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The Effect of Thermal and Irradiation Aging Simulation Procedures on Polymer Properties

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PREVIOUS PUBLICATIONS IN THIS SERIES

1. K. T. Gillen, R. L. Clough, G. Ganouna-Cohen, J. Chenion, and G. Delmas, Loss of Coolant Accident (LOCA) Simulation Tests on Polymers: The Importance of Including Oxygen, NUREG/CR-2763, SAND82-1071, July 1982.

2. K. T. Gillen, R. L. Clough, G. Ganouna-Cohen, J. Chenion, and G. Delmas, "The Importance of Oxygen in LOCA Simulation Tests," Nuclear Engineering and Design, 74 (1982), pgs. 271-285.

ABSTRACT

Prior to initiating a qualification test on safety-related equipment, the testing sequence for thermal and irradiation aging exposures must be chosen. Likewise, the temperature during irradiation must be selected. Typically, U.S. qualification efforts employ ambient temperature irradiation, while French qualification efforts employ 70°C irradiations. For several polymer materials, the influence of the thermal and irradiation aging sequence, as well as the irradiation temperature (ambient versus 70°C), has been investigated in preparation for Loss-of-Coolant Accident simulated tests.

Ultimate tensile properties at completion of aging are presented for three XLPO and XLPE, five EPR and EPDM, two CSPE (HYPALON), one CPE, one VAMAC, one polydiallylphtalate, and one PPS material.

Bend test results at completion of aging are presented for two TEFZEL materials.

Permanent set after compression results are presented for three EPR, one VAMAC, one BUNA N, one Silicone, and one Viton material.

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ACKNOWLEDGMENTS

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EXECUTIVE SUMMARY

As part of a joint French/U.S. sponsored research program, the influence of accident testing conditions on the behavior of polymer materials is being investigated. Test variables in the accident program include: irradiation temperature, oxygen presence during accident simulations, and simultaneous versus sequential accident exposures. In preparation for the accident tests, polymer materials were exposed to irradiation and elevated temperatures to produce an "accelerated age". Test variables in the aging program were irradiation temperature, the order of the aging sequence, and simultaneous versus sequential aging techniques. The influence of aging dose rate and accident oxygen concentration has previously been documented[1] and our current work is an extension of this previous effort.

In this report, test results at completion of the aging exposures are presented for several commercial insulation and jacket materials, compression and gasket materials, and thermosetting and thermoplastic materials. Our goals for the aging portion of the research program were:

1. To investigate whether polymer materials are sensitive to the sequential order in which they are irradiated and thermally aged. Previously published results[2] for low-density polyethylene (LDPE) and polyvinylchloride (PVC) indicated that irradiation followed by a thermal aging exposure was the more severe sequence. We wished to establish the applicability of this conclusion for a broader class of materials. Both irradiation followed by thermal exposure and thermal exposure followed by irradiation aging sequences were part of the testing program.

2. To investigate the importance of irradiation temperature during sequential aging exposures. French regulatory documents[3] require aging and accident irradiations for qualification testing to be performed at 70°C. In contrast, typical U.S. qualification efforts perform ambient temperature irradiations. Irradiations at ambient (~27°C) and 70°C were performed as part of the testing program.

3. To provide a variety of aged samples for use during LOCA simulation experiments.

Our experimental results at completion of the aging program indicate:

1. If sequential ordering of irradiation and thermal exposures was important to the aging degradation of tensile properties, usually the irradiation followed by thermal exposure sequence was most severe.

2. In general, the choice of irradiation temperature was secondary to the choice of aging sequence in its effect on polymer properties.

3. For several materials, tensile properties at completion of aging were only slightly affected by both the irradiation temperature and the order of the sequential aging environmental exposures.

4. At most, only slight differences in permanent set were noted as a result of the various aging techniques.

Ultimate tensile properties at completion of aging are presented for three cross-linked polyolefin (XLPO) and cross linked polyethylene (XLPE), five ethylene propylene rubber (EPR) and ethylene propylene diene terpolymer (EPDM), two chlorosulfonated polyethylene (CSPE, HYPALON), one chlorinated polyethylene (CPE), one acrylic polyethylene (VAMAC), one polydiallylphtalate, and one phenylene polysulfide (PPS) material.

Bend test results at completion of aging are presented for two TEFZEL materials.

Permanent set after compression results are presented for three ethylene propylene rubber (EPR), one acrylic polyethylene (VAMAC), one BUNA N, one Silicone, and one Viton material.

1.0 INTRODUCTION

When used as part of a safety-related system, equipment must meet certain qualification criteria. Several documents describe or suggest the tests and data necessary to establish qualification. For example, IEEE standards,[4] USNRC Rules (10CFR50.49)[5] and documents (NUREG-0588)[6] and French Documents (HM/63-7195/6)[3] provide guidance concerning qualification. These documents generally require qualification efforts to account for aging of components under normal operating conditions by performing accelerated aging of equipment or components prior to simulation of accident conditions.

The aging environment inside a nuclear reactor containment is a multistress environment including thermal and radiation stresses. Historically, aging in this multistress environment is simulated by a sequential application of single stress environmental exposures. For example, IEEE 323-1974[7] suggests a thermal aging followed by irradiation aging exposure. This exposure is considered the most severe sequence for most equipment and applications. "However, the sequence used shall be justified as the most severe for the item being tested."[7]

As part of a joint French/U.S. sponsored research program, we are investigating the influence of testing conditions on the behavior of polymer materials. Variables in the test program include: aging sequence, irradiation temperature, oxygen presence during accident simulations, and simultaneous versus sequential accident and aging exposures. The influence of dose rate and oxygen concentration has previously been documented[1] and our current work is an extension of this previous effort.

In preparation for LOCA research experiments, we have aged several commercial insulation and jacket materials, compression and gasket materials, and thermosetting and thermoplastic dumbbells. We have accumulated sufficient aging data to demonstrate that some materials are sensitive to the order and manner in which they are irradiated and thermally aged. Properties of other polymer materials did not depend on the order and technique of radiation and elevated temperature exposures. In this report we summarize our data and discuss its significance toward qualification testing of nuclear grade safety-related equipment.

2.0 SAMPLES

2.1 U.S. Samples

The U.S. samples consisted of six insulation materials, two jacket materials, and five compression materials. The insulation and jacket materials were carefully obtained by disassembling cable received from five U.S. manufacturers of Class 1E cables. The materials are:

EPR 1 — A radiation crosslinked fire-retardant EPDM insulation obtained from a shielded instrumentation cable.

EPR 2 — A chemically crosslinked fire-retardant EPDM insulation obtained from a 600V, 3-conductor control cable.

XLPO 1 — A crosslinked polyolefin insulation obtained from a shielded instrumentation cable.

XLPO 2 — A crosslinked polyolefin insulation obtained from a 600V, 3-conductor control cable.

TEFZEL 1 — A TEFZEL insulation removed from a thermocouple extension cable.

TEFZEL 2 — A TEFZEL insulation removed from a shielded instrumentation cable.

CSPE — A chlorosulfonated polyethylene jacket removed from a 600V, 3-conductor control cable.

CPE — A chlorinated polyethylene jacket removed from a 600V, 3-conductor control cable.

Each of the jacket and insulation specimens were cut to a length of 10.9 $\pm$ .3 cm (except for EPR 1 which had an initial length of 10.2 $\pm$ .3 cm). The insulation specimens were tubular in shape. The nominal insulation thickness is summarized in Table 2.1. The jacket specimens were cut with a die into rectangular pieces. The width of each specimen was .56 cm. The nominal jacket thicknesses are also presented in Table 2.1.

The five compression materials were obtained from the same U.S. manufacturer. Samples, received from the manufacturer as compression molded sheets with nominal thicknesses of .178 and .191 cm, were cut into small rectangles with approximate dimensions: .6 cm x 1.6 cm. The five materials were: EPR A, EPR B, BUNA N, SILICONE, and VITON.

Table 2.1

Nominal Insulation and Jacket Thicknesses for U.S. Samples

Material	Nominal Thickness (cm)
EPR 1	.064
EPR 2	.076
XLPO 1	.076
XLPO 2	.076
TEFZEL 1	.038
TEFZEL 2	.051
CSPE	.114
CPE	.114

2.2 French Samples

The French samples consist of six elastomer materials used in the manufacture of electrical cables (insulation and jacket materials), two O-ring seal materials and two thermoplastic and thermosetting materials used in the manufacture of connectors.

The electrical cable materials are in the form of either 110 mm-long pieces of insulating material stripped from the copper conductor (identified by "I"), dumbbells cut from jacket material and identified by "G", or standard dumbbells cut from compression molded sheets (identified by "H"). Elastomer dumbbell dimensions are shown in Figure 2.1. The six cable materials are:

82 I1 PRC — Chemically crosslinked polyethylene in the form of conductor insulation for 3-conductor cables.

82 I2 EPDM — Ethylene propylene diene terpolymer conductor insulation. Samples taken from a 3-conductor cable.

82 I9 EPDM — Alumina-loaded, ethylene propylene diene terpolymer conductor insulation. This material was removed from a 3-conductor cable.

82 G10 HYPALON — Chlorosulfonated polyethylene used in the manufacture of cable jackets.

82 H3 VAMAC — Acrylic polyethylene in the form of sheets from which dumbbells (Figure 2.1) were cut. Material is used in electrical cable jackets, mechanical parts, and connectors.

82 H4 EPR — Copolymer ethylene-propylene rubber in the form of 3 mm sheets from which dumbbells (Figure 2.1) were cut. Material is used in the manufacture of insulation for electrical cables sheathed with fire-proof EPDM.

The two O-ring seal samples (identified by "J") have an inner diameter of 12 mm and an outer diameter of 17 mm. They were enclosed and held under compression in aluminum grooves as shown in Figure 2.2. The O-ring seal materials are:

82 J3 VAMAC — Same material as 82 H3, used in the manufacture of O-ring seals.

82 J4 EPR — Same material as 82 H4, used in the manufacture of O-ring seals.

The two thermoplastic and thermosetting materials used in the manufacture of connectors were in the form of International Organization for Standardization (ISO) dumbbells. The dimensions for these dumbbells is illustrated in Figure 2.3. The two materials are:

82 H5 Polydiallylphtalate — Thermosetting polyester used in connectors and mechanical parts.

82 H6 PPS — Phenylene polysulfide used in the manufacture of switches and connectors.

The French sample identification code (82 I1, 82 H5, etc.) will be used in the remainder of this report. The code presents:

- The year of the starting investigation,
- The sample shape code letter,
- A number identifying the material type.

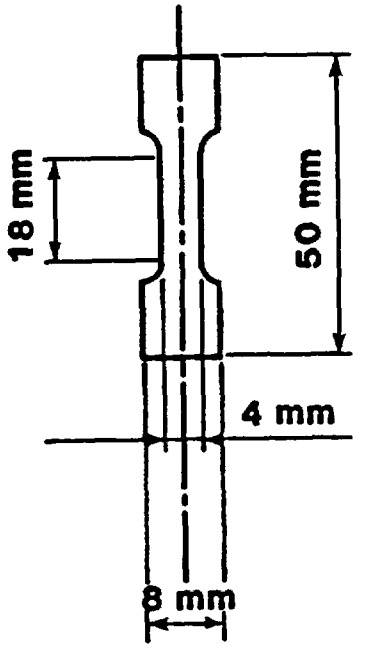


Figure 2.1. Dimensions for 82 G10, 82 H3, and 82 H4 French Dumbbell Samples - H3 French Standard (Commission of Normalization N° NF.T 51-034-June 1968)

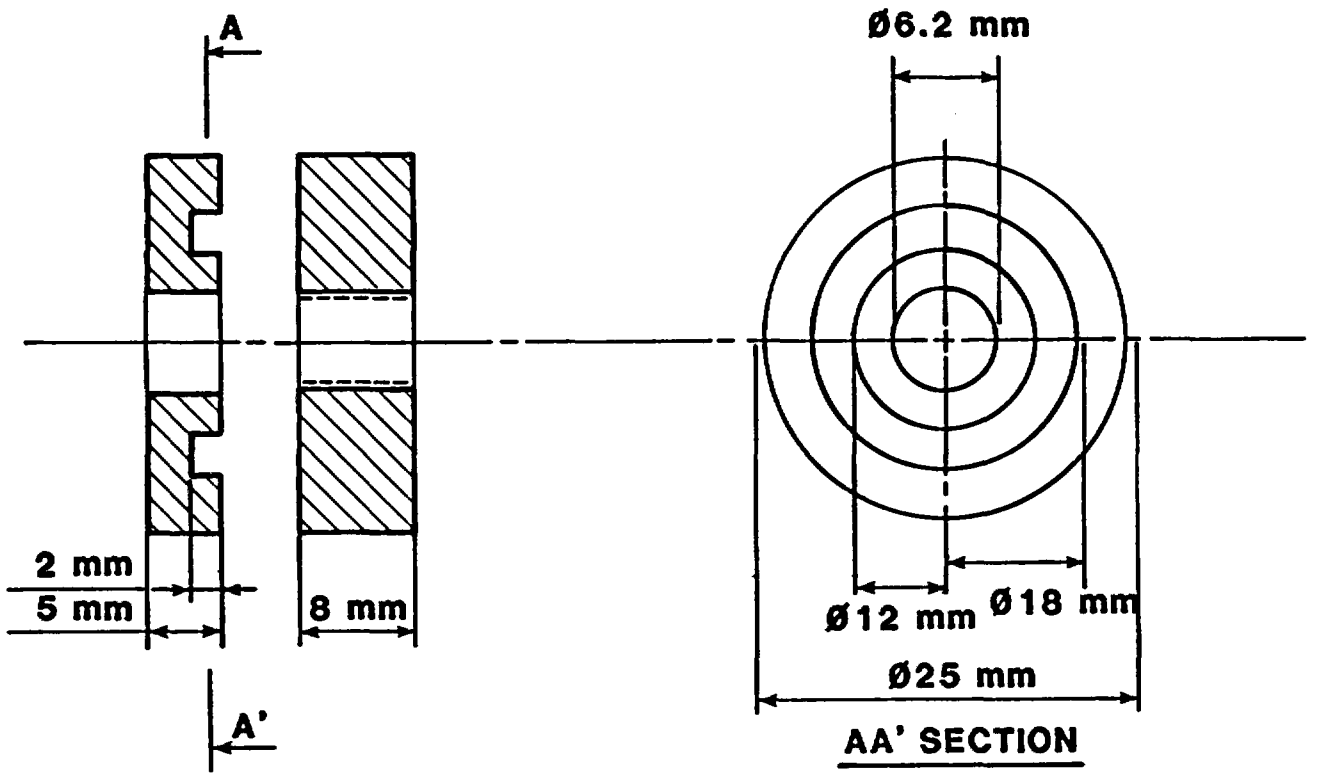


Figure 2.2. Compression Set Fixtures Used for 82 J3 and 82 J4 French O-ring Seal Samples

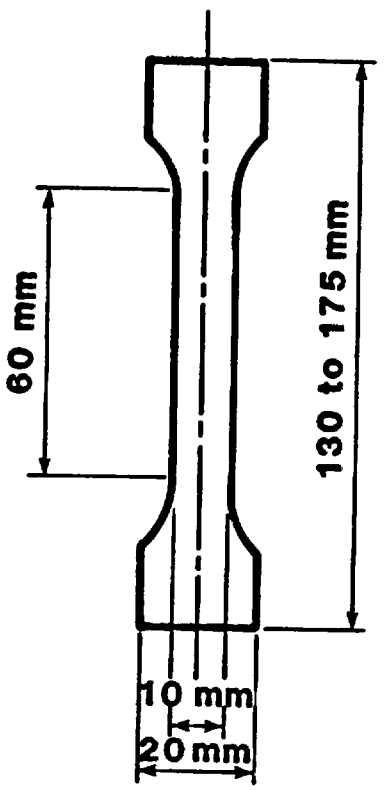


Figure 2.3. Dimensions for 82 H5 and 82 H6 ISO Dumbbell Samples

3.0 FACILITIES

3.1 Radiation Aging Facility[8]

Figure 3.1 shows an artist's rendition of the Low Intensity Cobalt Array (LICA) radiation facility. Approximately 16,000 curies of Co-60 is positioned at the bottom of a water-filled tank. Radiation aging is carried out in water-tight test cells by lowering the cells to the bottom of the tank. Water, separating the Co-60 from experimenters at the top of the tank, provides radiation shielding.

Approximately 10,000 curies of Co-60 is positioned in a circular array to provide high dose-rate exposures. This portion of the radiation facility was not used in this test. The remaining 6,000 curies of Co-60 is positioned in two parallel arrays as shown in Figure 3.2. Test cell holders, each containing four cylindrical holes, are oriented parallel to the linear cobalt holders. One holder is located between the two linear arrays of cobalt; the remaining holders are located on the two sides of the cobalt-60 sources at various distances from them. Test cells placed in any of the holes of a given holder receive comparable radiation dose rates.

The dose rate is governed by the distance of the cell from the cobalt array. Center-of-can dose rates are given in Figure 3.2. Gradients in dose rates occur in the individual test cells; for example, there is a dropoff in dose rates between the parts of the cell closest to, and furthest from, the cobalt. A Victoreen Model 550 Radicon III Integration/Rate Electrometer with a Model 550 air ionization probe was used to measure the dose rate at the center of the test cell. Accuracy was $\pm$ 5 percent. Harshaw TLD-400's (calcium fluoride manganese activated thermoluminescent detectors) were used to map the radiation gradients for several of the LICA positions. Figure 3.3 illustrates the positioning of the CaF_2 Mn wafers during gradient mapping; Table 3.1 summarizes the measured gradients. Accuracy of the CaF_2 wafers was $\pm$ 10 percent. For LICA can positions III A, III B, IV A, IV B, V A, and V B, center-of-can dose rates only were measured. Gradients are similar (by symmetry) to those measured for the respective C and D positions. Gradient effects were reduced by rotating the cans during the exposure or by appropriate positioning of the samples within the test cell.

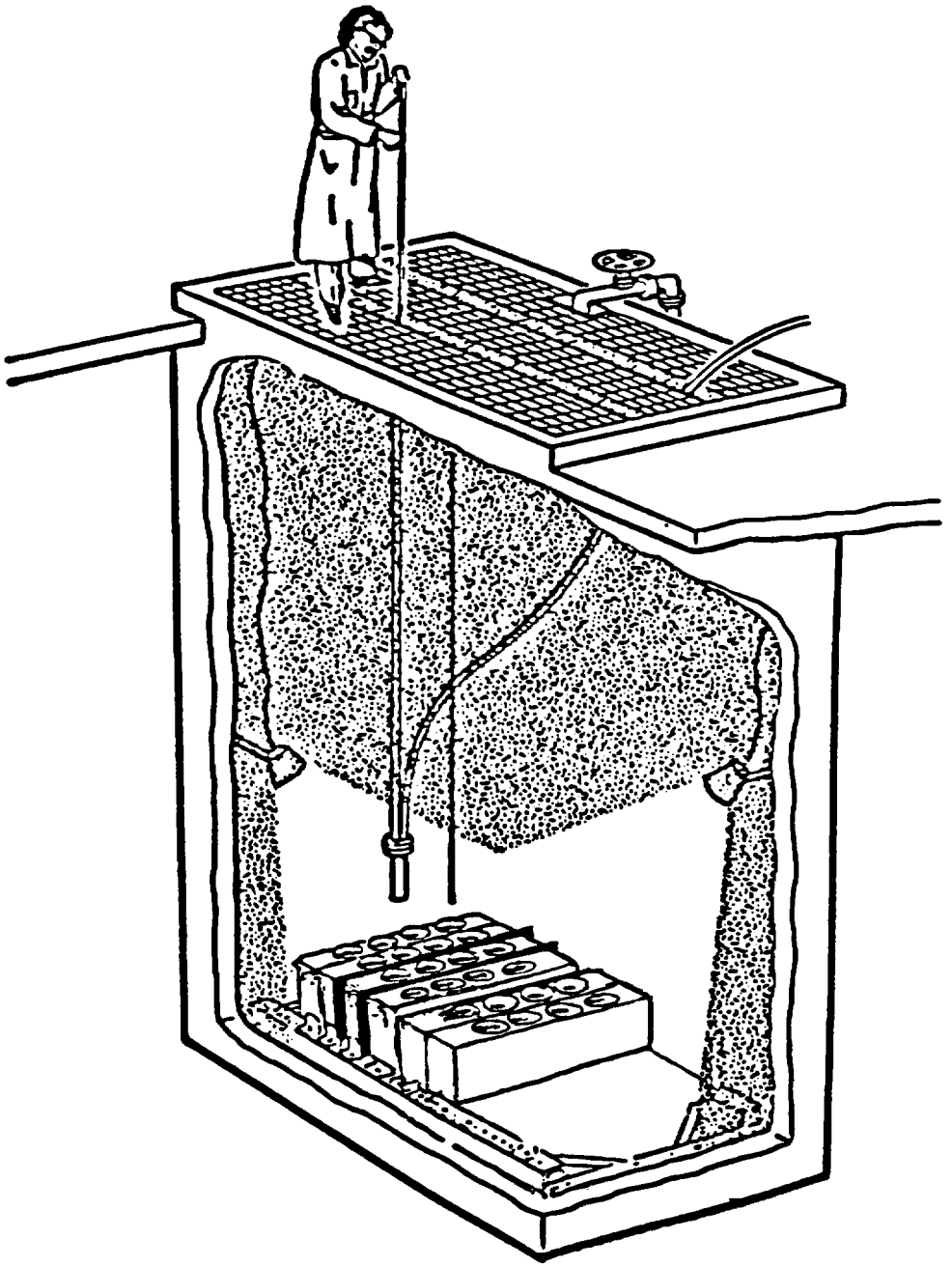


Figure 3.1. Artist's Rendition of Low Intensity Cobalt Array (LICA) Facility

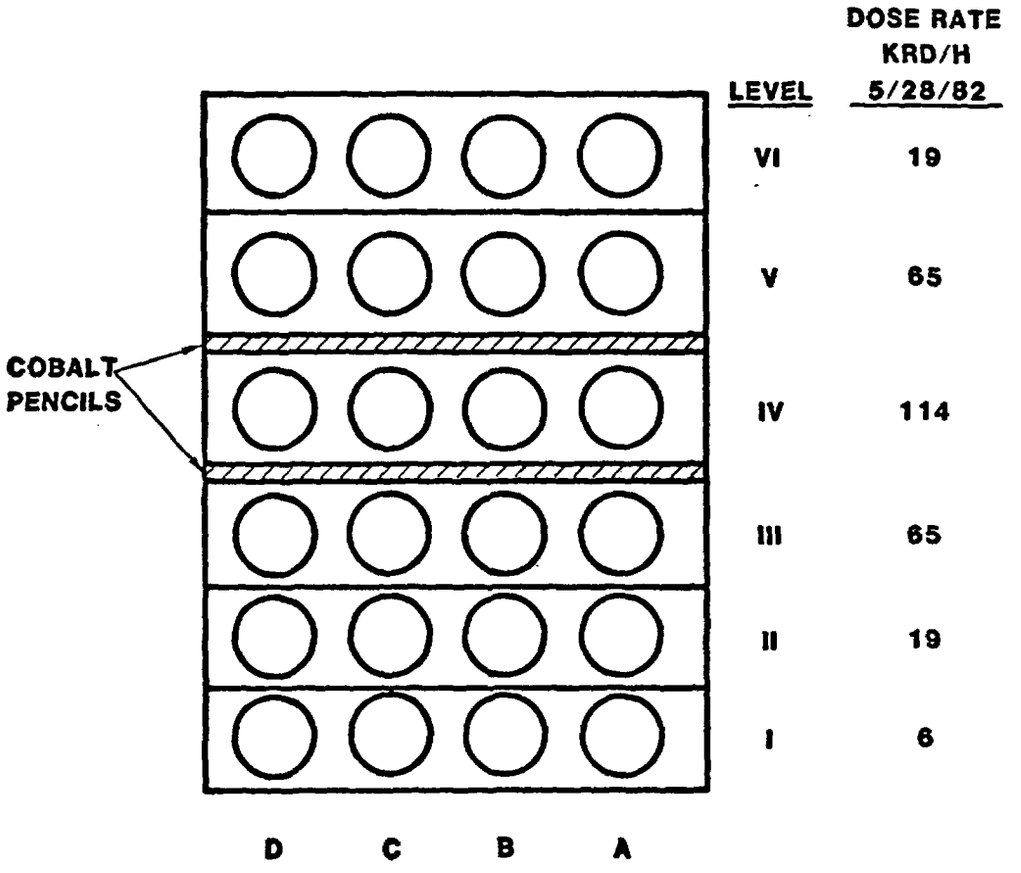


Figure 3.2. Arrangement of the Low Intensity Cobalt Array (LICA) facility for U.S./French aging program. French samples were aged using positions IV A-IV D. U.S. Samples were aged using positions III A-III D and V A-V D.

