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An Evaluation of the Effects of Gamma Irradiation on the Mechanical Properties of High Density Polyethylene

Prepared by D. R. Dougherty, J. W. Adams, R. E. Barletta

Brookhaven National Laboratory

Prepared for
U.S. Nuclear Regulatory Commission

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An Evaluation of the Effects of Gamma Irradiation on the Mechanical Properties of High Density Polyethylene

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Prepared by
D. R. Dougherty, J. W. Adams, R. E. Barletta

Brookhaven National Laboratory
Upton, NY 11973

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Division of Waste Management
Office of Nuclear Material Safety and Safeguards
U.S. Nuclear Regulatory Commission
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ABSTRACT

High-integrity containers (HICs) provide one option under 10 CFR Part 61 (Licensing Requirements for Land Disposal of Radioactive Waste) for meeting the stability requirements for Class B and C radioactive waste. High-density polyethylene (HDPE) is the material used currently to fabricate most HICs.

Mechanical tests following gamma irradiation and creep tests during irradiation have been conducted on high-density polyethylene (HDPE) to assess the adequacy of this material for use in high-integrity containers (HICs). These tests were motivated by experience in nuclear power plants in which polyethylene electrical insulation deteriorated more rapidly than expected due to radiation-induced oxidation. This suggested that HDPE HICs used for radwaste disposal might degrade more rapidly than would be expected in the absence of the radiation field.

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 testing on these materials following irradiation at 10-11°C showed no effects directly or solely attributable to radiation-induced oxidation. However, effects due to radiation-induced cross-linking, including an increase in yield strength and decreases in both elongation at yield and elongation at break, were observed. Irradiation at 60-63°C showed effects or radiation-induced oxidation including a decrease in yield strength. These effects were more marked in thinner test specimens. Creep testing during irradiation indicated that irradiation increases the creep rate but that the effect is really only significant at creep loads greater than about half the nominal yield strength under the conditions of these tests (10-11°C and 5 krad/h).

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FIGURES

FIGURES (Continued)

FIGURES (Continued)

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†Trademark of the high integrity containers vended by CHEM-NUCLEAR SYSTEMS, INC.

ACKNOWLEDGMENTS

The authors thank Drs. K. J. Swyler, P. L. Piciulo, R. E. Davis, and E. P. Gause and Ms. C. E. Shea for valuable comments on and assistance with this work. We would also like to acknowledge the efforts of Messrs. J. D. Smith, C. F. Ruege, and W. Becker for their contributions in building the devices for creep testing during irradiation and Mr. D. Horne for performing the tensile and bend tests. The authors express their gratitude to Ms. M. McGrath and N. Yerry for their preparation of the manuscript. Finally, the authors acknowledge Chem-Nuclear Systems, Inc. for providing the Marlex HIC material used in these tests.

AN EVALUATION OF THE EFFECTS OF GAMMA IRRADIATION ON THE MECHANICAL PROPERTIES OF HIGH-DENSITY POLYETHYLENE

1. INTRODUCTION

High-integrity containers (HICs) provide one option under 10 CFR Part 61 (Licensing Requirements for Land Disposal of Radioactive Waste) for meeting the stability requirements for Class B and C radioactive waste. The technical position on HICs states that they should have as a design goal a minimum lifetime of 300 yrs*. NRC guidance on HIC characteristics includes the following directive from the technical position.

> The high-integrity container design should consider the radiation stability of the proposed container materials, as well as the radiation effects of the wastes.
>
> Radiation degradation testing should be performed on proposed container materials using a gamma irradiator or equivalent. No significant changes in material design properties should result following exposure to a total accumulated dose of 10^8 rad. If it is proposed to design the high-integrity container to greater accumulated doses, testing should be performed to confirm the adequacy of the proposed materials. Test specimens should be prepared using the proposed fabrication techniques.
>
> Polymeric high-integrity container designs should also consider the effects of ultra-violet radiation. Testing should be performed on proposed materials to show that no significant changes in material design properties occur following expected ultra-violet radiation exposure.

HICs have been licensed by the State of South Carolina for disposal of radioactive waste in the Barnwell, SC, land burial site. High-density polyethylene (HDPE) is the material used to fabricate most of the HICs licensed by South Carolina.

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 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 copolymer. Chem-Nuclear Systems uses Marlex CL-100 to fabricate HICs by rotational molding. Chemplex 5701 is used by Plasti-Drum Company to blow mold 55 gallon drums, which are not HICs.

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*"Technical Position on Waste Form," p. 9, May, 1983, Rev. 0, in "Final Waste Classification and Waste Form Technical Position Papers," USNRC, May 1983, Rev. 0.

Part of the motivation for conducting these tests stems from nuclear plant experience in which polyethylene electrical insulation deteriorated more rapidly than expected.[1-4,28] The electrical insulation was described as low to medium density polyethylene in Reference 1 while it was described as simply polyethylene in References 2-4 and as polyolefin in Reference 28. The deterioration was reported to have been caused by radiation-induced oxidation of the polyethylene. This process was slow due to the low permeability of oxygen in polyethylene at the plant temperature of approximately 43°C and to the low dose rate. The maximum estimated dose in 12 years was 2.5 Mrad. They also showed that, since the mechanism for degradation of polyethylene resulting from radiation-induced oxidation is thermally activated, increasing the temperature results in increased degradation. Clough and Gillen concluded that traditional radiation resistance testing performed at high dose rates for short times has tended to underestimate the degree of deterioration that has occurred in long term, low dose rate exposure.

Radiation induced oxidation would clearly be a concern for any contemplated long-term storage of polyethylene HICs in air. Additionally, studies on trench gas compositions at radwaste burial sites indicate that oxygen may also be present in the trenches.[5-7] Specifically, data from the Beatty, NV, site suggest that trench gas at arid burial sites may have essentially the same oxygen content as air, and data from the West Valley, NY, and Maxey Flats, KY, sites indicate that, in trenches at wet disposal sites, the oxygen content of the trench gas may be depleted, but oxygen will still be present. The trench gas at West Valley appeared to have stabilized at about 3% oxygen while that at Maxey Flats had nearly the same oxygen content as air.

Another concern is whether the creep properties measured on unirradiated polyethylene provide realistic estimates of creep during irradiation. In fact, irradiation during creep testing increased the creep rates in all polymers tested,[8,9] although polyethylene itself was not tested. For all polymers tested including polymethylmethacrylate, a copolymer of 88% polyvinylchloride and 12% polyvinylacetate and polystyrene, creep rates increased during irradiation as the dose rate increased. Additionally, creep rates increased at a given dose rate as the thickness increased for all polymers except polystyrene.

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 ion exchange resins. Irradiations in which the HDPE specimens had unlimited access to air were conducted to determine a baseline of performance of the mechanical properties of HDPE. (The results of Clough and Gillen in Reference 1, suggest this may be the worst case.) This baseline performance of HDPE irradiated in air also provides the data base from this work to assist in the assessment of possible consequences of storage of HICs in air before disposal.

The purpose of the irradiations in the soils and IX resins was to provide a more realistic approximation to burial conditions. The HIC will certainly be in contact with backfill soil and 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. In fact, no interaction was observed in these tests. Comparison of the results of mechanical testing following irradiation in these environments with the results from the air irradiations did not confirm that irradiation in air is the worst case. No difference in the changes produced upon irradiation was noted between test specimens irradiated 10-11°C in air or the other environments. The changes we observed in mechanical properties could be more readily explained as having been due to radiation induced cross-linking in the polymer rather than to radiation induced oxidation. This may have been the result of the conditions of irradiation time and temperature used in these experiments—i.e., the irradiation times may have been too short for the conditions under which these experiments were conducted.

The results summarized in the previous paragraph led us to perform an irradiation in air at 60-63°C and the results of this experiment did indicate a degradation of the materials beyond what occurred at 10-11°C. This experiment at 60-63°C appears to be the only clear example of radiation-induced oxidation from these tests. Given this, it therefore appears that our results in soil and IX resins did not give a true picture of the effect, if any, of environments other than air in moderating radiation-induced oxidation. The IX resins may exclude oxygen entirely since they react with oxygen during irradiation.[10] Thus, one might reasonably expect the presence of IX resins to moderate radiation-induced oxidation of a container containing resins. However, to confirm this, irradiations would have to be conducted at higher temperatures and/or longer times than were conducted in these tests.

Creep during irradiation was found to have increased over that observed in the absence of irradiation. Generally, the higher the creep stress, the greater the increase in creep was observed. The effect appeared to be small below a relative creep loading of approximately half the nominal yield stress. However, above this stress creep increased rapidly over the level outside of the radiation field. This effect was the same in environments of both air and IX resins. The creep experiments were limited in that they were performed at only one temperature and dose rate—i.e., 10-11°C and 5 krad/h.

The results reported here indicate that polyethylene undergoes changes in its mechanical properties upon irradiation. Whether these changes are significant with respect to important properties of particular HIC designs would have to be addressed by the manufacturer or by independent analysis. In general, the changes produced by irradiation make polyethylene less tolerant of deformation before breaking. Therefore, for example, if a HIC is designed to deform upon loading or burial based on the properties of the unirradiated material, an irradiated container may not tolerate the deformation without breaking. This reduced tolerance of deformation occurs following irradiation whether air is present or not. Cross-linking in polyethylene during irradiation causes a loss of ductility and deformability whether air is present or not.(11) Although radiation-induced oxidation may speed up the loss of desirable characteristics, irradiation will change the mechanical characteristics of HDPE such that it will lose much of the deformability of the unirradiated material whether oxygen is present or not. One aspect of the loss of deformability from the HDPE materials studied here has been found to be dose rate dependent and been modelled. This model is presented in the conclusions of this report.

2. EXPERIMENTAL

The experimental efforts in this project consisted of two primary tasks. The first task consisted of mechanical testing of HDPE material following irradiation while the second task involved creep testing the HDPE material during irradiation. The simple irradiations of the first task involved tensile and bend testing following irradiations in environments of air, soils and IX resins. The second task, creep testing during irradiation, was performed in air and IX resins.

2.1 Materials

Two types of HDPE were used in these experiments, Chemplex 5701 and Marlex CL-100. Additionally the Marlex was from two different sources and of different thicknesses. One variety of Marlex CL-100 was actual HIC material and was approximately half an inch in thickness. The other Marlex CL-100 material was taken from a container purchased by BNL for this project. The walls of the purchased container are nominally an eighth-inch thick, although we found them to be somewhat thicker than this. These two varieties of Marlex CL-100 are hereafter referred to as "Marlex HIC material" and "Marlex non-HIC material." More detailed descriptions of these materials are given below.

Test specimens were stamped or machined from Chemplex 5701 and from two varieties of Marlex CL-100. The size of test specimens required was determined by the thickness of the material. Figure 2.1 shows the various test specimens used in these tests along with a ruler for scale.

2.1.1 Chemplex 5701 Test Material

Chemplex 5701 is a high-density, high molecular weight non-cross-linked polyethylene used by Plasti-Drum Corporation, Lockport, IL., to manufacture 55-gal drums. Chemplex 5701 is actually a copolymer of ethylene and hexene, according to a Chemplex product brochure.(12) However, the amount of hexene in the formulation is very small*.

Blow molding is a commonly used method of manufacturing hollow articles such as containers.(13) Generally, the process involves extruding a tube (parison) of heated polyethylene downward between the opened halves of a metal mold, closing the mold to pinch off and seal the parison at top and bottom, injecting air through a needle inserted through the parison wall, cooling the mass in contact with mold, opening the mold and removing the formed article. Many variations of the process exist.

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*Personal communication between D. Dougherty (BNL) and G. Kamykowski (Chemplex Co.), February 24, 1983. The actual amount of hexene in Chemplex 5701 is considered confidential.

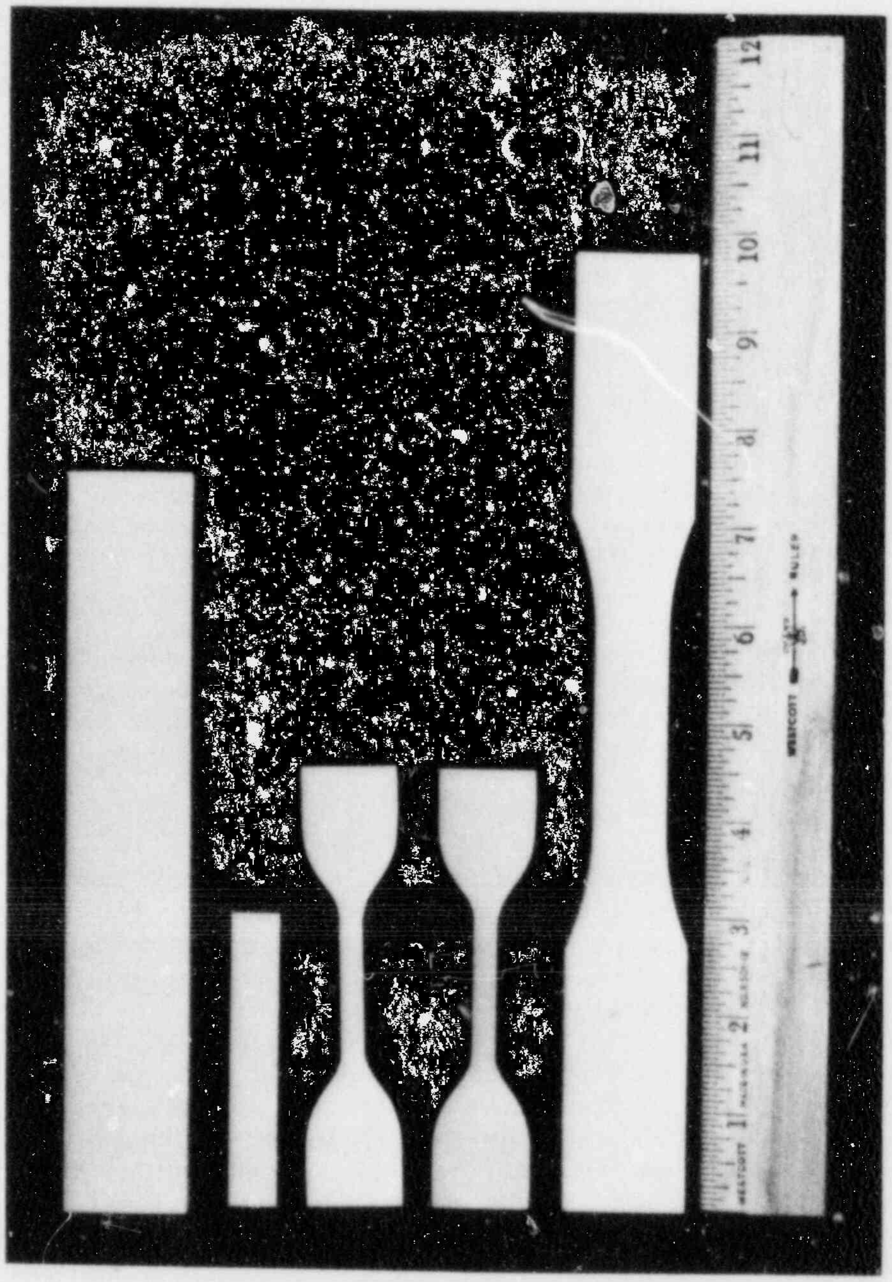

Figure 2.1. Photograph showing typical tensile and bend test specimens used in this task along with a ruler for scale. From left: Marlex HIC material bend specimen, Chemplex bend specimen, Chemplex Type IV tensile specimen, Marlex non-HIC material Type IV tensile specimen and a Marlex HIC material Type III tensile specimen.

The Chemplex 5701 material used in these experiments came from two 55-gal drums purchased from Plasti-Drum Corporation. These drums were received by BNL on January 19, 1983. According to Plasti-Drum*, the drums are generally no more than a few weeks old when shipped. The 55-gal drums such as used by BNL for these tests have been certified for Type A low level radioactive waste since March, 1982.

2.1.2 Marlex CL-100 Test Materials

Marlex CL-100 is a high-density highly cross-linked polyethylene used to fabricate containers using the process of rotational molding, which involves melting MARLEX CL-100 granules on the inside of a mold rotating simultaneously about two perpendicular axes.(14,15) The molding process results in different surface textures for the inner and outer container surfaces. The container outer surface, which is in contact with the mold during fabrication, has a dull textured finish. The container inner surface, which is exposed to hot air (typically 500-650°F) during the rotational molding process, has a glossy finish.(16) This glossy surface was found to exhibit different characteristics during mechanical testing than the rest of the material (i.e., the bulk material and the dull surface).

The Marlex CL-100 tested in this task included both HIC material and non-HIC material. The HIC material came from several EnviroSAFE† HICs. Chem-Nuclear Systems, Inc., generously supplied BNL with this HIC material for use in these tests. The non-HIC material was taken from a single container purchased by BNL for this project.

2.1.2.1 Marlex HIC Material

The Marlex HIC material used in these experiments was cut out of containers which were fabricated by the rotomolding method. Twenty-seven of these cut-outs, one per container, were shipped to BNL on October 29, 1983 by Poly-Processing, Inc., Monroe, LA., who manufactures the HICs for Chem-Nuclear Systems, Inc. These cut-outs came from any one of three sizes of HIC - 84 ft^3, 168 ft^3 or 195 ft^3. There are no obvious differences to indicate from which size HIC the cut-outs came. The age of the individual cut-outs was not available either. After a HIC is made a cut-out is taken to provide for the opening in the container. A portion of this cut-out is saved for quality control testing on the HIC while the rest of the cut-out, which is unlabelled, is put in a scrap pile. The HIC material sent to BNL was from this scrap pile**.

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*Personal communication between D. Dougherty and B. Ranworth (Plasti-Drum), January 10, 1983.

†Trademark of the high integrity containers vended by Chem-Nuclear Systems, Inc.

**Personal communication between D. Dougherty and F. Wimberly (Poly Processing) April 5, 1983.

The thickness of the cut-outs averaged 0.53 ± .04 inch. There are two surface textures evident among the cut-outs. The glossy surface texture is smooth in 24 of the cut-outs whereas it is mottled in the remaining three. These different surface textures are pictured in Figures 2.2 and 2.3. The glossy surfaces of the HIC material, both smooth and mottled surfaces, also contained blisters (~1/4- to 1/2-in. diam.) sparsely spread over the surface. The density of these blisters varied somewhat between pieces but averaged about 1 blister per 10 square inches of surface.

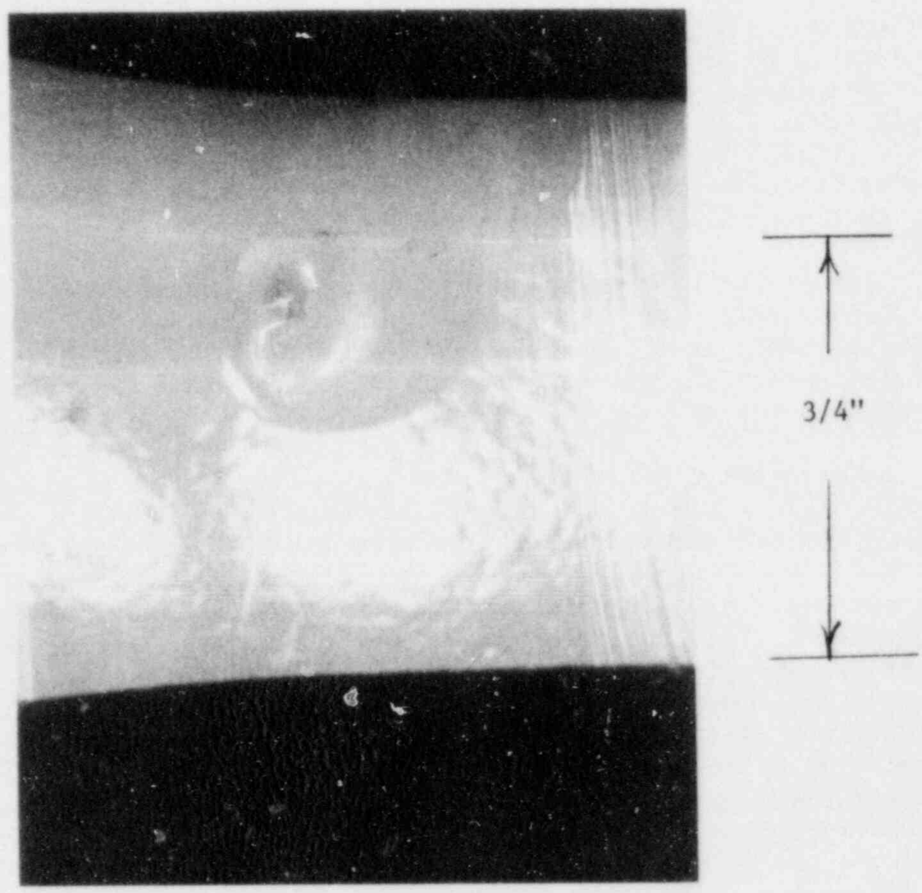

Figure 2.2. Closeup photograph of Marlex CL-100 HIC material smooth glossy surface including a blister.

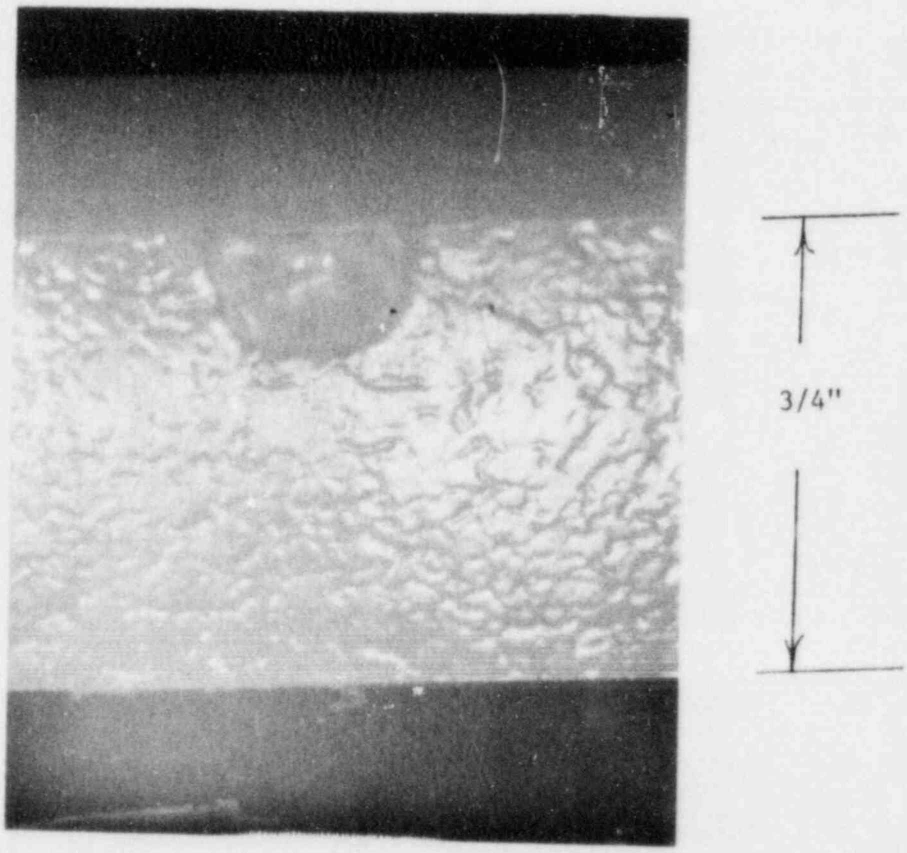

Figure 2.3. Closeup photograph of Marlex CL-100 HIC material mottled glossy surface including a blister.

2.1.2.2 Marlex non-HIC Material

The Marlex CL-100 non-HIC material was taken from a rotomolded 1260-gal container with a nominal 1/8-inch wall thickness specially ordered by BNL for this task from Poly Processing, Inc. The glossy surface of this material has a mottled texture similar to that of the HIC material shown in Figure 2.3. The mottled glossy surface of the non-HIC material is pictured in Figure 2.4. This container was fabricated on February 8, 1983 and arrived at BNL on February 24. The measured wall thickness of the container was 0.135-0.175 inch. This container, although not a HIC, was fabricated using the same material and process as is used in making HICs. The purpose of testing the thinner material, as well as the HIC material at container thickness, was to investigate possible effects of wall thickness on the mechanical properties following irradiation.

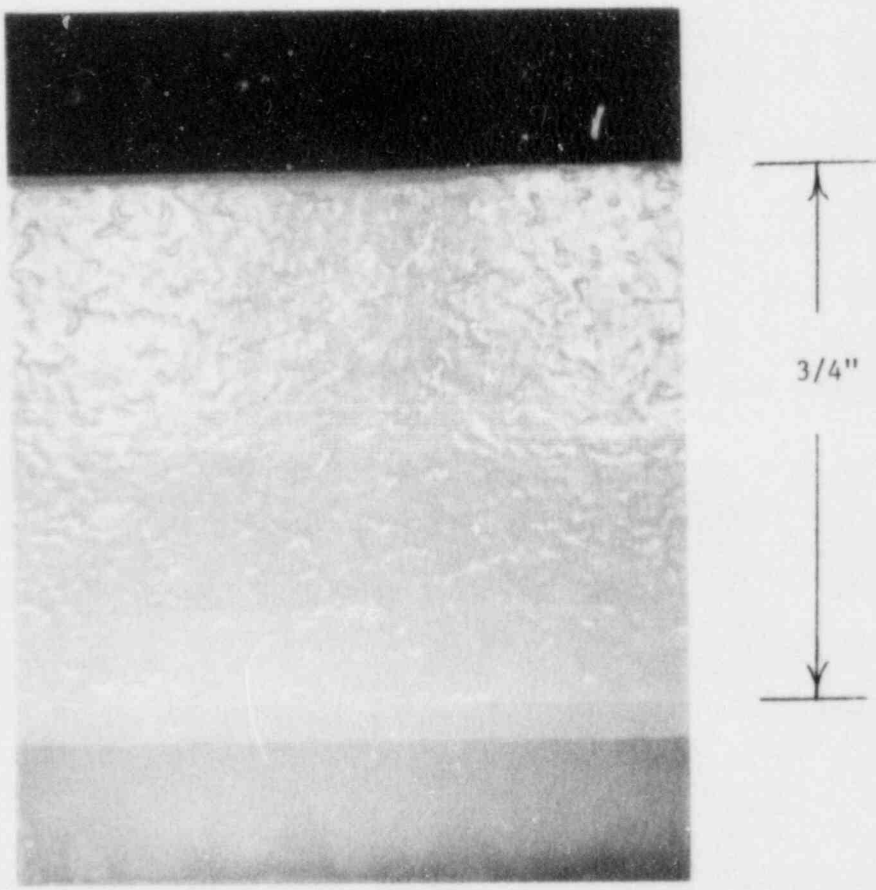

Figure 2.4. Closeup photograph of Marlex CL-100 non-HIC material mottled glossy surface.

2.2 Mechanical Testing

Both tensile and bend (flexure) testing were performed on the irradiated HDPE materials in this task. Polyethylene is normally evaluated from tensile data since tensile properties have been found to be most indicative of fundamental performance for thermoplastics.(17,18) Bend testing was included because this may correspond more directly to the kind of deformation that HDPE HICs are expected to experience.(19)

The mechanical testing was conducted following standard ASTM procedures. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) at a testing speed of 2 in./min. ASTM Type IV specimens, for material up to 0.160 inch in thickness, were used for the Chemplex and non-HIC Marlex materials while ASTM Type III specimens, for material 0.28-0.55 inch in thickness, were used for the Marlex HIC material. 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.

2.3 Irradiations

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 and in environments of air, backfill soils from the Barnwell, SC., and Hanford, WA. land burial sites and dewatered ion-exchange (IX) resins. 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 recommendation, as stated in the Technical Position on Waste Form, May 1983, that, "No significant changes in material design properties should result following ex posure to a total accumulated dose of 10^8 rad." The lowest dose rate used, 1.4 krad/h, was the lowest available. Test samples were placed in 3 in. diam. x 12 in. high Pyrex containers for irradiation. Air flowed through the container at a rate of approximately 100 cm^3/min for the air irradiations. For irradiation in the soils and IX resin, the test samples in the container were completely surrounded by and covered with well tamped soil or resin.

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.

Temperature during irradiation was monitored by observing pool temperature. This was found to be accurate to within 1°C by measurements using a thermocouple in the 4.6 M (15 ft) air tubes. For irradiations in the soils and IX resins the temperature was checked by inserting a thermometer into the medium immediately upon removal of the container from the air tube following irradiation. The temperature measured in this way was also found to be within 1°C of pool temperature. These measurements indicated that radiational heating was not a measurable factor (within 1°C) even in the soil and resin irradiations. The temperature of the test specimens undergoing irradiation was assumed to be equal to that of the surrounding air, soils or IX resin.

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 an analysis of spent PWR mixed bed IX resins.[20] The IX resins loaded with soluble and insoluble contaminants were used to more closely approximate the chemical environment that HIC material would experience in actual use.

2.4 Irradiation Under Stress - Creep Studies

Creep testing during irradiation has been performed on Type IV tensile specimens in equipment built for this study. Four Type IV tensile specimens are clamped into self-aligning holders and lowered down an air tube in the BNL gamma pool and locked into place. A half-inch diameter aluminum rod which runs down the center of the sample holder assembly is used to lower and raise the assembly and to rotate it to lock it into place at the bottom of the air tube used for irradiation. Figure 2.5 shows the sample holder assembly. Cables, which attach to the rings at the tops of the sample holders, pass over pulleys and are attached to weight pans. Weight added to the pans supply the creep stress and pan movement provides the creep measurement. Figure 2.6 shows the pulley and weight pan assembly attached to the air tube used for irradiations. The cables from the sample holders to the weight pans can be seen coming through the slotted lid on the top of the air tube.

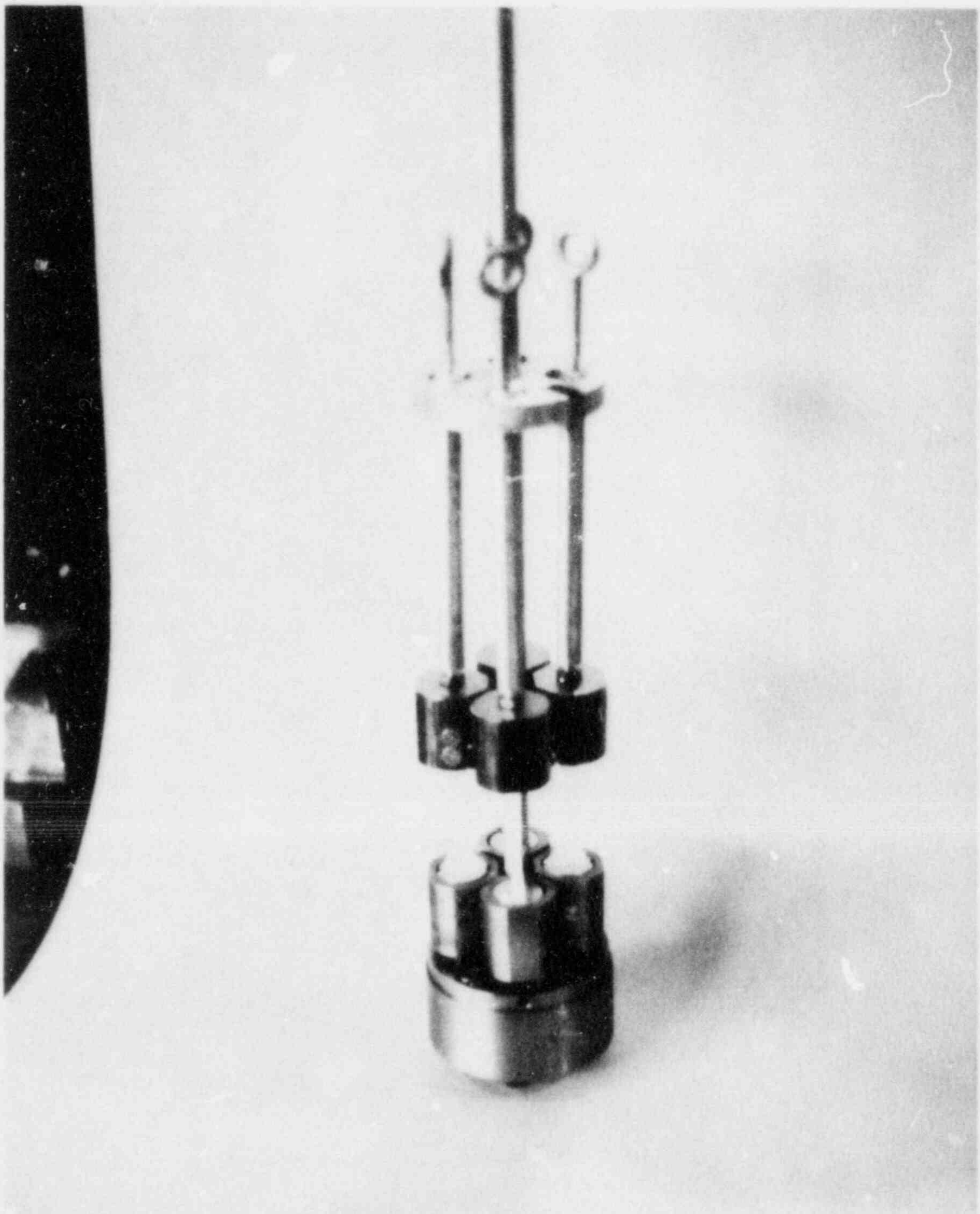

Figure 2.5. Photograph of a sample holder for creep testing during irradiation loaded with four Type IV tensile test specimens.

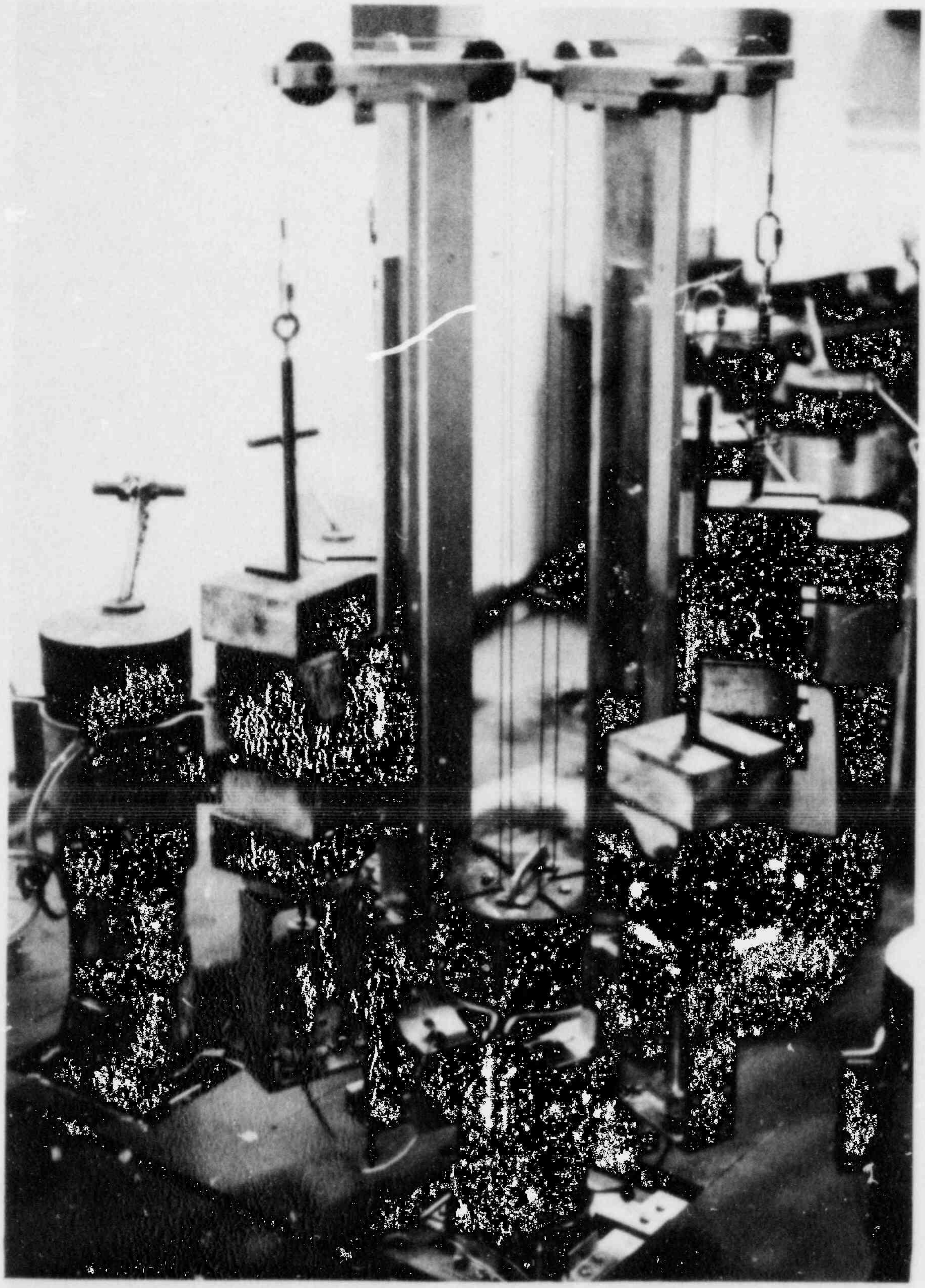

Figure 2.6. Photograph of the upper part of an irradiation-under-stress apparatus attached to the top of an air tube in the BNL gamma pool room.

3. TENSILE TESTING

Qualitatively, the tensile tests results on the three HDPE materials were quite similar. Differences were noted mainly in details. However, there was at least one notable difference in tensile characteristics between the Chemplex and Marlex materials which appeared to be related to the container manufacturing processes rather than to material differences. This difference was the fact that, for irradiated Marlex, the surface cracking that occurred above certain doses occurred almost exclusively in the glossy surface whereas the Chemplex cracked equally on both surfaces at the doses where cracking occurred.

3.1 Tensile Testing - General

Tensile testing of thermoplastic polymers such as polyethylene at room temperature typically results in a stress vs strain (or elongation) curve like that shown in Figure 3.1. The labelled parameters on the curve are: T_y = yield stress, E_y = elongation at yield and E_b = elongation at break. The star at E_b indicates the break point. The events that occur upon tensile testing of a specimen which result in a curve typified in Figure 3.1 are described as follows:

- The narrow section uniformly elongates up to the yield point.
- The neck develops from the yield point, E_y, until the curve levels off into the horizontal portion of the curve.
- The horizontal portion of the curve results from the neck spreading to include the entire gage length (i.e., narrow section) of the specimen.
- Once the neck has spread throughout the gage length, the stress again increases as the necked material uniformly elongates.
- The specimen breaks at some point on the curve depending upon the temperature, the rate of pulling and/or defects and inhomogeneities in the material. (Generally, the lower the temperature, the faster the rate of pull and/or the more severe any defects the sooner the break occurs.)

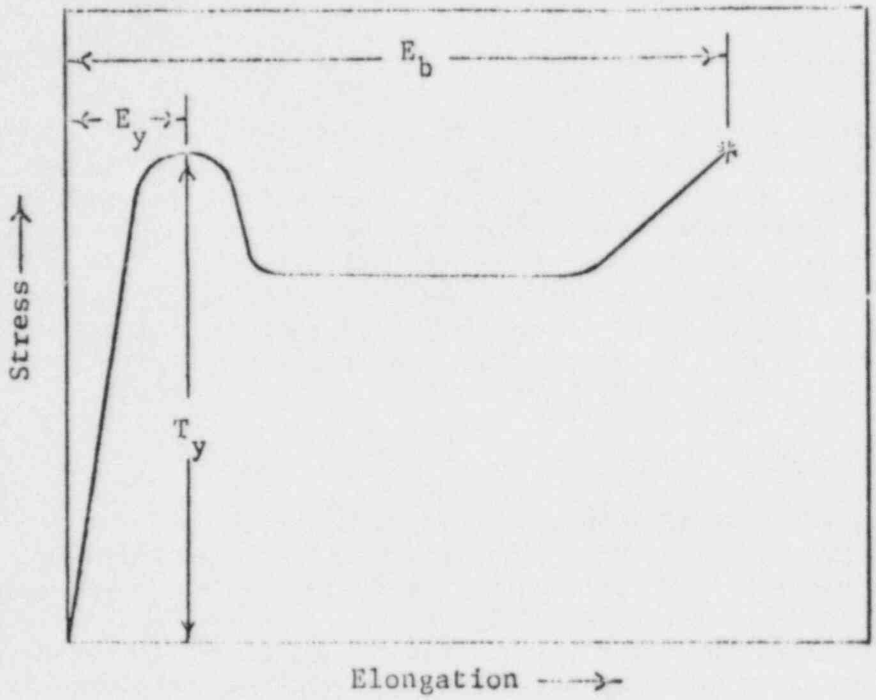

Figure 3.1. Schematic stress vs elongation (or strain) for typical polymer tensile behavior in the temperature region where necking occurs. E_y = elongation at yield, T_y = tensile stress at yield (yield stress) and E_b = elongation at break.

