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DOE-SPONSORED CABLE AGING RESEARCH AT SANDIA NATIONAL LABORATORIES

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ABSTRACT

Cables have been identified as critical components requiring detailed technical evaluation for extending the lifetime of Light Water Reactors beyond 40 years. This paper highlights some of the DOE-sponsored cable aging studies currently underway at Sandia. These studies are focused on two important issues: the validity of the often-used Arrhenius thermal aging prediction method, and methods for predicting lifetimes in combined thermal-radiation environments. Accelerated thermal aging results are presented for three cable jacket and insulation materials, which indicate that hardening of the outside surface has an Arrhenius temperature dependence and correlates well with reductions in ultimate tensile elongation. This suggests that the indentor approach is a promising NDE technique for cable jacket and unjacketed insulation materials installed in thermally-dominated regions of nuclear power plants.

INTRODUCTION

In the task of extending the lifetime of Light Water Reactors beyond 40 years, cables have been identified as important components requiring detailed technical evaluation. Polymeric components (e.g., insulation and jacketing materials) represent some of the most critical and potentially susceptible cable materials. In particular, NRC comments on the Low-Voltage, Environmentally-Qualified Cable Industry Report [1] focused on the uncertainties and possible insufficiency of data regarding (1) synergistic and dose-rate effects and (2) the Arrhenius thermal aging methodology. Our aging research program is attempting to develop unique experimental techniques and aging models that will help resolve both of these issues. We have derived an aging approach to handle synergistic and dose-rate effects [2-6]. This methodology, which we refer to as time-temperature-dose rate superposition, has been successfully applied to several important nuclear power plant cable materials. We are also developing a detailed understanding of the Arrhenius methodology and methods for testing its validity [7-8]. This paper highlights our recent investigations of nuclear cable materials and describes some counterintuitive combined environment studies on crosslinked polyolefin (CLPO) cable insulation materials in which mechanical degradation slows as the aging temperature is raised.

DISCLAIMER

RESULTS AND DISCUSSION

Thermal Aging Studies

Predictions and/or simulations of the lifetimes of nuclear power plant cable materials exposed to aging environments dominated by thermal effects are typically based on the Arrhenius methodology. This approach was derived from the knowledge that the rate of a chemical reaction is usually proportional to $\exp(-E_a/RT)$, where E_a is the Arrhenius activation energy, R is the gas constant and T is the absolute temperature. Although the polymers comprising cable materials degrade by a complex series of chemical reactions, steady state kinetic analysis of these reactions typically yields a rate expression with an approximately Arrhenius temperature dependence, where E_a represents an effective activation energy for the underlying mix of reactions. If the overall degradation mechanism remains unchanged throughout the experimental temperature range, the logarithm of the time to a given amount of degradation (property change) should be linearly related to the inverse absolute temperature, yielding Arrhenius behavior.

Figure 1 shows ultimate tensile elongation data versus aging time at five aging temperatures for a nitrile rubber material [7,8]. As shown in Fig. 2, these data display excellent Arrhenius behavior when analyzed at $e/e_0 = 0.75$, 0.5 and 0.25 [7]. A more rigorous analysis uses the time-temperature superposition principle to shift all of the data of Fig. 1 to a common reference temperature, T_{ref} [2,5]. This is accomplished by multiplying the times appropriate to experiments conducted at each temperature T by a shift factor, a_T, given by

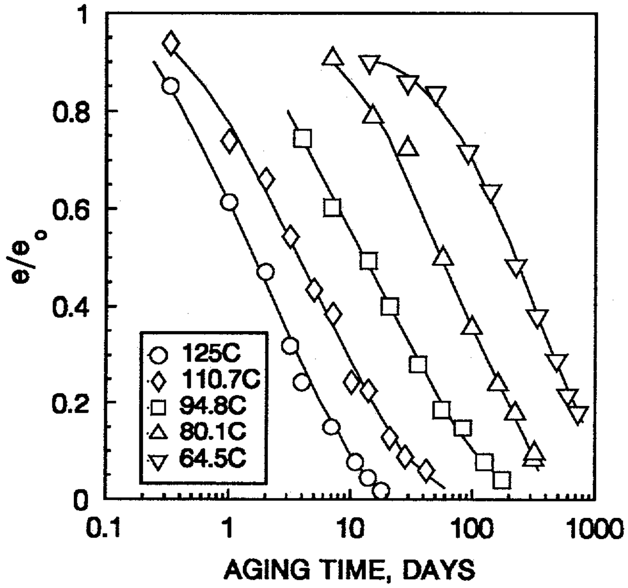

Fig. 1. Ultimate tensile elongation (e) of the nitrile rubber normalized to its unaged value (e_O) versus air-oven aging time and temperature.

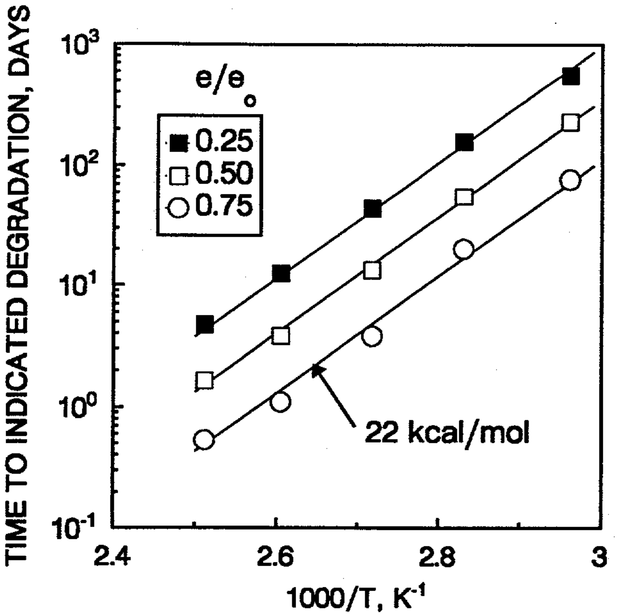

Fig. 2. Conventional Arrhenius plots of elongation results for the nitrile rubber.

$$a_T = \exp\{(E_a/R)\ [(T_{ref})^{-1} - (T)^{-1}]\}$$

Figure 3 shows time-temperature superposed results at a reference temperature of 50°C for the data of Fig. 1 using an E_a of 22 kcal/mol. Excellent superposition occurs, which is not surprising given both the linearity of the Arrhenius plots in Fig. 2 and the independence of the slopes to the amount of damage. In comparing the normal Arrhenius analysis approach with time-temperature superposition, it should be noted that the former uses a processed, truncated data set (typically a single degradation level such as $e/e_0 = 0.5$). The time-temperature superposition approach is superior because the entire data set is used to determine the activation energy (E_a) and to confirm Arrhenius behavior.

