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WANL-TME-1815
July 10, 1968

Westinghouse Astronuclear Laboratory

EXPERIMENT DESIGN FOR GTR-20

RADIATION EFFECTS TEST OF

BERYLLIUM, 37/W406
(U)

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I. Experimental Purpose

Beryllium will be used in the R-1 reflector as one of the presently conceived designs shown in Figure 1. It has good neutron moderating and reflecting properties as it is a light element and has a low neutron absorption cross section.

Material properties of beryllium are known for beryllium specimens irradiated to fast neutron fluences of 2.4×10^{17} and 7.6×10^{17} nvt ($E > 1$ MeV) in GTR-18 and GTR-19, respectively (Reference 1). The brittle fracture toughness is known for beryllium wedge-opening-loading (WOL) specimens irradiated to a fast neutron fluence of 5×10^{17} nvt ($E > 1$ MeV) in GTR-19 (Reference 2).

The A-6 reflector exhibited extensive cracking, which demonstrates the need for additional knowledge about beryllium brittle fracture toughness. It is assumed that cracking occurred in A-6 near the end of the run. This gives a fast neutron ($E > 1$ MeV) fluence of approximately 3×10^{18} nvt at the inner reflector radius, core midplane.

There is evidence in the literature of a synergistic effect between neutron fluence and applied load during irradiation for other materials. This is to be investigated for beryllium and the knowledge may contribute to the solution of the reflector cracking problem.

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The environment for beryllium in the R-1 reactor consists of fast neutron fluences ($E > 1$ MeV) between 1.5×10^{17} and 6.0×10^{18} nvt (Reference 3) and temperature between 100 and 550°R (Reference 4). The uncertainty in the neutron fluence is $\pm 50\%$, while the uncertainty in temperature is $+ 55\%$ and $- 33\%$. Hence a knowledge of beryllium material properties and fracture toughness is needed for fast neutron fluences up to 9×10^{18} nvt and for temperatures between 70 and 850°R.

The R-1 reactor is expected to undergo approximately 10 restarts, causing the reflector to anneal after each run. It is necessary to learn how much effect remains after each annealing.

II. Experimental Objectives

Following are objectives for 37/W406.

For group A-I determine:

1. 0.2% tensile yield strength
2. Ultimate tensile strength, and
3. Elongation

As a function of

1. Temperature from 140 to 540°R
2. Specimen orientation (longitudinal and transverse)
3. Fast neutron fluence from 0 to 1×10^{19} nvt, and
4. Two different cores

For group A-II determine the annealing effect on:

1. 0.2% tensile yield strength .
2. Ultimate tensile strength, and
3. Elongation

As a function of

1. Longitudinal and transverse specimen orientation
 2. Annealing times between 1 and 1000 minutes
 3. Two different cores, and
 4. Annealing temperatures between 273 and 540°R
-

[REDACTED]

For group A-III determine for notched tensile specimens:

1. Notched tensile strength
2. Elongation

As a function of:

1. Temperature from 140 to 540°R
2. Longitudinal and transverse specimen orientation
3. Fast neutron fluence from 0 to 5×10^{18} nvt, and
4. Two different cores

For group A-IV determine:

1. The brittle fracture toughness

As a function of:

1. Temperature between 140 and 540°R
2. Longitudinal and transverse specimen orientation, and
3. Fast neutron fluence from 0 to 1×10^{19} nvt

Examine the fractured surfaces of these specimens

For group A-V determine:

1. The annealing effect on fracture toughness of specimens irradiated in LN₂ (140°R) to 5×10^{18} nvt

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As a function of

1. Test temperature from 273 to 540°R, and
2. Annealing time from 10 to 1000 minutes

Examine the fractured surfaces of these specimens

For group A-VI determine for specimens held under a tensile load

1. 0.2% tensile yield strength
2. The ultimate tensile strength, and
3. Elongation

As a function of

1. Fast neutron fluence from 0 to 5×10^{18} nvt
2. Tensile load from 0 to 25 ksi
3. Test temperature between 140 and 540°R

III. Analysis Procedures

Standard techniques to perform an analysis of variance will be used to determine the material properties as a function of the independent variables discussed in Section II and the random variability of the specimens. These analyses will be performed by the WANL Reliability Department, and references as to their use can be found in many statistics text books, such as References 5 and 6. The matrices for each group of specimens used in the analysis of variance are given in Appendix A. A fractional factorial design is used. Results will be given in the form of functional equations expressing the property of interest as a function of the variables considered in the experiment plans. The standard deviation associated with the random variability of each property will also be given.