3.2 Tensile Testing - Results

Figures 3.2, 3.3 and 3.4 show a series of typical tensile stress vs elongation vs dose curves for Chemplex 5701, Marlex CL-100 HIC material and Marlex CL-100 non-HIC material, respectively, irradiated in air at 10-11°C at a dose rate of 93 krad/h. The unirradiated and 9.3 Mrad curves for the Chemplex, Figure 3.2, show stress rising to a maximum at the yield point followed by a decrease as the neck forms to a constant stress which finally increases again up to the break. The bumps in the unirradiated curve (in this particular curve at elongations of approximately 400% and 700%) are typical of Chemplex tensile curves. They appear somewhat randomly along the horizontal portion of the curve and in the final increase in stress before the break. These bumps apparently correspond to inhomogeneities in the plastic which are stronger than the surrounding material. In both Marlex materials, Figures 3.3 and 3.4, the unirradiated and 9.3 Mrad curves show stress rising to a maximum at the yield point and then decreasing as the neck forms toward the horizontal region of constant stress. The break occurs sometime after neck propagation has begun but before it has spread throughout the gage length in both Marlex materials. Irradiation eventually causes the loss of necking behavior in all three materials. The 47 Mrad and 93 Mrad curves in Figures 3.2-3.4 evidence this by the absence of the transition to the region of constant stress. Figures 3.5, 3.6 and 3.7 show the tensile specimens which produced the curves shown in Figures 3.2, 3.3 and 3.4, respectively.

(Continued Page 20)

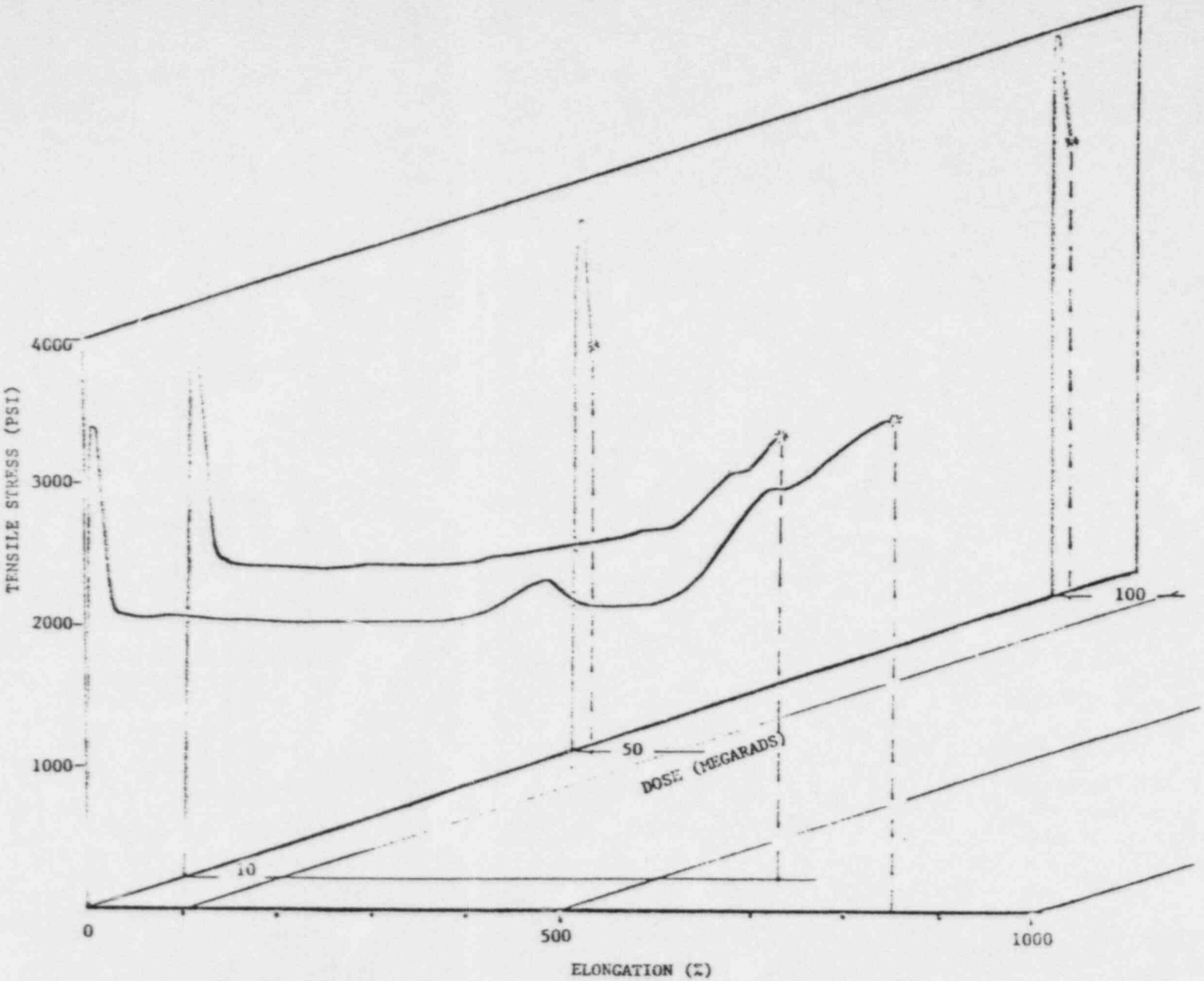

Figure 3.2. Three-dimensional plot of tensile stress (psi) vs elongation (%) vs gamma ray irradiation dose (Mrad) for CHEMPLEX 5701. The irradiations were performed in air at 10-11°C and 93 krad/h.

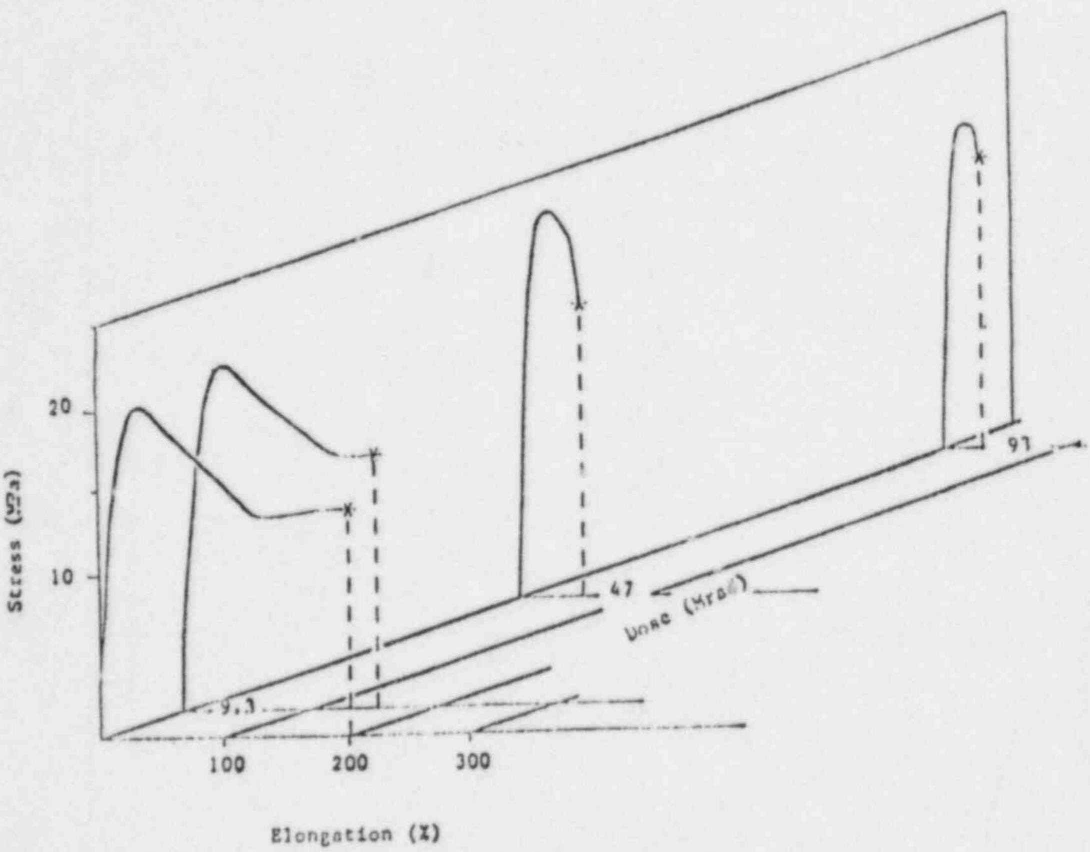

Figure 3.3. Three-dimensional plot of tensile stress (psi) vs elongation (%) vs gamma ray irradiation dose (Mrad) for Marlex CL-100 HIC material. The irradiations were performed in air at 10-11°C and 93 krad/h.

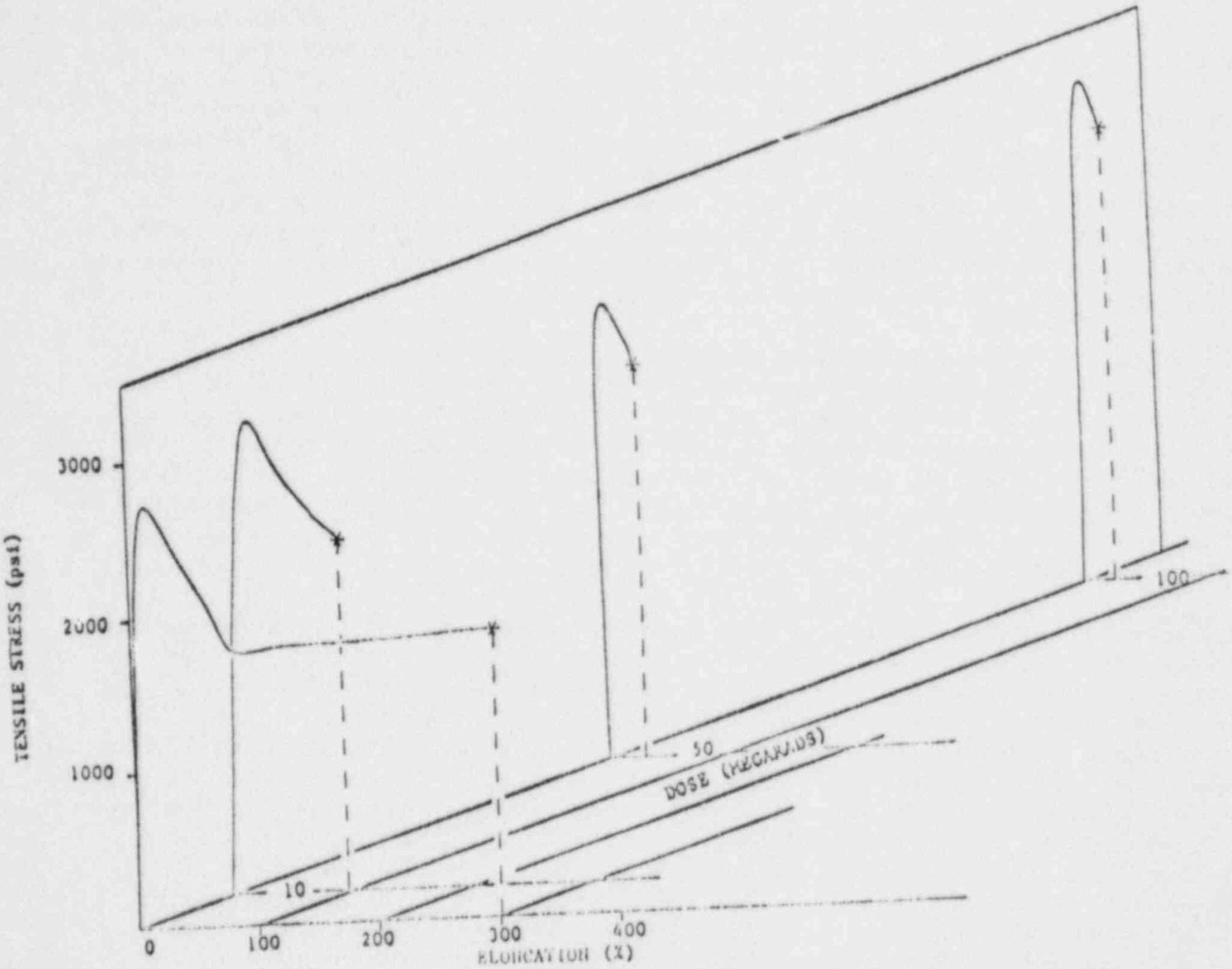

Figure 3.4 Three-dimensional plot of tensile stress (psi) vs elongation (%) vs dose (Mrad) for MARLEX CL-100 non-HIC material. The irradiations were performed in air at 10-11°C and 93 krad/h.

Unirradiated tensile specimens and irradiated specimens which still neck typically break somewhere along the necked region. In Chemplex, the break tends to occur suddenly, without warning. 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 seem 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 mechanism was observed quite clearly in the failure of several Marlex HIC material unirradiated specimens in which the bubble inclusions were clearly seen to pop and the tear which propagated through the specimen progressed from the popped bubble. This bubble defect mechanism of failure initiation for the Marlex materials may also explain the large variation in the elongation at break (vide infra).

Loss of necking behavior following irradiation is accompanied in all three materials by the appearance of cracks as they are pulled during tensile testing. 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. Figure 3.8 shows a closeup view of the cracks which occurred in the 47 Mrad specimens of Figure 3.3.

In both Marlex materials, the cracking always appears on the glossy surface, which corresponds to the inside surface of the container, and the cracks are generally evenly spaced along the entire narrow section of the test piece. An example of cracking in Marlex HIC material is shown in Figure 3.8. 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.[21] The glossy surface of the rotationally molded Marlex CL-100 has been found to be heavily oxidized, apparently by oxidation resulting from the contact with hot air during the molding process.[29]

Tensile data for the Chemplex, Marlex HIC material and Marlex non-HIC material irradiated at various dose rates in air, Barnwell and Hanford soils and IX resins are listed in Tables 3.1-3.3, respectively. Figures 3.9 thru 3.17 plot the data for yield stress, elongation at yield and elongation at break vs dose listed in the tables for each of these materials. Following the plots of tensile data vs dose, Figures 3.18-3.26 present plots of tensile data vs dose rate for selected data from Tables 3.1-3.3.

(Continued Page 45)

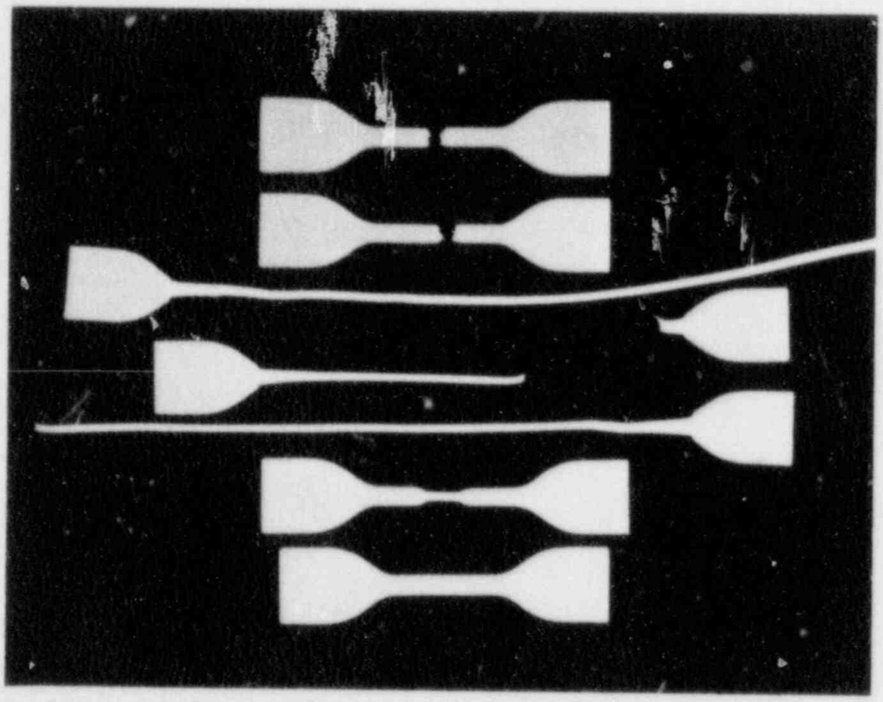

Figure 3.5. Chemplex tensile specimens including those which produced the curves in Figure 3.2. From bottom: an untested specimen, a specimen with a half-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specimen, the 47 Mrad specimen and the 93 Mrad specimen.

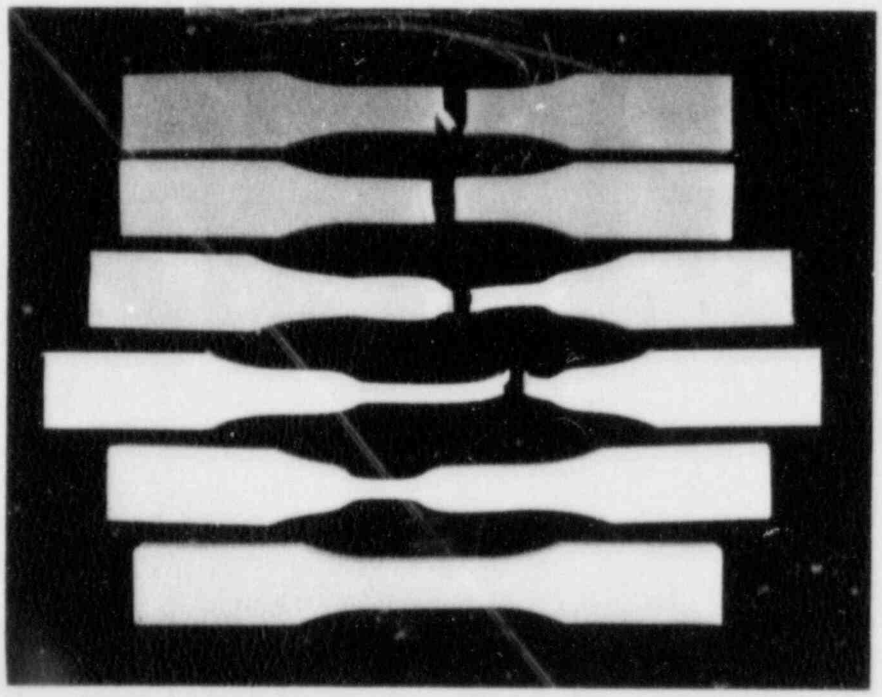

Figure 3.6. Marlex HIC material specimens including those which produced the curves in Figure 3.3. From bottom: an untested specimen, a specimen with a one-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specimen and the 93 Mrad specimen.

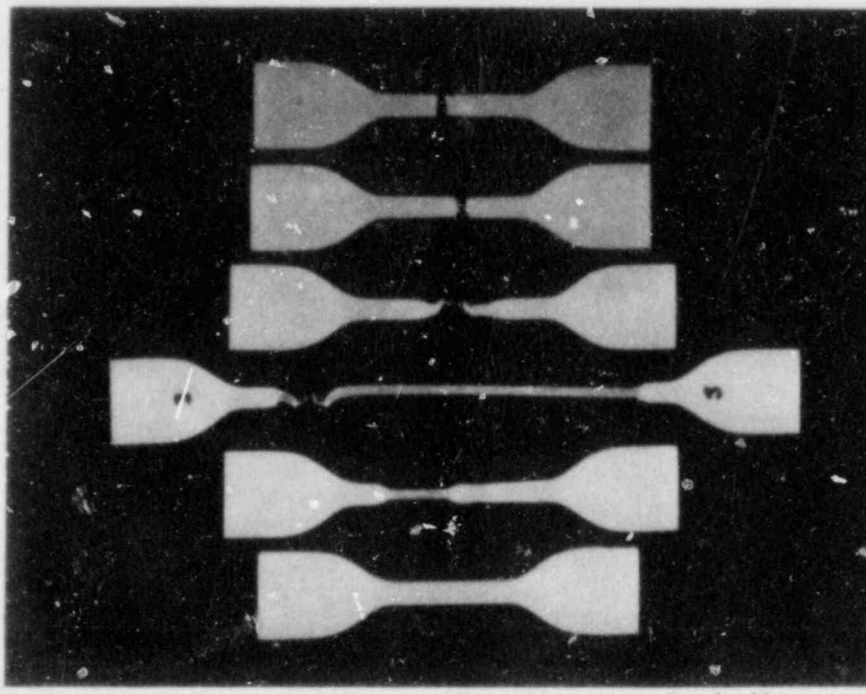

Figure 3.7. Marlex non-HIC material tensile specimens including those which produced the curves in Figure 3.4. From bottom: an untested specimen, a specimen with a three-fourth-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specimen, the 47 Mrad specimen and the 93 Mrad specimen.

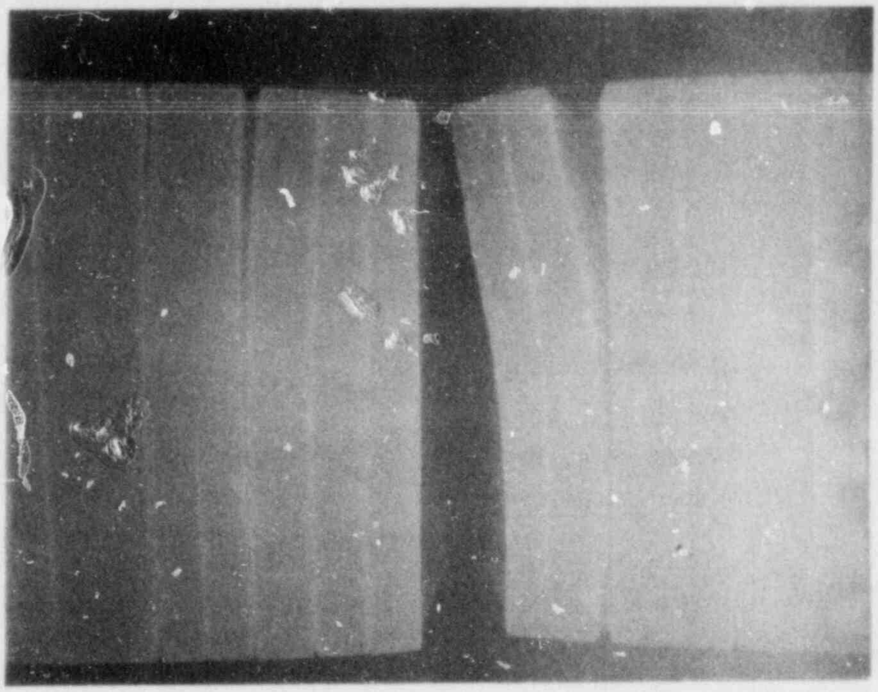

Figure 3.8. Closeup of the cracks which occurred in the Marlex HIC material tensile specimen irradiated to 47 Mrad of Figures 3.3 and 3.6.

Table 3.1

Tensile Test Data on Irradiated CHEMPLEX 5701[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
2-10	0[b]	----	----	3460 ± 120	15.4 ± 1.6	950 ± 40	N
3-28	0	----	----	3560 ± 30	16.7 ± 1.2	1030 ± 60	N
12-01	0	----	----	3740 ± 60	11.8 ± 0.5	1080 ± 10	N
12-29	0	----	----	3760[c]	10.8	1050	N
2-10	9.3	93	air	3760 ± 30	15.0 ± 0.5	660 ± 50	N
11-30	15	93	air	4210[c]	10.8	120	N
12-29	20	93	air	4600[c]	9.2	36	N
12-01	27	93	air	4240[c]	9.2	25	B
12-08	32	93	air	3940[c]	9.2	29	B
12-08	36	93	air	3970[c]	9.2	25	B
3-02	47	93	air	3740 ± 100	15.0 ± 0.1	32 ± 1	B
3-28	93	93	air	3950 ± 40	13.8 ± 0.1	27 ± 2	B
4-11	8.6	17	air	3730 ± 60	15.9 ± 0.9	730 ± 120	N
9-23	6.6	14	air	4200[c]	11.5	660	N
9-27	7.9	14	air	4300[c]	12.3	510	N
10-03	8.7	14	air	4100[c]	10.0	520	N
10-27	9.4	14	air	3970[c]	10.0	420	N
11-01	11	14	air	4100[c]	10.0	52	N
11-16	14	14	air	3970[c]	6.2	17	B
6-01	25	14	air	3980 ± 60	8.5 ± 2.0	20 ± 6	B
5-25	9.5	5.7	air	4050 ± 60	8.7 ± 1.7	18 ± 6	B
6-08	2.7	2.5	air	4350 ± 50	10.0 ± 1.3	820 ± 90	N
11-01	3.6	2.5	air	3980 ± 110	11.0 ± 0.4	640 ± 110	N
5-02	9.7	58	Barnwell soil	3730 ± 120	14.7 ± 0.6	120 ± 60	N
6-09	50	58	Barnwell soil	4690 ± 80	8.2 ± 0.9	21 ± 2	B
5-18	8.5	11	Barnwell soil	3790 ± 40	14.1 ± 1.2	30 ± 1	B
6-09	20	11	Barnwell soil	4540 ± 90	8.1 ± 0.9	22 ± 2	B
8-30	8.0	3.7	Barnwell soil	3660 ± 170	5.3 ± 2.8	11 ± 6	B
8-16	2.0	1.4	Barnwell soil	3930 ± 100	10.8 ± 0.1	830 ± 130	N
7-15	50	58	Hanford soil	4230 ± 100	9.2 ± 0.8	25 ± 1	B
6-28	8.5	11	Hanford soil	4050 ± 60	7.7 ± 0.1	23 ± 1	B
8-18	20	11	Hanford soil	4150 ± 20	11.0 ± 0.4	24 ± 1	B
10-06	3.0	4.0	Hanford soil	3950 ± 60	10.5 ± 0.5	680 ± 340	N
7-25	13	79	IX resin	4140 ± 30	10.0 ± 0.8	400 ± 190	N
8-18	49	79	IX resin	4370 ± 50	10.3 ± 0.5	26 ± 4	B
10-25	100	79	IX resin	4400 ± 80	9.2 ± 0.1	23 ± 3	B
9-14	20	11	IX resin	4080 ± 10	9.5 ± 0.9	25 ± 2	B
8-16	10	8.7	IX resin	3790 ± 50	9.2 ± 0.8	22 ± 3	B
7-25	3.0	5.7	IX resin	3930 ± 60	10.3 ± 0.5	910 ± 130	N

[a] Irradiations were performed in the BNL Co-60 gamma facility at 10°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using three Type IV specimens per test. Test specimens were stamped (ASTM D-412, die C) from material obtained from two plain (not color pigmented) 55-gal drums purchased from PLASTI-DRUM Co., Lockport, IL.

[b] Five unirradiated specimens were tested.

[c] Only one specimen was tested.

Table 3.2

Tensile Test Data on Irradiated EnviroSAFE*
High-Integrity Container Material[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
1-24	0[b]	----	----	2880 ± 180	32 ± 1	220 ± 100	N
4-12	0[c]	----	----	2970 ± 40	33 ± 1	210 ± 90	N
12-08	0	----	----	3005	31	203	N
3-28	9.3	93	air	3140	33	160	N
3-02	47	93	air	3410	33	51	B
3-28	93	93	air	2880	22	32	B
4-11	8.6	17	air	3200	31	66	B
6-01	25	14	air	3300	31	57	B
5-25	9.5	5.7	air	3150	29	47	B
6-09	2.7	2.5	air	3370	28	120	N
11-01	3.6	2.5	air	3070	30	66	B
5-02	9.7	58	Barnwell soil	3110	29	130	N
6-09	50	58	Barnwell soil	3700	22	44	B
5-18	8.5	11	Barnwell soil	3000	28	64	B
6-09	20	11	Barnwell soil	3570	28	60	B
8-30	3.0	4.0	Barnwell soil	3250	29	130	N
8-30	8.0	3.7	Barnwell soil	3240	27	43	B
8-16	2.0	1.4	Barnwell soil	3040	33	110	N
7-15	50	58	Hanford soil	3310	27	52	B
6-28	8.5	11	Hanford soil	3040	25	49	B
8-18	20	11	Hanford soil	3240	27	50	B
10-06	3.0	4.0	Hanford soil	3060	31	170	N
7-25	13	79	IX resin	2940	31	110	N
8-18	49	79	IX resin	3360	28	47	B
10-25	100	79	IX resin	3530	27	36	B
9-14	20	11	IX resin	3010	28	76	B
8-16	10	8.7	IX resin	2910	27	64	B
8-30	3.0	4.0	IX resin	3210	30	200	N
7-25	3.0	3.7	IX resin	3110	29	60	B
10-03	3.0	4.0	IX resin/ Barnwell soil	3100	28	56	B
11-16	10	11	IX resin/ Barnwell soil	3030	29	62	B

*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 in the BNL Co-60 gamma facility at 10-11°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using one Type III specimen per test. Test specimens were machined from HIC material cut out during container fabrication. These container cut-outs were obtained from Poly-Processing Co., Monroe, LA, who manufactures these HICs for CHEM-NUCLEAR SYSTEMS, Inc.

[b]These data are from seven unirradiated specimens.

[c]Three specimens were tested.

Table 3.3

Tensile Test Data on Irradiated Rotationally Molded Marlex CL-100[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
3-07	0[b]	----	----	2730 ± 30	21.0 ± 1.0	300 ± 100	N
10-10	0[b]	----	----	3230 ± 60	19.5 ± 0.7	230 ± 55	N
11-16	0[b]	----	----	3110 ± 60	16.8 ± 0.8	210 ± 70	N
12-01	0[b]	----	----	3150 ± 40	18.0 ± 0.9	240 ± 75	N
12-08	0[b]	----	----	3030 ± 70	16.6 ± 0.4	140 ± 60	N
12-29	0[b]	----	----	3090 ± 100	17.4 ± 0.7	180 ± 70	N
3-07	9.3	93	air	2890 ± 40	20.0 ± 1.0	100 ± 6	N
11-30	15	93	air	3430 ± 20	15.7 ± 0.5	74 ± 8	N
12-29	20	93	air	3320 ± 50	15.1 ± 0.5	60 ± 4	B
12-01	27	93	air	3410 ± 40	15.4 ± 0.1	55 ± 2	B
12-08	32	93	air	3310 ± 20	14.9 ± 0.9	42 ± 8	B
12-08	36	93	air	3230 ± 110	14.9 ± 0.9	46 ± 7	B
3-28	47	93	air	2870 ± 30	17.5 ± 0.5	42 ± 3	B
4-18	93	93	air	3130 ± 40	18.7 ± 1.7	35 ± 3	B
4-12	8.6	17	air	2910 ± 50	20.5 ± 0.5	66 ± 3	B
8-30	5.1	14	air	3140 ± 30	16.9 ± 0.9	170 ± 40	N
9-23	6.3	14	air	3260 ± 160	15.9 ± 1.8	90 ± 10	N
9-27	7.0	14	air	3320 ± 30	16.9 ± 0.1	68 ± 22	N
10-27	9.4	14	air	3190 ± 130	15.4 ± 0.1	52 ± 5	B
11-01	11	14	air	3190 ± 10	15.4 ± 0.1	57 ± 9	B
6-02	25	14	air	3140 ± 120	13.8 ± 0.8	31 ± 3	B
5-25	9.5	5.7	air	3040 ± 30	15.4 ± 0.1	43 ± 4	B
11-09	0.5	4.2	air	3140 ± 20	16.4 ± 0.4	105 ± 5	N
11-09	1.0	4.2	air	3020 ± 60	16.7 ± 0.4	200 ± 90	N
11-16	1.5	4.2	air	3230 ± 10	16.9 ± 0.1	175 ± 45	N
11-23	2.0	4.2	air	3160 ± 50	16.7 ± 0.4	135 ± 45	N
11-30	2.5	4.2	air	3280 ± 60	15.9 ± 0.9	105 ± 20	N
12-09	3.9	4.2	air	3130 ± 70	15.1 ± 2.0	55 ± 6	B
11-16	0.5	2.5	air	3100 ± 20	17.7 ± 1.4	180 ± 35	N
11-16	0.6	2.5	air	3170 ± 90	16.4 ± 0.9	170 ± 120	N
11-23	1.0	2.5	air	3210 ± 100	16.4 ± 0.4	125 ± 40	N
12-01	1.6	2.5	air	3170 ± 50	16.9 ± 0.8	200 ± 120	N
6-09	2.7	2.5	air	3570 ± 50	16.4 ± 0.9	95 ± 21	N
11-01	3.6	2.5	air	3160 ± 100	15.7 ± 0.5	60 ± 11	B
5-02	9.7	58	Barnwell soil	2950 ± 50	20.5 ± 0.9	83 ± 9	N
6-09	50	58	Barnwell soil	3750 ± 70	12.5 ± 0.9	37 ± 7	B
5-18	8.5	11	Barnwell soil	2910 ± 20	19.2 ± 0.1	62 ± 10	B
6-09	20	11	Barnwell soil	3510 ± 80	14.2 ± 0.7	39 ± 2	B
8-30	8.0	3.7	Barnwell soil	3110 ± 40	13.8 ± 0.1	35 ± 7	B
8-16	2.0	1.4	Barnwell soil	3110 ± 60	10.5 ± 0.5	70 ± 3	N
7-15	50	58	Hanford soil	3470 ± 40	13.5 ± 0.5	34 ± 6	B
6-28	8.5	11	Hanford soil	3190 ± 30	15.0 ± 0.1	56 ± 16	B
8-18	20	11	Hanford soil	3190 ± 40	14.9 ± 0.9	36 ± 6	B
10-06	3.0	4.0	Hanford soil	3260 ± 50	15.9 ± 0.9	86 ± 21	N
7-25	13	79	IX resin	3140 ± 150	16.2 ± 0.8	110 ± 20	N
8-18	49	79	IX resin	3500 ± 80	15.4 ± 0.1	43 ± 5	B
10-25	100	79	IX resin	3520 ± 20	13.1 ± 0.1	37 ± 9	B
9-14	20	11	IX resin	3110 ± 60	14.9 ± 0.9	42 ± 4	B
8-16	10	8.7	IX resin	3040 ± 60	14.6 ± 0.8	37 ± 7	B
7-25	3.0	3.7	IX resin	3190 ± 30	16.2 ± 0.8	60 ± 12	B

[a]Irradiations were performed in the BNL Co-60 gamma facility at 10°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using three Type IV specimens per test. Test specimens were stamped (ASTM D-412, die C) from the sidewall of a 1200-gal rotationally molded container purchased from Poly-Processing Co., Monroe, LA.

[b]Five unirradiated specimens were tested.