Although the Arrhenius model appears to work for many materials, closer scrutiny raises some significant concerns. For example, Arrhenius behavior is often observed for ultimate tensile elongation results, whereas the (typically unreported) ultimate tensile strength data from the same mechanical property tests are non-Arrhenius. This non-Arrhenius behavior is seen for the nitrile material in Fig. 4, when its tensile strength data are time-temperature superposed using the same 22 kcal/mol E_a found to apply to the elongation data. In fact, it is clear from this figure that the tensile strength data could never be superposed, since it drops in the latter stages at some temperatures and increases at others.

The apparent contradiction between the elongation and tensile strength results was resolved [7,8] through the use of our unique modulus profiling apparatus [9,10]. This technique allows us to quantitatively map the modulus of a material with ~50μm resolution. After aging, samples were cut and modulus measurements were taken across the sample cross-section. Figure 5 shows

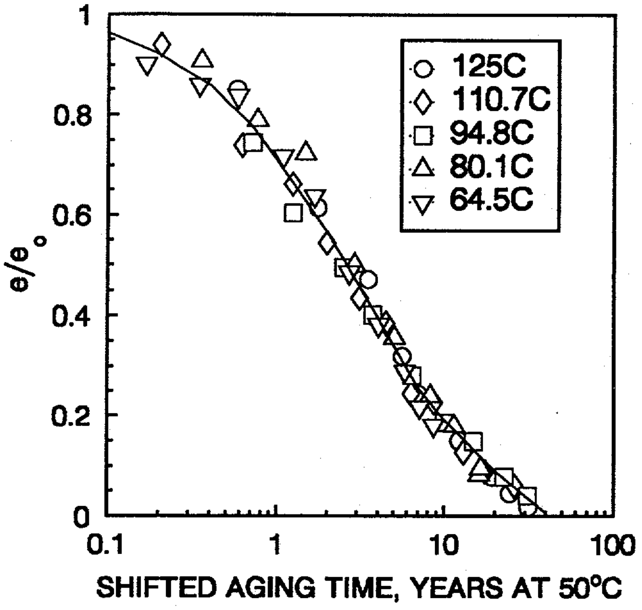

Fig. 3. Time-temperature superposition of the data from Fig. 1 using E_a = 22 kcal/mol.

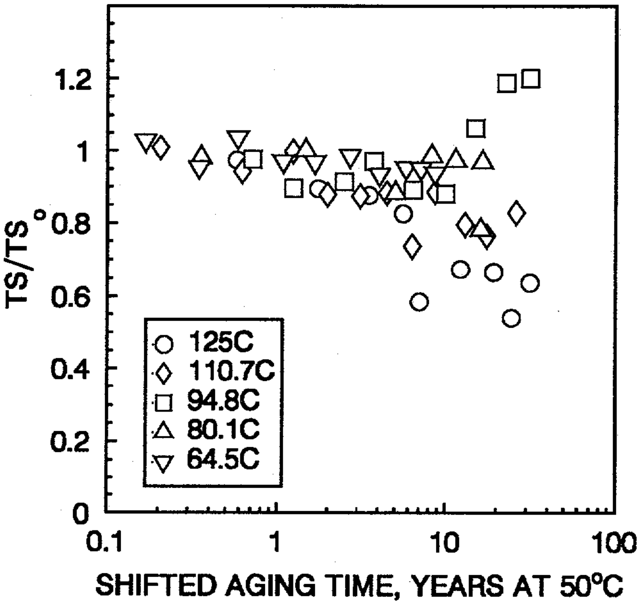

Fig. 4. Time-temperature superposition of the normalized nitrile ultimate tensile strength data using E_a = 22 kcal/mol.

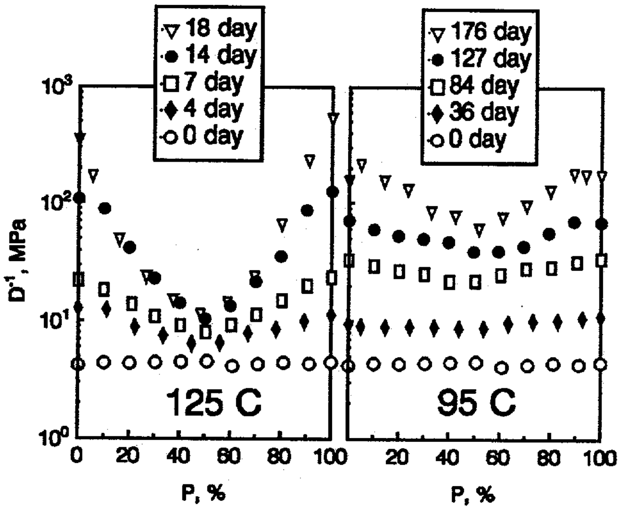

Fig. 5. Modulus profiles for 2.0-mm thick nitrile rubber samples after aging for various times at the indicated temperatures. The abscissa, P, refers to the percentage of the total distance from one air-exposed sample surface to the opposite air-exposed surface.

representative results at the highest temperature (125°C) and an intermediate temperature (95°C), where P refers to the percentage of the distance from one air-exposed surface of the sample to the opposite air-exposed surface. The heterogeneous behavior observed is very common under typical air-oven aging conditions and is due to diffusion-limited oxidation (DLO). This phenomenon leads to equilibrium oxidation at the air-exposed sample surfaces and reduced or non-existent oxidation towards the sample interior [7-11]. The importance of DLO depends upon sample thickness and the competition between oxygen consumption in the material and the diffusion rate of oxygen into the material [12,13]. Since the E_a for oxygen consumption is typically much larger than that for oxygen diffusion, the importance of DLO normally drops as the temperature is lowered, as seen in Fig. 5.