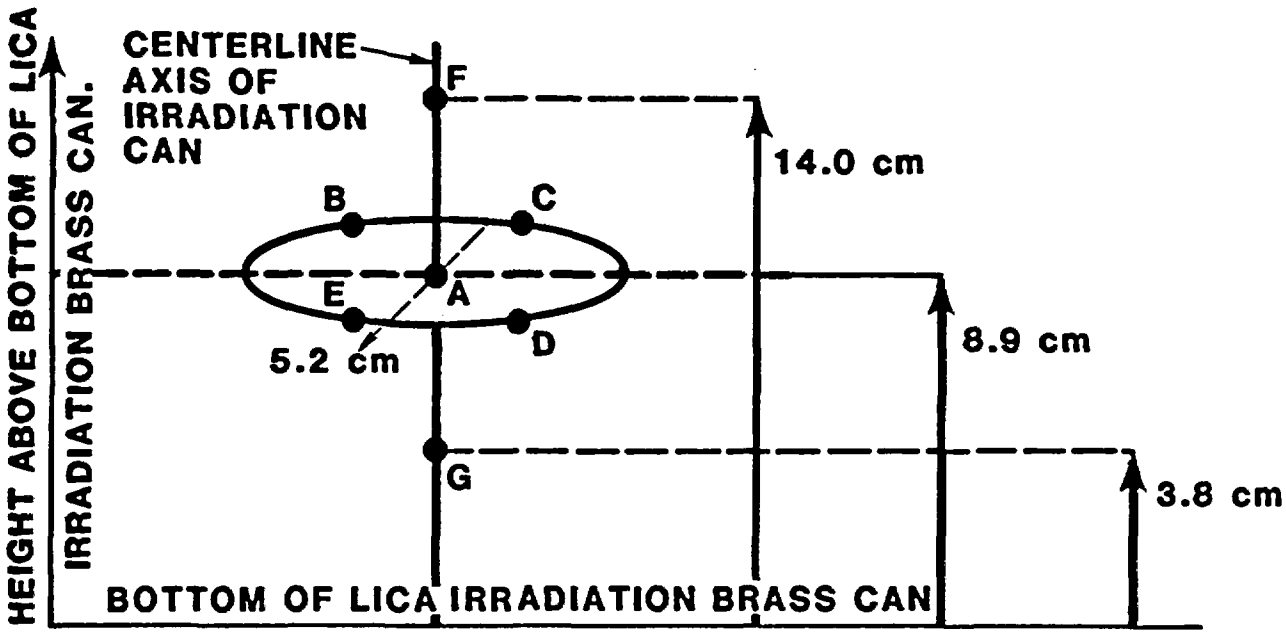


Figure 3.3. Locations of thermoluminescent CaF_2 wafers inside LICA irradiation can during dose rate gradient mapping. CaF_2 wafers were placed at Positions A, B, C, D, E, F, and G.

Table 3.1

Relative Dose Rate Gradients, R/R_A, for Several LICA Irradiation Positions. Measurement Locations A-G are Shown in Figure 3.3.

Measurement Location	LICA Position					
	IIIC	IIID	IVC	IVD	VC	VD
A	1.00	1.00	1.00	1.00	1.00	1.00
B	0.97	1.14	1.01	1.03	1.09	0.92
C	0.69	0.77	0.96	0.98	1.37	1.32
D	0.93	0.83	0.91	1.02	1.02	0.95
E	1.29	1.27	0.96	1.08	0.83	0.76
F	1.05	1.04	0.94	0.94	1.04	0.95
G	0.93	0.85	0.92	0.96	1.06	0.90

A detailed sketch of a test cell is shown in Figure 3.4. The cylindrical sample aging region (10 cm diameter and 23 cm long) is located inside a brass can, which in turn is suspended from the lid of a double-walled stainless-steel can. (The double-walled design provides some thermal insulation.) When the stainless-steel lid is lifted free of the stainless-steel can, a brass lid at the bottom of the brass can is easily removed, allowing access to the sample aging region. Figure 3.5 illustrates insulation specimens prepared to be inserted into the sample aging region.

The sample region is heated using an insulated nichrome wire wrapped around the sides of the brass can and also around a pancake heater which lies between the brass can and the stainless-steel lid. The auxiliary pancake heater was found to be useful for reducing rather substantial temperature gradients at the top of the sample aging region caused by the heat-sink effect of the massive top of the stainless-steel can. To accurately control and monitor the temperature in the sample region, two resistance temperature devices (RTDs) were incorporated in the design. A control RTD is directly clamped to the inside wall of the brass can and a monitor RTD is positioned close to the center of the sample-aging region. A 2.9 cm OD plastic tube runs from the top of the stainless-steel can up through the water tank. Lead wires for the heater and the two RTDs feed through this tube. In addition, a small tube inside the outer plastic tube is used to circulate air or other gaseous environments past the sample region at controlled flow rates. The temperature capabilities of the test cells are room temperature to 150°C.

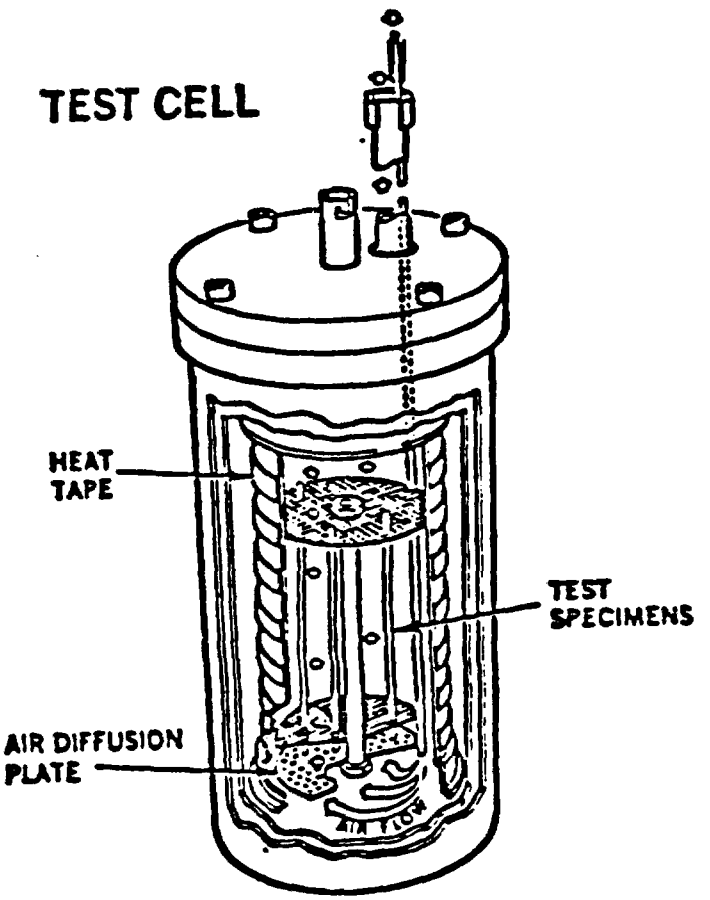


Figure 3.4. A LICA Irradiation Test Cell

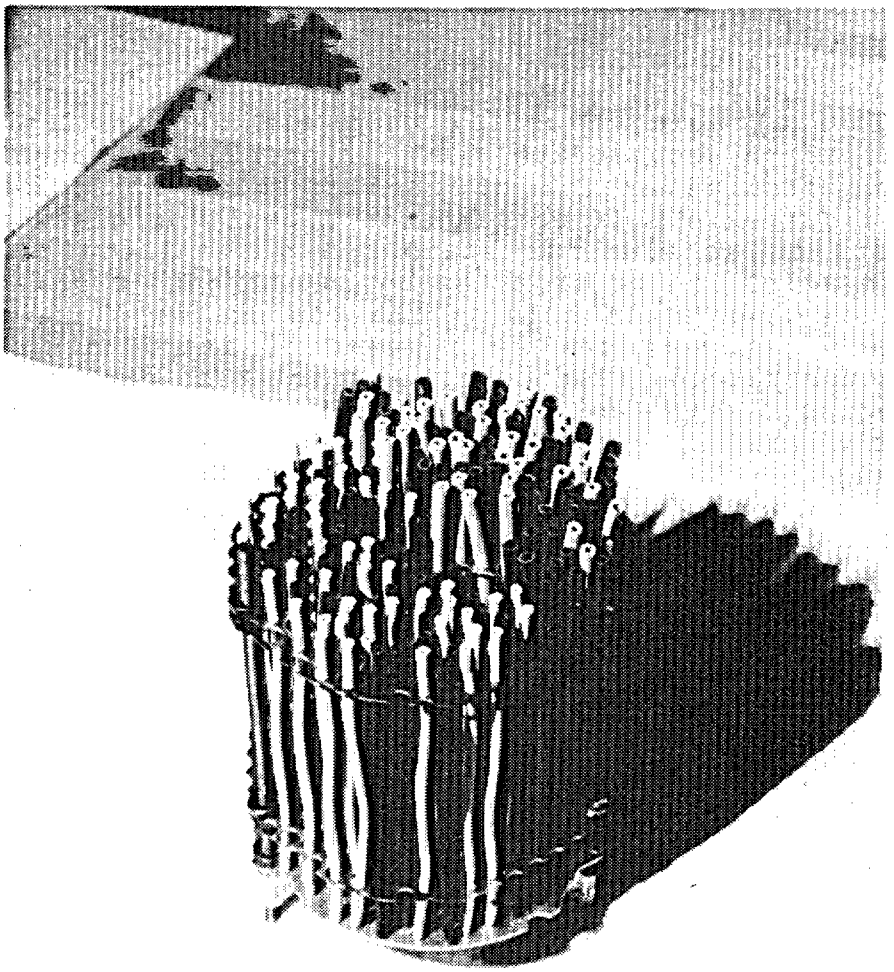


Figure 3.5. EPR 1 and EPR 2 insulation specimens prepared to be inserted into the sample aging region of a LICA irradiation test cell.

3.2 Thermal Aging Facility[8]

The Thermal Aging Facility uses air circulating ovens, each modified to accommodate a number of self-contained aging cells. A detailed sketch of one of these aging cells is shown in Figure 3.6. It consists of a bell jar glass chamber which rests on an aluminum stand. A gas inlet line enters the sample aging region from the bottom through a hole in the aluminum base. A small hole at the top of the glass chamber serves as a gas exit and as a means of introducing a permanently positioned thermocouple into the center of the sample region. Metal collars surrounding the glass bell jars improve the thermal stability and reduce temperature gradients in the sample aging region. Sample holders fit inside the glass chamber. The aging cells used for this test were constructed in three sizes with internal volumes of .9 liter, 2.5 liters, and 3.9 liters.

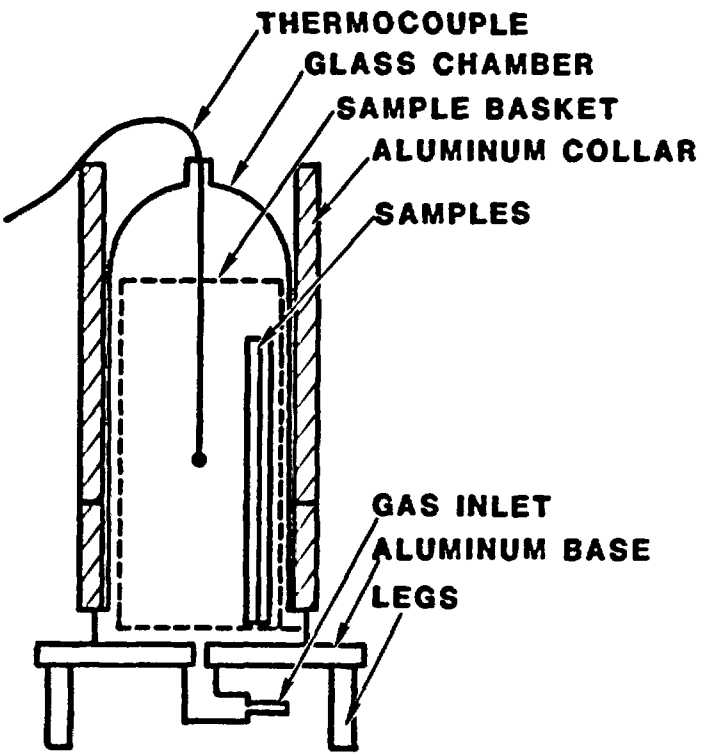


Figure 3.6. Oven Aging Cell

4.0 EXPERIMENTAL TECHNIQUES

4.1 U.S. Samples

The U.S. insulation and jacket samples were exposed to five different aging procedures. These procedures (and the shorthand codes used in the rest of the report to describe them) are:

A = $R_{70} \rightarrow 120°C.$: A 16-day irradiation at ~65 krd/h and 70°C followed by a 16-day thermal exposure at 120°C.

B = $R_{27} \rightarrow 120°C$: A 16-day irradiation at ~65 krd/h and ambient temperatures (~27°C) followed by a 16-day thermal exposure at 120°C.

C = $120°C \rightarrow R_{70}$: A 16-day thermal exposure at 120°C followed by 16-day irradiation at ~65 krd/h and 70°C.

D = $120°C \rightarrow R_{27}$: A 16-day thermal exposure at 120°C followed by a 16-day irradiation at ~65 krd/h and ambient temperatures (27°C).

E = R_{120}: A 16-day simultaneous exposure to 120°C thermal and ~65 krd/h irradiation environments.

The 120°C, 16-day deviated temperature exposure was chosen based on Arrhenius calculations which assumed a 40-year service operation at 45°C and cable material activation energy of 1.0 eV.

The insulation specimens were placed in sample fixtures as illustrated by Figure 3.5. EPR 1 and EPR 2 samples were irradiated together. Likewise, XLPO 1 and XLPO 2 samples were irradiated together. The jacket specimens (CSPE and CPE) were irradiated separately. During irradiations, air flow to the 1.8 liter irradiation cell was approximately 40 cc/min.

For thermal aging, 0.9 liter thermal aging cells were employed for the insulation specimens. Sample holders similar to those shown in Figure 5 (but smaller in diameter) were used to support each individual insulation specimen. EPR 1, EPR 2, XLPO 1, and XLPO 2 samples were each aged separately. The jacket specimens were aged using the 2.5 liter thermal aging cells. CPE and CSPE were thermally aged separately. Air flow to the thermal aging cells was ~20 cc/min for both the insulation and jacket specimens. This corresponds to

.5 to 2 "complete" air changes an hour. The air flow removes from the vicinity of the samples any thermal decomposition products that are outgassed by the samples.

For each material, approximately 80 sample specimens started each aging sequence. Several times during both the irradiation and thermal exposures, aging was momentarily interrupted to allow for removal of several sample specimens. These specimens were used to determine the degradation history during the aging exposures. Midway during the irradiation exposure, the sample cell was rotated ~180° to average radiation gradient effects. All samples pulled from the cell prior to completion of the irradiation were removed from the center of the cell. This minimized the effect of radiation gradients on intermediate exposure results.

Table 4.1 summarizes the total radiation dose and the thermal exposure time for each group of U.S. insulation and jacket specimens.

Tensile tests were performed at Sandia National Laboratories on the EPR 1, EPR 2, XLPO 1, XLPO 2, CSPE, and CPE samples. For each experimental condition, three or four samples were tested to determine the ultimate tensile elongation, e, and the ultimate tensile strength, T. Tensile measurements were performed at ambient temperatures (~23°C) using Instron 1125 (EPR 2 specimens; aging environments A, B, D, and E) and Instron 1130 machines (all other specimens). Samples were gripped using pneumatic jaws; initial jaw separation was 5.1 cm and samples were strained at 12.7 cm/min. The strain was monitored with an Instron electrical tape extensometer clamped to the sample. The systematic difference in normalized elongation and tensile strength due to the use of two different tensile macchines was approximately 12%. Data obtained on the 1130 was higher than that obtained by the 1125.

Bend tests were performed on the TEFZEL 1 and TEFZEL 2 samples rather than tensile tests since aged specimen tubes shattered when gripped by the pneumatic jaws of the Instron tensile testing machine. Bend radii between 75 and 6 times the radii of the TEFZEL specimens were employed. Each specimen was successively wrapped around tubes of smaller diameter until insulation cracking was visually observed.

The U.S. Compression Set Samples were exposed to three different aging procedures. These procedures (and the shorthand used in the rest of the report to describe them) are:

$R_{27} \rightarrow 120°C$ = A 16-day irradiation at ~60 krd/h and ambient temperatures (~27°C) followed by a 16-day thermal exposure at 120°C.

$120°C \rightarrow R_{27}$ = A 16-day thermal exposure at 120°C followed by a 16-day irradiation at ~60 krd/h and ambient temperatures (~27°C).

R_{120} = A 16-day simultaneous exposure to 120°C thermal and ~60 krd/h irradiation environments.

The compression set samples were exposed using a fixture as shown in Figure 4.1. Each layer of the fixture contained three .127 $\pm$ .001 cm spacers and four compression set samples. Each of the five layers of one fixture was used for a different compression sample material; namely EPR A, EPR B, BUNA N, VITON, or SILICONE. Two sample fixtures were irradiated in an aging cell. The fixtures were oriented so that the compression fixture surfaces were perpendicular to the Cobalt-60 array (see Figure 4.2). Air flow to the irradiation cell was approximately 40 cc/min. Thermal aging was performed using a 2.5 liter aging cell containing four fixtures. Air flow to the thermal aging cell was also 40 cc/min. Table 4.2 summarizes irradiation doses and thermal aging times for each fixture.

The compression set samples ranged in thickness between .165 and .205 cm. Prior to compression, each individual sample was measured for thickness and its position in the fixture noted. After exposures to radiation and thermal environments, the sample thickness was again measured. Prior to the final measurements, the fixture was disassembled (releasing the compressive strain) and a 30-minute recovery time allowed.

Our compression set samples have much smaller thicknesses than those recommended by ASTM Standard D395-78.[9] This Standard recommends (for Method B) test specimens with 1.25 cm thicknesses. Our sample thicknesses more realistically simulate use conditions, but generate much larger errors in the calculated values for compression set. Absolute error for our compression set values is $\pm$ 0.1.

Table 4.1

Radiation Dose and Thermal Exposure Time
for U.S. Insulation and Jacket Specimens

Sample Group	Total Radiation Dose[Mrd(air equiv.)]	Thermal Exposure Time (HRS)
XLPO 1A	24.2*	385 hours
XLPO 1B	24.2	385 hours
XLPO 1C	23.4	384 hours
XLPO 1D	23.1	385 hours
XLPO 1E	24.3	386 hours
XLPO 2A	22.7*	386 hours
XLPO 2B	24.2	385 hours
XLPO 2C	23.4	384 hours
XLPO 2D	23.0	385 hours
XLPO 2E	24.3	386 hours
EPR 1A	25.7	385 hours
EPR 1B	25.5	385 hours
EPR 1C	25.0	384 hours
EPR 1D	25.3	384 hours
EPR 1E	25.7	384 hours
EPR 2A	25.7	385 hours
EPR 2B	25.5	385 hours
EPR 2C	25.0	384 hours
EPR 2D	25.3	384 hours
EPR 2E	25.7	384 hours
CSPE A	25.0	389 hours
CSPE B	25.4	380 hours
CSPE C	24.2	389 hours
CSPE D	24.2	389 hours
CSPE E	24.6	384 hours
CPE A	23.9	389 hours
CPE B	23.4	389 hours
CPE C	23.1	385 hours
CPE D	23.1	385 hours
CPE E	23.4	384 hours
TEFZEL 1A	22.3	386 hours
TEFZEL 1B	22.9	377 hours
TEFZEL 1C	22.3	383 hours
TEFZEL 1D	22.3	384 hours
TEFZEL 1E	22.4	381 hours
TEFZEL 2A	22.3	386 hours
TEFZEL 2B	22.9	377 hours
TEFZEL 2C	22.3	383 hours
TEFZEL 2D	22.3	384 hours
TEFZEL 2E	22.4	381 hours

* XLPO 1A and XLPO 2A were irradiated separately.

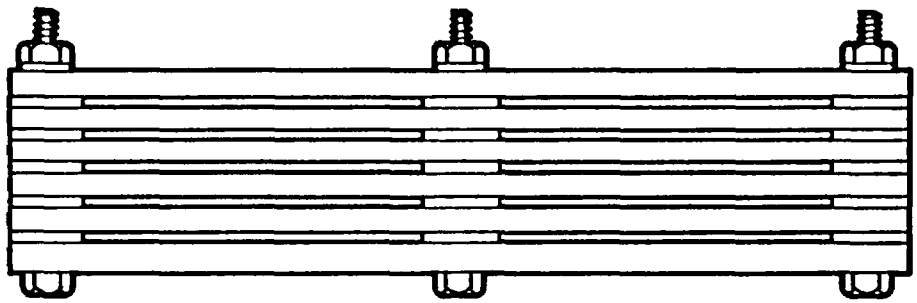


Figure 4.1. U.S. Compression Set Fixture

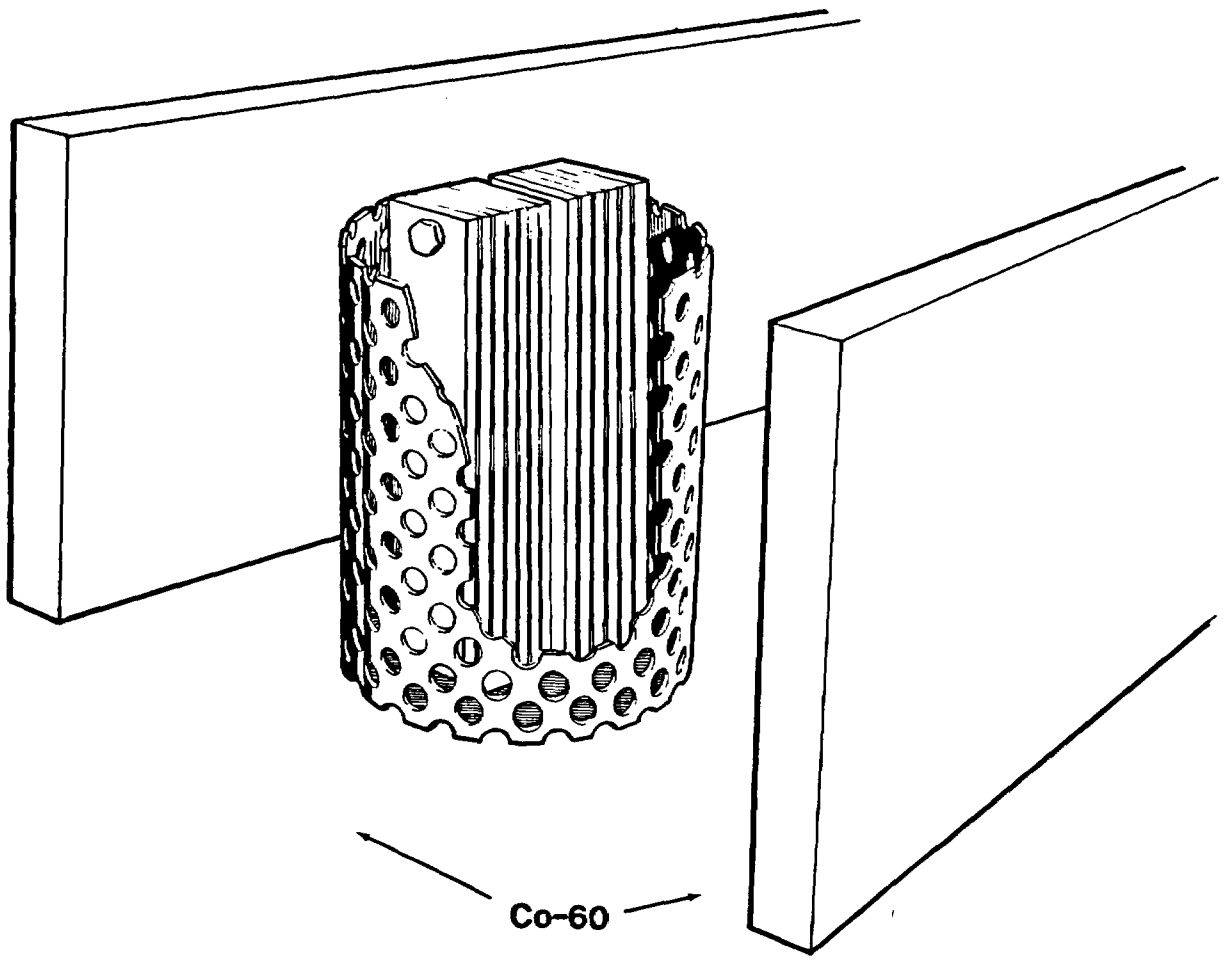


Figure 4.2. Orientation of U.S. Compression Set Fixtures in Radiation Field

Table 4.2

Radiation Doses and Thermal Aging Conditions
for Each U.S. Compression Set Fixture

Compression Set Fixture	Aging Sequence	Total Dose (Mrd)	Thermal Aging Time (hrs)
#38	Unaged	NA	NA
#12	R_{27}	23.8	NA
#37	120	NA	386
#32	R_{120}	22.9	382
#13	R_{27} → 120	23.8	386
#14	R_{27} → 120	23.8	386
#20	120 → R_{27}	23.4	386
#50	120 → R_{27}	23.0	386

4.2 French Samples

The French samples were exposed to four different aging procedures. These procedures (and the shorthand codes used in the rest of the report to describe them) are:

A = T → R_{70}: A 10-day thermal exposure followed by a 9- or 10-day irradiation at ~115 krd/h and 70°C.

