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THE EFFECT OF NEUTRON IRRADIATION  
ON THE MECHANICAL PROPERTIES OF WELDED ZIRCALOY-2

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by

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Chalk River, Ontario

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THE EFFECT OF NEUTRON IRRADIATION  
ON THE MECHANICAL PROPERTIES OF WELDED ZIRCALOY-2

by

D. G. Evans

SYNOPSIS

Zircaloy-2 tensile specimens, subsize impact bars and representative spigot welds were subjected to three NRX cycles in the X-5 loop. Average loop temperature was 260°C over the three cycles. One group of tensile specimens was heat-treated in vacuum at 900° C for 40 minutes, another group contained welded areas in the centre of the gauge length and a third group was hydrided after welding. Notches of the impact specimens were located in the fusion zone of the weld. Spigot welds were made on autoclaved and unautoclaved simulated production assemblies.

The transition temperature of Zircaloy-2 increased appreciably upon welding. This was accompanied by a decrease in absorbed energy values for all temperatures between 0° and 300°C. Neutron irradiation had no effect on the impact properties of welded Zircaloy-2.

Welding decreased the uniform and total elongation at room temperature and at 260°C, and increased the 260°C PL, YS and UTS. Hydriding to a nominal 100 ppm hydrogen had no effect on the unirradiated tensile properties at either test temperature. The heat treatment decreased the strength properties but did not affect the ductility. Neutron irradiation increased the YS of the welded and hydrided material by 20% and the heat treated YS by 40%. Irradiation also increased the 260°C strength properties of the as-welded material.



It was found that the unautoclaved spigot welds had a generally higher tensile strength than the autoclaved and welded specimens. For specimens welded in either condition, the outer welds of the 19-element bundle had a lower average breaking load than the inner welds. Neutron irradiation had no effect on the tensile strength of these welds. It was also demonstrated that a cup-and-cone type of fracture could be produced in a bend test. These fractures were similar to those observed in irradiated fuel bundles which had been damaged during transfer operations.

A large amount of scatter rendered some results inconclusive. This was especially the case for the room-temperature as-welded results and for the majority of the total and uniform elongation measurements.

Chalk River, Ontario  
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TABLE OF CONTENTS

## SYNOPSIS

1. INTRODUCTION
2. SUMMARY OF PREVIOUS WORK
3. EXPERIMENTAL
  - 3.1 Materials
  - 3.2 Welding
    - 3.2.1 End Plug Assemblies
    - 3.2.2 Zircaloy-2 Plate
  - 3.3 Specimen Types
    - 3.3.1 End Plug Assemblies
    - 3.3.2 Tensile Specimens
    - 3.3.3 Impact Bars
  - 3.4 Irradiation Conditions
4. RESULTS
  - 4.1 Mechanical Testing
    - 4.1.1 Tensile Specimens
      - 4.1.1.1 Room temperature, unirradiated properties
      - 4.1.1.2 Elevated temperature, unirradiated properties
      - 4.1.1.3 Irradiated, room temperature properties
      - 4.1.1.4 Irradiated, elevated temperature properties
    - 4.1.2 Spigot Welds
      - 4.1.2.1 Tensile tests
      - 4.1.2.2 Bend tests
      - 4.1.2.3 Torsion tests
    - 4.1.3 Impact Tests
  - 4.2 Evaluation of the Mechanical Test Results
    - 4.2.1 Tensile Specimens
    - 4.2.2 Spigot Welds
    - 4.2.3 Torsion Tests
  - 4.3 Hydrogen Analyses
  - 4.4 Metallographic Examination
    - 4.4.1 Tensile Specimens
    - 4.4.2 Spigot Welds
      - 4.4.2.1 Unirradiated specimens
      - 4.4.2.2 Irradiated specimens
      - 4.4.2.3 General

TABLE OF CONTENTS (cont'd)

- 5. DISCUSSION
  - 5.1 Mechanical Properties
    - 5.1.1 Tensile Specimens
      - 5.1.1.1 The effect of welding
      - 5.1.1.2 The effect of heat treating
      - 5.1.1.3 Effect of irradiation
    - 5.1.2 Impact Tests
    - 5.1.3 Spigot Welds
      - 5.1.3.1 Tensile tests
      - 5.1.3.2 Bend tests
  - 5.2 Recommendations for Further Work
- 6. SUMMARY
- ACKNOWLEDGEMENTS
- REFERENCES
- TABLES
- FIGURES

## 1. INTRODUCTION

The fuel bundle string which was used for the E-20 Mixing and Boiling Experiment<sup>(1)</sup> was damaged during post-irradiation handling<sup>(2)</sup>. All the fuel elements of the 19-element fuel bundles had separated from the end plates at the end plug spigots. The fracture path was through the weld-affected zone (Figure 1). All the components were Zircaloy-2.

Mechanical testing of specimens taken from the end plates showed a moderate decrease in impact strength and a corresponding increase in tensile strength<sup>(3,4)</sup> of the Zircaloy. The amount of hydrogen pickup in the sheaths and the end plates was unexpectedly high<sup>(4)</sup>. The conclusion was reached, however, that the type of failure was more a characteristic of impact loading, temperature, and the geometry and microstructure of the weld than a result of an embrittling environment<sup>(2)</sup>.

As a direct result of the E-20 failure it was apparent that the effect of neutron irradiation on the mechanical properties of welded Zircaloy-2 should be investigated. To this end, instron tensile specimens and subsize impact bars, both containing welded areas, and a number of representative end plug to end plate (spigot) welds were irradiated in the X-5 loop of the NRX reactor<sup>(5)</sup>. Mechanical testing, metallographic examination and hydrogen analyses were used to assess the effect of irradiation and of the high-temperature, high-pressure water environment on the weldments.

## 2. SUMMARY OF PREVIOUS WORK

There have been a large number of investigations conducted on welding techniques for Zircaloy-2<sup>(6)</sup> but relatively few solely concerned with the mechanical properties of the weld-deposited metal. No information on the effect of irradiation on the properties of welded Zircaloy-2 was found in the literature.

Grozier and Rubenstein<sup>(7)</sup> statistically analyzed the results of room temperature, 300°F (149°C) and 600°F (316°C) tensile tests on Zircaloy-2 specimens from PWR welded subassemblies. The results of this investigation showed that:



- (a)  $\alpha$ -phase annealing (1 hour and 8 hours in vacuum at 800°C) did not significantly alter the mechanical properties of hot-rolled or welded Zircaloy-2;
- (b) welding significantly increased the 0.2% offset yield strength (YS) and decreased the total elongation. The magnitude of this effect decreased with increasing testing temperature. The ultimate tensile strength (UTS) and the reduction in area were not significantly affected by welding.

However, in another investigation<sup>(8)</sup> Grozier and Rubenstein found that the UTS of weld metal was higher than that for the base metal. By taking specimens at various intervals from the centre line of a weld, they were able to show that the UTS and the YS, with one exception, increased from base metal through the heat affected zone to the weld metal. This effect did not appear to be very significant when a testing temperature of 600°F (316°C) was used.

Mock<sup>(9)</sup> also found that the UTS and the YS were higher for the weld metal than for the wrought material. The effect again was greater for the YS than for the UTS, and again the magnitude of the effect of welding decreased as the testing temperature increased.

Beitscher<sup>(10)</sup>, in a more extensive examination of the properties of Zircaloy-2 weld metal, reached the following conclusions:

- (a) The differences between the properties of fine-grained and welded Zircaloy-2 were small.
- (b) The weld metal had higher YS, lower ductility and about the same UTS as wrought material at both room temperature and at 600°F (316°C). However, the differences in properties were considerably less at the higher testing temperature.
- (c) The ductility decreased more rapidly with increasing hydrogen content for the weld metal than for the wrought material.
- (d) Strength properties were unaffected by  $\alpha$ -annealing (770°C for 1/2 h and furnace cooled); the ductility, however, increased about 50%.
- (e) Longitudinal specimens - those composed entirely of weld metal - had the same tensile properties as transverse specimens - those machined perpendicularly to the welding direction and therefore

containing a welded area only in the centre of the specimen. This implied that, for the latter, either the fractures all occurred in the weld metal, or the strength properties of the weld metal and of the heat affected zone were the same.

The impact properties of Zircaloy-2 weld metal were partially determined by Rubenstein(11). Only three temperatures were used: room temperature, 300°F (149°C) and 600°F (316°C). The energy absorbed increased almost linearly with increasing temperature. A slight overall increase in energy absorbed accompanied vacuum anneals of 1 and 7 hours at 800°C; it appeared that the longer the anneal, the greater the increase. The results quoted here refer to notches perpendicular to the welding direction.

Further insight into the probable mechanical properties of Zircaloy-2 weld metal can be obtained from the effects of heat treatment on the strength properties of wrought Zircaloy-2. Goodwin et al(12) studied these effects and found that:

- (a) Alpha heat-treatment had no effect on as-received mechanical properties.
- (b) There was essentially no change in the YS and the UTS values for heat-treating temperatures in the  $\alpha + \beta$  range (1500 to 1800°F) (800 to 980°C). However, the ductility (measured as either elongation or reduction in area) decreased with increasing temperature of anneal.
- (c) Annealing in the  $\beta$  range (above 1850°F : 1010°C) increased the YS, decreased the UTS and decreased the ductility with increasing temperature of anneal.
- (d) The rate of cooling had a pronounced effect on the mechanical properties:
  - i) Tensile strengths were greater for water quenched than for furnace cooled specimens for annealing temperatures in the  $\beta$ -range. This partially verified some earlier results(13).
  - ii) Furnace cooled specimens showed higher YS than quenched specimens in the range 1650° to 1750°F (899° to 954°C). Otherwise there was no real difference in YS for different cooling rates.

- (e) The time at temperature also appeared to be significant, having no effect on the YS or total elongation but decreasing both the reduction in area for all annealing temperatures above 1450°F and the tensile strength for annealing temperatures above 1700°F as time at temperature increased. Times of 1/2 h and 96 h were used.
- (f) Attempts to restore the ductility of the  $\beta$ -quenched specimens by  $\alpha$ -annealing at 1450°F for up to 8 h were unsuccessful.

In summary, therefore, the following conclusions have been verified by the majority of the investigations:

- (a) Welding Zircaloy-2 decreases the ductility(7, 10, 14) and increases the yield strength(7, 8, 9, 10, 14).
- (b) Alpha annealing of the weld metal does not affect the strength(7, 10).
- (c) The higher the tensile test temperature the smaller the difference between the welded and wrought metal mechanical properties(7, 8, 9, 10).

The results of the annealing experiments of Goodwin et al<sup>(12)</sup> predict the first two conclusions.

There is some disagreement between investigators on the following questions:

- (a) whether the UTS increases(8, 9, 14) or remains essentially constant (7, 10) upon welding;
- (b) whether the strength of the fusion zone of the weld is higher than (8, 9, 14) or the same as(10) the strength of the heat affected zone;
- (c) whether the ductility is unchanged(7) or increased(10, 11) upon  $\alpha$ -annealing the weldments.

The annealing studies<sup>(12)</sup> favour a slight decrease in the UTS when quenching from above 1750° - 1800°F. No change in ductility was experienced on  $\alpha$ -annealing " $\beta$ -quenched" specimens.