Since the tensile strength of a material is dependent upon the integrated force across a material at tensile failure, the presence of temperature-dependent DLO effects (Fig. 5) can be used to explain the failure of the Arrhenius model for this property. For the elongation, modulus values (related to hardness) are maximum at the surface, where equilibrium oxidation occurs. During tensile testing, cracks can be expected to initiate in the most embrittled portion of the material, which, due to DLO, will be the surface. If these cracks, once initiated, propagate quickly through the material, then the equilibrium surface oxidation will determine (and have the same E_a as) the tensile elongation. This turns out to be the case [7,8], as demonstrated in Fig. 6, which shows that the surface modulus values have an excellent correlation with the normalized elongation results.

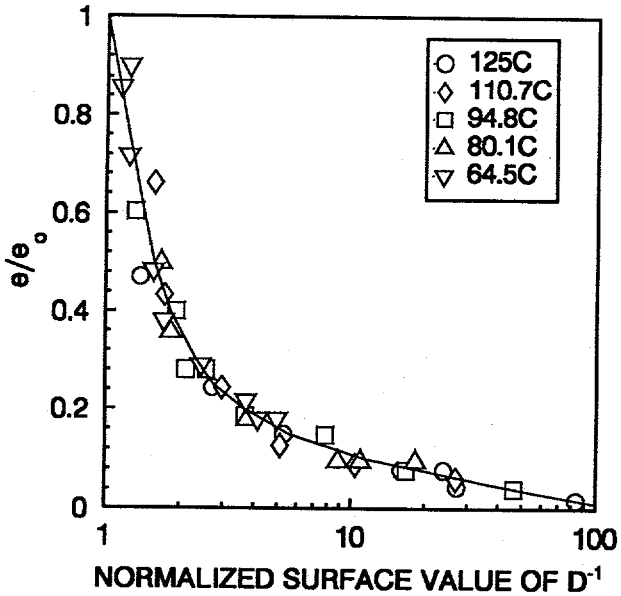

Fig. 6. Normalized elongation (e/e_o) plotted versus the normalized surface modulus for the nitrile rubber.

Many of the behaviors noted above for the nitrile material are commonly observed for other elastomeric materials. For instance, we have documented surface modulus increases (oxidative hardening), important DLO effects and immediate propagation of surface-initiated cracks for neoprene and styrene butadiene rubber materials [7]. Theoretical modeling of typical accelerated air-oven aging conditions indicates that DLO effects should be quite common for ~1 mm thick polymeric materials [14]. But these behaviors cannot be assumed to be universal, as we will now show for three nuclear cable materials.

One cable material studied was a 15 mil individual hypalon jacket wrapped over the EPR insulation from a three conductor Anaconda Flame-Guard FR-EP cable. The three hypalon individual jackets were all black but two had "painted" white and red surfaces to distinguish conductors. To minimize DLO effects, the material was separated from the overall jacket and insulation and then oven-aged at temperatures ranging from 100°C to 150°C. Modulus profiles were obtained on the material with the white outer surface; results for aging at 150°C are shown in Fig. 7. Except for a small increase at the outer surface presumably caused by the presence of the harder white "paint", the profiles are flat for both unaged and aged materials. Since similarly shaped profiles occur at lower temperatures, DLO effects are insignificant over the entire temperature range, not unexpected given the small sample cross-section.

The best time-temperature superposition of the tensile elongation results for this hypalon material occurs using an E_a of 25.5 kcal/mol; this is shown in Fig. 8 at a T_{ref} of 50°C. The same value of E_a also leads to excellent superposition of the tensile strength data (not shown), which is not surprising given the absence of any important DLO effects. Based on our earlier discussion, we expect the decrease in tensile properties to be based on the modulus increases resulting from

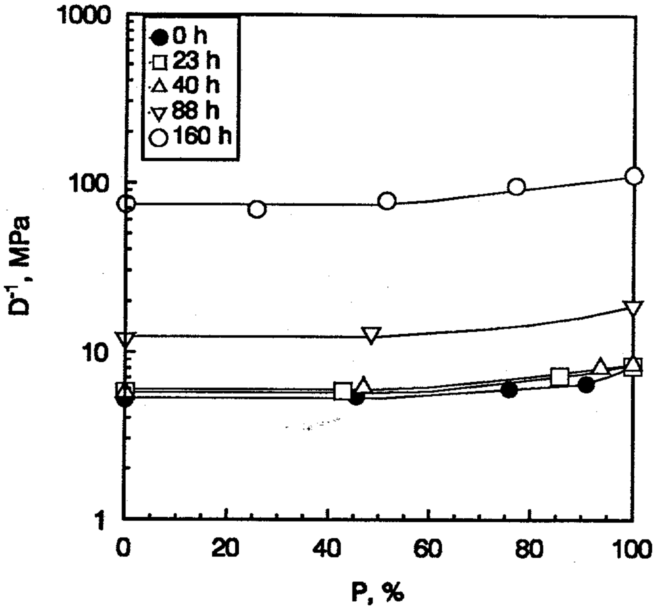

Fig. 7. Modulus profiles for 0.4 mm thick Anaconda hypalon samples after aging for the indicated times at 150°C.

