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Loss of Coolant Accident (LOCA) Simulation Tests on Polymers: The Importance of Including Oxygen

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ABSTRACT

Experiments were performed to survey the effects on material degradation of both aging conditions and the oxygen concentration during a LOCA simulation. Changes for a number of commercial materials commonly used as electric cable jackets and insulations in nuclear power plant applications were monitored in terms of weight, mechanical properties, solubility measurements and infrared spectroscopy. For a number of these materials (an EPR insulation, a chloroprene jacket and a PVC jacket), the concentration of oxygen during LOCA simulation was found to be an important parameter. For the first two materials, more degradation occurred when oxygen was present; for PVC, substantially increased swelling occurred as the oxygen concentration was lowered. Aging conditions were also found to have a very substantial influence. In particular, for a number of the materials, lowering the radiation dose rate used for aging led to enhanced degradation after both the aging and the LOCA simulation. The different materials examined showed very different behaviors in terms of the degradation resulting from aging and from LOCA simulation.

ACKNOWLEDGEMENTS

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INTRODUCTION

In order for a safety-related component to be used in a nuclear power plant application, it must pass certain qualification criteria. Various documents (for example references 1 and 2) describe or suggest the tests and data necessary for the qualification. The most severe requirements concern safety components located inside the containment building. These components must be able to withstand a so-called High-Energy Line Break accident, a general term which covers all forms of line breaks including the more familiar Loss-of-Coolant Accidents (LOCA) and Main-Steam-Line Break Accidents. Since the term LOCA is often used to generically describe such accidents, we will use either this term or the term LOCA simulation for the remainder of the paper. Although specific details of the requirements for LOCA simulations differ in different countries, the overall qualification usually includes a sequential procedure encompassing accelerated aging followed by accident simulation.

To account for the aging of components under normal operating conditions, accelerated environments must be used since certain components are installed for the 40 year design life of the nuclear power station. The exact procedures suggested for the aging simulation differ in different countries. In the US, for example, current documents for electric cables allow the dose to be applied at 1 Mrad/hr at room temperature (3). French qualification procedures require dose rates between 0.05 and 0.15 Mrad/hr to be administered at 70°C (2). In an extensive, continuing, aging simulation program at Sandia National Laboratories, we have shown that for numerous cable insulation and jacketing materials, important radiation dose-rate effects exist. In other words, the often used assumption of equal dose, equal damage, is wrong (4,5). The results indicate that for a given total radiation dose, more damage (in terms of mechanical embrittlement) occurs as the dose rate is lowered. There are numerous mechanisms which lead to these dose-rate effects, and every mechanism suggested to date involves oxidation. In fact, for many materials, excluding oxygen during radiation or heat aging leads to greatly decreased material degradation. In other words oxidation effects often dominate degradation.

These observations from aging studies have potentially significant implications when the LOCA situation is considered. At the start of a "real" accident, there will be approximately one atmosphere of air trapped in the containment; oxygen will therefore be available during the accident for reaction with oxidation-sensitive materials. LOCA simulations in the US often occur with significant or total oxygen depletion, since they are routinely performed with steam generation and autoclave apparatus which may effectively sweep out the oxygen at the initiation of the experiment, or remove it in dissolved form in the condensed steam which is withdrawn from the autoclave. Such conditions could lead to a severe underestimate of damage to oxidation sensitive materials. In France, on the other hand, the LOCA simulation includes the requirement that an overpressure of about one atmosphere of air be maintained throughout most of the high temperature, high pressure, steam exposure (2). This arrangement requires a somewhat different experimental setup, wherein both pressure and temperature must be automatically controlled for long periods of time.

To better assess the potential importance of oxygen concentration during LOCA simulation, a joint US-French cooperative research program was initiated. In the first series of experiments described in this paper, various cable insulation and jacketing materials were aged under a number of radiation conditions using Sandia's radiation aging facilities and then subjected to accident simulations at different levels of oxygen overpressure, using the Electricite de France facility at Les Renardiers, France.

EXPERIMENTAL

Materials

The materials studied are described in Table 1 together with the abbreviations which will be used throughout this paper. The first six materials were carefully stripped before aging from modern Class 1E (i.e., qualified for US nuclear power plant safety applications) low-voltage electrical cables obtained from a number of manufacturers. The silicone insulation material is also Class 1E qualified but was obtained from the manufacturer as an extruded tube. The PVC jacketing represented a material which was installed in an older US reactor (6). Finally an EPR compression molded sheet material was prepared at Sandia using a Burke Industries #1483 base compound to which flame retardant and curing ingredients were added (7). All insulation materials were aged as tubes, whereas rectangular samples, approximately 5.5 mm wide by 150 mm long were cut from the jacket materials and the EPR sheet.

Radiation Aging Exposures

The radiation aging was carried out in Sandia's radiation aging facility which has been described previously (8). The facility allows aging to be carried out using various radiation dose rates ranging from approximately 1 Mrad/hr to 1 krad/hr, at any temperature from ambient to 200°C. Dose rates at the facility were obtained using a Victoreen Model 550 Radocon III Integration/Rate Electrometer and thermoluminescent CaF_2 wafers; agreement between the two methods was excellent and the estimated uncertainties in the dose rates are ± 10%.

TABLE 1

MATERIALS

ABBREVIATION	MATERIAL DESCRIPTION
CLPO	Fire retardant radiation crosslinked polyolefin insulation
EPR	Ethylene propylene rubber insulation
CSPE	Chlorosulfonated polyethylene jacket
CP	Chloroprene rubber jacket
CLPE	Chemically crosslinked polyethylene insulation
Tefzel®	Tefzel-280 insulation
Silicone	Dimethyl siloxane rubber insulation
PVC	Polyvinyl chloride jacket
EPR-S	Compression molded ethylene propylene rubber sheet

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During the irradiation, air was circulated through the aging chambers at a high enough rate to ensure at least two complete air exchanges per hour.

Thermal Exposures

The air oven exposures were carried out in Sandia's thermal aging facility which allows control of both the gaseous environment and the gas flow rate during aging. This facility has been previously described (8). For the present experiments, the air flow rates were approximately 30 cc/min for aging chambers of approximately 500 cc volume.

Aging Conditions

Qualification of safety-related equipment in nuclear power plants generally includes radiation exposures to simulate both the aging of the component and the radiation exposure relevant to the accident. For electric cables, The Institute of Electrical and Electronics Engineers recommends an aging radiation dose of 50 Mrad at a dose rate not greater than 1 Mrad/hr (3). In practice, this is often combined with the accident radiation dose which typically runs 150 Mrad at 1 Mrad/hr. French documents (2) recommend an aging dose of 25 Mrad at 50-150 krad/hr and an accident dose of 60 Mrad at 150-750 krad/hr, both carried out at 70°C.

For the present experiments, the aging and accident radiation exposures were combined into a single radiation exposure, which for convenience we refer to as an aging exposure. To assess the effect of varying this aging exposure, three different aging conditions were used. The detailed conditions for all samples are listed in Table 2, where for convenience they are divided into 4 lots: lot 1 represents unaged materials, lot 2 represents materials exposed to approximately 21 Mrad at 880 krad/hr, lot 3 represents materials exposed to approximately 45 Mrad at 880 krad/hr and lot 4 represents materials exposed to approximately 23 Mrad at 24 krad/hr. By comparing the responses after LOCA simulation for the various lots of a material, we are able to determine the effect of both radiation dose and dose rate on material degradation. For various reasons, not all materials were exposed to all three radiation aging conditions. For example, lack of sufficient EPR sheet material limited its use to lots 1 and 2; Tefzel®, on the other hand, was not exposed in lot 3 due to its greatly reduced mechanical properties after such an exposure. Since the CLPE material became available after the aging exposures had started, it was exposed to only 15 Mrad in lot 4.

It is clear from Table 2, that the three conditions used for this series of experiments involve less radiation than the conditions suggested by the IEEE and French documents. In addition, the thermal aging exposures suggested in these documents were not included in our aging simulations. Thus the aging conditions used for the current experiments might be considered relatively mild, but still serve the scoping function intended for this study.