Differences in the welding technique used by the various investigators could account for the differences in results. Welding variables such as arc voltage, amperage, welding rate and number of passes would

control the cooling rate of the welded area and thus affect the mechanical properties. In addition, some investigators make no mention of compositional differences, if any, between base plate and filler rod. Such differences could affect the relative strengths of the fusion zone and unwelded material.

### 3. EXPERIMENTAL

#### 3.1 Materials

The filler rod and base plate analyses are given in Table 1. No differences in mechanical properties due to the small differences in composition were expected. Both the plate and the rod were in the annealed condition.

Two Zircaloy-2 end plug/end plate simulated production assemblies supplied by Canadian General Electric were used in this experiment. The components of one assembly were autoclaved 24 hours at 750°F in 800 lb/in<sup>2</sup> gauge steam prior to welding; the other assembly was welded in the as-pickled condition(15).

#### 3.2 Welding

##### 3.2.1 End Plug Assemblies(2, 15)

The end plugs were fusion welded to the end plate (Figure 2). Normally, the sheaths are first welded to the plugs followed by the spigot weld. In this case, however, the plugs were not attached to the fuel sheaths; the two plug-to-plate units were assembled using a jig consisting of 19 empty tubes. Close plate-to-plug contact was maintained by placing the longest tubes in the centre of the bundle and applying pressure to the periphery of a copper chill covering the end plate. The weld current was 90 amperes (low side of tolerance). Production procedures were followed as closely as possible.

Four units were made. Two were sent to Chalk River and two were torsion tested at Canadian General Electric.



### 3.2.2 Zircaloy-2 Plate

Unfortunately, no detailed record was kept of the welding conditions or sequence. Arc-welding was used in an inert-gas chamber. Figure 3 gives the details of the welding geometry and how the specimens were machined from the welded plate.

Each weld required two passes. The first pass served to fuse the sides of the notch. The filler wire was used in the second pass.

### 3.3 Specimen Types

#### 3.3.1 End Plug Assemblies

These assemblies were cut up as shown in Figure 4 to produce the two specimen types sketched in Figure 5. The stress conditions used to test these specimens to destruction in tension and in bending are also depicted in Figure 5. Figures 6 and 7 are photographs of the equipment used to produce the two stresses. An Instron Tensile Machine was used; all tests were done at room temperature.

#### 3.3.2 Tensile Specimens

All the tensile specimens (Figure 8) were machined from the same plate and were tested in the following conditions:

- (a) as-received;
- (b) as-received and heat-treated in vacuum at 900°C for 40 minutes followed by an oil-quench. This treatment was intended to produce a  $\beta$ -quenched structure. However, metallographic examination revealed that the temperature had not been high enough to produce this structure<sup>(16)</sup>.
- (c) containing a welded area in the centre of the gauge length;
- (d) as (c) with 100 ppm hydrogen added.

These specimens were pulled at room temperature and at 260°C using an Instron Tensile Machine.

#### 3.3.3 Impact Bars

Each subsized impact bar (Figure 8) had notches alternately in

welded and unwelded areas. In addition, one impact bar of as-received material was tested. Temperatures from 0° to 300°C were used to determine the transition curves.

A flowsheet for the specimens from the welded plate is shown in Figure 9.

### 3.4 Irradiation Conditions

A number of specimens from each of the above groups were irradiated in the X-5 loop of the NRX reactor for three reactor cycles. The specimens were contained in a stainless steel bundle (DHX) described elsewhere<sup>(5, 17)</sup>. A comparison of the conditions in the E-20 irradiation with those which prevailed in the X-5 loop is made in Table 2.

Apart from the number of shutdowns and consequent thermal cycling experienced by the DHX bundle, the X-5 irradiation was a fair approximation of the E-20 Mixing and Boiling test in that the two more important items, the total integrated flux and the average temperature were reasonably near the mixing and boiling test values.

Brief surface boiling on the fuel sheath probably occurred during the second cycle of the NRX irradiation<sup>(18)</sup>. Boiling also occurred in the E-20 loop around the upper (outlet) bundle during the mixing and boiling test<sup>(2)</sup>.

After each NRX cycle the bundle was allowed to cool from about 500°F (260°C) to about 125°F (52°C) over a period of 2-1/2 hours. The bundle was removed after remaining approximately 30 hours in the loop (probably at 125°F) to permit the activity to die down. After removal, the bundle was stored under water.

## 4. RESULTS

### 4.1 Mechanical Testing

#### 4.1.1 Tensile Specimens

The results of the mechanical testing of the tensile specimens are shown in Table 3. Each value is an average of two tests with the

exception of the welded and unirradiated specimens. These values are an average of three determinations.

#### 4.1.1.1 Room Temperature, Unirradiated Properties

The only readily apparent effects of welding were the decreases in the UTS and the total elongation. To a much lesser extent, welding decreased the YS and the PL from as-received values.

Hydriding the weld metal appeared to increase the YS and the PL from the as-welded quantities to above the as-received values. The UTS of as-welded specimens also increased, but remained somewhat lower than that for the as-received material. The total elongation was unchanged while the uniform elongation decreased.

Strength values decreased and the elongation (total and uniform) remained the same upon heat treating.

#### 4.1.1.2 Elevated Temperature, Unirradiated Properties

Tensile testing at 260°C produced results which conflicted somewhat with room temperature results. The mechanical properties of the welded and welded-and-hydrided specimens were identical. The strengths in these two conditions were greater than for the unwelded material and the total elongation appreciably lower. Heat treated specimens had a higher UTS and a lower elongation than the as-received material.

#### 4.1.1.3 Irradiated, Room Temperature Properties

Neutron irradiation had no effect on the as-welded properties. The strength of welded-and-hydrided specimens, however, increased appreciably and the ductility decreased slightly. Irradiation produced about a 90% greater increase in the PL and the YS of the heat-treated specimens compared to that for the welded-and-hydrided specimens, but the same small decrease in the elongation.

#### 4.1.1.4 Irradiated, Elevated Temperature Properties

Again the as-welded, and welded-and-hydrided strength values at the 260°C testing temperature were identical. This meant that both conditions responded equally to irradiation (in contrast to the results in 4.1.1.3 above). The heat-treated strengths increased by about the same amount as did the specimens in the other two conditions.

In all cases there was an appreciable amount ( $> 7\%$ ) of uniform elongation. That irradiation appeared to have little effect on this property was surprising. Previous results (19, 20) had shown that 4 - 7% could be expected at room temperature and about 2% at 280°C for irradiated Zircaloy-2.

#### 4.1.2 Spigot Welds

These results are presented in Table 4. A good deal of scatter characterized both methods of testing. Because of the inconsistency in the type of failure in the bend tests, no real comparisons of the strengths of the various conditions could be made. However, some tentative observations were made by considering only the specimens which had failed in a manner similar to a typical E-20 failure (Figure 1). This mode of failure will hereafter be referred to as a cup-and-cone fracture.

##### 4.1.2.1 Tensile Tests

The averages of the tensile results indicated no appreciable effect of irradiation on the weld strength:

	Breaking Load (lb)		
	<u>Irradiated</u>	<u>Unirradiated</u>	<u>Range</u>
Autoclaved	448	450	325 - 545
Unautoclaved	583	597	520 - 654

Assuming no irradiation effect, a further observation may be made: the outer welds (1 to 12 inclusive) were weaker than the inner welds (13 to 19 inclusive). This is shown in the following table:

	Ave breaking load (lb)		Ranges (lb)		No. of Tests
	<u>Autocl'd</u>	<u>Unautocl'd</u>	<u>Autocl'd</u>	<u>Unautocl'd</u>	
Outer welds	402	538	325-510	520-550	3
Inner welds	470	612	420-545	512-654	7

It was apparent too, that the unautoclaved specimens had higher average breaking loads than the autoclaved specimens. This result was contrary to the predicted behaviour<sup>(15)</sup>.

All specimens failed by the spigot pulling out of the end plate. No gross deformation of the end plate occurred, although plastic flow was evident in the metal immediately adjacent to the fracture path.



#### 4.1.2.2 Bend Tests

Perhaps the most important result of these tests was the increased incidence of the cup-and-cone failure in the irradiated specimens. There were 6 such failures in 9 specimens in the irradiated condition and only 3 out of 8 in the unirradiated condition. Of these, 6 out of 9 in the autoclaved condition and 3 out of 7 in the unautoclaved condition failed in this manner.

Although the strengths varied widely, the irradiated unautoclaved specimens had a higher average breaking load than the irradiated autoclaved specimens. This was in agreement with the tensile test results. No inner welds were bend tested.

#### 4.1.2.3 Torsion Tests<sup>(15)</sup>

Torsion tests done on duplicate assemblies showed that the autoclaved specimens had slightly higher average strengths than unautoclaved:

	Failure Torque in-lb	No. of Tests
Autoclaved	33.5	19
Unautoclaved	31.9	19

Some unautoclaved specimens did not break on the spigot major diameter.

#### 4.1.3 Impact Tests

These results are tabulated in Table 5, and plotted in Figure 10. The impact values for the alternate notches in unwelded material were not included in Figure 10 because the scatter was so large that the values are without significance. In fact, depending on the extent of the two adjacent weld-affected zones, the unwelded notch could lie in the base metal or in the heat-affected zone.

Figure 10 illustrates the effect of welding on Zircaloy-2 impact properties. Welding lowered the energy absorbed at all testing temperatures and raised the transition temperature.

Neutron irradiation did not appreciably affect the impact properties of the weld metal.

#### 4.2 Evaluation of the Mechanical Test Results

The scatter in the mechanical test results was appreciable, especially for the welded tensile specimens and for the spigot welds in both tension and bending. Since some of the changes noted were small and the number of determinations limited, a statistical evaluation of the averages was undertaken. The t-test(21) was applied to these averages; these calculations are tabulated in Table 6.

##### 4.2.1 Tensile Specimens

The following trends were found to be not significant:

- (a) the decrease in the room temperature PL, YS and UTS due to welding of the base plate;
- (b) the increase in the room temperature PL, YS and UTS due to hydriding the weld metal;
- (c) the increase in the 260°C PL, YS and UTS and the decrease in the total elongation due to heat treating the as-received material.

Of borderline significance were:

- (a) the decrease of the room temperature UTS upon heat treating the as-received material;
- (b) the increase in the welded, and welded-and-hydrided 260°C UTS upon irradiation.

All other trends were found to be significant. Previous results on the room temperature tensile properties of the base metal(20) were also used in this evaluation.

##### 4.2.2 Spigot Welds

No significance was found for the difference between the average tensile breaking loads of the autoclaved outer and inner welds or between the autoclaved and unautoclaved outer welds. However, this may have been due to one high value of one autoclaved outer weld. No evaluation of the bend test strengths was attempted.

#### 4.2.3 Torsion Tests

The higher average torsion strength of the autoclaved spigot welds was statistically significant.

#### 4.3 Hydrogen Analyses

A number of hydrogen analyses were done on active and inactive broken tensile specimens and spigot welds (Table 7). The analyses of the inactive heat treated and as-received specimens agreed very well with the manufacturer's analysis. There was no hydrogen pickup from the welding rod.

The hydrogen analyses on the whole, however, were erratic. Some specimens indicated increased and others decreased hydrogen contents after irradiation. There was also a wide variation between specimens in the same condition. Therefore, it could not be ascertained by chemical analysis alone whether there had been any hydrogen pickup during irradiation. However, metallography indicated no gross hydrogen pickup from the irradiation environment.

