time just as the acceptable material had done.

(2) The other approach to designing meaningful test methods is to study and understand fundamental properties in order to design tests for all eventualities. This paper is intended to be a step in that direction. In order to accomplish this, it is necessary first to assess the basic factors influencing the performance of sealants and consider their relative importance. The possible factors are: stress, strain, time (rate of extension), temperature, humidity, oxygen, light, and type and condition of substrate. Of these factors, stress, strain, time, and temperature are considered to be of primary importance. They have to be known simultaneously in order to describe a material at any given age, humidity, or substrate condition. Consequently, these four factors have to be included in any test method devised to measure mechanical properties, while the other variables have to be kept constant at values considered to be realistic.

SELECTING TEST METHOD

After deciding which variables should be investigated, the next step is to select the test method. A good performance-oriented test should be a fatigue test. It is a very complex test, however, and can be satisfactorily designed only if the material properties are well known from the results of tests using simpler loading patterns. Tensile extension at constant rate, stress relaxation under constant strain, and creep under constant stress are three of the test methods describing the material properties of polymers through the factors of stress, strain, time and temperature.

The tensile extension test was chosen in this in-

* Building Materials Section, Div. of Building Research, Ottawa, Canada, KIA OR6.

stance for several reasons: (1) it is the least time-consuming; (2) most laboratories are equipped for it; and (3) among the various ways sealants can be loaded in practice, this type of loading is most likely to lead to failure.

SELECTING SAMPLE GEOMETRY

The size and shape of the test specimen should be considered next. This was discussed in a previous paper[1] in which the temperature dependence of the mechanical properties of sealants was examined. The specimen used there, as well as in this work, was essentially a model representation of a sealant bead in a butt joint; the sealant bead dimensions were ½ × ½ × 2 in. (1.3 × 1.3 × 5.1 cm) as illustrated in *Figure* 1. A specimen with this geometry has a complicated stress field[2] as opposed to dumbbell- or ring-shaped specimens which have a parallel stress field that can be treated theoretically. The latter samples, however, are not useful in finding a simple test method that is performance oriented because the conditions of failure are extremely different from those in actual practice. This can be illustrated by a comparison of the values of extension at failure at room temperature determined at 0.05 in./min (0.127 cm/min) rate of extension for different types of samples with silicone sealant. The extension at failure averages approximately 80% on aluminum substrate with the model sample, while dumbbell-shaped specimens usually fail at over 200% elongation, in both cases cohesively. In addition, the model samples can fail adhesively as often occurs in practice. The example shows that there is no simple way in which performance-oriented test methods can be derived with samples having a parallel stress field, consequently, model samples have to be used. It is then necessary to establish whether theoretical considerations can be applied to test results obtained from specimens related to practical applications. If this can be done, the empirical content of the test method could be diminished to a point where laboratory testing relates directly to outdoor performance.

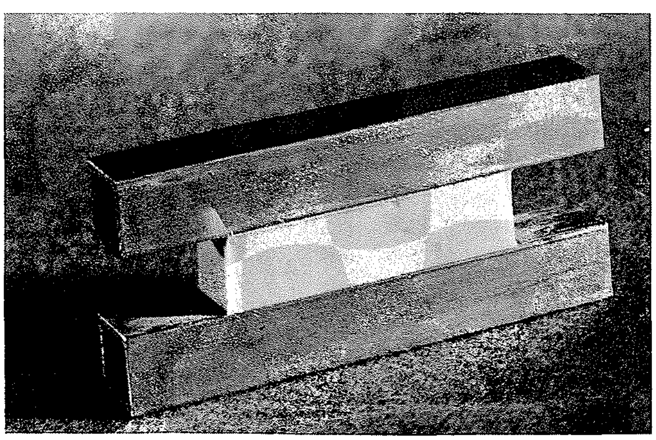

Figure 1—Model specimen

It has been demonstrated that the results of tensile experiments executed at constant rate of extension using the previously described specimens produce different curves as a function of temperature.[1] An examination of *Figures* 2 to 4 demonstrates that the tensile curve changes not only with temperature but with rate of extension as well. An infinite number of curves can be produced by varying either the temperature, or the rate, or both. The question arises which and how many of these conditions should be chosen in a laboratory testing method for a true representation of the mechanical properties of the sealant. The rate of extension is the time factor required above for the characterization of the material and is distinctly different from the time factor involved in the aging of materials; the latter will be kept constant in this work.

PIGMENTATION

Apart from the difficulty represented by the complicated stress field of the specimen, another difficulty in trying to apply test methods of theoretical value to sealants is that sealants are usually pigmented which may influence the results. The silicone sealants used, however, contained only a small amount of insoluble residue, presumably a thixotropic agent. The contribution to the strength of the cured sample of this material is negligible and its effect can be disregarded.

