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Karpati, K. K.

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Mechanical Properties of Sealants: III. Performance Testing of Silicone Sealants

by
K. K. KARPATI

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LES PROPRIETES MÉCANIQUES DES PRODUITS DE SCELLEMENT ESSAIS DE FONCTIONNEMENT DE PRODUITS DE SCELLEMENT DE SILICONE

SOMMAIRE

Le comportement mécanique d'un produit de scellement de silicone à une partie et de cure chimique est défini selon ses variables primaires: la contrainte, la déformation, le temps et la température, au moyen d'un échantillon modèle. Le matériau s'est montré indépendant de la température dans la région importante pour les produits de scellement, avec quelque déviation de la règle près de la température de cristallisation lorsque la déformation est élevée. Les trois autres variables décrivent le comportement dans un système de coordonnées à trois dimensions. La projection des points de rupture dans le plan du logarithme déformation vs le logarithme temps constitue le lien entre les essais en laboratoire et le fonctionnement à l'extérieur. En extrapolant la ligne de régression des points de rupture sur une période de six mois, il est possible de prédire la capacité d'allongement du matériau. On propose une méthode d'essai appropriée aux normes qui fait le lien entre les essais en laboratoire et le fonctionnement à l'extérieur.

Mechanical Properties of Sealants

III. Performance Testing Of Silicone Sealants

KLARA K. KARPATI
National Research Council of Canada*

The mechanical behavior of a one-part, chemically-curing, silicone sealant is defined by its primary variables: stress; strain; time; and temperature; using a model specimen. The material proved to be temperature independent in the region important for sealants, with some deviation from the rule near the crystallization temperature under high strain. The remaining three variables describe behavior in a three-dimensional co-ordinate system. Projection of the break points in the logarithm of strain vs logarithm of time plane is the link between laboratory testing and outdoor performance. By extrapolating the regression line of the break points to six months, extensibility of the material can be predicted. A testing procedure suitable for standards is proposed that links laboratory testing with outdoor performance.

KEY WORDS: Sealants; Building sealants; Silicone sealants; Mechanical properties of sealants; Rate of movement dependence of mechanical properties of sealants; Temperature dependence of mechanical properties of sealants; Performance testing of sealants.

INTRODUCTION

THIS is the third in a series of papers reporting the results of an investigation of the mechanical properties of sealants designed to provide a test method for predicting outdoor performance. Many variables govern the mechanical behavior of sealants[1,2] and the problem is complex. Because of the large number of variables, some must, as a first step, be kept constant; the most basic—stress, strain, time and temperature—are used here to define the properties. These variables are designated as basic because they are necessary in describing a sealant at any state defined by other variables.

The tensile test is used as a test method mainly because it simulates the loading that is, in practice, the most likely cause of failure. In order that the results can be related to practical situations, the test specimen is a model representation of the sealant bead used in butt joints of buildings. It is described in detail under "Experimental."

It has been illustrated[1] that a large number of different tensile curves can be obtained for silicone sealants at constant strain rate by varying the test temperature. Similarly, different tensile curves are obtained when the rate of extension is varied and the temperature is kept constant.[2] Some of these tensile curves correspond to the rates of joint movement in buildings and therefore represent practical situations. To conduct a test of this type to the break point is time-consuming and not suitable as a testing procedure. Tensile tests are economical at rates of extension several decades higher than the highest rate normally occurring on an expansion joint.

As it is the aim of this paper to establish a unified system of properties by which tests performed at ordinary test rates and temperatures can be used to predict tensile behavior at outdoor temperatures and outdoor rates of joint movement, theoretical background information has to be applied to the test results to achieve this goal. Theoretical considerations are known, however, to be easily applicable only to uniaxial deformation, and this the model sample representing practical conditions does not have. In fact, the sample has an extremely complicated stress field.[3] In spite of this, theoretical information has been adapted to the model sample with success within acceptable errors.[2] In the present paper, work is further pursued to define how a laboratory test method can be correlated with outdoor sealant activity.

EXPERIMENTAL

The proper choice of silicone sealant sample and substrate has been investigated.[1] As a conclusion of that study, only one brand of one part white silicone

* Building Materials Section, Div. of Building Research, Ottawa, Canada, K1A 0R6.

sealant of the chemically curing type is required for the present investigation, and one type of substrate, aluminum, is sufficient. The specimens have the same dimensions as those used previously: ½ × ½ × 2 in. (1.3 × 1.3 × 5.1 cm) sealant bead on ½ × ½ × 3¼ in. (1.3 × 1.3 × 8.1 cm) square aluminum tubing (*Figure* 1).

In a previous work[2] the sealant was cured before testing for three to four weeks at room conditions. This curing was sufficient to demonstrate that the tensile curves obtained with the model specimens at various rates of extension are superimposed on a single cumulative curve at a given temperature. At the same time, the relatively short curing period kept down the time involved in the experiment. Because only a small fraction of the lifetime of sealants is spent in the initial cured state, in this more intensive investigation the samples were heat aged, the fastest available approach to obtaining aged samples. The specimens were cast at 72 F (22.2 C) and 50% relative humidity on primed aluminum. They were cured for two weeks at the same conditions, then subjected to accelerated aging at 158 ± 2 F (70 C ± 1 C) in a ventilated oven for a month.

In order to establish a single master curve that would include tests done at different rates and temperatures, as proposed in the present work, the number of test temperatures was extended to seven from the previously used three: 100 F (37.8 C), 72 F, 20 F (−6.7 C), 0 F (−17.8 C), −10 F (−23.3 C), −20 F (−28.9 C), −40 F (−40.0 C). Temperature could be maintained within ± 1 F (± 0.6 C) accuracy in the test cabinet and within ± 2 F in the room when room temperature testing was required.

An Instron testing machine was used for tensile testing at the following rates of cross-head travel:

cm/min	*In./min*	*% Extension/ min*
5.000	1.969	393.7
1.000	0.394	78.7
0.5000	0.1969	39.37
0.1000	0.0394	7.87
0.05000	0.01969	3.937
0.01000	0.00394	0.787
0.005000	0.001969	0.394
0.001000	0.000394	0.079
0.0005000	0.0001969	0.039
0.0002000	0.0000787	0.016
0.00005000	0.00001969	0.004

Some of the lower rates of extension were of about the same order as the highest rate of movement estimated and measured on expansion joints: 0.05%/min.[2] The substrate was gripped along its full length by jaws having serrated and parallel surfaces during the tensile test. A duPont 900 Differential Thermal Analyzer was used to find the crystallization temperature of the sealant. Calculations and plottings were carried out with the aid of a computer.

