PROPERTIES OF IRRADIATED BERYLLIUM STATISTICAL EVALUATION

J. M. BEESTON

October 1976

IDAHO NATIONAL ENGINEERING LABORATORY

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STATISTICAL EVALUATION

J. M. Beeston

EG&G Idaho, Inc.

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PREPARED FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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PROPERTIES OF IRRADIATED BERYLLIUM - STATISTICAL EVALUATION*

ABSTRACT

Irradiated beryllium properties of tensile and compression fracture strength and growth or swelling were evaluated by statistical methods to determine the best estimate model from the data. The equations presented extend the properties to a fluence of $3 \times 10^{22} \text{ n/cm}^2$ $E > 1 \text{ MeV}$. The annealing behavior indicates a threshold temperature for 1% swelling of 425°C.

*Work performed under the auspices of the Energy Research and Development Administration under Contract EY-76-C-07-1570.

TABLE OF CONTENTS

	<u>Page</u>
1.0 Introduction	
1.1 Background	1
1.2 Objective	2
2.0 Materials and Irradiation Conditions	
2.1 Fluence Determination	4
2.2 Statistical Procedures	10
3.0 Growth	10
4.0 Compression Strength	15
5.0 Tensile Tests	
5.1 Flexure Tests	20
5.2 Splitting Tensile Tests	21
5.3 Analysis of Tensile Data	25
6.0 Annealed Density and Electron Microscopy	29
7.0 Summary	29
8.0 References	35

FIGURES

Figure 1. Fluence Contour Plots	
A. South Quadrant	5
B. West Quadrant	6
C. North Quadrant	7
D. East Quadrant	8
Figure 2. Growth Measurements on Beryllium Specimens	16
Figure 3. Compression Yield or Fracture Strength vs Fluence	17
Figure 4. Flexure Test Model	22

FIGURES (continued)

	<u>Page</u>
Figure 5. Failure Mode in Splitting Tensile Tests	23
Figure 6. Splitting Tensile Test Model	26
Figure 7. Tensile Strength as Function of Fluence	27
Figure 8. Annealed Density Changes	31
Figure 9. Bubbles Not Resolved	32
Figure 10. Bubbles Resolved	33

TABLES

Table I. Solid Chemistry of Hot Pressed Beryllium	3
Table II. Growth Measurements on Beryllium Specimens	12
Table III. Growth Measurements and Normalized Comparisons	14
Table IV. Compression Yield or Fracture Strength	19
Table V. Flexure Tests	24
Table VI. Splitting Tensile Tests	28
Table VII. Annealing Tests	30

1.0 INTRODUCTION

1.1 Background

The properties of irradiated beryllium are of interest in the determination of service life of reflectors for the ATR, and some components in other reactors (ETR, HFIR, BR-2). The first reflector was removed from the ATR in 1972 after the beryllium reflector cracked and the reactor had been operated to a planned safety analysis limit.⁽¹⁾ The safety analysis limit was related to the probability of additional cracking in other stressed regions of the reflector. The criterion for cracking is based upon the maximum stress theory which assumes failure when one of the principle stresses reaches the yield strength in simple tension or compression.⁽²⁾ The stress is principally the result of internal stresses from the transmutation product helium, since loading stresses including thermal stresses are generally small.

Although the failure mode is not limited to tensile loads, and a compressive loading could institute failure, it is the tensile strength which is the property of most concern for reflector designs and predicted lifetimes. The tensile stress in the ATR ligaments at the top or bottom of the reflector (low fluence regions) reaches the tensile yield strength for the limiting reflector life.⁽³⁾ For reflector III the stressed regions may change and perhaps compressive stress as well as tensile may limit the reflector life.⁽⁴⁾ The tensile strength is a difficult property to determine for a highly brittle material such as beryllium when irradiated to fluences above $0.5 \times 10^{22} \text{ n/cm}^2$ $E > 1 \text{ MeV}$ at temperatures of 100°C , since the alignment and grips introduce stresses resulting in premature failure. A method for combining flexural and splitting tensile tests has been reported,⁽⁵⁾ and contains the data with fluence limit of $2 \times 10^{22} \text{ n/cm}^2$.