TABLE 2

AGING CONDITIONS

LOT NUMBER 1	"Unaged Materials"
LOT NUMBER 2	"21 Mrad at 880 krad/hr"
PVC 828 krad/hr CSPE 880 krad/hr EPR 909 krad/hr CLPO 909 krad/hr CLPE 909 krad/hr Silicone 875 krad/hr Tefzel 810 krad/hr EPR-S 927 krad/hr CP 834 krad/hr	for 24 hr at 44°C
LOT NUMBER 3	"45 Mrad at 880 krad/hr
PVC 828 krad/hr CSPE 880 krad/hr EPR 927 krad/hr CLPO 909 krad/hr CLPE 909 krad/hr	for 50.5 hr at 44°C
LOT NUMBER 4	"23 Mrad at 24 krad/hr"
PVC 23.7 krad/hr CSPE 25.2 krad/hr EPR 26.5 krad/hr CLPO 24 krad/hr Silicone 20.8 krad/hr Tefzel 23.2 krad/hr	for 958 hr at 26°C
CLPE 24 krad/hr	for 622 hr at 26°C (15 Mrad)

LOCA Simulation Tests

The LOCA simulations were carried out at EDF, Les Renardieres, in a versatile facility that was designed and has been used primarily for LOCA simulations of various nuclear power plant safety-related equipment. The facility shown schematically in Fig. 1 includes a 0.2 m^3 vessel in which the test samples are placed. For the current experiments, the various materials were positioned vertically in stainless steel baskets with stainless steel wires used to separate different types of materials. A regulated steam generator supplies nine bars of saturated steam to two solenoid valves; one valve which is located on a large pipe leading to the test vessel, allows quick temperature and pressure rise times to be achieved at the beginning of a test; the second solenoid valve, in a small pipe connected in parallel, is used for fine control and regulation. The overpressurization gas (normally air) enters the test vessel through another inlet, which is also controlled by a solenoid valve. Outlet solenoid valves in the upper part of the vessel allow for gas exhaust during and at the end of the experiment. Connected to the lower part of the vessel are a condensate exhaust solenoid which is used to regulate the condensate level and a manual leak valve. Various pressure sensors and thermocouples are placed in the chamber in order to measure pressures and temperatures. The entire test sequence is automatically controlled by a Hewlett Packard HP 9825A computer, including the rapid temperature and pressure ramp at the beginning of the test and any chosen

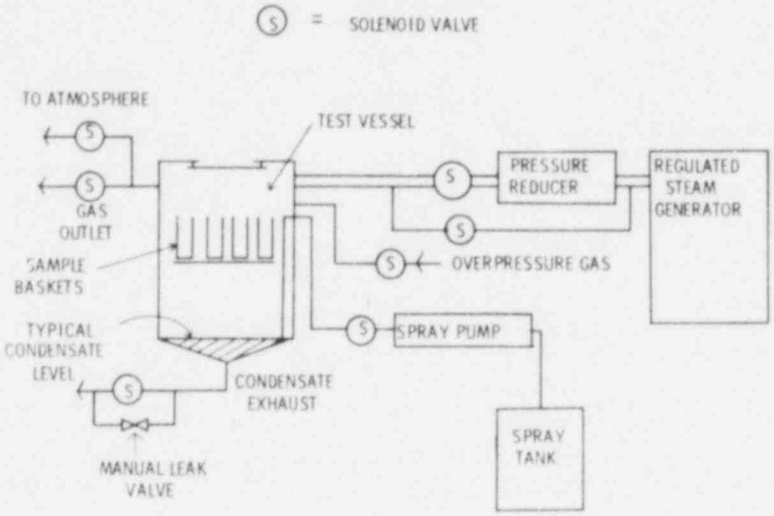

Figure 1. Schematic diagram of EDF-DER LOCA simulation facility.

temperature and pressure conditions thereafter. The computer is used to control the various solenoid valves to keep the temperature, pressure and condensate level within specified limits of the chosen profile. Various safeguards are included to protect the system from thermal or pressure runaway.

Typical LOCA test profiles used in the US (1) and France (2) are different; both involve relatively complicated time-dependent combinations of temperature, steam pressure and (for France) air overpressure. The profiles start at high temperature and pressure values (typically 171°C, 8 x 10^5 Pa steam in the US; 156°C, 5.5 x 10^5 Pa steam in France) and reach much lower values at the end of 4 days (121°C, 2 x 10^5 Pa steam in the US; 73°C, 4 x 10^4 Pa steam, 1.2 x 10^5 Pa air in France). Since the experiments described in this paper represent a first attempt to screen for the possible influences on materials of aging conditions and oxygen concentration during LOCA simulation tests, a greatly simplified constant test profile was chosen as a compromise representation of the more complicated profiles referred to above. The conditions were 96 hrs at constant conditions of 145°C, 4.1 x 10^5 Pa steam and 2 x 10^5 Pa overpressure gas. Three LOCA simulations were run with the only planned difference being the composition of the overpressure gas. In July of 1981, tests using nitrogen and air for the overpressure gas were run. In December of 1981, a test was run using an overpressure gas containing 10.5% ± 1% oxygen.

The starting conditions for all tests were 50 ± 10°C and atmospheric pressure of the gas used for overpressure. Within 10 sec, 144°C and 6 x 10^5 Pa total pressure was reached. During the 96 hr tests, the temperature in the middle of the sample region was maintained at approximately 145°C ± 3°C; there was an estimated 2-3°C gradient along the sample lengths. The pressure was maintained at 6.1 ± 0.2 x 10^5 Pa which at 145°C corresponds to a gas overpressure of approximately 2 x 10^5 Pa. A leak, manually introduced at the bottom of the test vessel, resulted in an average injected gas flow of 1.5 m^3/hr (measured at 2 x 10^5 Pa). With the test vessel volume of 0.2 m^3, a gas "exchange" took about 10 min. At the end of the 96 hrs, the inlet valves were closed and the exhaust valves opened (first the condensate valve then the atmospheric equilibrium valve); this caused the pressure to quickly drop to atmospheric. The temperature returned to ambient in a few hours.

Tensile Tests

For each experimental condition (i.e., for each material type aged under specified conditions and then subjected to a specific LOCA simulation), 2 or 3 samples were tensile tested at Sandia National Laboratories. An Instron Table Model 1020 Machine was used at an ambient temperature of 23 ± 1°C. Samples were gripped using pneumatic jaws with an air pressure of approximately 3 x 10^5 Pa. Initial jaw separation was 51 mm and samples were strained at 127 mm/min; the strain was monitored with an Instron electrical tape extensometer clamped to the sample. One additional sample was tensile tested at the CEA-ORIS laboratory using a Zwick and Co. KG Type 7025/3 Tensile Machine at 20.5°C ± 1°C. Samples were gripped using mechanically controlled clamps with a force feedback feature. Initial jaw separation was typically 30 mm and samples were strained at 50 mm/min; the strain was also monitored with an extensometer. Since the tensile testing conditions in the two laboratories differed (e.g., strain rate, test temperature, test equipment) the tensile results were somewhat different, as shown in Table 3 for the unaged materials. However, as is shown below, when comparisons are made between the normalized elongation (e/e_o) and tensile strength (T/T_o) value (i.e., the ultimate tensile property divided by the value for unaged material), agreement between the Sandia and CEA results is usually excellent.

TABLE 3

TENSILE PROPERTIES OF UNAGED MATERIALS

	SANDIA		CEA/ORIS	
MATERIAL	e_o (%)	T_o (MPa)	e_o (%)	T_o (MPa)
CLPE	350	16.2	313	17.6
TEFZEL®	290	54	245	55
SILICONE	430	7.6	307	5.3
EPR-S	360	12.2	387	13.4
CLPO	240	13.7	199	14.3
CSPE	300	24.4	239	17.4
PVC	300	20	209	18
CP	185	11	181	9
EPR	340	6.9	300	8.6

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Infrared Spectra

Spectra were obtained using potassium bromide pellets containing polymer samples that had been ground-up at -196 C. A Nicolet Model 7199 Fourier Transform Infrared spectrometer was employed.

Swelling Measurements

Samples of initial weight, W_i, ranging from 0.3 - 1.0 g were extracted with tetrahydrofuran at 50°C in a Soxhlet extractor for 24 hrs. After quickly removing excess solvent from the surface and capping the samples in a bottle to retard evaporation of solvent, the samples were weighed, yielding W_S; they were then dried to constant weight in a vacuum oven, yielding W_d. The weight swelling ratio (WSR) was then calculated as

$$WSR = \frac{W_S - W_d}{W_d}$$

and the soluble percentage was obtained from $(W_i - W_d)/W_i$.