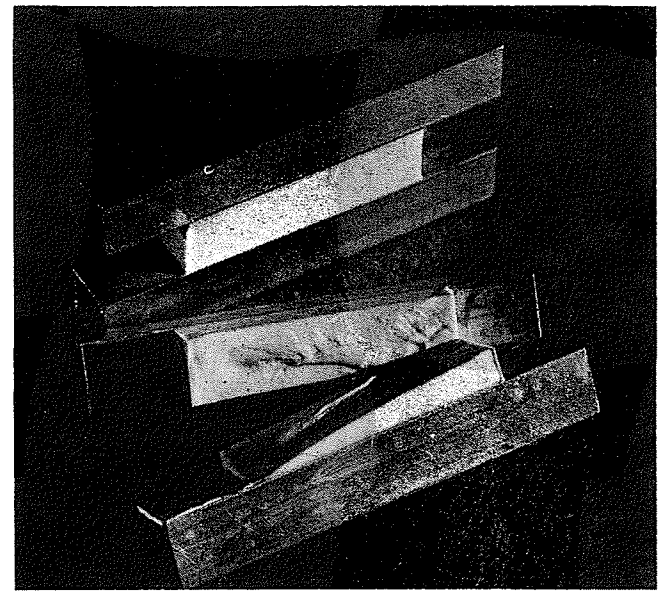

Figure 1—Test specimen before and after test

DISCUSSION

Smith[4] and authors quoted by him pointed out that the following equation can be derived for stress, S:

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

where R is strain rate, γ is strain, τ is the relaxation time, $M(\tau)$ is the relaxation distribution function, and $M(\tau)\ d \ln \tau$ is the contribution of those elastic mechanisms to the instantaneous tensile modulus that have their relaxation times between $\ln \tau$ and $\ln \tau + d \ln \tau$. As S/R is a function only of γ/R, the tensile curves obtained at different strain rates are superimposed on a single cumulative curve on a plot of log S/R vs log γ/R. Smith found, empirically, that a correction for true stress has to be applied; multiplying the stress by λ, the extension ratio, will achieve the expected superposition. The single curves obtained at each temperature can be shifted along the time axis, following the WLF equation,[5] after multiplying by T_s/T; T_s/T reduces the data to the reference temperature, T_s, from the test temperature, T, as the theory of rubber-like elasticity requires it. Single master curves were successfully obtained with different polymers.[4,6]

The above treatment was applied to the tensile curves of the artificially aged silicone specimens. As previously observed with fresh samples, the tensile curves of aged specimens could also be superimposed on a single straight line at each temperature. Three of the cumulative curves are illustrated in *Figures* 2 to 4, representing the results obtained at the lowest temperature, at room temperature, and at the highest one, respectively. The cumulative curves derived at other temperatures give similar plots.

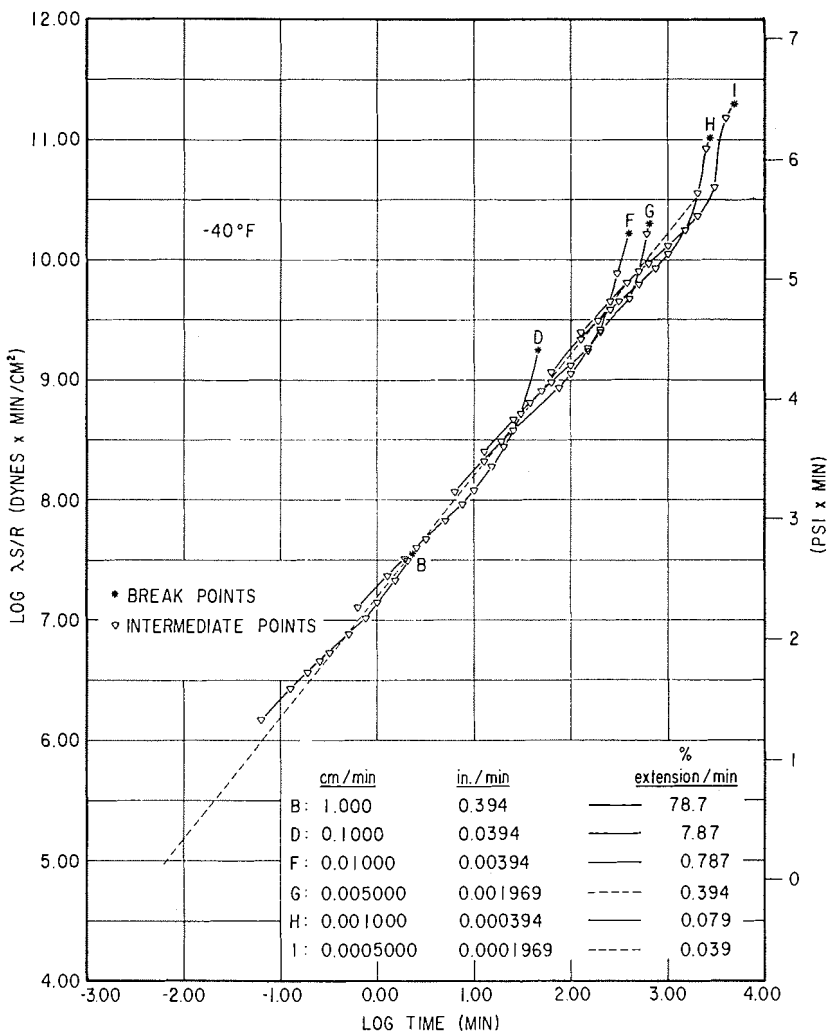

Figure 2—Tensile curves reduced by the corresponding rate; at −40 F

Statistical analysis of the data, in particular of the slopes and intercepts, reveals that the cumulative curves obtained at different temperatures are identical within experimental errors. *Table* 1(a) presents the detailed statistical analysis. With the exception of the curve traced at −40 F the slopes of all curves are the same, the values overlapping within the 95% confidence limits. The value of the slope at −40 F is slightly higher than those for other temperatures. One may observe in *Figure* 2 that the individual curves, marked D to I, (at 0.1 cm/min and lower rates) break away from the cumulative curve to higher levels on approaching their break points. The deviation of the single curves from the cumulative curve is the result of toughening of the material, better visualized by looking at *Figure* 5, which represents the original stress-strain curves at −40 F and 72 F. It may be seen (the scales being identical) that much higher stress and strain values are reached at failure at −40 F than at 72 F. There is a definite change in material properties and these deviating parts of the tensile curves should not be included in the data of a master curve.

The standard error obtained when analyzing the −40 F data in steps of different percentage extension readings varied as shown in *Table* 2. The standard error increases with the level of extension. At 160% it exceeds the highest standard error found at other than −40 F (see *Table* 1(a) where at a given temperature all data are included in each analysis). One may conclude that readings over 120% at −40 F represent material properties considerably different from the general behavior demonstrated at all other conditions. These readings should therefore be excluded from the master curve.

