PREDICTING LONG-TERM PERFORMANCE OF MATERIALS/SYSTEMS

A Role for Accelerated Creep Tests?

BY J.S. THORNTON, PH.D.

Background

There is valid skepticism about the value of some laboratory test results in assessing the performance of roofing components. In a discussion of destructive testing of single ply roof membranes, it has been pointed out that "there are no universally accepted values (for shear and peel strength) which signal an inferior lap.[1] Research and testing elsewhere has shown that bonded seams of EPDM rubber which had been prepared "with moisture, contaminated surfaces, and adhesive voids demonstrated shear strength values comparable to control specimens."[2] These observations are examples that initial strength of a bonded joint is not a reliable indicator of its service life. Yet another example is shown in *Figure 1*. In evaluating candidates for a certain moisture exposure application, the initial strengths of polyurethane rubber-to-metal bonded joints far exceeded those constructed with Neoprene rubber. However, in boiling water exposure tests, the Neoprene joints outlasted the polyurethane joints.

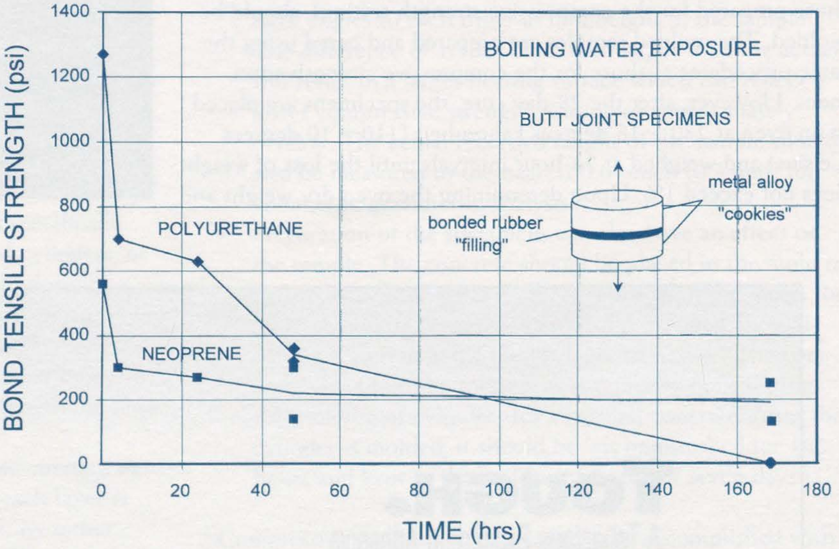


Figure 1. Effect of time in boiling water on the strength of rubber to metal bonds.

Some have referred to this test as an accelerated *death* test rather than an accelerated *life* test, but subsequent tests under more realistic (but still aggressive) moisture exposure conditions told the same story. How materials perform over the long term is what's important, not necessarily their properties when new as measured by index tests. After an introductory discussion about temperature effects on materials properties, a breakthrough testing protocol that would allow the roofing applications engineer to evaluate materials' long-term behavior in short-term laboratory performance-based tests will be described.

Effect of Temperature on Creep-Rupture

Rossiter and his coworkers at NIST have done extensive investigations of EPDM membrane seams for roofing applications. In a series of reports, this research team presented findings on the effects of adhesive joint thickness, type of joint (liquid adhesive or tape) contamination, cold temperature preparation, various environmental exposures prior to loading, and test temperature on the strength and creep-rupture properties of shear and peel specimens. Of particular interest are the results of the effects of temperature on creep-rupture properties.[3] *Figure* 2 shows one of Rossiter's results for creep-rupture of EPDM peel specimens bonded with "Tape System 1" and tested at 23°, 40°, and 70° Celsius. Note that at higher temperatures and higher loads, the time for the peel joint to rupture is greatly reduced. The time scale is logarithmic, so that the range of times represented by the data (5 minutes to 100 hours), spans three decades!