A method for analysis of irradiation induced stresses in beryllium and relating these to the irradiation swelling has been reported.⁽⁶⁾ For the prediction of the service life the interaction of the results of these two methods is evaluated.

1.2 Objective

The objective of this report is to extend the tensile and compression property data of irradiated beryllium to higher fluence and include statistical evaluation of the data. The statistical analysis was performed upon the property data collected from specimens removed from the ATR. The analysis was conducted using the Statistical Analysis System (SAS) Regression Procedure (REGR) in the Idaho National Engineering Laboratory (INEL) IBM 360/75 computer.

2.0 Materials and Irradiation Conditions

The hot pressed beryllium specimens used in this investigation were from pressings prepared for the first ATR reflector. That material was identified as lots 2811 and 2870. From the reflector pressings cylindrical surveillance specimens were fabricated 0.4" diameter by 1.23" long. The specimens were measured, weighed, encapsulated, and irradiated. The specimens were machined to a surface finish of 63 microinch or finer. The specimens were encapsulated in either stainless steel with a NaK thermal bond or in aluminum splined tube design sealed in argon during tube closure. No effect of NaK versus argon environment has been detected on the property measurements.

The pressings were obtained from specifications prepared by Babcock and Wilcox for EBASCO as Specification M-182, "ATR Specification for Beryllium Pressings of ATR Internals" for a nominal 2% BeO and 98% beryllium.

The mechanical properties were specified as 40 ksi UTS, 30 ksi YS, and an elongation of 1 percent in 1 inch (minimum). A density of 1.84 g/cc was required. The solid chemistry of these lots is given in Table I.

TABLE I
SOLID CHEMISTRY OF HOT PRESSED BERYLLIUM

Element	Lot 2811 Content %	Lot 2870 Content %
Be	98.67	98.26
BeO	1.67	1.70
C	0.110	0.100
Al	0.100	0.085
Cr	0.011	0.010
Fe	0.130	0.153
Mg	0.004	0.006
Mn	0.016	0.012
Ni	0.014	0.014
Cu	0.009	0.008
Si	0.030	0.030
Ti	0.024	0.021

The irradiation was conducted in the ETR and the ATR. In the ETR the capsules were contained in a special aluminum filler piece placed in the core in position K-6 in each of sixteen 5/8 inch drilled holes. A thermocouple which penetrated the top of four of the NaK capsules indicated a temperature < 121°C. A temperature of < 121°C was the design temperature for both the ETR and the ATR irradiation. In the ATR the capsules were placed in the active core in A and H hole positions. The gamma heating in the ETR K-6 position was 8 watts/g, while that in ATR A and H holes

with center hole at 55 MW was 18.3 watts/gram. The maximum specimen centerline calculated temperature⁽⁷⁾ for ATR conditions was 75°C for the aluminum splined capsules and 93°C for the NaK bonded capsules.

Hot pressed beryllium with a density of 1.85 g/cm³ has a structure of equiaxed Be grains with fine oxide particles at the grain boundaries and through the grains. The metallic impurities which exceed the solubility limit are finely dispersed primarily along grain boundaries. The average grain size of the ATR reflector-1 beryllium was about 25 microns by the intercept method. During the sintering step the grains recrystallized and underwent slight growth.

2.1 Fluence Determination

The fluence was determined from flux wire monitors (Fe, Ni, Co) placed in four vertical holes in the ETR filler piece and in A and H holes in the ATR. In the ETR the radial flux gradient was accounted for by the four flux wire positions and the vertical gradient by the relative height of the specimen with respect to the flux wire map. A log of the specimen fluence in the ETR was prepared for all specimens. Upon transfer of the capsules to the ATR, the positions of the specimens in the vertical plane were identified and the fluence log updated by reference to the flux wire maps and a PDQ contour printout of group 1 flux contour plots. The terminal fluence was fixed by the reactor removal date and computed from the fluence log update.