IV. Requirements for Experimental Data

A. Accuracy Requirements

The neutron fluence for specimens with the same fluence requirement in any given group shall be within +5%. Whenever a group has two required fluences (for example, group A-I has 24 specimens that require 5×10^{18} nvt and 12 specimens that require 1×10^{19} nvt) the ratio of high to low fluence shall be $2.00 \pm 10\%$. Variation in fluence between groups shall be within $\pm 30\%$.

The gamma dose of all specimens shall be reported to within $\pm 50\%$ to comply with the NERVA Trend Data Retrieval Program.

The Instron machine load shall be recorded with $\pm 0.5\%$.

The load on pre-stressed specimens shall be recorded within $\pm 1\%$.

All elongation measurements shall be made to $\pm 0.5\%$.

Dimensional measurements of length shall be to the nearest 0.5 mils.

Reduction of area measurements shall be to $\pm 1\%$.

Room temperature shall be recorded to the nearest °R.

IV. Requirements for Experimental Data (Continued)

The Instron machine grip temperatures shall be monitored with thermocouples, and they should be recorded to within $\pm 1^\circ\text{R}$. The specimen temperature shall be accurate to $\pm 5^\circ\text{R}$.

Annealing temperatures shall be accurate to $\pm 2^\circ\text{R}$ in the cryogenic range and to $\pm 5^\circ\text{R}$ at temperatures $\geq$ room temperature.

The annealing times shall be accurate to $\pm 1\%$.

B. Prediction of Results

Beryllium property changes (%) are given in the following table as a result of irradiations to fast neutron fluences of 2.4×10^{17} and 7.6×10^{17} nvt ($E > 1$ MeV) in GTR-18 and 19, respectively. These are taken from Reference 1. The ultimate tensile strength (U.T.S.), the 0.2% tensile yield strength, and % plastic strain (or elongation) are given for five test temperatures.

Test Temperature (°R)	Percent Change in the Indicated Property for Both Fluences					
	U.T.S		0.2% Y.S.		%Plastic Strain	
	<u>2.4(17)</u>	<u>7.6(17)</u>	<u>2.4(17)</u>	<u>7.6(17)</u>	<u>2.4(17)</u>	<u>7.6(17)</u>
140	-3.5	-13.3	*	*	~-100	~-100
240	-3.5	-7.3	*	*	~-100	~-100
340	11.7	10.3	56.0	*	-86.8	-99.3
440	10.8	8.7	18.1	19.2	-8.3	-18.0
540	-0.05	1.4	0.3	6.9	12.2	-14.4

* Failure before 0.2% yield

IV. (Continued)

These changes are extrapolated to the GTR-20 fluences of 5×10^{18} and 1×10^{19} nvt by drawing straight lines on semi-log paper through the two points at 2.4×10^{17} and 7.6×10^{17} nvt from the above table. This extrapolation is very crude and should not be relied on too heavily. It may not even be a good assumption that the extrapolation has this type of behavior. It is merely to give a rough idea of what the properties might be in GTR-20. The extrapolations are given in the following table.

Test Temperature (°R)	Extrapolated Property Changes (%) (Extrapolated to 5×10^{18} and 1×10^{19} nvt)			
	U.T.S.		0.2% Y.S.	
	<u>5(18)</u>	<u>1(19)</u>	<u>5(18)</u>	<u>1(19)</u>
140	-29	-35		
240	-14	-17		
340	7	6		
440	6	4	21	22
540	4	5	17	21

Prediction of fracture toughness is based on a comparison of unirradiated toughness to that in GTR-19 References 2 and 7. Irradiation at 140°R to a fast ($E > 1$ MeV) neutron fluence of 5×10^{17} nvt decreased the fracture toughness of beryllium by approximately 40% for test temperatures below 340°R.