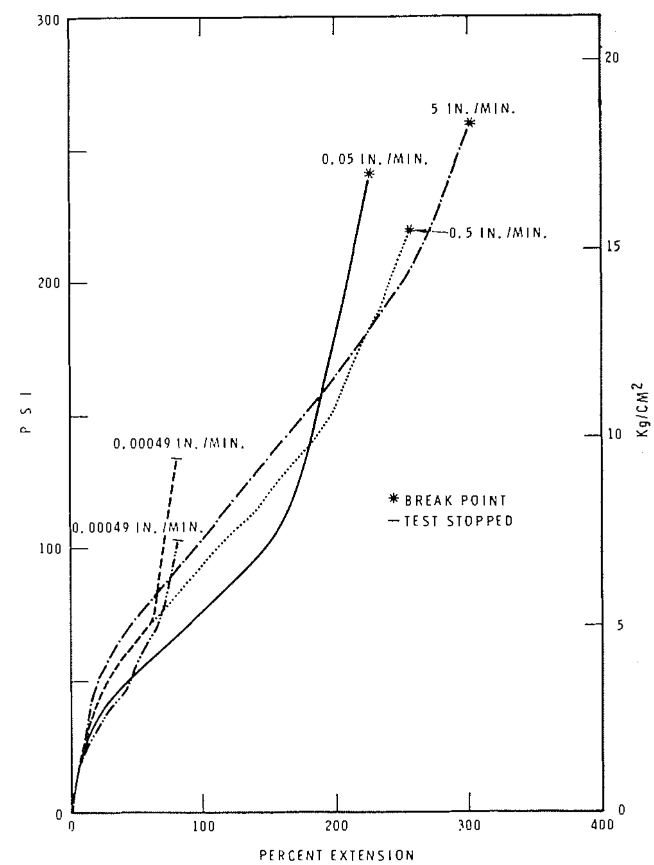

Figure 2—Tensile tests at various rates of extension, at —50 F (—46 C)

BACKGROUND INFORMATION

Thor L. Smith's experiment,[3] and others cited by him, demonstrated that the infinite number of stress-strain curves that can be obtained by varying time (i.e., strain rate) and temperature can be replaced by one master curve for polymeric materials. His samples were either ring- or dumbbell-shaped specimens that undergo deformation uniaxially and, therefore, lend themselves to theoretical calculations. Although only polyisobutylene rubber was used in Smith's investigation,[3] the procedure was applied in later works to other rubbers.

When a constant strain rate, R, is applied to a material, Smith gives the stress, S, as:

$$S/R = \int_{-\infty}^{\infty} M(\tau)\ \tau (1 - e^{-\gamma / R_t})\, d\, ln\, \tau \qquad (1)$$

where τ is the relaxation time, $M(\tau)$ is the relaxation distribution function, and γ is strain. He points out that equation (1) states that S/R is a function only of γ/R, i.e., when the stress and the corresponding strain are divided by the strain rate of the experiment all data obtained at a given temperature should superimpose on a single curve on a plot of log S/R vs log γ/R. His experiments substantiated this. Data obtained at different temperatures give different log S/R vs log γ/R curves for materials that are temperature dependent. A single master curve can be obtained from the curves obtained at different temperatures by applying time-temperature superposition according to the WLF equation.[4]

If the tensile curves obtained with model samples follow equation (1) at each temperature, i.e., the tensile curves superimpose to form a single curve, then the way is open to apply background information to a practical situation such as the performance of the sealant in the joints of buildings through the simple tensile tests used here.

Before proceeding to testing, one has to choose the experimental conditions that cover the temperature range and the rate of joint movement range occurring in practice. The latter is dependent on the former; they are discussed together in the next section.

RATE OF CYCLIC MOVEMENT IN BUILDING JOINTS

A reasonably good sealant material in a joint not subject to movement may last indefinitely, provided it has adhesion to the substrate. In a moving joint, however, the sealant is compressed or extended and if the