The influence of temperature on the time scale is quite spectacular. This, of course, is not a novel observation. Scientists and engineers have recognized the influence of temperature on rate processes in materials for at least 100 years. A body of knowledge has accumulated on the subject of time-temperature equivalence. It is an experimentally-observed fact that increasing temperature can reduce, predictably, the time for a rate process (such as creep or creep-rupture) to occur.

Applied to *Figure* 2, a procedure called time-temperature superposition (TTS) is a way to produce a single "master curve" of creep rupture behavior that is expressed at convenient "reference temperature," using "shift factors." Then, with the knowledge and availability of the master curve and the shift factors, curves at intermediate temperatures can be generated easily. Most generally, TTS is utilized to make long-term performance predictions based on short-term, elevated temperature data. *Figure* 3 presents the results of TTS applied to the data of *Figure* 2. The 70° data was shifted 2.08 log decades and the 40° data is shifted 0.95 log decades to achieve the master curve at a 23° reference temperature. The vertical logarithmic shifts at 70°

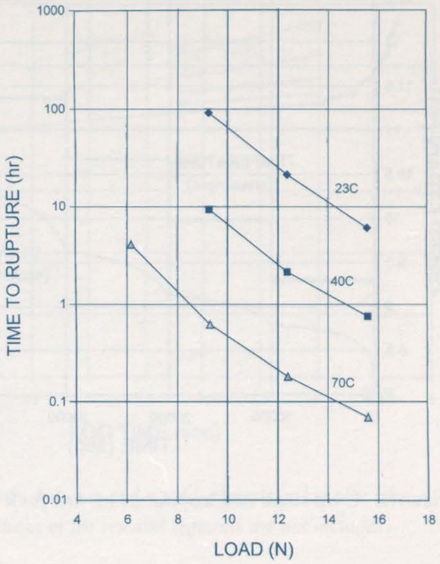


Figure 2. Effect of temperature on creep-rupture properties of Tape System 1 bonded EPDM T-peel specimens.

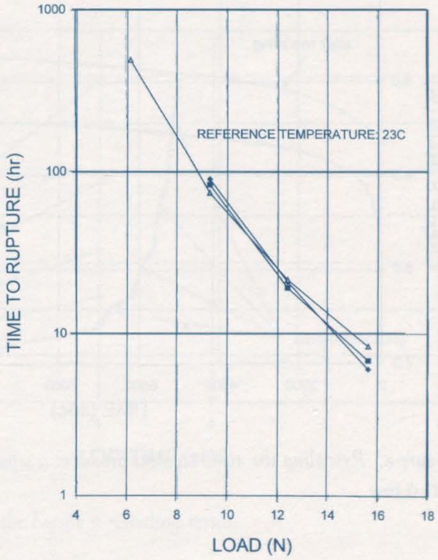


Figure 3. Master creep-rupture curve from the data in Figure 2.

TOUGHER.
Powerful Enough To Reroof Without Penetrations
BORN AND RAISED IN TORNADO ALLEY… DESIGNED TO SURVIVE

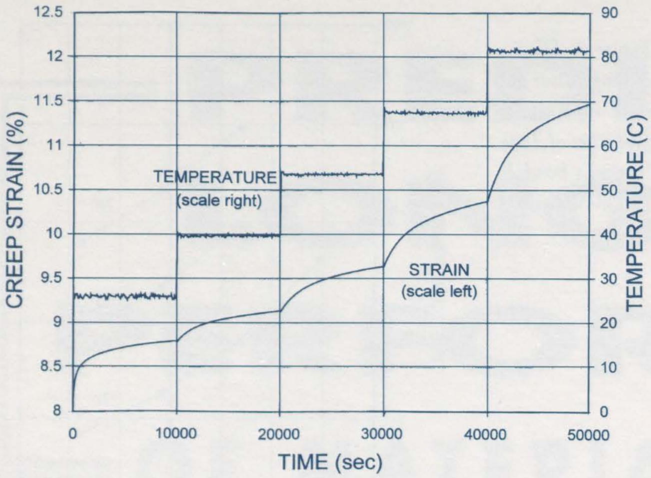


Figure 4. Creep strain and temperature vs. time for a SIM test at 62% UTS.