It is reasonable to suppose that at −40 F the material properties change as a result of crystallization under the effect of high stresses that increase the alignment of the molecular chains. Direct evidence of crystallization occurring in the polymer under the effect of high strain was not sought, it is a well-known phenomenon[7,8] and it was not the purpose of this paper to go into this detail. The temperature where crystallization occurs however, was checked by differential thermal analysis (*Figure* 6). The instrument

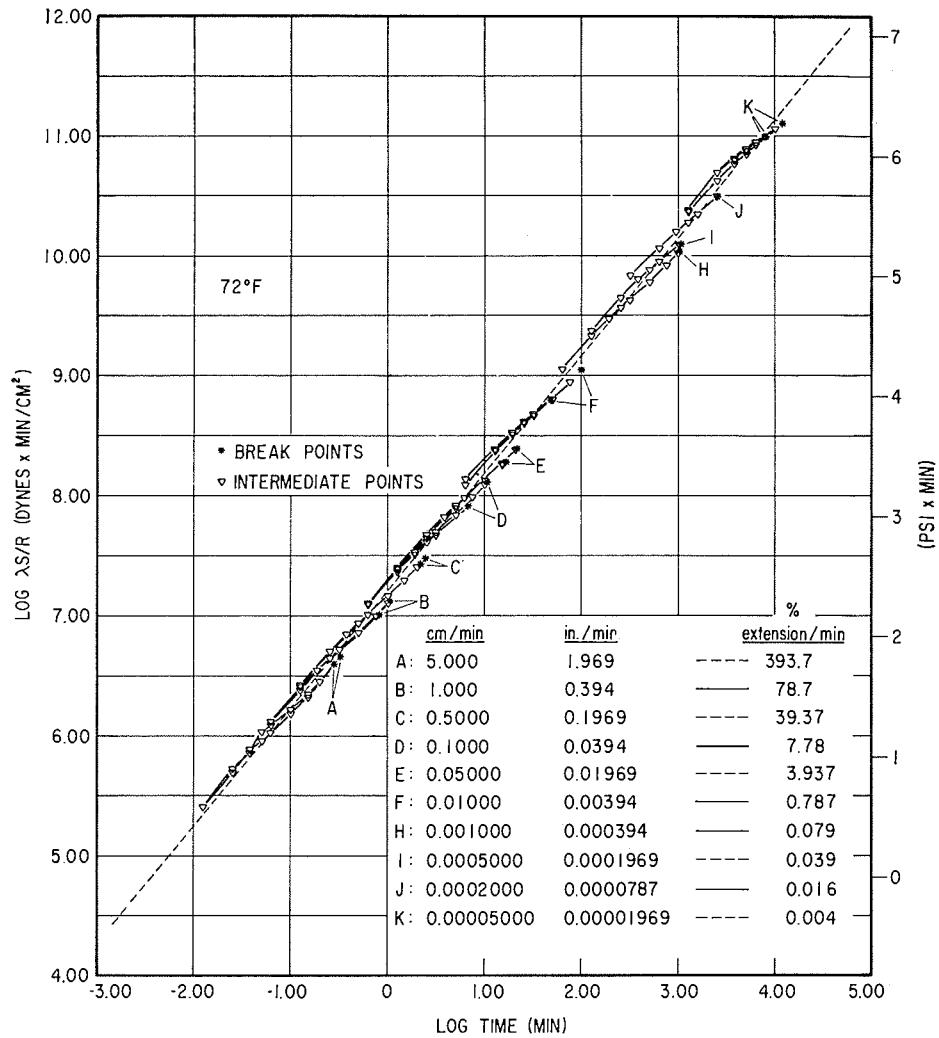

Figure 3—Tensile curves reduced by the corresponding rate; at 72 F

Table 1(a)—Statistics of log λS/R vs log γ/R

Test Temperature	n	r	a	ta	b	tb	s_e
100 F	51	0.997	7.20	±0.02	0.95	±0.02	0.064
72 F	142	0.999	7.20	±0.01	0.98	±0.01	0.075
20 F	55	0.997	7.22	±0.02	0.95	±0.02	0.064
0 F	65	0.998	7.22	±0.02	0.96	±0.02	0.066
—10 F	67	0.998	7.23	±0.02	0.96	±0.01	0.058
—20 F	54	0.998	7.23	±0.03	0.95	±0.02	0.071
—40 F	70	0.993	7.20	±0.06	1.01	±0.03	0.147

n — Number of readings
r — Correlation coefficient
a — Intercept
ta — Limits for the intercept at the 95 per cent confidence level
b — Slope
tb — Limits for the slope at the 95 per cent confidence level
s_e — Standard error

Table 1(b)—Regression Line, Up to and Including 120% Extension at —40 F

—40 F	51	0.998	7.22	±0.03	0.96	±0.02	0.071

Table 1(c)—Master Curve

All temperatures	485	0.998	7.21	±0.01	0.97	±0.01	0.070

(Excludes readings at 160% and at higher extensions at —40 F)

provides a constant rate of temperature change only while the material is being heated, but the onset of melting of crystallized phase of the polymer is not very well defined in heating mode alone. If the cooling curve also is obtained, although the rate of cooling is not so closely controlled, the crystallization temperature becomes quite well defined at —54 C (—65.2 F) by comparison of the curves.

As DTA is done on the unstrained sample, it may be seen that the crystallization temperature is much lower in the unstrained state than in the strained state, as is the case for tensile curves. The slower the rate of extension the lower the extension and the lower the stress at which the material starts to crystallize, as can be deduced from the placement of the inflexion points of the curves in *Figure* 5. At higher rates, failure occurs before crystallization can start (curve B). The dependence of crystallization on time may be seen directly from *Figure* 2 where the curves for decreasing rates of extension break away from the cumulative curve after increasing lengths of time have elapsed for the tensile tests.