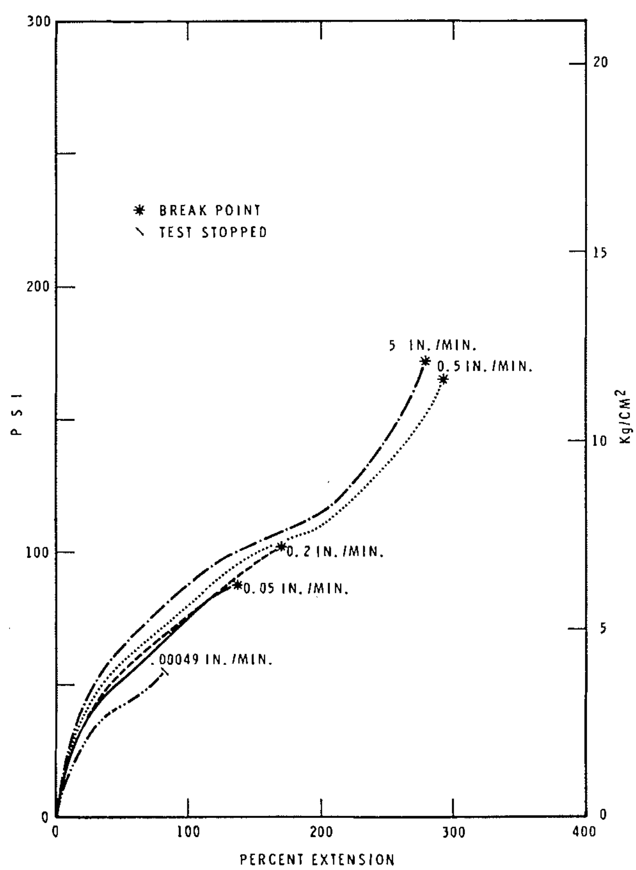

Figure 3—Tensile tests at various rates of extension, at —10 F (—23 C)

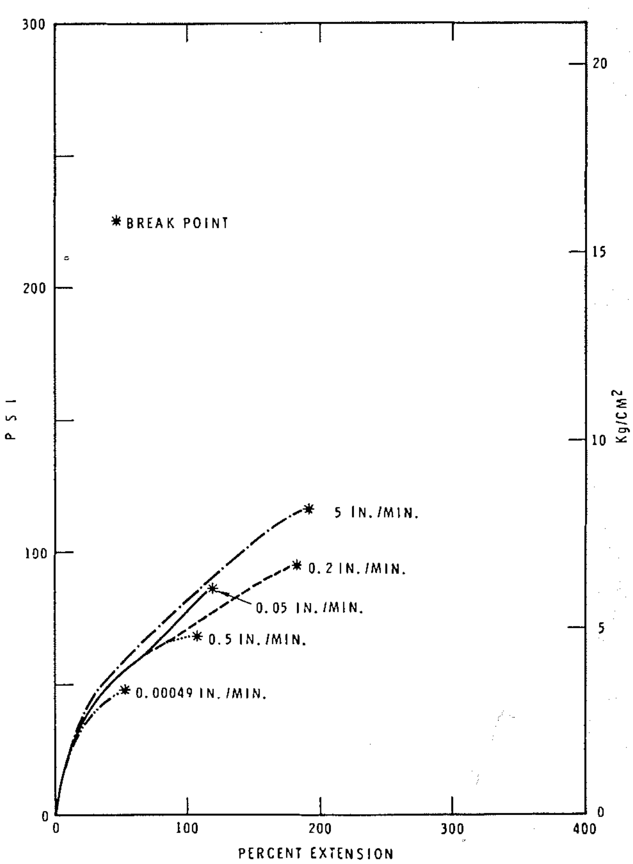

Figure 4—Tensile tests at various rates of extension, at 72 F (22 C)

stresses build up in the material to a critical level, failure may occur. It takes more time for failure to occur in slow movement than in fast movement. Consequently, the fastest joint movement has the greatest chance of failure because there is reversal of the movement almost every day due to the weather conditions. Therefore, it is the fastest joint movement that one has to estimate and include in the testing rates. At the same time, if this rate is the slowest testing rate, the time necessary to perform the work is kept within reasonable limits.

The fastest rate of joint movement can be estimated from meteorological data and from the maximum joint movement expected. It has been established[5] that there its a fairly high correlation between joint movement and meteorological temperature readings for a given geographical region. The joint width —or the strain in the sealant—changes approximately sinusoidally with the temperature, making one full cycle per year on which daily sinusoidal cycles are superimposed. In order to derive an estimate for the maximum strain rate in the sealant, values have to be assumed for the yearly and daily temperature changes and for the maximum allowable strain of the sealant. For the latter, the sealant manufacturers' recommendation, which is maximum ±25% joint width change, i.e., ±0.25 strain of the sealant, can be used. When this figure is reduced in the proportion of the yearly and daily temperature changes given here, one obtains ±8.3% joint width change, or ±0.083 strain of the sealant per day. The maximum daily strain rate is the slope of the assumed sinusoidal curve of the daily strain as a function of time as illustrated in *Figure* 5. The equation of the curve is:

$$\epsilon = A, \sin \omega t$$

where ϵ is strain, A is the amplitude and t is time.

The slope, i.e., the strain rate, is:

$$\dot{\epsilon} = A \omega \cos \omega t$$

If $A = 0.083$, $\omega = \frac{2\pi}{T}$ where $T = 24 \times 60$ minutes, and t is chosen as 1 minute, then the strain rate is: $\dot{\epsilon} = 0.0004$ minutes^{-1} (or, 0.04% width change per minute).

