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AN EVALUATION OF THE RADIATION RESISTANCE OF HIGH-DENSITY POLYETHYLENE*

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ABSTRACT

Mechanical tests following gamma irradiation and creep tests during irradiation have been conducted on high-density polyethylene (HDPE) to provide data to help assess the adequacy of this material for use in high integrity containers (HICs). Two types of HDPE, a highly cross-linked rotationally molded material and a non-cross-linked blow molded material, were used in these tests. Gamma-ray irradiations were performed at several dose rates in envrionments of air, Barnwell and Hanford backfill soils, and ion-exchange resins. The results of tensile and bend tests on these materials following irradiation are presented along with results on creep during irradiation.

INTRODUCTION

High integrity containers (HICs) provide an alternative to solidification for meeting the stability requirements for Class B and C radioactive waste under 10 CFR Part 61 (Licensing Requirements for Land Disposal of Radioactive Waste). The State of South Carolina has licensed HICs for disposal of low-level radioactive waste in the Barnwell, SC, land burial site. High density polyethylene (HDPE) is the material used to fabricate most HICs.

To provide a data base to assist in assessing the adequacy of HDPE for HICs, the U. S. Nuclear Regulatory Commission (NRC) contracted with Brookhaven National Laboratory (BNL) to test the radiation resistance of two types of HDPE, Marlex CL-100 and Chemplex 5701. Marlex CL-100 is a highly cross-linked HDPE produced by the Phillips Chemical Company while Chemplex 5701 is a non-cross-linked, high-molecular weight HDPE which contains a small percentage of hexene as co-polymer. The Marlex CL-100 material used in this study was rotationally molded while the Chemplex 5701 was blow molded.

To investigate the radiation resistance of HDPE, tensile and bend tests were performed following irradiation, and creep testing was conducted during irradiation. For the tensile and bend testing, samples were irradiated at several dose rates in air, backfill soils from the Barnwell, S.C. and Hanford, WA, radioactive waste burial sites and in ion-exchange (IX) resins. Creep tests during irradiation were conducted in air and in IX resins. Irradiation affects polyethylene by causing cross-linking and by introducing unsaturation and free radicals into the polymer.(1) Hydrogen evolution accompanies these changes, and the presence of unsaturation and radicals also causes the color to become yellow to brown as the dose increases. Oxidation of polyethylene occurs during irradiation in the presence of oxygen, as in air.(1,2) Cross-linking generally leads to some increase in tensile strength and a decrease in elongation at break.(1) Oxidation decreases cross-linking and introduces products of oxidation into the polymer which result in decreases in both tensile strength and elongation at break.(2)

Irradiations in the soils and IX resins were conducted at 10-11°C to provide a more realistic approximation to burial conditions. The HIC will certainly be in contact with backfill soil. IX resins are perhaps the most common type of radwaste disposed of in these containers. It was of interest to determine whether irradiation of HDPE in the soils or the IX resins might cause any interaction or reaction between an HIC and its external or internal environment. It was also of interest to determine whether mechanical testing following irradiation in the soils and IX resins would be be noticeably different from results following irradiation in air. The soils might limit oxygen availability to the HPDE specimens while the IX resins may exclude oxygen entirely since they react with oxygen during irradiation.(3)

EXPERIMENTAL

Irradiations were performed in the BNL Co-60 gamma pool facility at 10-11°C, at dose rates from 1.4 krad/h to 93 krad/h in environments of air, backfill soils from the Barnwell, SC, and Hanford, WA, land burial sites and dewatered ion-exchange (IX) resins. Temperature was monitored by observing pool temperature. For irradiations in air, this was found to be accurate to within 1°C by measurments using a thermocouple. For irradiations in the soils and IX resins the temperature was measured by inserting a thermometer into the medium immediately upon removal of the container from the air tube following irradiation. This was also found to be within 1°C of pool temperature. The highest dose rate used in these tests, 93 krad/h, was chosen to allow irradiation to 100 Mrad in a reasonable time (45 days). The dose value of 100 Mrad was based on the NRC's requirement, as stated in the Technical Position on Waste Form, May 1983, that, "No significant changes in material design properties should result following exposure to a total accumulated dose of 10^8 rads." The lowest dose rate used, 1.4 krad/h, was the lowest available. The IX resin formulation used was a 1:1 mixture of a strong-acid cation resin and a strong-base anion resin. The resin mixture was loaded with soluble contaminants and insoluble corrosion products (crud) according to a recipe from an analysis of spent PWR mixed bed IX resins.(4) Test samples were placed in 0.075 m x 0.3 m (3-in. diam x 12-in. high) Pyrex containers for irradiation. Air flowed through the container at a rate of 100 cm^3/min for the air irradiations. For irradiations in the soils and IX resin, the test samples in the container were completely embedded in well tamped soil or resin.