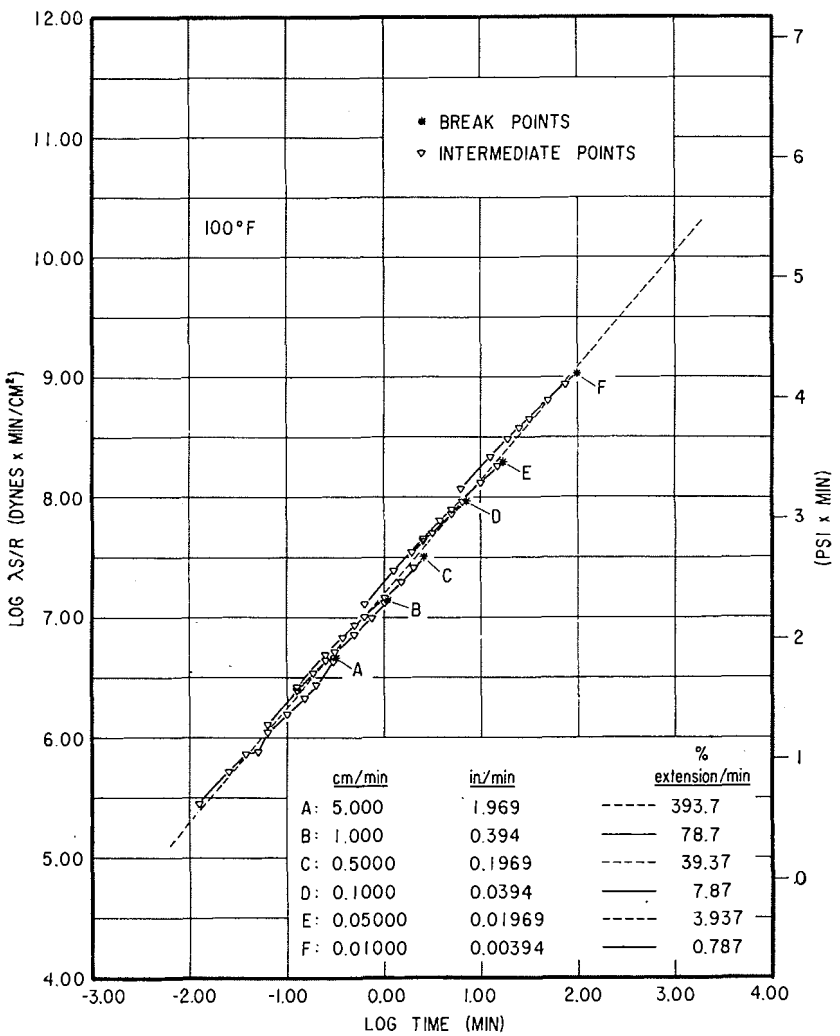

Figure 4—Tensile curves reduced by the corresponding rate; at 100 F

Differential thermal analysis revealed as well that artificial aging does not affect the crystallization temperature of the polymer. This can best be observed by comparing the position of peaks of *Figure* 5 (sample weights being identical) occurring while the material is subjected to a uniform heating rate of 10 C/min (18 F/min). There is no detectable change in the position of the peak or of the onset of melting due to artificial aging.

It should be noted that although only extension readings of 160% and higher were excluded from the final master curve, crystallization may occur at lower extensions but without marked effect.

A further examination of the data given in *Tables* 1(a) and 1(b) indicates that the intercept, a, is identical for the seven curves obtained at different temperatures because ta, the t test for the intercept at the 95% confidence level, gives values overlapping at all temperatures.

With the slope and the intercept constant within the 95% confidence level, one may consider that all readings belong to the same population (excluding readings at —40 F at and above 160% extension). This means that the material is temperature independent within the given errors in the temperature range observed and that all the stress-strain curves obtained at different temperatures can be treated together. A single curve, the master curve, can be derived for the properties of the silicone sealant. The results of the statistics of the master curve are summarized in *Table* 1(c).

Comparison of the cumulative curves obtained at a given temperature with the artificially aged samples used in this work and the fresh samples used in the previous work[2] reveals the effect of aging on mechanical properties. Comparisons can be made at room temperature and at —10 F because these test temperatures were used in both works. At each temperature the cumulative curves for aged and fresh samples are the same except for the intercept, which is higher for aged samples. The aging process hardens the material and raises the tensile stress 23% at room temperature and 28% at —10 F, calculated on a nonlogarithmic scale.

Time Dependence of Stress

Because the variable, temperature, proved to be a constant for the material in the temperature range important for sealants, the number of variables necessary to define the silicone sealant at any degree of aging is reduced to three: stress, strain, and time. They can be illustrated in a three-dimensional co-ordinate system that can be derived from plottings of log λS vs log γ/R ($=$ log t). These plots would represent the logarithm of true stress vs the logarithm of time for samples having uniaxial elongation. Although the model specimens have a complicated stress field, and one cannot talk about true stress, the above representation will prove useful. *Figures* 7 to 9 give this type of plot for the tests made at —40 F, 72 F, and 100 F, respectively. The individual stress-strain curves obtained at various strain rates are indicated by dotted lines. The continuous lines are the best fitting lines connecting readings obtained at constant (arbitrarily chosen percentage) extension levels of tensile curves traced at various rates of extension. The slopes of the curves are negative, indicating that the stress necessary to produce a certain elongation decreases with time. This means that the material properties are time dependent, i.e., some amount of stress relaxation occurs while the sealant is being stretched.

An analysis of the individual curves at different extension levels and temperatures illustrates that the slopes are identical up to and including 120% extension, but that errors are rather large at the 95% confidence level in comparison with the small value obtained for the slopes (tables omitted). The error could be reduced by increasing the number of readings. This can be done without increasing the number of tests because it has been found that the material is temperature independent and readings taken at different temperatures but at identical extension levels can be analyzed together. The slope of the combined data was —0.012 at 5% extension, progressively increasing to —0.023 at 80%, with not more than ±0.007 error at the 95% confidence level. These values illustrate that up to the 80% extension level the slopes are identical.

At the 120% extension level the slope is still in the same order as at lower levels, but the scatter increases;