As part of the work reported in reference 5, the maximum rate of joint movement was determined from direct readings of continuous charts obtained with an instrument described in reference 6. The maximum rate of movement was found to be 0.00038 in./min (0.00097 cm/min) for one building, and 0.00055 in./min (0.0014 cm/min) for another, during 1969-70. In both cases the nominal joint width was one inch.

For movements due to daily temperature changes, Patarcity and Giordano[7] indicate a rate of movement

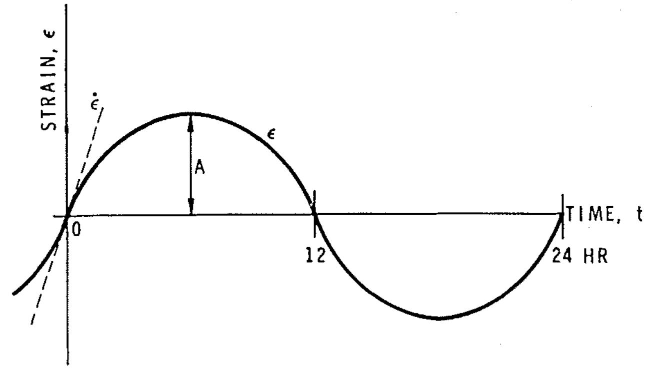

Figure 5—Assumed sinusoidal strain change as a function of time

of 0.0014 in./min (0.0036 cm/min) while Ryder and Baker[8] obtained less than 0.0008 in./min (0.0021 cm/min) maximum rate of movement on buildings. Patarcity and Giordano cited 6000 in./min (about 15,000 cm/min) as the rate of movement due to wind effects or vehicular traffic. None of the references, however, give the nominal widths of the joints on which the observations were made. It is evident that it is the percentage extension that matters from the point of view of sealant performance and it is this percentage value that should be used in comparing rates of movement for joints of various widths. As this was not available in the literature, the minimum rate of movement chosen for testing purposes was a compromise between the preivously estimated rate readings and practical considerations concerning the time required for the experiment. As a consequence, the lowest rate used was 0.00049 in./min (0.00124 cm/min). This is twice the rate of movement estimated on the building joints when expressed in percentage joint width because the model joint used for testing was only ½ in. wide and, therefore, the percentage elongation per minute (or strain per minute) was double: about 0.1 percent/min of the model joint width.

It has to be noted that much higher rates than those just mentioned can occur due to vibration or the effect of wind load on joints. The resulting movement, however, is likely to be a small percentage of the joint width and of the expected maximum width change. The rates used here cover the range that is important. At very high rates only the elastic component of viscoelastic behavior is likely to respond. This will not cause permanent deformation due to time-dependent stress relaxation; it should have little influence, therefore, on the failure properties when occurring only at small amplitudes.

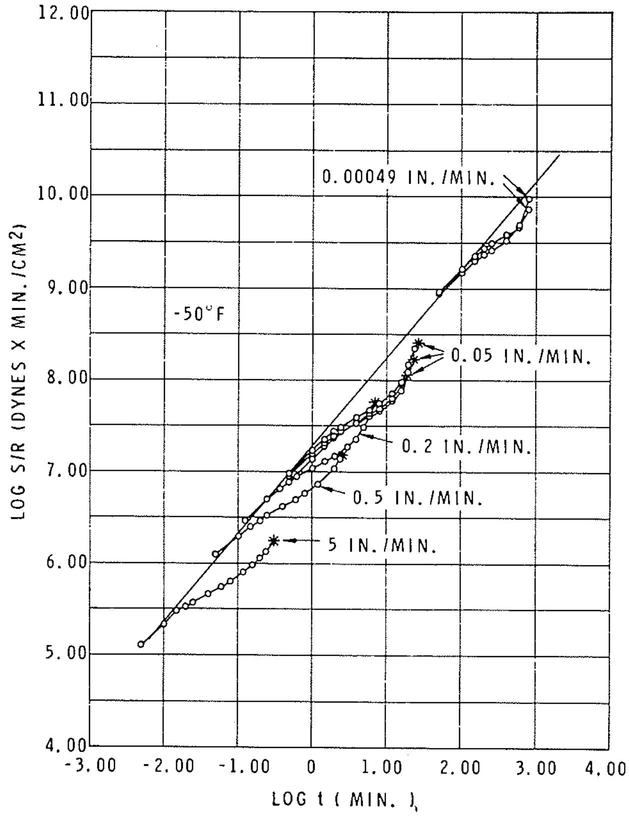

Figure 6—Tensile tests at various rates and at —50 F (—46 C)

EXPERIMENTAL

Based on conclusions from a previous paper,[1] one brand of a one-part chemically curing white silicone sealant was chosen for the investigation; it was applied on primed aluminum substrate. The samples were cured for three to four weeks under the same conditions as those in which they were cast, i.e., 72 F ± 2 F (22 C) and 50 ± 2% relative humidity.