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*Work carried out under the auspices of the U.S. Nuclear Regulatory Commission.

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Radiochromic film was used for dosimetry. The accrued dose may vary as much as ±10% from the value indicated by the film. The films used are regularly calibrated against other films which are traceable to the National Bureau of Standards.

Test specimens were stamped or machined from Chemplex 5701 and from two varieties of Marlex CL-100. The Chemplex was taken from blow-molded 55-gallon drums purchased from Plasti-Drum Company, Lockport, IL. One variety of Marlex CL-100, non-HIC Marlex, came from a rotationally molded container purchased from Poly-Processing, Inc., Monroe, LA. The other Marlex CL-100 was actual HIC material provided by Chem-Nuclear Systems, Inc.

Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) at a testing speed of 0.05 m/min (2 in./min). ASTM Type IV specimens, for material ≤0.004 m (0.160 inch) in thickness, sufficed for the Chemplex and non-HIC Marlex materials while ASTM Type III specimens, for material 0.007-0.014 m (0.28 - 0.55 in) in thickness, were required for the Marlex HIC material, which was typically about 0.013 m (0.500 in.) thick. The Type IV specimens were stamped, as recommended in D-638, using Die C as described in ASTM D-412. The Type III specimens were machined. Bend testing was performed according to ASTM D-790 (Flexural Properties of Plastics and Electrical Insulating Materials). Testing was performed within four days of the end of irradiation. Creep testing during irradiation was performed on Type IV tensile specimens in equipment built for this study. The Type IV tensile specimens were clamped into self-aligning holders and lowered down air tubes in the BNL gamma pool and locked into place. Cables from the sample holders passed over pulleys and were attached to weight pans. Weights added to the pans supplied the creep stress and pan movement provided the creep measurement.

RESULTS AND DISCUSSION

Qualitatively, the tensile results on the three HDPE materials were similar. Differences were noted mainly in details. However, there was at least one notable difference in characteristics between the Chemplex and Marlex materials which appeared to be related more to the container manufacturing processes than to material differences. Bend tests results showed a substantial difference between Chemplex and Marlex. Irradiation increased creep but the increase became noticeable only at relatively large stresses.

Tensile Testing

Figure 1 shows a series of typical tensile stress vs elongation vs dose curves for Marlex HIC material irradiated in air at 10-11°C at a dose rate of 9.3 krad/h. Curves similar to those shown in Figure 1 were also obtained for the non-HIC Marlex and Chemplex. For this reason, results for the different materials will not be presented separately. The unirradiated and 9.3 Mrad curves show stress rising to a maximum (which is defined as the yield point, stress or strength) followed by a decrease to a constant stress until the break occurs. In the region of decreasing stress following the yield point neck formation occurs. The constant-stress portion of the curve results from neck propagation.

Irradiation eventually causes the loss of necking behavior. The 47 Mrad and 93 Mrad curves in Figure 1 evidence this from the absence of the transition to a region of constant stress. Loss of necking behavior following irradiation is accompanied by the appearance of surface cracks in these materials. In a specimen irradiated to a dose beyond which no necking occurs, failure results when one of these cracks propagates through the specimen. The cracks appear prior to the yield point and at the yield point the one crack that will propagate through is apparent. The number of cracks increases with increasing dose.