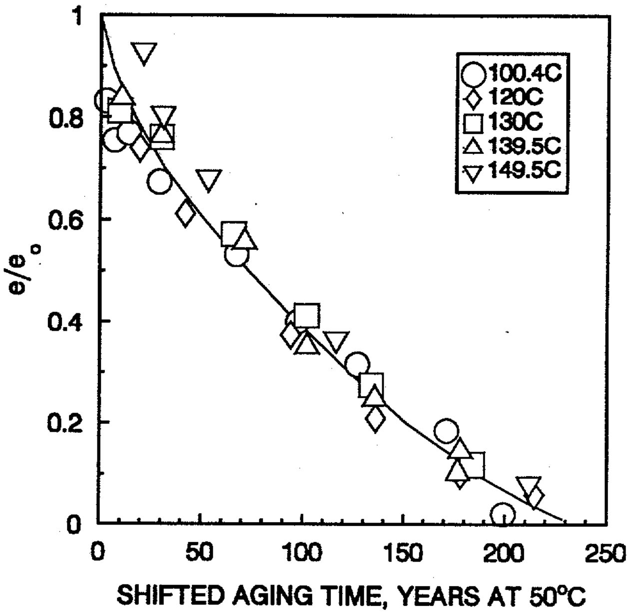

Fig. 8. Time-temperature superposition of the normalized elongation data for the Anaconda hypalon using $E_a = 25.5$ kcal/mol.

oxidative crosslinking. These modulus increases versus time and temperature are plotted in Fig. 9. As expected, excellent time-temperature superposition of these data occurs for E_a = 25.5 kcal/mol (Fig. 10). Finally, Fig. 11 shows the excellent correlation between modulus and tensile elongation.

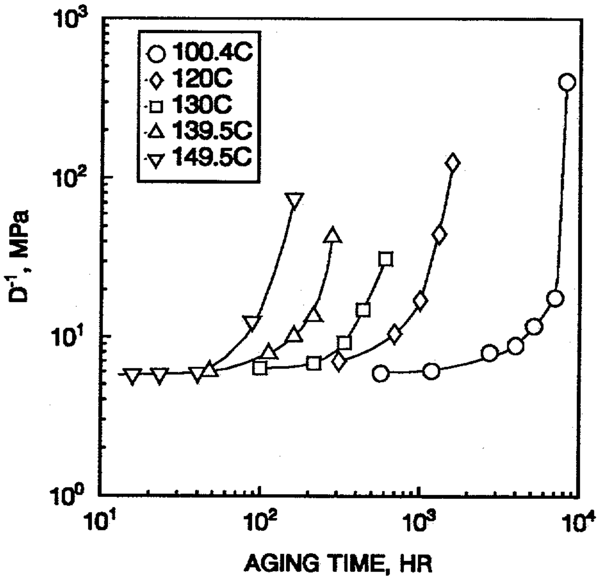

Fig. 9. Modulus values of the Anaconda hypalon versus aging time at the indicated air-oven aging temperatures.

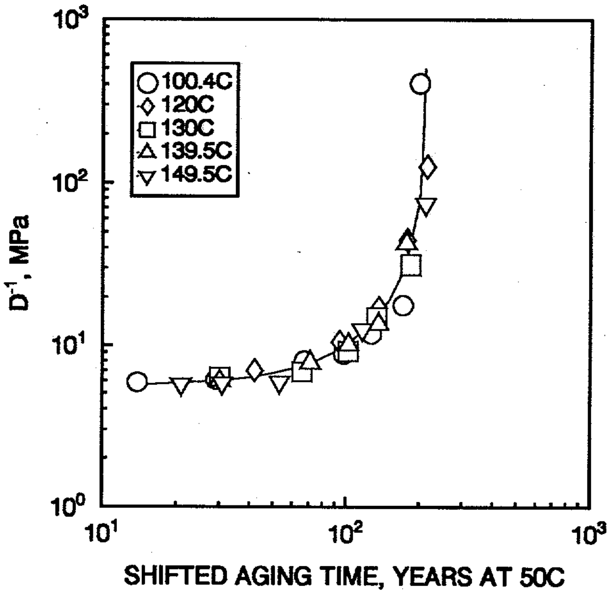

Fig. 10. Time-temperature superposition of the modulus results from Fig. 9 using E_a = 25.5 kcal/mol.

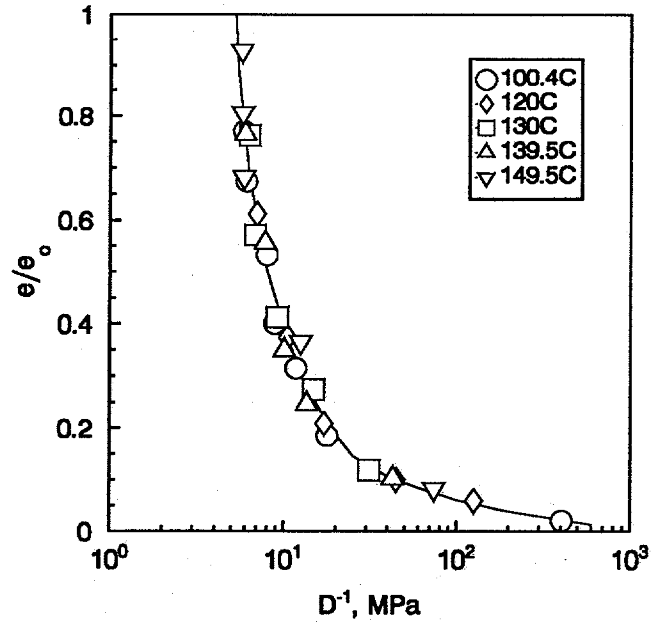

Fig. 11. Normalized elongation (e/e_O) plotted versus the normalized surface modulus for the Anaconda hypalon.

From this figure, we conclude that substantial material degradation occurs when the material modulus increases to ~30 MPa.