Legend for Data Figures

□ - air

○ - Barnwell soil

Δ - Hanford soil

X - IX resin

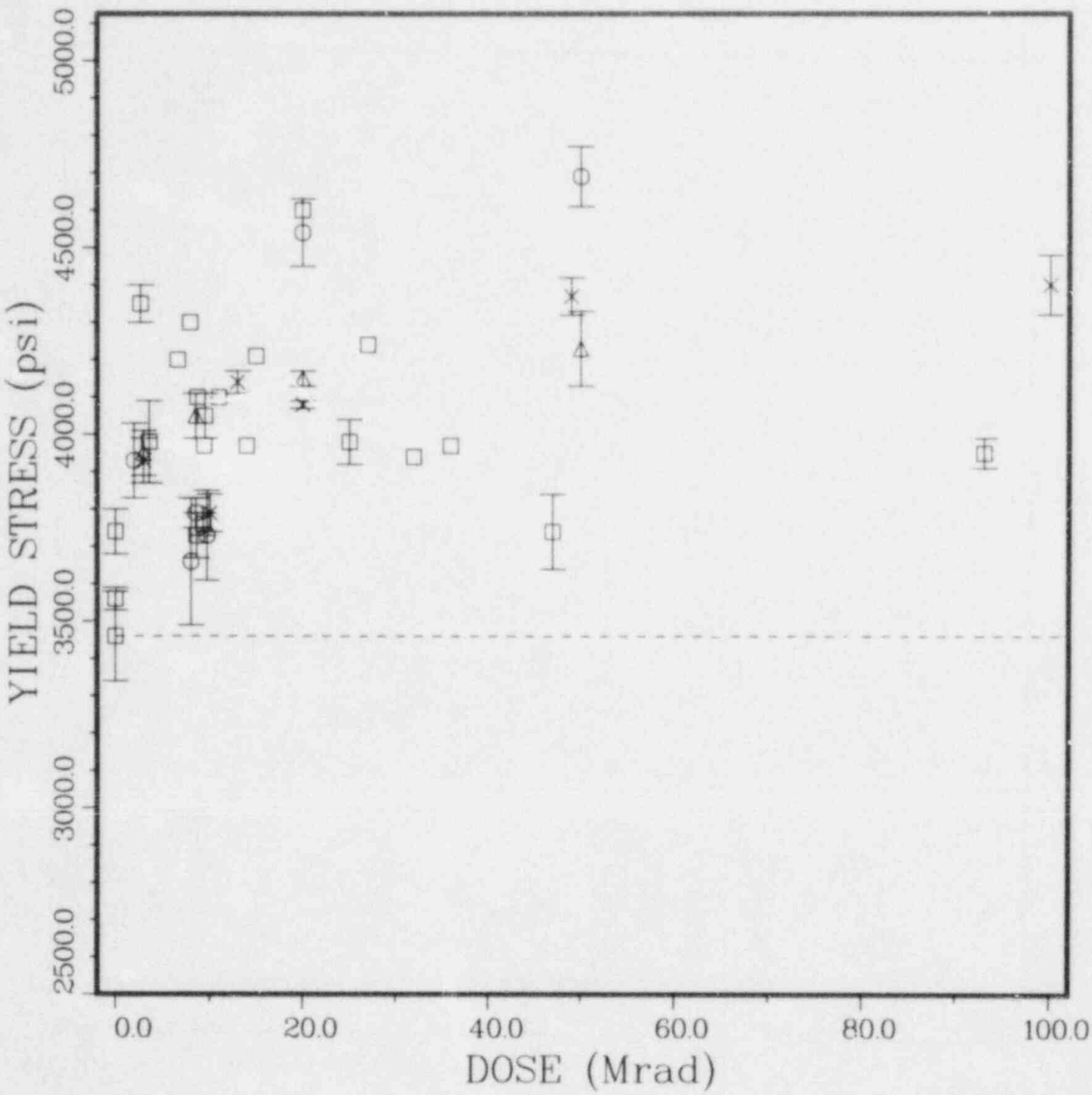

Figure 3.9. Yield stress (psi) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

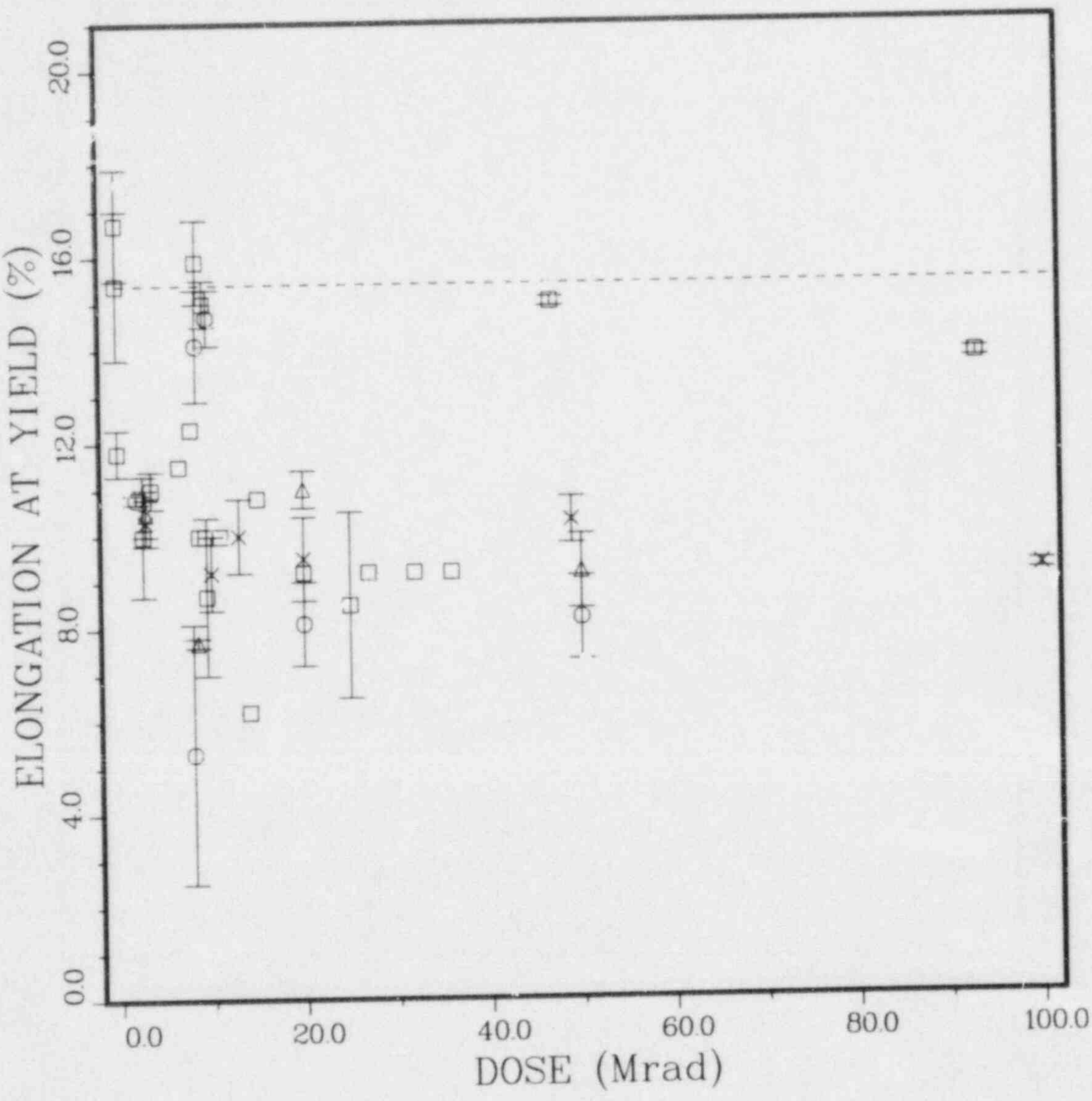

Figure 3.10. Elongation at yield (%) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

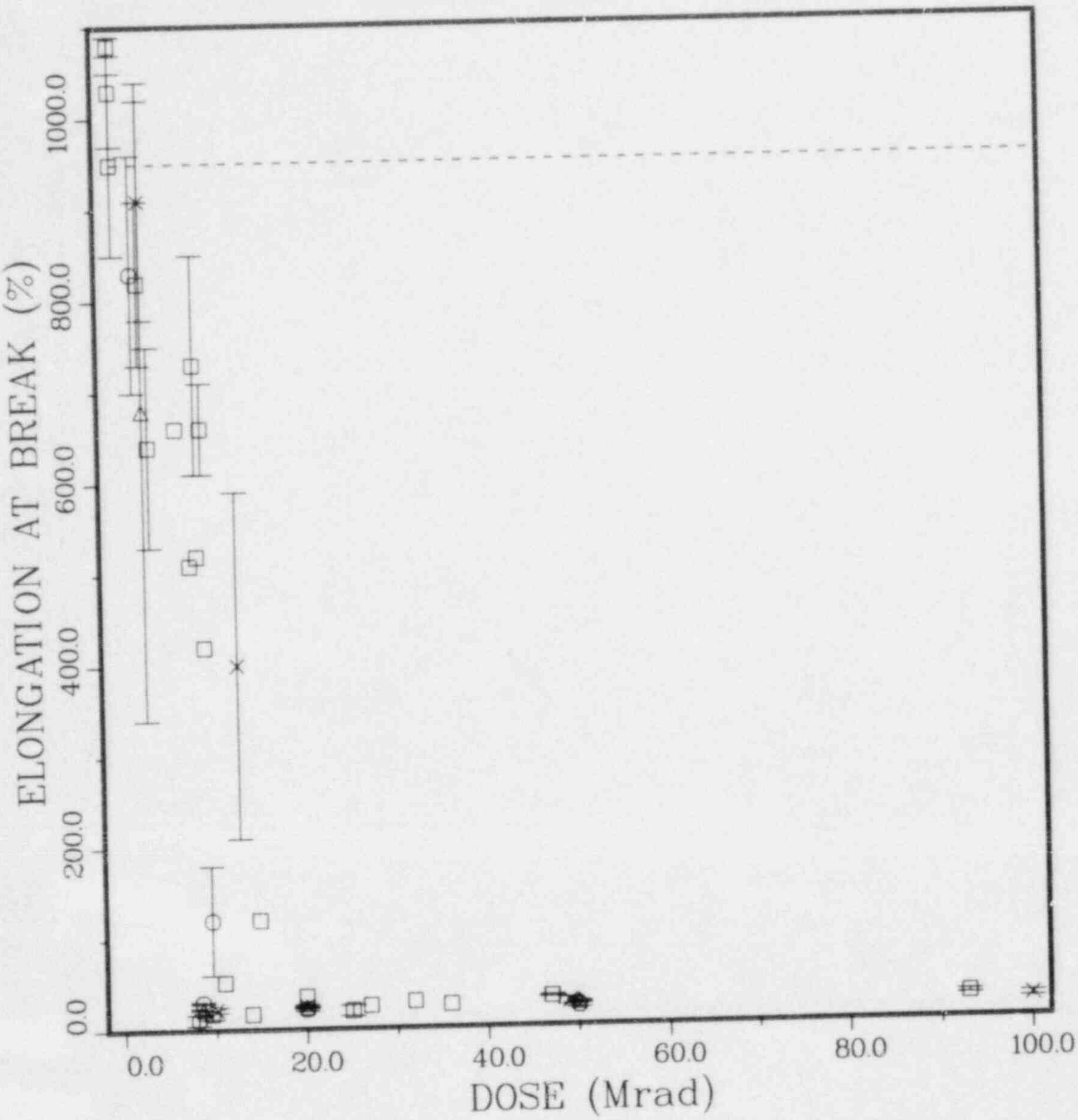

Figure 3.11. Elongation at break (%) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

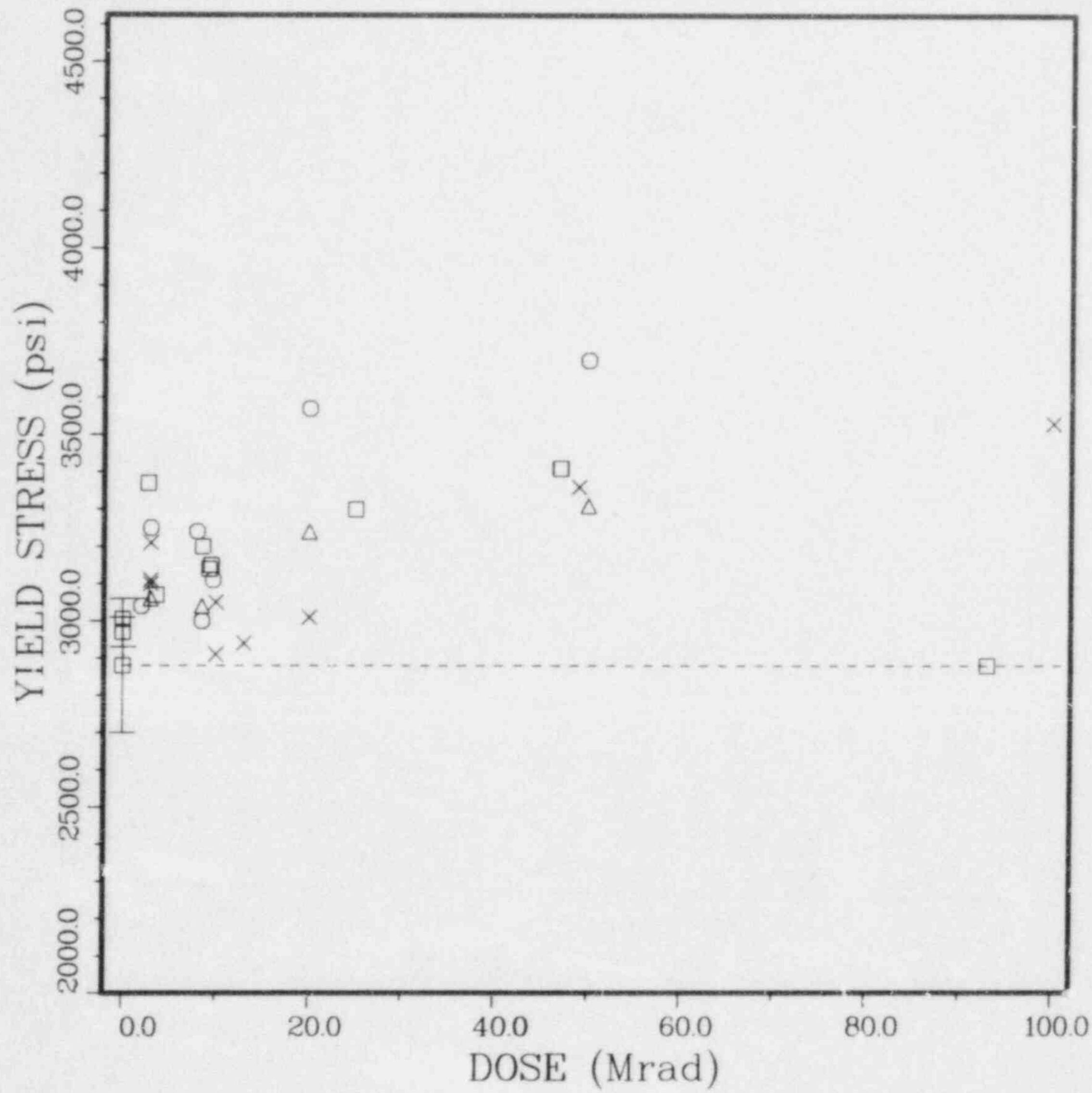

Figure 3.12. Yield stress (psi) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data are from Table 3.2. Symbols are defined in the Legend.

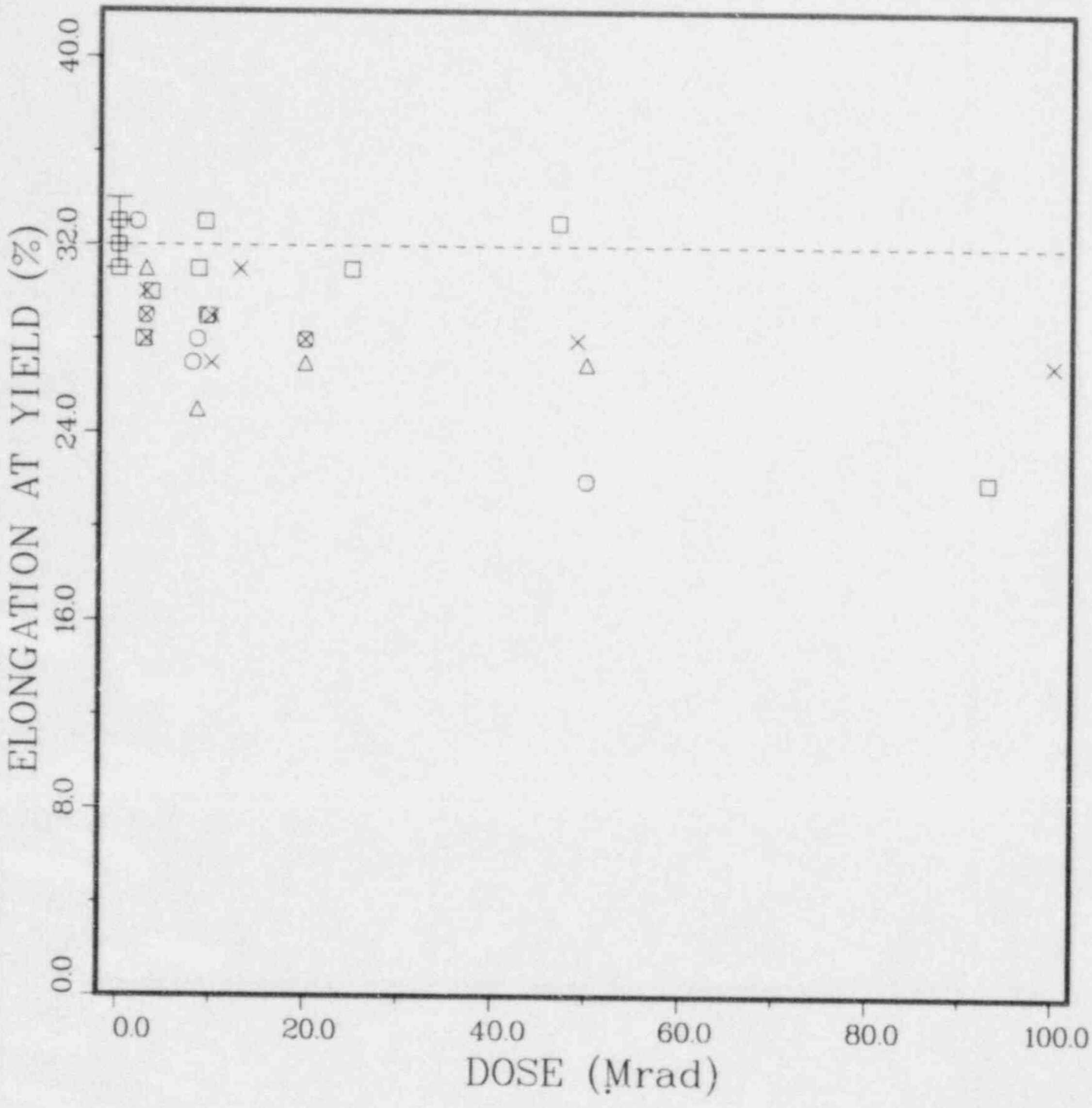

Figure 3.13. Elongation at yield (%) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data is from Table 3.2. Symbols are defined in the Legend.

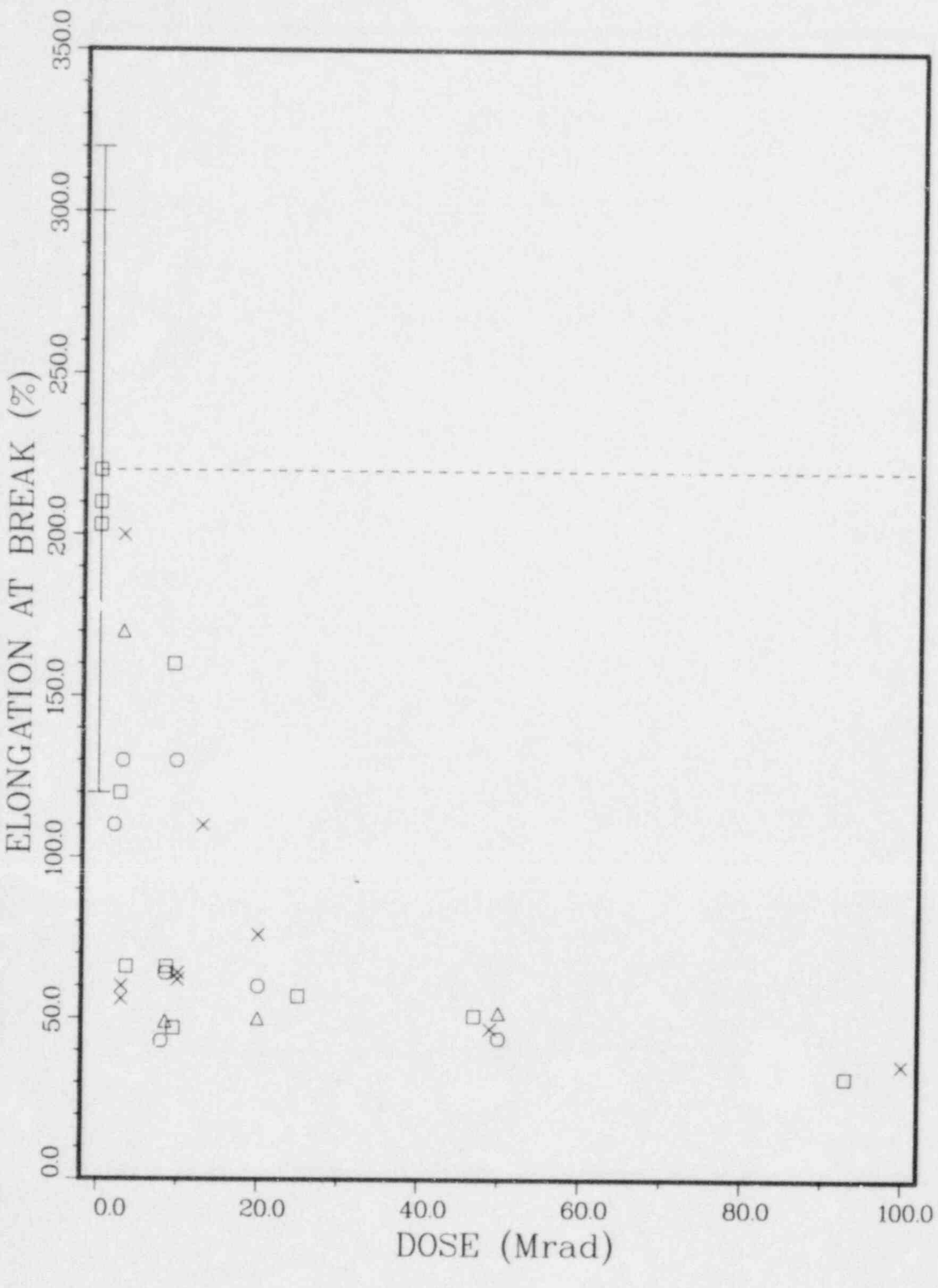

Figure 3.14. Elongation at break (%) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data is from Table 3.2. Symbols are defined in the Legend.

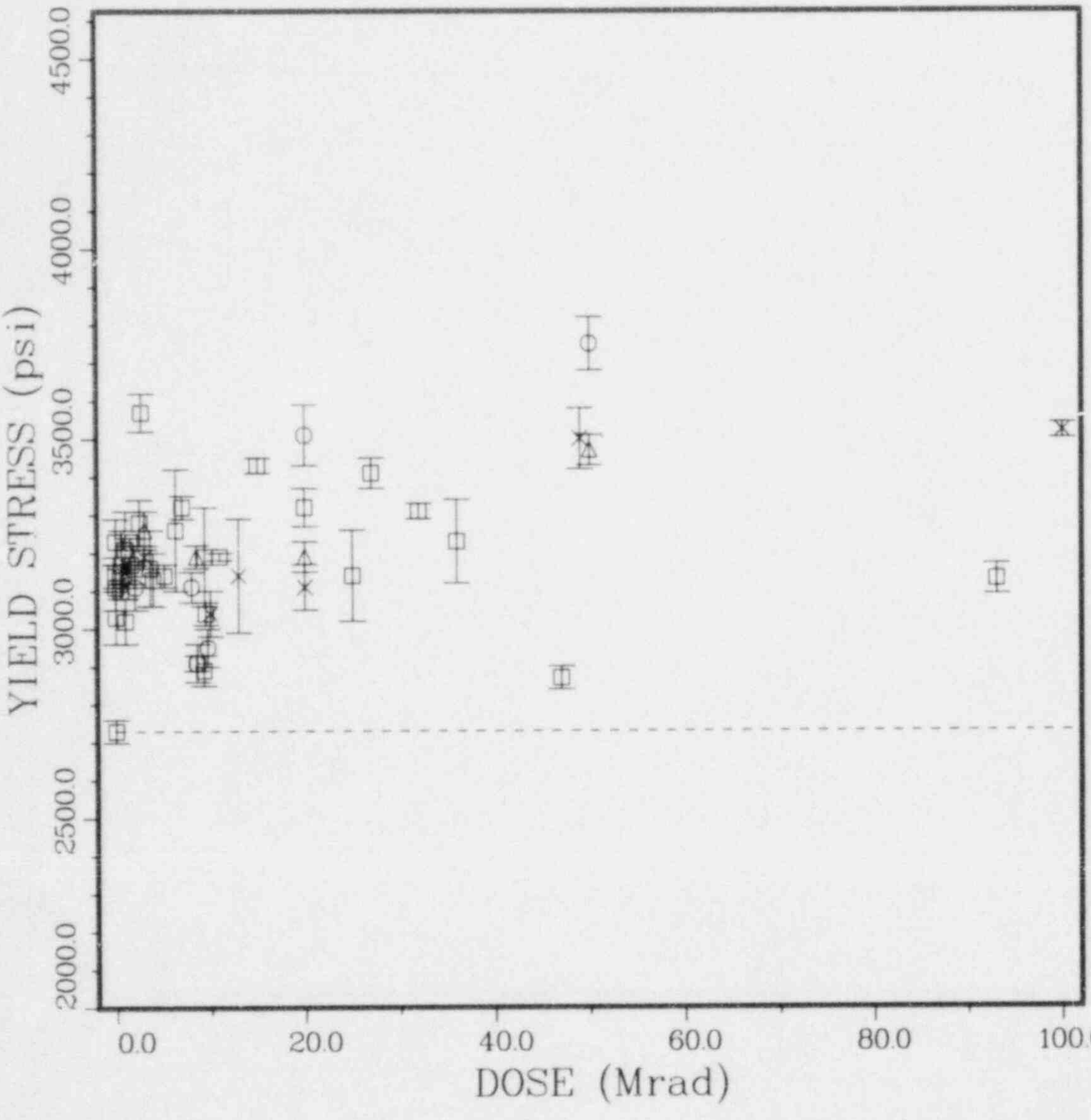

Figure 3.15. Yield stress (psi) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

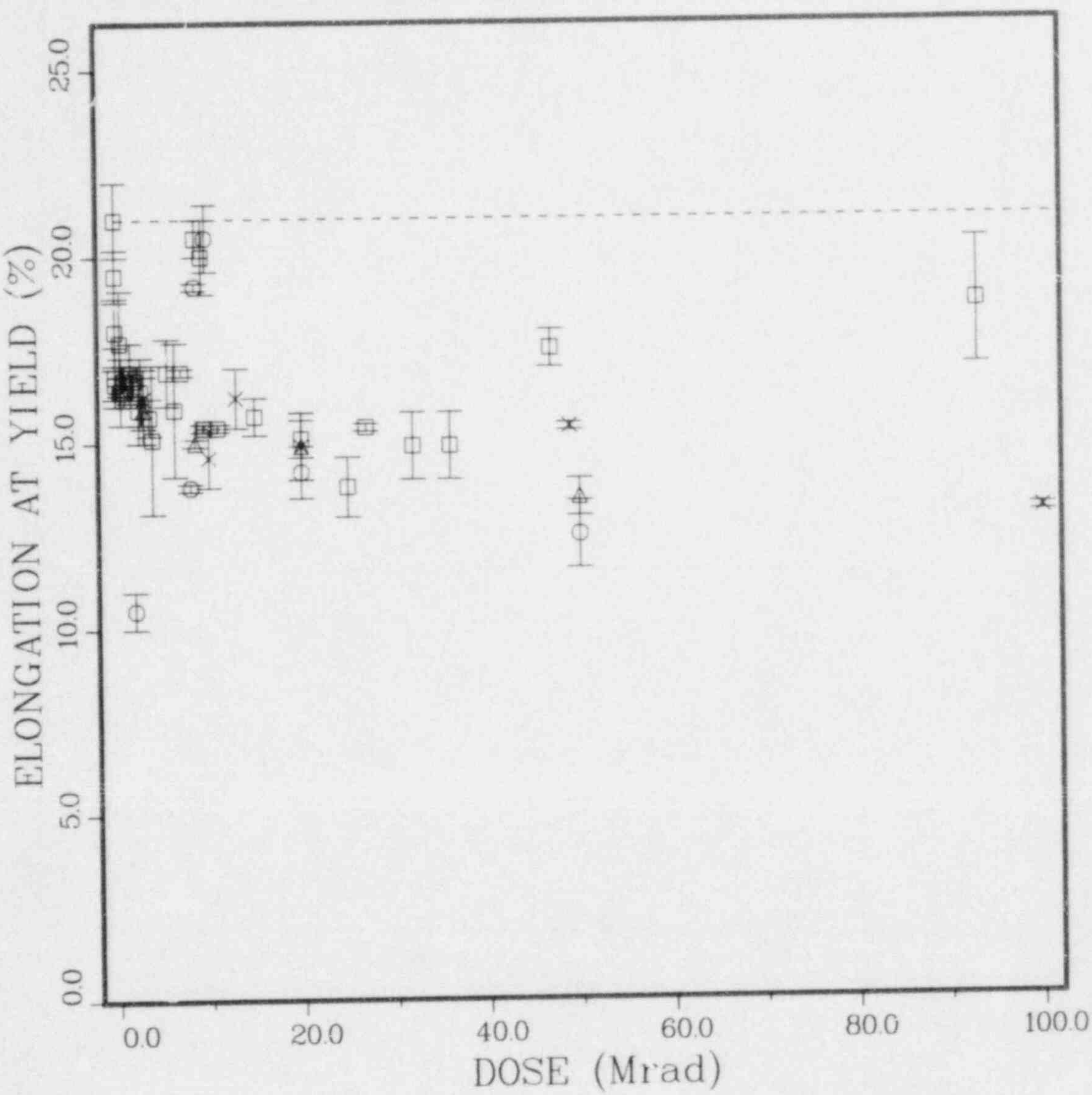

Figure 3.16. Elongation at yield (%) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

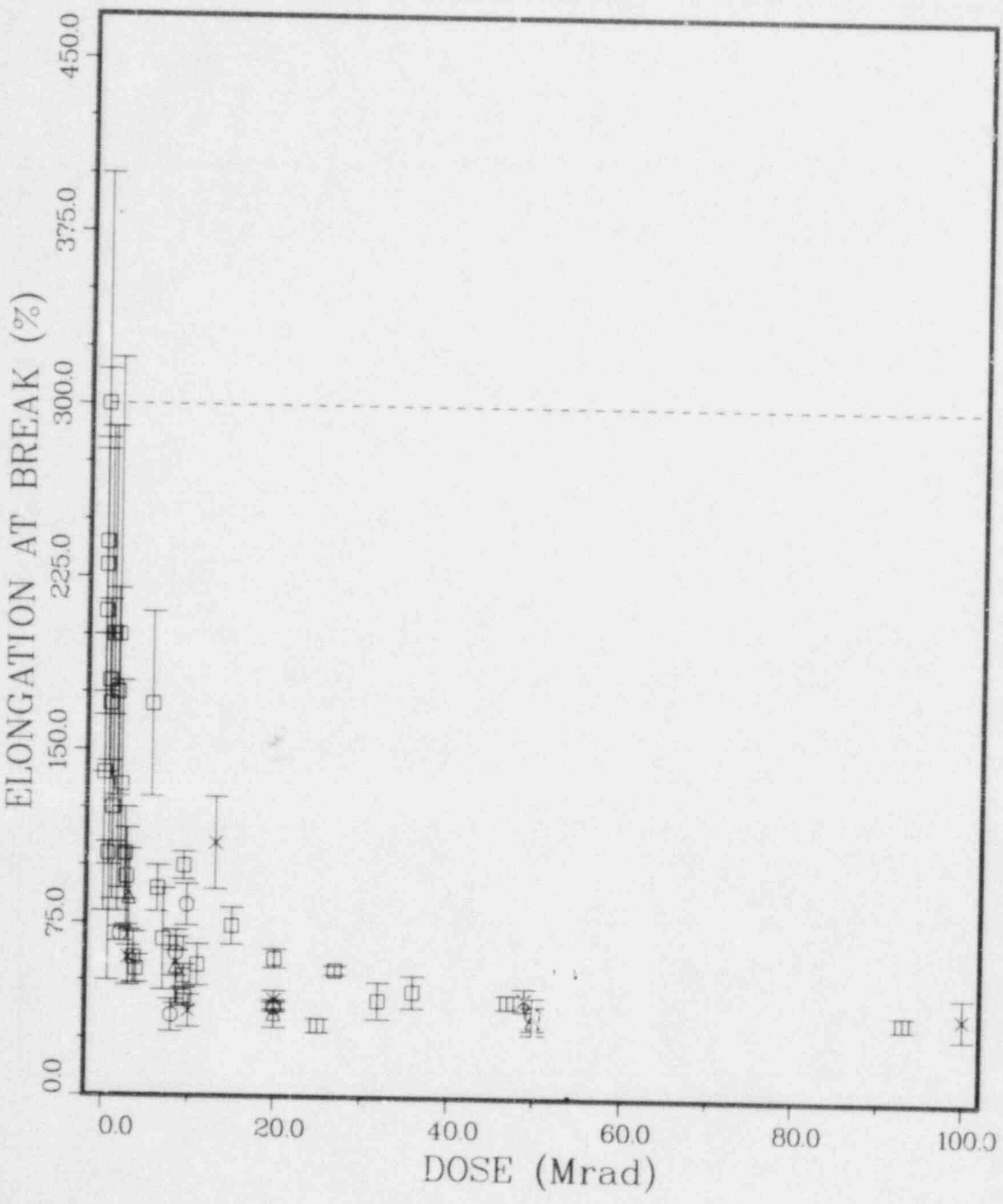

Figure 3.17. Elongation at break (%) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

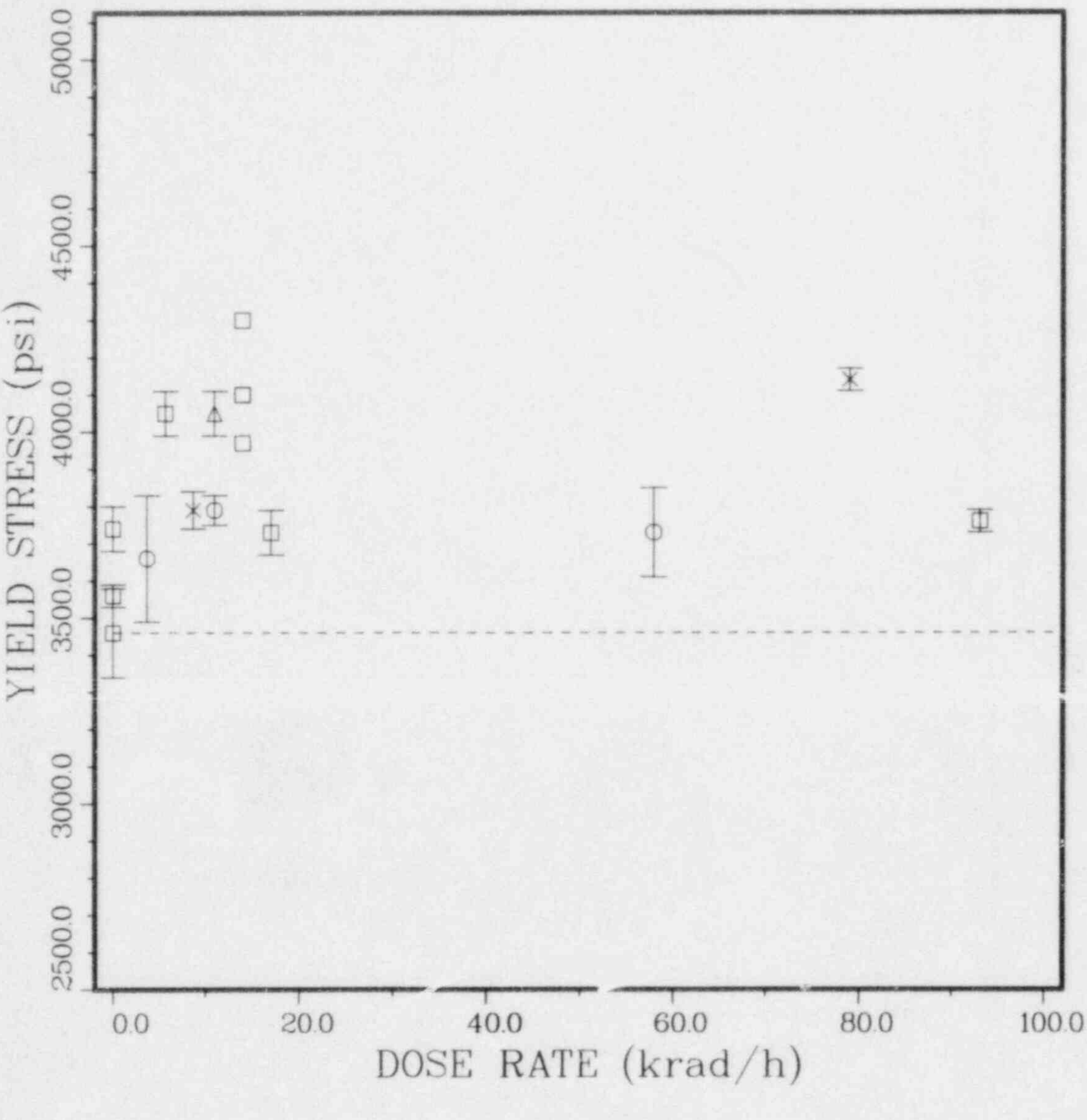

Figure 3.18. Yield stress (psi) vs dose rate (krad/h) of irradiated Chemplex 5701 for total doses from 7.9-13 Mrad. The data are from Table 3.1. Symbols are defined in the legend.

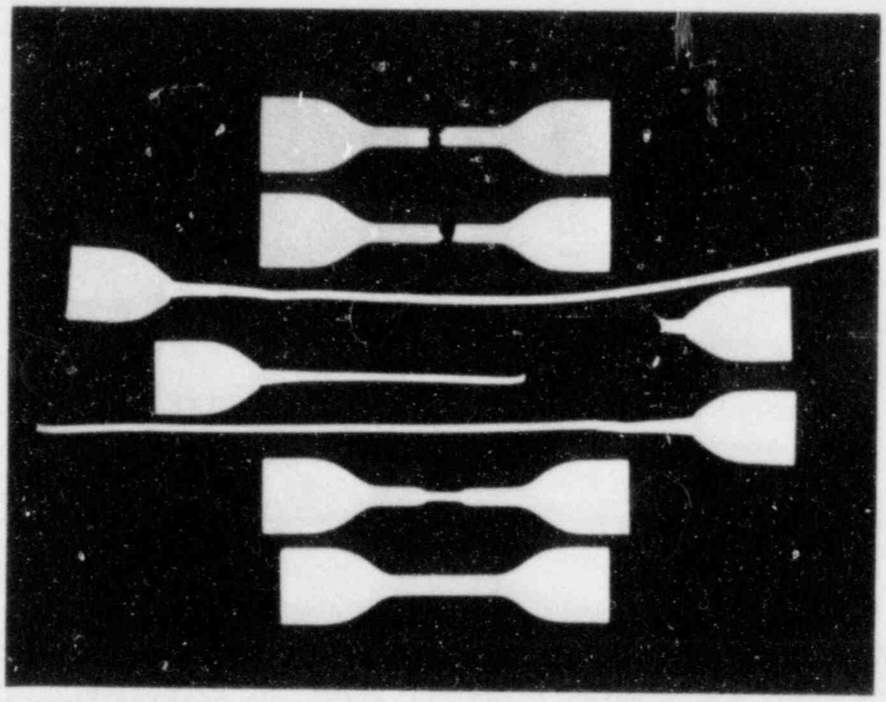

Figure 3.5. Chemplex tensile specimens including those which produced the curves in Figure 3.2. From bottom: an untested specimen, a specimen with a half-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specir , the 47 Mrad specimen and the 93 Mrad specimen.

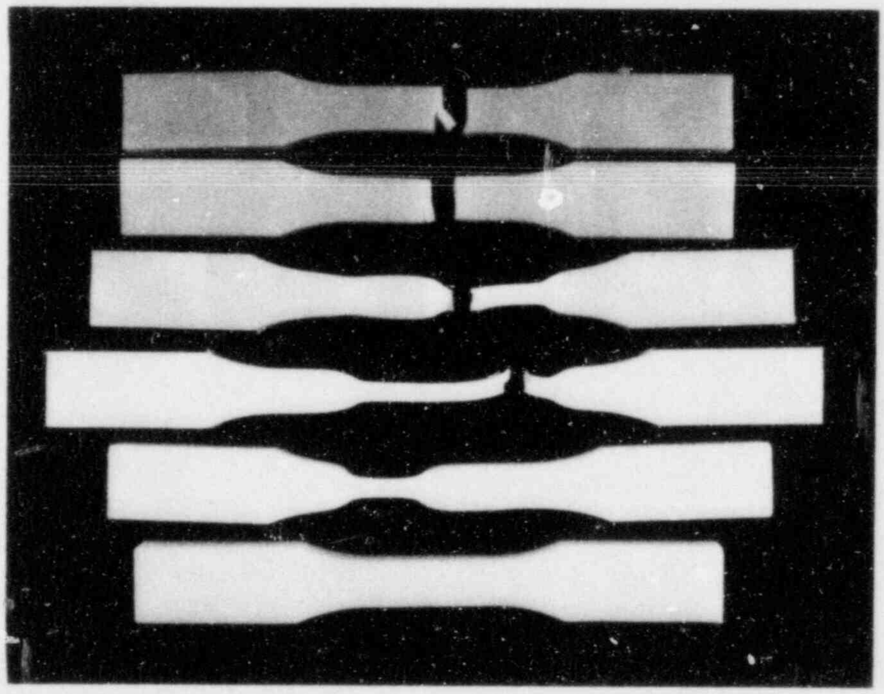

Figure 3.6. Marlex HIC material specimens including those which produced the curves in Figure 3.3. From bottom: an untested specimen, a specimen with a one-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specimen and the 93 Mrad specimen.

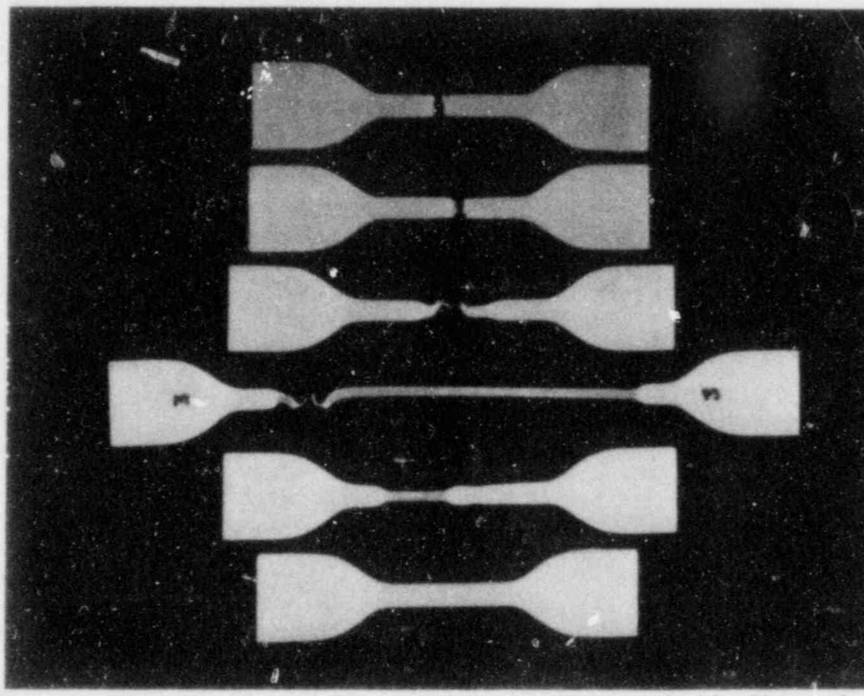

Figure 3.7. Marlex non-HIC material tensile specimens including those which produced the curves in Figure 3.4. From bottom: an untested specimen, a specimen with a three-fourth-inch neck, the unirradiated specimen pulled to the break, the 9.3 Mrad specimen, the 47 Mrad specimen and the 93 Mrad specimen.