B = R_{70} → T: A 9- or 10-day irradiation at ~115 krd/h and 70°C followed by a 10-day thermal exposure.

C = T → R_{27}: A 10-day thermal exposure followed by a 9- or 10-day irradiation at ~115 krd/h and ambient temperatures (~27°C).

D = R_{27} → T: A 9- or 10-day irradiation at ~115 krd/h and ambient temperatures (~27°C) followed by a 10-day thermal exposure.

The 10-day thermal exposure temperature depended on the specimen material. Table 4.3 lists each of the French

Samples and the appropriate thermal aging temperature. Various sample groups were irradiated over a several month period. Co-60 decay during this time period necessitated varying the irradiation exposure time from 9 days to 10 days so that each sample group was irradiated to a similar dose (~25 Mrd).

Table 4.4 summarizes the total radiation dose and the thermal exposure time for each group of French samples. The insulation specimens (82 I1, 82 I2, 82 I9) were irradiated and thermally aged using fixtures similar to that shown in Figure 3.5. 82 I1 and 82 I2 samples were irradiated together in the same aging cell. 82 I9 samples were irradiated alone. Air flow to each of the 1.8 liter irradiation cells was ~60 cc/min. Thermal aging of each group of insulation specimens was performed separately using a 0.9 liter aging cell and an air flow of ~30 cc min. The 82 H5 samples were irradiated as shown in Figures 4.3 through 4.5. Metallic washers were used to keep each dumbbell separate from all others. A similar arrangement was used for the 82 H6 samples (but without the basket). Air flow to the irradiation chambers was ~60 cc/min. The 82 H5 samples were thermally aged using 0.9 liter thermal cells and ~30 cc/min air flow. The 82 H6 samples were thermally aged using 3.9 liter aging cells and a ~100 cc/min air flow. 82 H3, 82 H4, and 82 G10 dumbbells were assembled as shown in Figures 4.6 and 4.7 and oriented for irradiation as shown in Figure 4.8. Air flow to the 1.8 liter irradiation cell was ~60 cc/min; air flow to the 0.9 liter thermal aging cell ~30 cc/min. The 82 J3 compression set assemblies were randomly placed in a basket and irradiated as shown in Figure 4.9. The 82 J4 compression set assemblies were stacked in a basket and irradiated as shown in Figure 4.10.

Air flow to the 1.8 liter irradiation cell was ~60 cc/min for the 82 J3 and 82 J4 samples. These samples were therm- ally aged using 0.9 liter aging cells with an air flow of ~30 cc/min.

The ultimate tensile elongation, e, and ultimate tensile strength, T, were measured at CEA-ORIS-LABRA in Saclay for French elastomer samples 82 I1, 82 I2, 82 I9, 82 H3, 82 H4, and 82 G10, the thermoplastic material 82 H6, and the thermosetting material 82 H5. A ZWICK Model 7025/3 test machine, located in a room with controlled temperature of 21°C, was used for the tensile tests. Dumbbells and conductor insulation samples were gripped in the jaws of the test machine. Initial spacing between the jaws was 40 mm for the 82 G10, 82 H3, and 82 H4 dumbbells, approximately 150 mm for the 82 H5 and 82 H6 dumbbells, and on the average 110 mm for the

82 I1, 82 I2, and 82 I9 insulation specimens. An extensometer was located at the center of each test piece. Its gap was 10.0 mm. The strain rate was 50 mm per minute.

Compression set measurements were performed at CEA-ORIS-LABRA in Saclay for French elastomer samples 82 J3 and 82 J4.

Table 4.5 summarizes the accuracy of the French measurements. Each measurement is made on five test pieces. If a measurement showed excessive discrepancy from the average it was rejected and a new average was computed.

Table 4.3

Thermal Exposure Temperatures For French Samples

	Sample Description	Sample Code	Exposure Temperature (°C)
1.	PRC Insulation	82 I1	140
2.	EPDM Insulation	82 I2	140
3.	EPDM Insulation	82 I9	140
4.	PPS Dumbbells	82 H6	160
5.	Polydiallylphtalate Dumbbells	82 H5	160
6.	VAMAC Acrylic Polyethylene Dumbbells	82 H3	120
7.	EPR Dumbbells	82 H4	140
8.	HYPALON Dumbbells	82 G10	140
9.	VAMAC Acrylic Poly-ethylene O-ring seal samples	82 J3	120
10.	EPR O-ring seal samples	82 J4	140

Table 4.4

Total Radiation Dose and the Thermal Exposure Time for Each Group of French Samples

Radiation Exposures

	Radiation Dose	Thermal Exposure Time
82 I1A	24.7 Mrd	240.5 hours
B	24.7 Mrd	240.2 hours
C	24.7 Mrd	240.5 hours
D	24.9 Mrd	240.2 hours
82 I2A	24.7 Mrd	240.5 hours
B	24.7 Mrd	240.2 hours
C	24.7 Mrd	240.5 hours
D	24.9 Mrd	240.2 hours
82 I9A	24.0 Mrd	258.5 hours
B	25.3 Mrd	238.8 hours
C	23.9 Mrd	258.5 hours
D	25.6 Mrd	241.1 hours
82 H6A	26.0 Mrd	238.6 hours
B	26.0 Mrd	238.7 hours
C	25.8 Mrd	238.6 hours
D	26.2 Mrd	238.7 hours
82 H5A	24.1 Mrd	240.2 hours
B	24.6 Mrd	238.6 hours
C	23.8 Mrd	240.2 hours
D	24.0 Mrd	241.0 hours
82 H3A	25.4 Mrd	240.0 hours
B	25.4 Mrd	240.2 hours
C	25.4 Mrd	240.0 hours
D	25.4 Mrd	240.2 hours
82 H4A	25.1 Mrd	258.5 hours
B	25.1 Mrd	268.8 hours
C	24.9 Mrd	258.5 hours
D	24.9 Mrd	268.8 hours
82 G10A	25.5 Mrd	239.0 hours
B	25.5 Mrd	243.9 hours
C	24.4 Mrd	239.0 hours
D	24.4 Mrd	243.9 hours

Table 4.4 (Continued)

Total Radiation Dose and the Thermal Exposure Time for Each Group of French Samples

Radiation Exposures

	Radiation Dose	Thermal Exposure Time
82 J3A	25.4 Mrd	240.0 hours
B	25.4 Mrd	241.3 hours
C	24.5 Mrd	240.1 hours
D	24.5 Mrd	240.2 hours
82 J4A	24.0 Mrd	245.5 hours
B	24.6 Mrd	243.9 hours
C	24.1 Mrd	245.5 hours
D	24.3 Mrd	239.0 hours

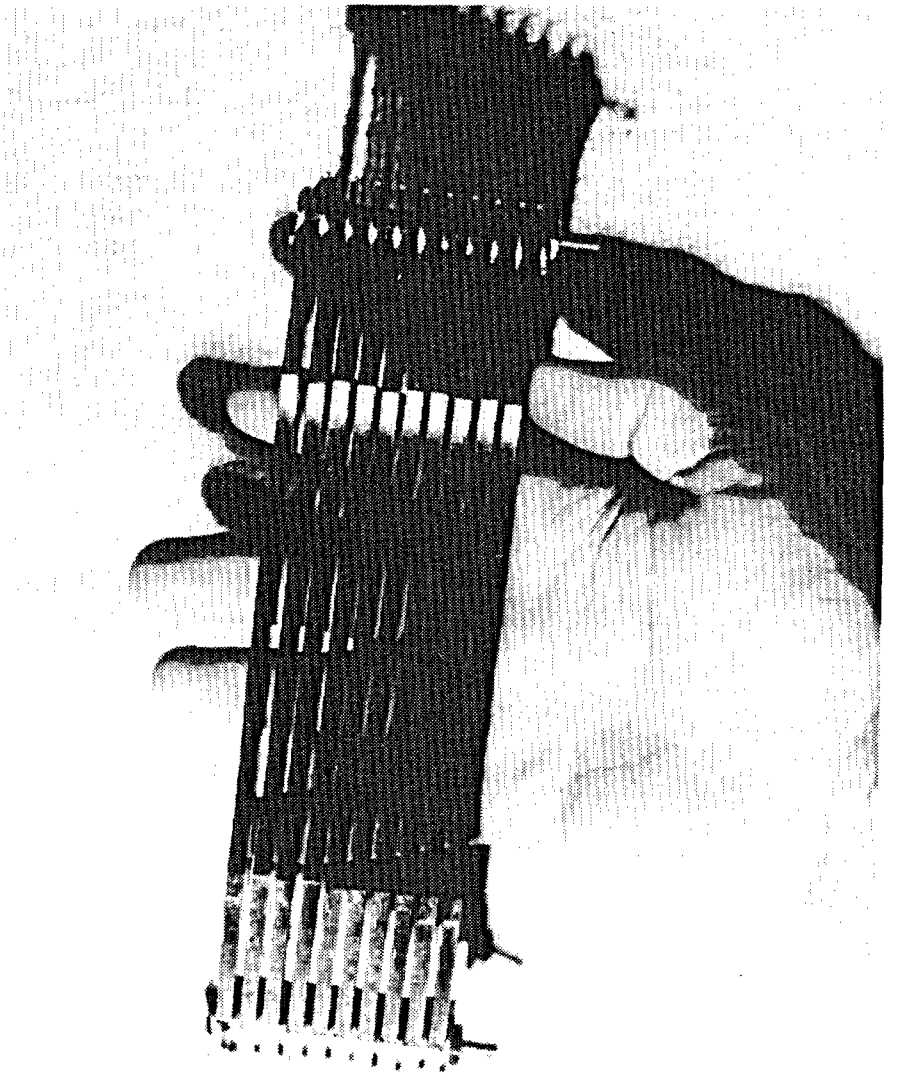


Figure 4.3. 82 H5 and 82 H6 Samples Prepared for Aging

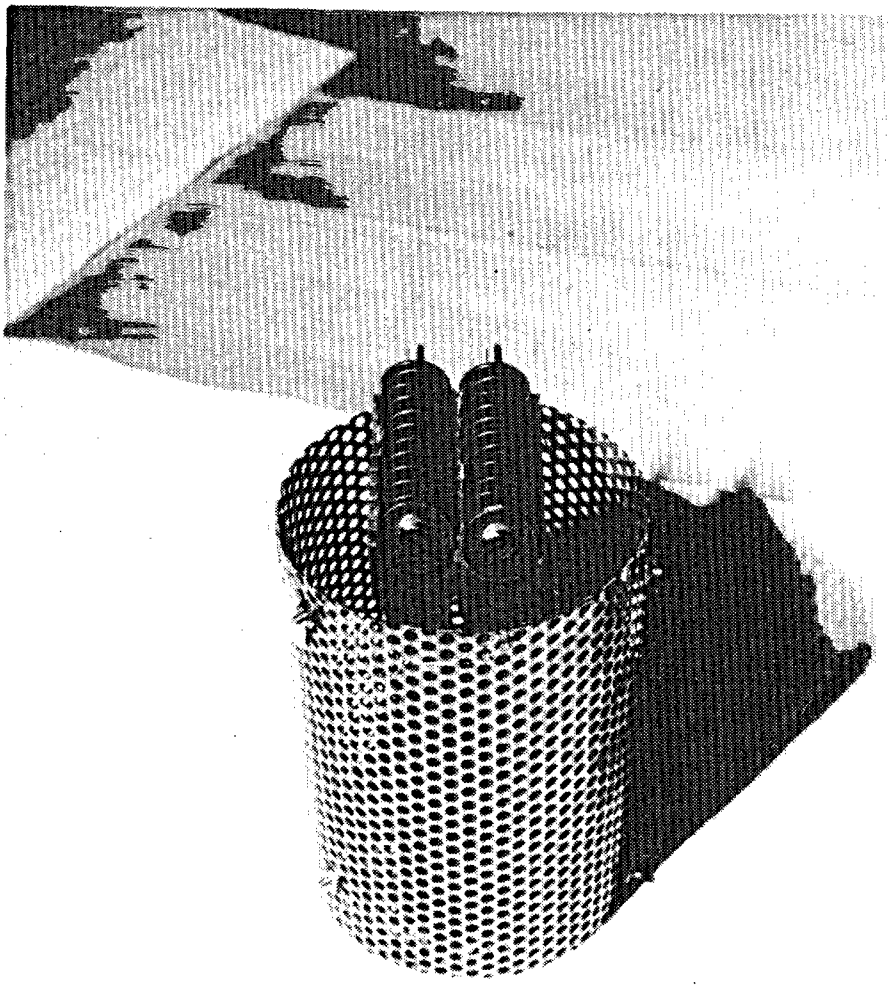


Figure 4.4. 82 H5 Samples Prior to Insertion into LICA Irradiation Cell

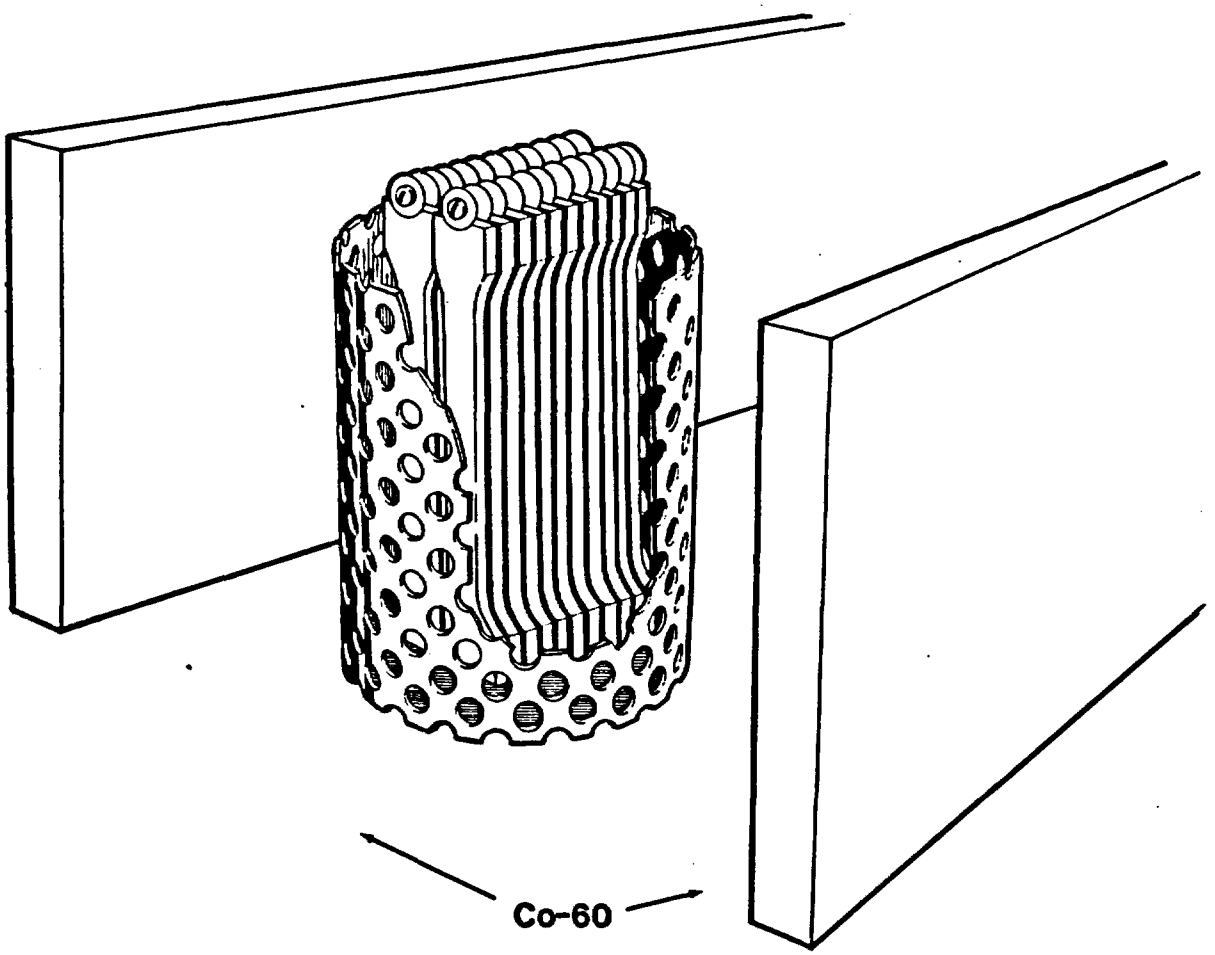


Figure 4.5. Orientation of 82 H5 and 82 H6 Samples During Irradiation

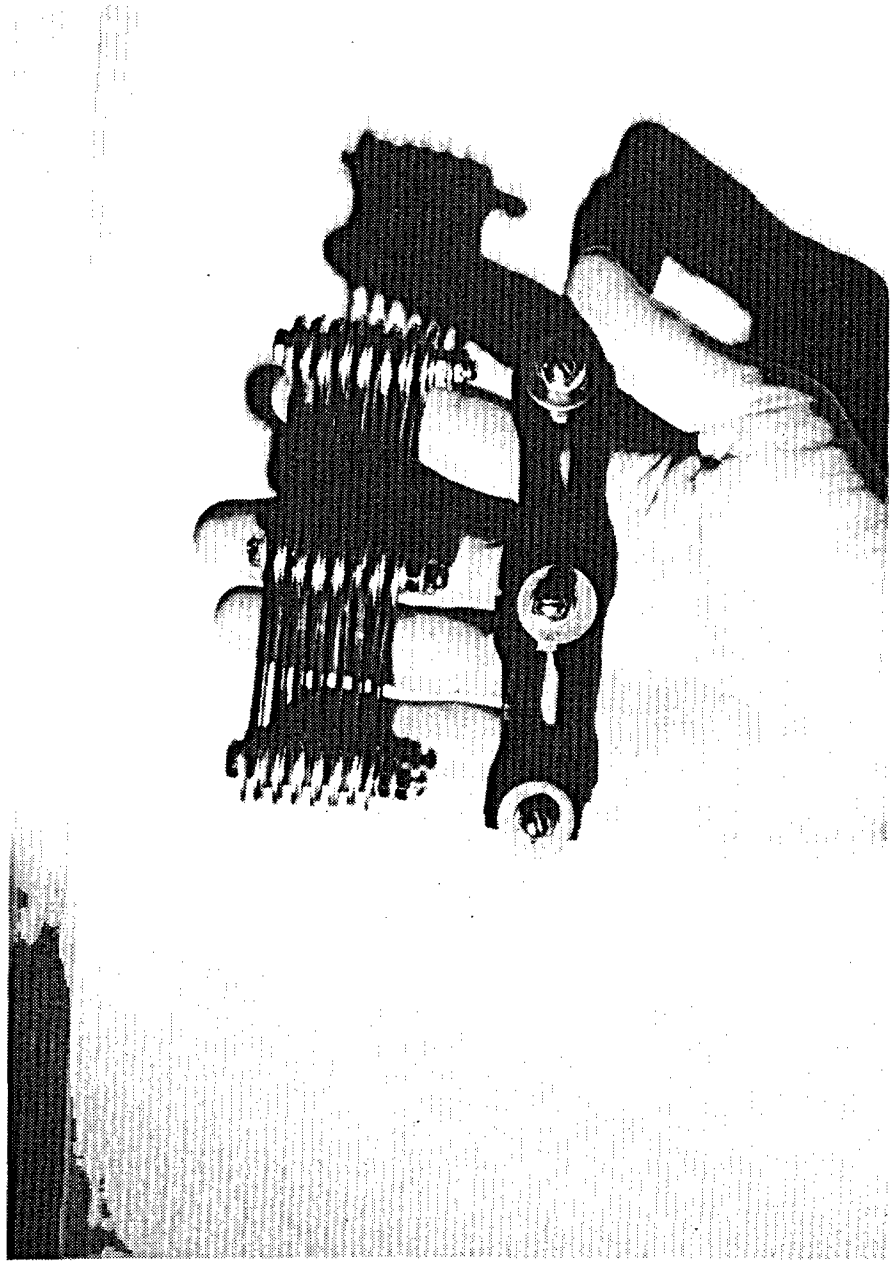


Figure 4.6. Preparation of 82 H3, 82 H4, and 82 G10 Samples for Aging

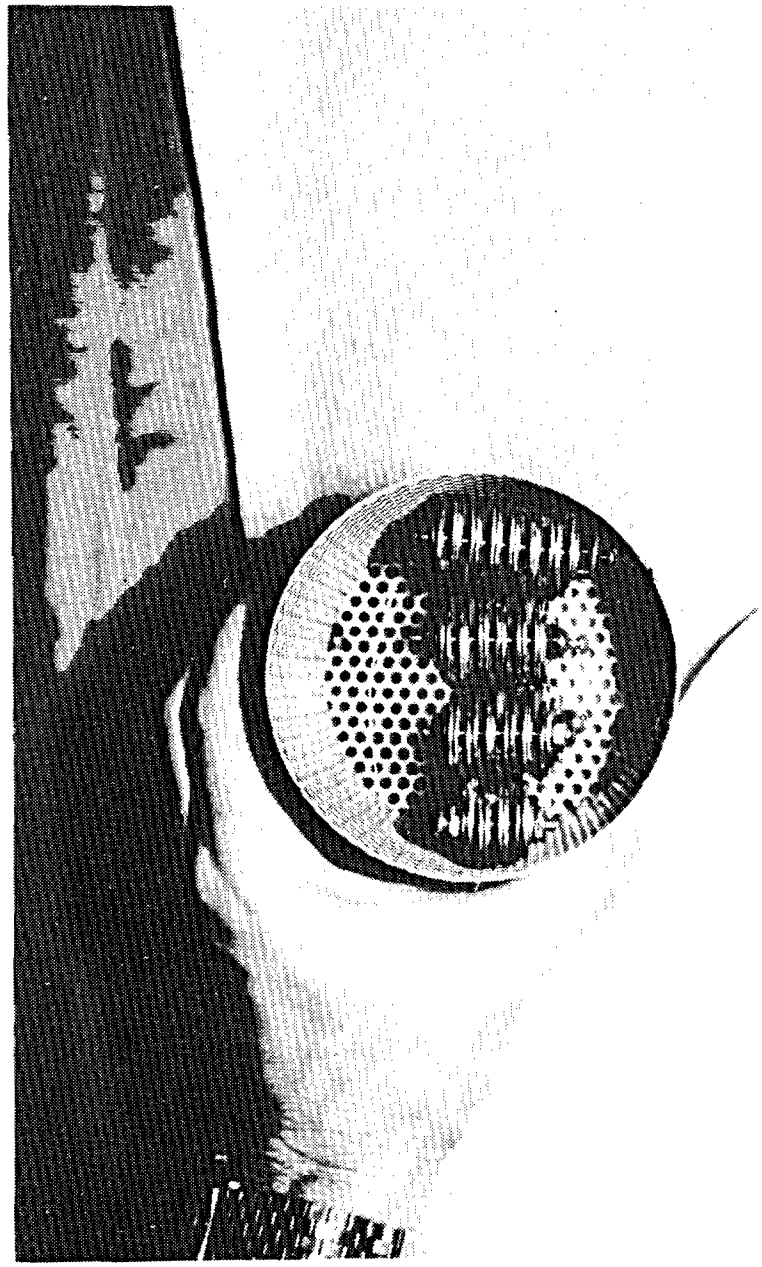


Figure 4.7. 82 H3, 82 H4, and 82 G10 Samples Shown as Prepared to be Inserted into LICA Irradiation Cell