The rates of extensions used were as follows: 5.0, 0.5, 0.2, 0.05, and 0.00049 in./min (12.7, 1.27, 0.508, 0.127, and 0.00124 cm/min, respectively). The crosshead speed accuracy was better than 0.5%. As previously discussed, the lowest rate used for testing was in the same order as the highest rate observed on expansion joints of buildings. The tests were done on a Tinius Olsen tensile machine that has a minimum rate of extension of 0.002 in./min. The lowest test rate was produced by an automatic on/off switch inserted in the circuit of the tensile machine which allowed the machine to pull for approximately ¾ of a minute out of three. Using the lowest crosshead speed (0.002 in./min) the average rate achieved was 0.00049 in./min. Because of the extremely time-consuming nature of this low extension rate test and because the aim was to establish whether a master curve could be obtained for the sealant using shapes of practical importance, it was considered sufficient to carry these extensions only to 80%.

Tests were performed at three different temperatures: 72 F, —10 F (—23 C), and —50 F (—46 C), using the previously mentioned rates at each one. An Aminco Sub-zero test cabinet was used to produce lower than room temperatures. The reproducibility of test temperatures was ±2 F (±1 C), the same as at room temperature.

DISCUSSION

As stated earlier, the individual tensile curves obtained at the conditions described should superimpose on a single curve on a plot of log S/R vs log γ/R.

Figures 2 to 4 represent the original data where, for clarity, some of the tests were omitted. When tests were repeated they usually followed the same shape of curve and relative position compared with the axes. The difference occurred in the breakpoints which stopped the curve at various phases. Because of the scatter of the breakpoints it is often difficult to see the pattern of behavior caused by the change in rate of elongation. A closer examination reveals, however, that at —10 F and at 72 F an increase in the rate of extension causes the curves to shift toward higher load values. At —50 F the same behavior applies below approximately 50% extension. At higher values the curves obtained at lower rates of elongation tend to

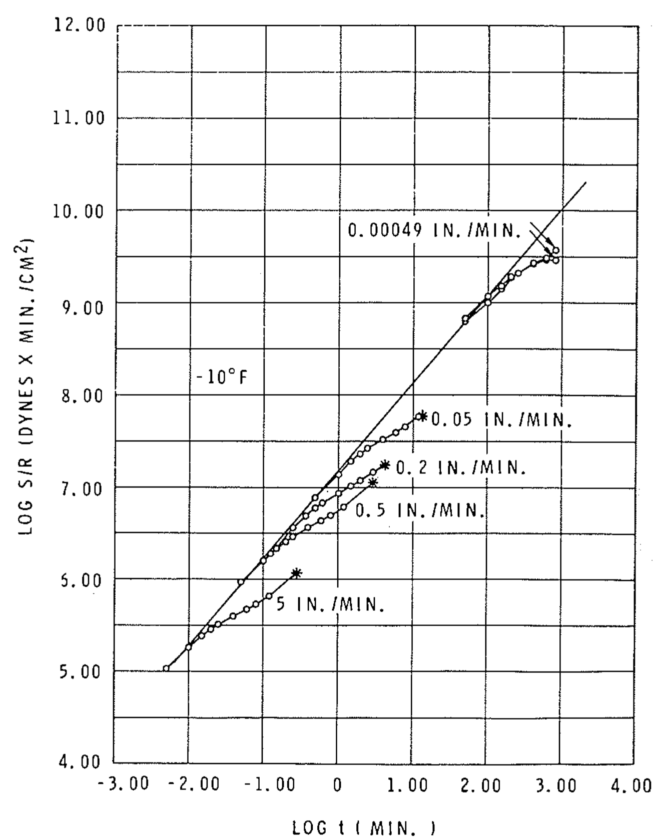

Figure 7—Tensile tests at various rates and at —10 F (—23 C)

cross the curves of higher rate and carry considerably higher load at the same percent elongations. The lower the rate of extension the lower is the percent extension at which the crossing starts.

Figures 6 to 8 illustrate the results on a plot of log S/R vs log t. The latter is derived from and equivalent to log γ/R; it is, therefore, the time necessary to reach a given elongation. Each short curve corresponds to one of the original tensile curves. In order to make calculations easy to program for a computer, each original tensile curve was read at the following percent extensions only: 5, 10, 15, 20, 25, 40, 60, 80, 120, 160, 200, and 240%, and at the breakpoint. The plotted points indicate these readings. The single straight line in all three figures is the best fitting line for the 5% readings. The smaller the percent elongation at which readings are taken the nearer to parallel pattern is the stress field of the sample. Due to experimental difficulties, 5% elongation was considered to be the smallest stress reading that could be taken with good accuracy. It can be seen from the curves that the 10% elongation readings, and even the 15% ones in most cases, fall reasonably close to the 5% regression line, i.e., the low extension readings approach the ideal situation of parallel stress field.