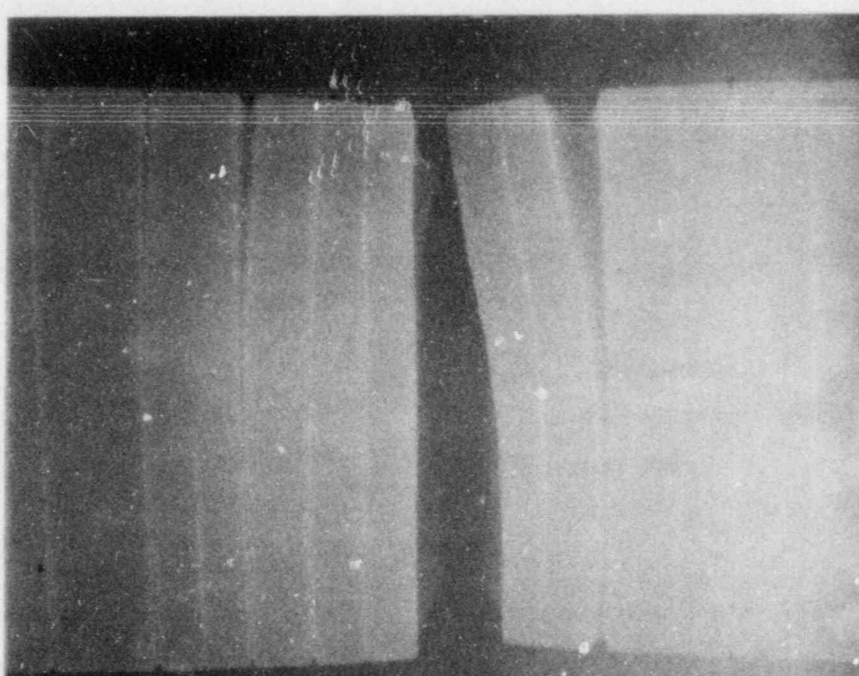

Figure 3.8. Closeup of the cracks which occurred in the Marlex HIC material tensile specimen irradiated to 47 Mrad of Figures 3.3 and 3.6.

Table 3.1

Tensile Test Data on Irradiated CHEMPLEX 5701[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
2-10	0[b]	----	----	3460 ± 120	15.4 ± 1.6	950 ± 40	N
3-28	0	----	----	3560 ± 30	16.7 ± 1.2	1030 ± 60	N
12-01	0	----	----	3740 ± 60	11.8 ± 0.5	1080 ± 10	N
12-29	0	----	----	3760[c]	10.8	1050	N
2-10	9.3	93	air	3760 ± 30	15.0 ± 0.5	660 ± 50	N
11-30	15	93	air	4210[c]	10.8	120	N
12-29	20	93	air	4600[c]	9.2	36	N
12-01	27	93	air	4240[c]	9.2	25	B
12-08	32	93	air	3940[c]	9.2	29	B
12-08	36	93	air	3970[c]	9.2	25	B
3-02	47	93	air	3740 ± 100	15.0 ± 0.1	32 ± 1	B
3-28	93	93	air	3950 ± 40	13.8 ± 0.1	27 ± 2	B
4-11	8.6	17	air	3730 ± 60	15.9 ± 0.9	730 ± 120	N
9-23	6.6	14	air	4200[c]	11.5	660	N
9-27	7.9	14	air	4300[c]	12.3	510	N
10-03	8.7	14	air	4100[c]	10.0	520	N
10-27	9.4	14	air	3970[c]	10.0	420	N
11-01	11	14	air	4100[c]	10.0	52	N
11-16	14	14	air	3970[c]	6.2	17	B
6-01	25	14	air	3980 ± 60	8.5 ± 2.0	20 ± 6	B
5-25	9.5	5.7	air	4050 ± 60	8.7 ± 1.7	18 ± 6	B
6-08	2.7	2.5	air	4350 ± 50	10.0 ± 1.3	820 ± 90	N
11-01	3.6	2.5	air	3980 ± 110	11.0 ± 0.4	640 ± 110	N
5-02	9.7	58	Barnwell soil	3730 ± 120	14.7 ± 0.6	120 ± 60	N
6-09	50	58	Barnwell soil	4690 ± 80	8.2 ± 0.9	21 ± 2	B
5-18	8.5	11	Barnwell soil	3790 ± 40	14.1 ± 1.2	30 ± 1	B
6-09	20	11	Barnwell soil	4540 ± 90	8.1 ± 0.9	22 ± 2	B
8-30	8.0	3.7	Barnwell soil	3660 ± 170	5.3 ± 2.8	11 ± 6	B
8-16	2.0	1.4	Barnwell soil	3930 ± 100	10.8 ± 0.1	830 ± 130	N
7-15	50	58	Hanford soil	4230 ± 100	9.2 ± 0.8	25 ± 1	B
6-28	8.5	11	Hanford soil	4050 ± 60	7.7 ± 0.1	23 ± 1	B
8-18	20	11	Hanford soil	4150 ± 20	11.0 ± 0.4	24 ± 1	B
10-06	3.0	4.0	Hanford soil	3950 ± 60	10.5 ± 0.5	680 ± 340	N
7-25	13	79	IX resin	4140 ± 30	10.0 ± 0.8	400 ± 190	N
8-18	49	79	IX resin	4370 ± 50	10.3 ± 0.5	26 ± 4	B
10-25	100	79	IX resin	4400 ± 80	9.2 ± 0.1	23 ± 3	B
9-14	20	11	IX resin	4080 ± 10	9.5 ± 0.9	25 ± 2	B
8-16	10	8.7	IX resin	3790 ± 50	9.2 ± 0.8	22 ± 3	B
7-25	3.0	3.7	IX resin	3930 ± 60	10.3 ± 0.5	910 ± 130	N

[a]Irradiations were performed in the BNL Co-60 gamma facility at 10°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using three Type IV specimens per test. Test specimens were stamped (ASTM D-412, die C) from material obtained from two plain (not color pigmented) 55-gal drums purchased from PLASTI-DRUM Co., Lockport, IL.
[b]Five unirradiated specimens were tested.
[c]Only one specimen was tested.

Table 3.2

Tensile Test Data on Irradiated EnviroSAFE*
High-Integrity Container Material[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
1-24	0[b]	----	----	2880 $\pm$ 180	32 $\pm$ 1	220 $\pm$ 100	N
4-12	0[c]	----	----	2970 $\pm$ 40	33 $\pm$ 1	210 $\pm$ 90	N
12-08	0	----	----	3005	31	203	N
3-28	9.3	93	air	3140	33	160	N
3-02	47	93	air	3410	33	51	B
3-28	93	93	air	2880	22	32	B
4-11	8.6	17	air	3200	31	66	B
6-01	25	14	air	3300	31	57	B
5-25	9.5	5.7	air	3150	29	47	B
6-09	2.7	2.5	air	3370	28	120	N
11-01	3.6	2.5	air	3070	30	66	B
5-02	9.7	58	Barnwell soil	3110	29	130	N
6-09	50	58	Barnwell soil	3700	22	44	B
5-18	8.5	11	Barnwell soil	3000	28	64	B
6-09	20	11	Barnwell soil	3570	28	60	B
8-30	3.0	4.0	Barnwell soil	3250	29	130	N
8-30	8.0	3.7	Barnwell soil	3240	27	43	B
8-16	2.0	1.4	Barnwell soil	3040	33	110	N
7-15	50	58	Hanford soil	3310	27	52	B
6-28	8.5	11	Hanford soil	3040	25	49	B
8-18	20	11	Hanford soil	3240	27	50	B
10-06	3.0	4.0	Hanford soil	3060	31	170	N
7-25	13	79	IX resin	2940	31	110	N
8-18	49	79	IX resin	3360	28	47	B
10-25	100	79	IX resin	3530	27	36	B
9-14	20	11	IX resin	3010	28	76	B
8-16	10	8.7	IX resin	2910	27	64	B
8-30	3.0	4.0	IX resin	3210	30	200	N
7-25	3.0	3.7	IX resin	3110	29	60	B
10-03	3.0	4.0	IX resin/ Barnwell soil	3100	28	56	B
11-16	10	11	IX resin/ Barnwell soil	3050	29	62	B

*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 in the BNL Co-60 gamma facility at 10-11°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using one Type III specimen per test. Test specimens were machined from HIC material cut out during container fabrication. These container cut-outs were obtained from Poly-Processing Co., Monroe, LA, who manufactures these HICs for CHEM-NUCLEAR SYSTEMS, Inc.

[b]These data are from seven unirradiated specimens.

[c]Three specimens were tested.

Table 3.3

Tensile Test Data on Irradiated Rotationally Molded Marlex CL-100[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
3-07	0[b]	----	----	2730 ± 30	21.0 ± 1.0	300 ± 100	N
10-10	0[b]	----	----	3230 ± 60	19.5 ± 0.7	230 ± 55	N
11-16	0[b]	----	----	3110 ± 60	16.8 ± 0.8	210 ± 70	N
12-01	0[b]	----	----	3150 ± 40	18.0 ± 0.9	240 ± 75	N
12-08	0[b]	----	----	3030 ± 70	16.6 ± 0.4	140 ± 60	N
12-29	0[b]	----	----	3090 ± 100	17.4 ± 0.7	180 ± 70	N
3-07	9.3	93	air	2890 ± 40	20.0 ± 1.0	100 ± 6	N
11-30	15	93	air	3430 ± 20	15.7 ± 0.5	74 ± 8	N
12-29	20	93	air	3320 ± 50	15.1 ± 0.5	60 ± 4	B
12-01	27	93	air	3410 ± 40	15.4 ± 0.1	55 ± 2	B
12-08	32	93	air	3310 ± 20	14.9 ± 0.9	42 ± 8	B
12-08	36	93	air	3230 ± 110	14.9 ± 0.9	46 ± 7	B
3-28	47	93	air	2870 ± 30	17.5 ± 0.5	42 ± 3	B
4-18	93	93	air	3130 ± 40	18.7 ± 1.7	35 ± 3	B
4-12	8.6	17	air	2910 ± 50	20.5 ± 0.5	66 ± 3	B
8-30	5.1	14	air	3140 ± 30	16.9 ± 0.9	170 ± 40	N
9-23	6.3	14	air	3260 ± 160	15.9 ± 1.8	90 ± 10	N
9-27	7.0	14	air	3320 ± 30	16.9 ± 0.1	68 ± 22	N
10-27	9.4	14	air	3190 ± 130	15.4 ± 0.1	52 ± 5	B
11-01	11	14	air	3190 ± 10	15.4 ± 0.1	57 ± 9	B
6-02	25	14	air	3140 ± 120	13.8 ± 0.8	31 ± 3	B
5-25	9.5	5.7	air	3040 ± 30	15.4 ± 0.1	43 ± 4	B
11-09	0.5	4.2	air	3140 ± 20	16.4 ± 0.4	105 ± 5	N
11-09	1.0	4.2	air	3020 ± 60	16.7 ± 0.4	200 ± 90	N
11-16	1.5	4.2	air	3230 ± 10	16.9 ± 0.1	175 ± 45	N
11-23	2.0	4.2	air	3160 ± 50	16.7 ± 0.4	135 ± 45	N
11-30	2.5	4.2	air	3280 ± 60	15.9 ± 0.9	105 ± 20	N
12-09	3.9	4.2	air	3130 ± 70	15.1 ± 2.0	55 ± 6	B
11-16	0.5	2.5	air	3100 ± 20	17.7 ± 1.4	180 ± 35	N
11-16	0.6	2.5	air	3170 ± 90	16.4 ± 0.9	170 ± 120	N
11-23	1.0	2.5	air	3210 ± 100	16.4 ± 0.4	125 ± 40	N
12-01	1.6	2.5	air	3170 ± 50	16.9 ± 0.8	200 ± 120	N
6-09	2.7	2.5	air	3570 ± 50	16.4 ± 0.9	95 ± 21	N
11-01	3.6	2.5	air	3160 ± 100	15.7 ± 0.5	60 ± 11	B
5-02	9.7	58	Barnwell soil	2950 ± 50	20.5 ± 0.9	83 ± 9	N
6-09	50	58	Barnwell soil	3750 ± 70	12.5 ± 0.9	37 ± 7	B
5-18	8.5	11	Barnwell soil	2910 ± 20	19.2 ± 0.1	62 ± 10	B
6-09	20	11	Barnwell soil	3510 ± 80	14.2 ± 0.7	39 ± 2	B
8-30	8.0	3.7	Barnwell soil	3110 ± 40	13.8 ± 0.1	35 ± 7	B
8-16	2.0	1.4	Barnwell soil	3110 ± 60	10.5 ± 0.5	70 ± 3	N
7-15	50	58	Hanford soil	3470 ± 40	13.5 ± 0.5	34 ± 6	B
6-28	8.5	11	Hanford soil	3190 ± 30	15.0 ± 0.1	56 ± 16	B
8-18	20	11	Hanford soil	3190 ± 40	14.9 ± 0.9	36 ± 6	B
10-06	3.0	4.0	Hanford soil	3260 ± 50	15.9 ± 0.9	86 ± 21	N
7-25	13	79	IX resin	3140 ± 150	16.2 ± 0.8	110 ± 20	N
8-18	49	79	IX resin	3500 ± 80	15.4 ± 0.1	43 ± 5	B
10-25	100	79	IX resin	3520 ± 20	13.1 ± 0.1	37 ± 9	B
9-14	20	11	IX resin	3110 ± 60	14.9 ± 0.9	42 ± 4	B
8-16	10	8.7	IX resin	3040 ± 60	14.6 ± 0.8	37 ± 7	B
7-25	3.0	3.7	IX resin	3190 ± 30	16.2 ± 0.8	60 ± 12	B

[a]Irradiations were performed in the BNL Co-60 gamma facility at 10°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using three Type IV specimens per test. Test specimens were stamped (ASTM D-412, die C) from the sidewall of a 1200-gal rotationally molded container purchased from Poly-Processing Co., Monroe, LA.

[b]Five unirradiated specimens were tested.

Legend for Data Figures

□ - air

○ - Barnwell soil

Δ - Hanford soil

X - IX resin

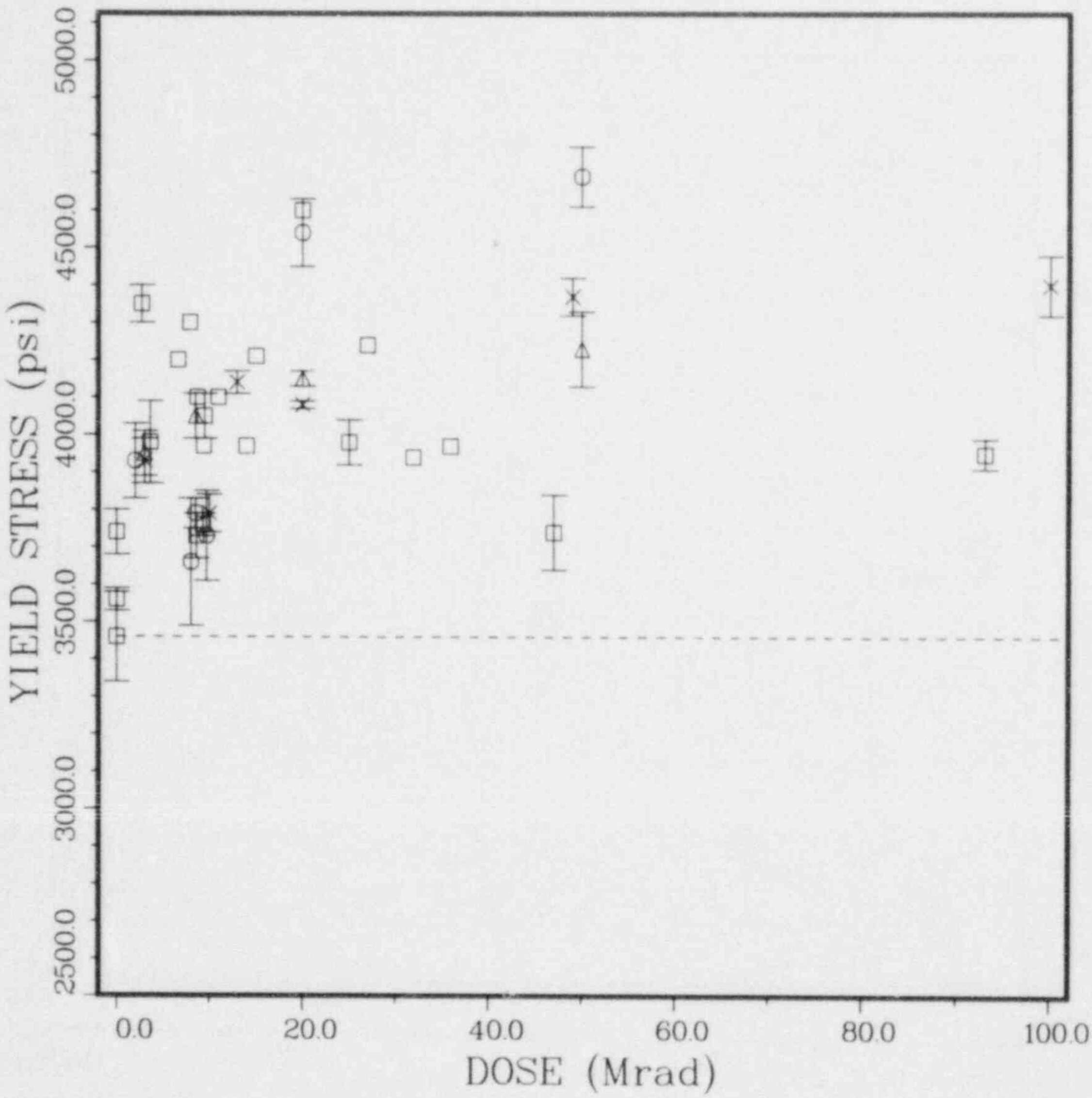

Figure 3.9. Yield stress (psi) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

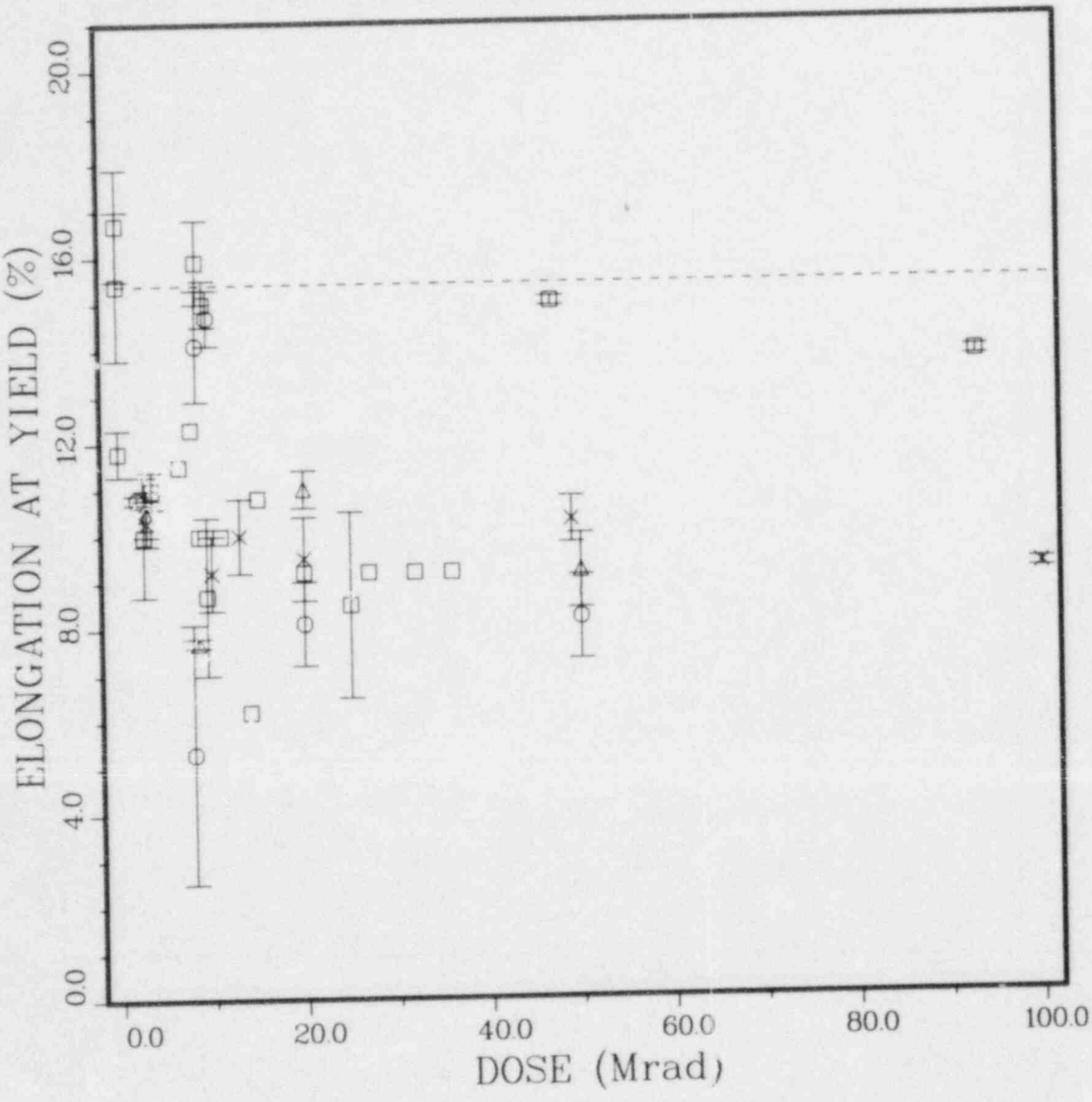

Figure 3.10. Elongation at yield (%) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

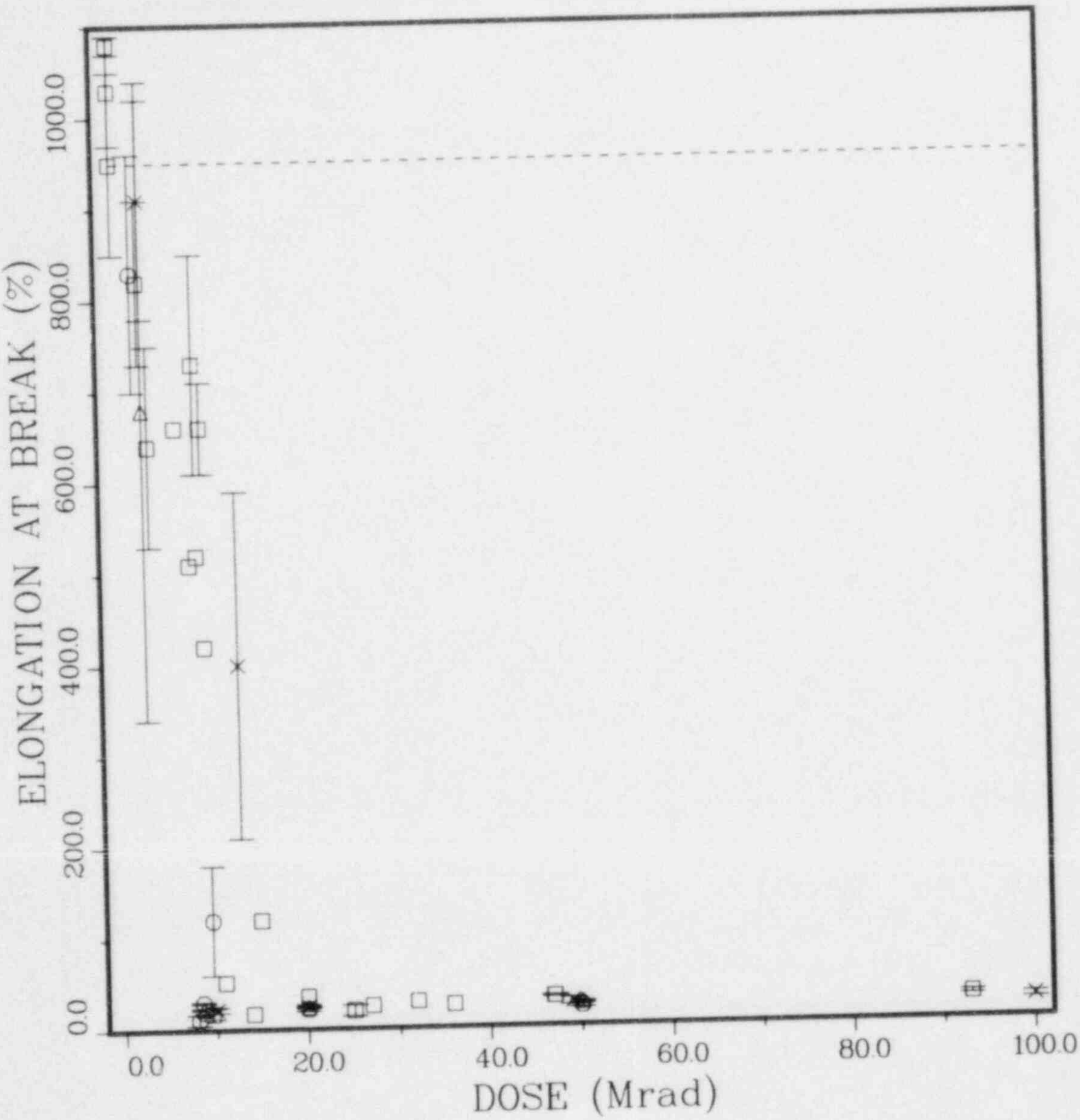

Figure 3.11. Elongation at break (%) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 3.1. Symbols are defined in the Legend.

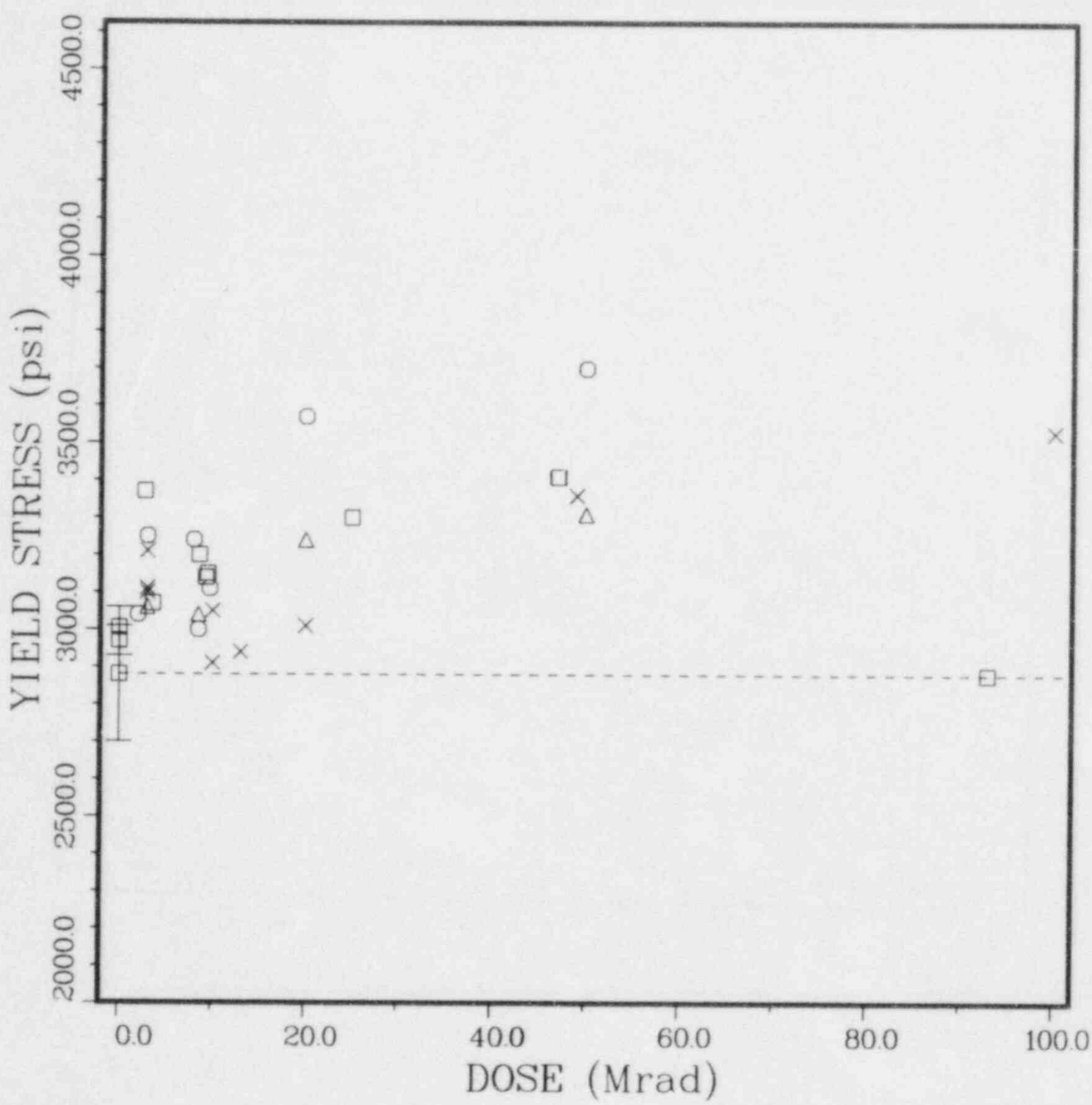

Figure 3.12. Yield stress (psi) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data are from Table 3.2. Symbols are defined in the Legend.

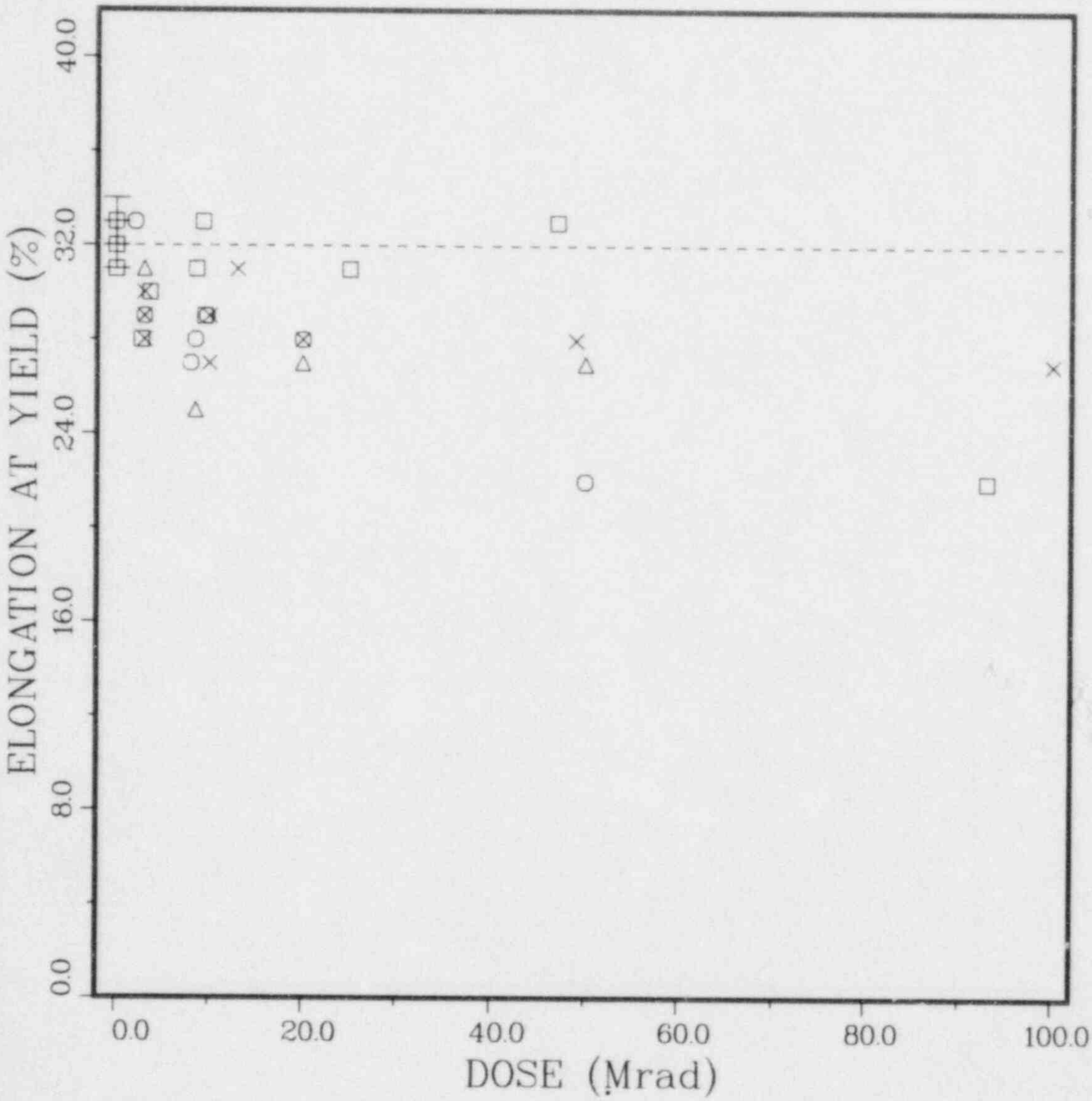

Figure 3.13. Elongation at yield (%) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data is from Table 3.2. Symbols are defined in the Legend.

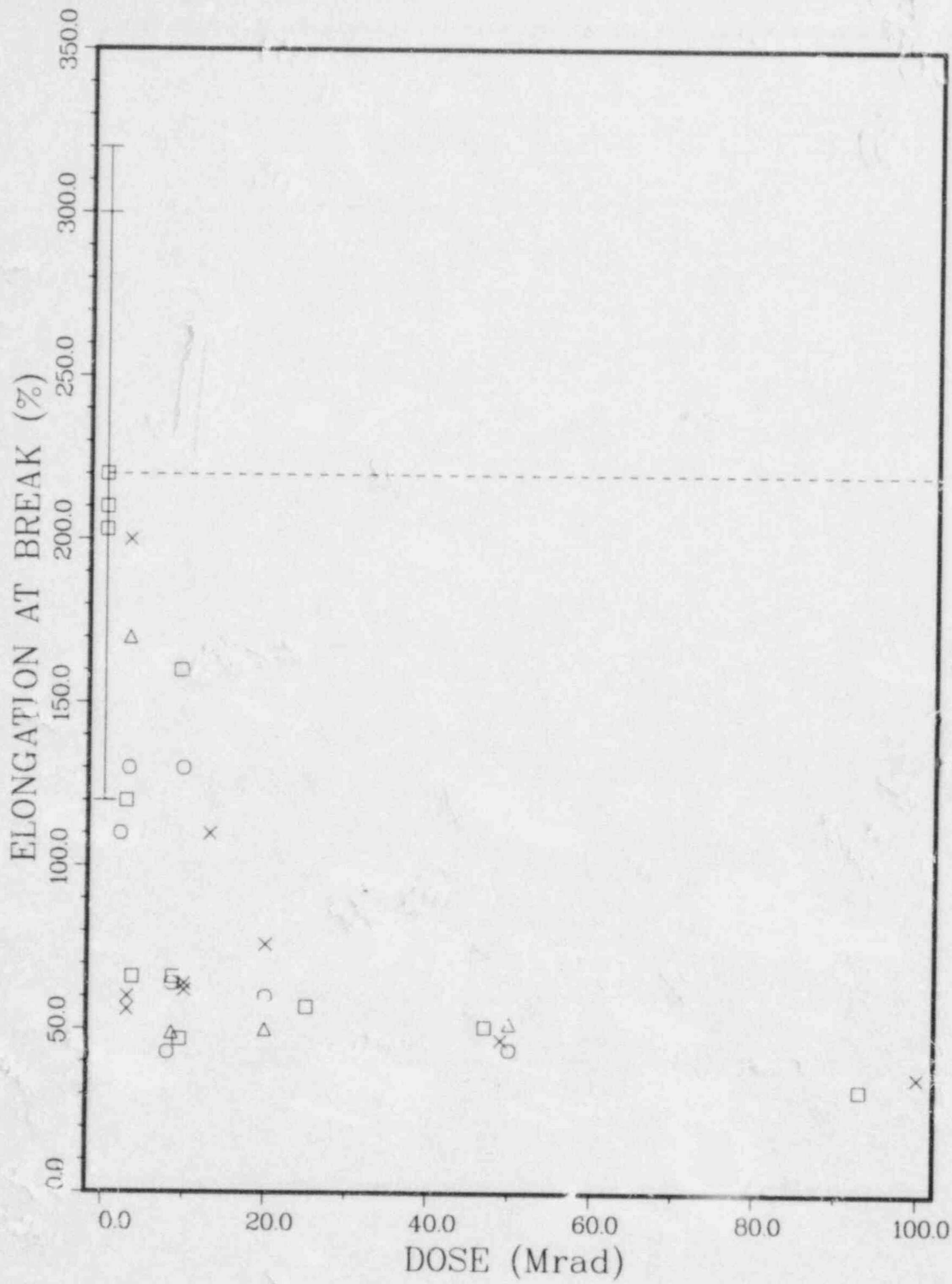

Figure 3.14. Elongation at break (%) vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data is from Table 3.2. Symbols are defined in the Legend.