#### 4.4 Metallographic Examination

##### 4.4.1 Tensile Specimens

Metallographic examination of a number of broken tensile weld specimens was undertaken in an attempt to explain the deviation of some of the present results from those of previous investigations. Fracture faces were inspected for weld porosity and blowholes. Microsections parallel to the stress axis were used to determine the relative hydride contents, distribution and orientation of the platelets, and the extent of the total weld-affected and fusion zones, and whether any stress raising factors were present.

Examination of the fracture faces of irradiated and unirradiated specimens revealed no blowholes in any of the specimens.

The boundaries of the fusion zone could not be resolved. The transition from the weld metal through the heat affected zone to the base material was very gradual. For some specimens the total weld-affected area extended over at least the gauge length and in many cases over the

full length of the tensile specimen. Therefore, a specimen which fractured close to the centre of the gauge length would probably have failed in the fusion zone.

Figure 11 shows the positions of the fractures of both the active and the inactive specimens corrected for the total elongation. The shaded area is the maximum width of the V-notch; the cross-hatched area is the V-notch width at half the plate thickness. Most of the fractures occurred within the shaded area. Those specimens which produced failures outside this area had essentially the same properties as those which failed within the estimated fusion zone. Average tensile properties were therefore calculated assuming no difference in properties between the heat-affected and fusion zones.

The unhydrided specimens exhibited very few hydride platelets. Hydrided specimens showed numerous hydride platelets uniformly distributed throughout the Zircaloy-2 matrix. The platelets showed a slight tendency to be oriented as illustrated in Figure 12.

#### 4.4.2 Spigot Welds

##### 4.4.2.1 Unirradiated Specimens

Of the seven specimens tested in bending, two failed by the spigot pulling out of the end plate, three by the cup-and-cone fracture and two broke across the spigot. Figures 13 and 14 show a typical cup-and-cone failure and a spigot failure respectively. Comparison of Figures 1 and 13 indicated that the stress conditions used in the bend tests were similar to those which caused the E-20 failures.

##### 4.4.2.2 Irradiated Specimens

In an attempt to explain the wide variation in the breaking loads of the bend specimens, the end plate portion of spigot welds 1 and 10 (unautoclaved), and 6 (autoclaved) were sectioned through the fractures. Such factors as hydride concentration and possible stress-raisers were examined.

There was no evidence of any hydride concentrations in any of the fractures. In fact, the hydride content was difficult to discern microscopically in any of the specimens. Moreover, there were no noticeable differences in the fractures or fracture paths of the low strength



specimens<sup>(16)</sup>. However, specimen No. 1 (unautoclaved) fractured partly across the spigot (Figure 15) while the specimens 6 and 10 produced smooth cup-and-cone fractures (Figure 16). The two types of failure exhibited in these specimens were either a product of variable welding penetration<sup>(16)</sup> or loading conditions as discussed later.

#### 4.4.2.3 General

Examination by means of a low power microscope of some of the bend and tensile fractures revealed unbonded areas on the conical spigot face. These unbonded areas were found consistently only on autoclaved specimens. Of the thirteen autoclaved specimens examined (both irradiated and unirradiated), nine had patches similar to that shown in Figure 17. Only one of nine unautoclaved specimens had any unbonded regions and these were very small (Figure 18). In addition, in the areas which had bonded there appeared to be a better pressure bond on the unautoclaved specimens. Figure 19 is typical of the eight other unautoclaved specimens examined.

Although the weaker welds generally exhibited unbonded areas, there was no definite correlation. This was possibly due to varying sizes of fusion zone and therefore the area over which the breaking force acted. Similarly there was no correlation between the amount of unbonded material and the position of the weld in the assembly

### 5. DISCUSSION

#### 5.1 Mechanical Properties

##### 5.1.1 Tensile Specimens

There were two principle difficulties in interpreting these results:

- (a) the scatter of the results;
- (b) the different responses of specimens in the same condition to the two testing temperatures and to neutron irradiation.

Furthermore, many results did not follow previously established trends; this was especially true for the welded material.

#### 5.1.1.1 The Effect of Welding

It would be expected from previous work<sup>(12, 13)</sup> that the cooling rate of the fusion zone would be of primary importance. Other factors such as the cooling rate from the hydriding temperature (750°C)<sup>(12, 13)</sup>, the annealing during hydriding<sup>(7, 10, 12)</sup> and the amount of hydrogen introduced into these specimens<sup>(22)</sup> should have no effect on the slow strain rate mechanical properties. However, grain size and possible stress raisers produced by welding could have an effect.

For the following reasons the cooling rate of the fusion zone was assumed to be relatively slow:

- (a) Two passes were made for each weld. The first pass would serve to preheat the plate.
- (b) The welds were closely spaced. Each weld would preheat the plate for the succeeding weld.
- (c) There was a gradual transition from the fusion zone to the base plate microstructure.
- (d) The  $\alpha$ -platelets in the fusion zone were relatively coarse.

The slow cooling rate from the  $\beta$ -range necessary for a decrease in total elongation with little change in strength properties<sup>(12, 13)</sup> was possibly present. However, the elevated temperature results appeared to be unaffected by the cooling rate and followed the previous trends found for welded Zircaloy-2<sup>(7-10, 14)</sup>.

Keeler<sup>(23)</sup> showed that the strength of  $\alpha$ -zirconium decreased with increasing grain size. It would be expected then, that the increased (prior  $\beta$ ) grain size produced by welding would have some effect when welded and as-received fine-grained material were compared.

With one exception, the scatter in the room temperature results was higher than in the 260°C results. This may indicate that some stress-raising mechanism was operative at the lower but not the higher testing temperature.

The interaction of these three variables may have produced the deviations from previous results. Presumably the adverse variables at room temperature, grain size and stress raisers, cancelled the

strengthening effect of welding but were less effective at 260°C. However, not enough experimental evidence was available to verify this.

#### 5.1.1.2 The Effect of Heat Treating

Since microscopic examination revealed that the specimens were not in the  $\beta$ -quenched condition, no definite conclusions could be drawn from the results. It may be noted that Goodwin et al(12) also found the YS (and presumably the PL) decreased when quenching from 1652°F (900°C). The UTS decreased slightly. However, in the present investigation the total and uniform elongations remained unchanged.

Any changes in the 260°C properties were masked by scatter. These results were inconclusive although one would expect them to follow the room temperature results.

#### 5.1.1.3 The Effect of Irradiation

Normally irradiation increases the strength and decreases the ductility of Zircaloy-2(19). The high post-irradiation total and uniform elongation of all specimens may have been due to in-loop annealing of the irradiation damage. Howe(24) showed that recovery of the uniform and total elongation of annealed Zircaloy-2 after an irradiation of  $7.7 \times 10^{19}$  (fast) n/cm<sup>2</sup> at 280°C was virtually complete after 1 to 2.3 hours annealing at 325°C. The UTS, YS and PL would have required considerably longer to return to their original values. For the  $0.27$  to  $1.9 \times 10^{19}$  (thermal) n/cm<sup>2</sup>\* irradiation received, some recovery of the elongation may have occurred during the 2-1/2 hour final shutdown. The prolonged heating at 260°C in the loop was assumed to have had no effect in improving the strength(7, 10) or the ductility(7) of the weld metal.

It is not understood why the as-welded material did not increase in strength after neutron irradiation. The welded and hydrided specimens did show an appreciable increase.

#### 5.1.2 Impact Tests

The mechanism causing the ductile-to-brittle transition in Zircaloy-2 is not understood. Howe, (22) using subsized impact specimens, detected no change over the transition range in the fracture mode

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\* The unperturbed fast neutron flux (>500 eV) in the X-5 loop is about 8% of the thermal flux(25).

or in the density of twinning adjacent to the fracture face. In contrast, there is a definite difference in appearance between the ductile fibrous fracture and the brittle cleavage fracture of zirconium<sup>(26)</sup> and most steels. However, it was assumed in this investigation that factors such as grain size and metallurgical condition affect the impact properties of Zircaloy-2 in the same manner as they do in steels and other metals.

In metals possessing a ductile-to-brittle transition, the transition temperature increases with increasing grain size. This effect has been studied primarily in body-centered cubic materials. Ogden et al<sup>(27)</sup> studied the effect of grain size and of  $\beta$ -quenching on the Charpy V-notch impact properties of a Ti-2.5 Cr-2.5 Mo alloy. They found that the impact strength of this alloy decreased with increasing primary- $\alpha$  grain size for all temperatures between  $-328^{\circ}\text{F}$  ( $-196^{\circ}\text{C}$ ) and  $750^{\circ}\text{F}$  ( $400^{\circ}\text{C}$ ). However, the impact strength was unaffected by prior- $\beta$  grain size over the same temperature range. It was also shown that the acicular  $\beta$ -quenched structure had a lower impact strength above  $32^{\circ}\text{F}$  ( $0^{\circ}\text{C}$ ) than an equiaxed  $\alpha$  structure of the same grain size. If a comparison may be drawn between the  $\beta$ -quenched Ti-2.5 Cr-2.5 Mo alloy and welded Zircaloy-2, one may speculate that the increase in the transition temperature, the overall decrease in impact strength (at least above  $0^{\circ}\text{C}$ ) and the narrowing of the transition range were caused by a combination of an increased grain size and the  $\beta$ -quenched structure produced by the welding process.

Howe<sup>(22)</sup> found that neutron irradiation had no effect on the impact properties of annealed or cold worked, fine grained, primary- $\alpha$  Zircaloy-2. It would therefore be expected that neutron irradiation would have no effect on the impact properties of the  $\beta$ -quenched structure of welded Zircaloy-2.

### 5.1.3 Spigot Welds

#### 5.1.3.1 Tensile Tests

The expectation that the autoclaved and welded specimens would exhibit higher tensile strengths was based on the torsion tests. The higher torsional breaking strength was felt to be indicative of better weld penetration in the autoclaved specimens<sup>(15)</sup>. However, the tensile tests appeared to indicate the opposite.

The strength of each weld will depend on<sup>(2)</sup> (see Figure 2):

- (a) the extent of the fusion zone. Normally, fusion will penetrate half the thickness of the end-plate;
- (b) the amount of pressure bonding on the conical spigot face. The area over which this type of bonding occurs depends very much on the contact area and on the force exerted over this area during welding.

Assuming the cooling rate and the weld penetration to be approximately the same for all welds, the tensile strength of the weldment depends on the amount of pressure bonding achieved. The amount of pressure bonding could be adversely affected by the following conditions:

- (a) the presence of the oxide layer on the autoclaved specimens(28, 29);
- (b) insufficient peripheral pressure exerted on the copper chill;
- (c) too much curvature in the end-plate due to the longer inner elements.

The presence of the black oxide layer on the autoclaved specimens would account not only for the unbonded patches on the conical face of these specimens, but also for the lower strengths of the autoclaved specimens in tension and in bending. The latter two points could produce the differences in strength between the outer and inner welds in either condition.

Irradiation had no effect on the tensile strength of the spigot welds. In these tests the shear strength of the weld metal was measured and the results seem to verify the room temperature results for the welded tensile specimens.

### 5.2.2 Bend Tests

The loading conditions in these tests were not satisfactorily reproducible. The tongue which applied the load to the end plate (Figure 7) bent after one or two tests. This, combined with the small angle of the plate to the vertical, would serve to concentrate an otherwise uniformly distributed load on the outermost point of the end plate. Therefore, the magnitude of the bending moment would increase with a consequent decrease in the breaking force.