V. Experimental Procedures

A. Specimen Irradiation Fluence and Temperature

All specimens shall be irradiated at 140°R in LN₂. The following table shows the required neutron fluence for each beryllium specimen to be irradiated and the group it is in. These group numbers correspond to those in Appendix A, the tabulation of specimen groups.

<u>Specimen Group</u>	<u>Number of Specimens To Be Irradiated</u>	<u>Required Fast Neutron Fluence (nvt)</u>
A-I	24 (Including 8 spares)	5×10^{18}
A-I	12 (Including 4 spares)	1×10^{19}
A-II	32	5×10^{18}
A-III	12 (Including 4 spares)	5×10^{18}
A-IV	12 (Including 4 spares)	5×10^{18}
A-IV	8 (Including 4 spares)	1×10^{19}
A-V	16	5×10^{18}
A-VI	12	5×10^{18}

The specimens scheduled to receive the high dose of 1×10^{19} nvt shall be placed on the inner surface of the dewar nearest the North face of the core within a 5 inch radius circle, as shown in Figure 2.* The center of the circle represents the projection of the core center. The dashed lines indicate the regions containing brittle fracture toughness specimens, which are directly underneath (or slightly farther away than) the beryllium shoulder loaded

* Based on estimated flux distribution provided by General Dynamics, Fort Worth.

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V. (Continued)

specimens. The specimens scheduled to receive the low fluence of 5×10^{18} nvt shall be placed on a spherical surface (as shown in Figure 3) that is 5 inches North of the inner surface of the dewar on a horizontal line through the core center perpendicular to the core North face and intersects a 10 inch radius circle on the inner surface of the dewar. This surface may be modified slightly and its deviation from spherical defined as a result of further dosimetry analysis and calculation of the attenuation from the high fluence specimens. The neutron fluence for specimens with the same fluence requirement in any given group shall be within $\pm 5\%$. Whenever a group has two required fluences (for example group A-I has 24 specimens that require 5×10^{18} nvt and 12 specimens that require 1×10^{19} nvt), the ratio of high to low fluence shall be $2 \pm 10\%$. Variation in fluence between groups shall be within $\pm 30\%$.

Specimen dimensions are approximated in the following table to demonstrate the space each specimen will occupy. The table also gives the drawing number(s) associated with each specimen for the exact dimensions. These drawings are Figures 4-8.

V. (Continued)

<u>Specimen</u>	<u>Approximate Specimen Dimensions (Inches)</u>			<u>Drawing Number(s)</u>	<u>Number of Specimens at Indicated Neutron Fluence (nvt)</u>		<u>Number of Unirradiated Control Specimens</u>
	<u>Length</u>	<u>Width</u>	<u>Thickness or Diameter</u>		<u>Fast Fluence 5 x 10¹⁸</u>	<u>1 x 10¹⁹</u>	
Shoulder Loaded	2.0		0.49	650A577, 650A578	68	12	12
WOL	1.46	1.01	1.00	650A587	28	8	8
Stressed Tensile	8.86	-	2.35	WT701590	12	-	12

B. Tensile Testing (Instron Machine)

Specimen test temperature shall be controlled with mixture of LN₂ and ambient N₂. Each specimen shall be held at its planned test temperature for 15 minutes before starting the crosshead. This time is approximate and may be changed by the WANL monitor at the time of testing. The Instron machine crosshead speed shall be 0.005 inches/min. for all beryllium tensile testing. Alignment checks shall be made intermittently during beryllium testing program. This shall be accomplished with three strain gages mounted 120° apart around the circumference of the gage section of a dummy specimen. The Materials Advisory Board recommends ≤5% misalignment for beryllium. The WANL monitor will determine whether the alignment of the system is adequate.