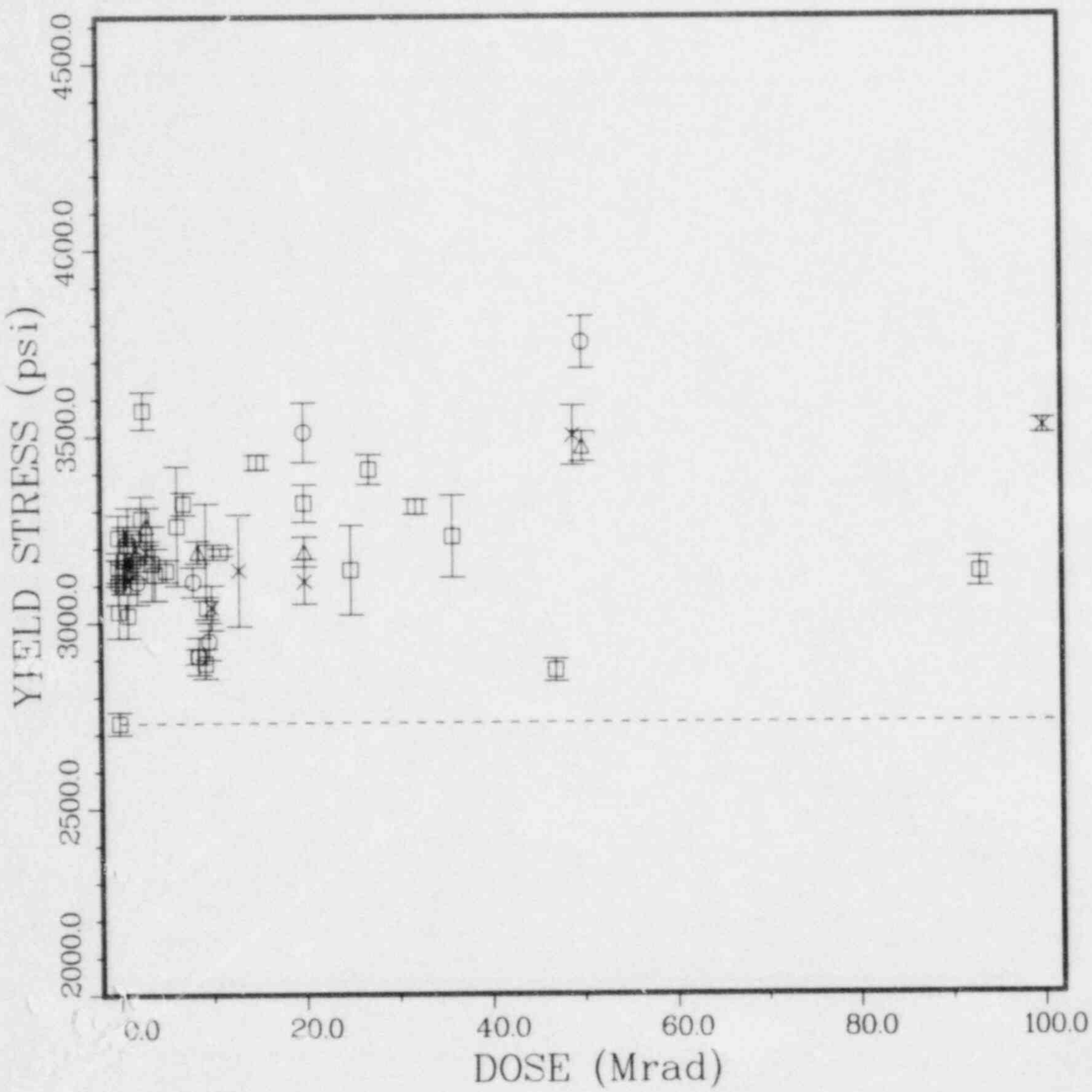

Figure 3.15. Yield stress (psi) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

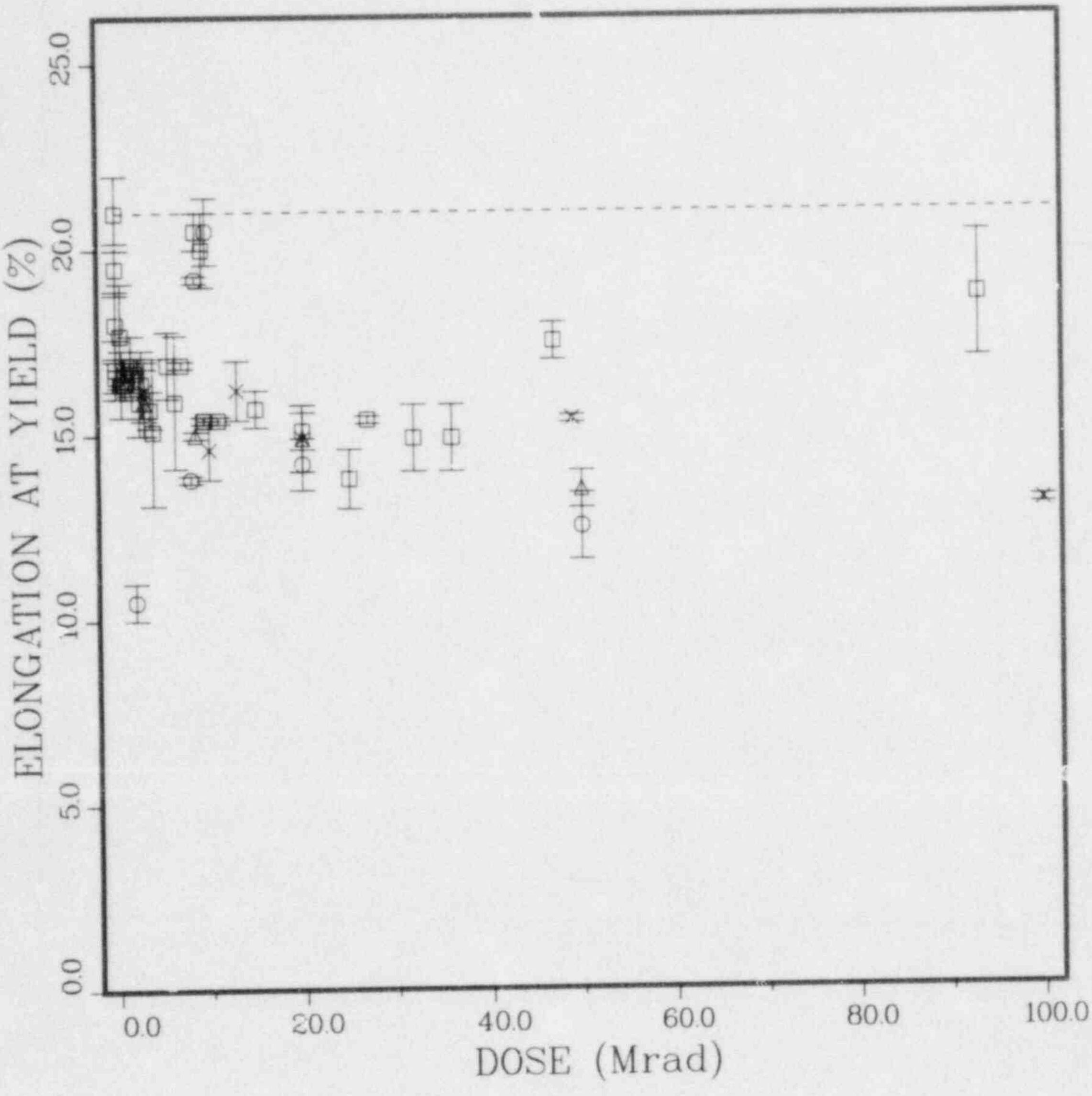

Figure 3.16. Elongation at yield (%) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

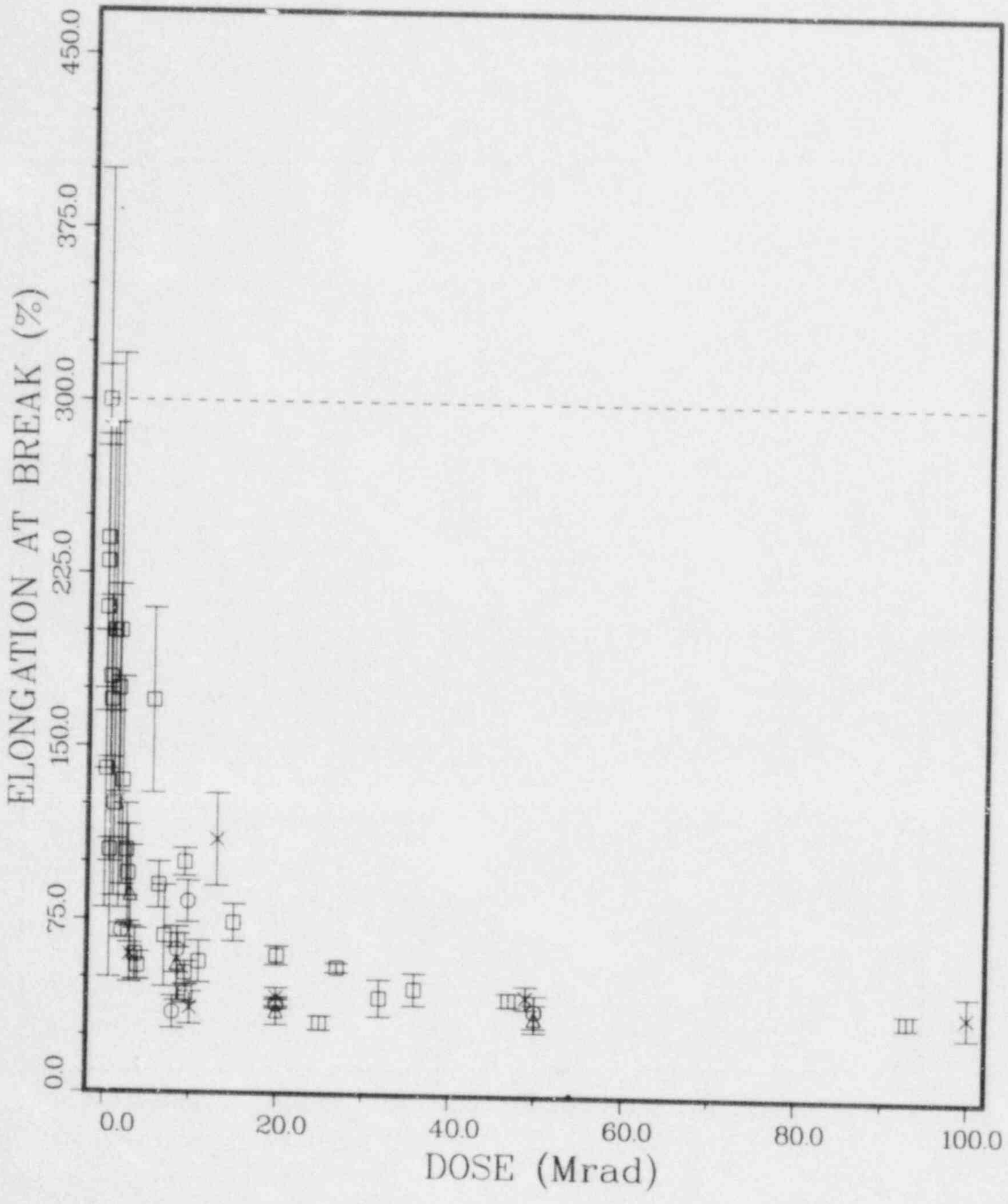

Figure 3.17. Elongation at break (%) vs dose (Mrad) of irradiated Marlex CL-100 non-HIC material. The data are from Table 3.3. Symbols are defined in the Legend.

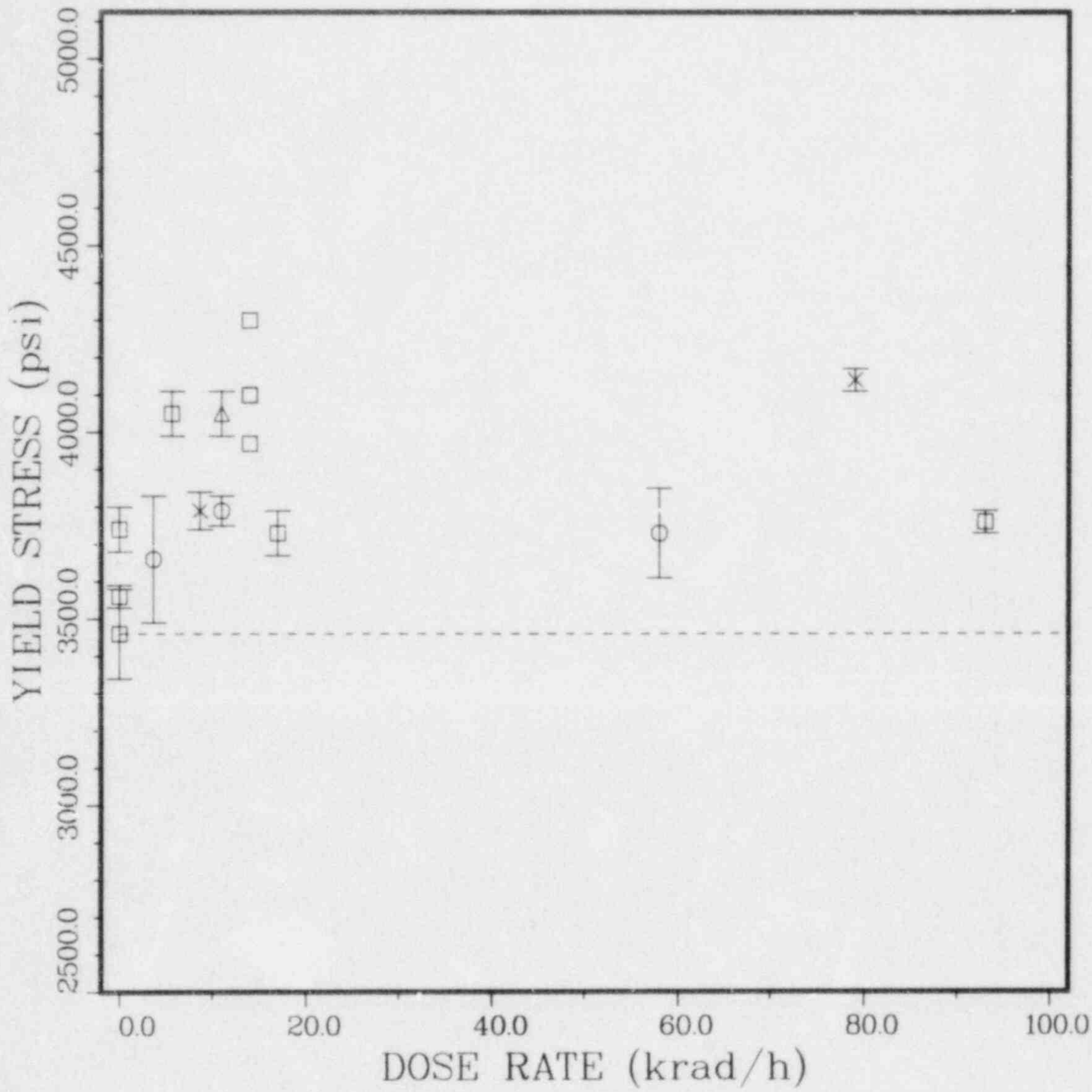

Figure 3.18. Yield stress (psi) vs dose rate (krad/h) of irradiated Chemplex 5701 for total doses from 7.9-13 Mrad. The data are from Table 3.1. Symbols are defined in the legend.

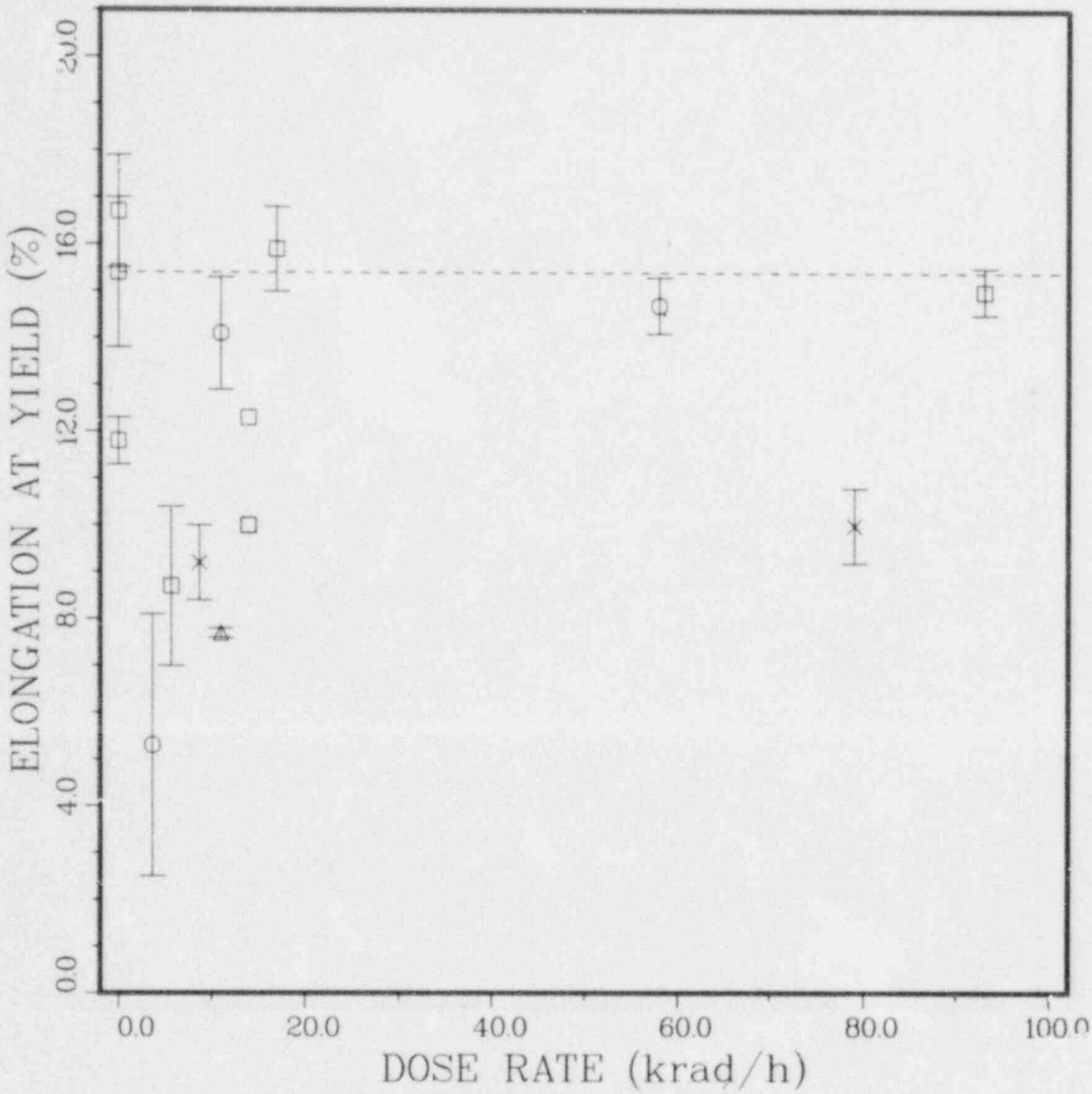

Figure 3.19. Elongation at yield (%) vs dose rate (krad/h) of irradiated Chemplex 5701 for total doses from 7.9-13 Mrad. The data are from Table 3.1. Symbols are defined in the legend.

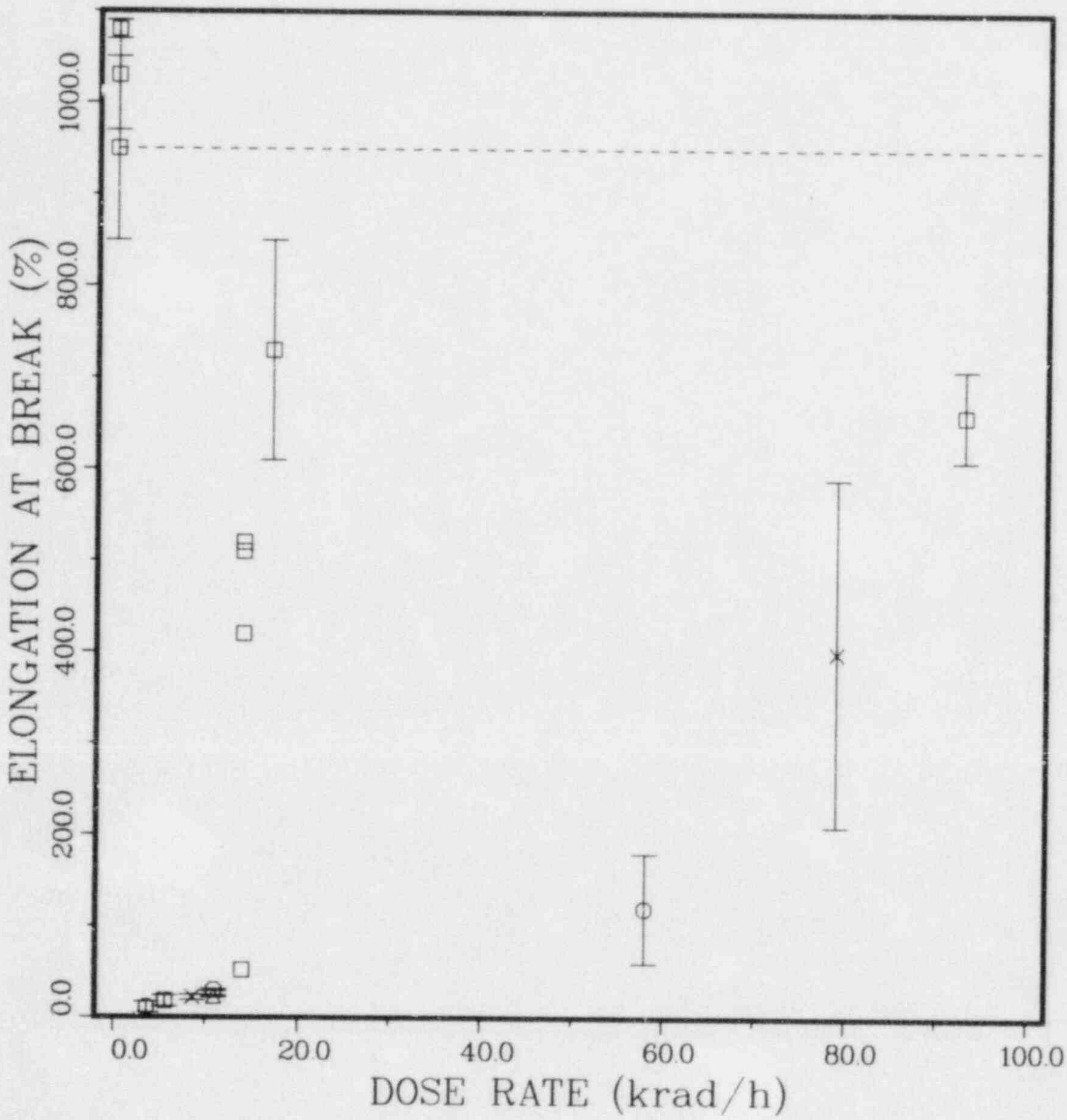

Figure 3.20. Elongation at break (%) vs dose rate (krad/h) of irradiated Chemplex 5701 for total doses from 7.9-13 Mrad. The data are from Table 3.1. Symbols are defined in the legend.

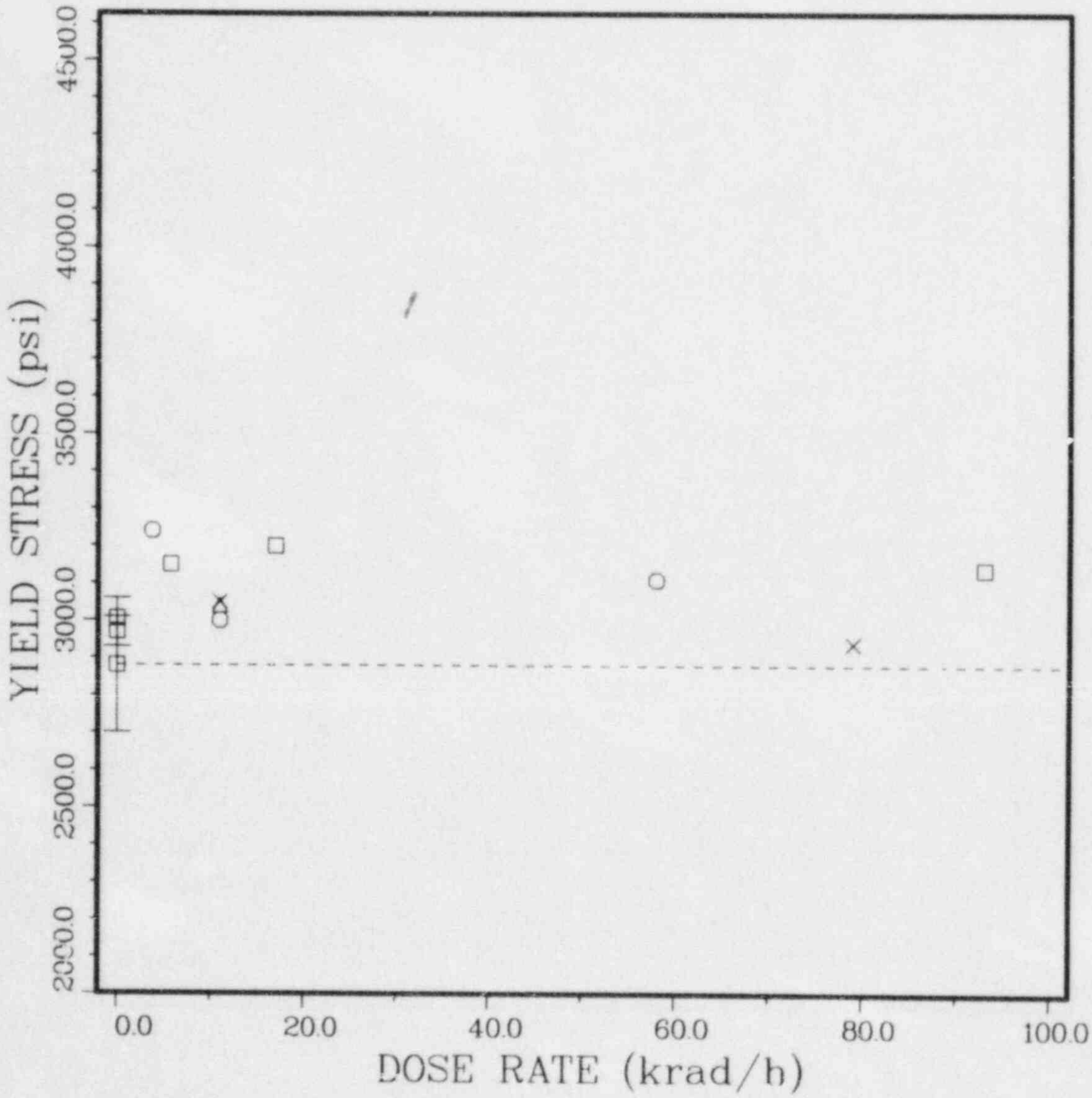

Figure 3.21. Yield stress (psi) vs dose rate (krad/h) of irradiated Marlex CL-100 HIC material for total doses from 8.0-13 Mrad. The data are from Table 3.2. Symbols are defined in the legend.

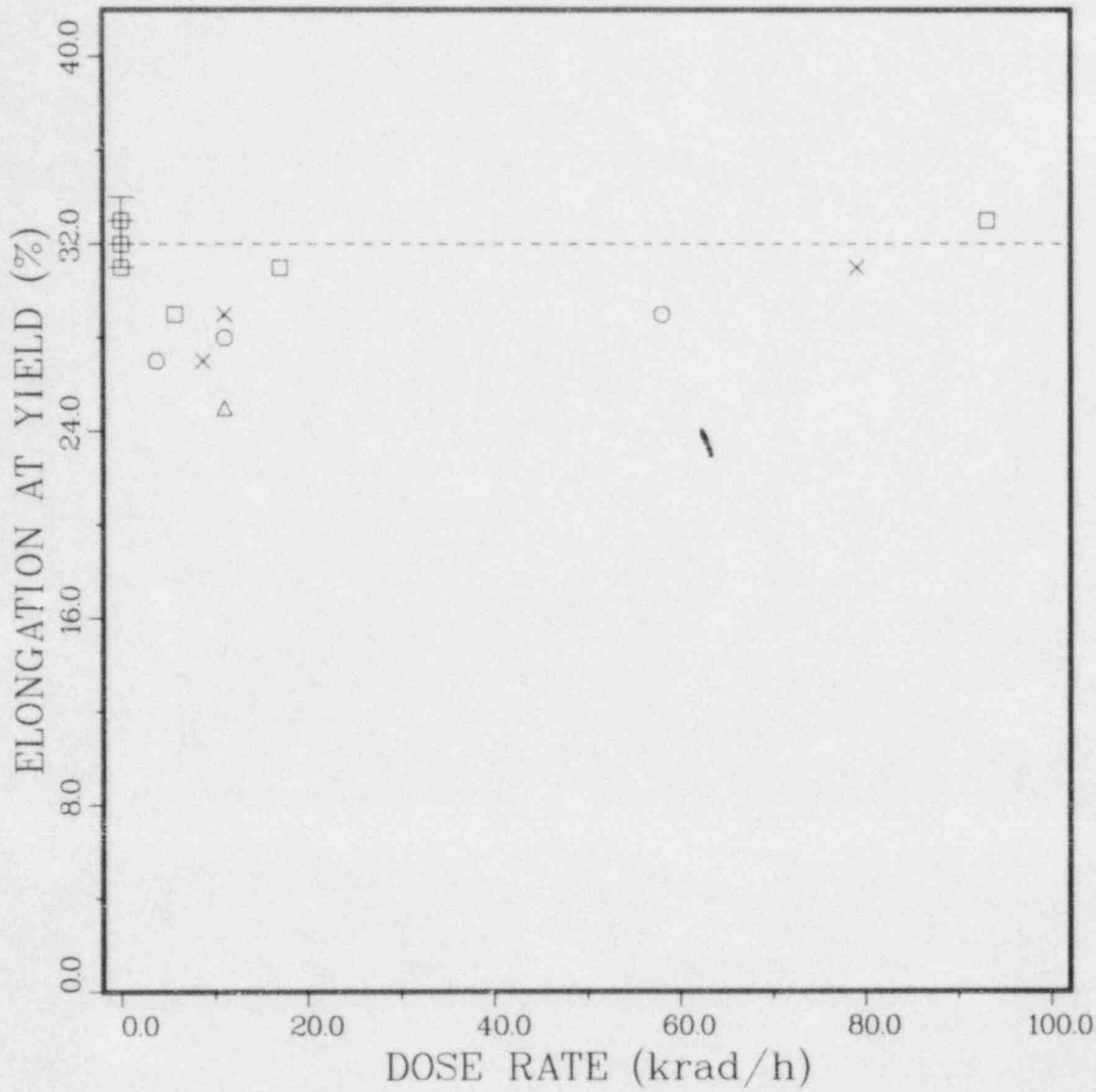

Figure 3.22. Elongation at yield (%) vs dose rate (krad/h) of irradiated Marlex CL-100 HIC material for total doses from 8.0-13 Mrad. The data are from Table 3.2. Symbols are defined in the Legend.

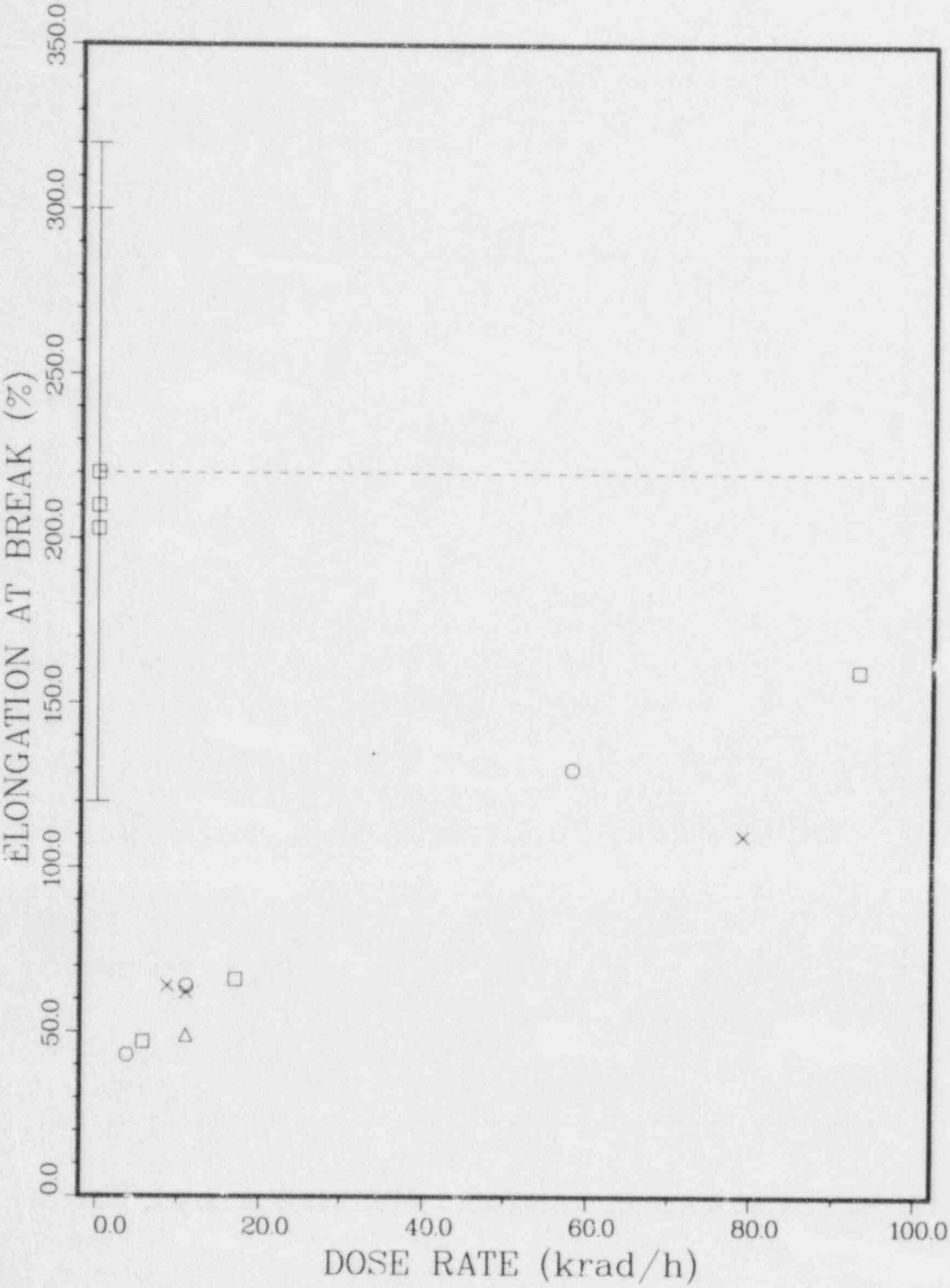

Figure 3.23. Elongation at break (%) vs dose rate (krad/h) of irradiated Marlex CL-100 HIC material for total doses from 8.0-13 Mrad. The data are from Table 3.2. Symbols are defined in the Legend.

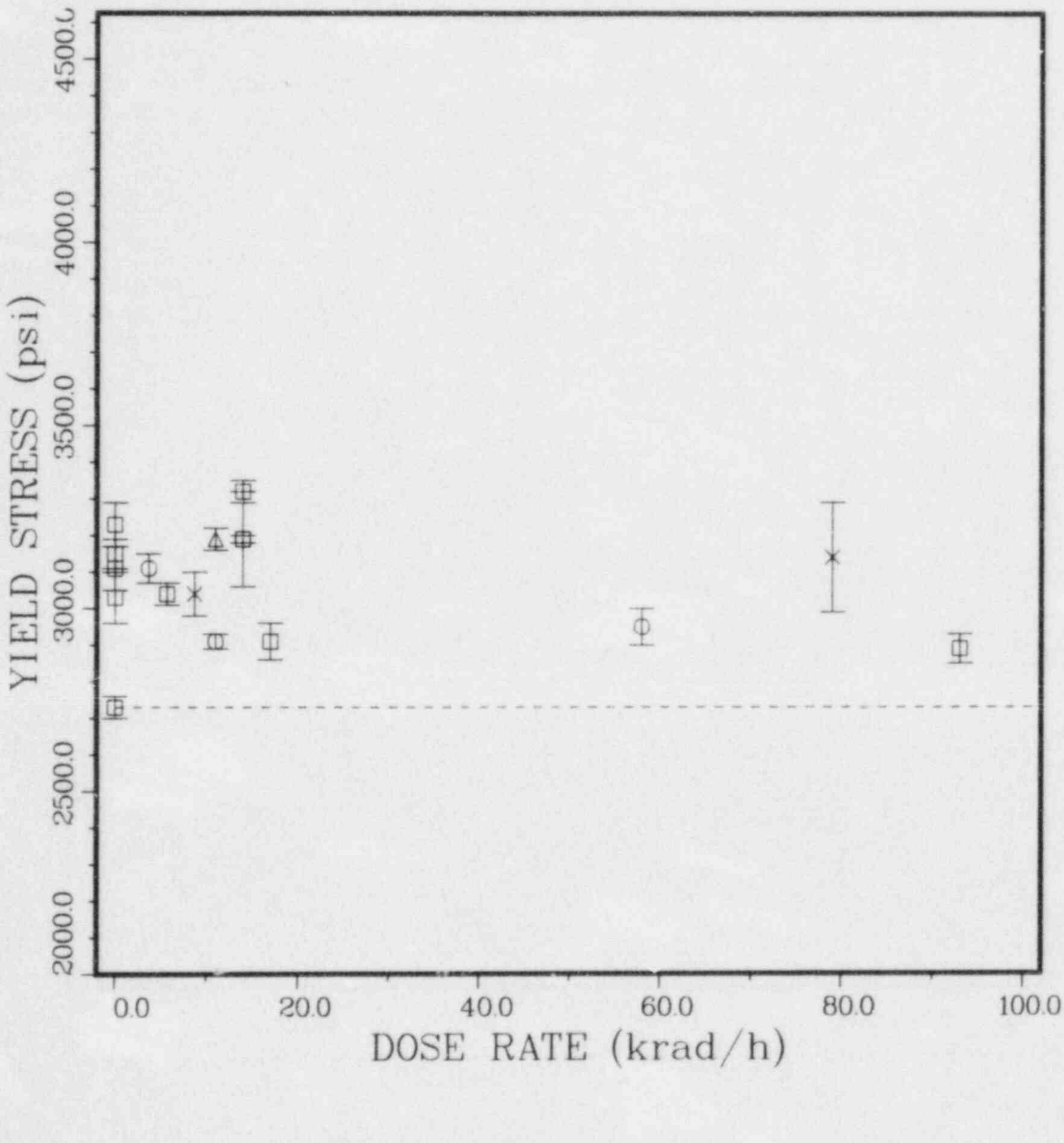

Figure 3.24. Yield stress (psi) vs dose rate (krad/h) of irradiated Marlex CL-100 non-HIC material for total doses from 7.0-13 Mrad. The data are from Table 3.3. Symbols are defined in the legend.

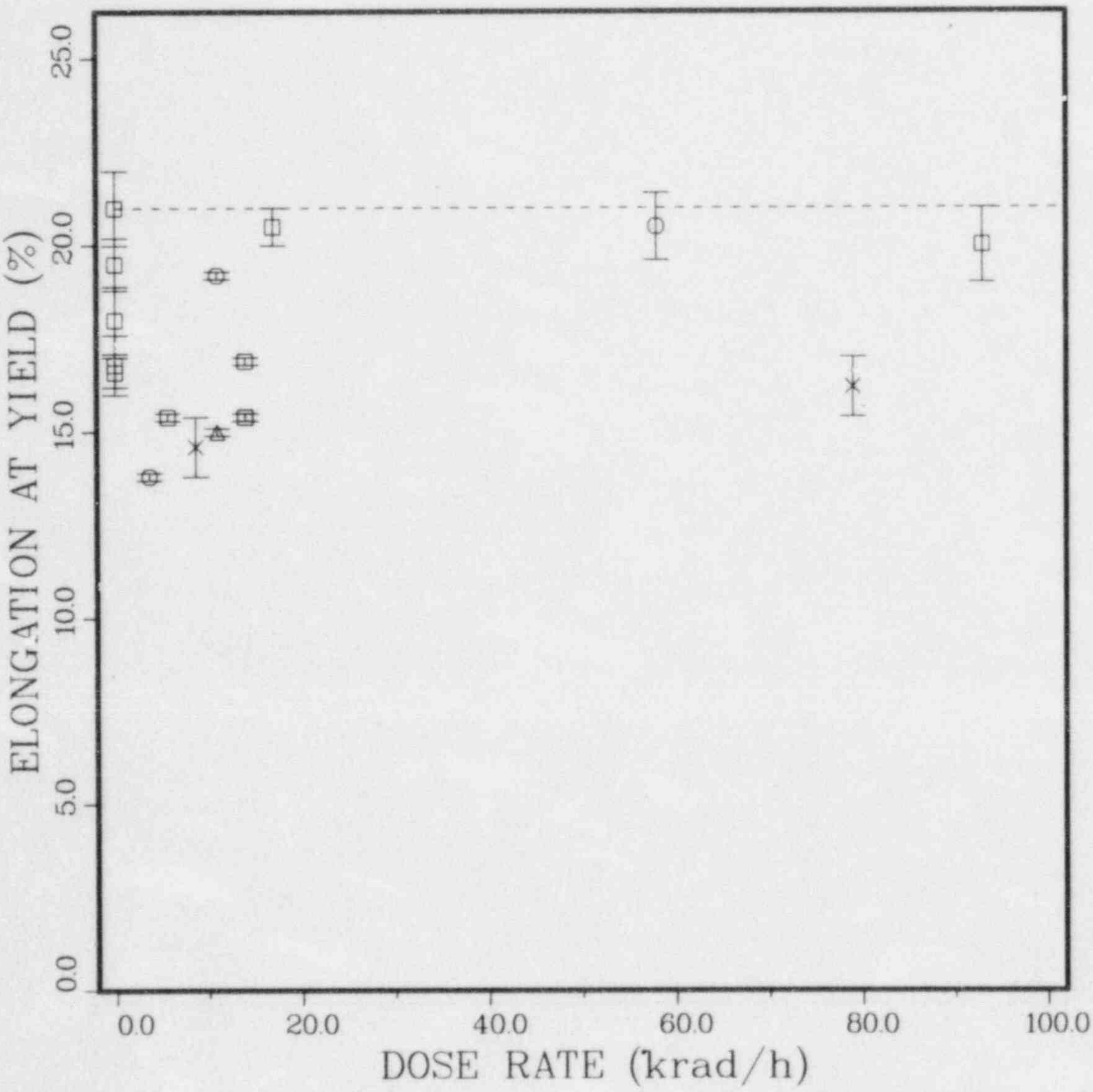

Figure 3.25. Elongation at yield (%) vs dose rate (krad/h) of irradiated Marlex CL-100 non-HIC material for total doses from 7.0-13 Mrad. The data are from Table 3.3. Symbols are defined in the legend.