Oxygen Gas Analysis

The gas mixture used for the overpressure gas in the December experiment which was specified to be 10% oxygen and 90% nitrogen, was analyzed by the supplier, AGA, and found to contain 9.75% oxygen. Additional samples were analyzed by CEA-ORIS and Sandia using gas chromatography techniques. The results from these labs were 10.2 ± 0.6% and 11.5% respectively. Thus, we estimate the oxygen concentration to be 10.5 ± 1%.

RESULTS AND DICUSSION

Weight Changes and Tensile Properties

Each lot and type of sample was weighed before LOCA simulations and as a function of time after removal from these tests. The approximate percentage changes determined at various times after the end of each LOCA simulation are listed in Table 4. For a number of the materials, in particular

TABLE 4
WEIGHT CHANGE DUE TO LOCA SIMULATION VS. TIME AFTER LOCA SIMULATION

MATERIAL	TEST % O_2	LOT	PERCENT WEIGHT CHANGE AFTER		
			6 HR	1500 HR	5000 HR
CLPE	0	1	0	0	0
		2	0	0	0
		3	0	0	0
		4	0	0	0
	10	1	0	0	
		2	+1	0	
		3	+1	0	
		4	+1	0	
	21	1	0	0	0
		2	+2	0	0
		3	+2	0	0
		4	+2	0	0
TEFZEL®	0	1	0	0	0
		2	0	0	0
		4	0	0	0
	10	1	0	0	
		2	-1	0	
		4	-1	0	
	21	1	0	0	0
		2	0	0	0
		4	0	0	0
SILICONE	0	1	-1	-3	-3
		2	+1	-4	-4
		4	+2	-4	-4
	10	1	0	-2	
		2	+1	-2	
		4	0	-3	
	21	1	-1	-1	-1
		2	-1	-1	-1
		4	-1	-2	-2
EPR-S	0	1	+1	0	-1
		2	+1	0	0
	21	1	0	0	0
		2	+1	0	0

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TABLE 4 - Continued

MATERIAL	TEST % O_2	LOT	PERCENT WEIGHT CHANGE AFTER		
			6 HR	1500 HR	5000 HR
CLPO	0	1	+2	0	0
		2	+2	-2	-2
		3	+10	0	-1
		4	+15	+3	-1
	10	1	+1	-1	
		2	+5	-1	
		3	+14	-1	
		4	+18	-1	
	21	1	+1	+2	+2
		2	+1	0	0
		3	+8	-1	-1
		4	+10	+1	0
CSPE	0	1	+16	-2	-2
		2	+12	-2	-2
		3	+17	-2	-2
		4	+18	-2	-2
	10	1	+12	0	
		2	+29	+1	
		3	+31	+3	
		4	+29	+2	
	21	1	+9	+1	+1
		2	+11	+1	0
		3	+14	0	-1
		4	+16	-1	-2
PVC	0	1	+93	+14	-4
		2	+220	+50	-1
		3	+347	+46	-4
		4	+330	+62	-7
	10	1	+53	-2	
		2	+150	-4	
		3	+360	-3	
		4	+230	-4	
	21	1	0	-1	-1
		2	+18	0	-2
		3	+39	+4	-2
		4	+35	+4	-3

TABLE 4 - Continued

MATERIAL	TEST % O_2	LOT	PERCENT WEIGHT CHANGE AFTER 6 HR	1500 HR	5000 HR
CP	0	1	+53	+15	-3
		2	+56	+19	-3
	10	1	+150	0	
		2	+84	-5	
	21	1	+93	-1	-2
		2	+71	0	-3
EPR	0	1	+5	-1	-1
		2	+8	-1	-2
		3	+19	+1	-2
		4	+23	+2	-2
	10	1	+3	-1	
		2	+9	-3	
		3	+7	-4	
		4	+10	-2	
	21	1	+4	+1	0
		2	+12	+6	+4
		3	+9	+1	0
		4	+10	-3	-4

CLPE, silicone, Tefzel® and EPR sheets, little if any changes in weight were caused by the tests. For the other four materials, moderate to large increases in weight occurred due to swelling by the steam environment. These weight changes were accompanied by large increases in physical dimensions, as evidenced for example in Fig. 2, which compares PVC samples before and immediately after the nitrogen test. As shown in Table 4, the water picked up by such samples slowly diffuses out of the sample, until after a few thousand hours, equilibrium weights are reached which are usually close to the original weights. Water dissolved in these polymers acts as a plasticizer which can affect the tensile properites. Since the tensile properties are being used in the present study as one monitor of chemical changes brought about by the various test profiles, the tensile properties were measured at a minimum of 1500 hours after the completion of the LOCA simulation. Comparison of results taken approximately 1500 hrs after a test with those taken approximately 5000 hrs after the test indicates that no significant changes in tensile properties occurred after 1500 hrs. As indicated in Fig. 2,

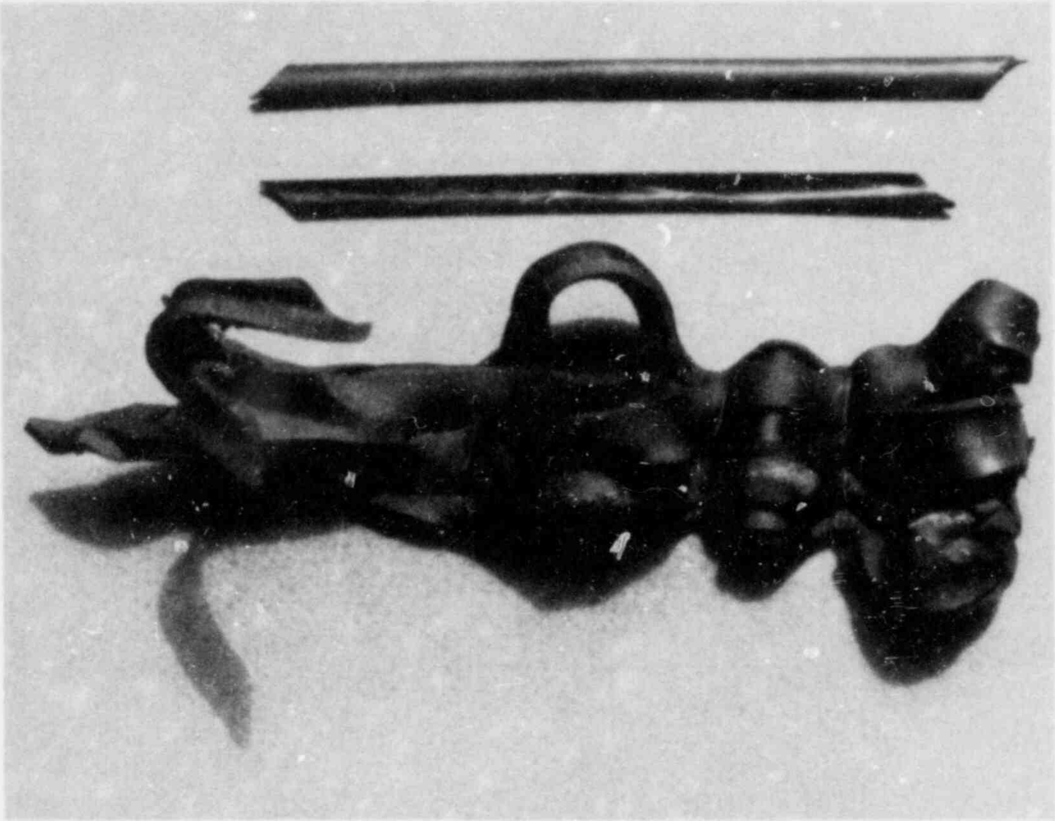
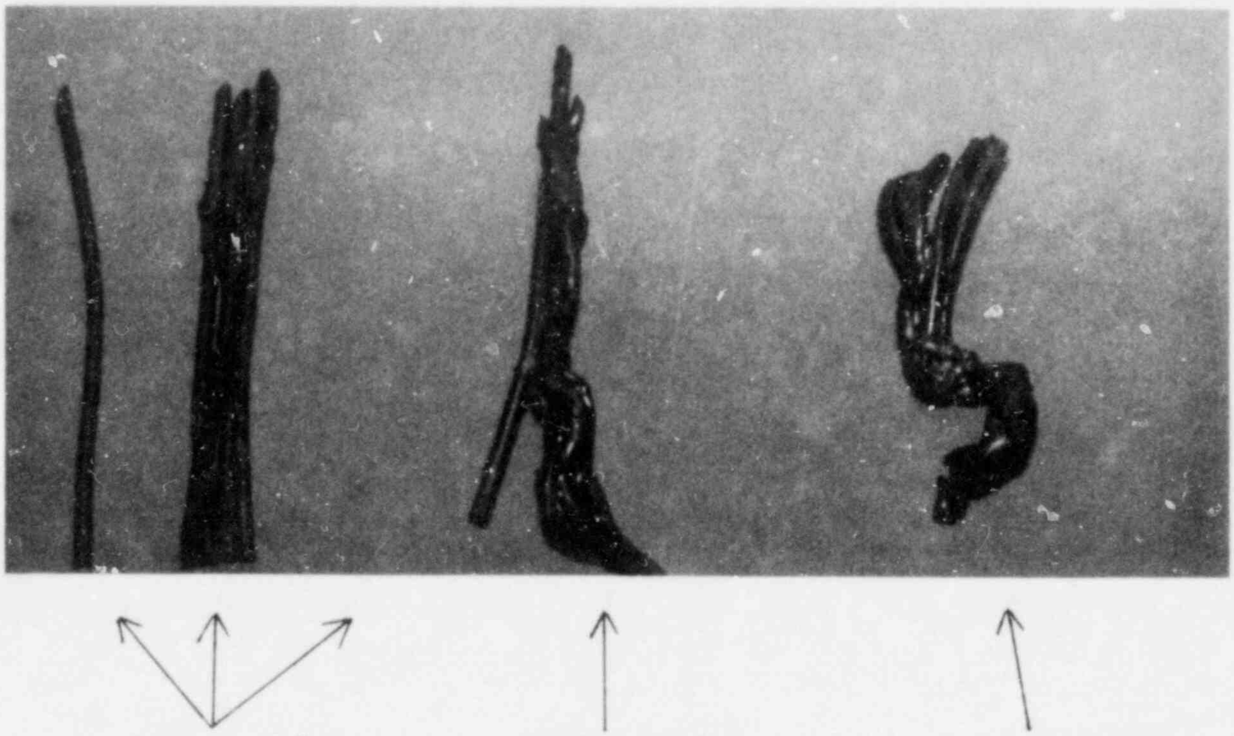