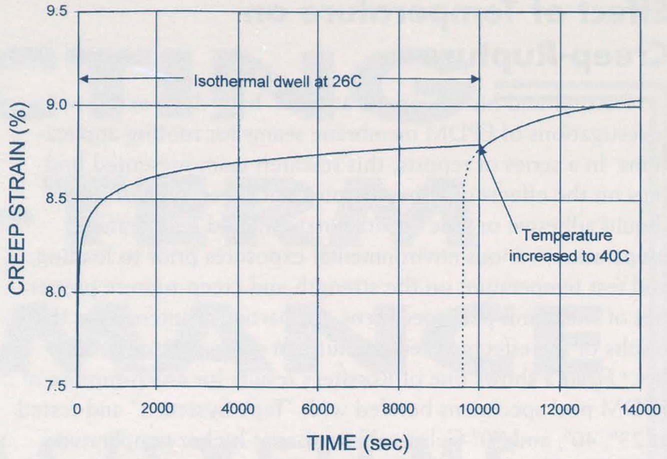


Figure 5. A close-up of creep strain vs. time for the initial portion of the SIM test in Figure 4.

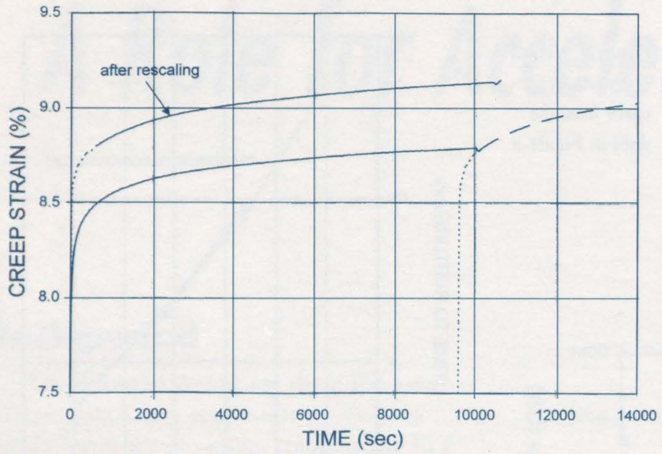


Figure 6. Rescaling the 40°C segment produces a separate curve starting at t-t=0 time.

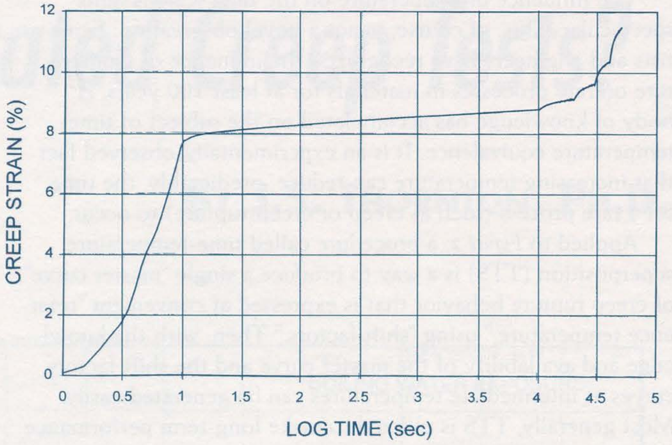


Figure 7. The creep data of Figure 4 plotted vs. log time.

and 40° are the equivalent of multiplication factors of 120 and 9, respectively. Note that the 6.2 N load data point at about 5 hours at 70° shifts to about 500 hours at 23°. This is over 400 hours beyond the real time data collected at 23°!

There is actually a problem with the TTS procedure we just performed. In reality, the shift factors for TTS are somewhat load dependent, so that using a single factor to shift a curve representing the results of several loads can lead to errors. A better approach for obtaining shift factors is to operate on the basic creep data generated at a single load but at different test temperatures. In the case of the T-peel results, the basic "creep" data would be the time-dependent extension of the specimen caused by the incremental failure of the bond. A master curve could then be generated based on shift factors so developed by expanding the test matrix to several different loads.