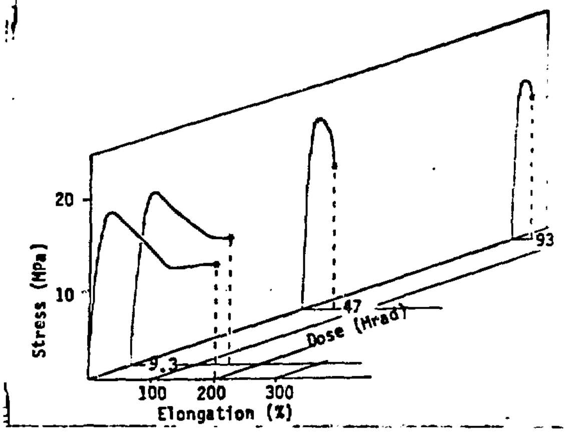

Fig. 1. Three-dimensional plot of tensile stress vs elongation vs gamma ray irradiation dose for Marlex CL-100 HIC material. The irradiations were performed in air at 10-11°C and 93 krad/h.

In Marlex, the cracking always appeared on the inside surface of the container and the cracks were generally evenly spaced along the entire narrow section of the test piece. Figure 2 shows a series of Marlex HIC material tensile specimens and Figure 3 shows a closeup of the cracking that occurs after necking behavior is lost. Examples of the cracking in Marlex HIC material is shown in Figure 3. The cracking observed in the Chemplex is usually limited to the vicinity of the break and occurs on both surfaces of the test piece. These differences in the nature of the cracking behavior appear to be related to surface differences arising in the manufacturing processes. There are no obvious differences between the inner and outer surfaces of the blow-molded Chemplex containers. However, the inside surfaces of the rotationally molded Marlex containers are very smooth and glossy whereas the outside surfaces are dull and somewhat roughly textured. Rotational molding involves melting resin beads on the inside of a mold rotating simultaneously about two perpendicular axes. The outside surface of a container during fabrication is in contact with the mold, whereas, the inside surface is in contact with hot air.(5)

Tensile data for Marlex HIC material irradiated at 10-11°C at various dose rates in air, Barnwell and Hanford soils and IX resins are listed in Table I. Figures 4-7 illustrate the data from Table I. Figure 4 plots yield stress vs dose while Figure 5 shows the effect of dose rate on yield stress for a dose of approximately 10 Mrad. Figure 6 plots elongation at break vs dose while Figure 7 shows the effect of dose rate on elongation at break for doses of about 10 Mrad. A quick scan of Figures 4-7 suggests that there is a lot of scatter in the data and that there is no clearly evident segregation of data points by environment (i.e., air, soils, or IX resin) in any of these plots.

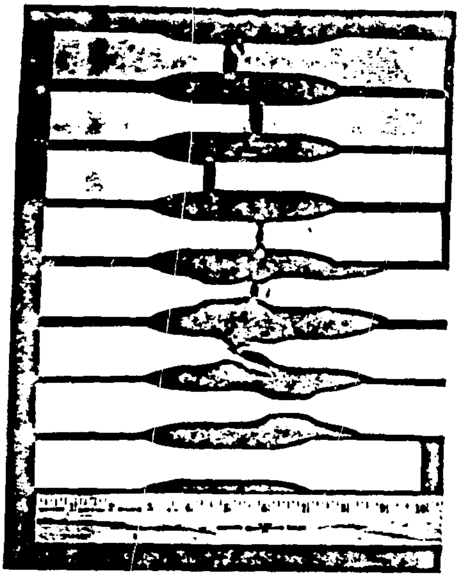

Fig. 2. Photograph of a series of Marlex HIC material Type III tensile specimens irradiated in air at 10-11°C which show the transition from necking to breaking without necking. From bottom: untested specimen; specimen with 1-in. neck; specimen irradiated 2.7 Mrad at 2.5 krad/h; specimen irradiated 9.3 Mrad at 93 krad/h; specimen irradiated 8.6 Mrad at 17 krad/h; specimen irradiated 25 Mrad at 14 krad/h; specimen irradiated 47 Mrad at 93 krad/h and, at top, a specimen irradiated 93 Mrad at 93 krad/h.