Figure 2. Photograph comparing PVC material aged for 958 hr at 23.7 krad/hr before and after LOCA simulation in nitrogen.

many of the samples which swelled significantly also tended to buckle and distort during the tests. Figure 3 shows that certain badly degraded EPR insulation samples tended to melt and partially fuse together. There were also indications of inhomogeneous degradation along individual samples with the damage slightly more severe at one end, (see Fig. 3) possibly caused by the small thermal gradients in the sample region. Such factors tended to increase the scatter in the tensile results for the PVC and EPR insulation materials. The tensile results for all the materials studied are summarized in Figs. 4-12, where the average values of the Sandia experiments (circles) and the CEA data (triangles) are both indicated for each material under its combination of aging and LOCA-simulation conditions. Where repeated runs were made, scatter in e/e_o and T/T_o for the PVC and EPR insulation materials was typically found to be $\pm$ 0.1 and $\pm$ 0.05 respectively; for the other materials it tended to be somewhat smaller. For



909 krad/hr
for 24 hr

927 krad/hr
for 50.5 hr

26.5 krad/hr
for 958 hr

Figure 3. Photograph showing EPR insulation materials after the LOCA simulation containing air. The 6 samples indicated on the left were aged for 24 hr at 909 krad/hr. The center samples were aged for 50.5 hr at 927 krad/hr. The group on the right were aged for 958 hr at 26.5 krad/hr.

Figs. 4-12, the numbers 1, 2, 3, and 4 listed on the abscissa refer to the lots listed in Table 2. BEFORE refers to the results for the indicated lot before LOCA simulation and L-0, L-10, and L-21 refer to the results after the nitrogen, the 10% oxygen and the air LOCA simulations respectively. (In a few instances, no samples were available for testing at CEA.) It is clear from Figs. 4-12, that on the basis of normalized tensile property changes, no significant differences exist between the results from the two laboratories, even though different testing equipment and conditions were utilized. The tensile results and weight-change results shown in Table 4 will now be used to discuss the materials individually.

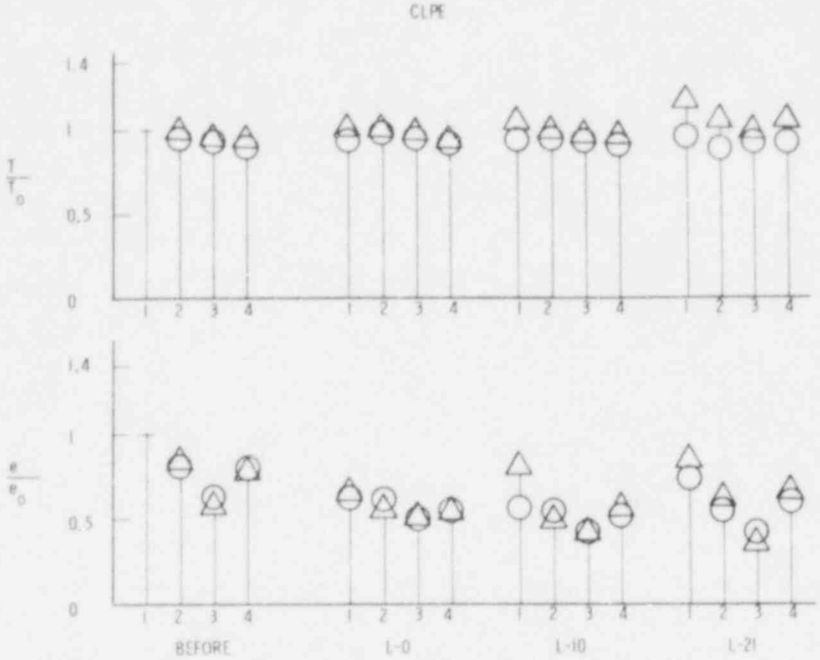


Figure 4. Tensile property results for the CLPE material. The tensile strength, T, divided by its unaged value, T_o (Table 3) and the ultimate tensile elongation, e, divided by the unaged value, e_o (Table 3) are plotted after the aging exposures and after the 3 LOCA simulations. The numbers 1, 2, 3, and 4 refer to the aging condition lot numbers (Table 2). BEFORE refers to the results before LOCA simulation, L-0, L-10, and L-21 refer to the results after the LOCA simulations containing nitrogen, 10% oxygen and air respectively. The circles give the results measured at Sandia, the triangles denote the results obtained at CEA-ORIS-STBR.

CLPE

No significant weight changes were observed for this material. Tensile property changes are plotted in Fig. 4. Within the experimental uncertainties, no significant effect of oxygen concentration during LOCA simulation is noted. Tensile strength values were unaffected; whereas tensile elongation values dropped slightly after the LOCA simulation, with the amount of change being essentially independent of the dose-rate and dose used for the aging sequence. In fact, the results for the 15 Mrad, low dose-rate aging exposures and the 21 Mrad, high dose-rate aging exposure were quite similar. This correlates well with the minor radiation aging dose-rate effects found recently for this material (9).

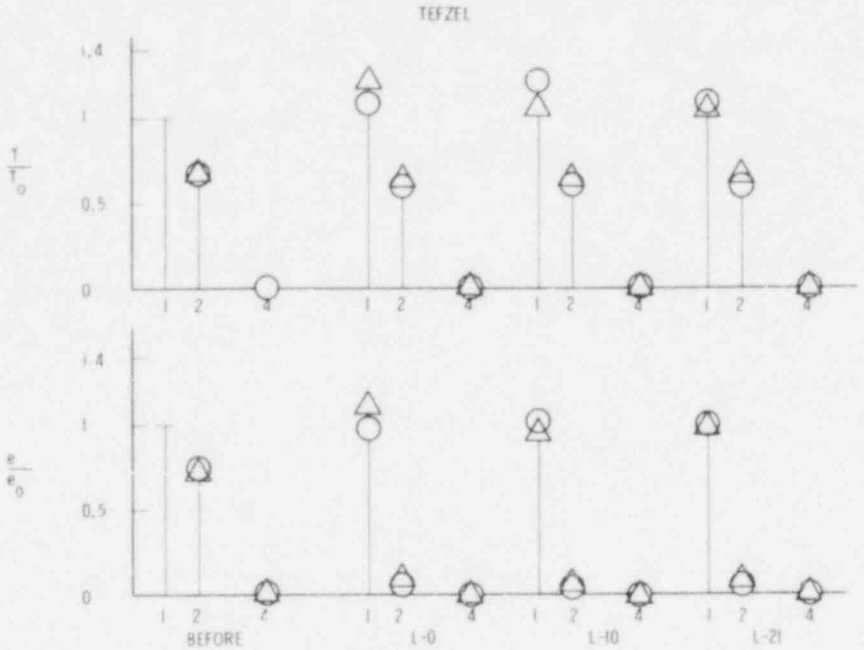


Figure 5. Tensile property results for Tefzel. See the caption for Fig. 4 for explanation.