The increased frequency with which the cup-and-cone failures occurred in the irradiated specimens suggested that irradiation hard-

ening may have been a contributing factor. There was noticeably less bending in the irradiated end plates and spigots. Therefore, the magnitude of the tensile force on the weld would decrease and the amount of direct bending would tend to become the controlling factor, thus producing a greater probability of cup-and-cone failures. That is, neutron irradiation increased the yield strength of the spigot and the end plate above the bend strength of the weld.

There were some minor differences between the observed E-20 failures (Figure 1) and the cup-and-cone failures produced in this investigation (Figures 13 and 16). This was quite probably due to the small angle between the force and the end plate (approximately  $30^\circ$ ) and to the difference in the rate of loading. Had a larger angle been used, a truer bend failure would have resulted which would have resembled more closely the type observed by Daniel and Parry<sup>(2)</sup>. Furthermore, the sharp angle would result in a combination of bend and shear forces which could also result in a failure similar to that in Figure 15.

### 5.3 Recommendations for Further Work

Some of the results of this work were inconclusive. To produce meaningful results, either the scatter must be reduced or the number of specimens increased. Some recommendations to help reduce scatter in results are:

- (a) The welding variables should be controlled so that an estimate of the cooling rate of the fusion zone can be obtained.
- (b) The tensile specimens should be all weld metal to eliminate the possibility of fracture in the heat-affected zone. Alternatively, a specimen should be cut perpendicular to the weld with a reduced area in the fusion zone<sup>(8)</sup>.
- (c) Supplement the welded results with mechanical tests using coarse and fine-grained  $\beta$ -quenched specimens. These specimens should be cooled at a rate approximating that of the fusion zone of the welded specimens.
- (d) Better control over the irradiation conditions would also be desirable. For instance, a room temperature irradiation in a transformer rod for one NRX cycle would eliminate many of the variables encountered in a loop irradiation and would give a better idea of the actual irradiation effect.

## 6. SUMMARY

The following results were found to be significant:

- (a) Welding decreased the room temperature and the 260°C total and uniform elongations, and increased the 260°C UTS, YS and PL.
- (b) Increasing the hydrogen content by a nominal 100 ppm did not affect the unirradiated weld metal properties at either test temperature.
- (c) Heat treating at 900°C for 40 minutes following by an oil quench decreased the room temperature UTS, YS and PL. The total and uniform elongation remained the same.
- (d) Neutron irradiation of  $0.3$  to  $1.9 \times 10^{20}$  thermal n/cm<sup>2</sup> raised the room temperature YS of welded-and-hydrided material 20% and the heat-treated YS 40%. The total and uniform elongation of the heat treated specimens remained constant and the UTS increased slightly. Irradiation also increased the 260°C strengths of the welded and welded-and-hydrided material.
- (e) Welding decreased the impact strength at all temperatures between 0° and 300°C, increased the transition temperature and decreased the transition range. Neutron irradiation did not affect the impact properties of welded Zircaloy-2.
- (f) For the spigot weld strengths, it was found that:
  - i) the unautoclaved welds were stronger in tension than the autoclaved specimens;
  - ii) the outer welds had a lower average breaking load in tension than the inner welds. This was probably true for both the autoclaved and unautoclaved specimens;
  - iii) neutron irradiation did not affect the tensile breaking strength of either the autoclaved or unautoclaved specimens;
- (g) It was demonstrated that the E-20 type of failure could readily be produced using the method of loading proposed by Daniel and Parry<sup>(2)</sup>.
- (h) A higher percentage of cup-and-cone fractures occurred in the irradiated specimens than in the unirradiated welds.

Most of the changes in the mechanical properties due to welding were small. Other changes were found to be non-significant. There was some indication that the welding conditions used in this investigation did not affect the room temperature properties of Zircaloy-2. Also, neutron irradiation did not appear to affect the room temperature strength of welded Zircaloy-2. However, these latter two points need verification.

#### ACKNOWLEDGEMENTS

This project was initiated by C.R. Cupp. The apparatus used for testing spigot welds was designed and operated by G.A. McCurrach. McCurrach and W.J. Langford carried out all the mechanical testing. Thanks are also due to W. Evans, A. Sawatzky, C.E.L. Hunt, J. Veeder and G.W. Parry for helpful discussions and information.

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Table 1

## Chemical Analysis of Materials Used

<u>Element</u>	<u>Plate</u>	<u>Filler Wire</u>
Sn	1.42%	1.45%
Fe	0.12%	0.12%
Cr	0.10%	0.0866%
Ni	0.05%	0.0587%
O <sub>2</sub>	-	1245 ppm
N <sub>2</sub>	-	17 "
H <sub>2</sub>	7 ppm	60 "
C	<300 "	203 "
Mn	< 20 "	< 15 "
Al	24 "	< 30 "
V	< 20 "	< 20 "
Mo	< 20 "	< 20 "
Hf	89 "	< 45 "
B	< 0.2 "	< 0.2 "
W	< 20 "	< 20 "
Mg	< 20 "	< 15 "
Co	< 20 "	< 5 "
Si	35 "	< 30 "
Zn	-	< 10 "
Cd	< 0.5 "	< 0.2 "
Cu	< 20 "	< 20 "
Pb	< 20 "	< 15 "
Tl	-	< 20 "

Table 2Comparison of the E-20 and X-5 Irradiations<sup>(30)</sup>

Quantity	E-20	X-5
Integrated Thermal Flux (approx) n/cm <sup>2</sup>	$1.5 \times 10^{21}$	0.27 to $1.9 \times 10^{20}$
Coolant Temperature °C	252 to 306	260°C (ave)
Average Coolant Flow (g(U. S. )/min)	220	80
Coolant Pressure (psig)	1390	1150
Number of Shutdowns	0	5

Table 3

## Mechanical Test Results of the Tensile Specimens

Unirradiated				Irradiated			Mechanical Properties	Testing Temp.
As- Received	Welded*	Welded & Hydrided	Heat- Treated	Welded	Welded & Hydrided	Heat- Treated		
44.4	41.0	46.5	34.8	45.1	60.1	49.8	PL (ksi)	RT
48.6	47.3	52.2	39.2	49.3	63.3	54.9	0.2% YS	
72.0	62.7	68.4	67.1	62.3	74.7	74.8	UTS	
24.4	11.9	12.0	25.3	14.3	11.9	24.2	% Elong	
13.3	10.2	8.4	13.4	11.4	7.7	12.0	% Uniform Elong	
19.6	22.3	22.3	20.8	29.4	28.2	26.4	PL	260°C
21.3	24.8	25.0	23.9	30.9	29.4	28.2	0.2% YS	
35.1	37.3	36.1	44.1	39.2	38.0	43.1	UTS	
41.8	21.3	21.5	35.4	18.5	23.5	29.1	% Elong	
13.3	12.3	11.7	13.8	9.3	11.6	8.6	% Uniform Elong	

\* Average of three specimens. All others average of two specimens at each temperature.

Table 4

## Mechanical Test Results on End Plug to End Plate Welds

<u>Specimen History</u>	<u>Specimen Number</u>	<u>Type of Test</u>	<u>Breaking Load (lb)</u>	<u>Type of Fracture</u>
Irradiated				
Unautoclaved	1	Bend	370	Cup-and-Cone
"	3	"	435	Grip broke
"	5	"	545	Grip broke
"	6	"	580	Sheared across spigot
"	10	"	190	Cup-and-Cone
"	12	Tensile	520	Spigot pulled out of end plate
"	13	"	620	"
"	15	"	612	"
"	17	"	512	"
"	19	"	650	"
Irradiated				
Autoclaved	1	Bend	260	Cup-and-cone
"	3	"	385	Spigot pulled out of end plate
"	5	"	118	Cup-and-cone
"	6	"	100	"
"	10	"	210	"
"	12	Tensile	370	Spigot pulled out of end plate
"	13	"	465	"
"	15	"	520	"
"	17	"	460	"
"	19	"	425	"
Unirradiated				
Unautoclaved	4	Tensile	550	Spigot pulled out of end plate
"	8	"	545	"
"	14	"	585	"
"	16	"	654	"
"	18	"	650	"
"	2	Bend	405	"
"	11	"	432	Cup-and-cone
"	7	"	430	Broke across spigot

(. . . cont'd)

Table 4 (cont'd)

<u>Specimen History</u>	<u>Specimen Number</u>	<u>Type of Test</u>	<u>Breaking Load (lb)</u>	<u>Type of Fracture</u>
Unirradiated				
Autoclaved	4	Tensile	510	Spigot pulled out of end plate
"	8	"	325	"
"	14	"	545	"
"	16	"	452	"
"	18	"	420	"
"	7	Bend	570	"
"	2	"	420	Cup-and-Cone
"	9	"	375	"
"	11	"	450	Broke across spigot

All specimens tested at room temperature.

Table 5

Results of Impact Tests on Welded and Unwelded Zircaloy-2					
Testing Temp (°C)	Energy Absorbed (in-lbs)				As Received
	Welded Irradiated	Unwelded Irradiated	Welded Unirradiated	Unwelded Unirradiated	
0	7.0	4.5	7.9	13.0	21.5
25	7.0	6.0	6.0	8.2	24.5
	8.5	6.5	10.7	14.0	
35			10.6	10.0	
40			10.5	15.5	
50	7.5	6.0			28.0
55				15.0	
60			13.3	4.5	
63			11.0		
75	10.5	12.0			28.0
80			7.5	17.5	
100	10.5	13.5	12.7	23.0	32.0
118			13.0	17.3	
125	16.0	16.0			
140			12.4	16.9	
			20.5	30.5	
			16.5		
145				24.2	
150	16.0	18.0			
155			19.4	18.9	
160	16.5				36.0
175	25.5	22.0	20.7	20.4	
180					42.5
200	25.0	24.0	24.5		
220			24.5	20.0	41.5
225	28.5	23.5			
230			25.3	20.0	
240	26.5	25.5			
250	36.0	30.0			
	24.5				
260	30.0	23.0			
275	25.5	25.0	29.9	32.6	42.0
	30.0	24.0			
300	35.0	26.5	34.1	33.1	