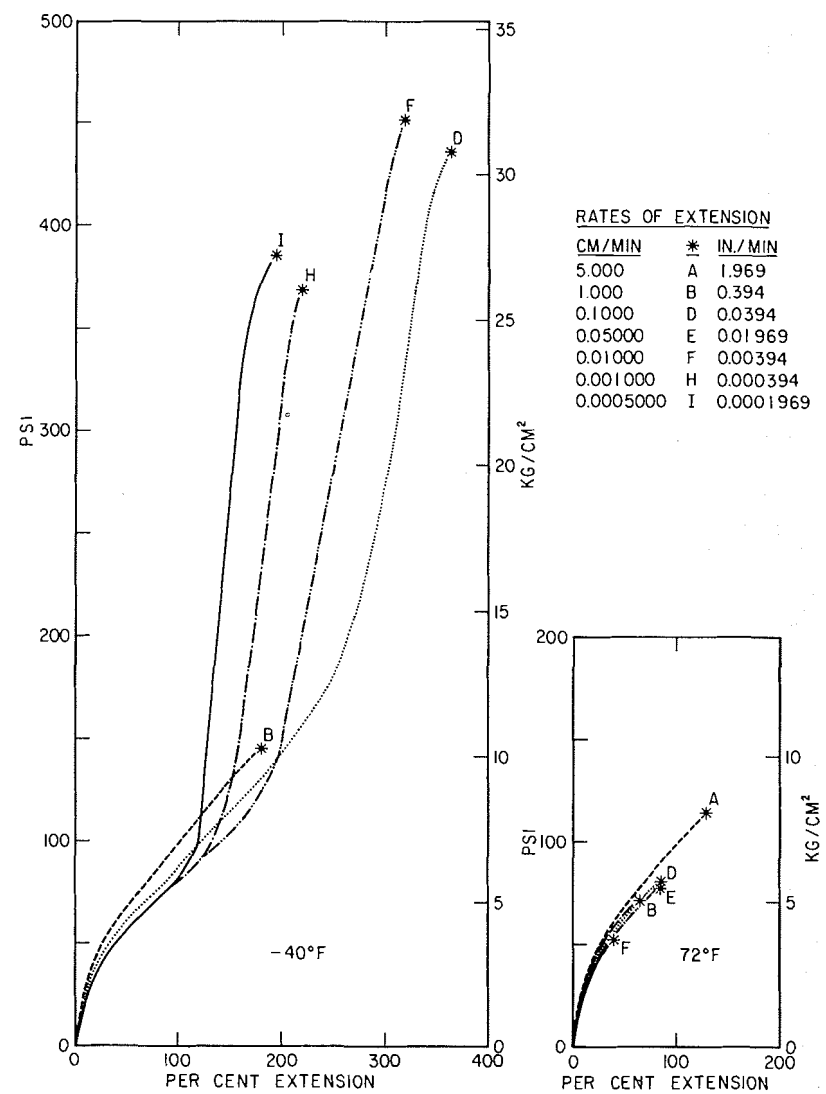

Figure 5—Tensile curves at —40 F and at 72 F at various rates of extension

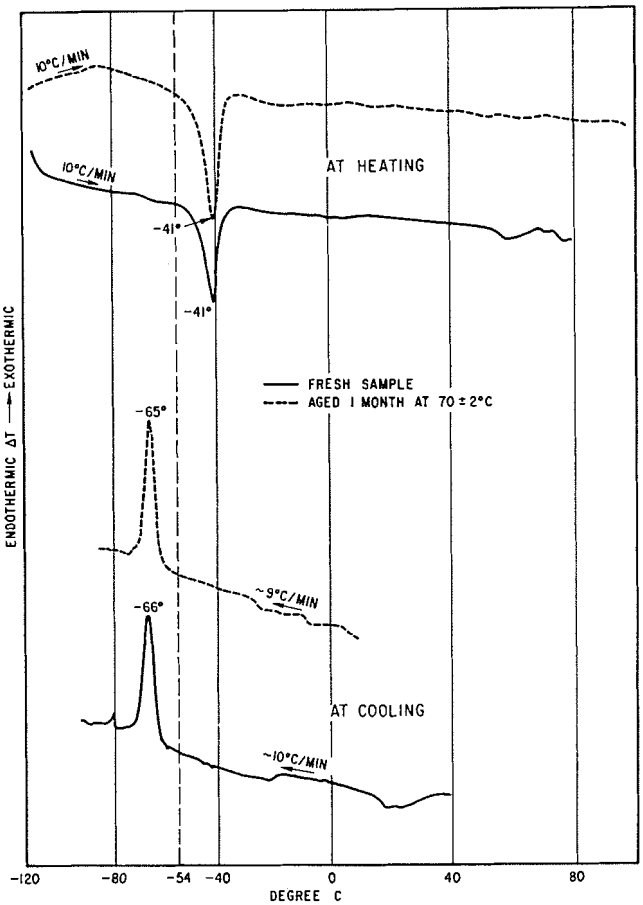

Figure 6—Differential thermal analysis of one-part silicone sealant

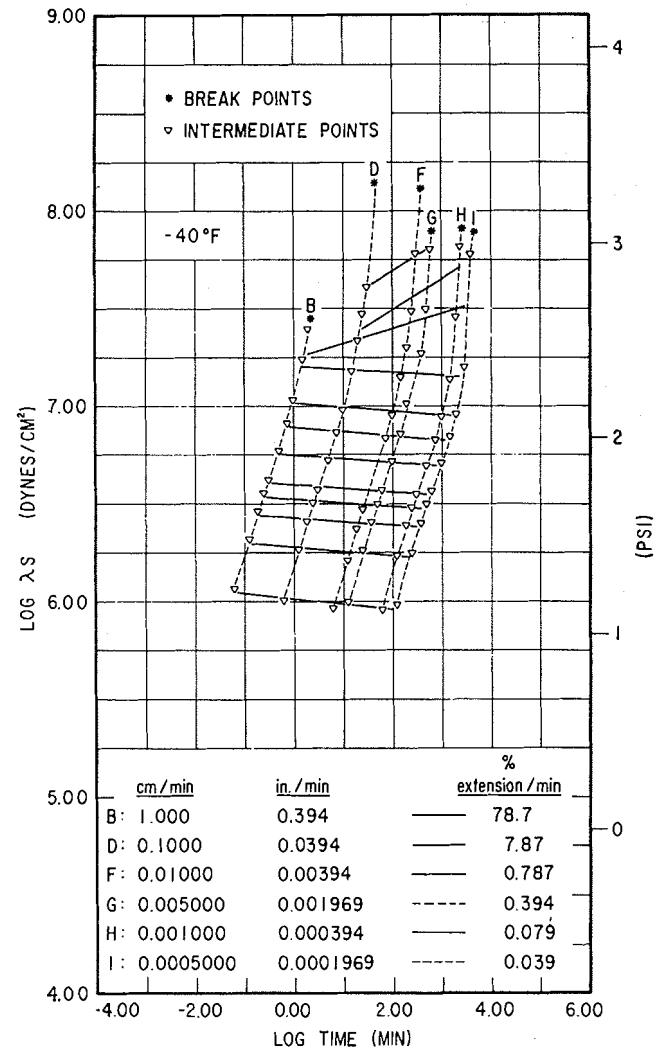

Figure 7—Time dependence of "true" stress; at −40 F

from 160% extension on and at −40 F (*Figure* 7) the slope changes to positive values as a result of the appearance of pronounced crystallization. At temperatures above −40 F failure occurs, with few exceptions, at lower than 160% elongation.

The break points of *Figures* 7 to 9 are scattered in rather broad bands and the regression lines fitted to the break points at each temperature have a much larger negative slope than the various extension levels. The bands shift to slightly higher stress values with increasing temperatures. The scatter looks essentially the same as that in *Figures* 8 and 9 for all temperatures and occurs as a result of the variation in time necessary to reach the break. There is an exception among the tests done at −40 F where, as illustrated in *Figure* 7, not all break points belong to the same band. A sudden drop occurs in both stress and strain when the strain rate is increased to 1 cm/min. As the other break points at −40 F follow the general pattern, this particular test has been repeated twice. The three break readings had a spread of ±18% error on a linear scale (in the direction of the y axis), whereas the other readings at −40 F were 300% different, based on averages. The good reproducibility of the 1 cm/min test does not leave much doubt that the sudden drop in break load at the increase of strain rate is a result of an inherent material property.

The explanation can be found in the type of failure. All samples tested at higher than −40 F failed cohesively. *Figure* 1 illustrates a typical failure. Samples tested at −40 F failed adhesively except at the highest rate of extension, 1 cm/min, where failure was cohesive. The probable explanation is that at the highest rate the crystallites do not have time to develop to the same extent as at lower rates. In the absence of the reinforcing effect of crystallites cohesive failure occurs. The adhesive strength is weakened by stresses built up in the interface owing to the difference developing in the linear coefficient of thermal expansion of the substrate and in the sealant with decreasing temperature.