Tefzel®

No significant weight changes were observed for Tefzel.® As shown in Fig. 5, the oxygen concentration during LOCA simulation had no perceptible effect on the tensile properties. As might be anticipated from Tefzel's® excellent thermal properties, the LOCA simulations did not effect the properties of unaged samples. The effects of radiation aging were much more dramatic. For the samples aged to 19 Mrad at high dose-rate, e/e_o dropped to 0.72 after the aging; after LOCA simulation, it dropped more than an order of magnitude further to around 0.05, although the tensile strength remained essentially unchanged. The samples aged to 22 Mrad at low dose rates were so brittle after the LOCA simulations that they often broke just from handling. Again this data correlates well with the radiation aging results found for this material, where both low radiation tolerance and indications of dose-rate effects were found (9).

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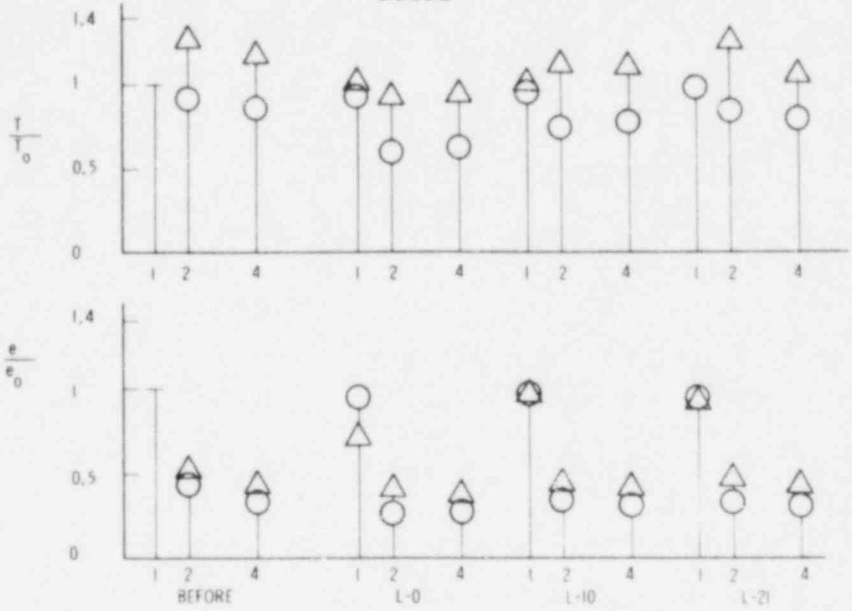

Figure 6. Tensile property results for silicone. See the caption for Fig. 4 for explanation.

Silicone

For the silicone material, weight changes are not significant. Tensile property changes are plotted in Fig. 6. The major change in the tensile elongation in this material occurred as a result of the aging rather than the LOCA simulations. There are indications of a slight effect of the oxygen concentration with the tensile properties after the nitrogen test slightly lower, but the effects are not large. The results for the high- and low-dose-rate, radiation-aged samples are similar, as might be expected from the minor dose-rate effects previously found for this material (9). Although T/T_o values measured by CEA averaged approximately 30% higher than the Sandia measured values, the trends were identical. This difference may be due to the 40% higher T_o value measured at Sandia (Table 3).

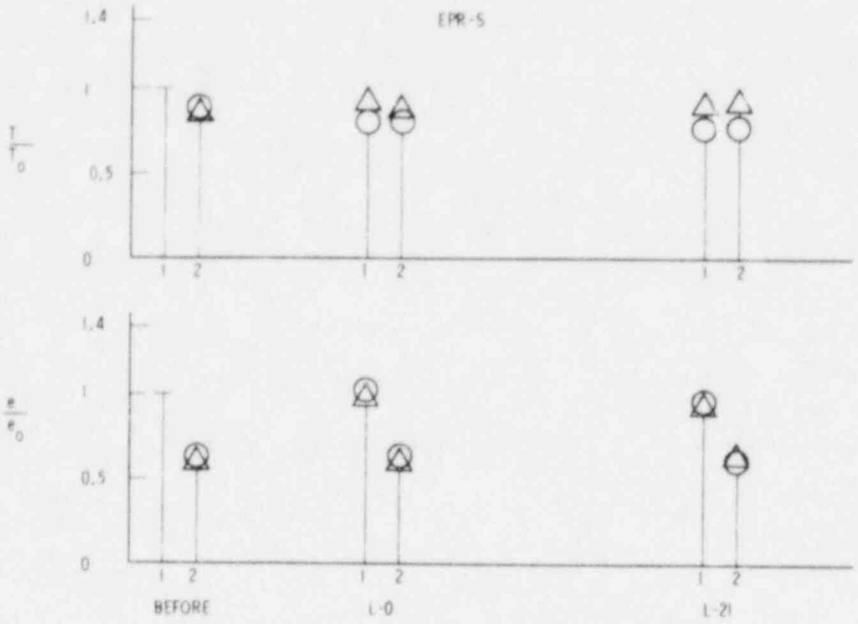

Figure 7. Tensile property results for EPR-S. See the caption for Fig. 4 for explanation.

EPR Sheet Material

For this material, no samples were available for the 10% oxygen test. The other LOCA simulations caused no significant weight changes. The results, plotted in Fig. 7, show that the oxygen concentration is not an important parameter and that the LOCA simulations had little effect on the tensile properties. Although radiation aging was carried out at only high dose rates, evidence that aging dose-rate effects are minimal for this material (7) allows us to predict that no substantial differences would be expected for lower dose-rate aging exposures.

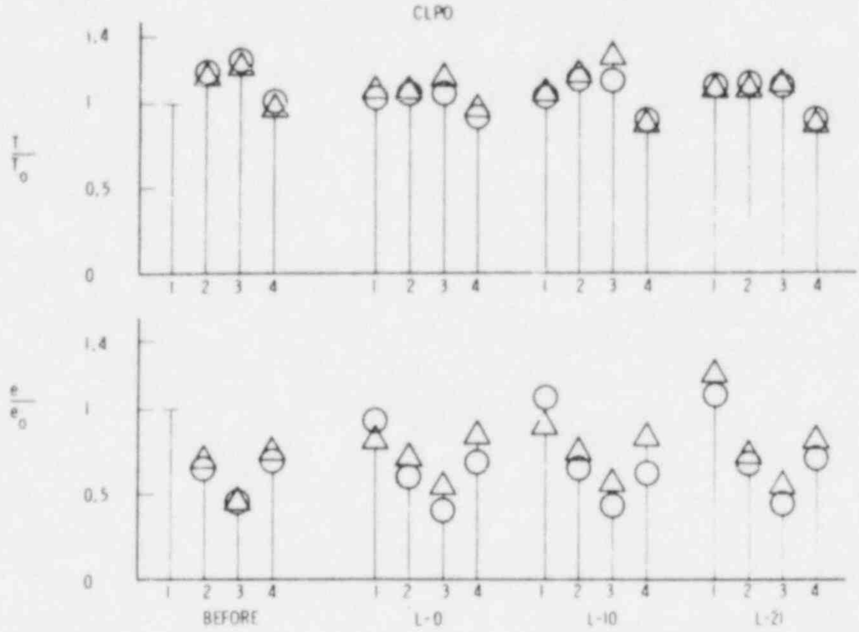

Figure 8. Tensile property results for CLPO. See the caption for Fig. 4 for explanation.

CLPO

For this material, moderate weight gains occurred as seen in Table 4. The tensile results are shown in Fig. 8. Radiation aging resulted in a significant decrease in tensile elongation of the CLPO. In contrast, the results indicate that the LOCA simulation itself had no major effect on the material properties, regardless of which oxygen concentration was used in the test. The tensile strength results show the existence of small radiation dose-rate effects after radiation aging. This is in agreement with our earlier dose-rate data for this material (4).