The second cable material is another hypalon, the FR jacket of a single conductor Kerite FR cable. Although this material had a number of unusual behaviors, it will be apparent that its overall behavior is generically similar to that of the Anaconda hypalon. Mechanical property data were taken versus time at approximately 10°C intervals from 160°C to 90°C. Figure 12 shows a conventional Arrhenius plot for four levels of change in ultimate tensile elongation value. For the five lowest temperatures, parallel lines corresponding to an Arrhenius activation energy of 26 kcal/mol accurately describe the data, but slight deviations are apparent at the three highest temperatures of 140°C, 150°C and 160°C. It turns out that modulus profile results and theoretical calculations [13,14] indicate the presence of important DLO effects above approximately 130°C, implying that the observed deviation from Arrhenius behavior is due to DLO anomalies. Experimental evidence for these DLO effects is seen in Fig. 13, which shows modulus profile results at 160°C, where $P = 0$ and $P = 100$ correspond to the outer and inner surfaces, respectively. Because the Arrhenius model fails above 130°C due to DLO effects, we restrict time-temperature superposition analysis to the data from 130°C to 90°C. The result, shown in Fig. 14, indicates that excellent superposition of the data from this temperature range is possible with an E_a of 26 kcal/mol. The E_a value and the resulting predictions at 50°C for this hypalon material are quite similar to those for the earlier Anaconda hypalon material (25.5 kcal/mol and the predictions shown in Fig. 8).

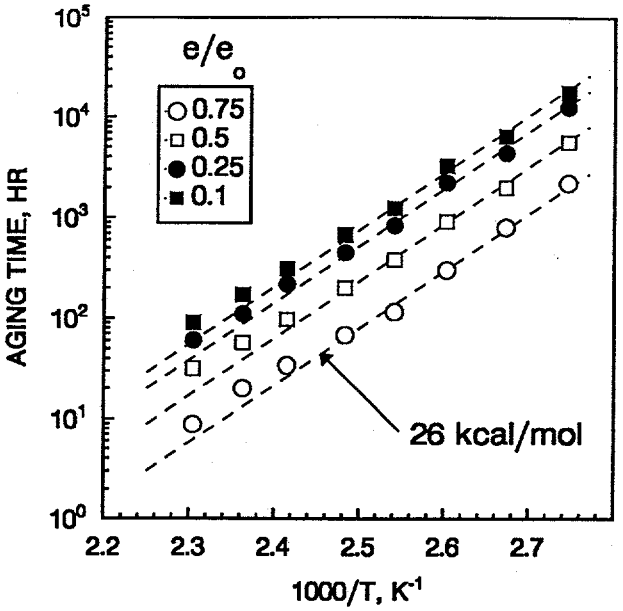

Fig. 12. Conventional Arrhenius plot of elongation results for the Kerite hypalon jacket.

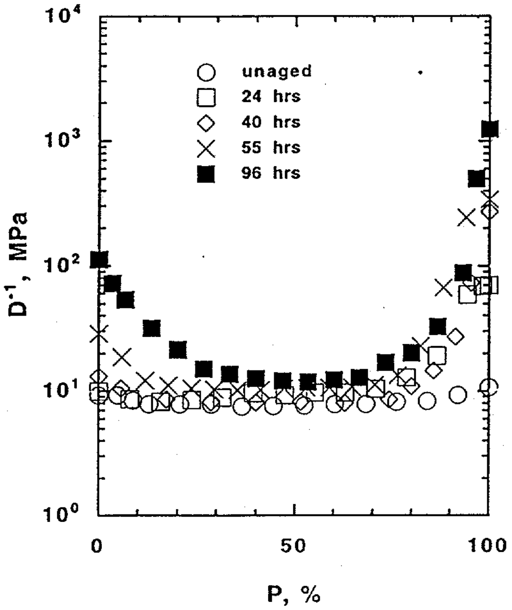

Fig. 13. Modulus profiles for 1.5 mm thick Kerite hypalon jacket materials after aging at 160.7°C for the indicated times. The outside surface of the jacket corresponds to P = 0.

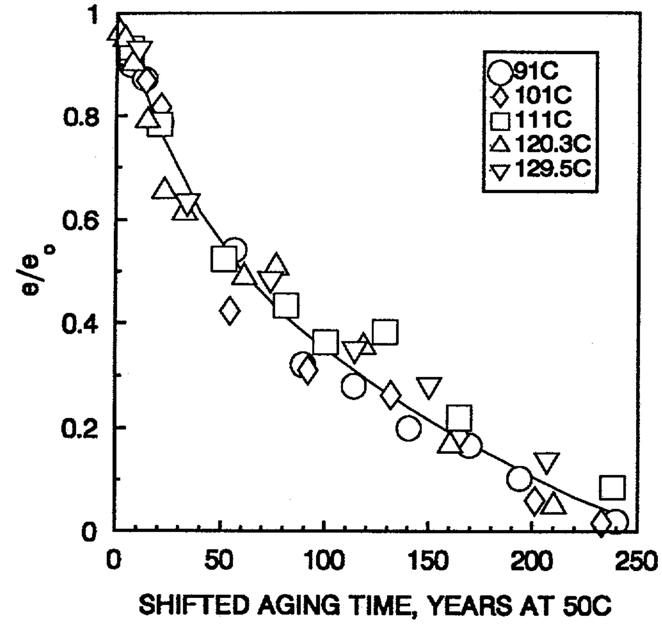

Fig. 14. Time-temperature superposition of the normalized elongation data for the Kerite hypalon jacket using E_a = 26 kcal/mol. Data above 130°C were not used in this plot.

Besides showing strong evidence of DLO effects, the modulus profile results of Fig. 13 show that an enhanced oxidation mechanism occurs near the inside surface of the material (P = 100). It turns out, however, that the enhanced inside oxidation is not correlated to mechanical failure (manuscript in preparation). We therefore examine the Arrhenius behavior of the modulus at the outside surface of the material, since this will be correlated to the equilibrium oxidation in the absence of DLO effects. Following the procedure used in Figs. 9 and 10 for the Anaconda hypalon, we carry out time-temperature superposition on the outside modulus results for the Kerite jacket, and find that 26 kcal/mol gives excellent superposition, as seen in Fig. 15. This is the same E_a determined for the elongation data at temperatures ranging from 90°C to 130°C, which adds confidence to the predicted extrapolations of Fig. 13 and confirms that the outside surface modulus is an ideal parameter for determining the Arrhenius E_a and is an excellent predictor of jacket condition. The slight deviations from Arrhenius behavior for elongation data above 130°C indicates that cracks do not immediately propagate at the higher temperatures where DLO effects are present.