Three-Dimensional Co-ordinate System

Based on the present findings, the mechanical properties of silicone sealants can be characterized in a three-dimensional co-ordinate system. Readings

should be taken at a given time at each extension level from plots of the type shown in *Figures* 7 to 9 (or combined ones). The logarithm of stress vs logarithm of strain should be plotted at each time chosen and the plots spaced along the third axis, log t, according to the time intervals used for the readings. Owing to the difficulty of visualizing a three-dimensional co-ordinate system, projections of the break points in the three different planes are used since one endeavors, ultimately, to define the failure conditions.

One of the projections is in the plane of logarithm of "true" stress (log λS) vs logarithm of "true" strain (log ϵ) and is illustrated in *Figure* 10. Quotation marks are used because the model samples cannot be characterized by true stress or true strain owing to the complicated stress field, but the values have been treated as if the ideal conditions had been realized. *Figure* 10 is the so-called failure envelope[9] consisting of a band within which the failure points of all tests occur. (The continuous lines are drawn in arbitrarily.) The scatter is not very great considering the type of

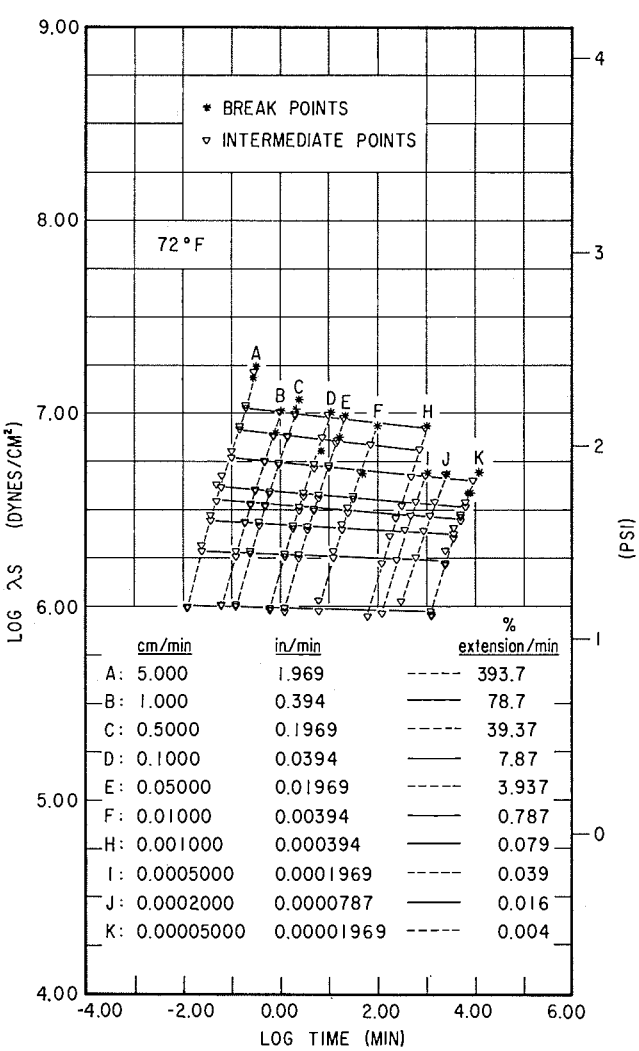

Figure 8—Time dependence of "true" stress; at 72 F

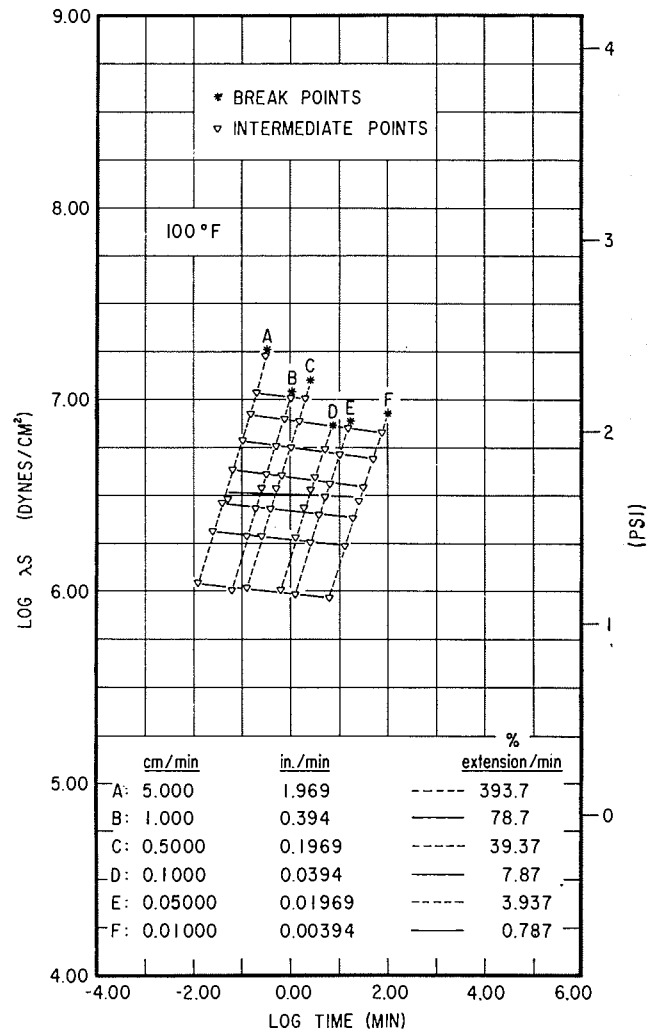

Figure 9—Time dependence of "true" stress; at 100 F

samples used for testing and compared with results that can be obtained on polymer samples having uniaxial elongation, as data published in the literature indicate.

The same failure envelope could be established through stress relaxation or creep tests. In the former case the sample has to be stretched to a constant elongation to a point such as example A in *Figure* 10. Strain is maintained while stress decays as a function of time, following line A-B. Failure occurs when the stress reaches the failure envelope. In a creep test in which the stress is constant, the strain increases, following arrow A-C, causing failure when the failure envelope is reached. If the strain of a relaxation experiment or the stress of a creep test were very small, failure might never be reached, but an equilibrium situation might be, where neither stress nor strain would change.[10]

The second projection of the three-dimensional system could be in the plane of logarithm of "true" stress (log λS) vs logarithm of time (log t) necessary to reach failure. The projection in this plane is not

Table 2—Variation of the Standard Error With the Level Of Extension

(Tests at −40 F)

Readings Up To and Including: % Extension	Number of Readings	Standard Error, s_e
5	6	0.024
10	12	0.029
15	18	0.032
20	24	0.038
25	29	0.042
40	34	0.054
60	40	0.067
80	46	0.071
120	51	0.071
160	57	0.095
200	61	0.104
240	64	0.110
All data	70	0.147

illustrated because stress is of secondary importance for sealants. It develops as a consequence of the strain imposed on the material.