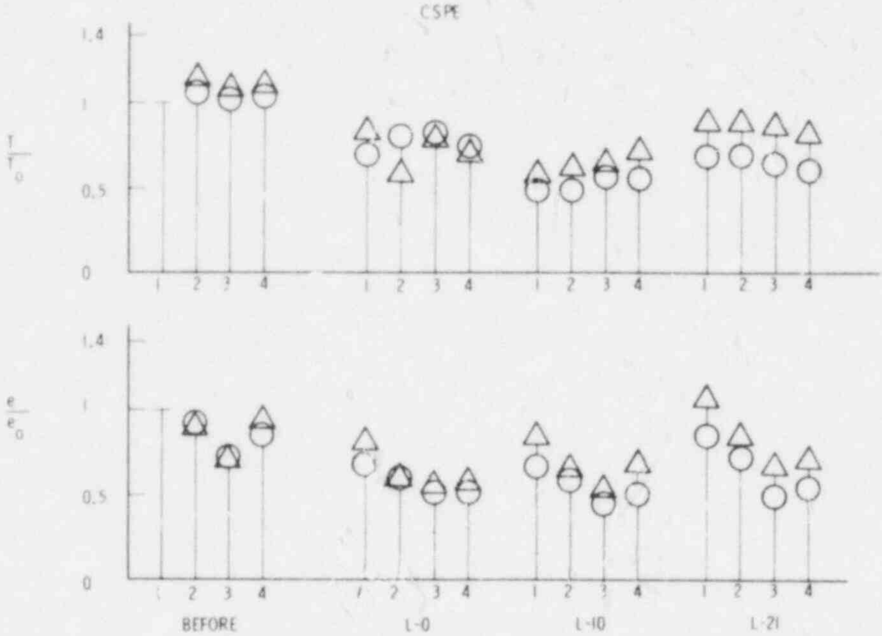

Figure 9. Tensile property results for CSPE. See the caption for Fig. 4 for explanation.

CSPE

Table 4 gives the weight change results. The tensile properties of the CSPE samples are plotted in Fig. 9. It should be noted that for CSPE and a number of other materials, the swelling and tensile results sometimes go through slight maxima or minima as the oxygen concentration in the LOCA simulation is raised, perhaps indicating some unknown variable is slightly influencing the results. An attempt was made to use identical conditions (besides oxygen concentration) for the three tests, but since the 10% oxygen experiment was run 5 months after the air and nitrogen experiments, subtle differences in experimental conditions might be anticipated. In any case, even though this effect appears to be larger in CSPE than in any of the other materials studied, no important trends with oxygen concentration can be established. The effect of the aging dose rate is also minor, which again correlates well with the minor dose-rate effects found for the aging of this material(4).

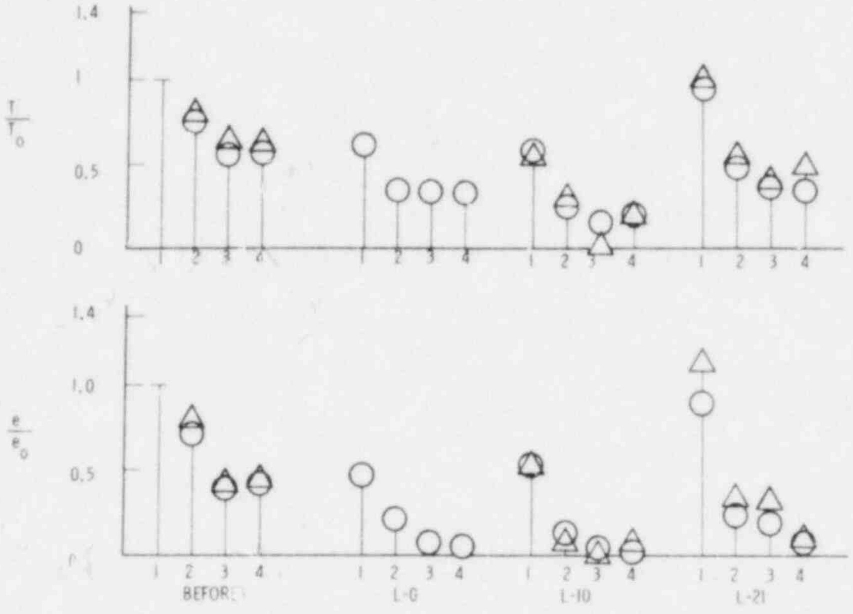

Figure 10. Tensile property results for PVC. See the caption for Fig. 4 for explanation.

PVC

Weight changes and tensile properties of this material are shown in Table 4 and Fig. 10, respectively. The weight-change data indicates that substantial differences occurred as the oxygen concentration during the LOCA simulations increased. Depending on the aging conditions, weight increases ranging from 100-350% were measured after the nitrogen test, compared to 0-40% after the air test. These differences in water sorption are reflected in dramatic differences in swelling caused by the various LOCA simulations. Figure 2 shows the effect that the nitrogen test had on the physical dimensions of samples aged to 23 Mrad at 24 krad/hr. During the LOCA simulation, the samples were partially constrained by the sample holders; the tremendous expansion of sample dimensions due to the swelling mechanism resulted in distorted ribbon-like samples. As evidenced in Fig. 2, an approximate doubling of the physical dimensions of these samples occurred, not surprising in

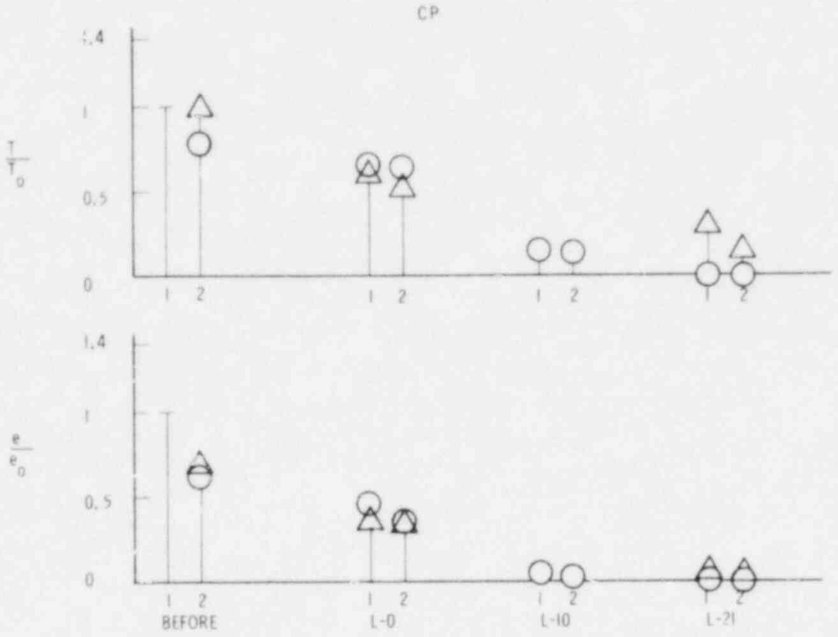

Figure 11. Tensile property results for CP. See the caption for Fig. 4 for explanation.

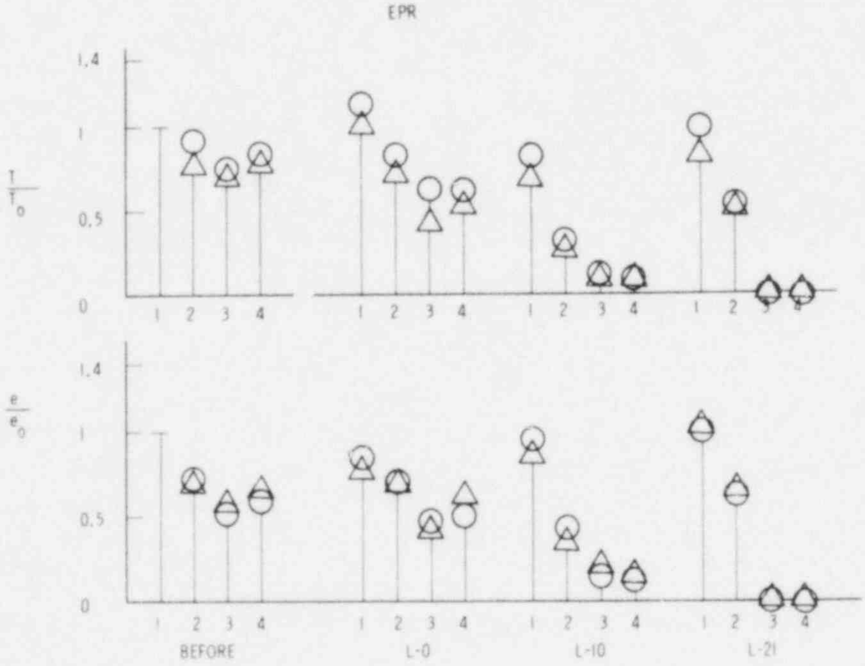

Figure 12. Tensile property results for EPR. See the caption for Fig. 4 for explanation.

light of their 330% increase in weight. Figure 13 compares the results for samples aged to 42 Mrad at 828 krad/hr. The swelling which occurred in the nitrogen test is clearly much greater than the swelling caused in the air test. After a few thousand hours, the weights of the swelled samples dropped back close to the weights before the LOCA simulations, indicating that most of the absorbed water was lost by diffusion. This indicates that the water was not chemically incorporated into the polymer. It should be noted however, that the physical dimensions of the polymer did not return to original as the weight recovered. This "set" of the PVC material makes comparisons of tensile property changes somewhat more difficult, since many of the nitrogen and 10% oxygen tested samples are longer than the air tested samples at the start of tensile tests.