The resulting master curve would look very much like the one in *Figure 3*, but would differ in detail. The load dependency effect just mentioned would probably extend the 6.2 N point to longer times, perhaps beyond 1000 hours.

The Stepped Isothermal Method (SIM)

A new procedure, called the "stepped isothermal method" (SIM), developed initially for the geosynthetics industry, enables TTS to be applied to the creep behavior of a single specimen as it is subjected to stair step series of temperatures followed by isothermal dwells.[4]

As we have just seen, use of TTS is a way to obtain creep information for times longer than the longest test times. Conventional TTS has been used successfully for many decades,[5] and has worked especially well in the laboratory on linear viscoelastic response regimes where it is possible to perform repeated creep tests on the same sample (after allowing time for recovery between creep exposures). Repeated testing on the same sample, which eliminates lot-to-lot variation among samples and the uncertainty in quantifying the shift factors for certain ideal materials, doesn't work in the real world. For nonlinear viscoelastic response situations, which probably include most engineered polymers subjected to strains above a few percent, it has been necessary to test multiple replicate specimens to reduce uncertainty. Since creep tests are relatively expensive, there tends to be little enthusiasm for testing numerous replicate specimens.

The SIM appears to overcome this problem. An SIM creep test sequence begins with an ordinary creep test under constant load at a reference temperature in an environmental chamber. After a specified exposure time (often 10,000 seconds) and without releasing the applied load, the exposure temperature is increased rapidly by a specified amount, such as 14° C. Each temperature step must be accomplished rapidly. Moreover, the temperature step cannot be large because a key assumption of the method is that the state of the specimen before the step and the state after the step are corresponding states. Furthermore, the states before and after the steps should be steady states that would be readily obtainable in conventional creep tests at those temperatures. The load applied to the specimen is maintained through the temperature step to prevent any creep recovery from taking place. The temperature steps followed by isothermal dwells can be repeated until a temperature limit is achieved or the specimen ruptures.

In analyzing the SIM creep response data, the history that has accumulated in the specimen from the beginning of each new temperature exposure is accounted for in a simple and direct manner. This is by the selection of an equivalent earlier starting time, t', while rescaling the creep exposure times that were conducted at the temperatures above the reference temperature. The selection of t' is used to manipulate the slopes at the beginning of each creep segment. The boundary conditions for achieving smooth master curves are that 1) the slopes at the beginning of each new temperature segment are the same as those at the end of the previous segment; and 2) the creep strains are (nearly) the same at the points of juxtaposition of the resulting master curves.

To illustrate how SIM rescaling and shifting works, we examine creep and creep-rupture tests of a three-ply, 1000 denier, polyester yarn. *Figure 4* shows a creep strain curve

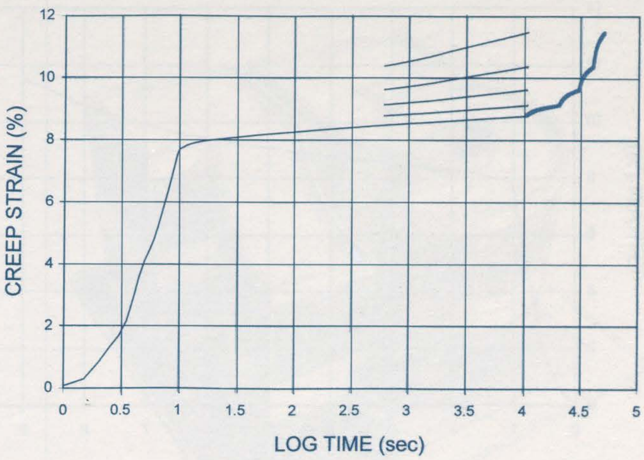


Figure 8. Rescaling the 40°, 54°, 68° and 82° C segments in log time (the hypothetical ramp-up portions of the rescaled segments are not included).