Strain at Break vs Time

It is the strain at break that is the most important characteristic of a sealant. A projection of the break points of the three-dimensional co-ordinate system into the plane of logarithm of strain ($\log \gamma$) vs logarithm of time ($\log t$) necessary to reach failure is illustrated in *Figure* 11. At first glance the data seem to be difficult to interpret, but closer examination reveals useful conclusions. At each test temperature the break points are arranged in a broad band sloping toward lower extension values as time increases. *Table* 3 summarizes the results of the linear regression analysis for the break points at each temperature. The bands are characterized by slope, b, and the intercept, a, with their respective 95% confidence levels. Although the confidence limits are rather wide, the slopes at different temperatures are approximately the same. The intercepts, however, shift to progressively higher strain levels as temperature decreases. The results at −20 F represent an exception to this trend, but they can be disregarded because only the six tests at −20 F oppose it (the trend is based on 49 tests). It is probable that an increase in the number of tests at −20 F would eliminate the anomaly.

Table 3—Linear Regression Analysis of Break Points Obtained at Various Temperatures and Rates of Extension

($\log \gamma$ vs $\log t$)

	n	r	a	ta	b	tb	s_e
100 F	6	−0.665	−0.02	±0.15	−0.09	±0.15	0.104
72 F	18	−0.779	−0.04	±0.09	−0.09	±0.04	0.122
20 F	6	−0.762	0.17	±0.19	−0.11	±0.12	0.082
0 F	7	−0.803	0.23	±0.18	−0.12	±0.10	0.092
−10 F	7	−0.707	0.29	±0.22	−0.11	±0.12	0.116
−20 F	6	−0.788	0.09	±0.11	−0.05	±0.05	0.048
−40 F	5	−0.965	0.81	±0.20	−0.14	±0.07	0.034

This projection of the failure points in the logarithmic strain vs logarithmic time plane is the basis on which performance testing can be built. The band of failure can be extrapolated to the region of six months, the maximum amplitude of the cyclic joint movement in the direction of extension.

Shifting of the failure bands to higher extension values means that as temperature decreases the material is capable of maintaining higher extensions. To simplify a testing procedure, therefore, it is sufficient to test the silicone sealant at the highest temperature. The extra extensibility at lower temperatures can be considered an added safety factor. A further simplification of the test procedure would be to use room temperature for testing. This can be done for two reasons: first, because above room temperature the sealant will probably undergo compression, not extension; second, because the band of break points at 100 F and at 72 F shows ample overlapping.

To decrease the width of the failure band at 72 F, i.e. to increase the accuracy of the prediction, the number of tests was tripled in relation to other test temperatures and the range of rate of strain was extended. *Figure* 12 illustrates the results. The continuous line represents the best fitting line: the dashed lines represent the 95% confidence levels; the dotted ones, the 90% levels.

As *Figure* 12 illustrates, readings extend through nearly five time decades. Although an extrapolation to six months may not be strictly valid, the six-month reading on the plot is less than 1.5 time decades away from the region of testing. Thus, the extrapolation is reasonably small.

The extrapolated part of the straight line is inaccessible by stress and strain experiments for practical reasons and should be verified in relaxation experiments, putting the sealant samples on appropriate spacers and observing the time to failure. Although very simple, this is a time-consuming experiment and has not yet been attempted.

With the help of the linear time and extension scales one can read at six months the extensibility of the sealant specimen on the regression line and at the chosen confidence levels. The regression line indicates an extensibility of 28%. At the 95% limit the range is from 14 to 57%. The upper and lower 90% confidence limits are 51 and 16%, respectively. The 14% extensibility at the 95% limit means that 2.5% of sealant specimens may fail at lower than 14% extension within six months. Similarly, for the 90% limit there is a 5% chance that failure will occur at lower than 16% extension.

Standards and manufacturers generally recommend that this type of sealant should not be used in joints moving more than ±25%. At 25% extension the regression line indicates failure at just under two years. The lower confidence limit is reached, however, in about a day at the 95% level and in about two and a half days at the 90% level.

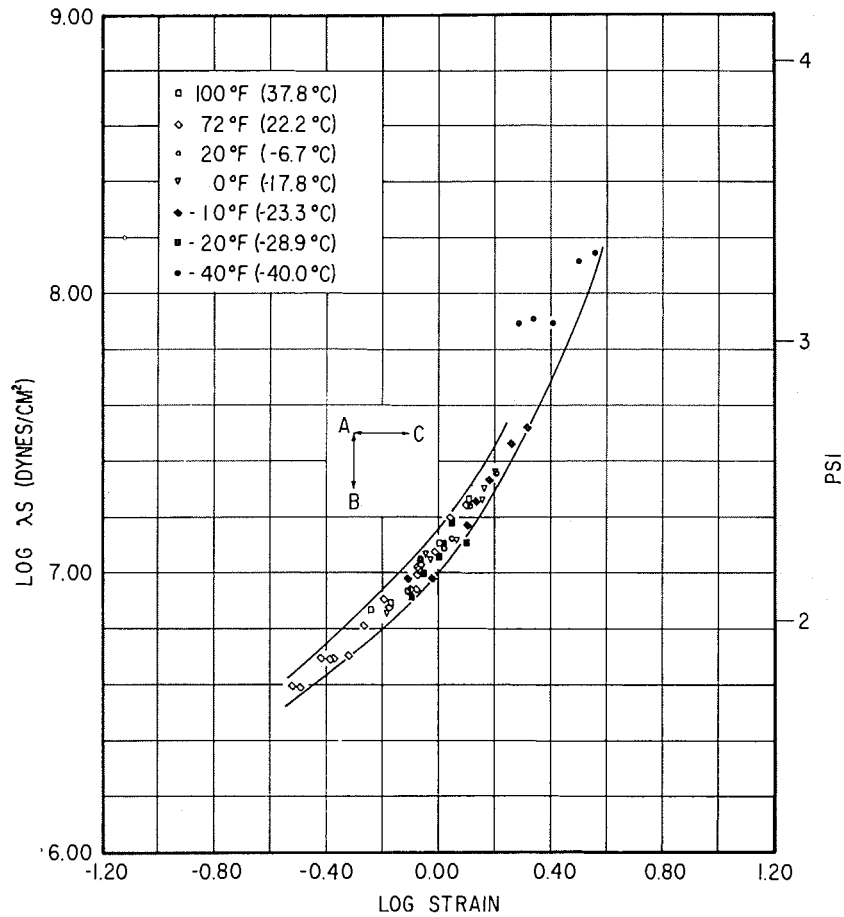

Figure 10—Stress vs strain at break; all temperatures

The above values include a safety factor because extensibility increases as temperature decreases. Calculating the extensibility at different temperatures, according to the best fitting lines given in *Table* 3, gives the following values at six months:

72 F — 28% extensibility
20 F — 40% extensibility
0 F — 40% extensibility
—10 F — 50% extensibility
—20 F — 65% extensibility
—40 F — 120% extensibility

If one calculates the extension at the lower 90% confidence limit at —20 F, one finds 43%. At —40 F this increases to 81%. The lowest winter temperature in most regions of Canada is between these temperatures, around —30 F, with the lower confidence limit between 43% and 81% extension. The latter, therefore, will probably be higher than the extension at the upper 90% confidence limit obtained from the room temperature data: 51%. Consequently, instead of giving confidence limits, the characterization of the sealant could be done in a more simple manner by giving a single figure, the extensibility defined by the regression line at six months from the 72 F data. This is 28% for the silicone sealant tested. This characteristic number includes an ample safety factor.