For convenience, the length after LOCA simulation was taken as the reference initial length, L_o, in the calculation of the tensile elongation. If the sample length before LOCA simulation was chosen as L_o, then the e/e_o values after the nitrogen and 10% oxygen LOCA simulations would increase a greater relative amount than the e/e_o values after the air LOCA simulation.

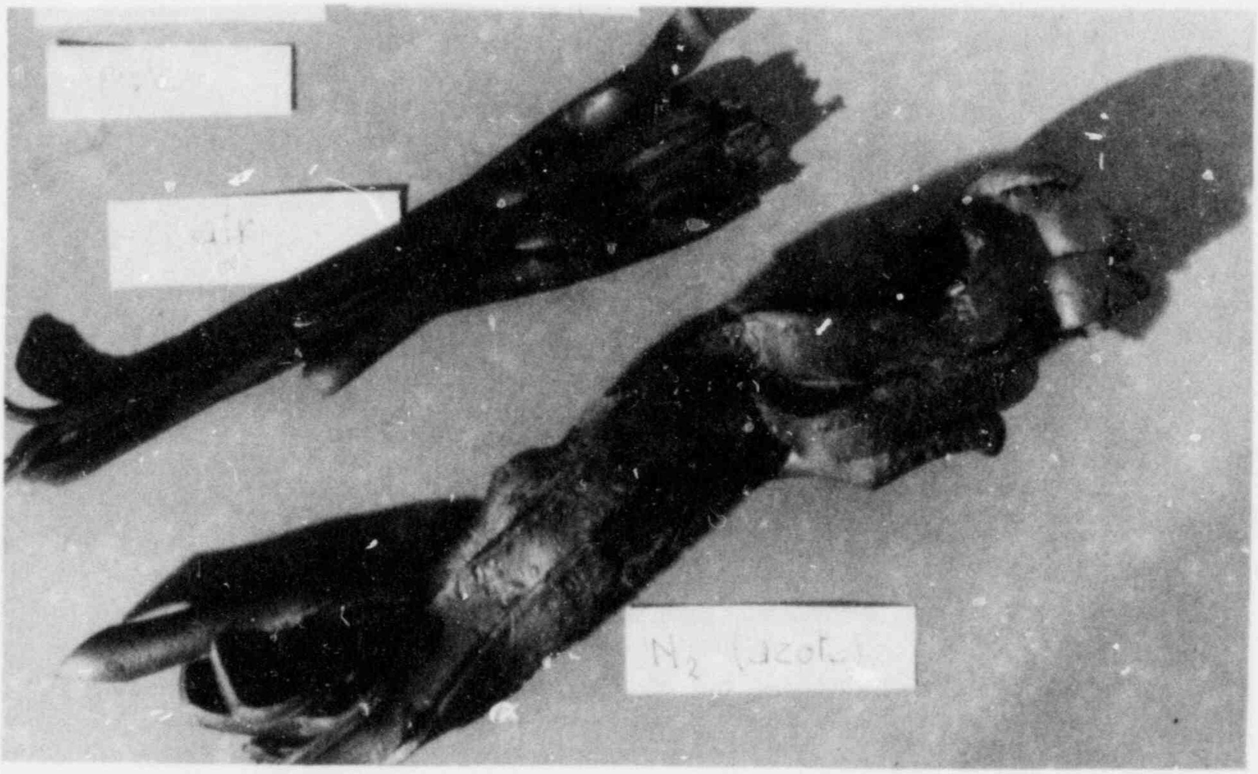

Figure 13. Photograph comparing PVC material which was aged for 50.5 hr at 828 krad/hr after the LOCA simulations containing nitrogen and air.

Figures 2 and 13 also show that certain of the samples tended to partially fuse together during LOCA simulations. Although individual samples could usually be separated for tensile testing, small flaws introduced by the separation could also influence the tensile properties. For the unaged samples, the tensile results shown in Fig. 10 indicate perhaps a small trend towards less deterioration as the oxygen concentration was increased. But given the uncertainties caused by the effects of swelling (ribboning, set, fusion and distortion), definitive conclusions about oxygen effects on mechanical property changes cannot be ascertained from the results. Certain of the LOCA simulations caused substantial drops in tensile properties, especially for the samples aged in radiation environments. In addition, these samples showed the expected trend based on the large dose-rate effects found for the aging of PVC in previous work (5); that is, the decrease in the tensile elongation for the 23 Mrad, low-dose-rate samples is similar to that of the 42 Mrad, high-dose-rate samples. Solubility and swelling experiments in THF solvent were carried out on the 23 Mrad, low-dose-rate aged samples after the three LOCA simulations. Only 28% of the nitrogen-exposed sample was soluble, compared to 24% soluble for the 10% oxygen-exposed sample and 56% for the air-exposed sample. For the material which remained insoluble, the swelling ratios were 6.9 nitrogen, 7.4 (10% oxygen) and 0.64 (air), which is reasonably consistent with the steam swelling results in Table 4. The THF results again show the large effect of oxygen concentration on this PVC material. The fact that PVC may sustain more damage in a nitrogen (or inert) LOCA simulation underlines the importance of knowing the oxygen content in a real LOCA, since the potential disappearance of oxygen through reaction with various materials during a LOCA could lead, in the later stages of a LOCA, to an enhanced degradation rate in those materials having an inverse degradation dependence on oxygen concentration.

Chloroprene

This material swelled and absorbed substantial amounts of water during the LOCA simulations. Also, the presence of oxygen had a pronounced influence on its tensile properties as shown in Fig. 11. Only unaged samples and samples aged to 20 Mrad at high dose-rates were exposed to the LOCA simulations. In both cases, the nitrogen test caused only moderate decreases in tensile properties, but the addition of oxygen led to greatly enhanced degradation.

EPR Insulation

The response of certain of the EPR samples also showed a pronounced dependence on the oxygen concentration during LOCA simulation, as evidenced in Fig. 12. The various LOCA simulations barely affected the tensile properties of unaged samples. The samples aged to 22 Mrad at a high dose rate are moderately influenced by the LOCA simulations and show slightly greater degradation at higher oxygen concentrations. The samples aged to 25 Mrad at low dose rate and 47 Mrad at high dose rate show a much larger dependence on oxygen concentration, with the samples tested in air so degraded that they melted (Fig. 3). The observation that the 25 Mrad, low-dose-rate samples show degradation behavior similar to the 47 Mrad, high-dose-rate samples is consistent with the radiation aging dose-rate effects found for this material in an earlier study (4). THF extractions on the 47 Mrad high-dose-rate samples after the three tests gives solubilities of 17% (nitrogen), 29% (10% oxygen) and 39% (air), consistent with the the expectation of increased oxidative scission as the oxygen concentration increases. The insoluble material from the nitrogen test has a swell ratio of 1.74; whereas, the swell ratios of the insoluble fractions LOCA -tested in the presence of oxygen cannot be measured since they are powdery residues, consistent with increased oxidative scission. One final piece of evidence for increased oxidative scission comes from infrared spectroscopy; the carbonyl peak at 1720 cm^{-1} is much stronger for the air-exposed sample, as shown in Fig. 14. Carbonyl species (esters, acids, ketones, aldehydes) are among the end products of oxidative degradation pathways and their presence is viewed as confirmation of the oxidation process. Further differences between the air and nitrogen samples are indicated in the 1500 - 1550 cm^{-1} region and at 1622 cm^{-1}, where the latter would seem to indicate the formation of more double bonded species during the LOCA simulation containing air.