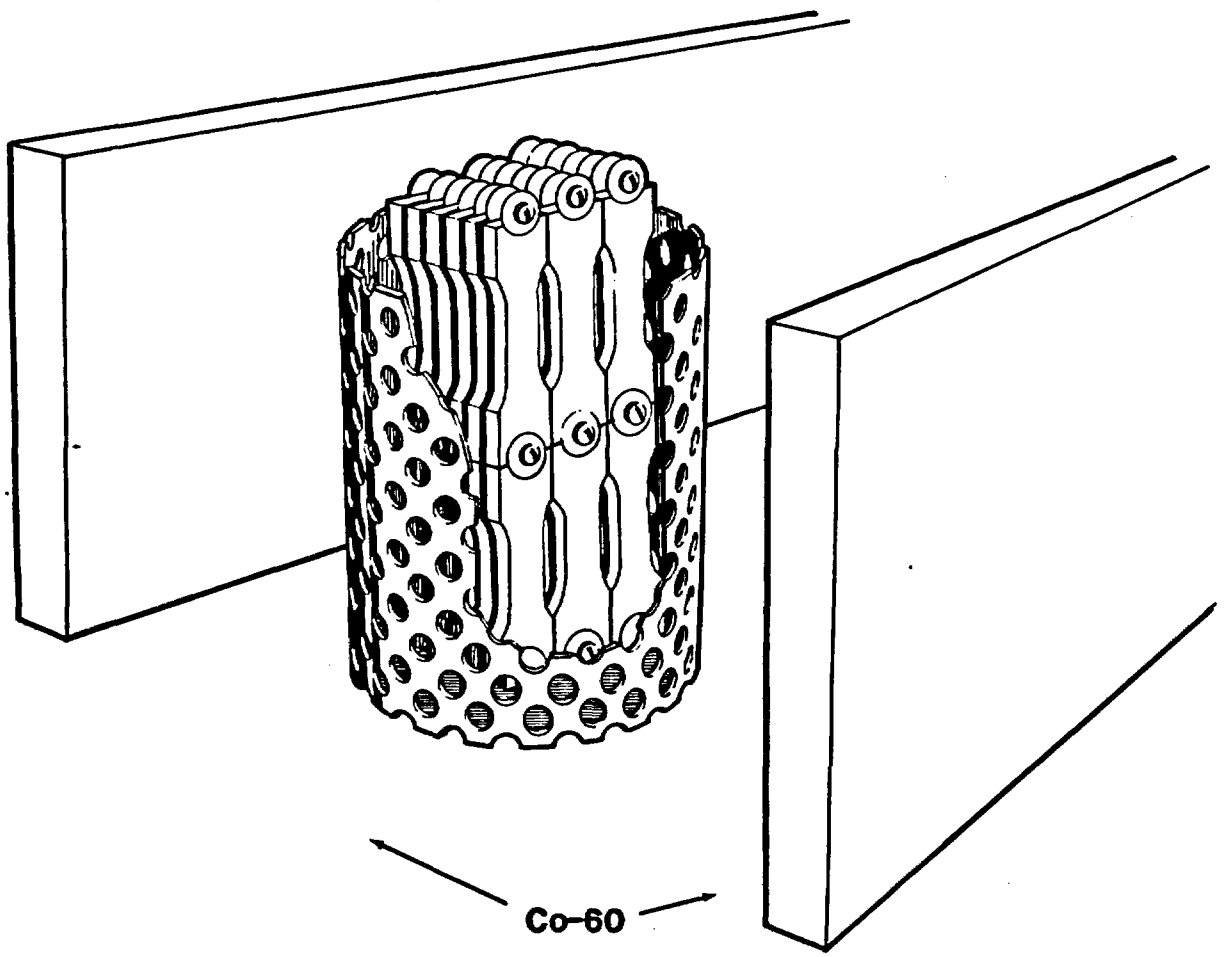


Figure 4.8. Orientation of 82 H3, 82 H4, and 82 G10 Samples During Irradiation

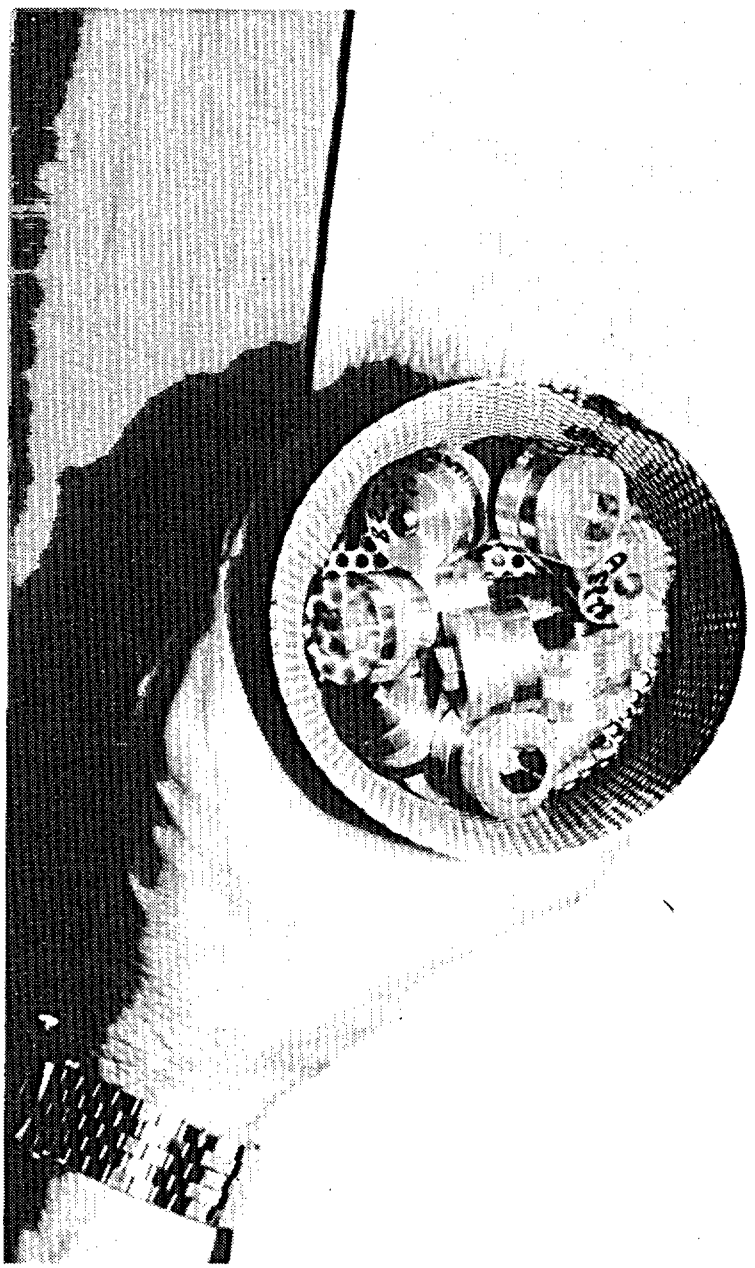


Figure 4.9. 82 J3 Samples Prepared to be Irradiated

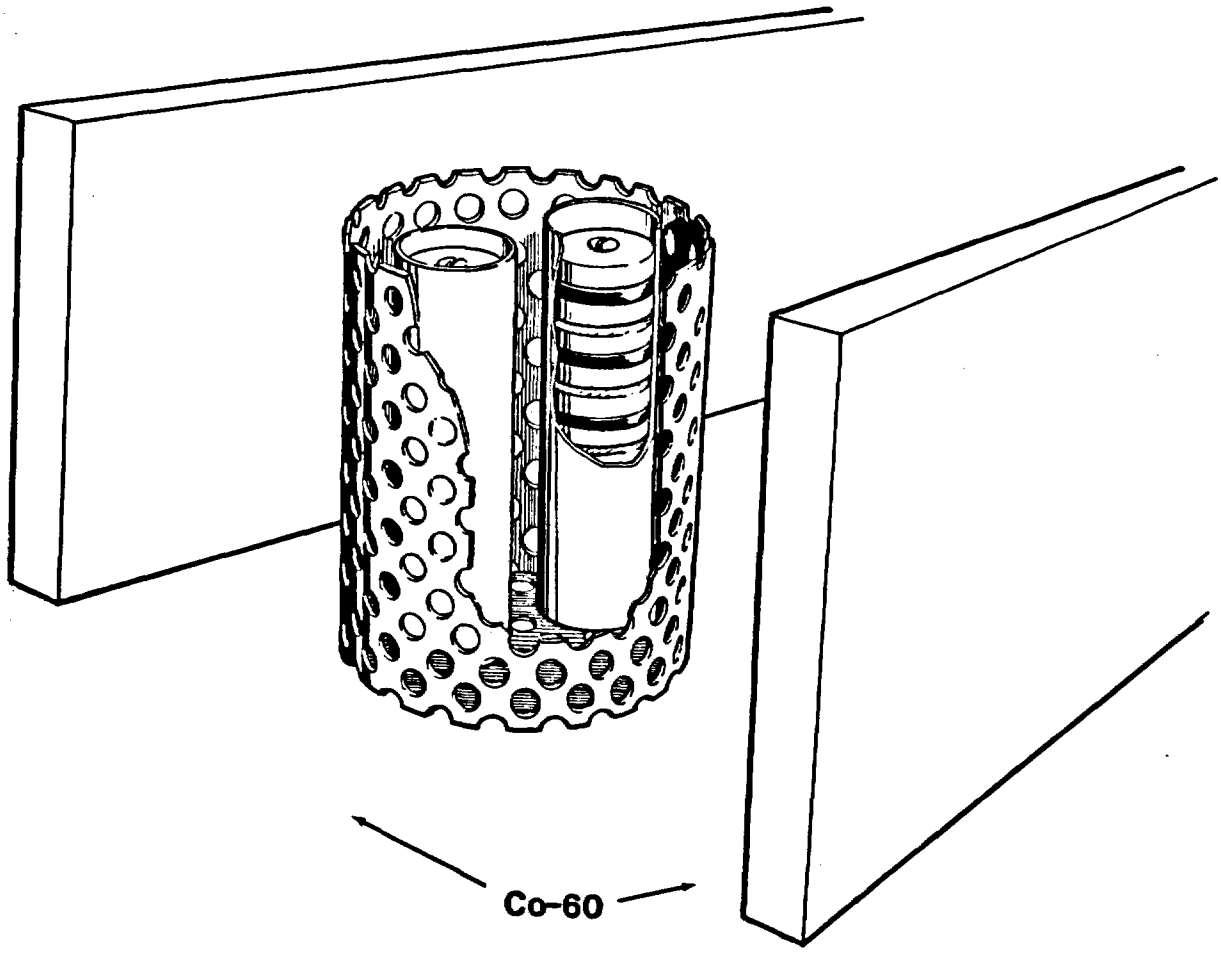


Figure 4.10. Irradiation Aging Configuration Used for 82 J4 Samples

Table 4.5

Accuracy of French Measurements

Measured Parameter	Accuracy
Dimensions	
Dumbbell Size	± 0.1 mm
Insulation Outer Diameter	± 0.5 mm
Insulation Inner Diameter	± 0.3 mm
Wire Thickness	± 0.2 mm
Tensile Strength	
Dumbbells	± 5%
Conductor Insulation	± 15%
Tensile Elongation	
Dumbbells	± 5%
Conductor Insulation	± 5%
Compression Set (Permanent Set)	± 9%

5.0 RESULTS

5.1 U.S. Samples

Sequential exposures to irradiation and thermal environments were employed for aging sequences A, B, C, and D (Section 4.1). Each environmental exposure lasted for ~380 hours (Table 4.1). Thus the total sequential exposure time for both the irradiation and thermal aging environments was ~760 hours. Several times during this 760-hour exposure period, aging was momentarily interrupted to allow for removal of several sample specimens. These specimens were used to determine the degradation history during the aging exposures. In this section we plot tensile property degradation versus exposure time for the CSPE, CPE, EPR 1, EPR 2, XLPO 1, and XLPO 2 materials. For each aging sequence, the first 380 hours of our plots represents one of the environmental stress exposures (irradiation or thermal), while the second 380 hours represents the second environmental stress exposure of the sequence. Aging exposure E was a simultnaeous exposure to irradiation and 120°C thermal environments. It lasted ~380 hours (see Table 4.1). Tensile property degradation during the E aging exposure is also shown in our figures.

5.1.1 CSPE and CPE Jacket Materials

The ultimate tensile properties of CSPE and CPE depended on the order of the sequential exposures. Figures 5.1 and 5.2 illustrate tensile property degradation of CSPE as a function of exposure time to radiation and thermal environments. The R_{70} → 120°C exposure most severely degrades both the ultimate tensile elongation and the ultimate tensile strength. The R_{120} exposure yields comparable results. The 120°C → R_{27} sequence is the least degrading sequence. For CSPE, jacket degradation during sequential sequences depends on whether the irradiation is performed at ambient or 70°C temperatures; 70°C irradiations are more severe.

CPE elongation degradation does not depend on irradiation temperatures (see Figure 5.3). The R → 120 sequences produces more elongation degradation than does the 120 → R sequence or the R_{120} simultaneous exposure. Ultimate tensile strength results, Figures 5.4 and 5.5, indicate that thermal exposures reduce tensile strength while ambient irradiation exposures increase tensile strength. Irradiation exposures at elevated temperatures do not strongly affect CPE's tensile strength properties.

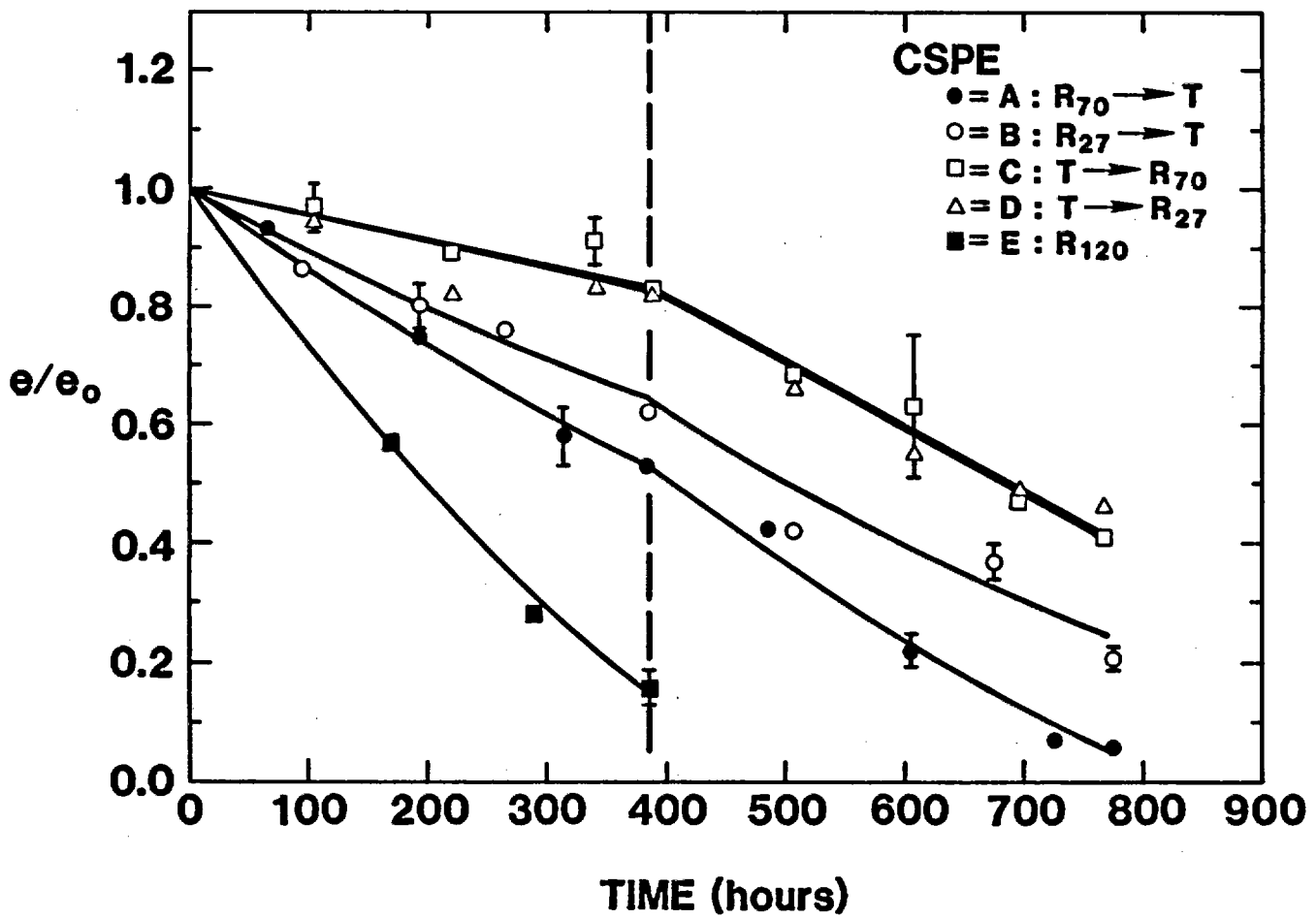


Figure 5.1. Ultimate Tensile Elongation of CSPE in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

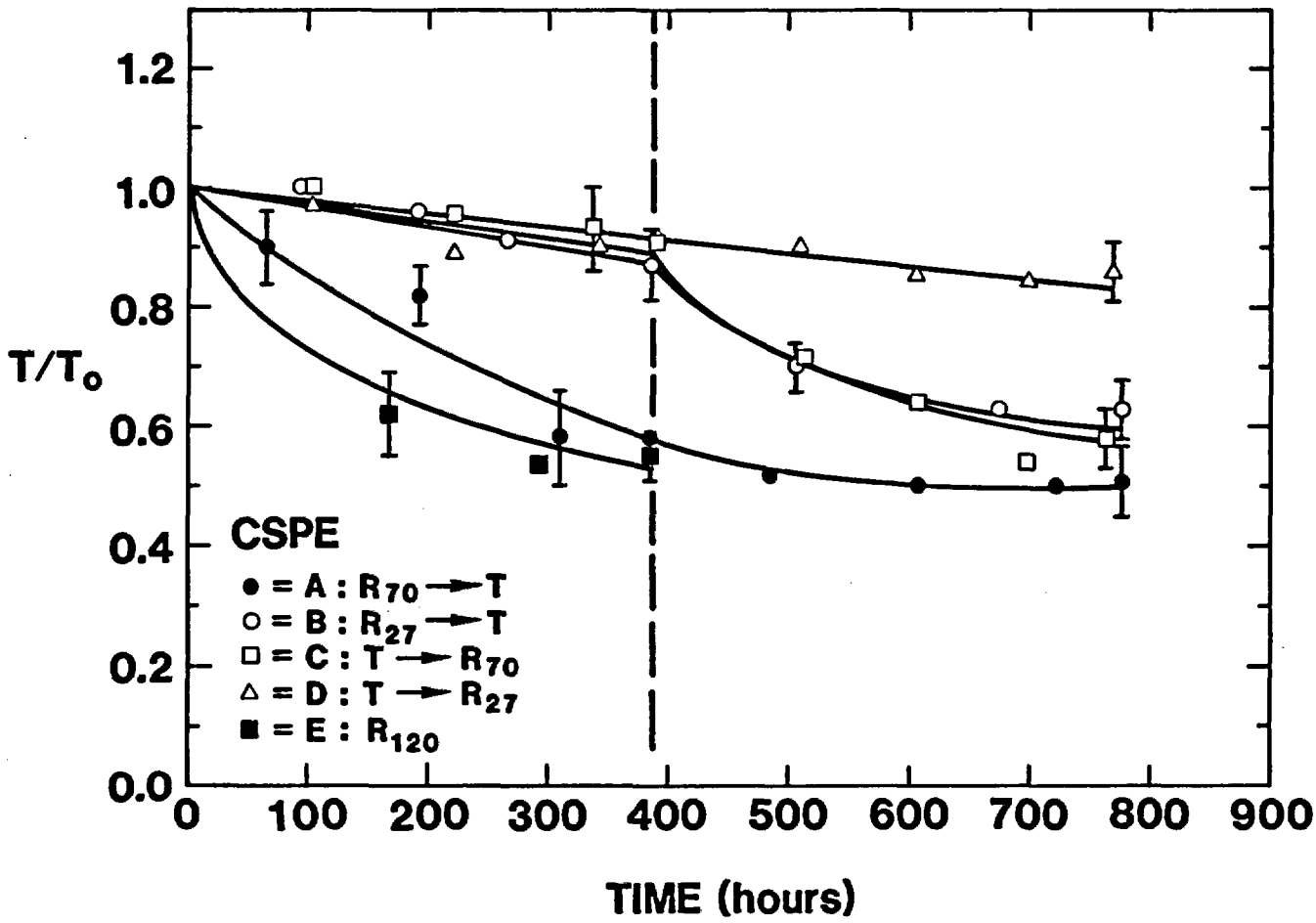


Figure 5.2. Ultimate Tensile Strength of CSPE in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

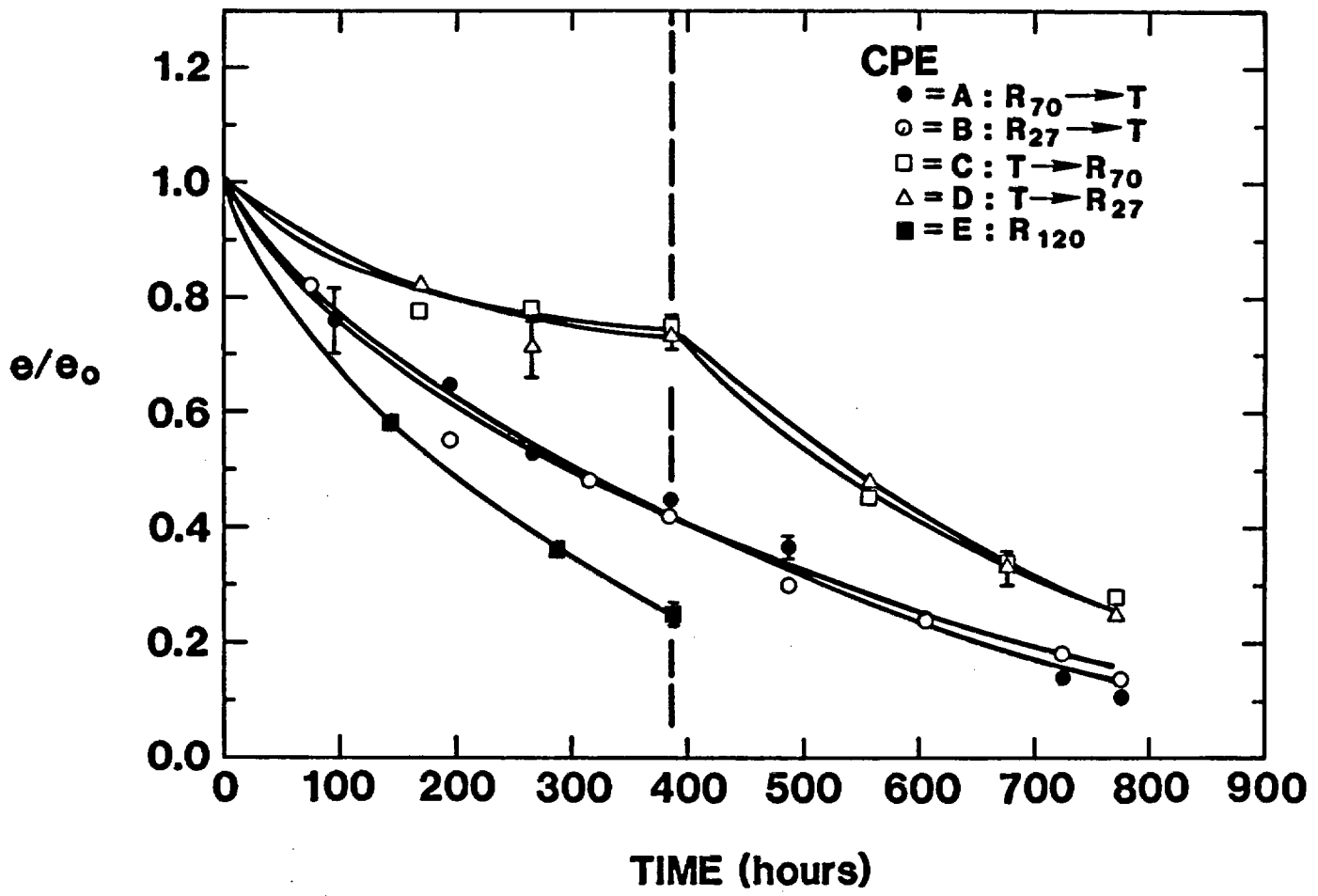


Figure 5.3. Ultimate Tensile Elongation of CPE in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