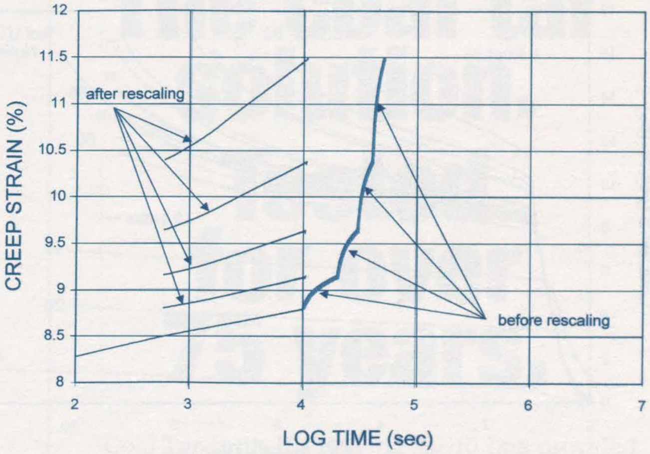


Figure 9. Close-up of the Figure 9 rescaling result.

FB FleeceBACK

TOUGHEST!

FOR SUPERIOR STRENGTH AND DURABILITY CHOOSE:
CARLISLE'S FLEECE-BACK FULLY-ADHERED ROOFING SYSTEM WITH FAST ADHESIVE

AVAILABLE IN 100- AND 115-mil THICKNESSES

YOUR SINGLE-PLY SOLUTION™
800-4-SYNTEC
www.carlisle-syntec.com

CARLISLE
Carlisle SynTec Incorporated

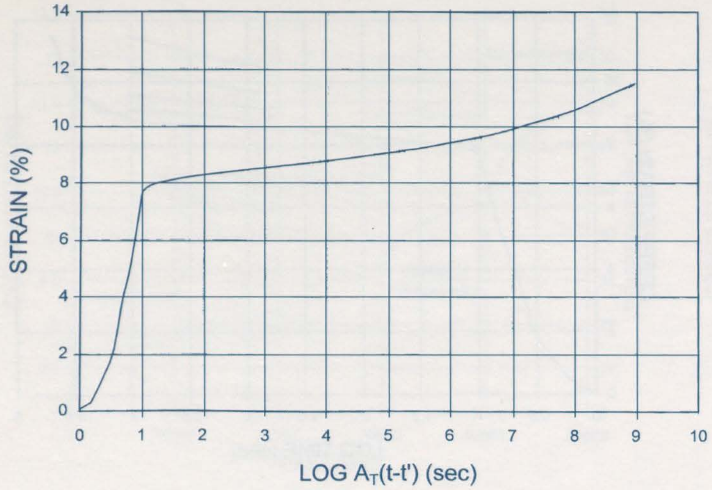


Figure 10. The master curve of creep strain vs. accelerated time results from horizontal shifting of the rescaled segments.

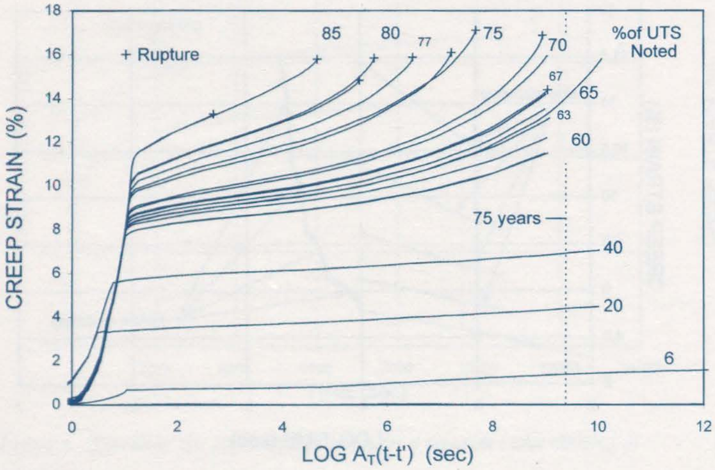


Figure 11. Family of master creep strain curves for a polyester yarn.