Table 6

## Tests of Significance of Some of the Mechanical Test Results

Specimen Condition	Property	Standard Deviation	Degrees of Freedom	t	Significance
As-received Welded	RT YS	$\pm 2.02$ psi $\pm 4.59$ "	2 5	1 14	None
As-received Welded	RT UTS	$\pm 1.87$ psi $\pm 5.05$ "	2 33	2.45	None
As-received Heat-treated	RT UTS	$\pm 1.87$ psi $\pm 0.05$ "	4.91	3.32	Fairly
As-received Heat-treated	RT PL	$\pm 1.61$ psi $\pm 0.07$ "	4.55	12.67	Very
Welded As-received	RT % Elong	$\pm 3.63$ psi $\pm 1.71$ "	2.05	4 88	Fairly
Welded and hydrided As-received	RT YS	$\pm 1.70$ psi $\pm 2.02$ "	-	1.13	None
Welded Welded and hydrided	RT YS	$\pm 4.59$ psi $\pm 1.70$ "	-	1.68	None
Welded and hydrided, unirrad. " " " irradi.	RT UTS	$\pm 1.00$ psi $\pm 0.04$	1.26	8.59	Slight
As-received Welded	260°C PL	$\pm 0.03$ psi $\pm 0.04$ "	2 46	8.21	Very
As-received Heat-treated	260°C UTS	$\pm 0.06$ psi $\pm 7.63$ "	1.00	1.66	None
Welded, irradiated " unirradiated	260°C YS	$\pm 1.41$ psi $\pm 1.17$ "	1.76	5.00	Fairly
Welded, irradiated " unirradiated	260°C UTS	$\pm 0.04$ psi $\pm 1.11$ "	2.72	2.69	Slight
Autoclaved, Inner welds " Outer welds	Tensile breaking load	$\pm 46.6$ lbs $\pm 96.5$ "	-	1.21	None
Unautoclaved, Outer welds " Inner welds	Tensile breaking load	$\pm 16.1$ lbs $\pm 50.7$ "	7.85	3.47	Very
Autoclaved, Inner welds Unautoclaved, Inner welds	Tensile breaking load	$\pm 46.6$ lbs $\pm 50.7$ "	11.9	5 44	Very
Autoclaved, Outer welds Unautoclaved, Outer welds	Tensile breaking load	$\pm 96.3$ lbs $\pm 16.1$ "	1.96	2.41	None
Autoclaved Unautoclaved	Failure Torque	$\pm 1.12$ in-lbs $\pm 0.88$ "	3.40	48.6	Very

Table 6 (cont 'd)

<u>Specimen Condition</u>	<u>Property</u>	<u>Standard Deviation</u>	<u>Degrees of Freedom</u>	<u>t</u>	<u>Significance</u>
Welded, irradiated " unirradiated	260°C UTS	± 0.04 psi ± 1.11 "	2.72	2.69	Slight
Autoclaved, Inner welds " Outer welds	Tensile breaking load	± 46.6 lbs ± 96.5 "	-	1.21	None
Unautoclaved, Outer welds " Inner welds	Tensile breaking load	± 16.1 lbs ± 50.7 "	7.85	3.47	Very
Autoclaved, Inner welds Unautoclaved, Inner welds	Tensile breaking load	± 46.6 lbs ± 50.7 "	11.9	5.44	Very
Autoclaved, Outer welds Unautoclaved, Outer welds	Tensile breaking load	± 96.3 lbs ± 16.1 "	1.96	2.41	None
Autoclaved Unautoclaved	Failure Torque	± 1.12 in-lbs ± 0.88 "	3.40	48.6	Very

Table 7

## Hydrogen Analyses

<u>Specimen Number</u>	<u>History</u>	<u>Hydrogen Content (ppm)</u>
Inactive		
2	Welded, hydrided, tensile specimen	32
6	" " " "	122
8	" " " "	81
4	Unautoclaved end plug	28
16	" " "	12
8	Autoclaved end plug	22
18	" " "	8
10	Welded tensile specimen	8
20	" " "	8
22	Heat-treated tensile specimen	5
-	As-received tensile specimen	7
Active		
1	Welded, hydrided, tensile specimen	58
5	" " " "	74
9	Welded tensile specimen	33
13	" " "	10
27	Heat-treated tensile specimen	<1

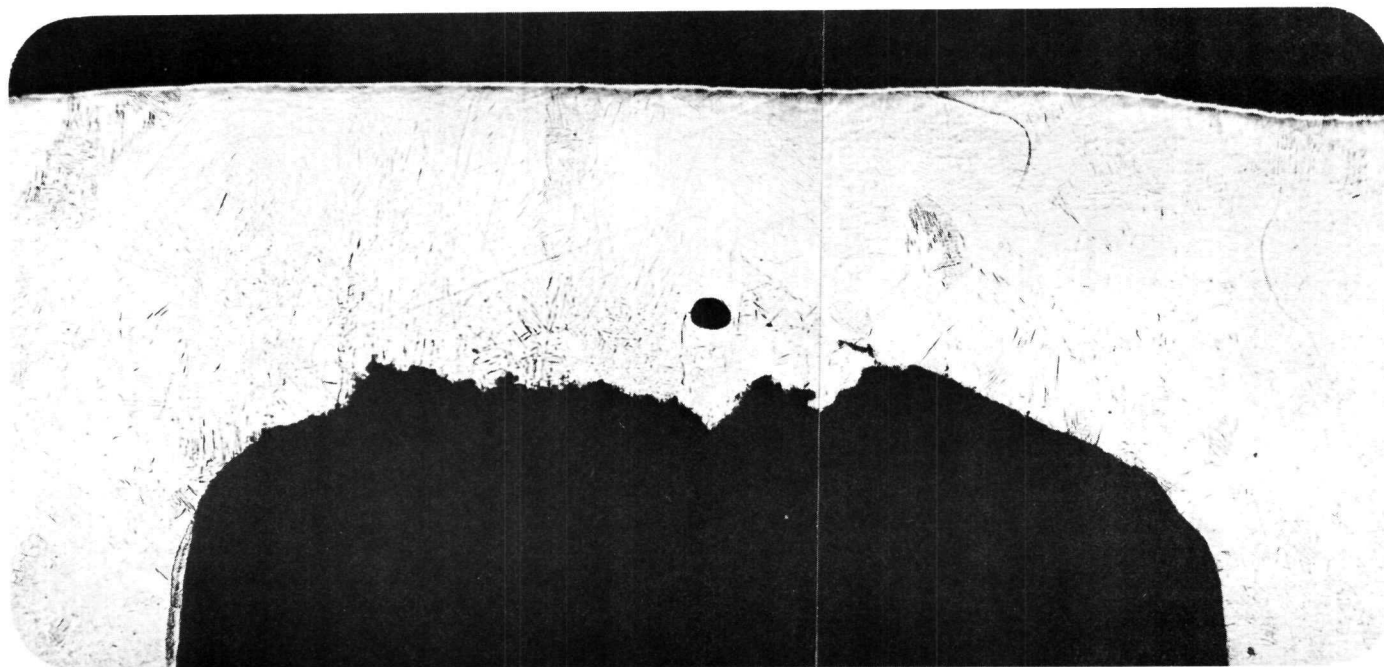
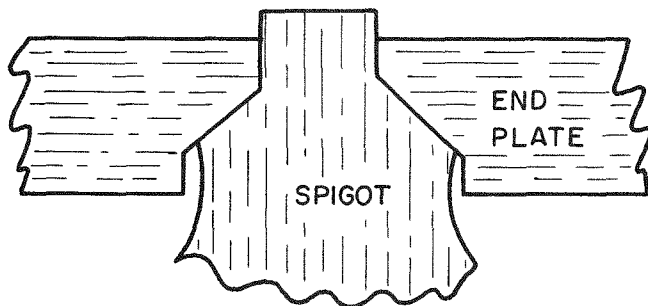


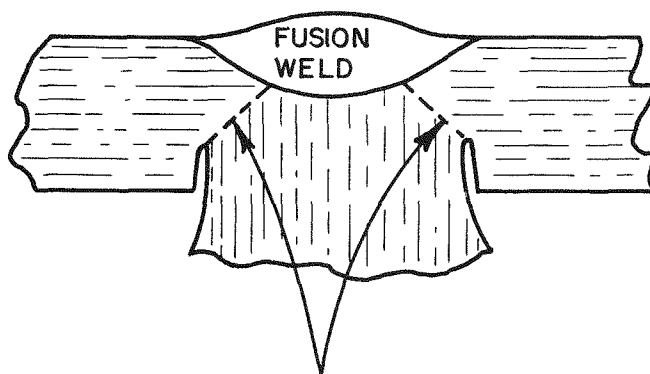
Figure 1: (Met Z70D2)  
Typical E-20 Failure (from Exp-NRU-1312; Figure 25)

X50

FIG. 2  
TYPICAL SPIGOT WELD  
(FROM EXP-NRU-1312; FIG. 5 & 6)