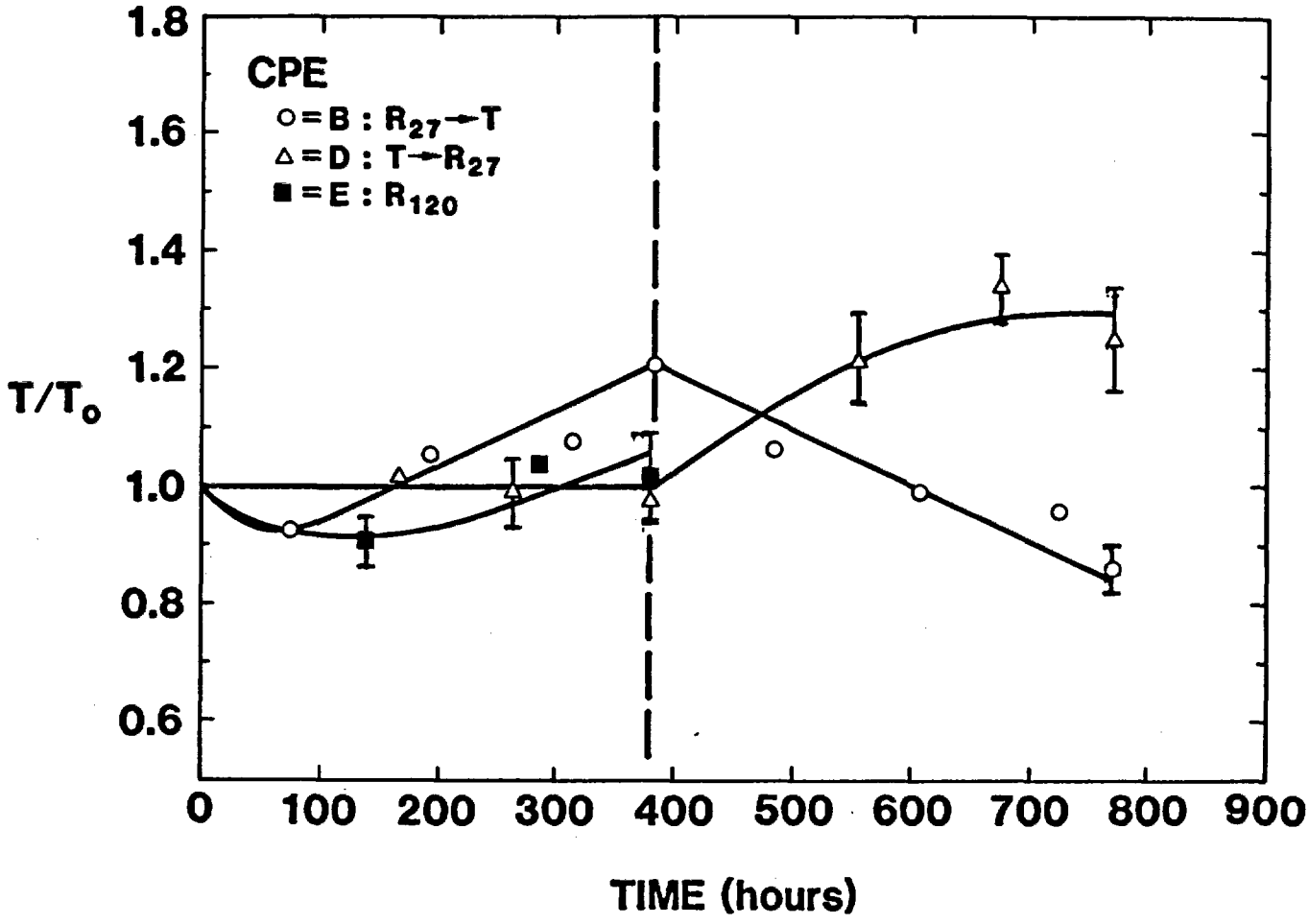


Figure 5.4. Ultimate Tensile Strength of CPE in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

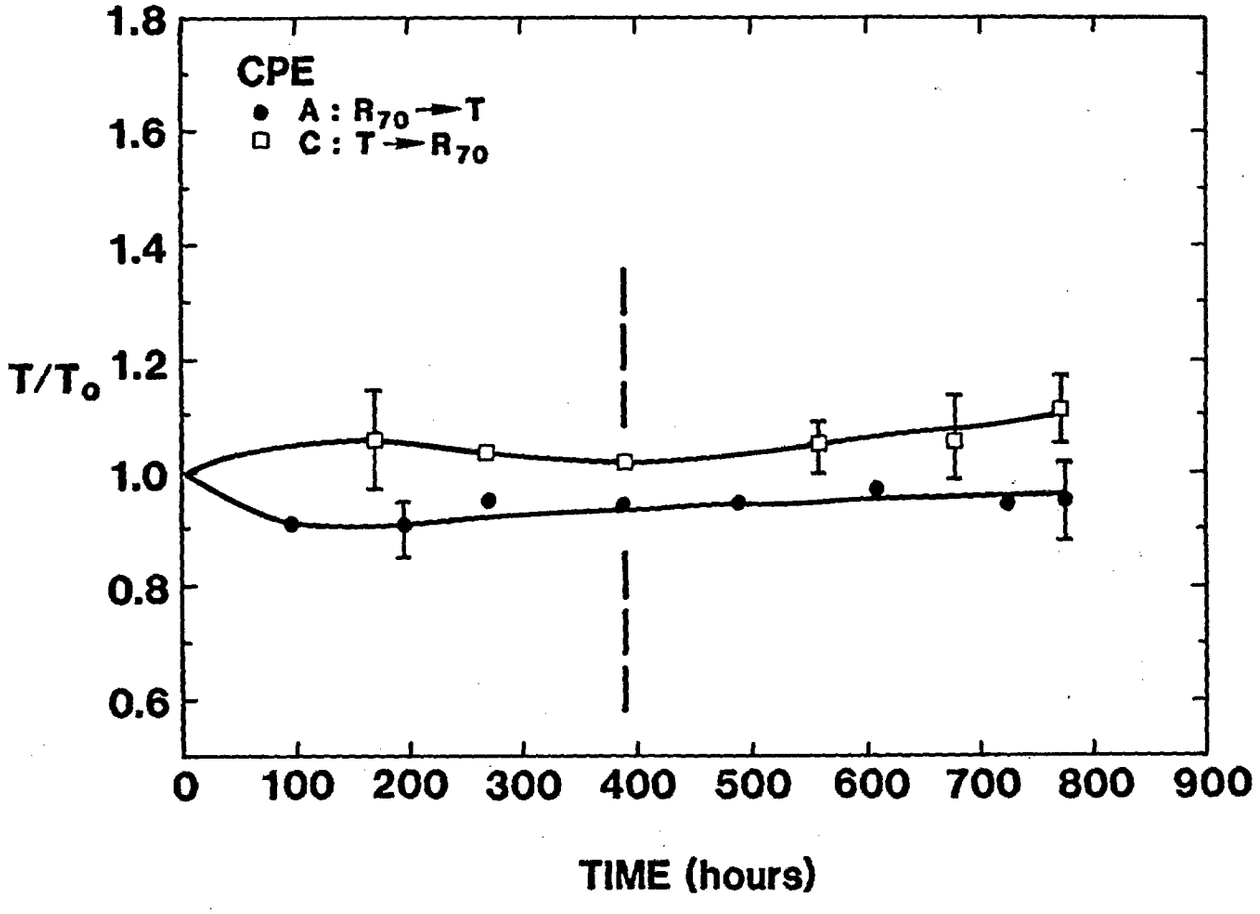


Figure 5.5. Ultimate Tensile Strength of CPE in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

For some CPE samples, irradiations were terminated when our irradiation cells mistakenly filled with water. The exposure sequence was restarted with new samples. Figures 5.6 and 5.7 demonstrate tensile property results for the two sets of exposures: one sequence successfully completed, the other identical sequence terminated prior to completion. The data illustrates the extent of repeatability of results.

5.1.2 EPR Insulation Materials

Ultimate tensile property degradation for EPR 1, a radiation cross-linked FR-EPDM, is illustrated in Figures 5.8 and 5.9. Irradiation reduces both the ultimate tensile elongation and ultimate tensile strength. Degradation is worst for the R_{27} exposure, with lesser degradation noted for the R_{70} and R_{120} irradiations. The opposite effect is noted for EPR 2, a chemically cross-linked FR-EPDM (Figures 5.10 and 5.11). For this material, the R_{120} and R_{70} irradiations more degrade the EPR tensile properties than does the R_{27} exposure. When irradiation and thermal stresses are applied sequentially, neither EPR 1 nor EPR 2's ultimate tensile elongation properties depended on the sequential ordering. EPR 1 and EPR 2 tensile strength properties are also not affected by sequential ordering.

5.1.3 XLPO Insulation Materials

XLPO 1 and XLPO 2 tensile properties are illustrated in Figures 5.12-5.15. Only the ultimate tensile strength of XLPO 2 was sensitive (mildly) to sequential ordering of radiation and thermal stresses. The R → T sequences increased the tensile strength more than did the T → R sequences.

5.1.4 TEFZEL Materials

Tables 5.1 and 5.2 present bend test results for TEFZEL 1 and 2 respectively. Four specimens (two white and two colored) were tested for each aging condition. If all four samples passed the bend test, a P is shown in the table. Those table locations where samples failed the bend test are marked with an F. TEFZEL degradation is clearly dependent on irradiation temperature. We experienced no bend test failures for TEFZEL specimens aged using ambient temperature irradiations. We did observe bend test failures when aging procedures employed either 70°C or 120°C irradiations. For both TEFZEL materials, the 120°C -> R_{70} sequence was more severe than the R_{70} -> 120°C exposure.

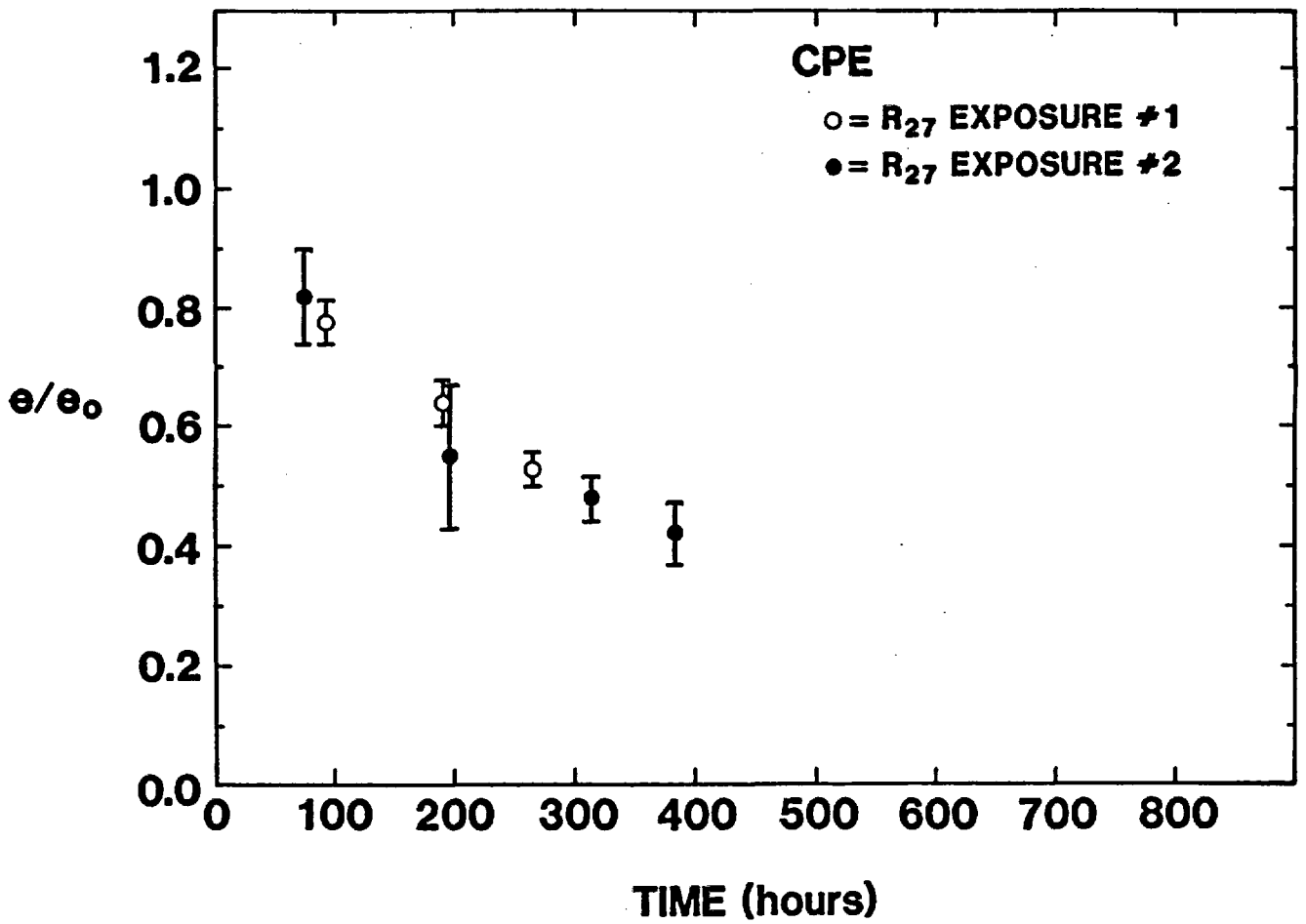


Figure 5.6. Repeatability of Ultimate Tensile Elongation Results for CPE During Two R_{27} Exposures

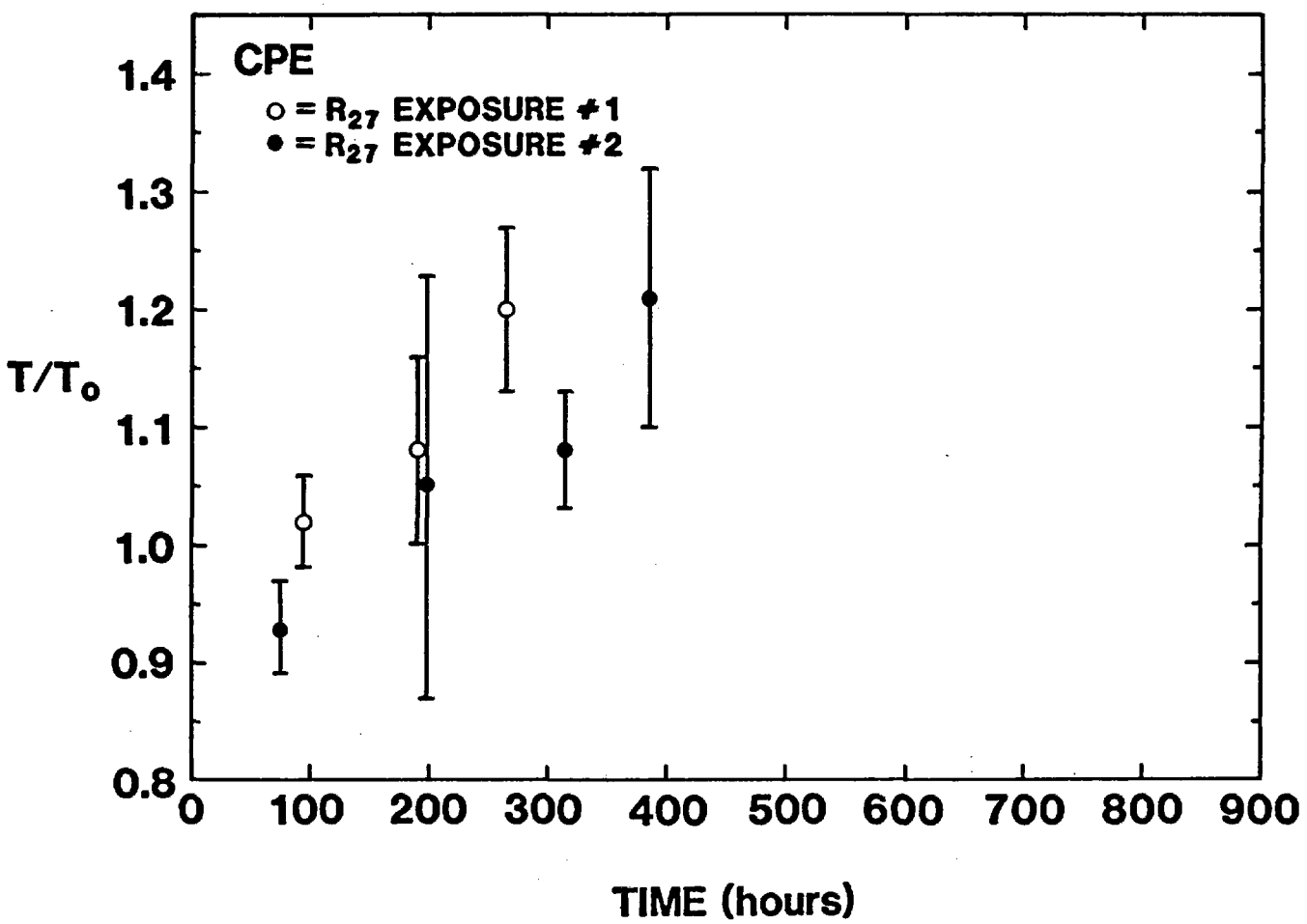


Figure 5.7. Repeatability of Ultimate Tensile Strength Results for CPE During Two R_{27} Exposures

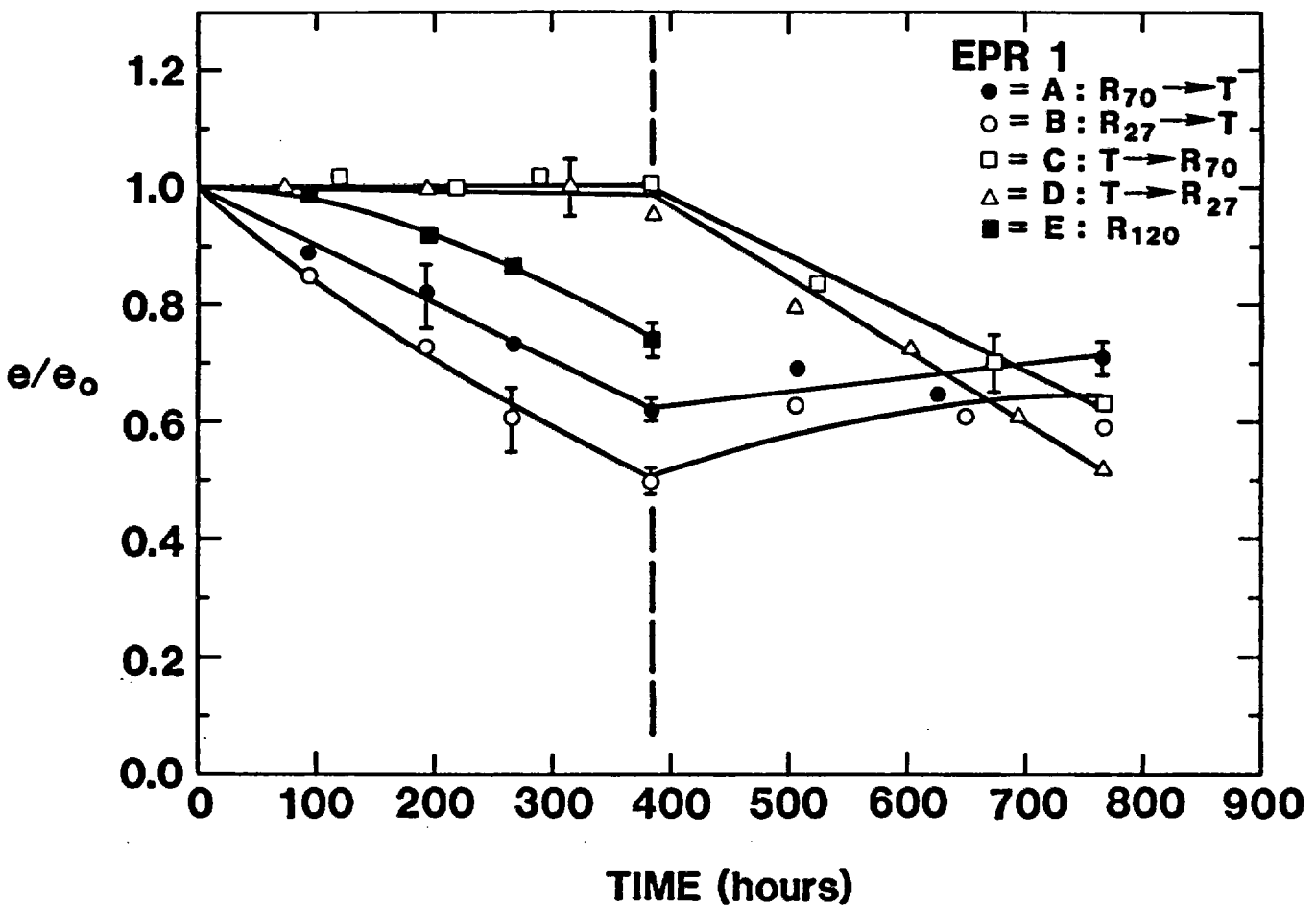


Figure 5.8. Ultimate Tensile Elongation of EPR 1 in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

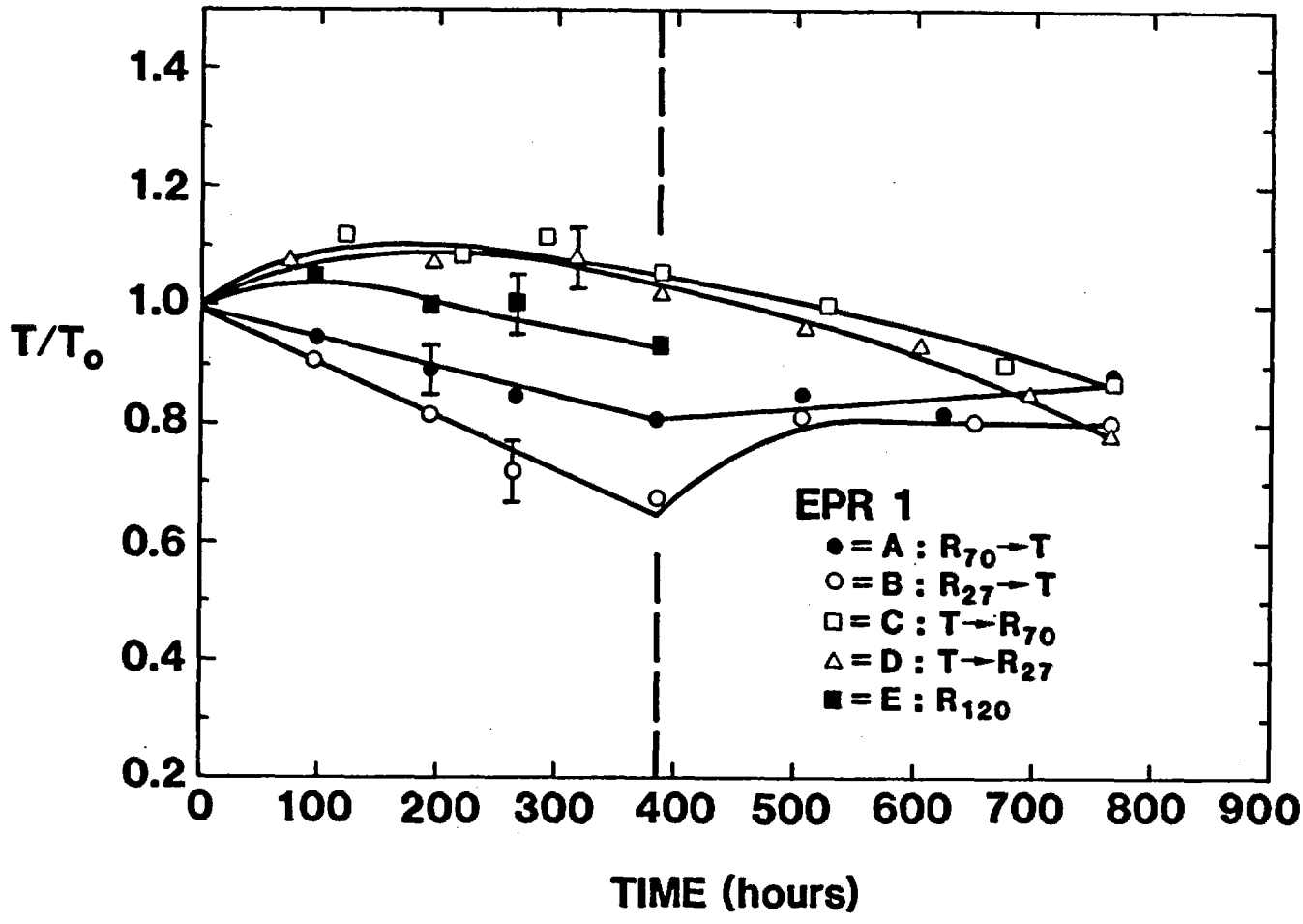


Figure 5.9. Ultimate Tensile Strength of EPR 1 in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