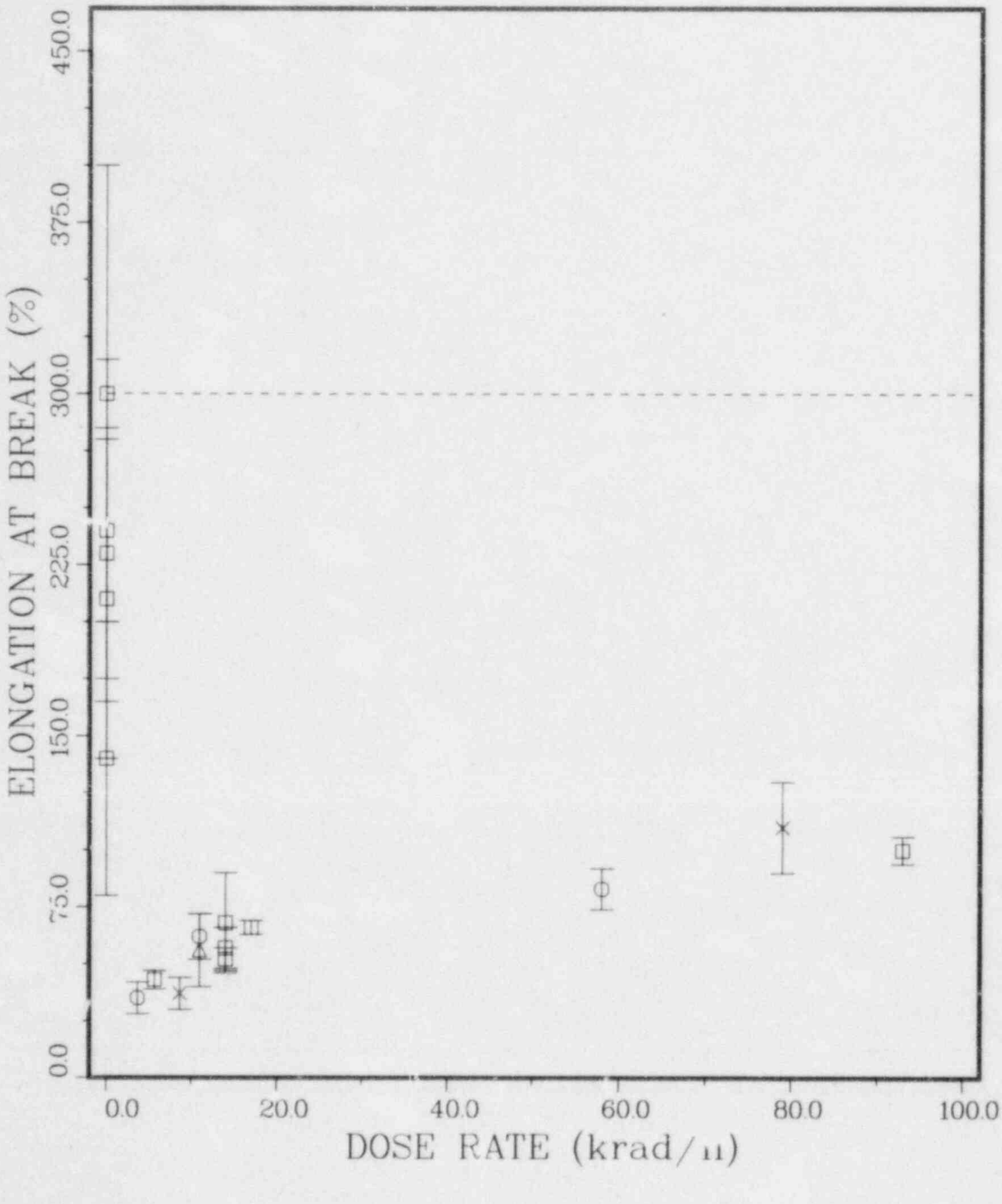

Figure 3.26. Elongation at break (%) vs dose rate (krad/h) of irradiated Marlex CL-100 non-HIC material for total doses from 7.0-13 Mrad. The data are from Table 3.3. Symbols are defined in the legend.

The tensile data plotted in Figures 3.9-3.17 is presented in the same order as the data is listed in Tables 3.1-3.3. Figure 3.9 plots yield stress vs dose, Figure 3.10 plots elongation at yield vs dose and Figure 3.11 plots elongation at break vs dose for Chemplex. Similarly, Figures 3.12-3.14 plot yield stress, elongation at yield and elongation at break, respectively, vs dose for Marlex HIC material and Figures 3.15-3.17 plot yield stress, elongation at yield and elongation at break, respectively, vs dose for Marlex non-HIC material. The dashed line corresponds to the value of the plotted parameter measured at the start of this task. The nominal initial value for many of the tensile parameters changed as the material aged during the course of the project, as discussed below.

A quick scan of Figures 3.9-3.17 suggests that there is a lot of scatter in the data and that there is no evident segregation of data points by environment (i.e., air, soils or IX resin) in any of these plots. However, in Figures 3.11, 3.14 and 3.17 for doses greater than approximately 20 Mrad, the scatter in the data is much reduced and the elongation at break appears to have plateaued or to be only slowly decreasing from 20-100 Mrad. In contrast, the decrease in elongation at break is quite pronounced from that of the unirradiated material to approximately 20 Mrad. We hasten to note at this point that there is nothing fundamentally significant about a dose of 20 Mrad, as will become clear later.

Analysis of the information contained in Figures 3.9-3.17 must take into account at least three complicating factors, as listed and discussed below.

(1) There is a great deal of scatter in the data. This is indicated both by the scatter of the individual data points themselves and by the magnitude of the error bars attached to the data points for which multiple specimens were tested. Some of this may be due to inhomogeneities in the HDPE materials themselves. The bumps in the stress vs elongation vs dose curve for unirradiated Chemplex, Figure 3.2, suggest that there are inhomogeneities in the Chemplex. The Marlex HIC material contained voids, which are presumably air bubbles trapped during the molding process. Bubbles were observed several times when they surfaced and popped as the specimen necked during tensile testing. Although no bubbles were observed in the Marlex non-HIC material, they probably were there but perhaps too small to be seen. It is difficult to imagine that some air would not be trapped between the melting Marlex granules as the container was formed. For all three materials the cross-sectional area varied along the length of the tensile test specimen. This was the result of varying wall thickness in all of the container materials. For the practical purpose of not having to discard most of the test specimens, Type IV tensile specimens were accepted with variations in thickness along the gage length of up to 0.010 inch. This variation in thickness for individual test specimens as well as possible errors in measurement—i.e., if the narrowest part of the specimen was not where the measurement was taken—probably contributed to scatter in the data. Finally, only the non-HIC Marlex came from a single container. The Chemplex was taken from two containers and the Marlex HIC material tensile specimens came from cut-outs from seven HICs.

(2) The tensile properties of these materials appear to change with age. The differences in measured parameters listed in Tables 3.1 to 3.3 for the unirradiated material do indicate changes in their mean values with time, however, in most cases, the changes are within the sample-to-sample variation observed in the testing. This is particularly true of the Marlex HIC material. Variation in the mean values was most evident for the Marlex non-HIC material which was the youngest of the materials tested. However, in this case, property changes appear to be minimal by the end of the experiments. For Chemplex and the Marlex non-HIC materials, an increase in yield strength and decrease in yield elongation were observed. While the effects of aging cannot be quantitatively separated from the effects due to irradiation in this study, it is felt that they contribute little to the qualitative effects observed and, in particular, have little or no effect upon the transition from necking to breaking failure.

(3) At least some of the data for irradiated material is affected by dose rate as well as dose. (Dose rate effects are discussed following this section on dose effects.) Since these experiments were conducted at several different dose rates, as shown in Tables 3.1-3.3, dose rate effects, where they occur, will have affected the dose data. Dose rate differences between irradiations in air and those in the soils and the IX resin were unavoidable because of the attenuation of the gamma ray flux by the soils and resins. The soils reduced the dose rate to 60-65% of that in air while the IX resin caused a reduction to 78-85% of the dose rate in air. Thus, for example, the 93 krad/h irradiation in air, the 58 krad/h irradiation in the soils and the 79 krad/h irradiations in IX resin were all conducted in the same air tube.

In the following paragraphs each of the Figures 3.9-3.17 is discussed. Conclusions arrived at for dose rate effects from Figures 3.18-3.26 are referenced here even though these results are discussed following this section on dose effects. (This is necessary because dose effects and dose rate effects could not be kept strictly isolated in our irradiation studies. Since these effects have been qualitatively shown to occur in polyethylene, we accepted this and directed our experiments toward an attempt to quantify this effect to provide a model of the consequences of irradiation on polyethylene. This model is discussed in the CONCLUSIONS section of this report). Following the discussion of dose effects and dose rate effects in the individual figures the changes observed in tensile properties following irradiation for these materials are summarized.

Figure 3.9 - Chemplex yield stress vs dose: All the data for irradiated material lie above the dashed line which marks the initial (nominal) value for the unirradiated material. Additionally, all but a few of the irradiated data points lie above the unirradiated data taken toward the end of this task. These observations indicate that irradiation increases the yield strength of the Chemplex.

It may be significant that, for doses greater than approximately 40 Mrad, all of the data for irradiation in soils and IX resin lie above those in air. Part of this probably results from aging since the irradiations in the soils and IX resin were conducted after those in air, as shown in Table 3.1.

However, dose rate effects (c.f. dose rates for the soil and IX resin irradiations were lower than for the air irradiations) seem unlikely to have contributed to this since no dose rate effect is apparent from Figure 3.18. It appears that irradiation in the soils and IX resin lead to a larger increase in yield strength than occurred in air. A possible explanation for this may be that the soil and resin environment reduced or excluded oxygen access to the test specimens during irradiation. Radiation induced cross-linking is known to increase the strength of polyethylene and radiation-induced oxidation decreases it.[11] This is also shown later in this report. The larger increase in strength for irradiation in media which may exclude oxygen is consistent with an increase in strength due to cross-linking which is not competing with degradation due to oxidation. For irradiation in air both processes are at work and tend to offset one another.

Figure 3.10 - Chemplex elongation at yield vs dose: The majority of data suggests that there is a decrease in yield elongation following irradiation. Data for unirradiated material, Table 3.1, at the beginning of this task and toward the end indicate that some of this decrease is due to aging. The data from Figure 3.19 suggest that there may be a dose rate effect at rates below about 10 krad/h which would tend to enhance the decrease in yield elongation at the lower dose rates.

Figure 3.11 - Chemplex elongation at break vs dose: The data in Figure 3.11 shows that there is a significant decrease in break elongation with irradiation. Part of the reason for this is that the unirradiated material elongates by a factor of about 10 due to necking. However, once necking in this material is lost the break elongation is reduced dramatically. Aging is not a factor since no decrease in break elongation of the unirradiated material with age was observed. However, there is a large dose rate effect, as shown in Figure 3.20. As noted earlier in the text, break elongation decreased to about 11-30% and then did not noticeably decrease further up to 100 Mrad.

Figure 3.12 - Marlex HIC material yield stress vs dose: Most of the data for irradiated material lies above the nominal yield strength at the beginning of the task, as indicated by the dashed line, and also above the values for the unirradiated material measured toward the end of the task, listed in Table 3.2. Thus, irradiation appears to increase the yield strength of the Marlex HIC material. Aging appears to affect this result only slightly, if at all, since the yield strength measured at the end of the task remains within one standard deviation of the initial value indicated by the dashed line. Also, no dose rate effect is apparent from Figure 3.21.

It is interesting in Figure 3.12 that there is no separation of the data for irradiation in air from that in the soils and in resin in the 47-50 Mrad dose range, as was observed for the Chemplex (see Figure 3.9) and Marlex non-HIC material (see Figure 3.15). However, at 93-100 Mrad the data point for IX resin is well above that for air as also occurs for the two other HDPE materials. This may be due to the thickness of the HIC material, which would tend to protect the bulk material from radiation-induced oxidation in a similar way that the presence of the soils and IX resin may slow down degradative effects by limiting oxygen availability to the plastic. Thus, the segregation of data in the soil and IX resin environments compared to air occurs at lower doses (and irradiation times) in thinner specimens than in thicker specimens.

Figure 3.13 - Marlex HIC material elongation at yield vs dose: The majority of data indicates that yield elongation decreases upon irradiation. Aging does not appear to be a factor, as shown by the unirradiated data in Table 3.2 from project beginning to end. However, there appears to be a dose rate effect, as shown in Figure 3.22, which leads to a decrease in yield elongation at a given dose as the dose rate decreases.

Figure 3.14 - Marlex HIC material elongation at break vs dose: This figure has two striking features. The first is the significant decrease in break elongation with dose. The second is the large error associated with the break elongation of the unirradiated material. The decrease in break elongation also shows a large dose rate effect, as shown in Figure 3.23, in which the break elongation decreases with dose rate for a given dose. Aging does not appear to be a factor and, in any case, would be difficult to evaluate as a consideration given the magnitude of the error bars. The loss of necking behavior causes the break elongation to plateau out at approximately 40-50%. As discussed earlier in this section, the large variation in break elongation of the unirradiated material may result from the bubbles trapped in the HIC material during container fabrication.

Figure 3.15 - Marlex non-HIC material yield stress vs dose: All of the irradiated data lies above the dashed line denoting the yield strength at the beginning of the task, as does the unirradiated data taken at the end of the task. The aging effect in this material is relatively large, as the unirradiated data shows in Figure 3.15 and in Table 3.3. The several sets of unirradiated specimens tested at different times at the end of this task provide an estimate of the reproducibility of the data over a period of time short enough that aging is not a factor. The relatively large aging effect limits any at tempt to comment on any but the most obvious features of Figure 3.15 and Table 3.3. These include the observations that the data for doses up to 3.9 Mrad taken at the end of the task at dose rates of 4.2 and 2.5 krad/h indicate that no increase in yield strength has occurred. However, the data taken at 93 krad/h at the end of the task for doses from 15 to 36 Mrad indicate that an increase in yield strength has occurred in these irradiations. Thus, irradiation does increase the yield strength of the Marlex non-HIC material even though the effect is masked in much of the data by the aging effect in this material.

As in the Chemplex but unlik the Ma HIC material, the data for the 47-50 Mrad irradiations in the soils and IX resins lie well above that in air. Similarly, in the 93-100 Mrad irradiations the data in IX resin lies above that in air. Aging almost certainly contributes to this since the irradiation in air were conducted before those in the soil and IX resins. Dose rate effects should not affect these results since the dose rate effect is evident in Figure 3.24. The pattern of the data from 47-100 Mrad may be no more than coincidence. However, it may also be that the soils and resin protected the specimens undergoing irradiation from radiation - induced oxidation, which may have occurred in the air irradiation and which would have counteracted the cross-linking which was strengthening the material.

Figure 3.16 - Marlex non-HIC material elongation at yield vs dose: The majority of the data indicates that irradiation decreases the yield elongation. Aging has also reduced the yield elongation in the unirradiated material. However, most of the data for irradiated material also lies below that for the unirradiated material. A dose rate effect may be evident, Figure 3.25, at dose rates below approximately 10 krad/h.

Figure 3.17 - Marlex non-HIC material elongation at break vs dose: The pattern of the data in Figure 3.17 for elongation at break in similar to that for the other two HDPE materials - i.e., a large initial decrease in break elongation with dose which plateaus out and then remains constant constant up to 100 Mrad in all four irradiation environments. The large decrease occurs as necking behavior is lost and the plateau is reached when necking no longer occurs. Figure 3.26 indicates that the decrease in break elongation is dose rate dependent. Additionally, there are large errors associated with the break elongation as long as necking occurs. As was discussed for the Marlex HIC material, this large variation in break elongation as long as necking occurs may be due to bubbles trapped in the material during container fabrication.

In the following paragraphs each of the Figures 3.18-3.26 is discussed. These figures plot selected data from Tables 3.1-3.3 for the same tensile parameters and in the same order as plotted in Figures 3.9-3.17. The dose range of approximately 10+3 Mrad over which the data at different dose rates were plotted was selected somewhat arbitrarily. However, this limited dose range (ideally the data would be at a single dose value) seemed to provide an adequate picture of dose rate effects for the cases in which dose rate effects were observed.

Figure 3.18 - Chemplex yield stress vs dose rate: No dose rate effect is evident from this figure. The data do not tend to either increase or decrease as the dose rate changes.

Figure 3.19 - Chemplex elongation at yield vs dose rate: The data at dose rates below approximately 10 krad/h appear to tend toward lower values whereas no trend is apparent at higher dose rates. Thus, decreasing the dose rate below approximately 10 krad/h appears to result in a lower yield elongation compared to the same dose administered at a higher dose rate.

Figure 3.20 - Chemplex elongation at break vs dose rate: There is clearly a decrease in break elongation with decreasing dose rate in this data. The decrease occurs rather abruptly below approximately 14 krad/h for the air irradiations. Note that the decrease may occur at a higher dose rate for soil and IX resin irradiations, as suggested by the data at 58 and 79 krad/h. However, this is not certain because of lack of data for dose rates between 11 and the higher dose rates listed in the preceding sentence. The decrease in break elongation parallels the loss of necking behavior.

The data plotted in this figure at approximately 14 krad/h appears not to follow the overall trend in the data. This is because, for Chemplex, the transition from necking to breaking failure occurs in the range of total dose plotted. This is evident in Figure 3.27 which plots the data for elongation at break vs total dose at 14 krad/h. The three points plotted in this figure above 10 Mrad are samples in which failure occurred by breaking. The remaining four points (below 10 Mrad) failed via necking. Although the variation in these latter points is large, the transition from necking to breaking appears to be quite sharp. At a dose rate of 14 krad/h, it appears to occur over less than 2 Mrad in a total dose.

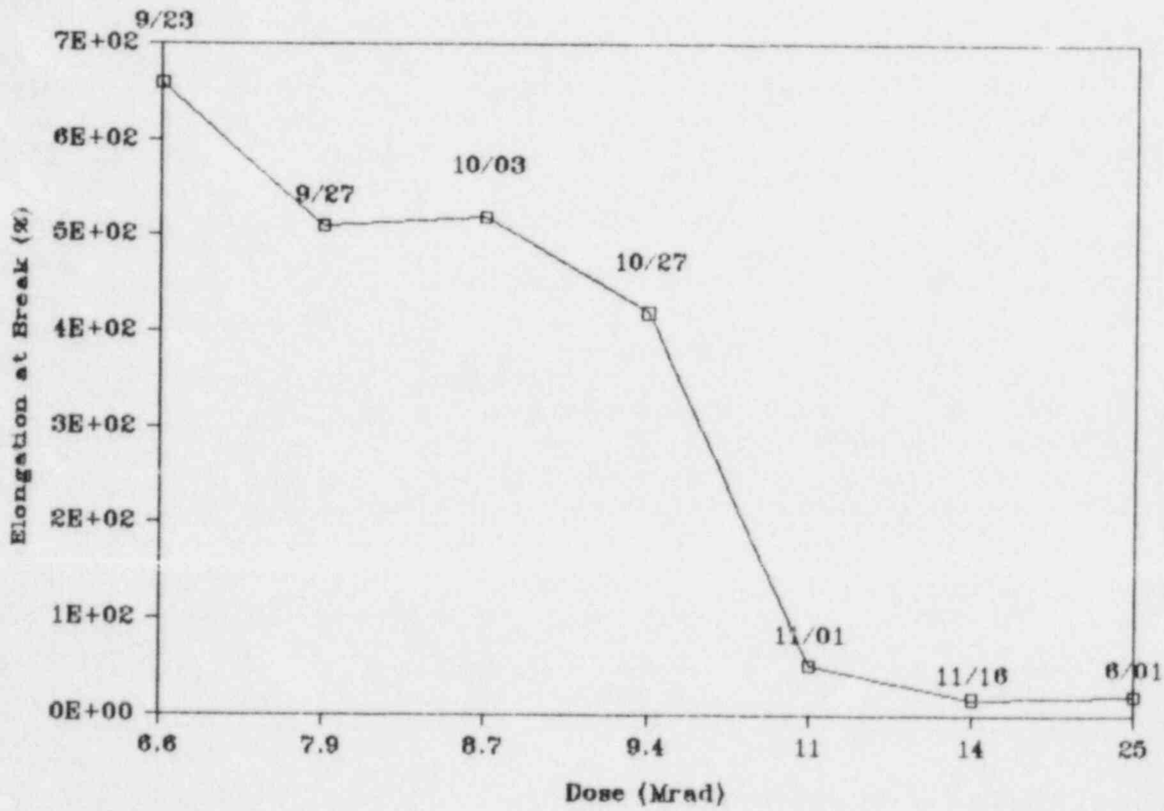

Figure 3.27 Elongation at break (%) vs dose (Mrad) of Chemplex 5701 irradiated at a dose rate of 14 krad/h. Dates above points indicate date on which sample was tested in 1983.

Figure 3.21 - Marlex HIC material yield stress vs dose rate: No date rate effect is apparent from these data.

Figure 3.22 - Marlex HIC material elongation at yield vs. dose rate: There is a trend in these data toward lower yield elongation with decreasing dose rate. It may be significant that the data for irradiation in the soils and IX resin generally lie below that in air. However, the effect is rather small even though it is consistent in the data.

Figure 3.23 - Marlex HIC material elongation at break vs. dose rate: These data clearly trend toward decreased break elongations with decreasing dose rates. The effect is uniform in all four irradiation environments.

Figure 3.24 - Marlex non-HIC material yield stress vs. dose rate: No dose rate effect is evident from these data.

Figure 3.24 - Marlex non-HIC material elongation at yield vs. dose rate: A dose rate effect may be evident from these data for dose rates less than approximately 10 krad/h. However, the variation in the irradiated data is no greater than that in the unirradiated data from aging. Based on the results for Chemplex and Marlex HIC material a dose rate effect at the lower dose rates might be expected.

Figure 3.26 - Marlex non-HIC material elongation at break vs. dose rate: A decrease in break elongation with decreasing dose rate is evident in these data. The effect in this figure is not as pronounced as it appeared in the Chemplex and Marlex HIC material figures.

As this project progressed it was clear that we had seen the same effect (i.e., a severe decrease in elongation at break for irradiated HDPE material compared to unirradiated material) that Clough and Gillen(1-4,28) had observed in the deterioration of nuclear power plant polyethylene electrical insulation. There seems little doubt that, for container purposes, the loss of deformability that the decrease in break elongation portends may not be desirable. However, the strength of the irradiated materials, as measured by the yield stresses listed in Tables 3.1-3.3, did not decrease but actually increased. It was clear from these data that the material had changed but it was not clear to what extent this change could be considered degradation. Decreases in break elongation result from cross-linking due to radiation.(11,26,27) In sum, the test specimens irradiated at 10-11°C in this task did not appear to have suffered a significant amount of degradation although they certainly had changed. There is little doubt, from the results of Clough and Gillen,(1-4,28) that radiation-induced oxidation was occurring. However, the effects of radiation-induced cross-linking seem to have counteracted most, if not all, of the effects of radiation-induced oxidation in these experiments.

To accelerate the process of radiation-induced oxidation in hopes of seeing a decrease in strength (i.e., yield stress), an irradiation was conducted at 60-63°C (140-145°F). [Note that cross-linking is also increased by increasing temperature.(26)] This temperature range was chosen for two reasons: (i) to be near, but not to exceed, the 170°F limit on polyethylene HICs set by the State of South Carolina,(25) and (ii) to be below the approximately 70°C (158°F) temperature at which the smaller crystalline regions in polyethylene began to soften.(11) Two sets of tensile specimens were used for this experiment, one set was irradiated while an unirradiated set of controls was maintained at temperature in an oven for the same length of time. The irradiation at 60-63°C lasted for 66 days at a dose rate of 5.7 krad/h, for a dose of 9.0 Mrad. The elevated temperature accelerated the degradation of the HDPE materials although we do not know how much of an acceleration occurred. However, using the rule-of-thumb that, for reactions near room temperature, the reaction rate doubles for each 10°C increase in temperature it can be estimated that the degradation reaction was approximately 32 times faster at 60-63°C than at 10-11°C, everything else being equal.

Tensile testing results for these specimens plus the unirradiated controls are listed in Table 3.4. The yield strength of the irradiated, heated specimens has clearly decreased. An effect related to specimen thickness is suggested by the fact that the yield strength of the Marlex non-HIC material has decreased relatively more than that of the thicker Marlex HIC material. The yield strength and other data for the unirradiated, heated specimens have not changed noticeably from the data for the unirradiated material listed in Tables 3.1-3.3. The loss of strength in the irradiated and heated specimens appeared to result from a change in the surface layer on all sides of each of these specimens. Since no such change was observed for the unirradiated, heated specimens or for the specimens irradiated at 10-11°C, it appears that this change was caused by radiation-induced oxidation. Additionally, all four sides of the Marlex CL-100 HIC and non-HIC materials cracked - not just the glossy surface as occurred in the specimens irradiated at 10-11°C. The change appeared to be an embrittled surface layer approximately 0.04-0.05 inch deep around the circumference of the break. The broken surfaces of this embrittled layer were shiny and strongly contrasted with the dull appearance of the broken surfaces of the bulk material inside the surface layer. This is shown in Figure 3.28, which is a picture of the broken cross-section of one of the HIC material tensile specimens irradiated at 60-63°C. Similar effects were observed for the Marlex non-HIC material and the Chemplex.

The results from tensile testing these materials can be summarized in the following statements:

- The tensile properties of these materials appear to change with age, however, in most cases, the changes are within the sample-to-sample variation observed in the testing. This is particularly true of the Marlex HIC material. While the effects of aging cannot be quantitatively separated from the effects due to irradiation in this study, it is felt that they contribute little to the qualitative effects observed and, in particular, have little or no effect upon the transition from necking to breaking failure.

- The relative tensile properties of Chemplex 5701 and the two types of Marlex CL-100 investigated in this task changed in generally similar ways following irradiation under the conditions of this study.

- The yield strength generally increased following irradiation.

- The majority of points indicate no dose rate effect on the yield strength.

- The elongation at yield generally decreased following irradiation. However, the magnitude of the decrease was not large.

- The majority of points support no dose rate effect on the elongation at yield for dose rates above approximately 10 krad/h. However, data at lower dose rates and low total doses suggest the possibility of a dose rate effect at dose rates below 10 krad/h.

- The elongation at break decreased significantly with irradiation. This was consistent with the transition from necking to breaking without necking.

- The elongation at break decreased with decreasing dose rate in the four irradiation environments, i.e. air, Barnwell and Hanford soils and IX resin, used in this task.

Table 3.4

Tensile Test Data on HDPE Irradiated at 60-63°C in Air[a]

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)
Chemplex 5701					
12-01	0	----	3840 ± 110	13.5 ± 0.5	950 ± 40
12-01	9.0	5.7	2750 ± 780	3.1 ± 1.6	5 ± 2
Marlex CL-100 HIC Material					
12-01	0	----	2900 ± 125	32.9 ± 2.4	260 ± 80
12-01	9.0	5.7	2750 ± 10	31.7 ± 2.9	54 ± 4
Marlex CL-100 Non-HIC Material					
12-01	0	----	3220 ± 30	18.5 ± 1.1	120 ± 20
12-01	9.0	5.7	2100 ± 40	9.5 ± 1.6	14 ± 2

[a]Irradiations were conducted in the BNL gamma pool at 5.7 krad/h. Each line represents results from three test specimens.

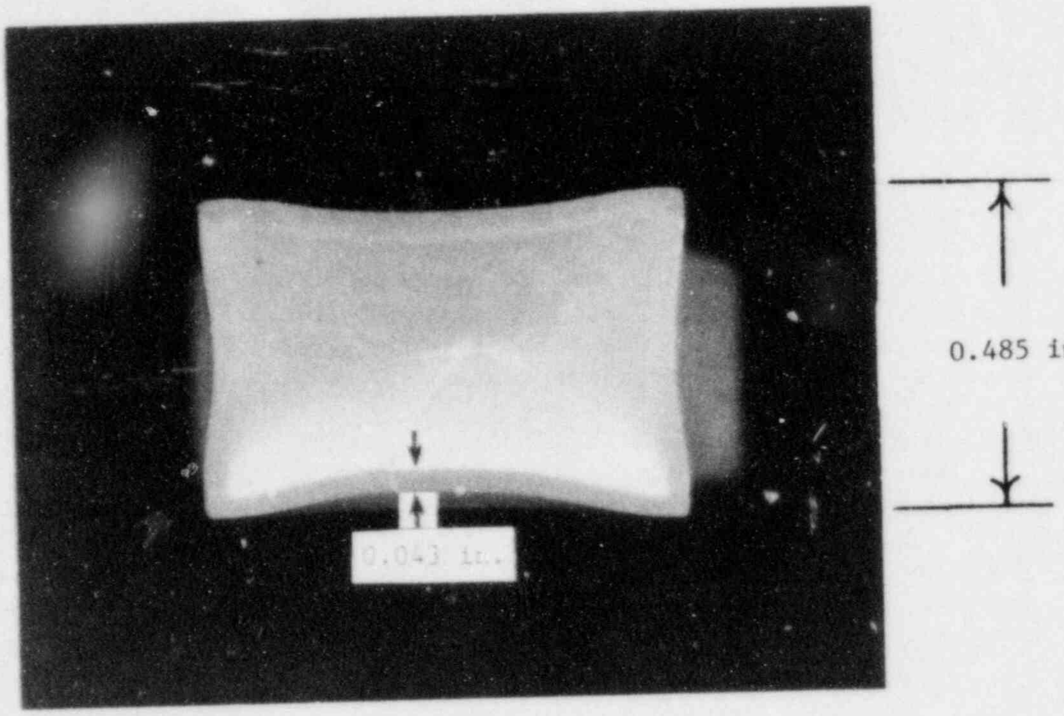

Figure 3.28 Cross-section of a broken Marlex HIC material tensile specimen irradiated in air at 60-63°C and at 5.7 krad/h for 66 days. The circumferential band, which is approximately 0.04-0.05 in. wide, appears to have been embrittled as a result of radiation-induced oxidation.

The final observation regarding the results of the irradiations in the soils and IX resin is that no interaction or reaction between the irradiated HDPE materials and the soils or resins was observed. Specifically, the soils and IX resins did not discolor (over and above that caused by irradiation), etch, stick to or even leave spots on the HDPE material surfaces. The irradiated HDPE appeared to be no less inert to the soil and IX resin environments, under the conditions of these tests, than is the unirradiated material.

4. BEND TESTING

Bend testing of Marlex HIC material and the Chemplex was conducted to determine any effects of irradiation on the stiffness and flexibility of these materials. The Marlex became stiffer upon irradiation whereas the Chemplex did not become noticeably stiffer or less flexible.

4.1 Bend Test - General

Bend tests on MARLEX CL-100 HIC material and CHEMPLEX 5701 have been performed according to ASTM D-790 (Flexural Properties of Plastics and Electrical Insulating Materials). Specifically, Method II, Procedure B (which utilizes four point loading on a test specimen and is used particularly for materials that undergo large deflections) from D-790 was chosen as most appropriate for these tests. The experimental setup for this testing is illustrated schematically in Figure 4.1.

Flexural strength is defined as being equal to the maximum stress at the moment of break. Since the polyethylenes tested in this task do not break, D-790 is not formally applicable.* However, this test does provide useful information on the effect of irradiation on the stiffness of the polyethylene being tested.

The flexural strength is calculated from the following formula:

Flexural Strength = $S = PL/bd^2$

S = stress in the outer fiber

P = the load at a specified point on the load versus deflection curve

L = the support span (Figure 10)

b = width of the test piece

d = thickness of the test piece**

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*ASTM D-790 is formally applicable only for those materials which break at no more than 5% strain. Other tests, e.g. ASTM D-638, are recommended when the 5% limit is exceeded. The results of testing according to D-638 are presented in the section on tensile testing in this report.

**The support span and the width of the rectangular test piece are dependent upon the thickness of the material being tested. They are determined from a Table of Recommended Dimensions in D-790.

The tangent modulus, M, is related to the intial slope of the load vs deflection curve and is related to the strength of the material in the region of elastic deformation. A high value of M is desirable since the stress required to give a specific amount of deformation is proportional to this value.

$$\text{Tangent Modulus} = M = 0.21\ L^3m/bd^3$$

m = slope of the tangent to the initial portion of the load versus deflection curve

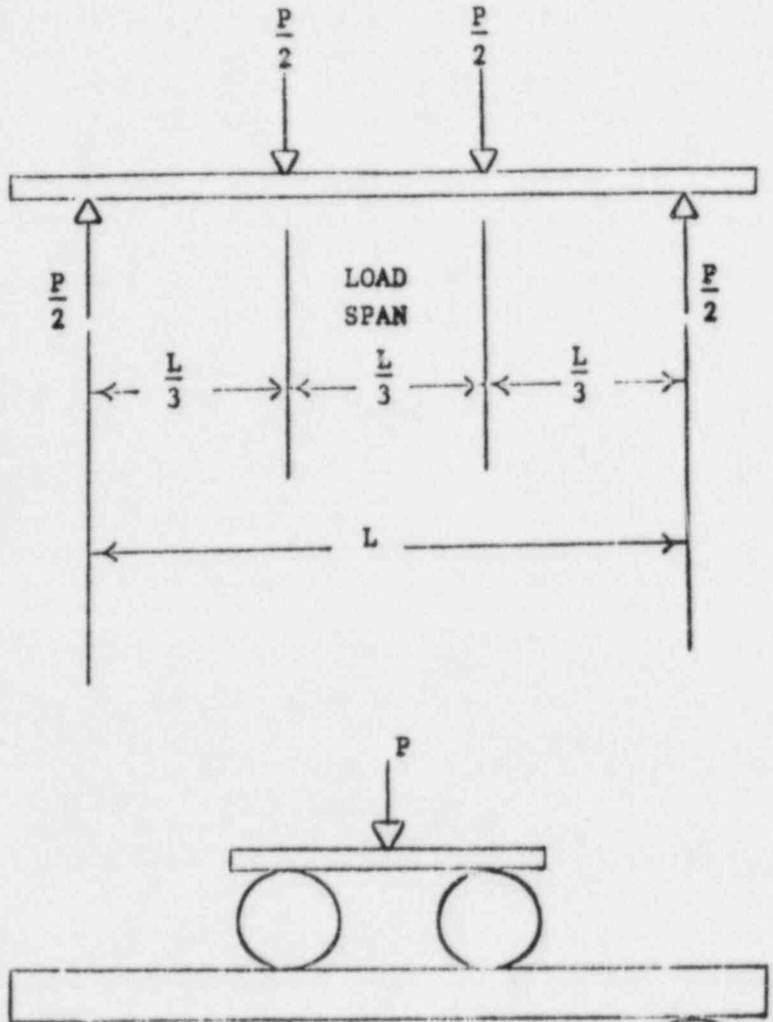

Figure 4.1. Schematic illustration of test setup for ASTM D-790, Method II, Procedure B. The upper diagram shows the loading on the specimen and the load span, where the maximum deformation occurs. The lower diagram illustrates at half scale the setup for testing the HIC material bend test specimen.

4.2 Bend Test - Results

The results of bend tests on irradiated Marlex HIC material are illustrated by the curves in Figure 4.2. These data show that the stiffness of the Marlex HIC material increased upon irradiation. As in the tensile test results described above, these tests also showed that the inside surface of this material cracks upon bending. However, 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.

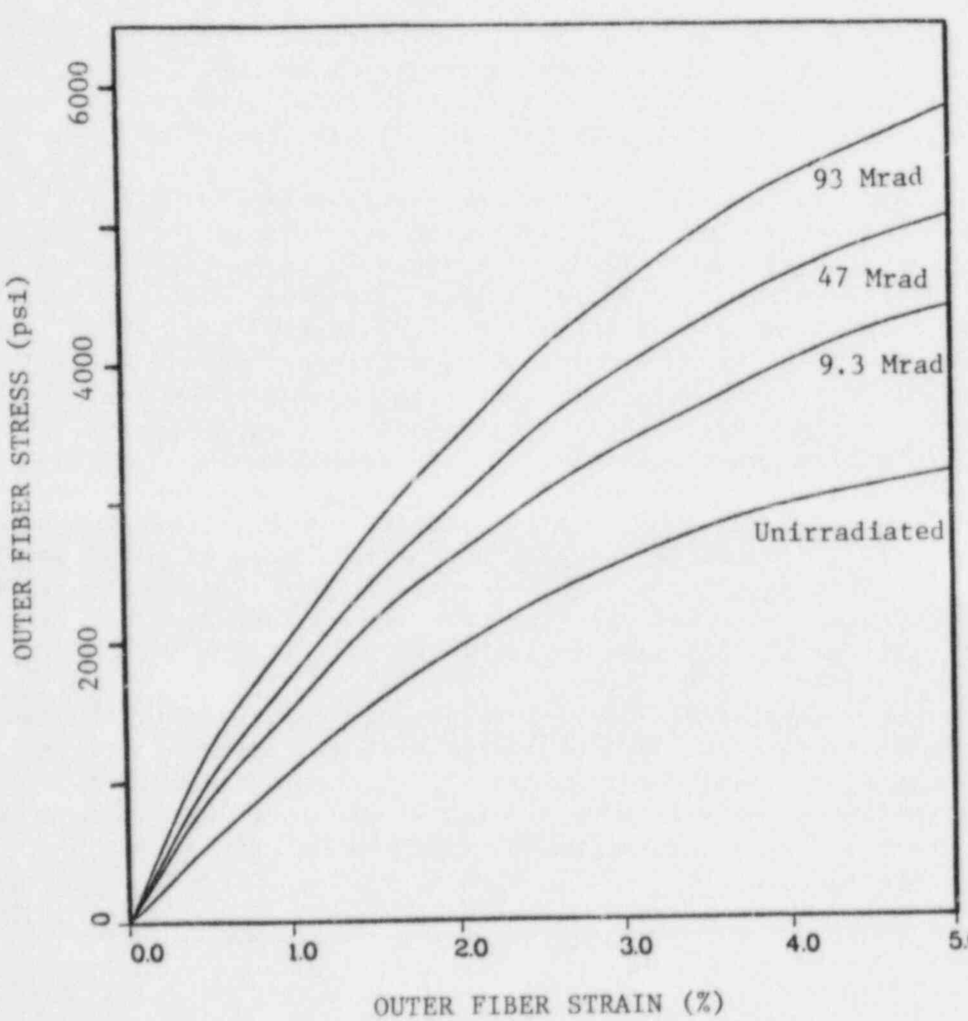

Figure 4.2. Bend test curves for irradiated Marlex CL-100 HIC material tested according to ASTM D-790.

The Chemplex behaved differently in the bend testing. It did not get noticeably stiffer following irradiation until doses of approximately 50 Mrad had been attained and the effect was much smaller than that observed for the Marlex. The Chemplex did not crack upon cycling in the bend test machine.

The data obtained to date from the bend testing of the Chemplex 5701 are listed in Table 4.1, while those for the Marlex CL-100 HIC material are listed in Table 4.2. The data from these tables are shown in Figures 4.3-4.6. The reported values are flexural strength and tangent modulus. Since the material does not break, the reported flexural strength is actually a measure of the force required to bend the test specimen a specific amount, which, in these tests, is to 5% strain.

The conclusions from the bend test results are summarized below:

- There appears to be no significant effect of irradiation on the stiffness, as measured by flexural strength or tangent modulus, of Chemplex 5701 up to approximately 50 Mrad. For doses greater than this there may be an increase in stiffness in all four irradiation environments.

- The bend test measurements on the Chemplex generally have a rather large associated error. These large errors mean that any effects of irradiation and environment would need to be large to become apparent from these data. The variability of the data in the bend tests is probably a property of the material. The bend test is not as severe a test as is the tensile test, which stresses the test piece to failure and for which the associated error is typically smaller. It may be that the individul differences in test specimens are more apparent in this material when not tested to failure.

- The bend test results for the Marlex CL-100 HIC material suggest that there is a noticeable increase in the stiffness of this material upon irradiation in all four environments. No statistics were gathered for these tests since only one specimen was run per test due to space limitations in the gamma irradiators.