With polymer samples undergoing uniaxial elongation Smith found empirically[3,9] that equation (1) was suitable to represent a polymer provided the stress was replaced by the true stress, i.e., the force divided

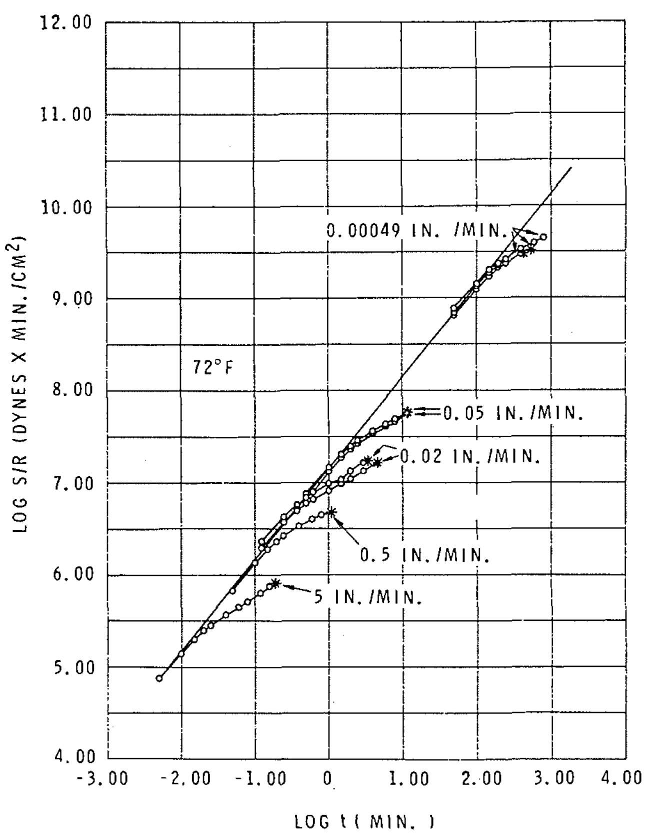

Figure 8—Tensile test at various rates and at 72 F (22 C)

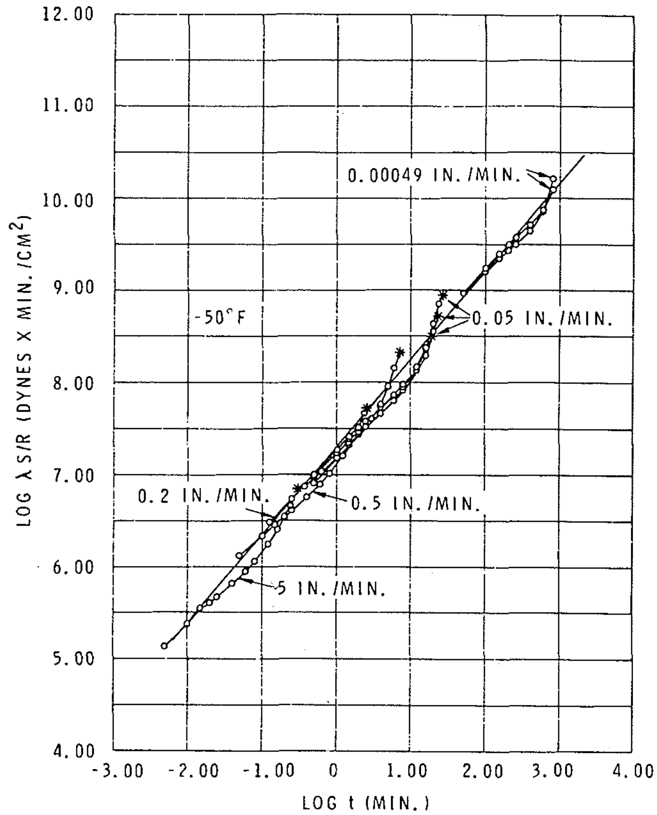

Figure 9—Tensile tests at various rates and at —50 F (—46 C), load corrected by extension ratio

by the cross-sectional area of the elongated specimen. The true stress for elastomers was found by Smith to be λS where λ is the extension ratio. This correction applied to the data in *Figures* 6 to 8 is illustrated in *Figures* 9 to 11. The multiplication by λ brings all the points within close range of a straight line, representing the best fitting line of the 5% readings, at —10 F and at 72 F. The scatter is larger at —50 F because of the upturn demonstrated by the original tensile curves illustrated in *Figure* 2.