The third cable material studied was the FR insulation of the Kerite FR single conductor cable, a proprietary compound. Oven aging was run at temperatures ranging from approximately 160°C to 120°C and the tensile elongation results gave excellent time-temperature superposition with an E_a of 23 kcal/mol, as seen in Fig. 16. The same value of E_a led to superposition of the outside surface modulus values from modulus profiling results. Therefore we again find a reasonable correlation between the modulus changes and tensile elongation results (Fig. 17).

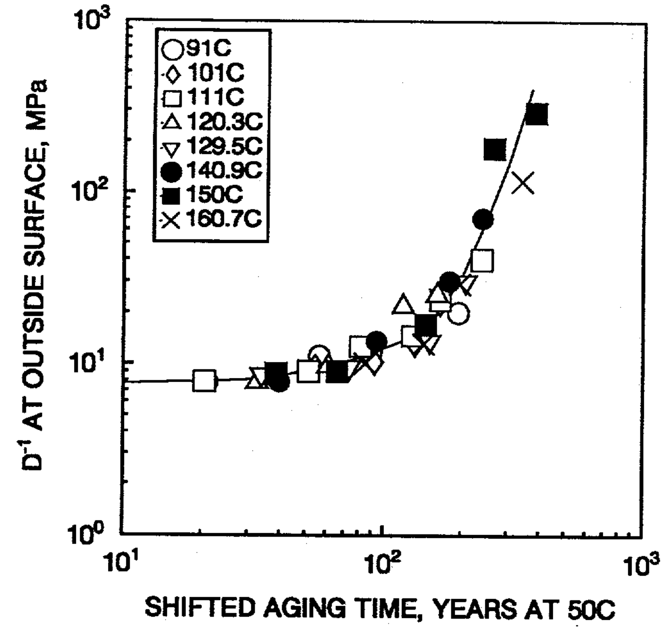

Fig. 15. Time-temperature superposition of the outside surface modulus values for the Kerite hypalon jacket material using $E_a = 26$ kcal/mol.

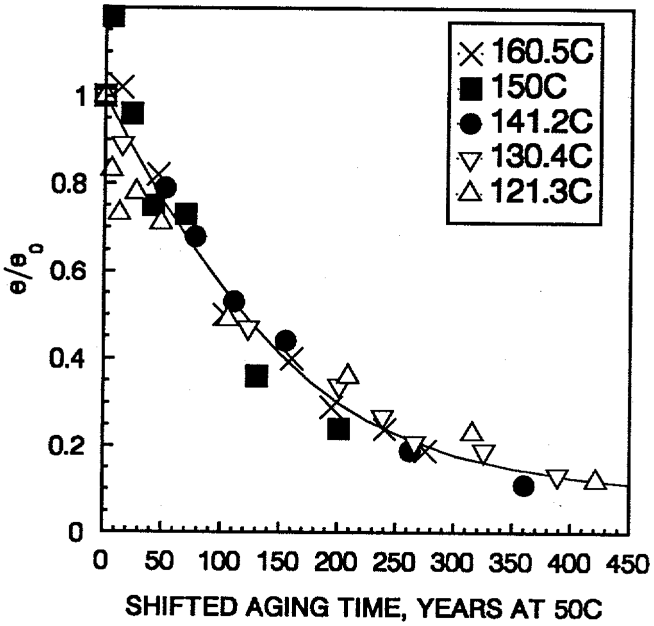

Fig. 16. Time-temperature superposition of the normalized elongation data for the Kerite FR insulation using $E_a = 23$ kcal/mol.

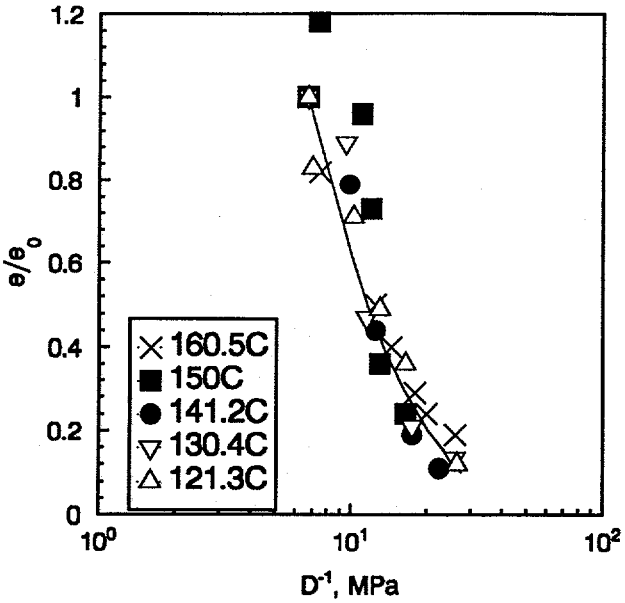

Fig. 17. Normalized elongation (e/e_0) plotted versus the normalized surface modulus for the Kerite FR insulation.

Superposition is not achieved, however, when the same E_a is used to shift the tensile strength data, as seen in Fig. 18. Unlike earlier materials where DLO effects caused non-superposition of the tensile strength data, modulus profile results indicate that DLO effects are unimportant for the Kerite insulation material, except for slight effects at 160°C. Therefore the tensile strength results implicate changes in the chemical mechanism. They, in fact, suggest that the relative mix between scission and crosslinking events (the former tending to decrease tensile strength, the latter tending to raise it) favors a relative decrease in scission as the temperature is lowered. This evidence of a change in the mix of reactions as the temperature is lowered raises concerns over the extrapolation of the high temperature results (160°C to 120°C) to the temperatures of relevance to nuclear power plant aging (e.g., 50°C). For this reason, we are currently using ultrasensitive techniques to obtain oxygen consumption data for this material from 160°C down to temperatures approaching 50°C. These data should allow us to determine whether there is evidence of a change in E_a for oxygen consumption over the extrapolation region. If no evidence is found, it would suggest that the E_a for elongation and modulus should not change significantly.