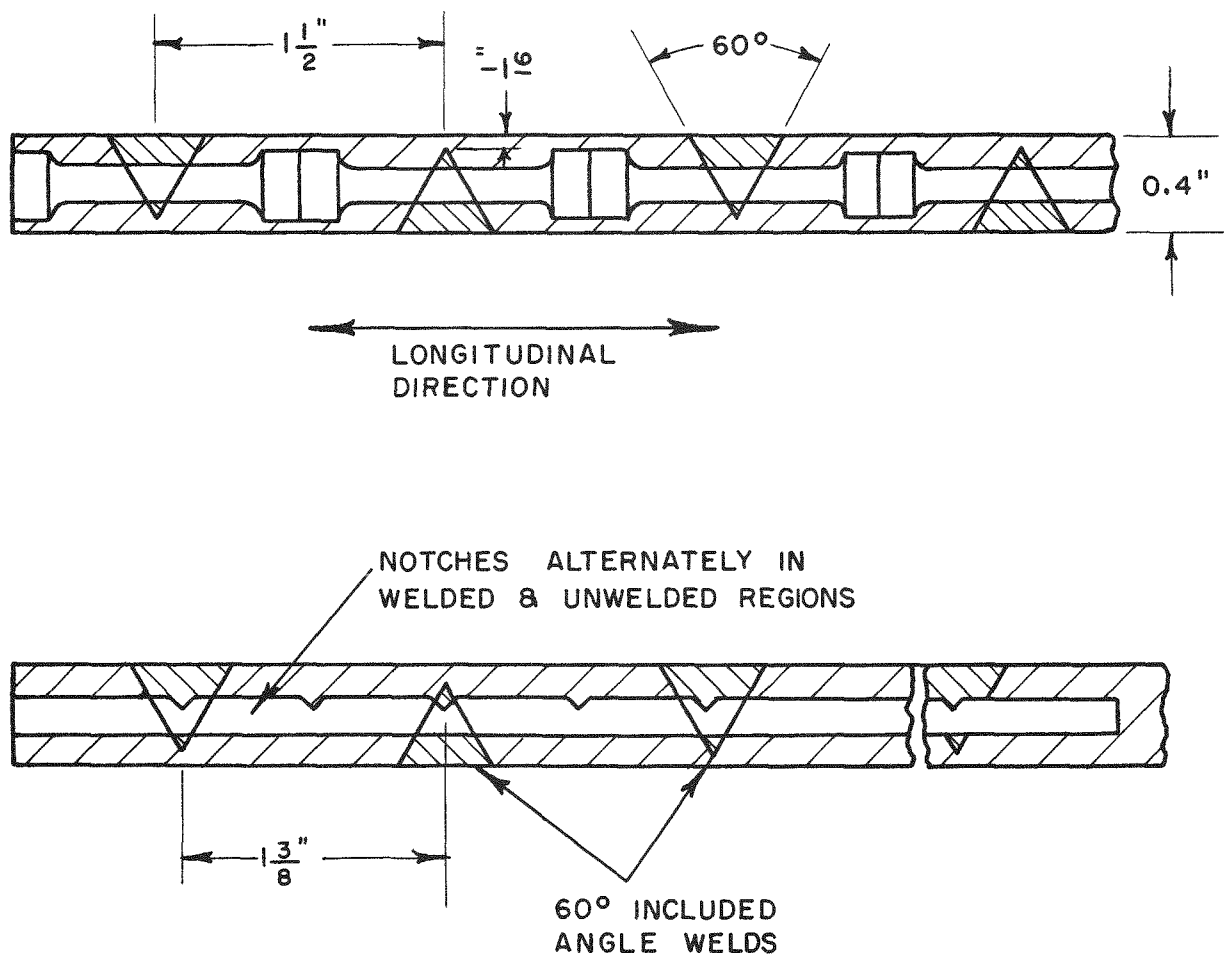
BEFORE  
WELDING



AFTER  
WELDING

INTERMITTENT PRESSURE  
WELDING ON CONICAL FACE

FIG. 3  
METHOD OF SPECIMEN PREPARATION



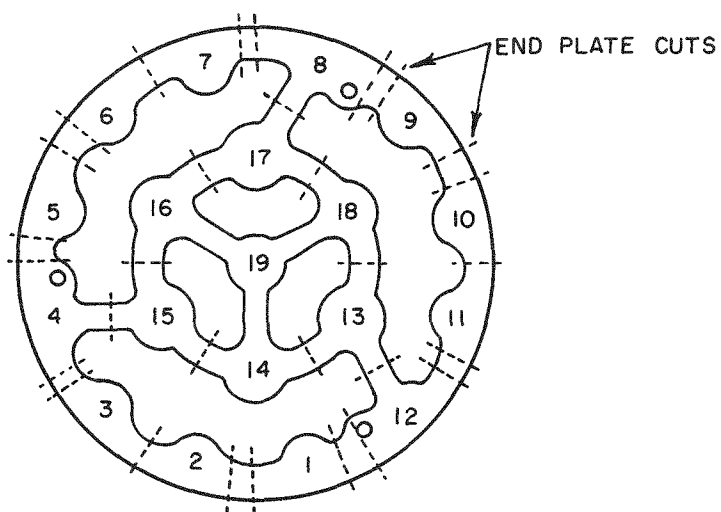


FIG. 4  
TOP VIEW OF END PLATE SHOWING SPECIMEN POSITIONS

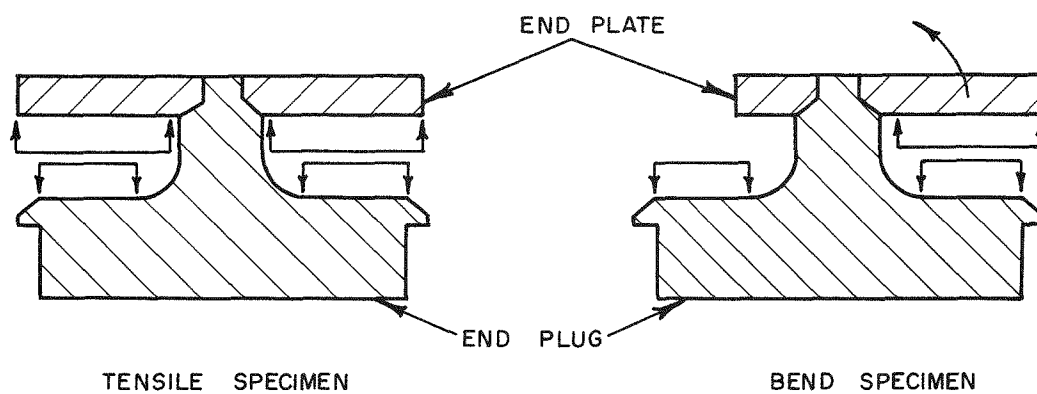


FIG. 5  
STRESS CONDITIONS AND SPECIMEN TYPES USED

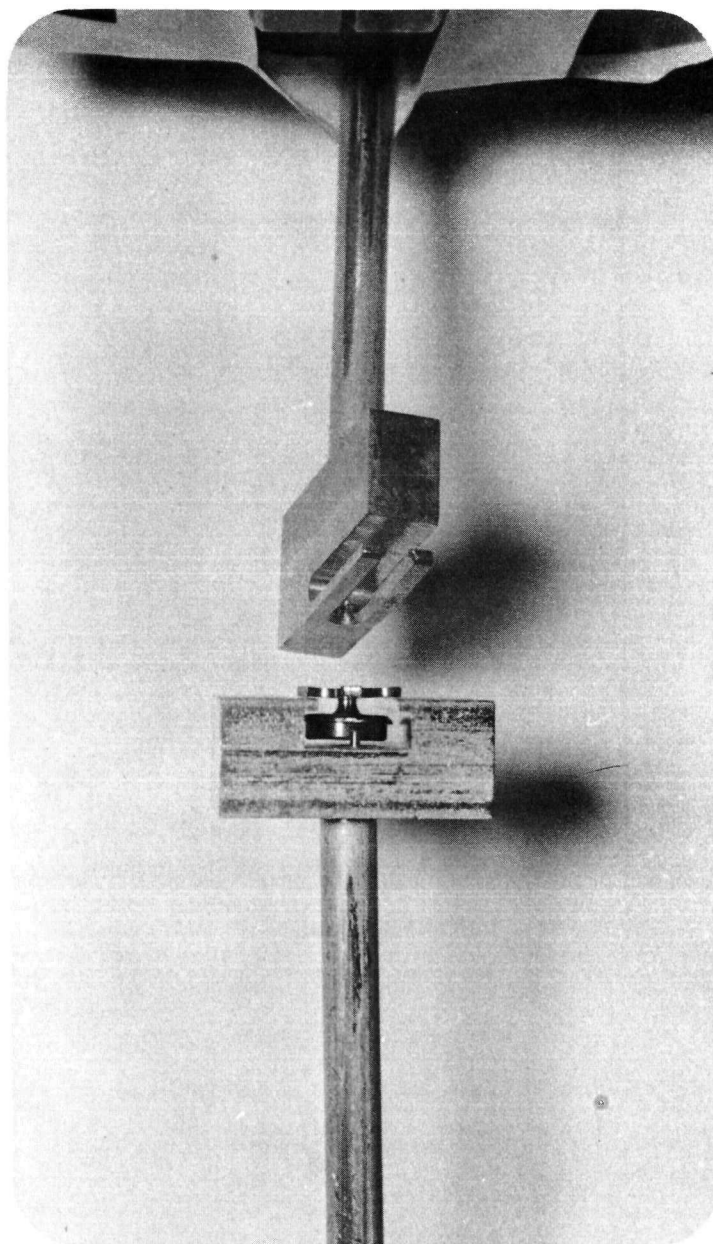


Figure 6.      Tensile Testing Apparatus



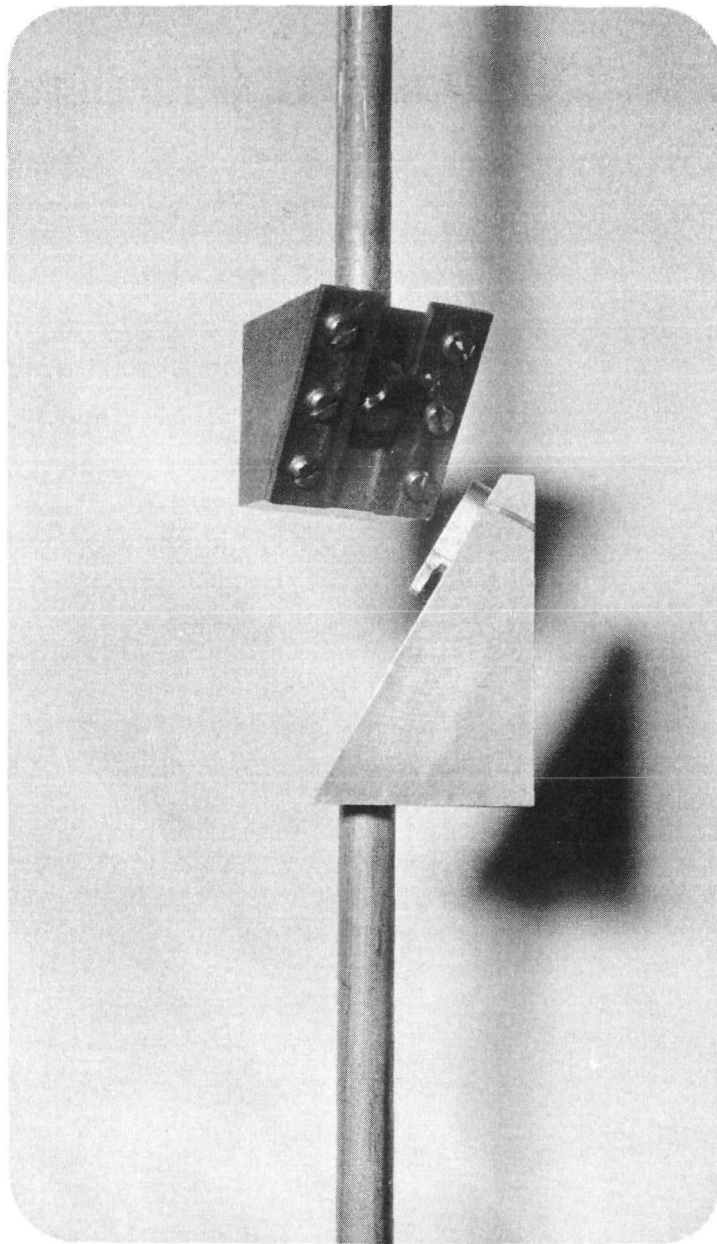
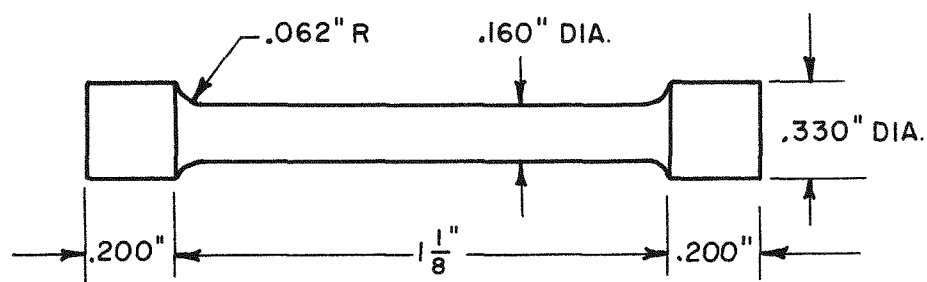
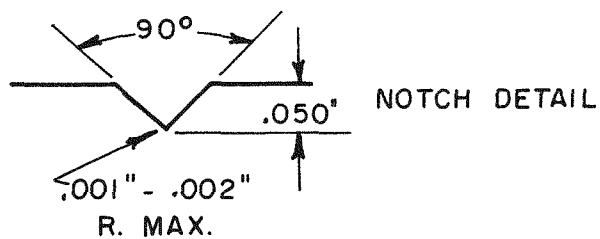
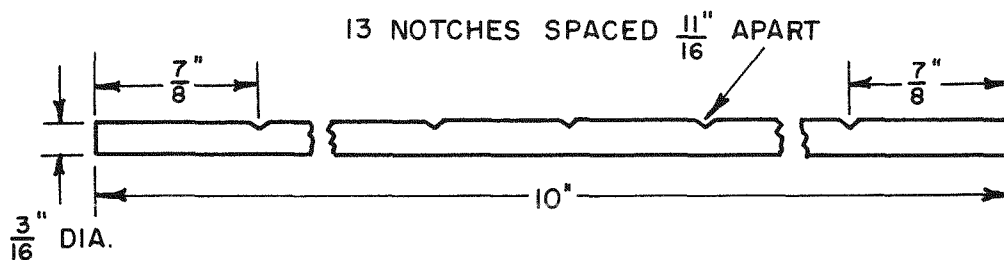