The increase in stiffness of the Marlex, as indicated in the bend test results, contrasts with the lack of increase in stiffness of the Chemplex up to 50Mrad. 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.

Table 4.1

Flexure Test Data on Irradiated CHEMPLEX 5701[a]

Dose (Mrad)	Dose[b] Rate (krad/h)	Environment	Outer Fiber Stress (psi) at 5% Strain	Tangent Modulus (psi)
0	----	----	4200 ± 500	168,000 ± 15,000
9.3	93	air	4800[c]	161,000
47	93	air	5300[c]	219,000
93	93	air	5800[c]	225,000
8.6	17	air	4200 ± 800	171,000 ± 39,000
25	14	air	3400 ± 100	126,000 ± 7,000
9.5	5.7	air	3800 ± 500	144,000 ± 31,000
2.7	2.5	air	4300 ± 200	184,000 ± 5,000
3.6	2.5	air	3600 ± 300	171,000 ± 7,000
9.7	58	Barnwell soil	4400 ± 2400	125,000 ± 83,000
50	58	Barnwell soil	4600 ± 200	188,000 ± 3,000
8.5	11	Barnwell soil	4300 ± 200	174,000 ± 25,000
20	11	Barnwell soil	3700 ± 200	154,000 ± 4,000
8.0	3.7	Barnwell soil	4800 ± 100	234,000 ± 13,000
2.0	1.4	Barnwell soil	4300 ± 200	178,000 ± 24,000
50	58	Hanford soil	5200 ± 500	237,000 ± 43,000
8.5	11	Hanford soil	3400 ± 1100	132,000 ± 54,000
20	11	Hanford soil	4700 ± 200	235,000 ± 22,000
3.0	4.0	Hanford soil	4500 ± 400	194,000 ± 23,000
13	79	IX resin	4800 ± 300	220,000 ± 23,000
49	79	IX resin	5600 ± 600	226,000 ± 12,000
100	79	IX resin	4600 ± 200	244,000 ± 25,000
20	11	IX resin	4400 ± 100	238,000 ± 35,000
10	8.7	IX resin	4300 ± 100	206,000 ± 16,000
3.0	3.7	IX resin	4700 ± 100	185,000 ± 13,000

[a]Flexure testing was performed according to ASTM D-790 (Flexural Properties of Plastics and Electrical Insulating Materials) Method II, Procedure B, using three specimens per test. Test specimens were machined from sidewall material obtained from plain (not color pigmented) 55-gal drums purchased from PLASTI-DRUM Co., Lockport, IL.
[b]Irradiations were performed in the BNL Co-60 gamma facility at 10-11°C.
[c]Only one specimen tested.

Table 4.2

Flexure Test Data on Irradiated EnviroSAFE[a] High-Integrity Container Material[b]

Dose (Mrad)	Dose[c] Rate (krad/h)	Environment	Outer Fiber Stress (psi) at 5% Strain	Tangent Modulus (psi)
0[d]	----	----	3200 ± 200	122,000 ± 8000
9.3	93	air	4400	250,000
47	93	air	5100	325,000
93	93	air	5900	351,000
8.6	17	air	4500	189,000
25	14	air	4800	168,000
9.5	5.7	air	4700	162,000
2.7	2.5	air	4700	164,000
3.6	2.5	air	4300	114,000
9.7	58	Barnwell soil	7200	137,000
50	58	Barnwell soil	5300	166,000
8.5	11	Barnwell soil	4600	134,000
20	11	Barnwell soil	5200	188,000
8.0	3.7	Barnwell soil	4800	183,000
2.0	1.4	Barnwell soil	4400	140,000
50	58	Hanford soil	5800	204,000
8.5	11	Hanford soil	5200	190,000
20	11	Hanford soil	4900	167,000
3.0	4.0	Hanford soil	4600	146,000
13	79	IX resin	5000	149,000
49	79	IX resin	5100	167,000
100	79	IX resin	5400	203,000
20	11	IX resin	4500	170,000
10	8.7	IX resin	4500	152,000
[illegible].0	3.7	IX resin	5000	154,000
3.0	4.0	IX resin/ Barnwell soil	3600	165,000
10	11	IX resin/ Barnwell soil	4600	148,000

[a]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.
[b]Flexure testing was performed according to ASTM D-790 (Flexural Properties of Plastics and Electrical Insulating Materials) Method II, Procedure B, using one specimen per test. Test specimens were machined from HIC material cut out during container fabrication. These container cut-outs were obtained from Poly-Processing Co., Monroe, LA, who manufactures these HICs for CHEM-NUCLEAR SYSTEMS, Inc.
[c]Irradiations were performed in the BNL Co-60 gamma facility at 10-11°C.
[d]Five unirradiated specimens were tested.

IMAGE EVALUATION
TEST TARGET (MT-3)

150mm

6"

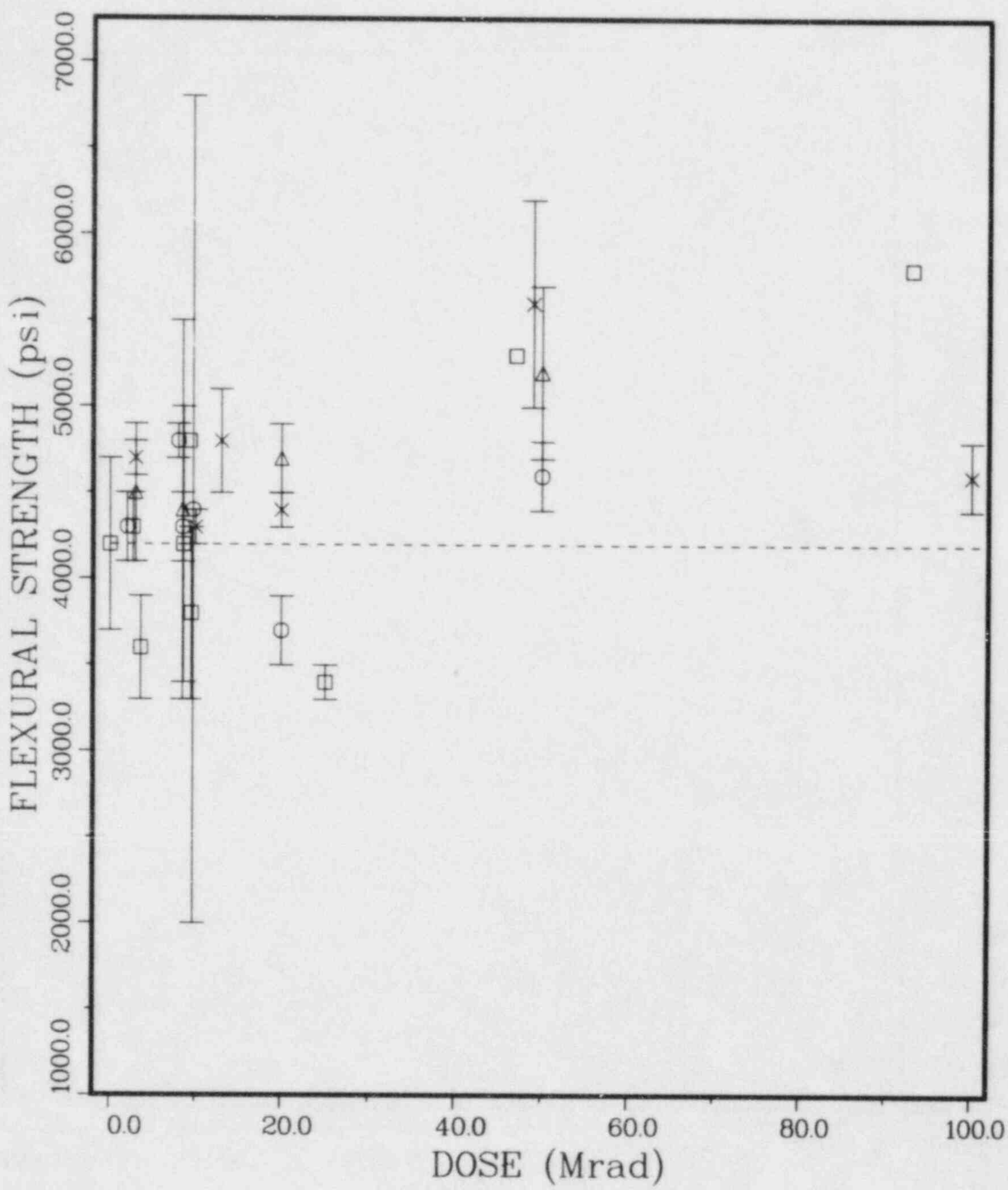

Figure 4.3. Outer fiber stress (psi) at 5% strain vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 4.1. Symbols are defined in the Legend.

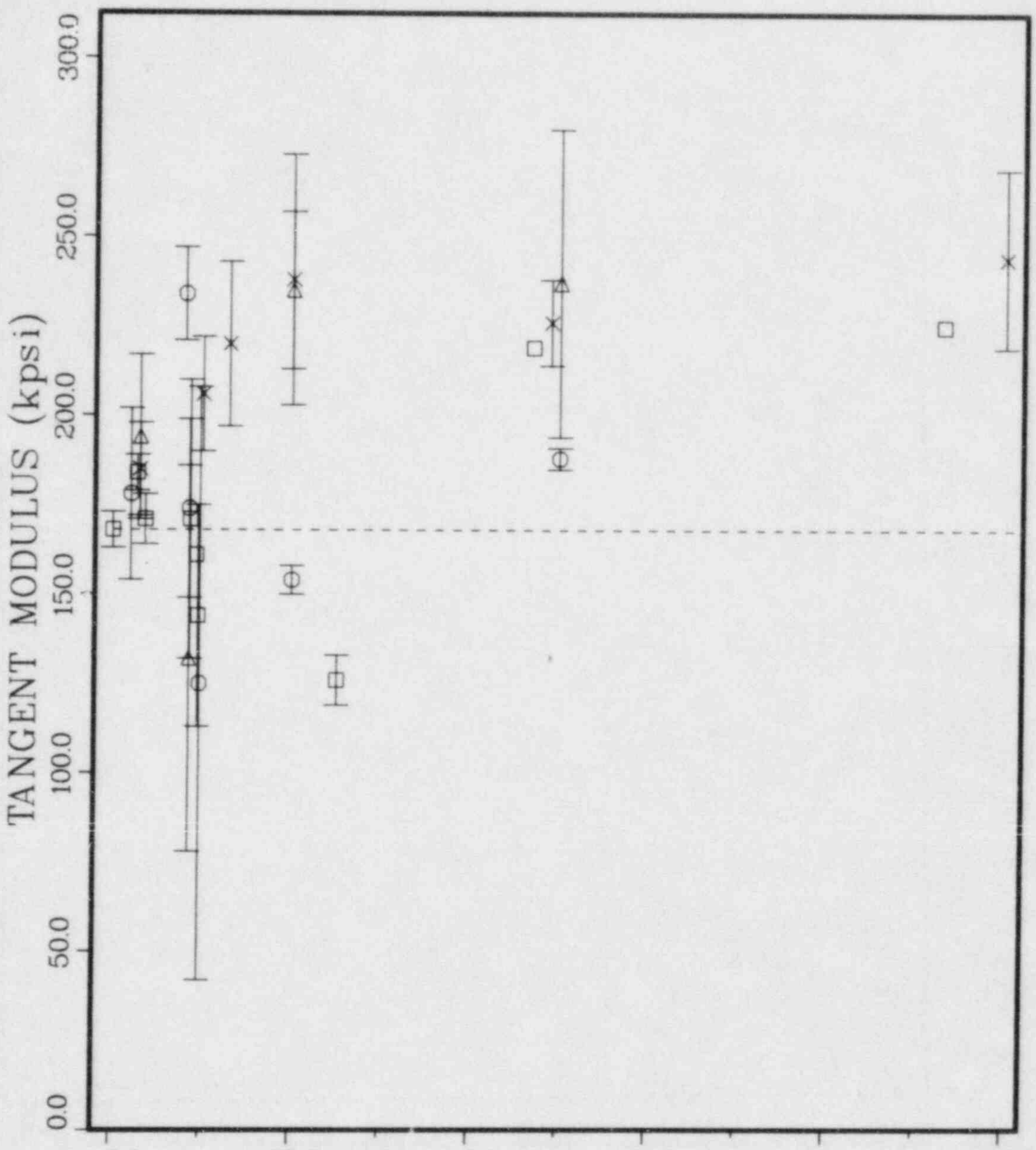

Figure 4.4. Tangent modulus (kpsi) vs dose (Mrad) of irradiated Chemplex 5701. The data are from Table 4.1. Symbols are defined in the Legend.

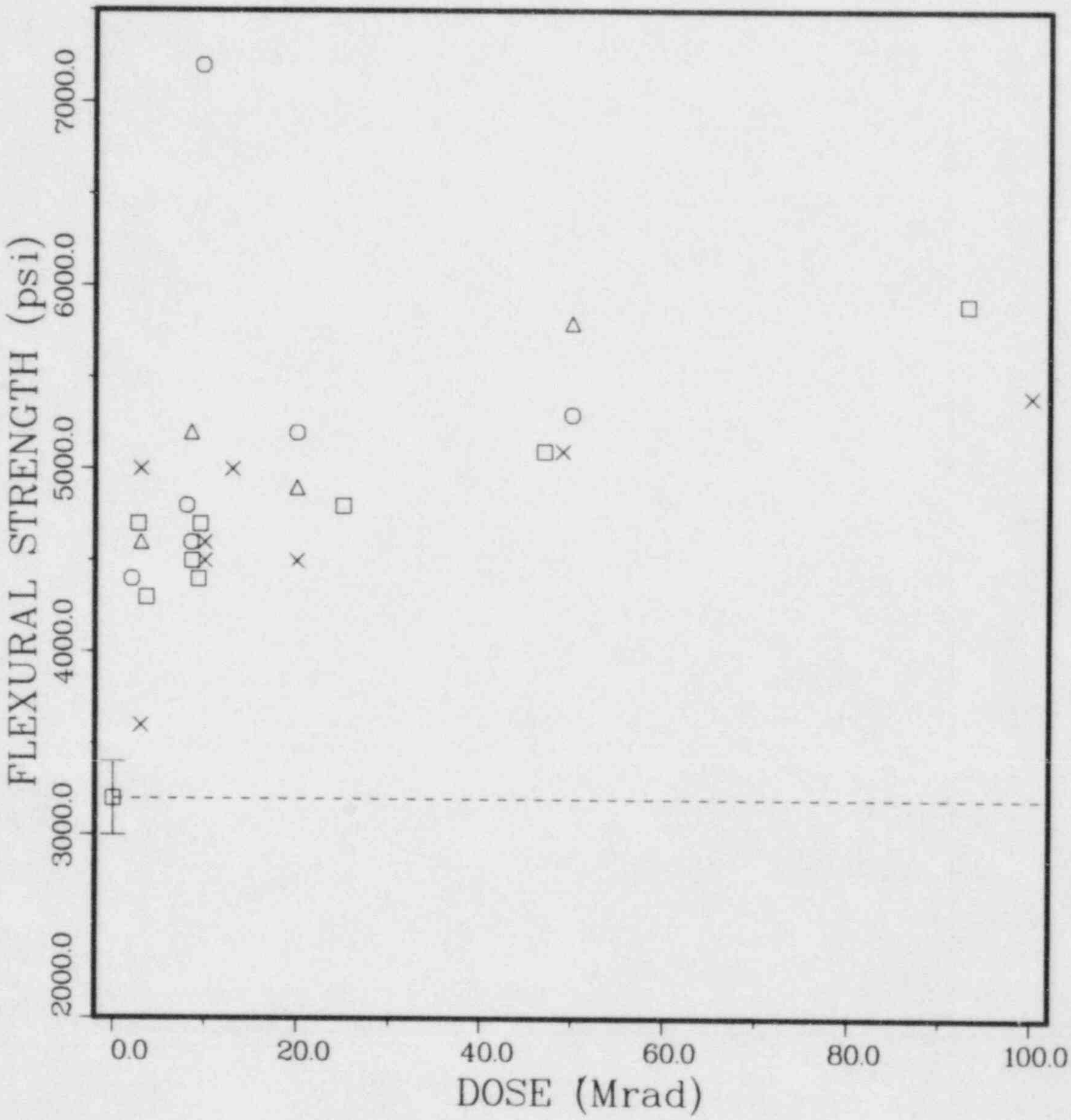

Figure 4.5. Outer fiber stress (psi) at 5% strain vs dose (Mrad) of irradiated Marlex CL-100 HIC material. The data are from Table 4.2. Symbols are defined in the Legend.

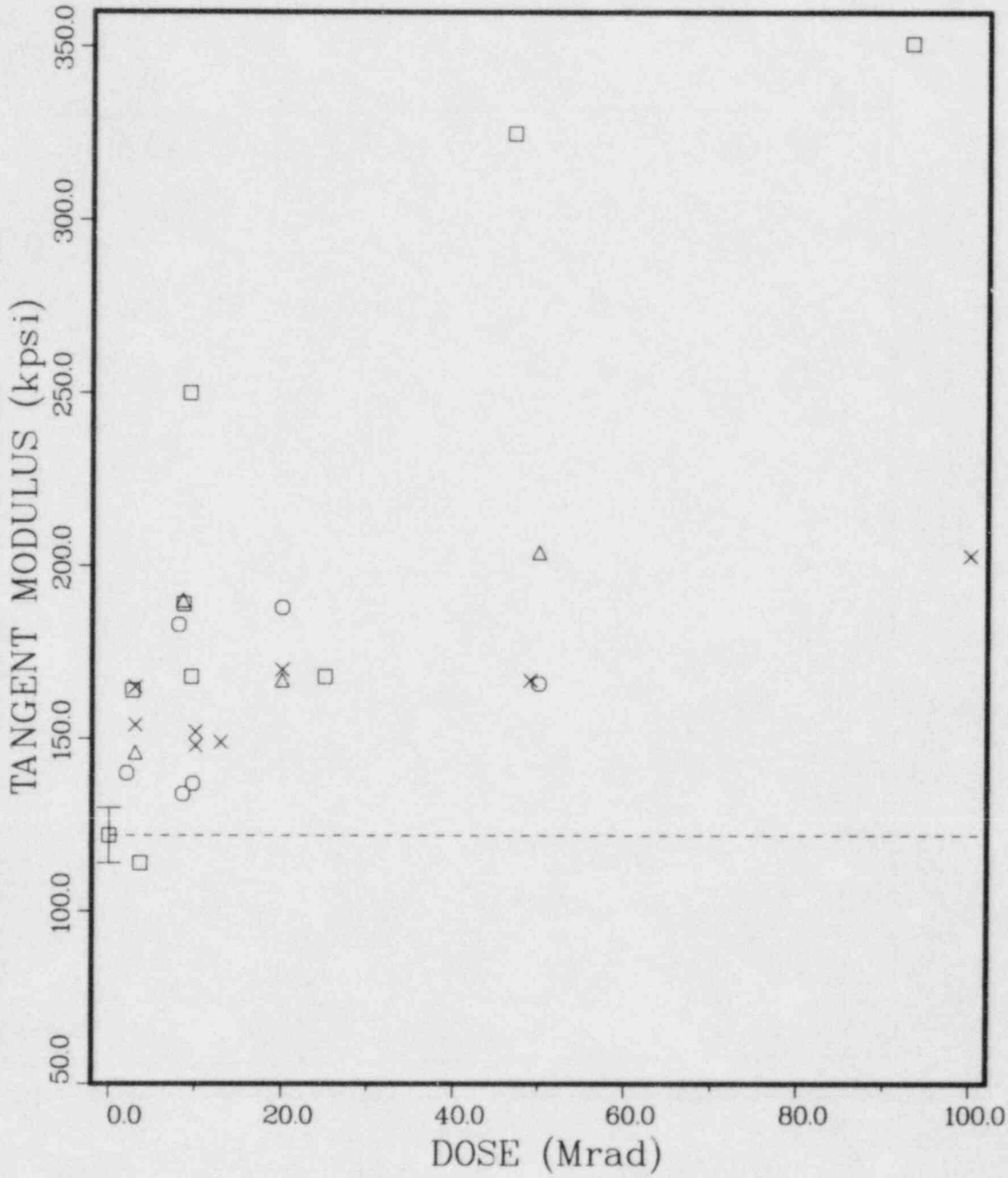

Figure 4.6. Tangent modulus of irradiated Marlex CL-100 HIC material. The data are from Table 4.2. Symbols are defined in the Legend.

5. CREEP DURING IRRADIATION

Irradiation of polymeric materials under stress has been shown to increase creep rates compared to creep in the absence of irradiation.[8,9] Since the TP recommends that, "for polymeric material, design mechanical strengths should be extrapolated from creep test data," creep in polyethylene was measured during irradiation. We have found that creep does indeed increase during irradiation in HDPE. The increase appears to be stress-dependent, i.e., the larger the stress, the larger the increase in creep during irradiation. The effect of dose rate was not investigated.

5.1 Creep - General

The deformation that occurs in a material under a steady load for a long period of time is called creep. Measurements of creep behavior are usually conducted by applying a constant load, or stress, and measuring the resulting deformation, or strain, of the test specimen at a constant temperature. Figure 5.1 presents a schematic creep curve which is typical of most materials.[22] In the first stage, often called primary creep, the creep rate starts out at a relatively high value and decreases toward a constant rate. The second stage is characterized by the constant rate of deformation and is called secondary creep. In the third stage, the creep rate increases up to the break. Although primary creep always occurs under load because of the elastic properties of materials, the other two stages need not appear. At low enough loads and/or low enough temperatures the secondary creep rate is zero. At high loads and/or temperatures the decelerating creep rate in the first stage passes immediately into the accelerating creep rate of stage three, leading rapidly to the break. Secondary creep behavior at the temperatures and loads typical of operational service is used for design purposes and for estimates of useful lifetimes for real systems.

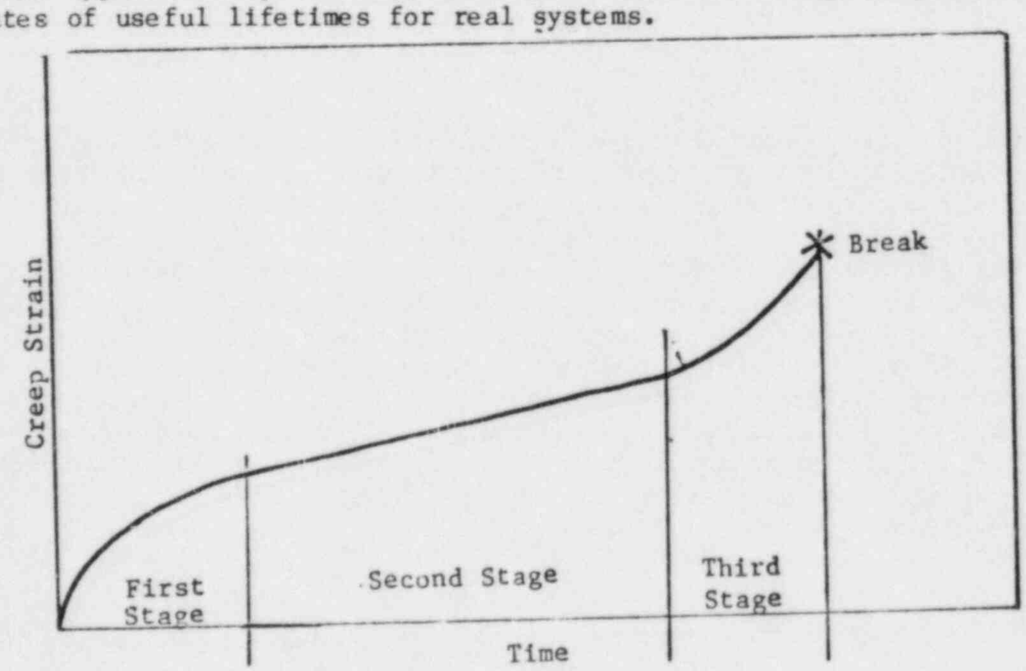

Figure 5.1. Schematic creep curve showing the three stages of creep.

5.2 Creep During Irradiation - Results

Figure 5.2 shows data from the first experiments conducted in the devices built to study creep during irradiation under this task. These were performed as scoping tests to guide the follow-on tests. The creep loads chosen for these first tests were chosen based on the results reported for creep at room temperature.[23] The data of Arora and Dayal[23] show that very little creep occurred at 900 psi, whereas, significant creep (nearly 100% elongation in 1-5 days) occurred at room temperature under loads of 1500-1600 psi. The change from room temperature (20-25°C) to the 10-11°C (irradiated specimens) and 6-12°C (unirradiated specimens) for the data shown in Figure 5.2 resulted in significantly less creep at the higher loads. Little effect was noted at the lower loads, where only a small amount of creep occurred at both temperatures. As a consequence of these results, stress loadings in following experiments were increased to 1600 psi and 1800 psi.

An additional consideration for the data in Figure 5.2 is that these specimens were irradiated for four days before the creep loads shown were applied. The four-day delay was to conduct dosimetry on the system in the loaded configuration. The stress loads applied to all specimens during the dosimetry was approximately 300 psi. No creep whatsoever was noted during this four-day period. The effect of dose accumulated during this four-day period is not known, but comparison of the 900 psi curves in Figures 5.2 and 5.3 may indicate that this four-day pre-irradiation may have resulted in an increased creep rate for a day or two following application of the 900 psi creep load. The large jumps in creep for the Marlex, 918 psi specimen between days 14-21 and 42-49 may be due to the apparatus. Although no evidence of sample slip-page in the grips was noted upon removal of the specimen from the holders, it is unlikely that these jumps are real. Therefore, the Marlex 918 psi curve probably does not reflect the actual creep behavior of the material. This curve was retained because the data for the first 14 days appear to be reliable.

Figure 5.3 shows creep data for unirradiated controls at stress loadings of 900 and 1600 psi. The unirradiated controls were tested in an apparatus identical to that used for the irradiated specimens, but in a different pool so as to be completely out of the radiation field. The temperature of the pool in which the unirradiated specimens were tested was nominally 10-11°C, but over a period of 9 days after testing began on these specimens the temperature drifted down to 6°C and then up to 12°C before it stabilized at 10-11°C. We do not know if these temperature excursions would noticeably affect the creep results shown in Figure 5.3 since the elongations recorded during this 9-day period were not more than 7%. Arora and Dayal under NRC contract FIN A-3027 observed that creep behavior of Marlex CL-100 was not noticeably affected by temperature variations of 3-4°C near room temperature.[22]

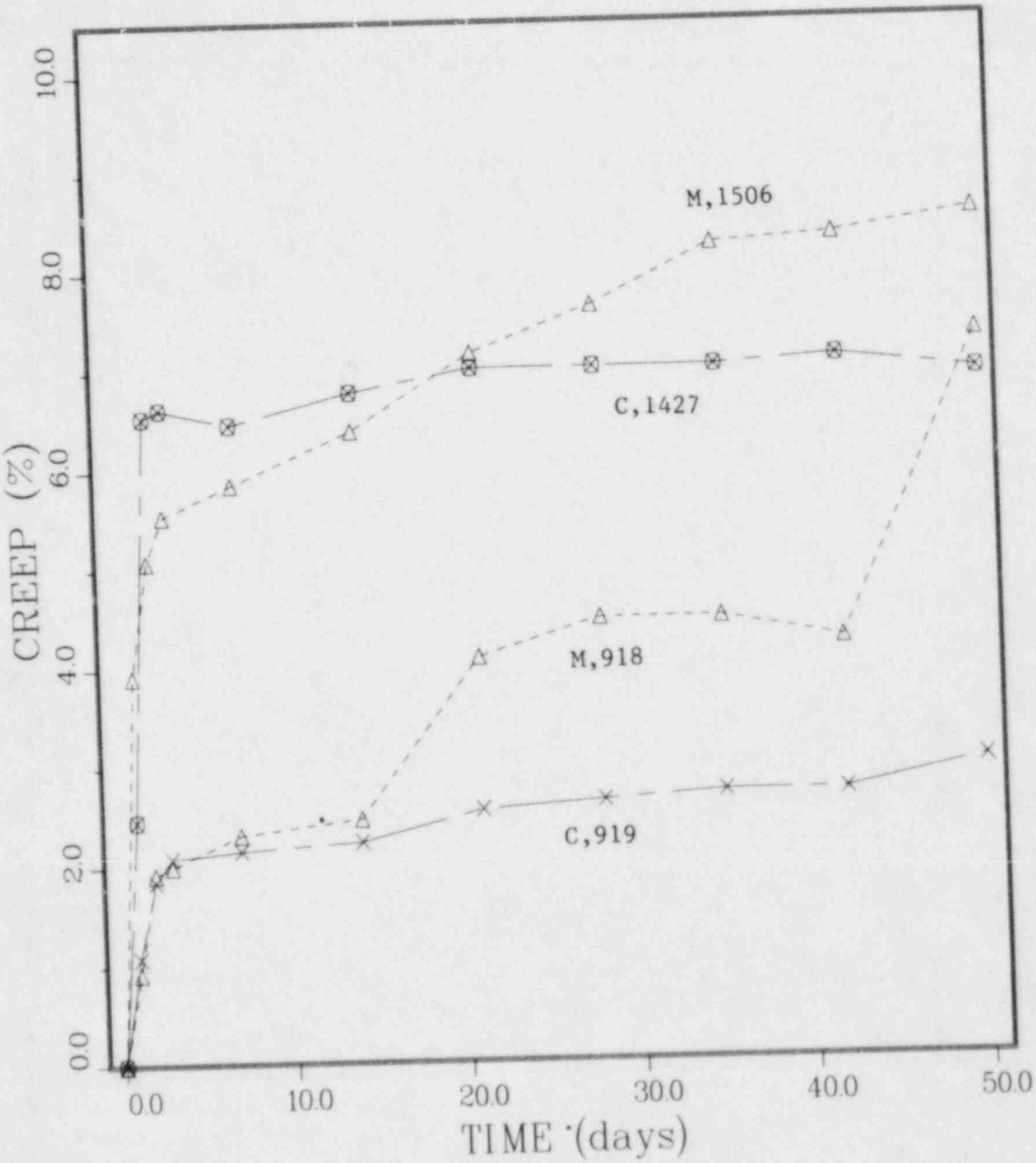

Figure 5.2. Creep (%) vs time for ASTM Type IV tensile specimens of Marlex CL-100 non-HIC material and Chemplex 5701 undergoing creep during irradiation in air at 10-11°C and at 5 krad/h. The curves are labelled by material (M for Marlex and C for Chemplex), load (psi).

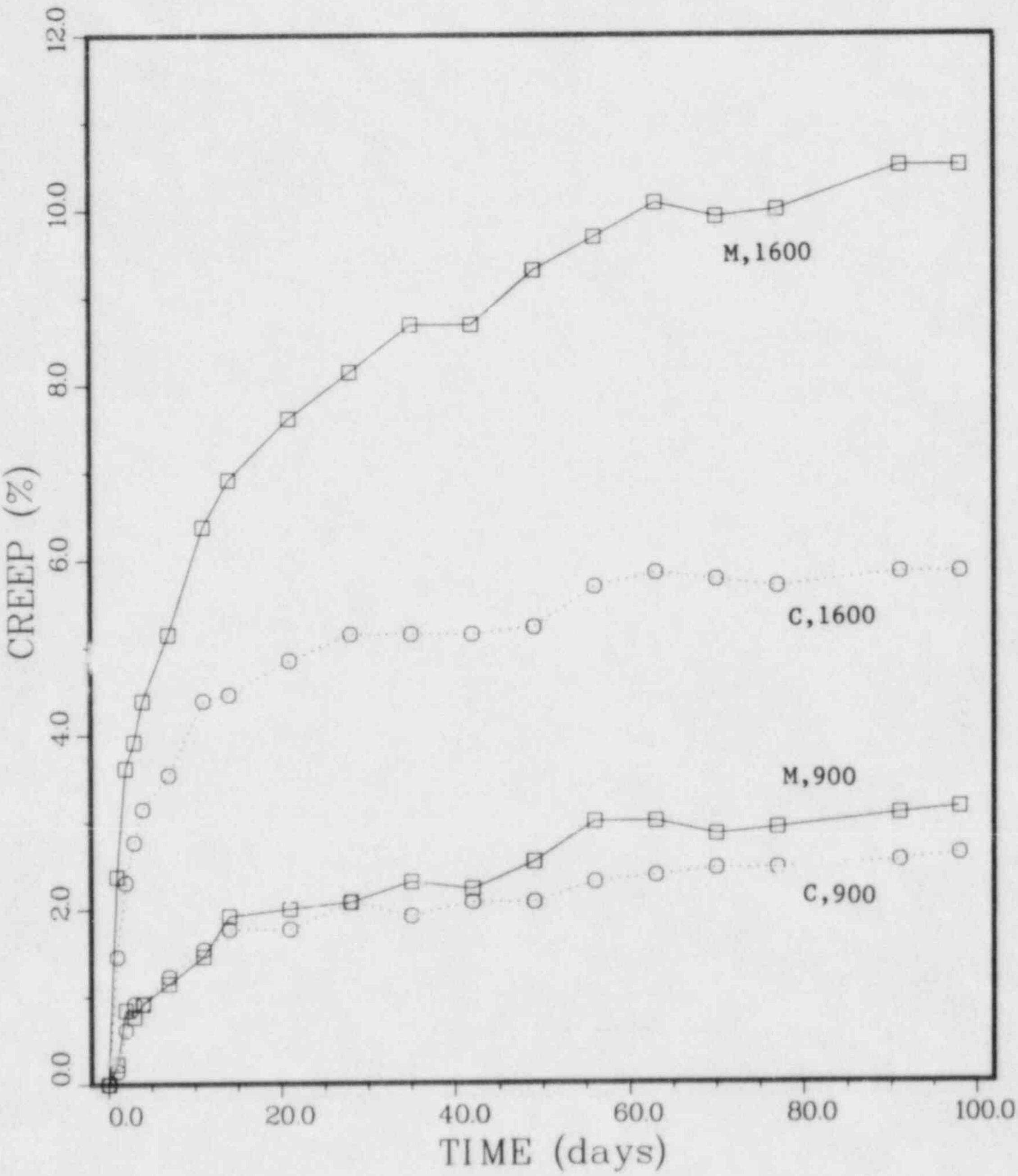

Figure 5.3. Creep (%) vs time for ASTM Type IV tensile specimens of Marlex CL-100 non-HIC material and Chemplex 5701. The curves are labelled by material (M for Marlex and C for Chemplex), load (psi). These curves are for the unirradiated controls.

Figure 5.3 shows that there appears to be little, if any, difference in creep between the Marlex and Chemplex at the lower stress, whereas, at higher stress the Marlex creeps more than Chemplex. The creep results at the higher stress are consistent with the measurements of yield stress in Tables 3.1 and 3.3, wherein Chemplex has been found to be the stronger material.

Figure 5.4 shows results on creep during irradiation in air at 10-11°C and 5 krad/h at loads of 1600 and 1800 psi. The Marlex creeped faster than the Chemplex, especially at the 1800 psi load. Comparison of the M,1600 psi curves in Figures 5.3 and 5.4 reveals no significant difference in creep between the test specimens undergoing irradiation and those not in the radiation field. These experiments were stopped on day 71 when the 1800 psi Chemplex specimen broke. (This is discussed later in this section.)

Figure 5.5 shows the results obtained for a set of unirradiated controls which underwent creep in air at 1600 and 1800 psi. Both the 1600 psi and 1800 psi unirradiated Chemplex test specimens creeped less than the irradiated 1600 psi Chemplex specimen in Figure 5.3. The 1600 psi Marlex specimen in Figure 5.5 also creeped less than the 1600 psi Marlex specimen in Figure 5.3.

The difference in creep between the unirradiated specimens in Figures 5.3 and 5.5 may be normal variation between test specimens or it may be due to the excursion to higher temperature that occurred in the early stages of the experiment, the data for which is shown in Figure 5.3. We were not be able to determine the reason for the difference.

Figure 5.6 shows the results for irradiation under stress experiments in IX resin at 1600 and 1800 psi. Each of the curves in Figure 5.6 show more creep than the equivalent curve in Figure 5.5. It is particularly evident that the 1800 psi Marlex specimens creep faster in Figure 5.6 than the equivalent specimen in Figure 5.5.

The data shown in Figure 5.4 for irradiation under stress in air, compared to Figure 5.5, also suggest that creep rates are higher during irradiation, other things being equal, than in the absence of irradiation. This effect again is more evident in the 1800 psi Marlex data than in the other data curves. The 1800 psi Marlex curves in Figures 5.4 and 5.6 are very similar and both are approximately 50% higher than the equivalent unirradiated curves in Figure 5.5.

The irradiation under stress experiments suggest the following results for creep rates.

- Irradiation under stress increases the creep rate. At the dose rate used in these tests, 5 krad/h, this effect does not become apparent until the higher stresses are applied. Thus, although the creep at 1800 psi for Marlex Cl-100 in the radiation field appears to be approximately 50% greater than that in the absence of the radiation field, at the lower stresses the effect is not really apparent.

- The limited amount of data presently available suggest that the presence of IX resin makes no significant difference compared to air on the creep rate during irradiation under the conditions of temperature and dose rate (i.e., 10-11°C and 5 krad/h) used in these tests.

Between day 70 and 71, the 1800 psi Chemplex specimen broke while undergoing irradiation. The creep data for this test specimen is the curve labelled C,1800, in Figure 5.4. Examination of the pieces showed that the specimen had undergone creep rupture by brittle fracture. Creep rupture is defined as a break that occurs under a continuously applied stress which is below the nominal tensile strength.(24) The 1800 psi creep load which led to creep rupture in the radiation field in 70 - 71 days is approximately half of the nominal tensile strength of approximately 3460 to 3740 psi, as listed in Table 3.1.

Figure 5.7 is a picture of the Chemplex tensile specimens from the irradiation under stress experiment from Figure 5.4. The broken piece, top, is the creep ruptured 1800 psi specimen. Second from the top is the 1600 psi specimen. The ruler and the untested tensile specimen at bottom are for scale.

Figure 5.8 shows the Marlex 1800 psi specimen, top, the 1600 psi specimen, middle, and an untested specimen and ruler. The 1800 psi Marlex piece had some surface cracks, but did not break even though it creeped more than three times as much as the Chemplex 1800 psi specimen. A closeup of the 1800 psi Marlex specimen is shown in Figure 5.9. The average crack spacing is approximately 1/16-inch. None of these cracks had penetrated into the specimen to a depth greater than their approximate width. The 1600 psi specimen showed no surface cracks. The total dose absorbed by these specimens during the irradiation was 8.4 Mrad.

Following creep testing the creep specimens were tensile tested to measure the effect, if any, of creep on the tensile properties on both irradiated and unirradiated material. Table 5.1 lists the tensile data following creep for the Chemplex specimens and Table 5.2 lists similar data for the Marlex non-HIC material specimens.

The Chemplex data in Table 5.1 compared to approximately equivalent data in Table 3.1 suggests that creep tended to have increased the yield stress while the elongation at yield was not significantly affected. Elongation at break for the unirradiated Chemplex was not changed from that of uncreeped material. However, for the irradiated creeped Chemplex the break occurred at somewhat greater elongations than would be expected for similarly irradiated uncreeped material (i.e., compared to the 9.5 Mrad material irradiated at 5.7 krad/h in Table 3.1).

The Marlex non-HIC material data in Table 5.2 compared to approximately equivalent data in Table 3.3 suggests that creep has not affected yield stress or elongation at yield, except for the 8.4 Mrad specimen which creeped 35%, for which the elongation at yield has increased. Elongation at break for the irradiated creeped specimens appears unchanged.