The above results make it clear that perhaps the two best parameters for measuring degradation and making lifetime predictions are the ultimate tensile elongation and the surface modulus of the material. When DLO effects are important, tensile strength data are often meaningless, but elongation and surface modulus results can still be correlated if cracks that initiate at the hardened surfaces of materials quickly propagate through the material. When surface-initiated cracks do not propagate, elongation data can become non-Arrhenius and only the surface modulus data remain predictive of the true E_a for the degradation mechanism. This

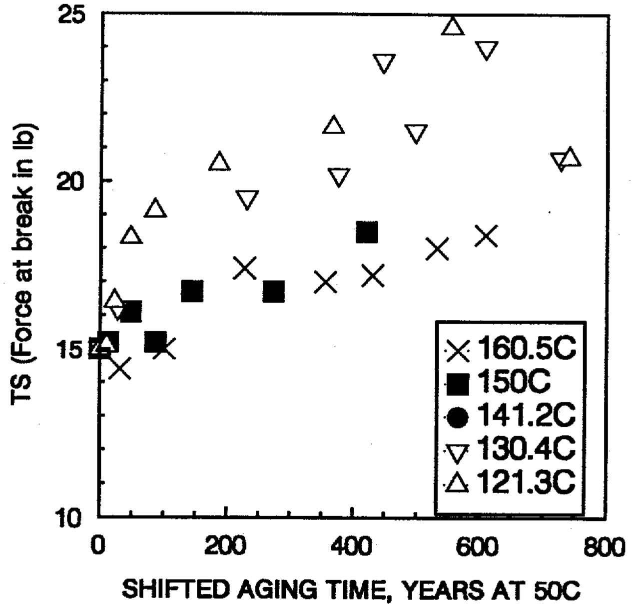

Fig. 18. Time-temperature superposition of the tensile strength data for the Kerite FR insulation using the same activation energy (23 kcal/mol) found to superpose the elongation results in Fig. 16.

discussion offers compelling evidence that the indentor approach is a promising non-destructive evaluation technique for thermal-dominated regions of nuclear power plants [15-17]. This specially designed portable test apparatus measures the penetration force of a blunt, conical probe as a function of penetration depth into the jacket of a cable. A slope (units of force per length) is extracted from the force versus penetration curve. This leads to the so-called "indenter modulus", which, though not a true modulus (units of force per area), is closely related to modulus. Although the indenter modulus is sensitive to more than just the surface modulus value, the relative importance of subsurface values diminishes with depth, implying that it is dominated by near surface modulus values. In addition, under the long-term, low-temperature aging occurring under ambient nuclear power plant conditions, DLO effects will be minimal, implying that relatively homogeneous modulus values might be expected. If correlations like those shown in Figs. 6, 11 and 17 can be made for important cable materials and if the resulting surface modulus values can be correlated with indenter modulus results, the indenter approach may prove to be the most viable NDE technique for assessing the condition of thermally-dominated, in-plant cable jackets.

Combined Radiation Plus Temperature Studies- Yankee Rowe cables

In earlier reports [2-6], we introduced and tested a time-temperature-dose rate superposition methodology which can be used for making predictions under combined radiation plus temperature environments, even in the presence of dose-rate and synergistic effects. When applicable, the methodology allows predictions to be made at the low dose rates existing under ambient nuclear power plant aging conditions. The methodology was successfully applied to many nuclear cable materials, and extrapolated predictions from the model were verified by comparisons with intermediate-term (7 to 12 year) results for similar or identical materials aged in nuclear environments. The methodology failed for several EPR and CLPO materials in which it was found that, at a constant dose rate, a decrease in aging temperature surprisingly increased the rate of mechanical degradation.

We are currently receiving naturally aged cables from Yankee Rowe, some of which were installed around 1960 (silicone, polyethylene, PVC and butyl materials), others in the late 70's and early 80's (CLPE, CLPO, neoprene, hypalon materials). We are attempting to obtain materials that came from high radiation aging areas of the plant (dose rates greater than 1 R/hr) and high temperature regions (greater than 45°C) either singly or in combination. Yankee Rowe will provide best estimates of the average environments seen for each section of cable. If available, Yankee Rowe will also supply us with "unaged" warehouse cables corresponding to the plant-aged cables, and sections of the plant-aged cables from benign aging environments. These materials will be used to estimate baseline properties and as sources of "unaged" materials for accelerated simulations.

We will use various techniques (e.g., tensile properties, infrared analyses, swelling, solubility, density, modulus profiling) to determine the levels of degradation in the materials. For materials removed from higher radiation environments, we will generate combined environment data at our radiation-aging facility to test the time-temperature-dose rate superposition methodology. If the material is from a generic class of previously studied materials [2-4], we will compare the generic model predictions with the ambient-aged cable material results. Yankee Rowe materials dominated by thermal aging will be used to test the Arrhenius thermal aging methodology.

Combined Radiation Plus Temperature Studies- Anomalous Materials

We find unusual behaviors for several CLPO and EPR materials, where the combined environment degradation rate under constant dose-rate conditions can *increase* as the temperature is lowered. As one example, combined environment experiments were run on a Brandrex CLPO insulation material using dose rates ranging from 17 Gy/h to 5200 Gy/h (1 Gy = 100 rad) at temperatures from 22°C to 120°C. Tensile tests versus aging time at each combined environment were used to estimate the dose required for the ultimate tensile elongation to decrease to 100% absolute (initial value was ~310%). The resulting values of dose to equivalent damage (DED) are plotted in Fig. 19 versus dose rate and temperature. DED plots are useful for discussing and analyzing combined environment data, since a dose-rate effect at a selected temperature can be immediately recognized if the DED values depend on dose rate. For the Brandrex CLPO, the higher temperature results (60°C and higher) shown in Fig. 19 are consistent with expectations. That is, under constant dose-rate conditions, the degradation rates increase as the temperature increases. But the results at 41°C and 22°C (solid symbols) are counterintuitive, since they show that the degradation rate at these temperatures are approximately 2.5 to 3 times faster than at 60°C. Gel content data, shown in Fig. 20, give further evidence for a fundamental difference in degradation mechanism at these lower temperatures. The gel content is sensitive to the polymer network properties and intrinsically related to the competition between chain scission and crosslinking events, which are the molecular level basis of the degradation (the former tends to