Several factors contributed to data scatter including inhomogeneities in the HDPE materials and dimensional variations in the test specimens. The Marlex materials contained small bubbles, which presumably were air bubbles trapped during the container fabrication process. Additionally, the inside and outside surface of the Marlex were noticeably different. The inner Marlex surfaces were also oxidized, presumably from exposure to hot air during the rotational molding process.(6,7) The surfaces of the materials corresponding to the inside and outside of the container were left as received and not machined smooth. Thus, the thickness of each test specimen varied at different places along its length. The HIC material varied up to a millimeter in thickness along the length of any individual test specimen while the non-HIC Marlex and Chemplex varied up to a quarter of a millimeter. The thinnest section of each test specimen was used for calculating the cross sectional area.

In Figure 4, which plots yield stress vs dose, most of the data lies above the value for the unirradiated material, as indicated by the dashed line. This indicated that irradiation tended to increase the tensile strength under the conditions of these tests. The unirradiated value is the average of 11 specimens tested over the time period of this task along with the standard deviation about this average. This result was typical of all three HDPE materials tested. Some differences due to irradiation environment appeared at higher doses. The 100 Mrad IX resin point is well above that for air at 93 Mrad. A similar effect occurred in both the Chemplex and non-HIC Marlex data. Additionally, the IX resin and soil irradiation data for the Chemplex and non-HIC Marlex lie well above that for air at 40-50 Mrad.

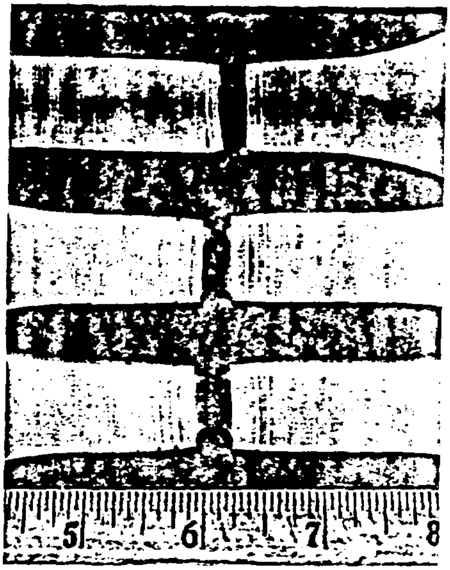

Fig. 3. Closeup of the cracks that occur in irradiated Marlex CL-100 once necking behavior is lost. From bottom: specimen irradiated in IX resins to 10 Mrad at 8.7 krad/h, specimen irradiated in Barnwell soil to 20 Mrad at 11 krad/h and, top, specimen irradiated in Hanford soil to 50 Mrad at 58 krad/h. Irradiations were conducted at 10-11°C.

Figure 5 shows no dose rate effect on the yield stress. There is no trend for these data to either increase or decrease with dose rate. There does not appear to be a noticeable effect of irradiation environment on these results.

There are two striking features evident in Figure 6. First, there is a significant decrease in break elongation with dose. Second, there is a large uncertainty associated with the break elongation of the unirradiated HIC material. The decrease in break elongation correlated with the loss of necking behavior. The break elongation plateaus out at about 50% once necking behavior is lost. Unirradiated Marlex specimens typically fail from a tear which starts at one of the four corners in the necked portion of the specimens. These tears often appear to be started by a small bubble in the material coming to the surface near a corner during formation of the neck and popping. This bubble defect mechanism of failure initiation for the Marlex materials may explain the large variation in the elongation at break. The variation in break elongation for Chemplex was much smaller and occurred at much greater elongation.

Table I

Tensile Test Data on Irradiated Marlex CL-100 HIC* Material[a]

Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (MPa)	Elongation at Break (%)
0[b]	---	---	20.1 ± 1.1	220 ± 90
9.3	93	air	21.7	160
47	93	air	23.5	51
93	93	air	19.9	32
8.6	17	air	22.1	66
25	14	air	22.8	57
9.5	5.7	air	21.7	47
2.7	2.5	air	23.2	120
3.6	2.5	air	21.2	66
9.7	58	Barnwell soil	21.4	130
50	58	Barnwell soil	25.5	44
8.5	11	Barnwell soil	20.7	64
20	11	Barnwell soil	24.6	60
3.0	4.0	Barnwell soil	22.4	130
8.0	3.7	Barnwell soil	22.3	43
2.0	1.4	Barnwell soil	21.0	110
50	58	Hanford soil	22.8	52
8.5	11	Hanford soil	21.0	49
20	11	Hanford soil	22.3	50
3.0	4.0	Hanford soil	21.1	170
13	79	IX resin	20.3	110
49	79	IX resin	23.2	47
100	79	IX resin	24.3	36
20	11	IX resin	20.8	76
10	8.7	IX resin	20.1	64
3.0	4.0	IX resin	22.1	200
3.0	3.7	IX resin	21.4	60
3.0	4.0	IX resin/ Barnwell soil	21.4	56
10	11	IX resin/ Barnwell soil	21.0	62