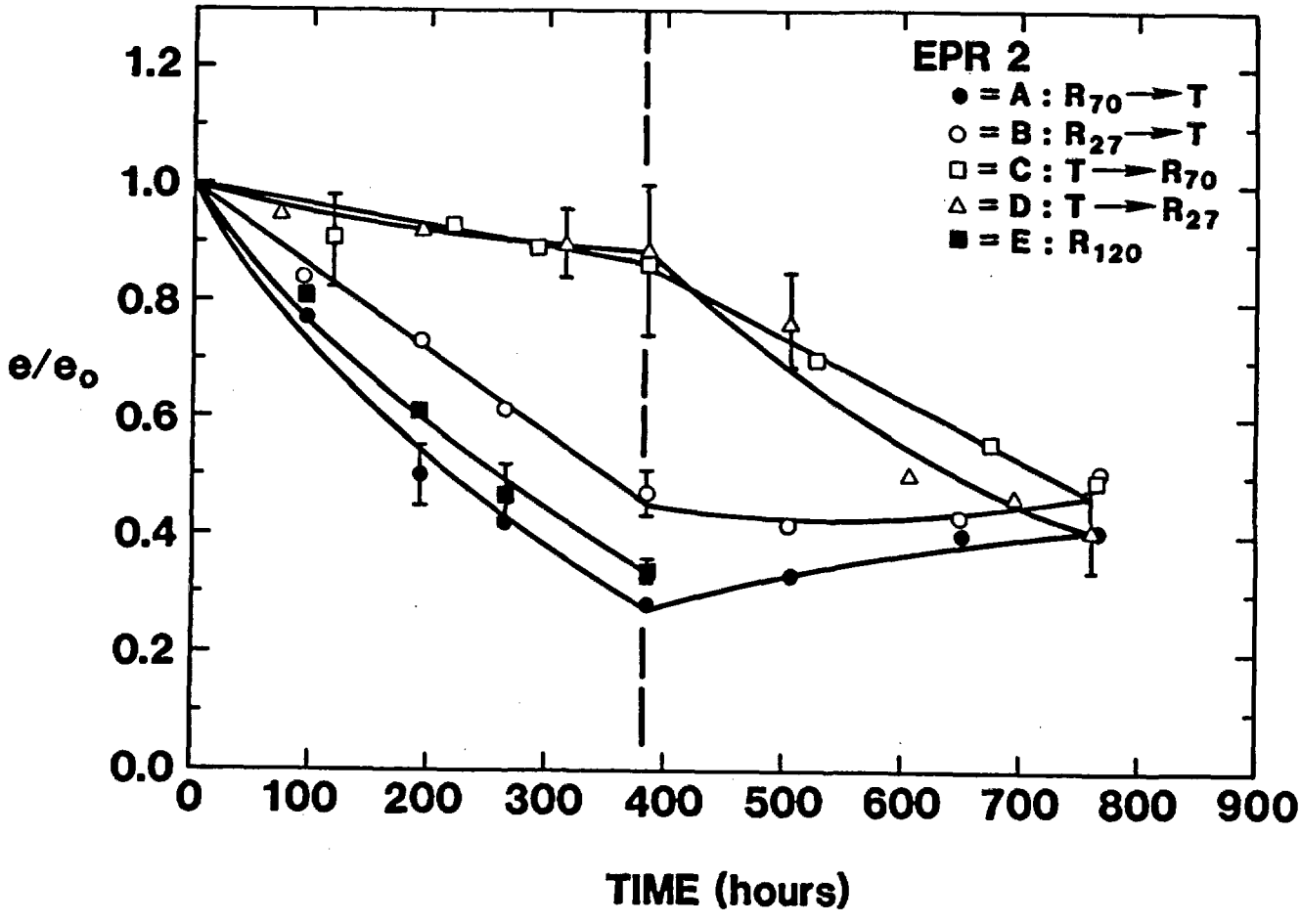


Figure 5.10. Ultimate Tensile Elongation of EPR 2 in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

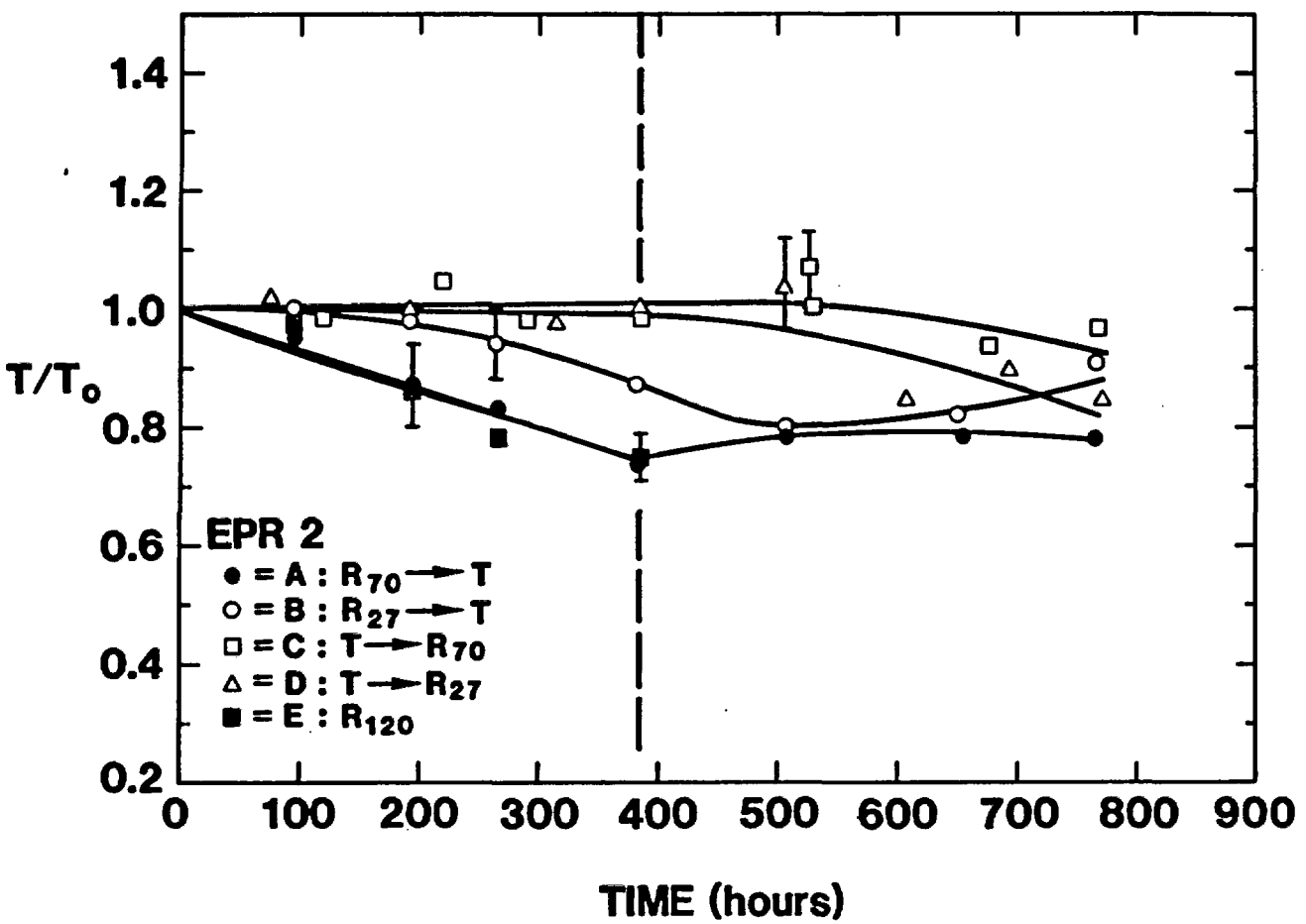


Figure 5.11. Ultimate Tensile Strength of EPR 2 in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

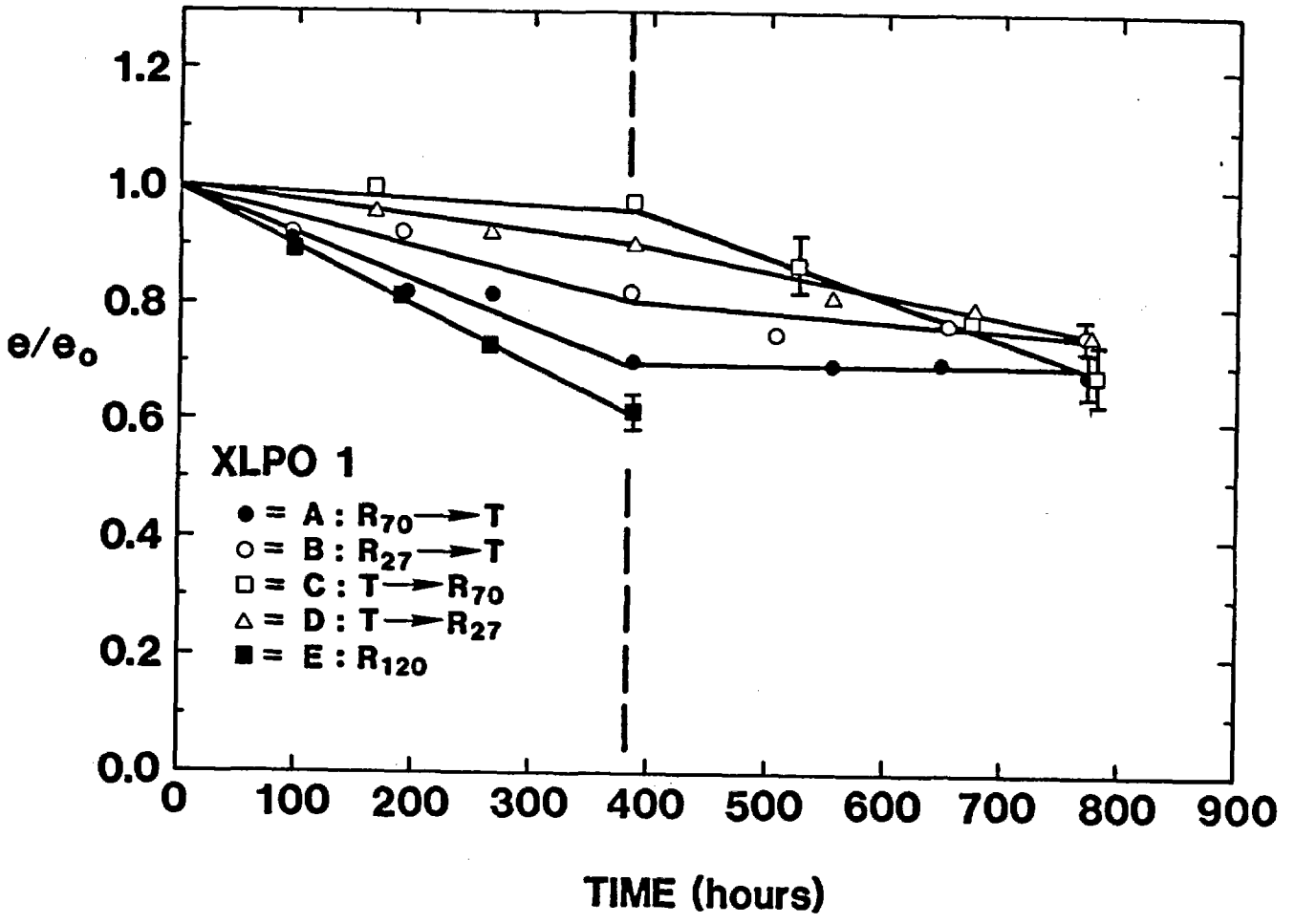


Figure 5.12. Ultimate Tensile Elongation of XLPO 1 in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

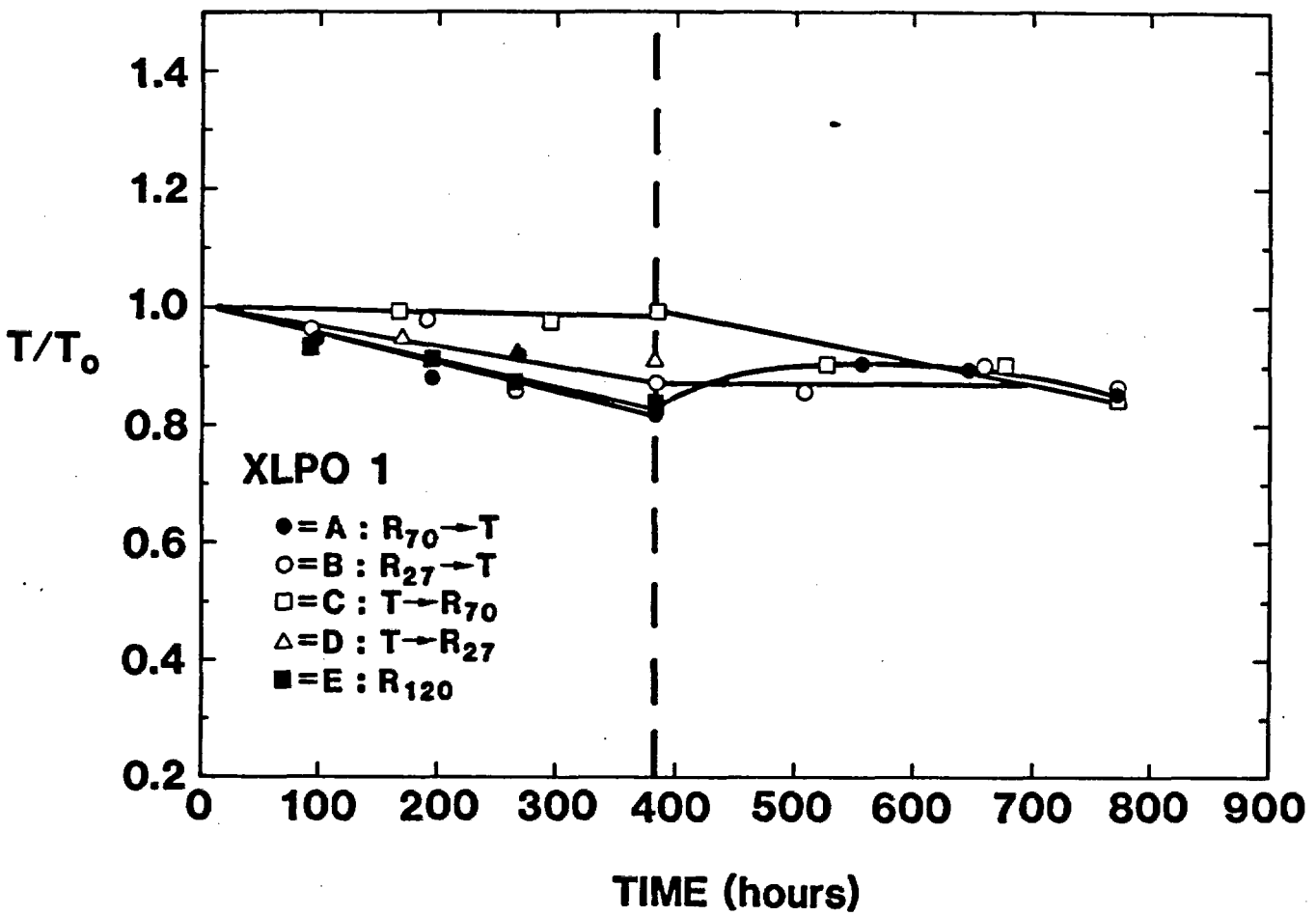


Figure 5.13. Ultimate Tensile Strength of XLPO 1 in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

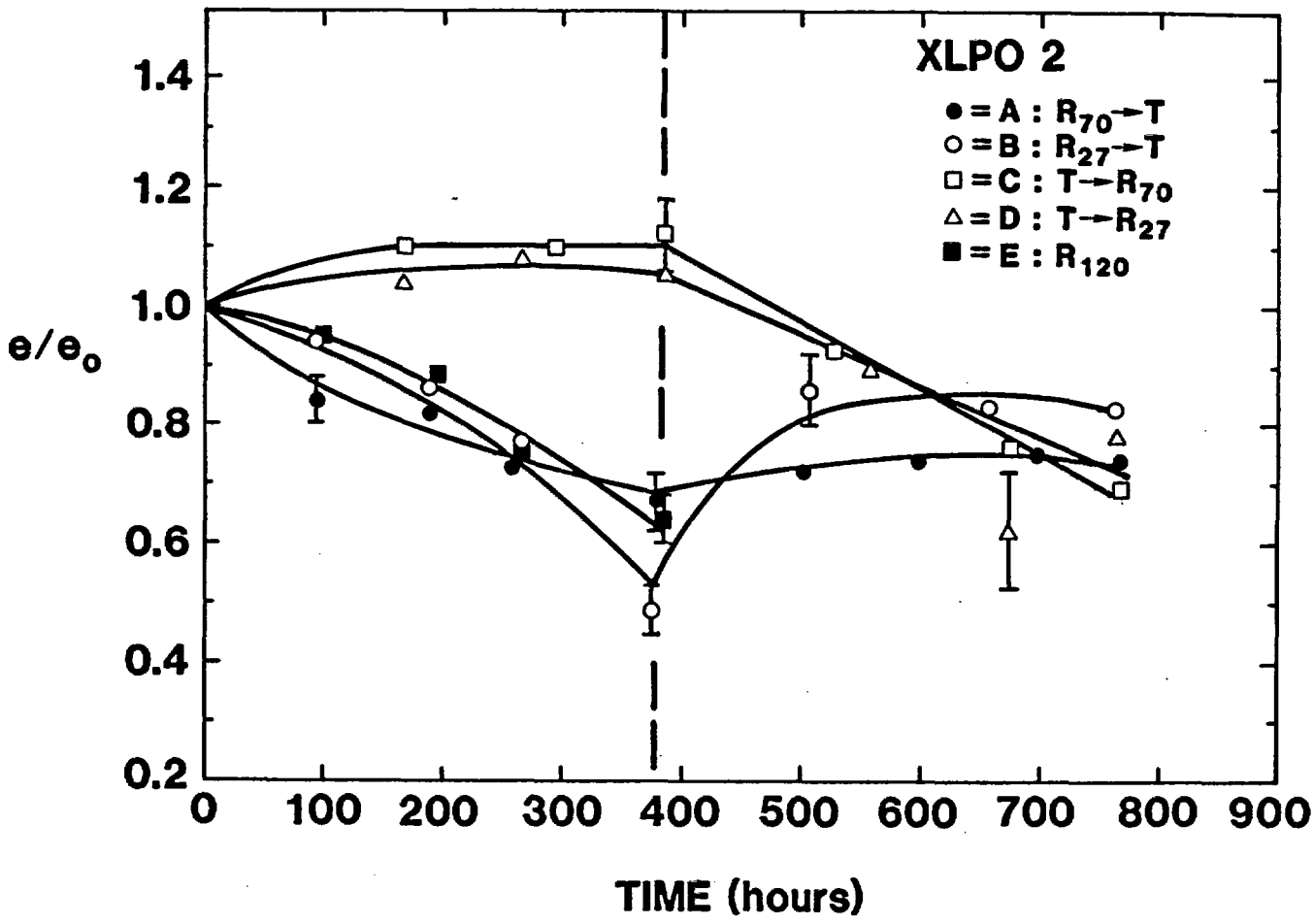


Figure 5.14. Ultimate Tensile Elongation of XLPO 2 in Various Environments. Sample tensile elongation divided by initial (unaged) elongation is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

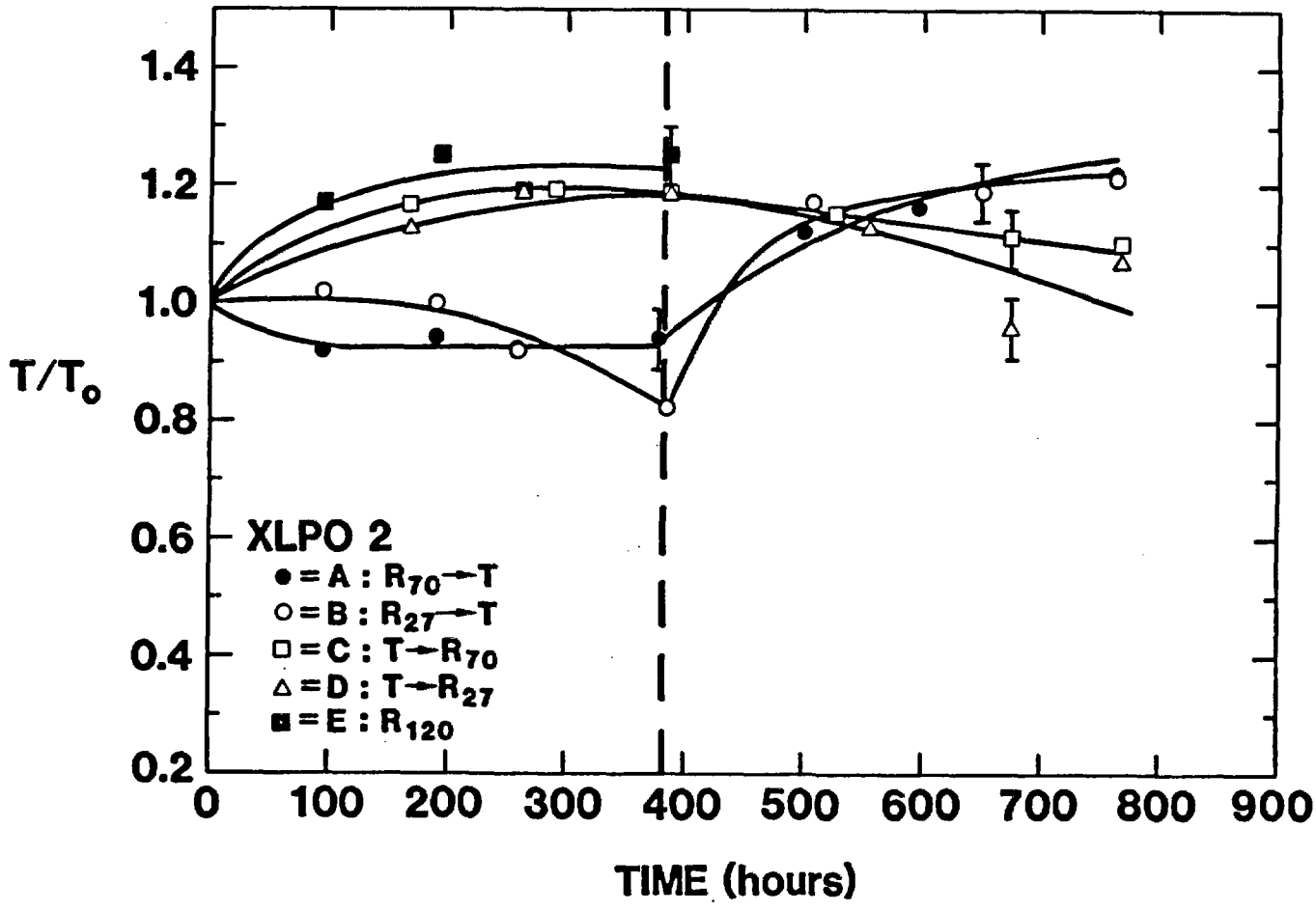


Figure 5.15. Ultimate Tensile Strength of XLPO 2 in Various Environments. Sample tensile strength divided by initial (unaged) strength is plotted versus aging time. Each portion of the sequential exposures lasted ~380 h.

Table 5.1

Bend Test Results for TEFZEL 1

Bend Radius/Insulation Radius

		75X	69X	56X	50X	44X	31X	22X	11X	6X
F:	Unaged	P	P	P	P	P	P	P	P	P
B:	$R_{27} \rightarrow 120$	P	P	P	P	P	P	P	P	P
D:	$120 \rightarrow R_{27}$	P	P	P	P	P	P	P	P	P
A:	$R_{70} \rightarrow 120$	P	P	P	P	P	P	F	F	F
C:	$120 \rightarrow R_{70}$	P	P	P	P	P	F	F		
E:	R_{120}	P	P	P	P	F	F		F	

P = Pass Bend Test

F = Fail Bend Test

Table 5.2

Bend Test Results for TEFZEL 2

Bend Radius/Insulation Radius

		75X	69X	56X	50X	44X	31X	22X	11X	6X
F:	Unaged	P	P	P	P	P	P	P	P	P
B:	$R_{27} \rightarrow 120$	P	P	P	P	P	P	P	P	P
D:	$120 \rightarrow R_{27}$	P	P	P	P	P	P	P	P	P
A:	$R_{70} \rightarrow 120$	P	P	P	P	P	P	F	F	
C:	$120 \rightarrow R_{70}$	P	P	P	F		F			
E:	R_{120}	P	P	P	P	P	P	F	F	

P = Pass Bend Test

F = Fail Bend Test

5.1.5 Compression Set Tests

Our compression set tests employed compression samples with thicknesses ~.18 cm. As mentioned in Section 4, this thickness represents actual use conditions but produces large uncertainties in the value of compression set, C:

$$C = \frac{t_o - t_i}{t_o - t_n}$$

where t_o = original thickness

t_i = final thickness

t_n = compressed thickness.