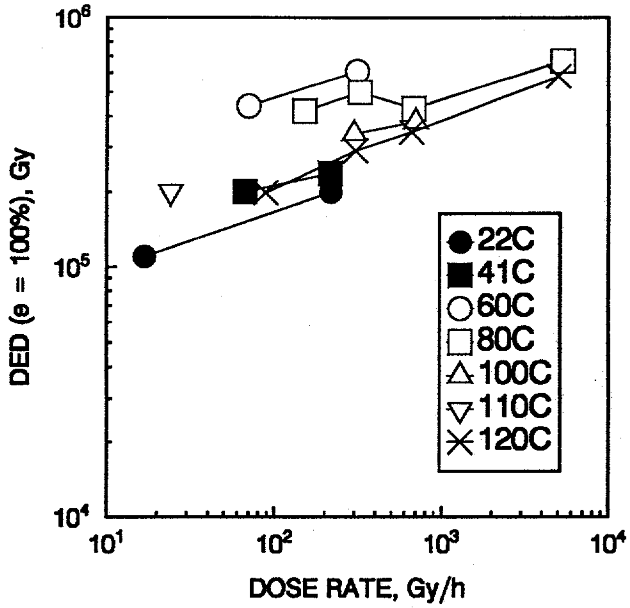

Fig. 19. Dose required for the ultimate tensile elongation of the Brandrex CLPO to decrease to 100% (initial value of 310%) versus dose-rate and temperature.

decrease gel content, while the latter tends to increase it). The results shown in Fig. 20 indicate that high temperature dominance by crosslinking switches abruptly to low temperature dominance by chain scission.

Similar behaviors exist for other CLPO materials. As an example, selected results from our studies and from a long-term, low dose-rate program [18] are plotted in Fig. 21 for a Rockbestos CLPO. Degradation rates are again faster at 22°C and 40°C than at higher temperatures, but for this material, the rates continue to decrease until the temperature reaches approximately 100°C.

Since ambient nuclear power plant aging temperatures are often in the range of approximately 20°C to 50°C, these counterintuitive effects must be understood before confident lifetime predictions for such materials can be generated. These CLPO materials are semi-crystalline, and melting plus reforming of crystallites (equivalent to molecular rearrangements) occurs over most of the temperature ranges investigated in Figs. 19 and 21. In fact, the dangers of extrapolating accelerated aging results across a transition of a material or modeling results through a transition region, such as the crystalline melting point region, are well recognized [2-6]. The relative changes in the amorphous fraction and the relative molecular mobility at different temperatures will clearly be one contribution to the observed behavior. We have also determined that reactivities of hydroperoxide species, which are important intermediates in the chemistry, appear to influence these anomalous effects. We are currently using a variety of techniques, including annealing experiments, oxygen consumption measurements, differential scanning

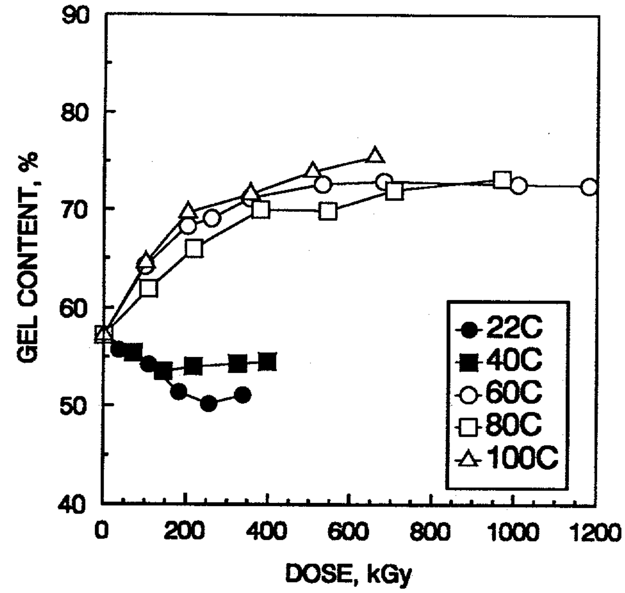

Fig. 20. Gel content results versus temperature for combined environment aging of Brandrex CLPO at dose rates of ~250 Gy/h.

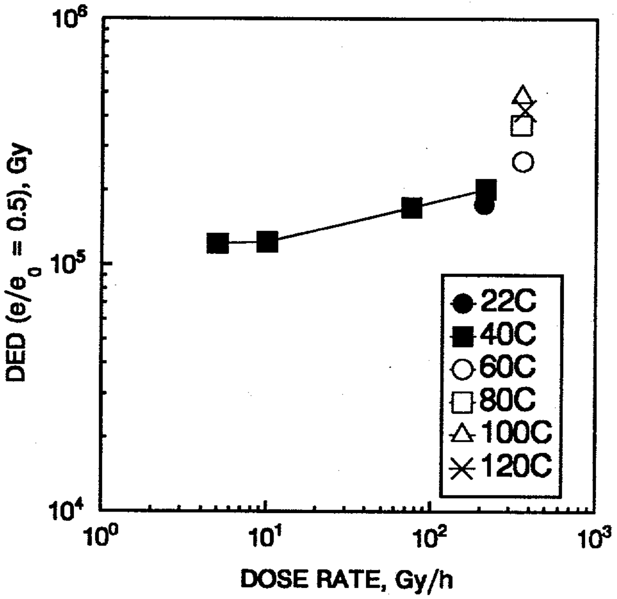

Fig. 21. Dose required for the normalized ultimate tensile elongation (e/e_0) to reach 0.5 versus dose-rate and temperature.

calorimetry, infrared analyses, density, solubility and swelling, to further understand the basis and extent of these complex effects.

ACKNOWLEDGMENT

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