It has to be noted that the test method and the derived single-characteristic extension figure relate to the model specimen of ½ × ½ in. (1.3 × 1.3 cm) square intersection chosen to conform to standards. It is usually recommended for this material, however, that the depth be half the width and that unattached surfaces be concave, a change from the model specimen that should allow much greater extensibility. This aspect, together with the specimen length and the effect of the shape of the ends of failure requires further study, using the method outlined above as the link between laboratory testing and practical performance.

CONCLUSION

A method has been outlined of predicting the failure of silicone sealants under practical conditions from the results of laboratory tensile testing. Prediction is possible because background information can be applied successfully to the model specimens. The stress-strain curves reduced by the strain rate give a single cumulative curve at each temperature for tests made at various strain rates. The resulting curves are identical for the silicone sealant studied, i.e. the material is independent of temperature within the range observed except where crystallization interferes. The remaining variables studied, stress, strain, and time, can represent the mechanical properties of the sealant in a three-dimensional co-ordinate system. From the point of view of sealant performance the most important aspect of the three-dimensional representation is the projection of break points in the logarithmic strain vs logarithmic time plane. This projection is the

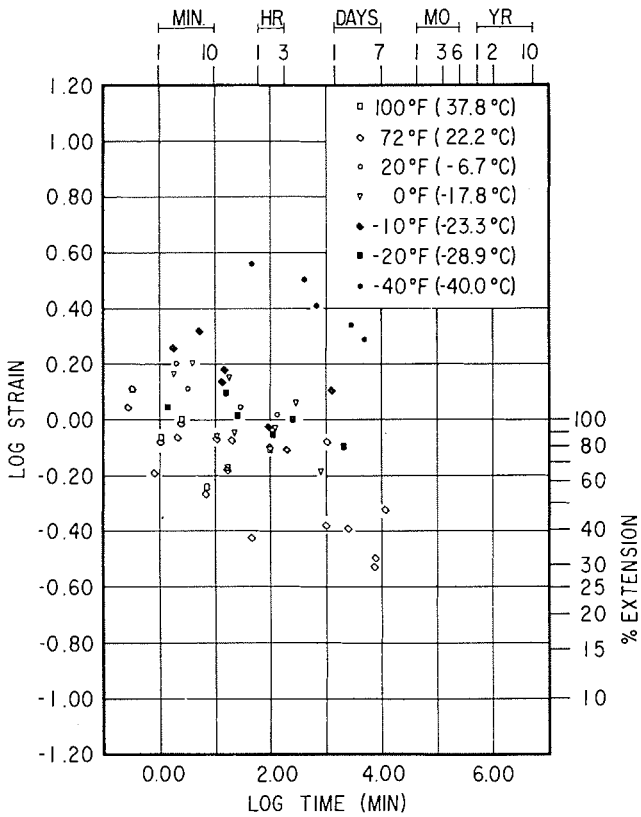

Figure 11—Time dependence of strain at break; all temperatures

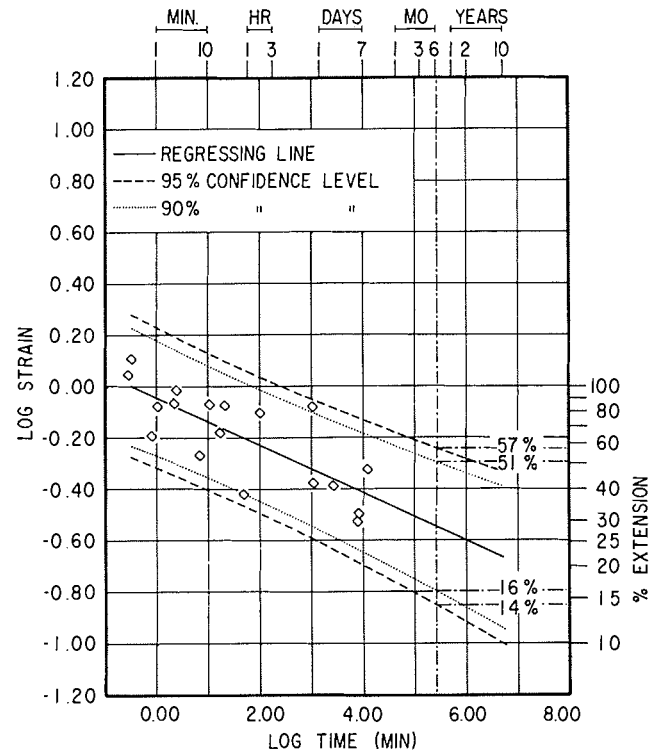

Figure 12—Time dependence of strain at break; at 72 F

key to performance testing because it includes testing times to break and time to reach failure (the latter by extrapolation). The failure points obtained from tests are scattered along a straight line with rather wide confidence limits, as is the nature of break tests. The tensile testing rates extended from about 2 cm/min (0.8 in./min) to 0.00002 cm/min (0.000008 in./min), covering five time decades with the broad band of scattered points sloping toward longer times. If the failure band is extrapolated a further 1.5 cycle, one reaches six months, the time spent in tension by the sealant during a yearly cycle. The extensibility at six months is a characteristic figure that can serve the architect in designing a joint. The results indicate, as well, that failure is improbable in a daily movement.

The shape of the specimen tested was kept constant to conform to specifications. It is, however, not the shape recommended by the sealant manufacturers for practical applications. This latter shape tends to allow more extension without failure. The shape of the specimen is a variable that needs further examination as does the choice of the heat-aging process.

Based on the present state of available information about silicone sealant behavior, it is suggested that standard testing of silicone should be done at room temperature on heat-aged samples covering as wide a range of rate of extension in tensile testing as possible (at least the range covered in this work). A minimum number of 15 tests would be necessary to keep accuracy at an acceptable level. Until the question of the shape of the specimen is clarified by further work, the model specimen should be retained. The logarithm of strain at break vs the logarithm of time to break should be statistically analyzed and the extensibility determined for the six-month period by extrapolation. The latter figure should be considered to be a characteristic of the material on which its acceptance should be based. The fact that the sealant can be extended more with decreasing temperatures should be regarded as a safety factor. The crystallization temperature is another characteristic that should be given for identification of the material.

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