A Possible Test to Screen for LOCA Sensitivity

Since LOCA simulations require highly specialized and expensive experiments, it would be useful to develop some simple screening tests to determine material sensitivity to a LOCA environment. Such tests would not replace LOCA simulations but could be extremely useful for initial component design by allowing the elimination of materials likely to be severely degraded by the LOCA environment. Since the temperature, and in certain cases, the oxygen concentration, may be the most significant stresses during a LOCA, one possible screening test involves the use of air oven aging as a replacement for the LOCA environment. Coupled with radiation aging, this screening technique would be similar to sequential aging experiments

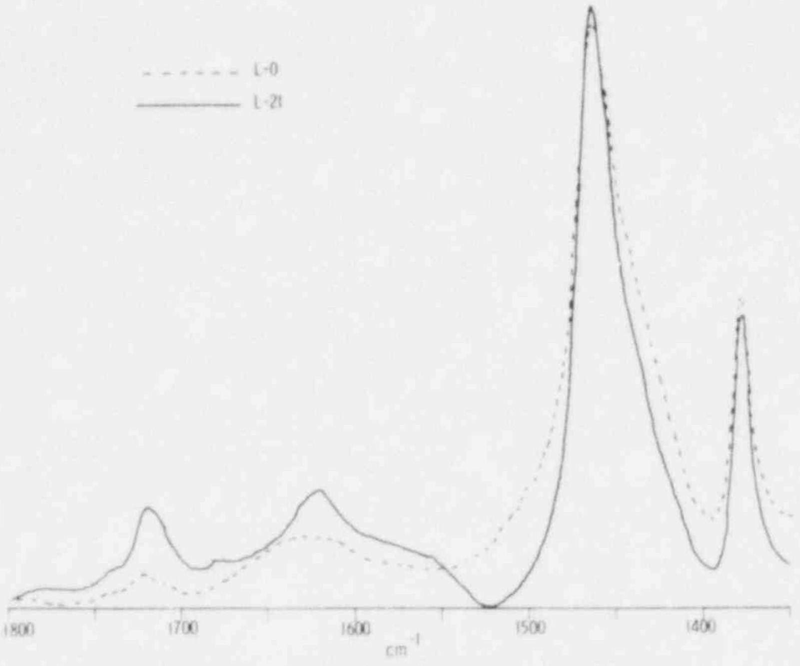

Figure 14. Infrared spectra after LOCA simulation of EPR insulation material which had been aged for 50.5 hr at 927 krad/hr. The solid spectra shows the result after the LOCA simulation with air whereas the dashed spectra is after the LOCA simulation with nitrogen.

of radiation followed by elevated temperature exposures. In such sequential aging experiments, we have previously documented the fact that the thermal exposure can bring about rapid degradation for certain polymeric materials which have been presensitized by previous radiation exposure (5).

Using Sandia's thermal aging facility which allows control of the gaseous environment, a 96 hr thermal exposure at 145°C was used on various unaged and radiation aged samples in a flowing air environment. After the thermal exposures, tensile tests were carried out and the resulting e/e_o values are summarized in Table 5 in the column labelled "AIR OVEN". These results are compared in the table to the results for similarly aged samples after exposure [illegible] the 10% oxygen LOCA simulation ("L-10" in the table). The [illegible]0 test was chosen since at 2 x 10^5 Pa total pressure, [illegible]ygen corresponds to approximately 2 x 10^4 Pa oxygen partial [illegible]ure, similar to its partial

TABLE 5

SEQUENTIAL SCREENING COMPARISONS

		e/e_o	
MATERIAL	LOT	AIR OVEN	L-10
CLPE	1	0.65	0.68
	3	0.05	0.42
	4	0.56	0.53
TEFZEL®	1	0.92	1.02
	4	0	0.04
SILICONE	1	0.93	0.96
	2	0.24	0.38
	4	0.17	0.36
CLPO	1	0.79	0.98
	2	0.33	0.70
	3	0.24	0.49
	4	0.26	0.72
CSPE	1	0.40	0.74
	2	0.27	0.61
	3	0.19	0.48
	4	0.15	0.58
PVC	1	0.57	0.53
	2	0.18	0.09
	4	0.04	0.03
CP	1	0.01	0.05
	2	0.01	0.03
EPR	1	0.77	0.89
	2	0.49	0.38
	3	0.12	0.17
	4	0.14	0.12

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pressure in air. The results from the table indicate that the very simple application of a thermal oven to approximate LOCA environments may be useful as a screening device for comparing and choosing materials and formulations for safety component applications.

The only unaged material severely degraded by the air oven exposure is chloroprene which is consistent with the L-10 test results. For most of the materials studied, the general trends observed for the air oven results as the aging conditions are changed correlate well with the L-10 results. The notable exception is the CLPE sample aged to 40 Mrad, which shows substantially more degradation in the air oven exposure. This might indicate that the air oven screening test is not always valid, but it might also indicate that this material can become more sensitive to oxygen-containing LOCA simulations as the aging radiation exposure increases. More experiments to test this possibility and to further assess the utility of the sequential screening technique are planned.

CONCLUSIONS

The most important conclusion of this study is the observation that the oxygen concentration during a LOCA simulation can have a significant influence on the chemical and physical changes occurring in certain polymeric materials. The chloroprene material studied appeared to suffer significantly more damage in LOCA simulations containing oxygen, a conclusion which was valid even for samples which were not aged. Samples of the EPR insulation material which were aged in lots 3 and 4 had substantially decreased tensile properties when oxygen was included in the LOCA simulations. Solubility, swelling and Fourier Transform Infrared Spectroscopy results on these EPR materials indicated that increased oxidative scission was responsible for this enhanced degradation. For a PVC material, on the other hand, the results indicate that the damage may become more severe as the oxygen content is lowered (slightly greater mechanical damage, substantially greater swelling). Thus it is possible that a LOCA simulation without oxygen may underestimate the changes which occur in some materials while overestimating the changes which occur in other materials.

These results have important implications for material qualification testing relative to a LOCA. Since very low leak rates are designed into containment, it would appear that the original oxygen content in the air is largely trapped in the containment area for the duration of a "real" accident. Since no mechanism for oxygen replenishment exists, its concentration might decrease as the accident progresses due to both leakage and reaction with the materials in containment. It is therefore important to derive a more precise estimate of the time-dependent oxygen concentration during a LOCA and to use this information as a guide to the proper oxygen concentration to use in LOCA simulations.

The second major conclusion of this study concerns the importance of aging conditions on material response during LOCA. For the EPR insulation, which previously showed dose-rate effects during radiation aging studies (4), evidence was found for dose-rate induced effects being further amplified during the LOCA simulation. Since the lowest dose rate used in the present study was approximately 25 krad/hr, future studies should be carried out to see if further lowering of the aging dose rate leads to more enhancement of the degradation during LOCA simulation. Such an effect might be expected based on aging results obtained previously which indicate the occurrence of dose-rate effects for certain materials at dose rates below 25 krad/hr (4,5).

Different materials exhibited widely varying responses to the aging, LOCA simulation sequences, in terms of both the nature and the magnitude of the induced degradation. Some materials (PVC and chloroprene) exhibited huge steam-induced swelling. Certain materials (such as the CLPO and silicone) were degraded by the aging, but showed little or no further deterioration on subsequent LOCA simulation. Other materials, such as Tefzel, chloroprene and PVC showed moderate to significant aging-related degradation, and this degradation was strongly amplified by the subsequent LOCA simulation.

Many of the materials (Tefzel®, EPR insulation, PVC) exhibited substantial degradation as a result of the combination of aging and LOCA simulation, yet showed little or no damage when unaged samples were exposed to the LOCA simulation. Some of the materials (including Tefzel®, chloroprene and PVC) were very badly degraded (i.e., tensile values reduced to near zero) after exposure to various of the aging, LOCA simulation sequences employed in this study. Other materials (including CLPE, CSPE and CLPO) exhibited only modest damage under all of the testing conditions.

Finally, we have shown that the use of thermal air ovens may be a helpful screening device for assessing the importance of aging conditions on material response to LOCA. Since LOCA simulation experiments are expensive and difficult, this suggested sequential procedure could offer a relatively easy screening method for choosing materials and optimizing material formulations for applications to nuclear power plant safety equipment.

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