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*EnviroSAFE is the trademark of the high integrity containers vended by CHEM-NUCLEAR SYSTEMS, Inc. Containers are rotationally molded using MARLEX CL-100 high density, highly cross-linked polyethylene.

[a] Irradiations were performed at 10-11°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using one Type III specimen per test.

[b] These data are from 11 unirradiated specimens tested over the time period of this task.

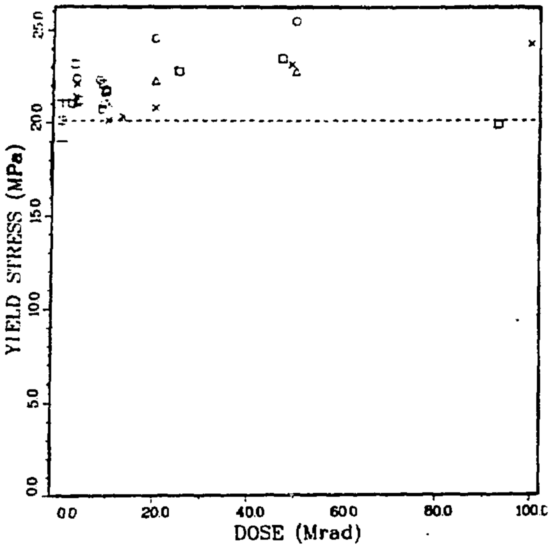

Fig. 4. Yield stress vs dose of Marlex CL-100 HIC material irradiated at 10-11°C. Symbols indicate irradiation environment: air-□, Barnwell soil -○, Hanford soil -△, IX resin - X.

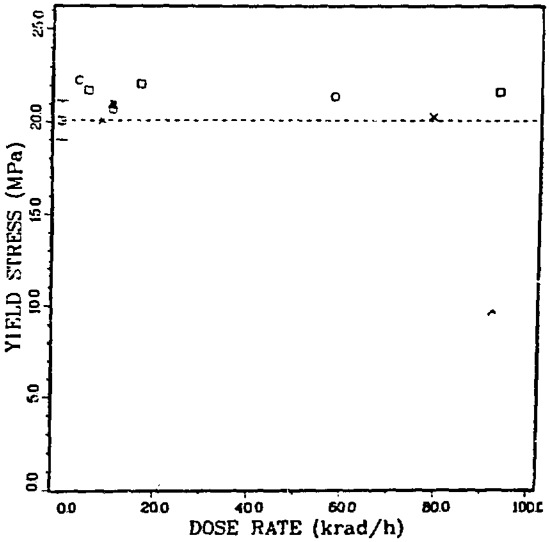

Fig. 5. Yield sress vs dose rate of Marlex CL-100 HIC material irradiated at 10-11°C for total doses from 8.0-13 Mrad. Symbols indicate irradiation environment: air-□, Barnwell soil -○, Hanford soil -△, IX resin -X.