Figure 7. Bend Test Apparatus

FIG. 8  
MECHANICAL TEST SPECIMEN

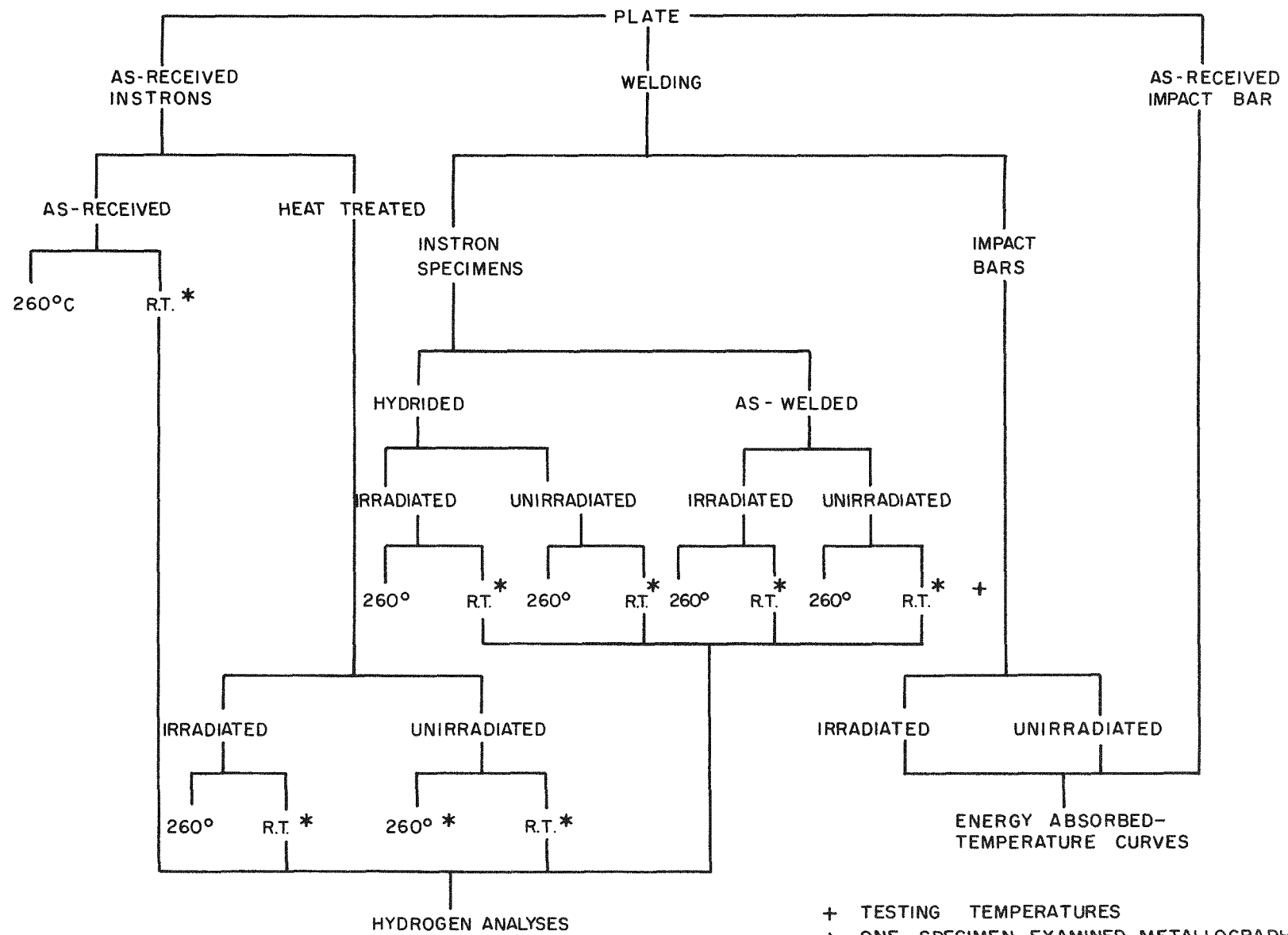


INSTRON TENSILE TEST SPECIMEN



SUB-SIZE IMPACT SPECIMEN

FIG. 9  
 FLOWSHEET FOR SPECIMENS FROM WELDED PLATE



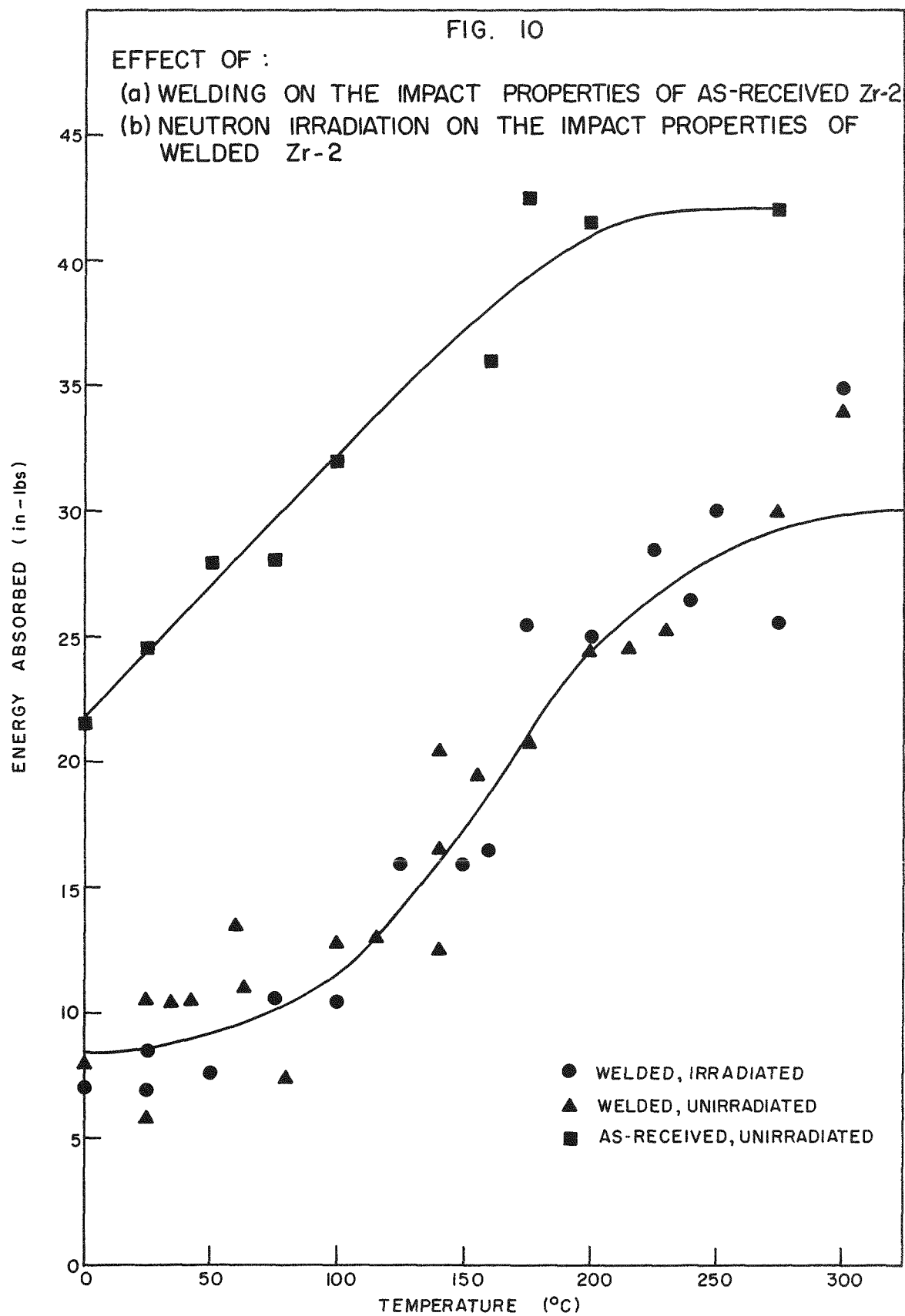
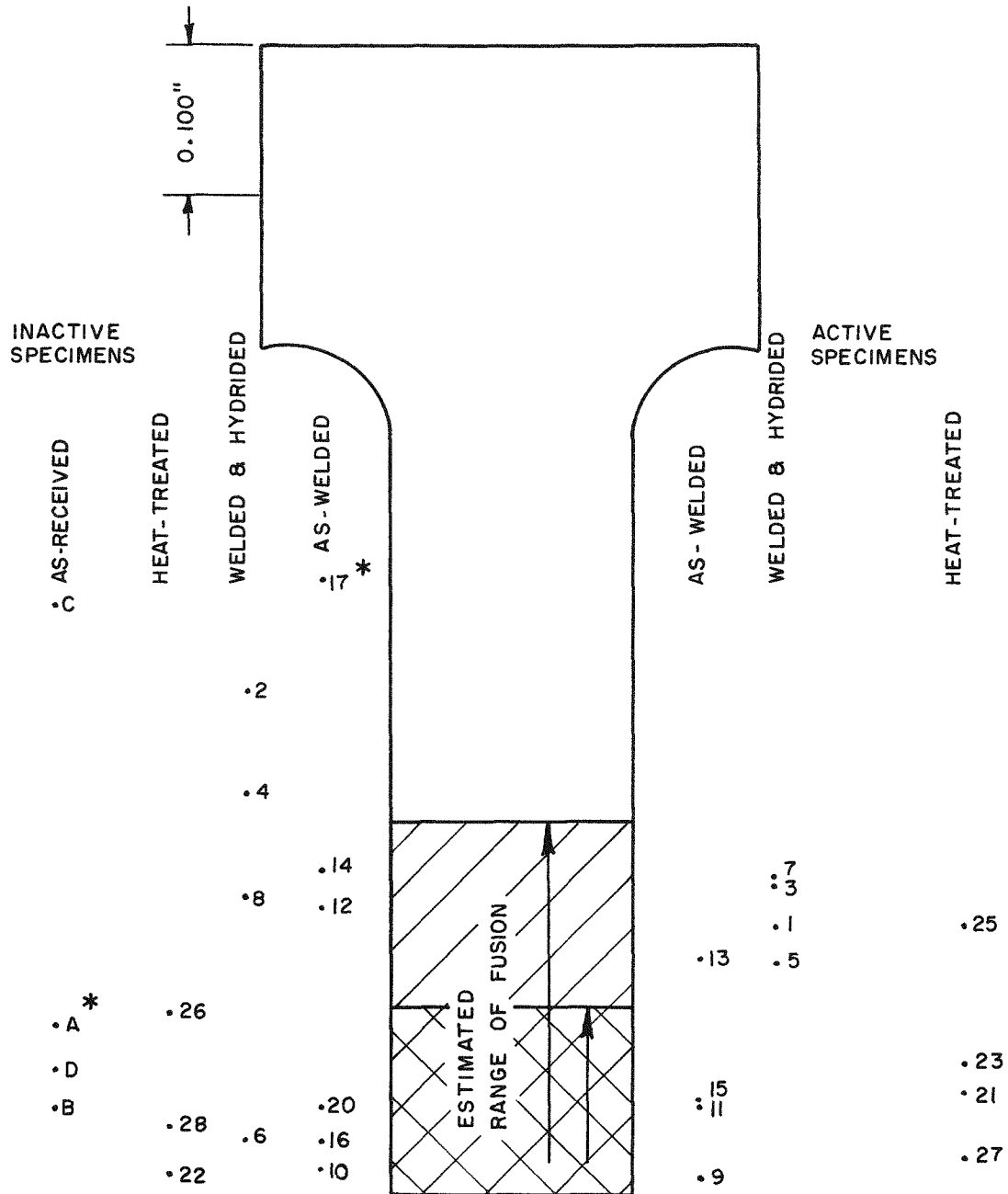


FIG. II  
FRACTURE POSITIONS OF ACTIVE  
AND INACTIVE SPECIMENS



\* SPECIMEN IDENTIFICATION NUMBERS & LETTERS



Figure 12 (Met. E13-62)  
Hydride Orientation in Hydrided Specimens.