As all readings are close to the 5% regression line, it is reasonable to do a regression analysis on all readings at each temperature. The results are summarized in *Table* 1. The comparison of the 5% regression line with that of all readings at a given temperature illustrates that the slopes are identical within the 95% confidence limits, because the confidence limits overlap. A comparison of the intercepts at the same confidence level does not show, as a whole, as good agreement as that for the slopes. There is overlapping at 72 F but not at the other temperatures. There would be, however, ample overlapping if the confidence limits were widened to the 99% level. This would not change ta (the limits for the intercept at the 95% confidence level) of all readings at —10 F at all and would raise it to $\pm$0.03 at —50 F. For the 5% readings ta would increase considerably due to the small number of observations. As ta = $\pm$0.02 is equivalent to less than $\pm$ 5% error in the intercept on a linear scale

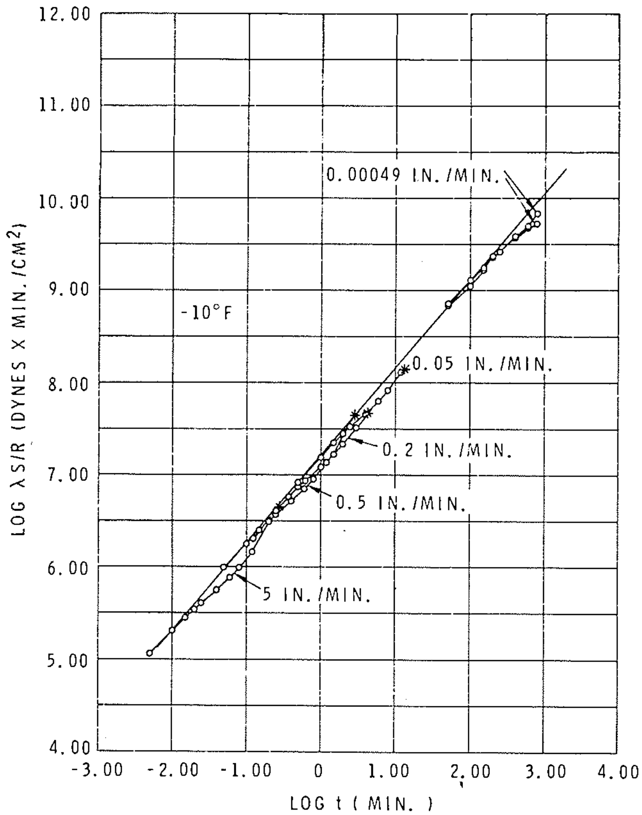

Figure 10—Tensile tests at various rates and at —10 F (—23 C), load corrected by extension ratio

when all readings are considered for both 72 F and —10 F and at both confidence levels, one can conclude that the accuracy is good even if all readings are treated together at a given temperature.

As a result of the analysis so far, it can be seen that the individual tensile curves superimpose on a single curve at each temperature with acceptable accuracy.

One could proceed now to a comparison of the regression lines of all readings obtained at different temperatures. The comparison of the available data indicates that there are some differences in the slope and there is a very small increase in the intercept as the temperature decreases. The latter differences are nowhere near the values that would be required to make a time-temperature superposition of the data. It is considered, however, that the data obtained at three temperatures only are not sufficient to demonstrate convincingly the absence of differences in the behavior at different temperatures, or to establish the feasibility of a time-temperature superposition. The experiments need to be extended to other temperatures before a conclusion can be drawn in this respect.

CONCLUSION

It has been demonstrated that the specimen geometry approximately representing the silicone sealant in butt joints used on buildings provided data to which theoretical considerations could be applied within acceptable accuracy. The proof was that the individual tensile curves obtained with the samples at different rates of extension superimposed on a single curve at each temperature. Because, ultimately, one would like to correlate laboratory test rates to rate of movement occurring in joints, the rates of extension used were chosen to cover both situations. Rate of movement expressed in percentage joint width, not being available in the literature, was estimated from the slope of the assumed sinusoidal change of strain with time and was also determined experimentally. The highest rate estimated, as well as observed, was of the same order as the lowest testing rate used. Due to the large variation of conditions which influence the rate of movement of joints one cannot give an exact figure for the highest rate but it was estimated to be approximately 0.0004 in./min (0.00102 cm/min) which was equivalent to 0.04%/min movement of the building joint width. It has been pointed out that the rate of movement of joints should be expressed in percentage joint width per minute (or strain per minute) instead of inches per minute in order to make the data of different experimentors comparable.