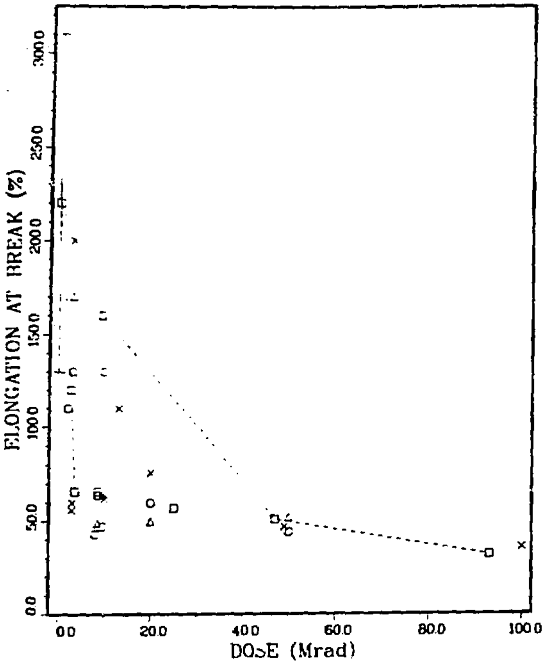

Fig. 6. Elongation at break vs dose of Marlex CL-100 material irradiated at 10-11°C. Symbols indicate irradiation environment: air-□, Barnwell soil -○, Hanford soil -△, IX resin - X.

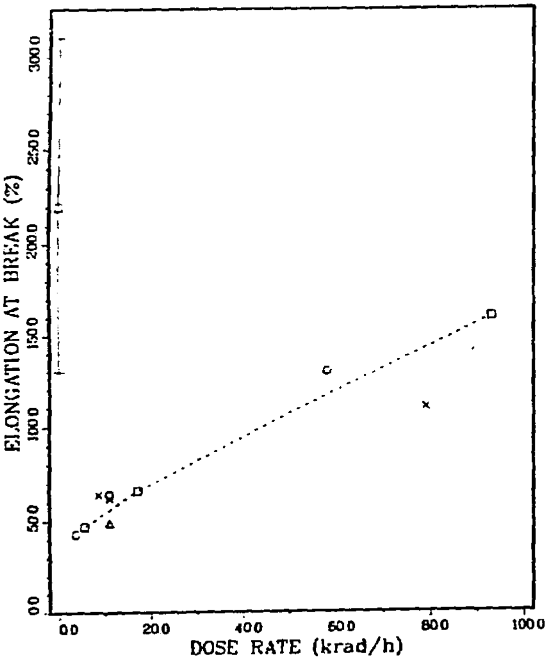

Fig. 7. Elongation at break vs dose rate for Marlex CL-100 HIC material irradiated at 10-11°C for total doses from 8.0 - 13 Mrad. Symbols indicate irradiation environment: air - □, Barnwell soil -○, Hanford soil -△, IX resin - X

The two dashed lines in Figure 6 connect air-irradiation points at the highest and lowest dose rates used. The upper line connects points at 93 krad/h, while the lower line, which appears nearly vertical, connects data at 2.5 krad/h. This, plus the data in Figure 7 (see below) shows that break elongation is sensitive to dose rate.

Figure 7 shows that break elongation decreases as the dose rate decreases. This effect occurred in all four irradiation environments and no differences between irradiations in the different irradiation environments were noted. The dashed line connects the air irradiation points in Figure 7.

Bend Testing

The results of bend tests on irradiated Marlex HIC material are illustrated by the curves in Figure 8. These data show that the stiffness of the Marlex HIC material increases significantly upon irradiation. As in the tensile tests the inside surface of this material cracks upon bending. This surface only cracked in tension and not in compression in samples irradiated up to up to 50 Mrad. As in the tensile tests, the onset of cracking in the bend tests was dose and dose rate dependent but was not noticably affected by the environment (i.e., air, soil, or resin). For specimens irradiated up to 50 Mrad, while cycling in the bend test machine did cause cracks, these cracks did not propagate beyond the surface.

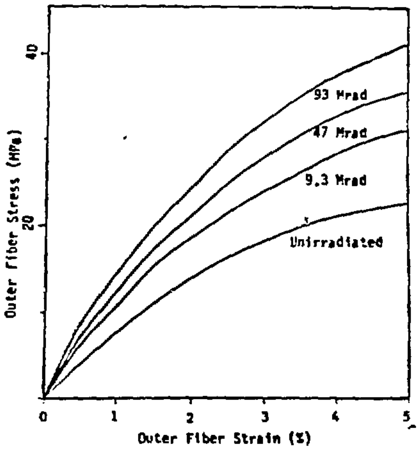

Fig. 8. Bend test curves for irradiated Marlex CL-100 HIC material tested according to ASTM D-790.