X250

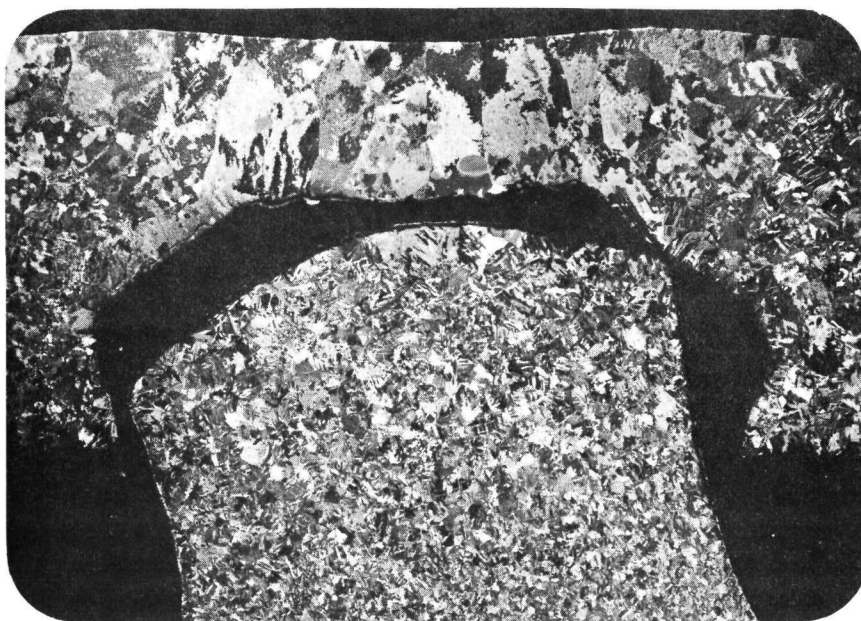


Figure 13. (Met. D369-A1)  
Unirradiated Cup-and-Cone Failure.

X30

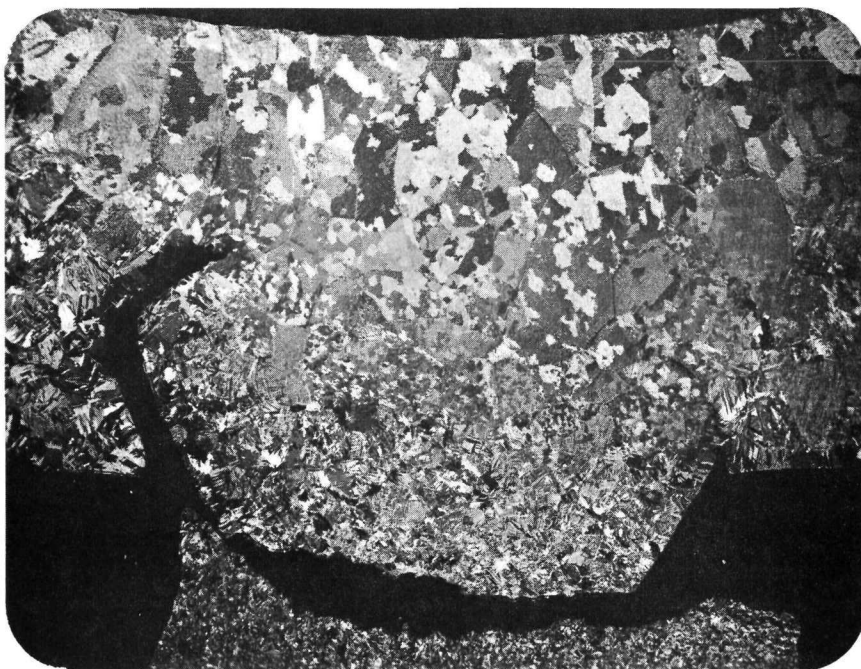


Figure 14. (Met. D369-B1)  
Unirradiated Spigot Failure.

X30

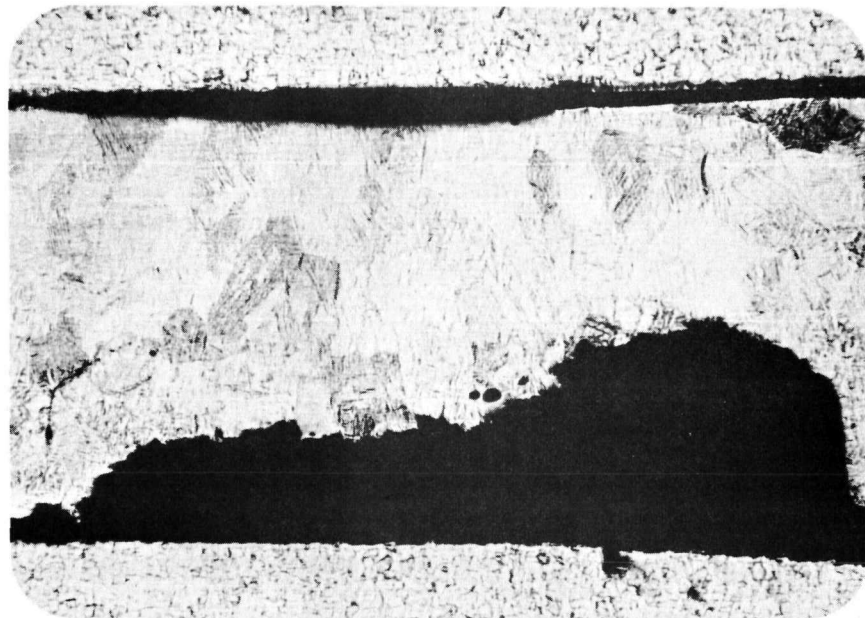


Figure 15. (Met. Y24-A1)  
Failure Mode of Specimen No. 1.

X32

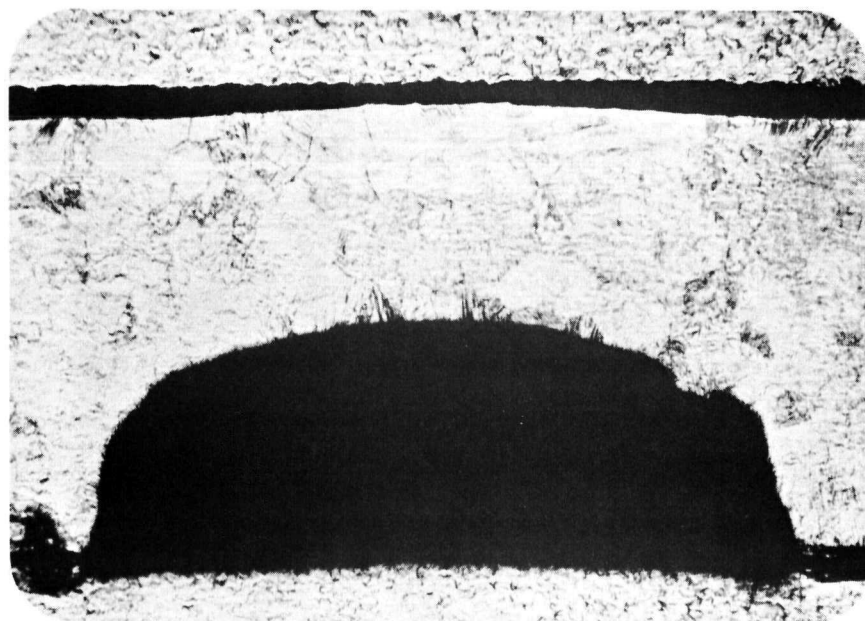


Figure 16. (Met. Y24-C1)  
Typical Failure of Irradiated Low-Strength Specimen.

X32



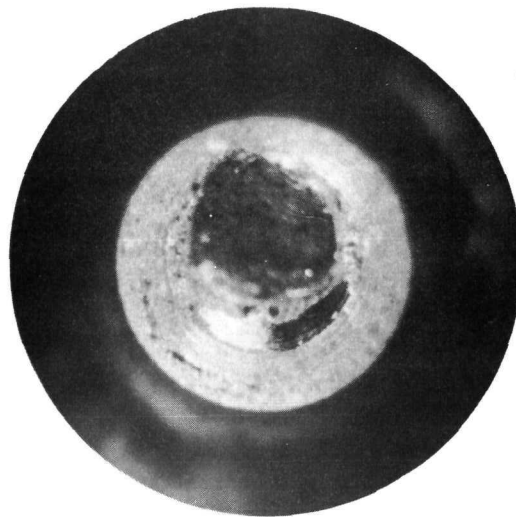


Figure 17. (Ref. No. 6595) X13  
Specimen No. 19A Autoclaved; Showing Unbonded Area.

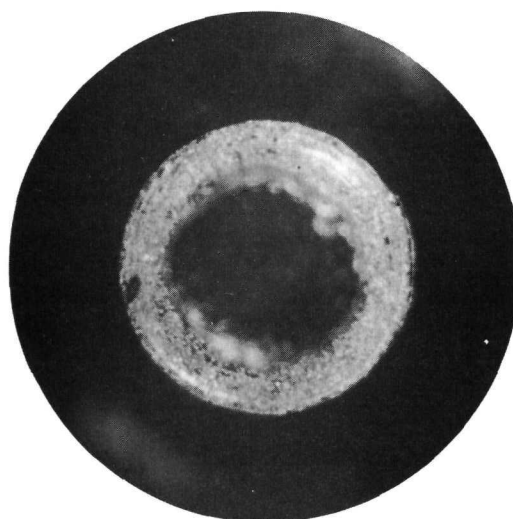


Figure 18. (Ref. No. 6596) X13  
Specimen No. 17. Only Unautoclaved Specimen  
Exhibiting an Unbonded Area (Upper Right).

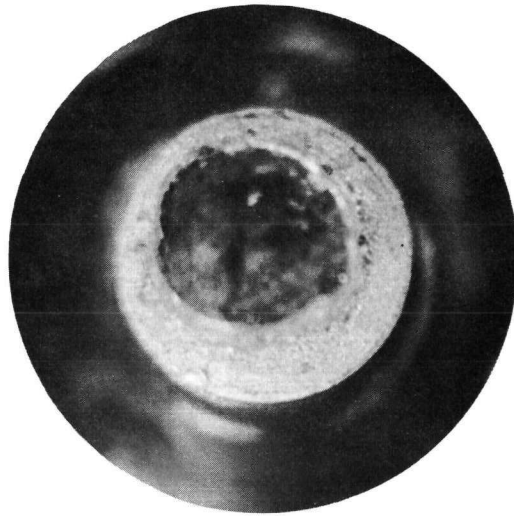


Figure 19. (Ref. No. 6597) X13  
Conical Spigot Face Showing Typical Fracture of  
an Unautoclaved Specimen (No. 15).