V. (Continued)

C. Spring Loaded Beryllium Specimens

All 24 stressed specimens are to be strain gaged at WANL. Fixtures shall be shipped to GD/FW unloaded. They shall be assembled and proper alignment determined at GD/FW under WANL supervision as late as possible before insertion into irradiation dewars. The control specimens shall be held at LN₂ temperature during the irradiation. Before insertion into the dewar the spring compressed length shall be measured. After irradiation the compressed length, free length, and spring constant of all springs shall be measured at LN₂ temperature. These 12 specimens will then be tested at the conditions given in Appendix A.

D. Metallography

Macrographs with a magnification of 2 to 4 shall be taken of the fractured surface of specimens numbered 2, 10, 11, 17, 74, 76, 77, 80, 81, 84, 85, and 87. (See Appendix A for specimen numbering.)

Electron fractographs (having suggested magnification 6000) of the fractured surface and microphotographs (having magnification 100 and 500) of the polished longitudinal area including the fracture shall be taken of specimens numbered 3, 4, 11, 15, 74, 76, 77, 80, and 119. WANL shall receive replicas of the electron fractographs.

V. (Continued)

Each beryllium brittle fracture specimen (numbers 73-104) shall have a macrograph and a microphotograph of the fractured surface taken after fracture. The macrograph shall have a magnification of 10, while the microphotograph shall have a magnification of 200.

E. Data Reduction

The data to be taken shall be that outlined in Sections A, C, and D plus the following: The ultimate and 0.2% offset tensile yield strength, the Instron chart elongation, the plastic elongation at maximum stress, the total plastic elongation obtained with bench measurement, and reduction of area using photography methods and/or micrometer measurements. Data shall be based on actual WANL reported gage length. Original or legible reproduced copies of all load-deflection curves, temperature recording curves, and raw data tabulations are to be made available to WANL as soon after testing as possible.

F. Background Information

For the tensile and stressed tensile specimens in GTR-20 pressing number 4055 will be used. This same pressing was tested in GTR-18 and 19, which is discussed in Reference 1. Also vendor reported tensile data on unirradiated beryllium from pressing 4055 is given in Reference 8.

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V. (Continued)

Brittle fracture specimens in GTR-20 are from pressing 4263. This pressing has not been tested, but the same type of brittle fracture specimen, made from pressing 3840, has been tested and is discussed in Reference 7. The chemical composition and the density of pressing 4263 are given below.

1. Chemical Analysis

Beryllium	98.43
Beryllium Oxide	1.69
Carbon	.13
Aluminum	.07
Chromium	.008
Iron	.11
Magnesium	.006
Manganese	.01
Nickel	.015
Copper	.008
Silicon	.04
Titanium	.020

2. Density

1.856

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V. (Continued)

The fabrication procedure for beryllium is given here. The beryllium was obtained from the trepanned cores of actual reflector pressings. Briefly, the production of such pressings consists of ore reduction, vacuum melting of the metal, attritioning to powder and vacuum, hot-pressed sintering. The main ore, beryl, contains about 5% beryllium and is mined largely as a secondary product. Because of large grain size and poor as-cast properties, the metal is subsequently attritioned to an average particle size of 35 microns. The powder is then hot pressed at about 100 psi and at a temperature of 2000°F while under a vacuum of 50 microns. The chemical composition of typical, finished material is 98.3% beryllium (including that present in the oxide) with 1.8% beryllium oxide and about 0.4% additional metallic impurities. The following shows the composition, as well as density, for pressing 4055.

Total Be	BeO	Fe	Al	Mg	Si	C	Density (gm/cm ³)
98.33	1.85	0.13	0.07	0.01	0.03	0.09	1.859

The vendor reported tensile data at room temperature are given below for pressing 4055 for both longitudinal and transverse specimen orientations.

Ultimate Strength (KSI)		0.2% Yield Strength (KSI)		Elongation (%)	
Long.	Trans.	Long.	Trans.	Long.	Trans.
44.8	47.5	33.2	34.5	1.5	2.4

[REDACTED]

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V. (Continued)

These values are all greater than limits given in WANL-PDS-30018-E.
The microstructures are given in Figure 9 for both longitudinal and
transverse ring pressings.