The Chemplex behaved differently in the bend testing (data not shown). This material did not get noticeably stiffer following irradiation until doses of approximately 50 Mrad had been attained and, above that dose, the increase in stiffness was smaller than that observed for the Marlex. The Chemplex did not crack in the bend test.

Creep During Irradiation

Creep testing on Type IV tensile specimens of Chemplex and non-HIC Marlex irradiated at 5 krad/h and 10-11°C indicates that the creep rate is faster in the irradiated test samples than in the unirradiated controls. The increase appears to be stress-dependent, i.e., the larger the stress the greater the increase in creep during irradiation. This is illustrated in Figure 9, which shows creep curves for Marlex CL-100 under tensile creep loads of 11.0 and 12.4 MPA (1600 and 1800 psi) at 10-11°C. Curves are shown for unirradiated specimens and for specimens undergoing irradiation at 5 krad/h in IX resin. Similar results are obtained for creep during irradiation in air.

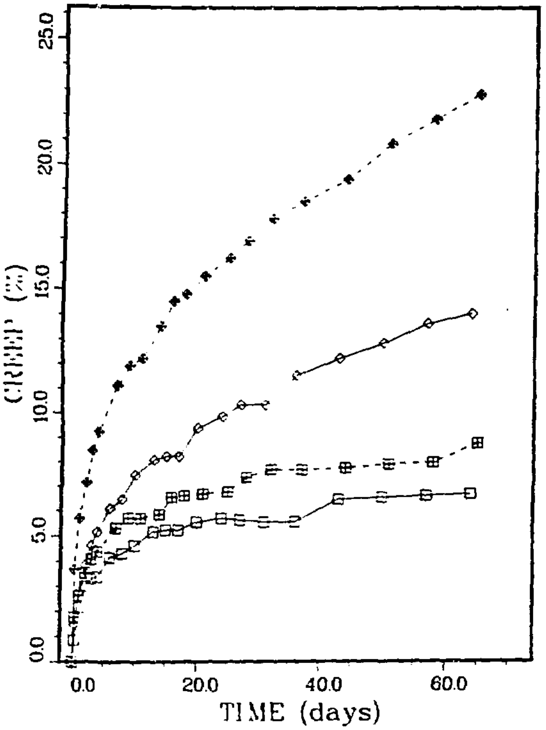

Fig. 9. Creep curves for Marlex CL-100 at 10-11°C under tensile stresses of 11.0 and 12.4 MPa (1600 and 1800 psi). Curves are for unirradiated controls and for specimens undergoing irradiation at 5 krad/h in IX resin. Unirradiated, 11 MPa -□; irradiated 11 MPA -⊞; unirradiated, 12.4 MPA -◇; irradiated, 12.4 MPa -⊕

The data for creep during irradiation for Chemplex (not shown) were similar to the Marlex curves shown in Figure 9. However, there was a significant difference. The Chemplex specimens stressed to 12.4 MPa ruptured about 10 weeks into the test. One specimen irradiated in air ruptured at 71 days and another specimen irradiated in IX resin ruptured at 84 days. This creep rupture during irradiation occurs at approximately half the normal tensile strength of 24.8-25.5 MPa recorded for unirradiated Chemplex from tensile testing in this task. The unirradiated Chemplex controls did not exhibit creep rupture at 12.4 MPa.

CONCLUSIONS

Mechanical testing of HDPE following irradiation at 10-11°C has shown that irradiation resulted in no loss of strength. However, irradiation caused these materials to become less tolerant of deformation. The usual terminology for such changes would be that the material had embittled. However, since test specimens of these materials irradiated to 50 Mrad tolerated tensile deformation exceeding 10% and did not break under cyclic bend testing, the term brittle hardly seems appropriate.

The effects of the different irradiation environments (air, soils and IX resins) in modifying the changes in characteristics produced by irradiation were variable. There was some indication that the soils and IX resin moderated attack from air during irradiation to large doses. The effect became noticeable at approximately 50 Mrad in the thinner Chemplex and Marlex non-HIC specimens while it was not observed until 100 Mrad, if at all, in the thicker Marlex HIC specimens.