(Continued Page 79)

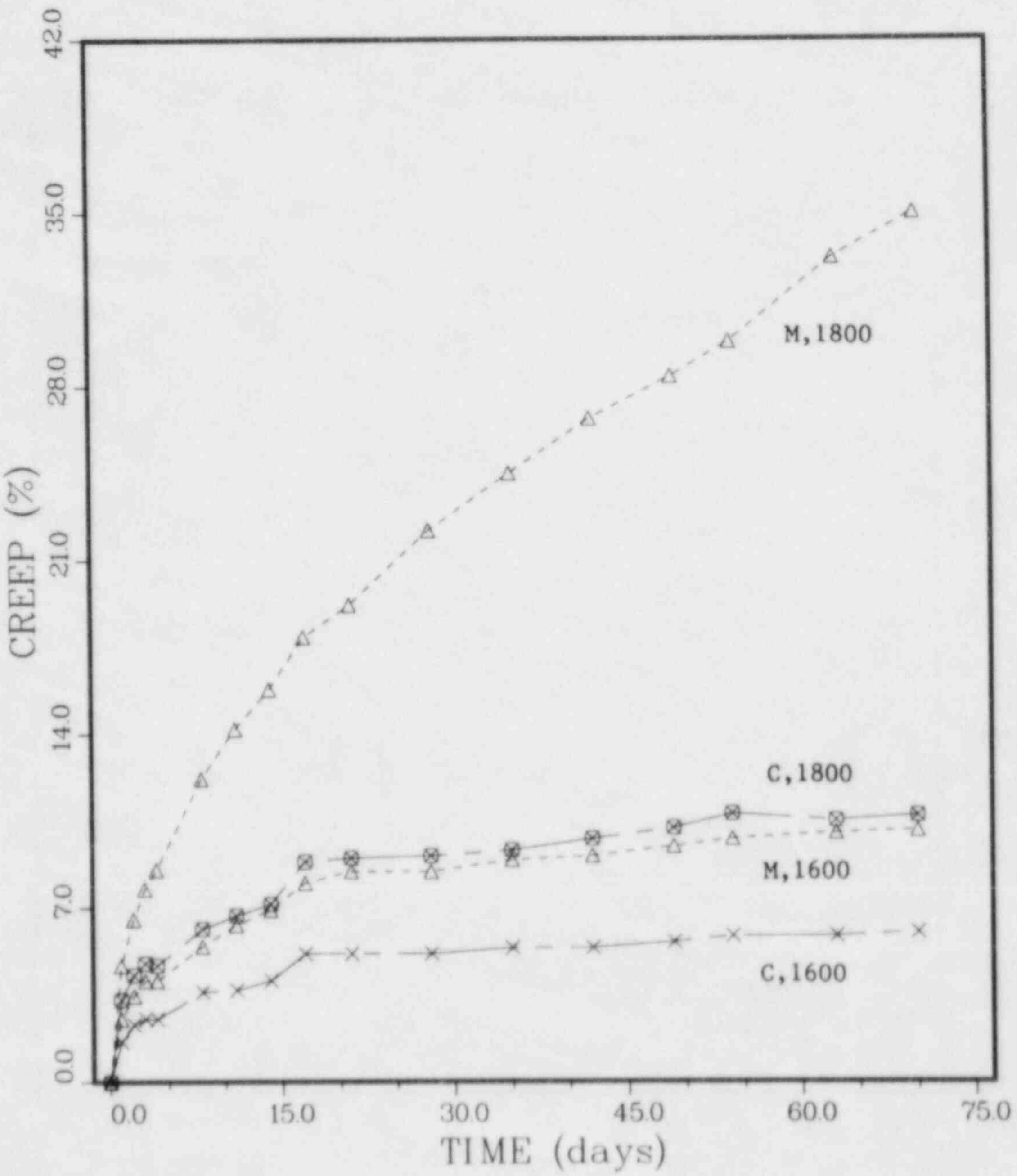

Figure 5.4. Creep (%) vs time for ASTM Type IV tensile specimens of Marlex CL-100 non-HIC material and Chemplex 5701 undergoing creep during irradiation in air at 10-11°C and at 5 krad/h. The curves are labelled by material (M for Marlex and C for Chemplex), load (psi). These experiments were stopped at day 71 when the C,1800 specimen broke.

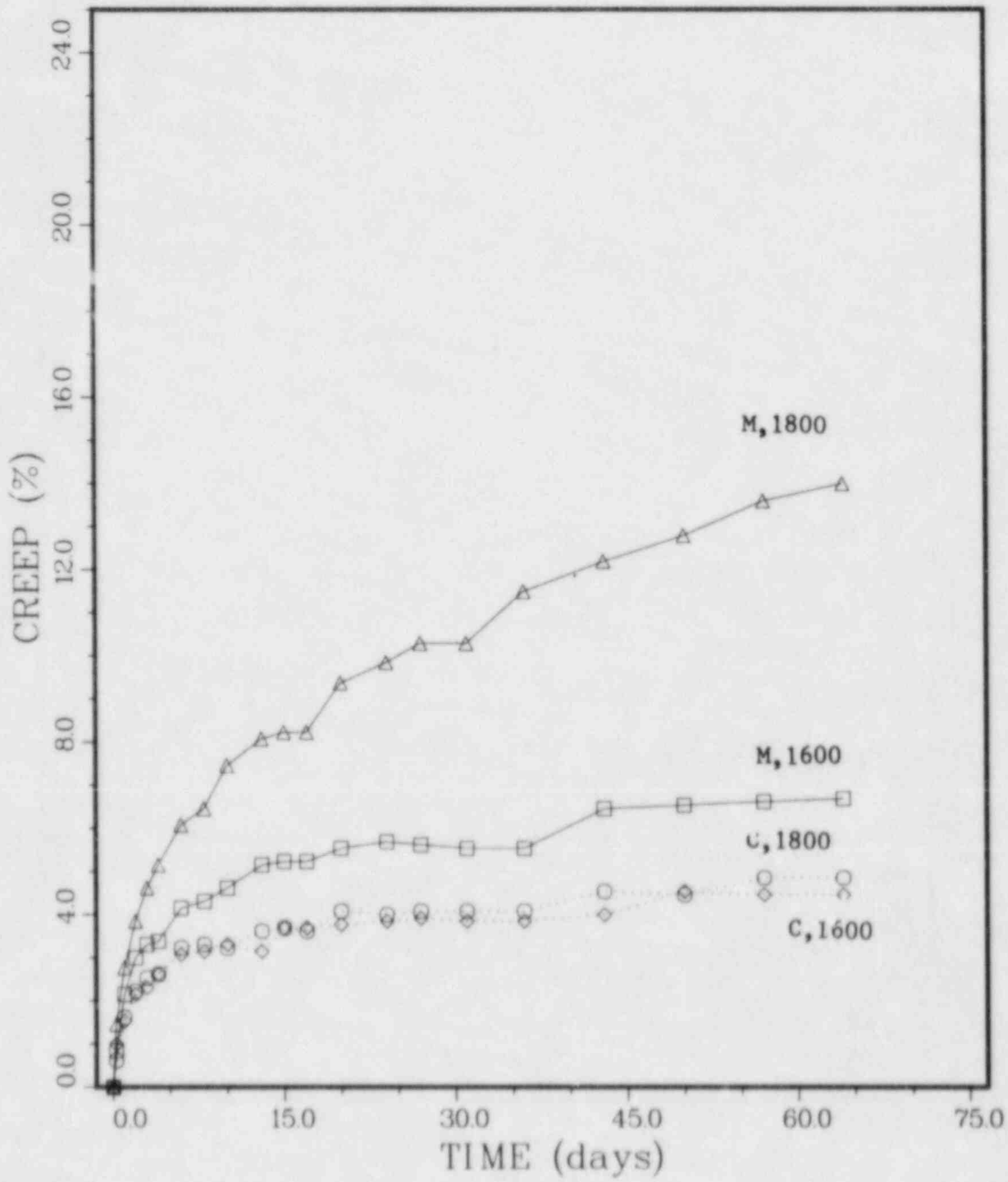

Figure 5.5. Creep (%) vs time for ASTM Type IV tensile specimens of Marlex CL-100 non-HIC material and Chemplex 5701. The curves are labelled by material (M for Marlex and C for Chemplex), load (psi). These curves are for the unirradiated controls.

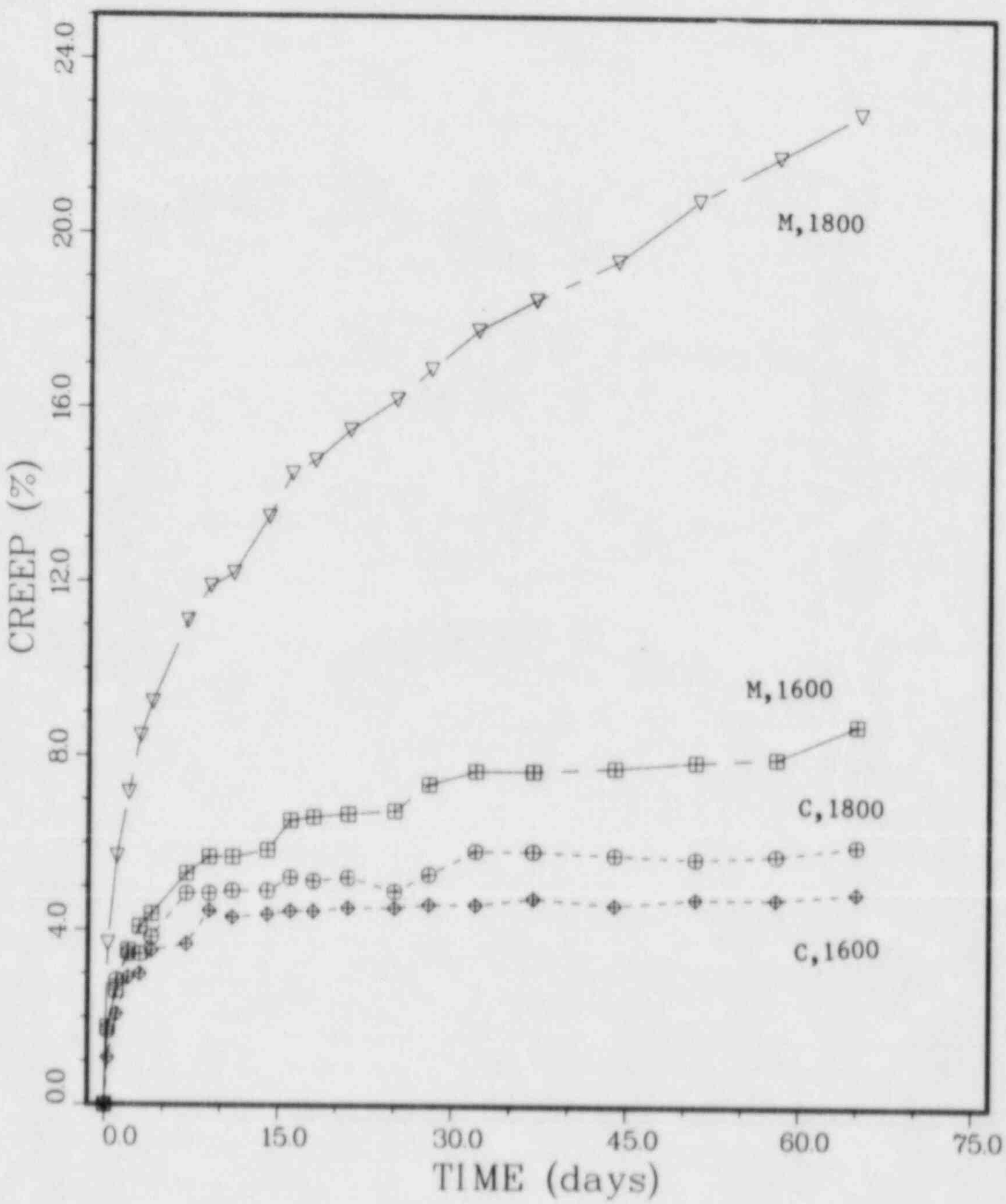

Figure 5.6. Creep (%) vs time for ASTM Type IV tensile specimens of Marlex CL-100 non-HIC material and Chemplex 5701 undergoing creep during irradiation at 5 krad/h in IX resin. The curves are labelled by material (M for Marlex and C for Chemplex), load (psi).

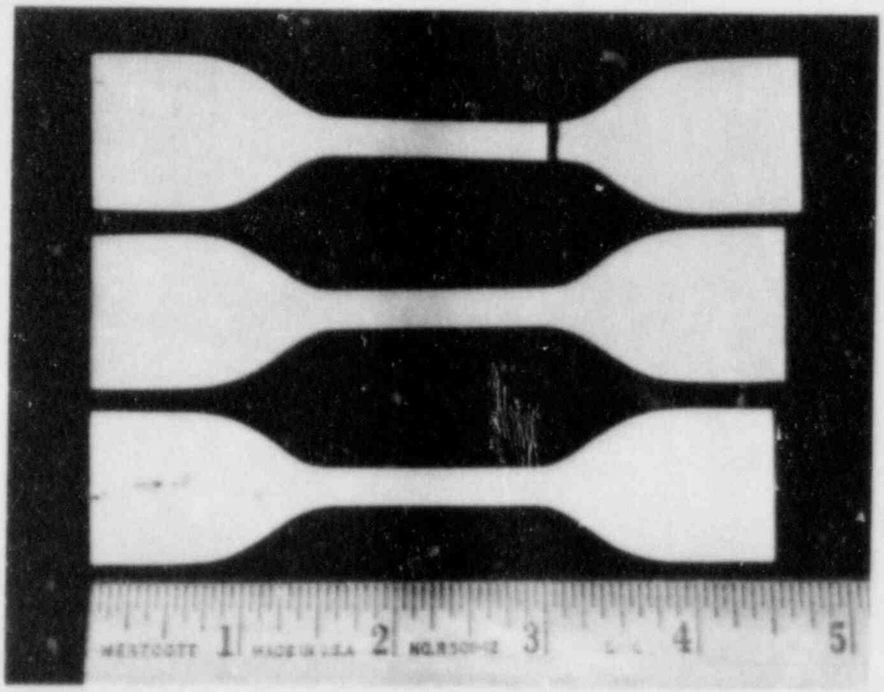

Figure 5.7. Chemplex 5701 tensile specimens from the irradiation under stress experiment in air at 10-11°C in air. The top (broken) specimen was stressed at 1800 psi. The middle specimen was stressed at 1600 psi. The bottom specimen is untested and the ruler (scale in inches) is for scale. The top specimen broke between days 70 and 71 of the test.

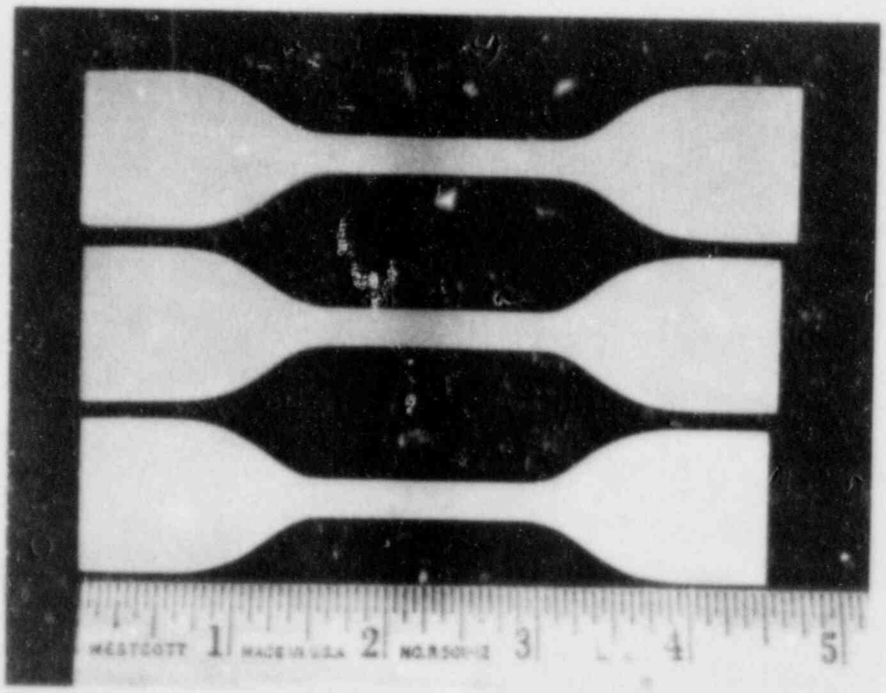

Figure 5.8. Marlex C1-100 tensile specimens from the irradiation under stress experiment in air at 10-11°C. The top specimen was stressed at 1800 psi. The middle specimen was stressed at 1600 psi. The bottom specimen is untested and the ruler (scale in inches) is for scale.

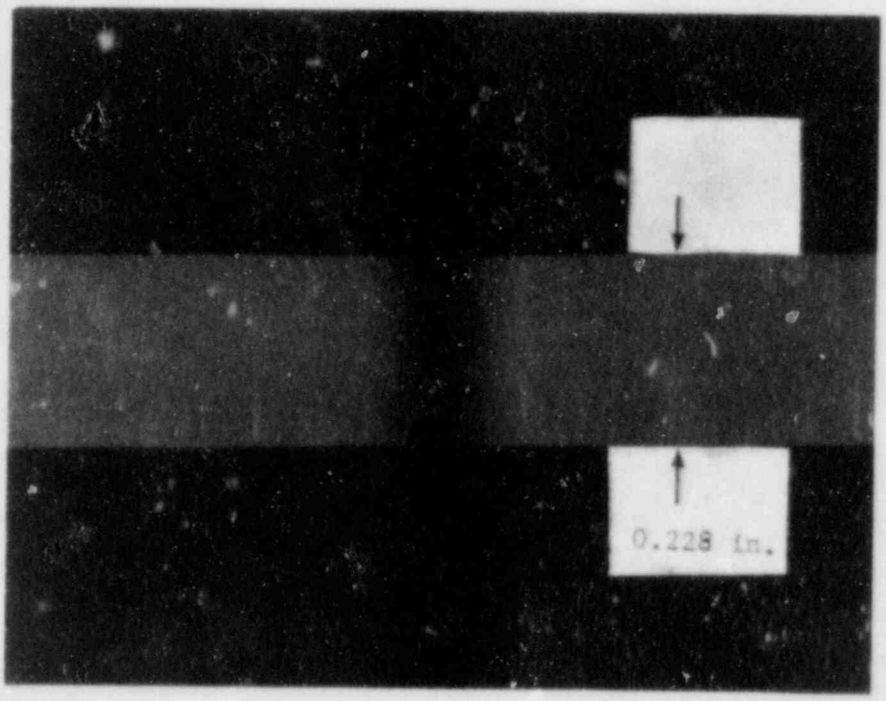

Figure 5.9. Closeup of the narrow section of the 1800 psi Marlex specimen in the irradiation under stress experiment showing the cracks that developed during the test.

Table 5.1

Tensile Test Data on Chemplex 5701 Following Creep Testing

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Creep Load (psi)	Creep (%)	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
11-30	0	----	air	900	2.62	4090	11.5	1190	N
11-30	0	----	air	1600	5.85	3970	10	850	N
9-08	6.8	5.0	air	919	3.00	4600	10	34	B
9-08	6.8	5.0	air	1427	6.92	5080	10	31	B
11-23	8.4	5.0	air	1602	6.00	4280	11	44	B
11-23	8.4	5.0	air	1800	10.7	----*	----*	10.7*	B

*Specimen creep ruptured.

Table 5.2

Tensile Test Data on Rotationally Molded Marlex CL-100 Following Creep Testing

Date Tested (1983)	Dose (Mrad)	Dose Rate (krad/h)	Environment	Creep Load (psi)	Creep (%)	Yield Stress (psi)	Elongation at Yield (%)	Elongation at Break (%)	Neck (N) Break (B)
11-30	0	----	air	900	3.15	3190	17	115	N
11-30	0	----	air	1600	10.5	3280	17	86	N
9-08	6.8	5.0	air	918	----*	3380	15	45	B
9-08	6.8	5.0	air	1506	8.54	3320	12	42	B
11-23	8.4	5.0	air	1613	10.1	3140	15	37	B
11-23	8.4	5.0	air	1795	35.0	3000	22	66	B

*Probable problems with the apparatus resulted in unreliable creep data after two weeks into the experiment.

6. TRANSITION IN FAILURE MODES FOR HIGH DENSITY POLYETHYLENE

During irradiation, there is a transition from behavior characteristic of unirradiated material (i.e., necking behavior) to behavior characterized by surface cracking and breaking without necking. This change makes these materials less tolerant of deformation before failure. When failure mode (i.e., N or B) is plotted as a function of dose vs dose rate, the transition from necking to breaking appears linear on a log-log scale. The relationship obtained from these plots for Chemplex is

$$D_N = 550000\ (R)^{0.32} \qquad (6.1)$$

and for Marlex non-HIC material is

$$D_N = 77000\ (R)^{0.48} \qquad (6.2)$$

where D_N is the dose (rad) up to which necking predominates at a dose rate of R(rad/h). The relationship for the Marlex HIC material is presumed to be the same as that for the non-HIC material in Equation 6.2.

Plots on a log-log scale of dose (Mrad) vs dose rate (krad/h) of the data in Tables 3.1-3.3 are shown in Figures 6.1-6.3, respectively. The points are labelled N or B for neck before break or break without necking. The line corresponding to Equations 6.1 or 6.2 is plotted on the appropriate graph. Each of these figures is discussed in the following paragraphs to indicate how the transition line was determined.

Figure 6.1 - Chemplex: The line specified by equation 6.1 was determined by the two air irradiation points (14 krad/h, 11 Mrad) and (93 krad/h, 20 Mrad). These points were chosen because the test specimens were just on the verge of the transition between necking and breaking without necking. It would have been better to augment these points with data at a lower dose rate. However, none of the irradiations at lower dose rates in air broke as the doses were not large enough to reach the transition.

This line is not an absolute demarcation between N and B behavior since there are three B points below the line. Two of these three points are for irradiations in soil and the other is for a specimen which had undergone creep during irradiation and so may not be strictly comparable to simple irradiations in air. Additionally, since the two points used to determine the line were from only one test specimen each, the possibility that one or both is anomalous is large. If the line specified in Equation 6.1 is incorrect, the error should lie in the exponent of 0.32. It may be less than a value that might be determined with more data over a wider range. The reason for this is suggested from the results for Marlex non-HIC material, discussed below, for which more data are available and which show essentially a square root dependence.

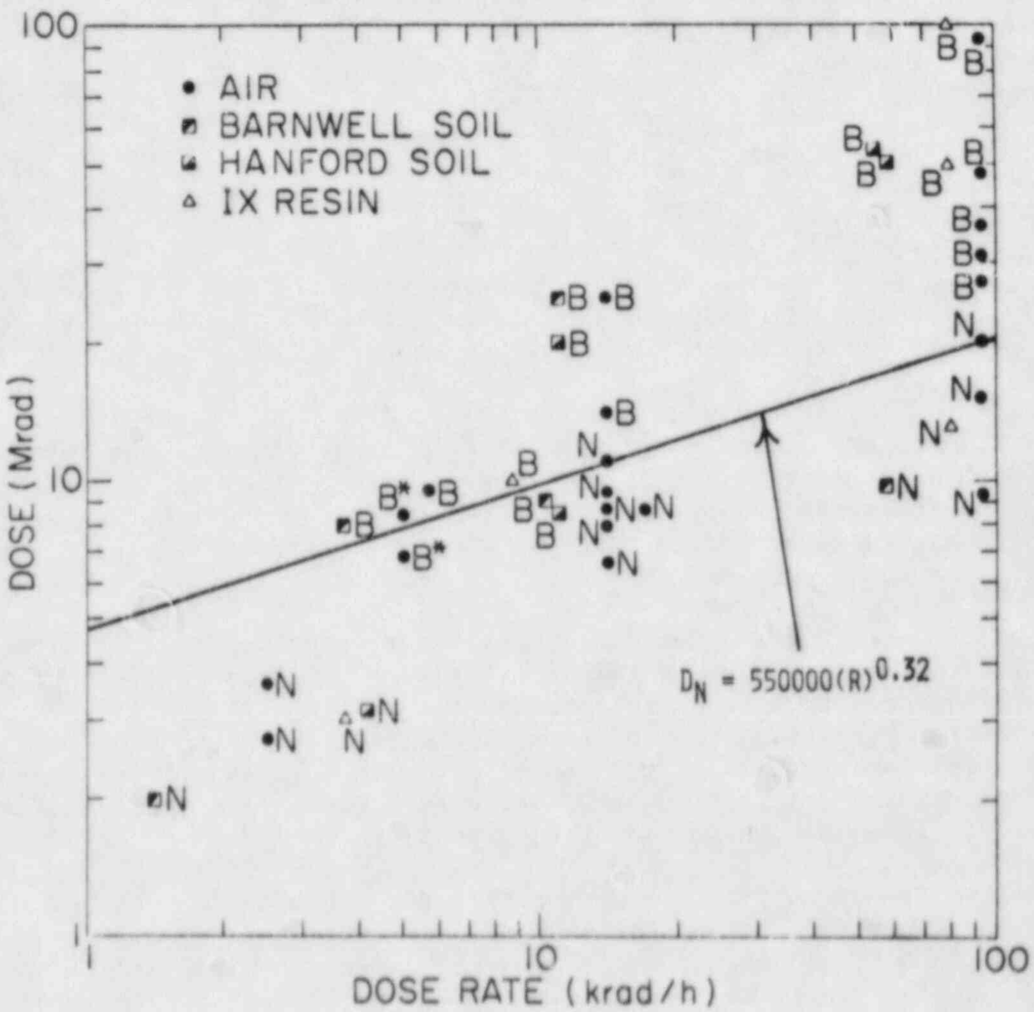

Figure 6.1. Plot on a log-log scale of the data in Table 3.1 for Chemplex 5701 indicating whether the irradiated test specimen necked (N) or broke without necking (B).

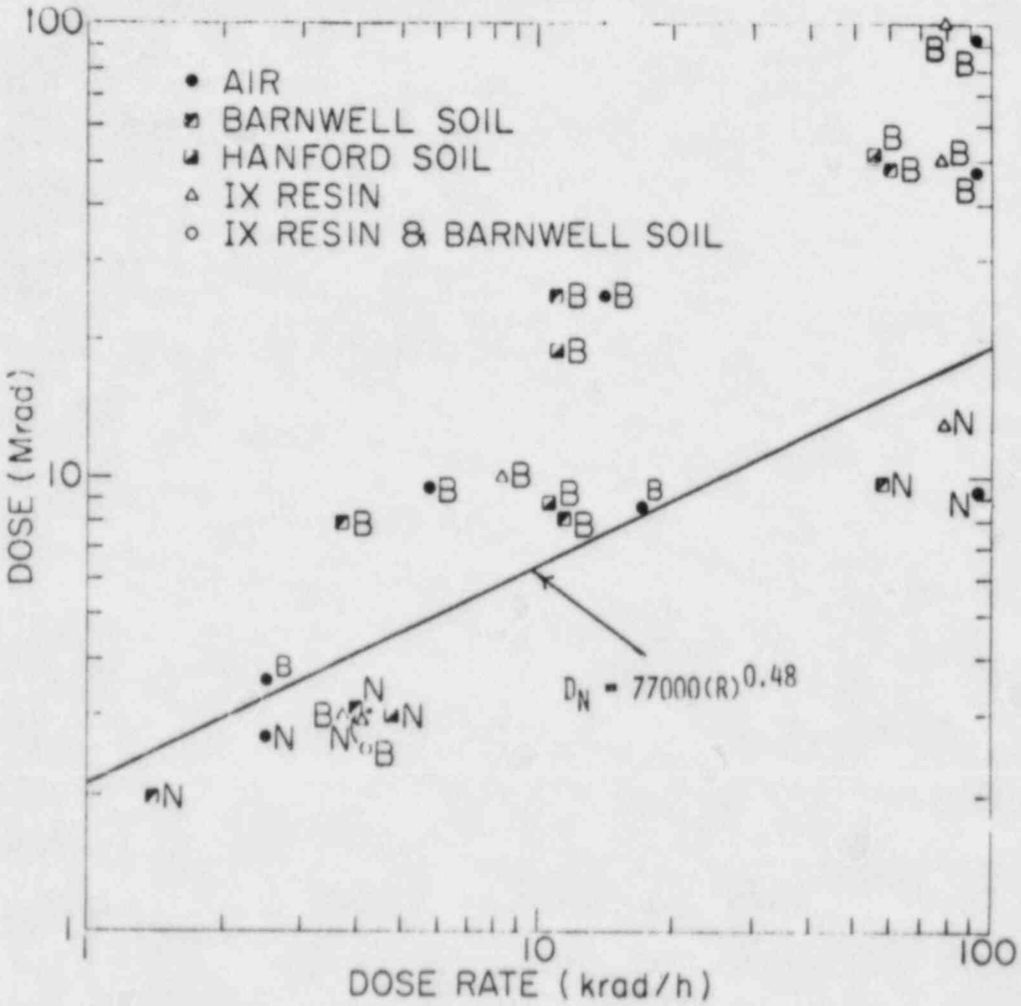

Figure 6.2. Plot on a log-log scale of the data in Table 3.2 for Marlex CL-100 HIC material indicating whether the irradiated test specimen necked (N) or broke without necking (B).

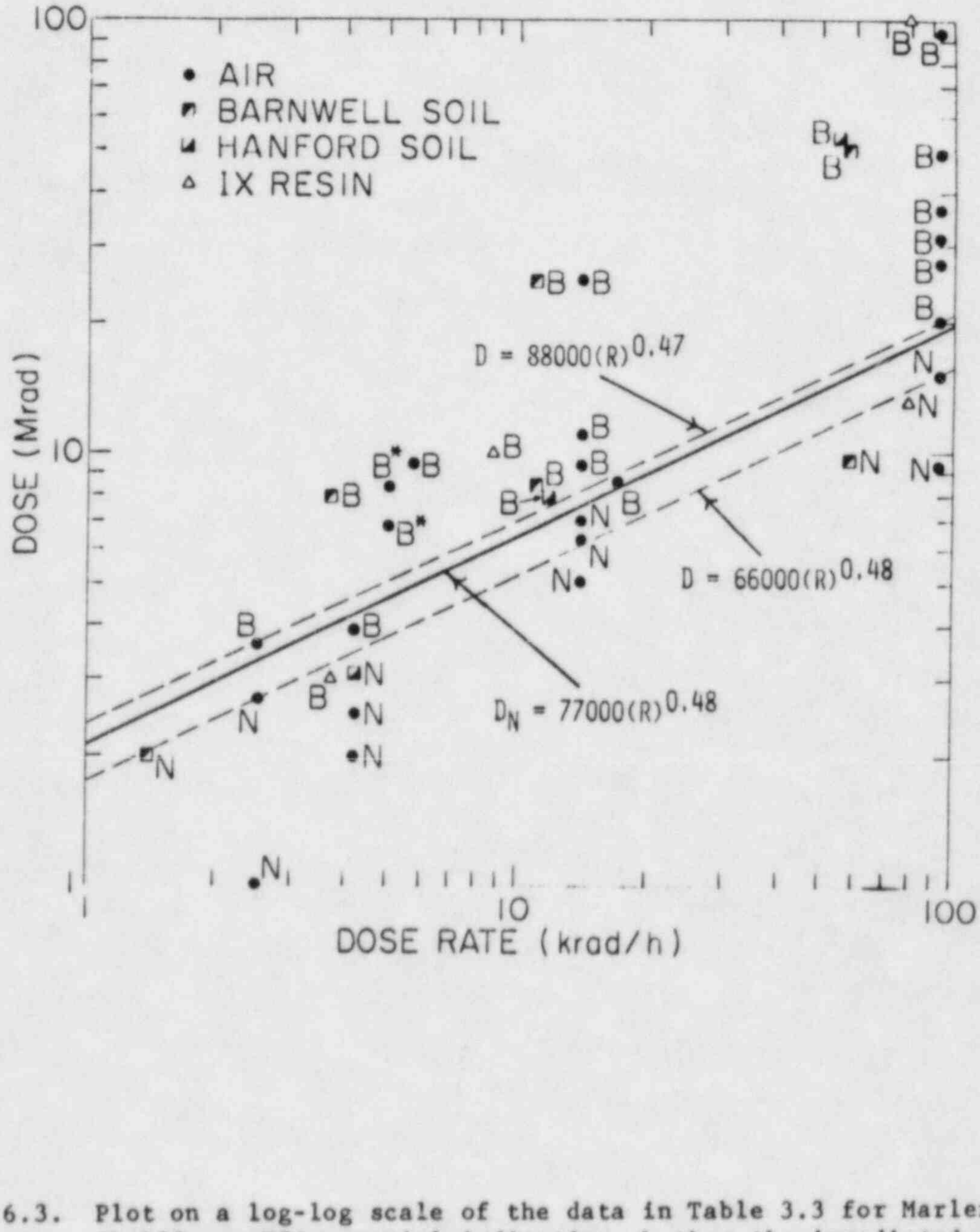

Figure 6.3. Plot on a log-log scale of the data in Table 3.3 for Marlex CL-100 non-HIC material indicating whether the irradiated test specimen necked (N) or broke without necking (B).

Figure 6.2 - Marlex HIC material: The transition line in this plot is presumed to be the same as that determined for the non-HIC material, as discussed below. The transition itself seems valid from the data although there are two B points below the line at the lower dose rates. It is also noteable that data for two specimens irradiated in IX resin at essentially the same dose rate show one sample breaking and the other necking (see the last two entries in the IX resin section in Table 3.2).

Figure 6.3 - Marlex non-HIC material: The transition region appears well defined from these data. Since so much data was available for this material three lines were determined. The lower dashed line is defined by the N points (2.5 krad/h. 2.7 Mrad) and (93 krad/h, 15 Mrad). Below this line necking occurs in all but one case (not in an air irradiation) for the rest of the data. The upper dashed line is defined by the B points (2.5 krad/h, 3.6 Mrad) and (93 krad/h, 20 Mrad). Above this line every test specimen broke without necking. The parameters in equation 6.2 are the arithmetic average of these two boundary lines. (The solid line corresponding to equation 6.2 is not centered between the two dashed lines on the plot because of the variable spacing between integer numbers on a log scale.) The dashed boundary lines appear to provide a good estimate of the transition region in which either N or B behavior may occur.

Comparing the data in Figures 6.2 and 6.3 it seems that there is no significant variation (where corresponding data points are available) between the data for the non-HIC material and the much thicker HIC material. Thus it appears that the thickness of the Marlex CL-100 may not be a factor in the applicability of Equation 6.2 for predicting the behavior of irradiated Marlex CL-100 material. However, it seems likely that the temperature during irradiation may affect these transition models. If the effect of temperature is "simple" it would only change the pre-multipliers and not the exponents in Equations 6.1 and 6.2. However, since our experiments were performed at only one temperature, we do not know what effect, if any, irradiation temperature might have on these models.

Table 6.1 presents estimates of D_N (i.e., the dose below which necking will predominate in irradiated material) and the time to reach D_N for several dose rates using these equations. The values of D_N and the time to D_N at the lower three dose rates in Table 6.1 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, as described below.

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 are probably conservatively high since they neglect container geometry and self-shielding by the resin wastes. Using these dose rates as a benchmark for expected field conditions, however, leads one to conclude that the transition could occur within 2 months to a year. While there are uncertainties associated with this estimate, it would appear that the consequences of such embrittlement during storage should be considered in the design of HICs made from HDPE. In any case, the transition appears to be dose rate dependent and occurs at lower total doses for lower dose rates.

Table 6.1

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[a]	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[b]	2000	6.3	130
Chemplex 5701	1000	5.0	209
Chemplex 5701	500	4.0	335
Chemplex 5701	100	2.4	1000

[a]Calculated from $D_N = 77000\ (R)^{0.48}$.
[b]Calculated from $D_N = 550000\ (R)^{0.32}$.

The information implicit in the model depends on knowing, or being able to bound at the upper limit, the dose rate. For radwaste in a HDPE HIC, this particularly means being able to estimate the beta dose to the inner wall of the HIC, as well as measuring the gamma dose rate at the outer wall. Failure to take account of beta activity could result in embrittlement before anticipated from the model equation.

7. CONCLUSIONS

The results of mechanical testing of HDPE following irradiation at 10-11°C have shown that these irradiations resulted in no loss of strength in these materials. However, it caused these materials to become less tolerant of deformation. We did not observe effects attributable solely to, or dominated by, radiation-induced oxidation from the irradiations at 10-11°C (see also the following paragraph). Specifically, there was no decrease in tensile strength. The tensile results for irradiation at 10-11°C were con sistent with radiation-induced cross-linking in polyethylene. It was not necessary to assume radiation-induced oxidation to explain these results.[11,26,27] This suggests that degradation due to radiation-induced oxidation at 10-11°C may be a very slow process.

Surface cracking accompanied (and may have caused) the transition from necking to breaking without necking. This cracking appeared to result from an embrittlement of the surface - particularly the glossy surface of the Marlex materials. The obvious possibilities are that radiation alone causes the surface embrittlement, that radiation-induced oxidation caused it or that both contributed to the embrittlement. Irradiation in soils and particularly the IX resin showed surface embrittlement just as did specimens irradiated in air. Since soils and the IX resins presumably protect the specimens from oxidation during irradiation, it seems unlikely that the presence of air during irradiation is required for the embrittlement to occur. These experiments indicate that, for the real environments HICs will experience, irradiation would cause surface embrittlement whether air is present or not.

Few effects of the different irradiation environments (air, soils and IX resins) in modifying the changes in characteristics produced by irradiation were apparent. The result that irradiation in the soils and IX resin tended to retard some of the degradative effects caused by irradiation in air seems to be the most important environmental effect. Although the evidence for this from this work is more circumstantial than undeniable (see the results for yield stress vs dose, Figures 3.9, 3.12 and 3.15 plus the accompanying discussion), it is certainly reasonable. Thus a buried HIC should have a longer service life than one exposed directly to air. However, the data presented here provide no evidence as to how much longer the buried HIC might last.

Irradiation in air at 60-63°C 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 embrittlement of the surface which progressed into the bulk material as the irradiation in air continued. This was the only result attributable with certainty to radiation-induced oxidation in this work. This, combined with the results for irradiation at 10-11°C plus results cited in the literature for irradiations in inert atmosphere, suggest that radiation-induced oxidation may enhance or speed up the decrease in yield elongation and break elongation that result from cross-linking, but does not cause these changes. The important point for HICs appears to be that an irradiated HIC will lose much of its ability to tolerate deformation at some radiation dose, which depends on dose rate (see below), long before any loss in strength becomes apparent.

A transition in failure mode, from necking-to-breaking was observed as a result of irradiation. For the Marlex material studied, the dose at which this transition occurs is proportional to approximately the square root of the dose rate measured over the dose rate range of 1400 rad/h to 93,000 rad/h. Estimates of the time to this transition were made for both materials studied for containers in which the dose rate varied from 100 to 2000 rad/h. Under these conditions, the time to transition varied from 292 to 63 days for Marlex CL-100 and 1000 to 130 days for Chemplex 5701. The total absorbed dose at which the transition would occur varied from 0.7 to 3.0 Mrad and 2.4 to 6.3 Mrad for the two materials, respectively.

It was felt that these transition times and doses could possibly be achieved by HICs containing highly loaded radwaste under conditions of storage. Hence, the effects of such potential embrittlement should be addressed in the design of a HIC. Since the effects of the embrittlement on a given container are design-dependent, it is not possible to set generic guidelines for these considerations. However, this concern may be addressed by a vendor of a HIC in a number of ways:

1. Loading of the container could be restricted such that embrittlement does not occur within 300 years. This dose may be determined from a knowledge of container geometry and the radioisotopes being disposed of. The threshold doses indicated above, however, indicate that the loading limits thus calculated may be low and, hence, not practical.

2. If the design and loading of the HIC is such that embrittlement could occur, then the effects of such embrittlement should be expressly considered by the vendor in the design analysis to determine that the container can withstand the stresses imposed during loading, storage, transportation, and burial conditions. Credible accident scenarios should also be considered in this analysis. The maximum allowable stresses are necessarily design specific.

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16. ABSTRACT (200 words or less)

Mechanical tests following gamma irradiation and creep tests during irradiation have been conducted on high-density polyethylene (HDPE) to 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 testing on these materials following irradiation at 10-11°C showed no effects directly or solely attributable to radiation-induced oxidation. However, effects due to radiation-induced cross-linking, including an increase in yield strength and decreases in both elongation at yield and elongation at break, were observed. Irradiation at 60-63°C showed effects or radiation-induced oxidation including a decrease in yield strength. These effects were more marked in thinner test specimens. Creep testing during irradiation indicated that irradiation increases the creep rate but that the effect is really only significant at creep loads greater than about half the nominal yield strength under the conditions of these tests (10-11°C and 5 krad/h).

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