We predict our absolute uncertainties for C to exceed .10. For all five materials, our uncertainty exceeds the compression set variation caused by aging differences. (See Table 5.3.)

5.2 French Samples

Each French aging sequence included two environmental stress exposures - an irradiation exposure and a thermal aging exposure. Mechanical tensile tests and compression set measurements were performed at the completion of each exposure. Results are presented in Table 5.4.

Measurement results are shown for each material in Figures 5.16 through 5.24. Measurement values are normalized with respect to those relating to unaged control specimens of the same material (blank samples U). For each aging sequence, usually two data values are plotted. The first value reflects test results at completion of the first exposure of the aging sequence, while the second value indicates results at the completion of the aging exposure. For a few test conditions, only data at the completion of the sequence were obtained. This single data value is plotted.

Table 5.3

U.S. Compression Set Test Results

	EPR A	EPR B	BUNA A	SILICONE	VITON
Unaged (#38)	.09 ± .01	.11 ± .02	.07 ± .01	.07 ± .03	.17 ± .01
Partially Aged:					
R_{27} (#12)	.54 ± .04	.78 ± .03	.48 ± .01	.82 ± .02	.80 ± .05
120 (#37)	.31 ± .07	.73 ± .05	.39 ± .02	.17 ± .01	.27 ± .03
Aged:					
R_{120}(#32)	.74 ± .03	1.0	.68 ± .01	1.0	.97 ± .02
$R_{27} \rightarrow 120$					
#13	.67 ± .02	.96 ± .03	.63 ± .01	.83 ± .02	.88 ± .01
#14	.67 ± .03	.98 ± .03	.59 ± .02	.86 ± .01	.89 ± .01
$120 \rightarrow R_{27}$					
#20	.68 ± .04	.91 ± .02	.68 ± .02	.72 ± .01	.80 ± .01
#50	.79 ± .06	.95 ± .01	.71 ± .02	.81 ± .01	.83 ± .02

*Errors Reflect ± σ for Four Samples. Absolute Error is ± .10.

Table 5.4

Tensile and Compression Set Results for French Samples

MATERIAL	Θ	TESTS**	U		A***		B***		C***		D***
Elastomeric Materials:											
PRC	140	T	1.3	1.1	1.2	1.1	0.9	1.1	1.1	1.0	1.0
82 I1		e	370	332	267	264	30	332	269	245	43
EPDM	140	T	0.6	0.6	0.3	0.4	0.4	0.6	0.5	0.4	0.2
82 I2		e	240	241	82	124	1.5	241	118	135	0.5
Fire-proof EPDM	140	T	0.6	0.6	0.7	0.7	0.4	0.6	0.8	0.7	0.4
82 I9		e	245	222	83	113	32	222	84	107	34
EPR	140	T	1.7	1.5	1.3	1.5	0.8	1.5	1.4	1.6	0.8
82 H4		e	174	186	99	124	81	186	93	116	73
EPR 82 J4	140	Compression Set	8.2	--	67	--	71	--	58	--	57
VAMAC	120		1.8	1.7	1.8	1.8	1.8	1.7	1.8	1.8	1.8
82 H3			312	308	245	248	238	308	207	217	215
VAMAC 82 J3	120	Compression Set	9.6	--	56	--	71	--	68	64	76
HYPALON	140	T	0.9	0.7	0.7		0.6	0.7	0.7	0.8	0.7
82 G10		e	422	332	249		205	332	204	284	214
Thermoplastic material:											
PPS	160	T	8.7		8.0	8.4	8.1		8.0	8.4	7.8
82 H6		e	0.7		0.6	0.7	0.7		0.6	0.7	0.6
Thermoset material:											
Polydial-lylphtalate	160	T	4.6	4.5	4.8	4.4	5.0	4.5	4.8	4.6	4.7
82 H5		e	0.3	0.4	0.3	0.3	0.3	0.4	0.3	0.4	0.3

*Code = U = Blank sample
A = Thermal aging at Θ°C, followed by irradiation at 70°C
B = Irradiation at 70°C, followed by thermal aging at Θ°C
C = Thermal aging at Θ°C, followed by irradiation at 27°C
D = Irradiation at 27°C, followed by thermal aging at Θ°C
Θ = Thermal aging temperature

** T = ultimate tensile strength (Units = 10 MPa)
e = ultimate tensile elongation (units = %)

*** First column = Results at completion of first exposure of the sequence
Second column = Results at completion of second exposure of the sequence

5.2.1 PRC (82 I1)

Figure 5.16 illustrates the normalized tensile properties for chemically cross-linked polyethylene (PRC). The order of aging exposures does not seem to affect the tensile strength. The deviation of the measured data from the average value is ± 10 percent, which is not significant.

However, tensile elongation is influenced by sequential exposure order to a much greater extent than the tensile strength. The PRC material is strongly degraded by the irradiation then thermal exposure sequences (sequences B and D), whereas degradation is slight for the thermal exposure then irradiation sequences (sequences A and C). In all cases, irradiation at 70°C or 27°C causes identical changes in tensile strength or tensile elongation, with or without a prior thermal exposure at 140°C.

5.2.2 EPDM (82 I2 and 82 I9)

Samples 82 I9 are fire-proof EPDM material, whereas the 82 I2 samples are not. Figure 5.17 illustrates measurement results for 82 I2, while Figure 5.18 presents results for 82 I9.

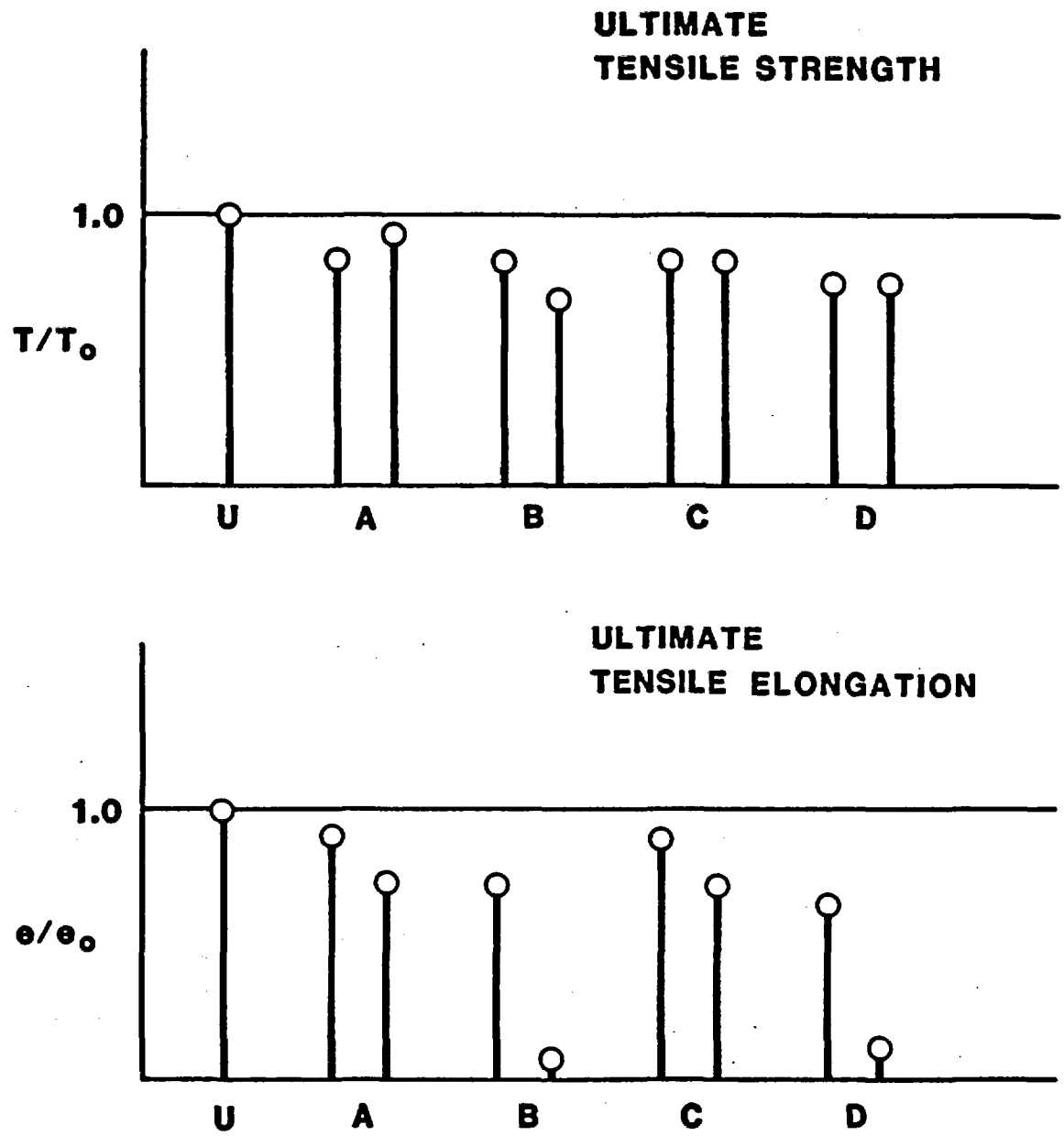
5.2.2.1 EPDM (82 I2)

When irradiation is performed at 70°C, the order of aging exposures does not affect the tensile strength (within measurement accuracy of ± 15 percent - see Table 4.5). At 27°C however, the irradiation followed by thermal exposure sequence (exposure sequence D) degrades the tensile strength more than does the thermal followed by irradiation sequence (exposure sequence C). Hence for tensile strength behavior of the 82 I2 material, irradiation temperature is important.

Ultimate tensile elongation is more degraded by the irradiation followed by thermal exposure sequences (sequences B and D).

5.2.2.2 Fire-proof EPDM (82 I9)

Both thermal followed by irradiation sequences (A and C) increased the tensile strength, whereas both irradiation followed by thermal sequences (B and D) decreased the tensile strength. Interestingly, for both of these latter sequences (B and D), the irradiation exposures at 70°C and 27°C increased the tensile strength, contrary to the behavior of the 82 I2 EPDM samples when irradiated. For all sequences, elongation was decreased. The material appears to become reticulated during aging exposures.



U = UNAGED

A = 140→R_{70}

B = R_{70}→140

C = 140→R_{27}

D = R_{27}→140

Figure 5.16. Ultimate Tensile Properties for PRC (82 I1)

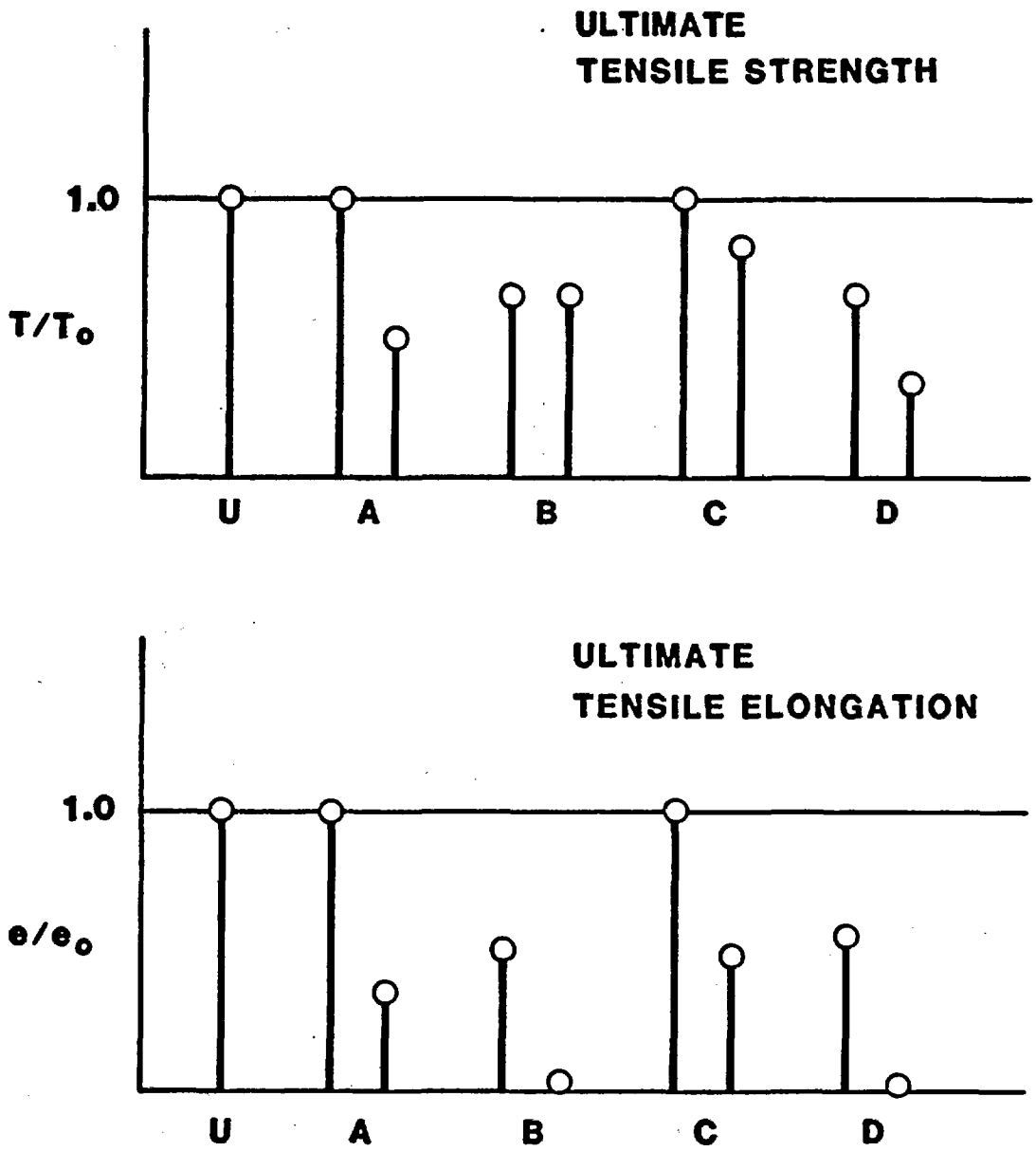
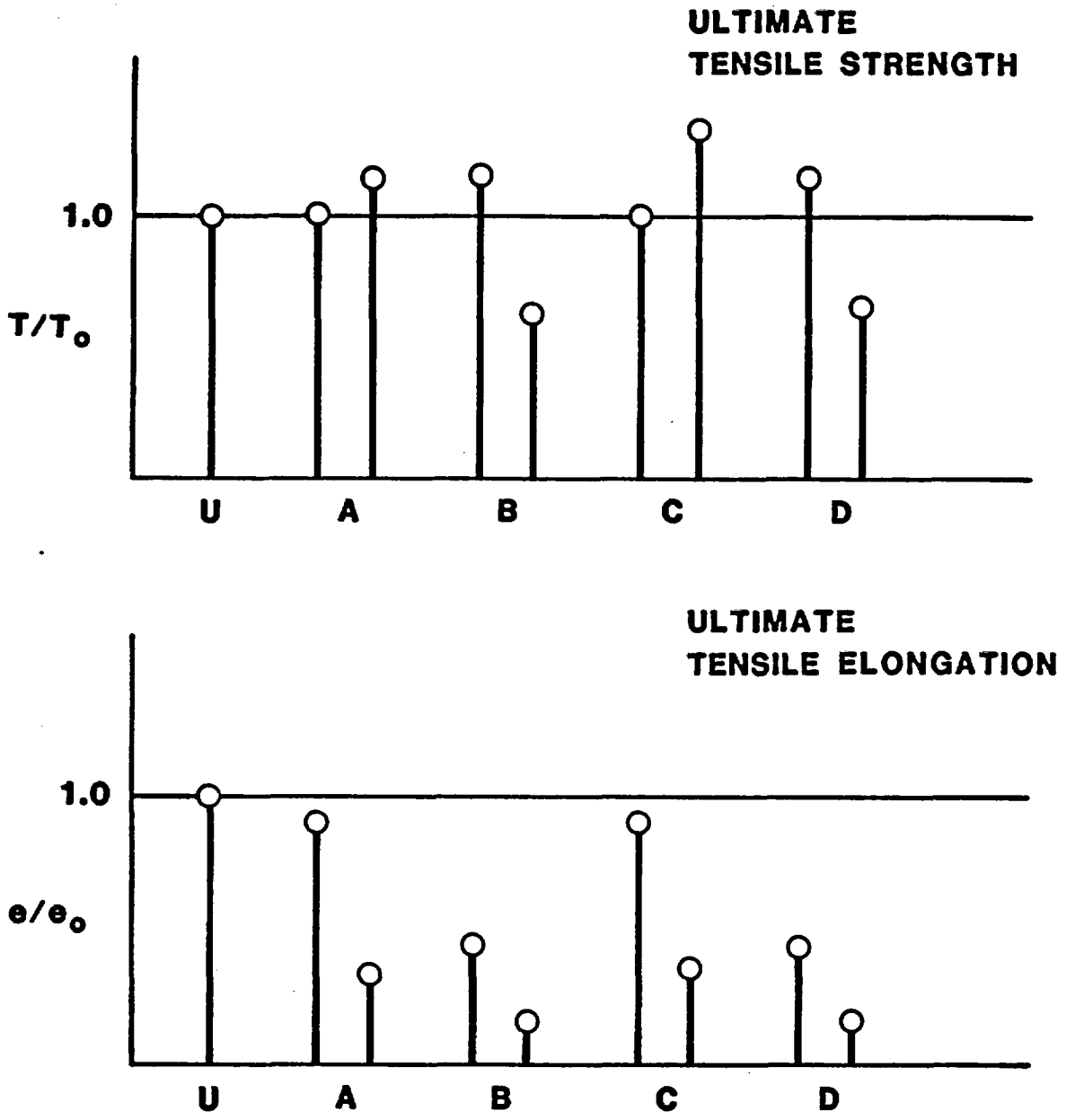


Figure 5.17. Ultimate Tensile Properties for EPDM (82 I2)



U = UNAGED

A = 140→R_{70}

B = R_{70}→140

C = 140→R_{27}

D = R_{27}→140

Figure 5.18. Ultimate Tensile Properties for Fire-proof EPDM (82 I9)

5.2.3 EPR (82 H4 and 82 J4)

The same EPR material was used to make 3 mm-thick sheets of EPR and also O-ring seals. Dumbbells (82 H4) as shown in Figure 2.1 were cut from the sheets and used for ultimate tensile strength and ultimate tensile elongation measurements. O-ring seals (82 J4) with an inner diameter of 12 mm and an outer diameter of 17 mm were supplied by the same manufacturer as the sheet EPR material. These were used for compression (permanent) set measurements. Ultimate tensile measurement results for the 82 H4 samples are shown in Figure 5.19. Permanent set after compression measurements for the 82 J4 samples are illustrated in Figure 5.20.

For the 82 H4 tensile specimens, irradiation at 27°C and at 70°C have the same effect on ultimate tensile properties. Identical changes in tensile strength and tensile elongation were observed for the two thermal followed by irradiation sequences (A and C). A similar observation is relevant for the two irradiation followed by thermal exposure sequences (B and D).

The effect of irradiation is more substantial for ultimate tensile elongation than for ultimate tensile strength. Both the 27°and 70°C irradiations reduced the tensile elongation by ~30 percent whereas the tensile strength was only reduced by 10-20 percent.

There are only slight differences in permanent set caused by the different aging sequences (Figure 5.20).

5.2.4 VAMAC (82 H3 and 82 J3)

The same VAMAC material was used to make 3 mm-thick sheets of VAMAC and also O-ring seals. Dumbbells (82 H3) as shown in Figure 2.1 were cut from the sheets and used for ultimate tensile strength and ultimate tensile elongation measurements. O-ring seals (82 J3) were used for compression (permanent) set measurements. Ultimate tensile measurement results for the 82 H3 samples are shown in Figure 5.21. Permanent set after compression measurements for the 82 J3 samples are illustrated in Figure 5.22.

For the 82 H3 tensile specimens, irradiations at 27°C and at 70°C have the same effect on ultimate tensile properties. The material tensile properties are also unaffected by the order of the aging sequence. Both the tensile strength and the tensile elongation are unaffected by the thermal aging

exposure. Only the irradiation exposure changed the tensile properties of VAMAC.

There are only slight differences in permanent set caused by the different aging sequences (Figure 5.22).

5.2.5 HYPALON (82 G10)

An electric cable was stripped of its HYPALON jacket. (HYPALON is the trade name of Dupont for CSPE.) Dumbbells as shown in Figure 2.1 were cut from the material and used for ultimate tensile strength and ultimate tensile elongation measurements.

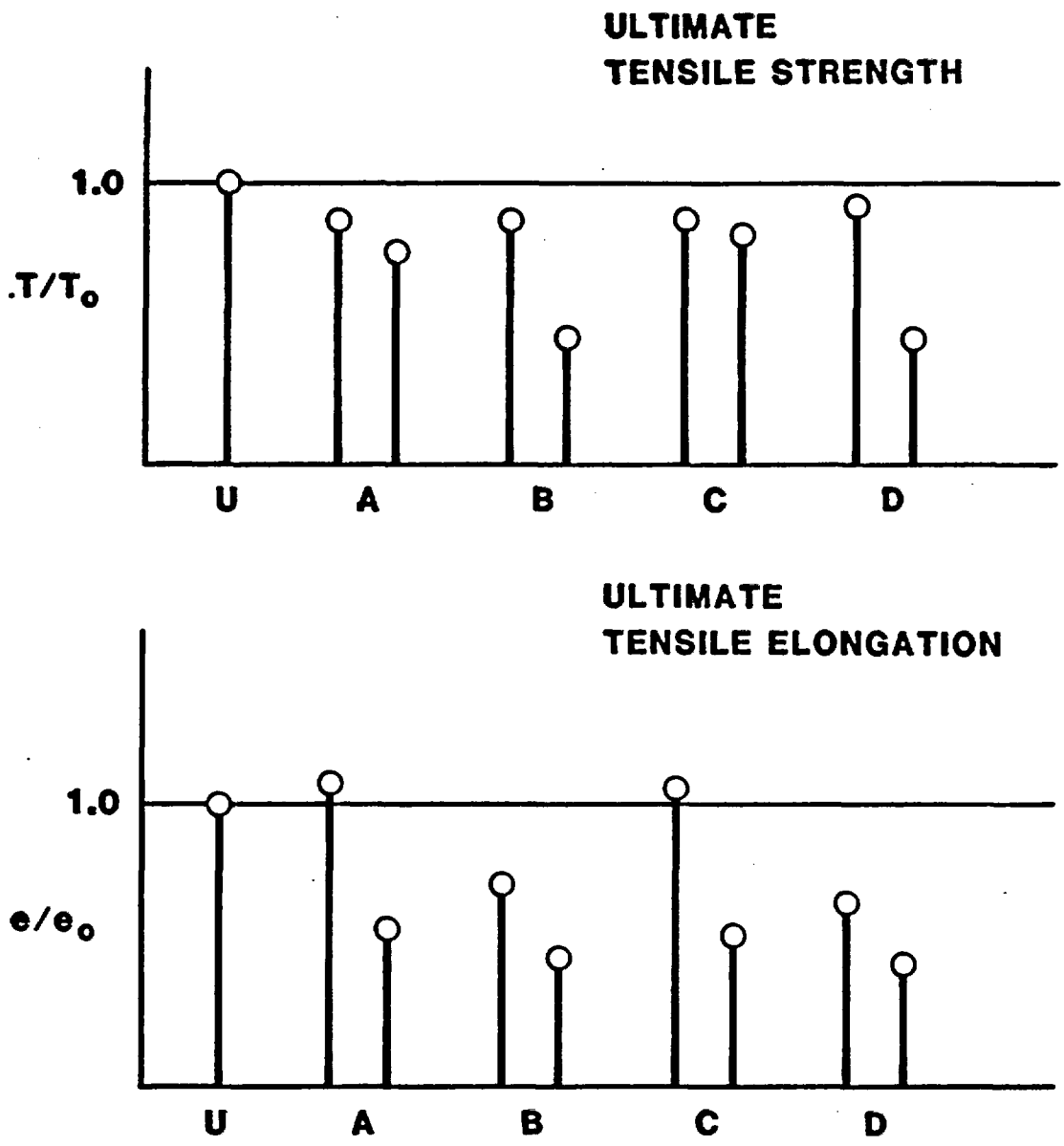
For this material (Figure 5.23), irradiations at 27°C and at 70°C have the same effect on ultimate tensile properties. Changes in ultimate tensile strength and elongation are also independent of the order of the aging exposures (irradiation and thermal).