G. Disposition of Spare Specimens

Spare specimens will be used to replace damaged specimens if
there are any. If spares are not used to replace damaged specimens
they will be used for replication at the most stringent test
temperature (140°R).

[REDACTED]

[REDACTED]

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APPENDIX A

The following pages give tabulation of all Beryllium specimens, the group number of each group, and the independent variables.

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A-I Beryllium - Plain Tensile

Test Conditions

<u>Specimen No.</u> & <u>Test Order</u>	<u>Core</u>	<u>Orientation</u>	<u>Fluence</u> (Fast) (nvt)	<u>Test</u> <u>Temperature</u> (°R)
11	↓ #4	Long.	0	140
29		↓	↓	406
15			5×10^{18}	140
36			↓	273
27			↓	406
3			↓	540
31			10^{19}	273
7			↓	540
33		Trans.	0	273
2		↓	↓	540
14			5×10^{18}	140
39			↓	273
21			↓	406
8			↓	540
20			10^{19}	140
22			↓	406
32	↓ #7	Long.	0	273
4			↓	540
19			5×10^{18}	140
34			↓	273

A-I Beryllium - Plain Tensile
(Continued)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Core</u>	<u>Orientation</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
30	↓ #7 ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓	Long.	5×10^{18}	406
5		↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓	↓	540
17			10^{19}	140
26			↓	406
18			0	140
25			↓	406
12			5×10^{18}	140
37			↓	273
23			↓	406
1			↓	540
38			10^{19}	273
10			↓	540

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Spare Specimens

A-I Beryllium - Plain Tensile

<u>Specimen No.</u>	<u>Core</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
395	#4	Long.	0
396		↓	5×10^{18}
397			↓
398			10^{19}
399		Trans.	0
400	↓	↓	5×10^{18}
401			↓
402		↓	10^{19}
403			0
404			5×10^{18}
405		↓	↓
406			10^{19}
407		Trans.	0
408		↓	5×10^{18}
409			↓
410			10^{19}

A-II Beryllium - Plain Tensile - Annealing Study
5 x 10¹⁸ nvt Fast Fluence

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Core</u>	<u>Orientation</u>	<u>Annealing Temperature (°R)</u>	<u>Annealing Time Minutes</u>
42	#4	Long.	273	1 (1)
71			↓	100 (3)
70			406	10 (2)
55			↓	1000 (4)
64			540	1 (1)
52				10 (2)
65				100 (3)
41			↓	1000 (4)
57		Trans.	273	10 (2)
62			↓	1000 (4)
67			406	1 (1)
61			↓	100 (3)
43			540	1 (1)
72				10 (2)
44				100 (3)
50			↓	1000 (4)
48	#7	Long.	273	10 (2)
49	↓	↓	↓	1000 (4)

A-II Beryllium - Plain Tensile - Annealing Study
 5×10^{18} nvt Fast Fluence
 (Continued)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Core</u>	<u>Orientation</u>	<u>Annealing Temperature (°R)</u>	<u>Annealing Time Minutes</u>
68	↓	Long.	406	1 (1)
66		↓	↓	100 (3)
59			540	1 (1)
63			↓	10 (2)
54				100 (3)
56		↓		1000 (4)
51		Trans.	273	1 (1)
58		↓	↓	100 (3)
45			406	10 (2)
47			↓	1000 (4)
46			540	1 (1)
60			↓	10 (2)
69				100 (3)
53				1000 (4)

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A-III Beryllium - Notched Tensile - 5×10^{18} Fluence (Fast) nvt
(To Be Combined with Some Data From I.)

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Core</u>	<u>Orientation</u>	<u>Test Temperature (°R)</u>
16	#4	Long.	140
28	#4	Long.	406
40	#4	Trans.	273
9	#4	Trans.	540
35	#7	Long.	273
6	#7	Long.	540
13	#7	Trans.	140
24	#7	Trans.	273

Spare Specimens

A-III Beryllium - Notched Tensile - All Specimens
Irradiated to 5×10^{18} n/cm² (E > 1 MeV)

<u>Specimen Number</u>	<u>Core</u>	<u>Orientation</u>
411	#4	Long.
412	#4	Trans.
413	#7	Long.
414	#7	Trans.