For the silicone sealant the mechanical properties seem to be independent of temperature within the range observed. The exact dependence on temperature, or its independence of it, cannot be established

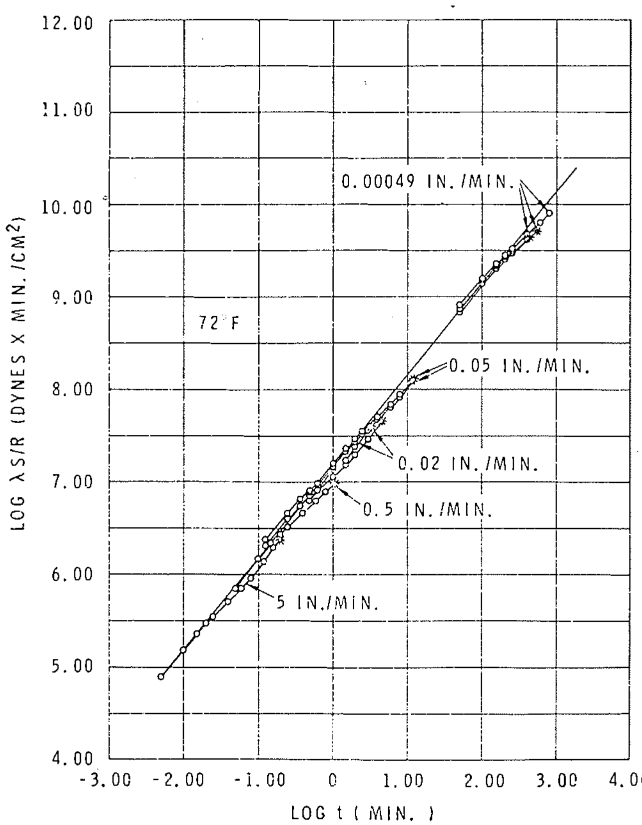

Figure 11—Tensile tests at various rates and at 72 F (22 C), load corrected by extension ratio

Table 1—Statistics of Log λS/R (dynes × min/cm²) vs log γ/R(min)

Test Temperature	5% Regression Line*							Regression Line of all Readings						
	n	r	a	ta	b	tb	s_e	n	r	a	ta	b	tb	s_e
72 F (22 C)	9	0.999	7.17	±0.05	0.99	±0.03	0.06	79	0.999	7.11	±0.02	0.98	±0.01	0.07
—10 F (—23 C)	6	1.000	7.21	±0.04	0.95	±0.03	0.03	56	0.999	7.13	±0.02	0.94	±0.01	0.06
—50 F (—46 C)	8	0.999	7.31	±0.05	0.96	±0.04	0.06	90	0.996	7.22	±0.02	0.97	±0.02	0.11

* n: number of readings
r: correlation coefficient
a: intercept
ta: limits for the intercept at the 95% confidence level
b: slope
tb: limits for the slope at the 95% confidence level
s_e: standard error

with accuracy, however, from the small number of data available.

The usefulness of the practical sample for the particular purpose of this paper being established, a link exists between the practical situation and a theoretical treatment of sealant behavior problems. Consequently, in future work, the criteria of failure can be studied as a function of the primary variables, stress, strain, time and temperature. Experiments have been started in this direction on artificially-aged silicone sealant samples with the ultimate aim of correlating tests with performance.

It is hoped that the idea of applying theory to a practical situation within probably large but acceptable errors can be extended to other types of sealant materials too, and as a consequence a firm basis for designing test methods can be established.

ACKNOWLEDGMENTS

The author wishes to thank S. W. Raymond and G. M. Polomark for their assistance in executing the tests and H. F. Slade for his help with the instrumental set-up. This paper is a contribution from the Div. of Building Research, National Research Council of Canada, and is published with the approval of the Director of the Division. ♦

References

(1) Karpati, K. K., JOURNAL OF PAINT TECHNOLOGY, *44*, No. 565, 55 (1972).
(2) Catsiff, E. H., Hoffman, R. F., and Kowalewski, R. T., *J. Appl. Polymer Sci.*, *14*, 1159 (1970).
(3) Smith, T. L., *J. Polymer Sci.*, *20*, 89 (1956).
(4) Williams, M. L., Landel, R. F., and Ferry, J. D., *J. Am. Chem. Soc.*, *77*, 3701 (1955).
(5) Karpati, K. K., and Gibbons, E. V., Mat'ls. Res. & Std., *10*, 4, 16 (1970). Karpati, K. K., American Concrete Institute Conference (1970).
(6) Gibbons, E. V. and Karpati, K. K., Mat'ls Res. & Std., *9*, 4, 18 (1969).
(7) Patarcity, A. and Giordano, J., Building Research Institute Conference (1968).
(8) Ryder, J. F. and Baker, T. A., International Symposium for the Construction Industry (1970).
(9) Smith, T. L., *Trans. Soc. Rheo.*, *6*, 61 (1962).