5.2.6 PPS (82 H6)

Phenylene polysulfide (PPS) dumbbells as shown in Figure 2.3 were injection molded by the material manufacturer. The effects of aging sequences on this material's ultimate tensile properties are shown in Figure 5.24. This material is highly resistant to aging. Its mechanical properties are so little affected by the aging sequences that it is difficult to detect any meaningful differences between the various aging sequences.

5.2.7 Polydiallylphtalate (82 H5)

Polydiallylphtalate dumbbells as shown in Figure 2.3 were molded by the material manufacturer. The effects of aging sequences on this material's ultimate tensile properties are shown in Figure 5.25. The first aging exposure (160°C thermal exposure or 27°C irradiation) results in an increase in tensile elongation, possibly caused by lengthening of the macro-molecular chains. Irradiation at 70°C does not increase the elongation. The second aging exposure of the aging sequence (either irradiation or thermal exposures) causes an increase in ultimate tensile strength and a reduction in ultimate tensile elongation. This possibly reflects additional cross linking of the material. For polydiallylphtalate, the order of the aging sequence does not affect the ultimate tensile properties.



U = UNAGED

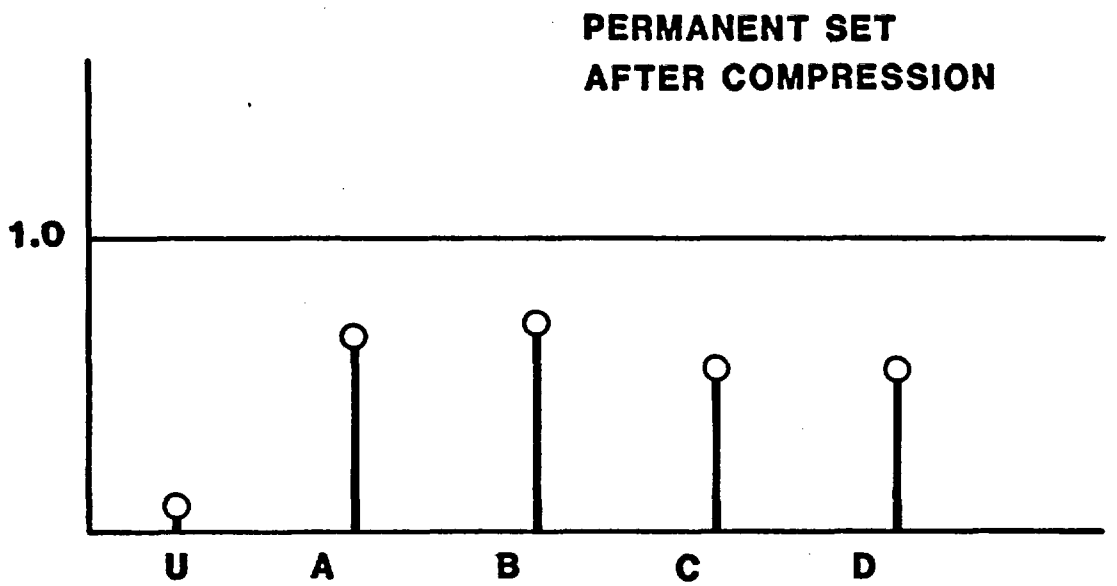
A = 140→R_{70}

B = R_{70}→140

C = 140→R_{27}

D = R_{27}→140

Figure 5.19. Ultimate Tensile Properties for EPR (82 H4)



U = UNAGED

A = 140→R_{70}

B = R_{70}→140

C = 140→R_{27}

D = R_{27}→140

Figure 5.20. Permanent Set After Compression for EPR (82 J4)

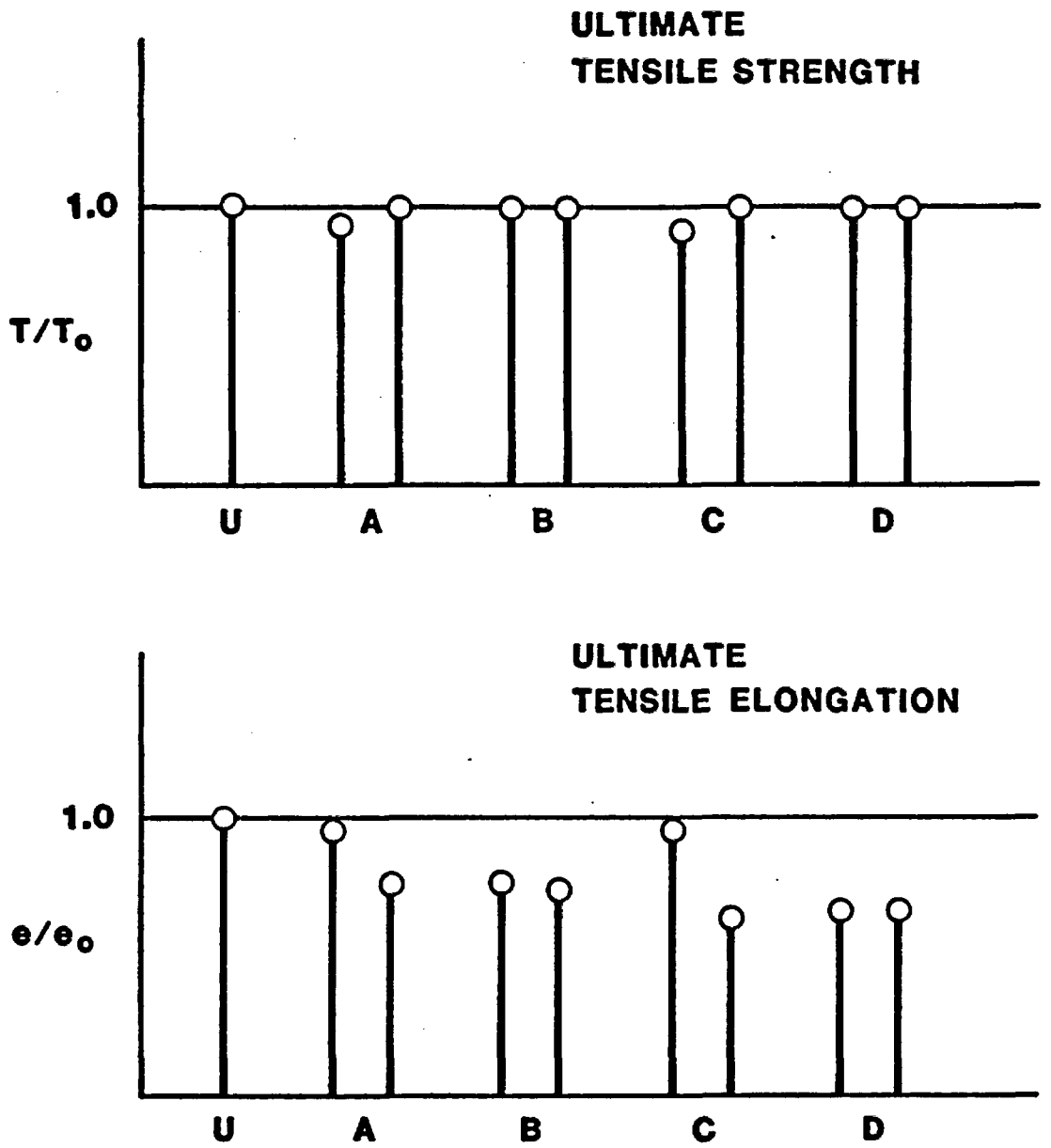
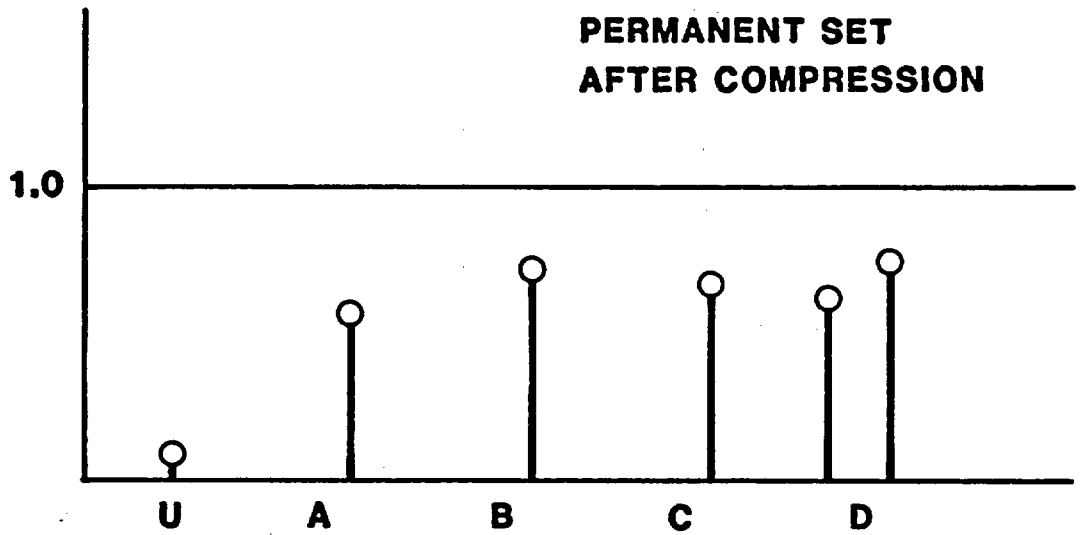


Figure 5.21. Ultimate Tensile Properties for VAMAC (82 H3)



U = UNAGED

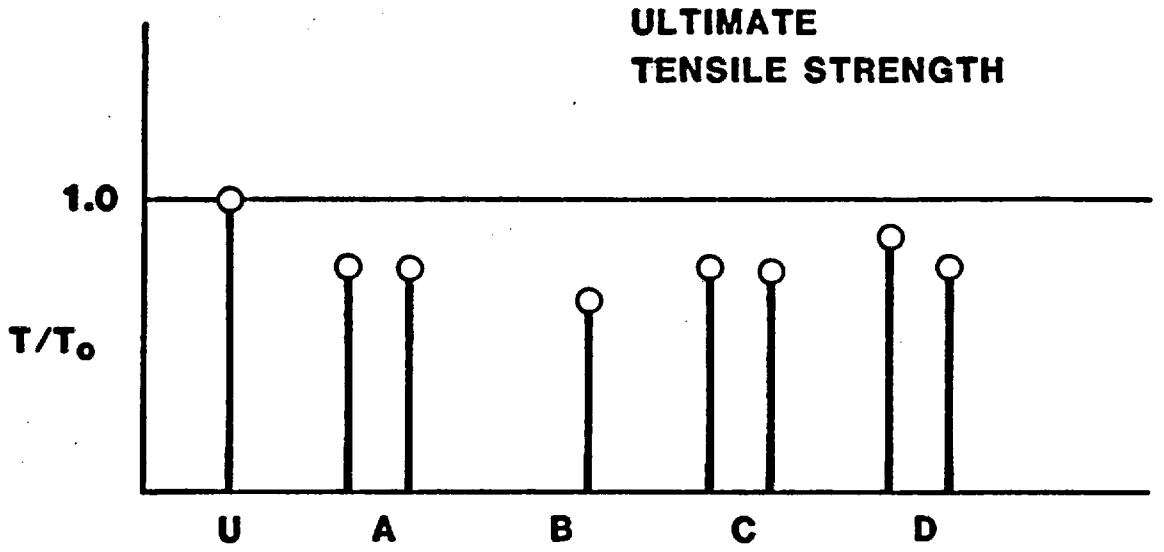
A = 120→R_{70}

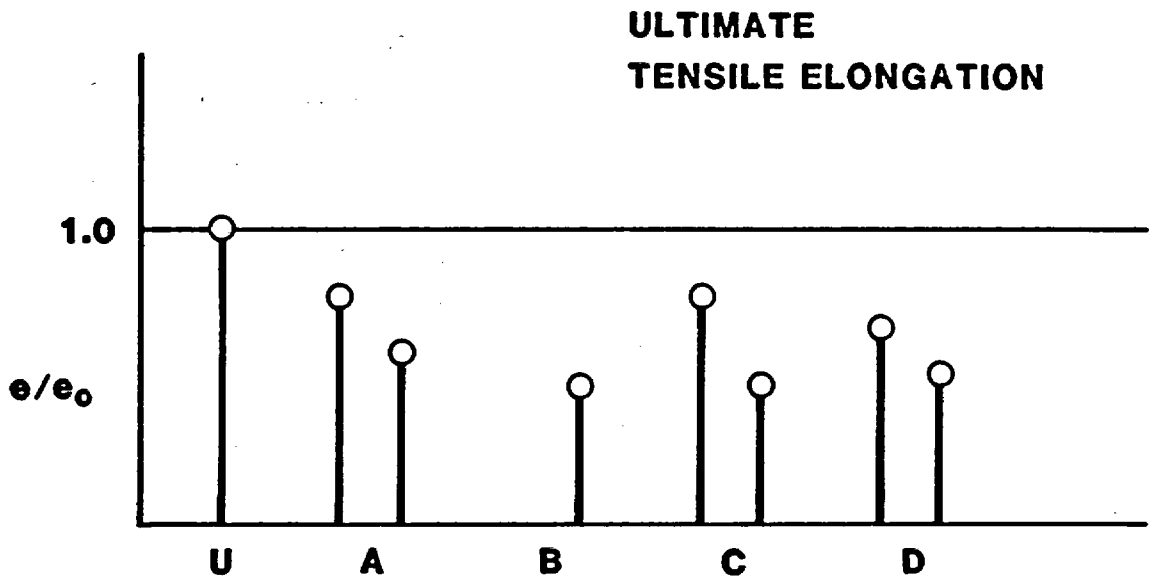
B = R_{70}→120

C = 120→R_{27}

D = R_{27}→120

Figure 5.22. Permanent Set After Compression for VAMAC (82 J3)





U = UNAGED

A = 140 → R_{70}

B = R_{70} → 140

C = 140 → R_{27}

D = R_{27} → 140

Figure 5.23. Ultimate Tensile Properties for HYPALON (82 G10)

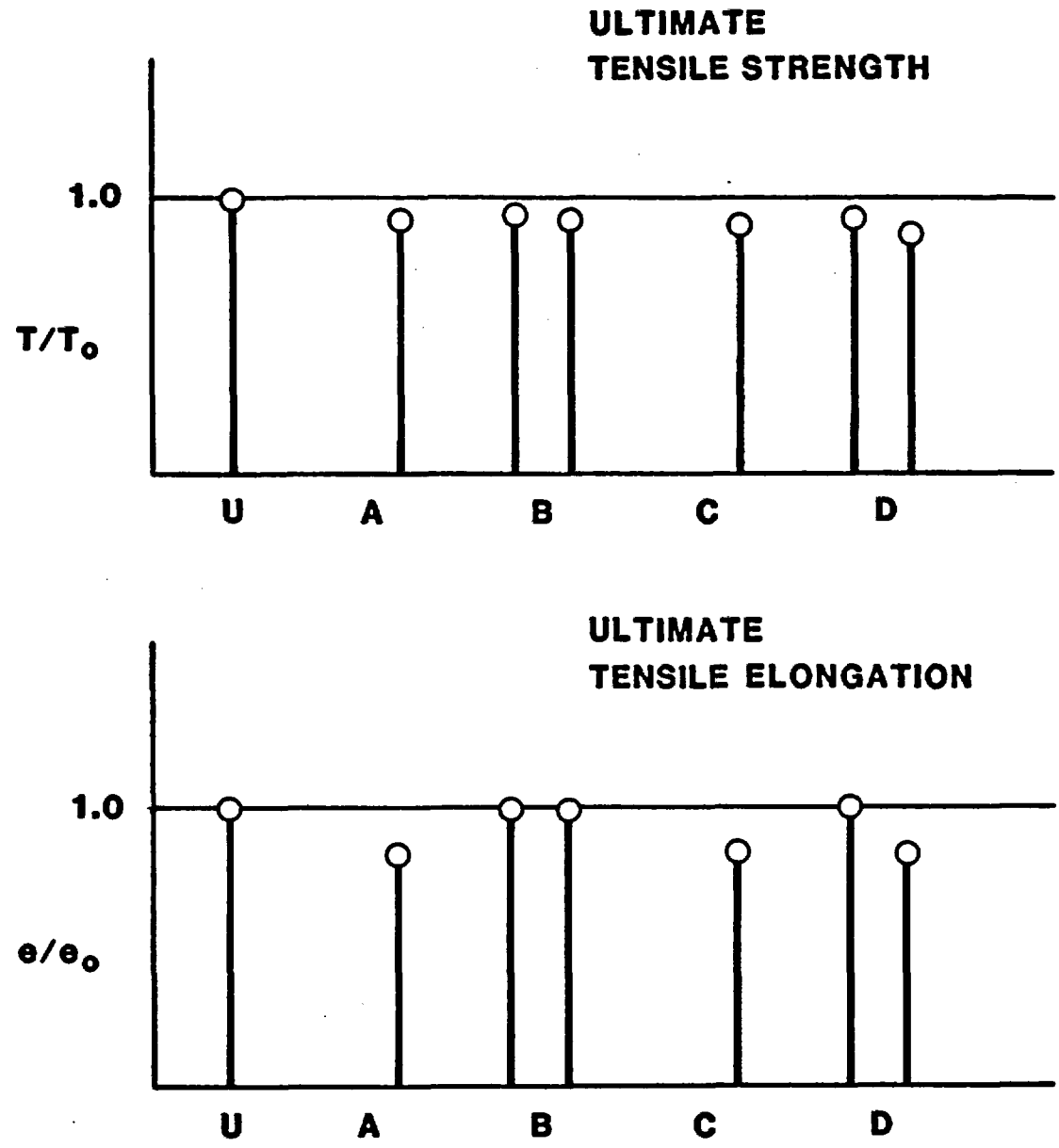


Figure 5.24. Ultimate Tensile Properties for PPS (82 H6)

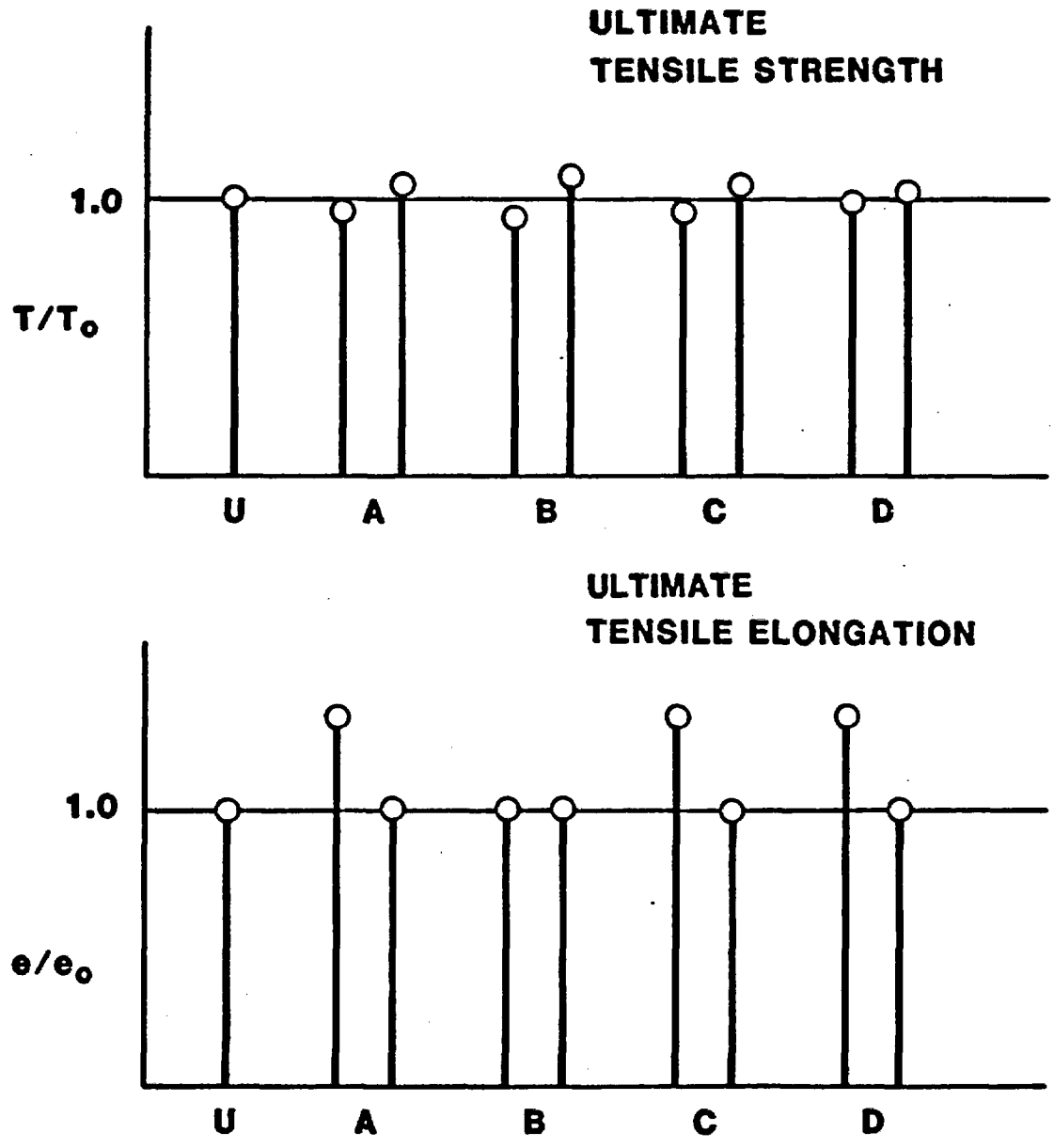


Figure 5.25. Ultimate Tensile Properties for Polydiallylphtalate (82 H5)

6.0 CONCLUSIONS

As part of a joint French/U.S. sponsored research program, we are investigating the influence of testing conditions on the behavior of polymer materials. In this report we have summarized the response of several commercial insulation and jacket materials, compression and gasket materials, and thermosetting and thermoplastic materials to various aging environments.

Ultimate tensile properties measured during our aging exposures exhibited several general trends:

1. If sequential ordering of irradiation and thermal exposures was important to the aging degradation of tensile properties, usually the irradiation followed by thermal exposure sequence was most severe. Examples are elongation and tensile strength for CSPE, CPE, EPDM (82 I2 and 82 I9), and elongation for PRC (82 I1). A counter example is TEFZEL 2 which was more degraded by the thermal followed by the 70°C irradiation exposure rather than the reverse sequence. The observation that irradiation followed by thermal exposures is the more severe sequence is consistent with previously published results for low density polyethylene (LDPE) and polyvinylchloride (PVC).[2] Neither LDPE nor PVC materials were included in this study.

2. During the sequential irradiation and thermal exposures, we performed irradiations at both ambient (~27°C) and 70°C temperatures. In general, the choice of irradiation temperature was secondary to the choice of aging sequence in its effect on polymer properties. Irradiation temperature did influence the degradation behavior of some materials. Examples are bend test results for TEFZEL 1 and TEFZEL 2, elongation and tensile strength data for CSPE, tensile strength data for EPR 2, and tensile strength data for EPDM (82 I2).

3. For several materials, tensile properties at completion of aging were only slightly affected by both the irradiation temperature and the order of the sequential aging environmental exposures. Examples are XLPO 1, VAMAC (82 H3), PPS (82 H6), and polydiallylphtalate (82 H5).

In addition to tensile measurements, we performed permanent set after compression measurements for several gasket and O-ring seal materials. At most, only slight differences in permanent set were noted as a result of the various aging techniques.

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14 ABSTRACT (200 words or less)

Prior to initiating a qualification test on safety-related equipment, the testing sequence for thermal and irradiation aging exposures must be chosen. Likewise, the temperature during irradiation must be selected. Typically, U.S. qualification efforts employ ambient temperature irradiation while French qualification efforts employ 70°C irradiations. For several polymer materials, the influence of the thermal and irradiation aging sequence as well as the irradiation temperature (ambient versus 70°C) has been investigated in preparation for Loss-of-Coolant Accident simulated tests.

Ultimate tensile properties at completion of aging are presented for three XLPO and XLPE, five EPR and EPDM, two CSPE (HYPALON), one CPE, one VAMAC, one polydiallylphtalate, and one PPS material.

Bend test results at completion of aging are presented for two TEFZEL materials.

Permanent set after compression results are presented for three EPR, one VAMAC, one BUNA N, one Silicone, and one Viton material.

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