A-IV Beryllium - Brittle Fracture

Test Conditions

Specimen No. & Test Order	Orientation	Fluence (Fast) (nvt)	Test Temperature (°R)
77	Long.	0	140
81	↓	↓	406
80	↓	5×10^{18}	140
85	↓	↓	273
84	↓	↓	406
76	↓	↓	540
88	↓	10^{19}	273
75	↓	↓	540
87	Trans.	0	273
74	↓	↓	540
79	↓	5×10^{18}	140
86	↓	↓	273
82	↓	↓	406
73	↓	↓	540
78	↓	10^{19}	140
83	↓	↓	406

Spare Specimens

A-IV Beryllium Brittle Fracture - Ring Pressing Material

<u>Specimen Number</u>	<u>Core</u>	<u>Orientation</u>	<u>Fluence (nvt)</u>
415	#4	Long.	0
416	↓	↓	5×10^{18}
417		↓	10^{19}
418		Trans.	0
419		↓	5×10^{18}
420		↓	10^{19}
421	#7	Long.	0
422	↓	↓	5×10^{18}
423		↓	10^{19}
424		Trans.	0
425		↓	5×10^{18}
426		↓	10^{19}

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A-V Beryllium - Brittle Fracture - Annealing Study
 5×10^{18} nvt Fast Fluence

Test Conditions

<u>Specimen No.</u>	<u>Orientation</u>	<u>Annealing Temperature (°R)</u>	<u>Annealing Time (min.)</u>
92	Long.	273	5 (1)
94		↓	100 (3)
103		406	10 (2)
96		↓	1000 (4)
102		540	5 (1)
95		↓	10 (2)
97		↓	100 (3)
89		↓	1000 (4)
104	Trans.	273	10 (2)
101		↓	1000 (4)
90		406	5 (1)
98		↓	100 (3)
99		540	5 (1)
93		↓	10 (2)
91		↓	100 (3)
100		↓	1000 (4)

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A-VI Beryllium - Stressed Tensile

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Load KSI</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
120	12.5	0	140
128			340
108			540
118			140
123			340
111			540
113		5 x 10 ¹⁸	140
126			340
112			540
117			140
124			340
107			540
116	25.0	0	140
121			340
109			540
115			140
125			340
105			540

A-VI Beryllium - Stressed Tensile

Test Conditions

<u>Specimen No. & Test Order</u>	<u>Load KSI</u>	<u>Fluence (Fast) (nvt)</u>	<u>Test Temperature (°R)</u>
114	25.0	5×10^{18}	140
127			340
110			540
119			140
122			340
106	↓		540
129	0		140
130			140
131			140
132			140
	↓	↓

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FIGURE 1 R-1 Reactor Assembly

F-2

FIGURE 2 Loading of High Fluence Specimens

612531-8B

612531-7B

FIGURE 3 Loading of Low Fluence Specimens

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2- ALL DIAMETERS $\text{A} \text{ } .005 \text{ TIR}$

1- UNLESS OTHERWISE SPECIFIED TOLERANCES ARE:
.XXX = ±.005

CODE 3

FIGURE 4 Beryllium Shoulder Loaded Tensile Specimen

FIGURE 5 Beryllium Notched Shoulder Loaded Tensile Specimen

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1 ALL SIDES $1 A .002$

CODE 3

FIGURE 6 Beryllium Brittle Fracture (WOL) Specimen

RESTRICTED DATA

CONFIDENTIAL

FIGURE 7 Beryllium Stressed Tensile Specimen

RESTRICTED DATA

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RESTRICTED DATA
Atomic Energy Act - 1954

F-8

FIGURE 8 Photo of Beryllium Stressed Tensile Specimen

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Transverse

Longitudinal

FIGURE 9 Microstructures of Transverse and Longitudinal Ring Pressings

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