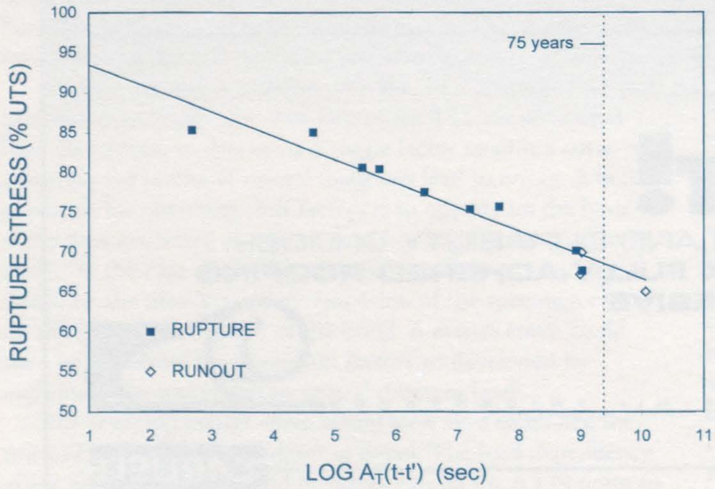


Figure 12. Master creep-rupture curve from the rupture data of Figure 11.

for an applied load of 62% of the ultimate tensile strength (UTS) of the yarn as a function of linear time for five exposures at different temperatures. Note that the times of the isothermal exposures are 10,000 sec each, and starting with about 26° C, the temperature steps are about 14° C each. An expanded view of the initial portion of the creep curve of *Figure 4* is shown in *Figure 5*. The first 10,000 sec segment of the data in *Figure 5* is the creep response at 26° C. Then at the 10,000 second point, the temperature is abruptly increased to 40° C and the new creep begins. Had the new segment of the creep curve been started from a zero load, the response to the load ramp might have had the shape shown by the dashed line. The new clock for this new segment of the creep curve can be conceptualized as starting with this hypothetical ramp at old clock time, t', at about 9600 sec. The new clock starts at t-t'=0. When the new segment is rescaled to the new clock time, we obtain the result shown in *Figure 6*. The new segment, which had extended to 20,000 sec on the old clock, is simply moved to the left as shown.

When viewed on log time plots instead of a linear time plot, the creep data we have just examined takes on a different appearance. The total strain curve, including the ramp up from zero strain, is shown vs. log time in *Figure 7*. The effect of rescaling is shown in *Figure 8* and *Figure 9* where *Figure 9* gives a close-up view of the curve shapes. Note that the rounded shapes of the creep segments shown in *Figures 4-6* become more linear in the semi-log plots after rescaling. Note also that we have picked the set of "ts" wisely since the slopes of the curves are appropriately matched to satisfy the boundary conditions. Hence, when the rescaled segments are shifted to the right, the smooth master curve of *Figure 10* is the result. The abscissa of the master curve is Log A_T(t-t') to signify that the results have been rescaled and shifted in accordance with TTS principles.

A rather complete family of creep strain curves is shown in *Figure 11*, including a number of ruptures (signified by "+s") at loads of 67% of UTS and higher. The orderly progression of the creep strain curves from loads of 6% up to 85% is worthy of note. The creep-rupture curve of *Figure 12* is drawn from the "+s," the rupture points of *Figure 11*. The longest test time for any individual creep curves in this example was less than 18 hours and yet the creep-rupture times extend beyond 75 years. Real time tests out to 10,000 hours have been performed that validate the results shown in *Figures 11* and *12*. Based on excellent agreement between 10,000-hour real time creep and SIM tests and the tremendous time savings offered by the latter, SIM has become accepted in the geosynthetic industry.

Summary Comments

Already within the geosynthetics community, SIM has been used by manufacturers to define quality vs.

economy relationships for raw materials, to qualify potential raw material sources, and to determine the long-term performance of potential new products. SIM has also been used to verify already established creep and stress rupture performance behavior. The use of SIM has made the previously unaffordable become feasible in terms of cost and turn-around times.