Marlex became stiffer following irradiation, as indicated in the bend test results. Up to 50 Mrad, Chemplex did not. We do not know whether this difference in the relative stiffness of the two irradiated materials is related to the fact that, before irradiation, the Chemplex is non-cross-linked while the Marlex is highly cross-linked or to the different container fabrication processes or to other factors.

The results of the irradiations conducted at 10-11°C may be explained by radiation-induced cross-linking, except for the environmental effect at large doses discussed two paragraphs previously. In order to accelerate radiation-induced oxidation, irradiations in air at 60-63°C were conducted. (These results were not discussed in this article. They are included in the complete report, including all of the data, for this study.(8)) These higher temperature irradiations resulted in a loss of strength as well as decreases in elongation at yield and elongation at break. The loss of strength appeared to result from degradation of the surface which progessed into the bulk material as the irradiation in air continued. This result is attributable to radiation-induced oxidation. This, combined with the results for irradiation at 10-11°C plus results cited in the literature for irradiations in inert atmosphere,(1,2) suggest that radiation-induced oxidation may enhance or speed up the decrease in break elongation that results from cross-linking, but does not solely cause this change. Thus, an irradiated HIC may lose much of its ability to tolerate deformation at some radiation dose, which depends on dose rate, before any loss in strength becomes apparent.

Irradiation under tensile stress resulted in increased creep. Under the conditions of these tests (10-11°C and 5 krad/h) the increase in creep did not become significant until stress loads of approximately half the normal tensile strength or greater were applied. The increase in creep during irradiation occurred in both air and IX resin. The Chemplex creep ruptured during irradiation in both air and IX resin.

During irradiation, there is a transition from behavior characteristic of unirradiated material (i.e., necking behavior) to behavior characterized by cracking and breaking without necking. When the type of failure is plotted as a function of dose vs dose rate, the transition from necking to breaking without necking behavior appears linear on a log-log scale. The relationship obtained from these plots for Marlex CL-100 is

$$D_N = 77000\ (R)^{0.5}$$

and for Chemplex is

$$D_N = 550000\ (R)^{0.3}$$

where D_N is the dose (rad) up to which necking predominates at a dose rate of R(rad/h).

Table II presents estimates of D_N and the time to reach D_N for several dose rates using these equations. The values of D_N and the time to dose at dose rates less than 2000 rad/h in Table II represent extrapolations from the data obtained in this study. The dose rates were chosen to bracket estimated initial dose rates for highly loaded IX resin waste. For wastes whose activity is dominated by isotopes with half lives on the order of 30 yrs (e.g., Cs-137), such that the total accumulated dose would be 10^8 rad, the dose rate to which the container may be exposed upon loading would be approximately 250 rad/h. Based on this loading, one year after loading, the accumulated dose would be approximatley 2.3 Mrad. Similarly, wastes whose activity is dominated by isotopes with half lives of 5 years (e.g., Co-60), loaded such that the total accumulated dose would be 10^8 rad, the dose rates to which the container would be exposed upon loading is approximately 1500 rad/h. In this case, one year after loading, the accumulated dose would be approximately 13 Mrad. It should be noted that these estimates of anticipated dose rates and doses may be conservatively high since they neglect container geometry and self-shielding by the resin wastes. On the other hand, it should be remembered that these estimates are based on irradiation data taken at 10-11°C. If the higher temperatures that might be encountered in storage speed up the transition, then these estimates may not be conservative. Using these dose rates as a benchmark for expected field conditions leads one to conclude that embrittlement of HDPE HICs could occur within a few months to a year. It would appear that the consequences of such embrittlement during storage and following burial should be considered in the design of HICs made from HDPE.

Table II

Estimates of the Dose and Time-to Dose for the Necking to Breaking Without Necking Transition for Marlex CL-100 and Chemplex 5701

Material	R(rad/h)	D_N(Mrad)	Time to D_N (Days)
Marlex CL-100	2000	3.0	63
Marlex CL-100	1000	2.1	88
Marlex CL-100	500	1.5	125
Marlex CL-100	100	0.7	292
Chemplex 5701	2000	6.3	130
Chemplex 5701	1000	5.0	209
Chemplex 5701	500	4.0	335
Chemplex 5701	100	2.4	1000

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