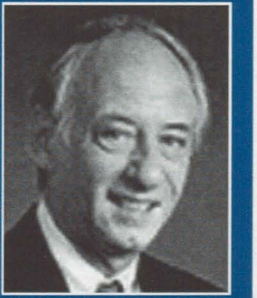
Like the geosynthetics industry, the roofing community can benefit from long-term performance data. Indeed, recent work has suggested that long-term performance data may be the only way to differentiate roofing variables. SIM testing holds promise as an exciting new tool to reach this goal.

References

1. Hogan, L., "The Province of Testing," *Interface*, March 1997, pp 10-12.
2. Rossiter, W.J., "The Effect of Application Parameters on Adhesive Bonded Seams in Single Ply Membranes," *Second International Symposium on Roofing Technology*, 1985, p. 386.
3. Rossiter, W.J., Vange, M.G., and Kraft, K.M., "A Performance of Tape-bonded Seams of EPDM Membranes: Factors Affecting the Creep-rupture Response of Tape-bonded and Liquid-adhesive-Bonded Seams," *NIST Building Science Series 177*, U.S. Dept. Of Commerce, Washington, D.C., July 1998.
4. Thornton, J.S., Allen, S.R., Thomas, R.W. and Sandri, D., "The Stepped Isothermal Method for Time-temperature Superposition and its Application to Creep Data on Polyester Yarn," *Sixth International Conference on Geosynthetics*, Atlanta, GA, 1998, pp 699-706.
5. Ferry, J.D., *Viscoelastic Properties of Polymers*, 3rd Edition, John Wiley and Sons, New York, NY, 1980. ■

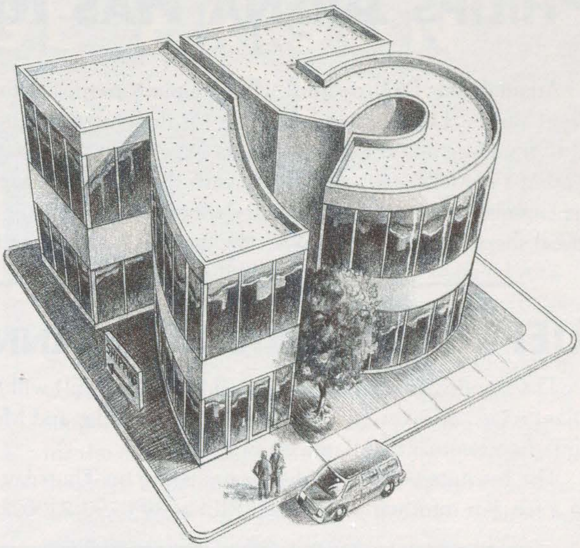
ABOUT THE AUTHOR

Scott Thornton is chairman and principal scientist of Texas Research International, Inc., Austin, TX (512-263-2101, Ext. 138). His research interests include accelerated reliability and life testing of materials and structures. He developed the SIM and applied it to a geosynthetic product in 1996. Thornton is a member of the American Society of Mechanical Engineers, the Adhesion Society, the American Society of Testing and Materials, ASM International, and the International Geosynthetics Society.



SCOTT THORNTON

RCI: On the Frontier of a New Millennium
Reno, Nevada
March 24–28, 2000



The coal tar solution. Tested for over 75 years.

Coal Tar Built-Up Roofing (BUR) has provided weather protection for over 75 years. (Actually, longer than that, but our records only go back that far.)

The preferred system for flat or low-sloped roofs, Coal Tar BUR is durable, easily maintained, and has unique cold-flow properties.

Coal Tar BUR provides long-lasting protection for your customers. And peace of mind for specifiers.

For more information, visit our web site at **www.koppers.com**.

KOPPERS INDUSTRIES

Koppers Industries, Inc.
Commercial Roofing Dept.
Koppers Building • Pittsburgh, PA 15219
1-800-558-2706