The contour plots, Figure 1, A, B, C, and D, were produced from the integrated power history of the ATR and from the PDQ contour printout of group 1 flux contour plots for ATR cycle 24B, in the following manner:

1. From the integrated power history the average fraction of power in each lobe was calculated as: NW-.2362, NE-.1535, C-.2111, SW-.2378, SE-.1612 for cycles 15F thru 29C-1. The average power

24B(10) AT 220 MW VERT M/A : 1.40 S QUAD CP 1 FLUX

Figure 1. Fluence Contour Plots
A. South Quadrant

248(110) AT 220 MW .VERT M/A : 1.40 W QUAD GP 1 FLUX

24B(10) AT 220 MM VERT M/A : 1.40 N QUAD CP 1 FLUX

Figure 1. Fluence Contour Plots
C. North Quadrant

248(110) AT 220 MW VERT M/A : 1.40 E QUAD GP 1 FLUX

Figure 1. Fluence Contour Plots
D. East Quadrant

in each lobe (total 220 MW) would be: NW-51.97, NE-33.78, C-46.45, SW-52.33, SE-35.47 MW's.

2. The average power in each lobe (normalized to 250 MW total reactor power) was then compared at the same total reactor power to the list of ATR CSA calculations for cycles 15F thru 29C to select the PDQ power distribution most closely representative of the average lobe power. The variance was minimized and resulted in the selection of the PDQ case for ATR cycle 24B at 10 days. The lobe power (total reactor power 220 MW) was as follows: NW-51.48, NE-33.792, C-45.584, SW-53.68, SE-35.464 MW's. The fraction of power in each lobe was NW-.2340, NE-.1536, C-.2072, SW-.2440, and SE-.1612.
3. From the neutron flux measurements and PDQ plots at the same lobe power basis and in the same space position (peak flux), a normalization factor was calculated as

$$N. F. = \left(\frac{\phi \text{ measured at 44 MW}}{\phi \text{ PDQ at 44 MW}} \right) \left(\frac{\text{Power Lobe Ave.}}{\text{Power PDQ 24B}} \right)$$

All fast flux measurements were used to calculate an average flux at a space position as

$$\phi \text{ measured at 44 MW} = \frac{\sum \text{MWd's} \times \phi}{\sum \text{MWd's}}$$

4. The space positions and normalization factors are indicated on the flux maps. The lobe power shown corresponds to a total reactor power of 220 MW.
5. The effective time at 220 MW for cycles 15F thru 29C-1 (111, 168 MWd's) is 4.366×10^7 seconds.
6. The fluence for cycles 15F thru 29C-1 at a particular space position and the peak flux is calculated by multiplying the PDQ contour value in a lobe by the normalization factor and by the effective time.

7. Estimation of flux at another lobe power is obtained by multiplying by the ratio of the estimated lobe power to the average power of the lobe (Item I).

The absolute fluence is expected to differ from calculated values by less than 20%.

2.2 Statistical Procedures

The data were analyzed⁽⁸⁾ by the least squares technique to determine the best estimate model of the data. A regression analysis of variance was then performed to assess the overall significance of fitting the regression equation using the F statistic. The Student's t statistic was also used to test the hypothesis that each derived least squares coefficient was zero. All data listed were used in the final analysis.

The R value, or Pearson product-moment correlation coefficient given in each regression analysis is a measure of the variance due to regression and is defined as the square root of the regression sum of squares divided by the total sum of squares, and is hereafter referred to as the correlation coefficient.

A 99% prediction interval about the point estimated value $\hat{y} = b_0 + b_1x$ was calculated.

Five sets of data were analyzed to determine the relationship of fluence to the various properties of irradiated beryllium. These sets were:

1. Growth measurement or change in length as a function of fluence.
2. Flexure strength as a function of fluence.
3. Splitting tensile strength as a function of fluence.
4. Tensile strength as a function of fluence.
5. Compression strength as a function of fluence.

3.0 GROWTH

Pre-irradiation length and diameter measurements were made^(9, 10) to 20 microinches. After irradiation the measurements were repeated on

a Bausch and Lomb optical gage, DR-25B to the same precision. The tolerance range for the change in length (square root of the sum of the squares of the deviation of individual measurements) is then 5.7×10^{-5} inches or 0.0046% of the specimen length. These measurements should be more precise than those of density wherein the weight was measured to ± 0.1 milligram, and the water temperature was measured to the nearest degree Celsius, since the tolerance range for the density change is 4×10^{-4} g/cm³ or 0.022% of the specimen density. The data from 66 specimens is given in Table II. The change in length ($\Delta L/L$ %) measurement was not available for seven (7) data points. The change in density ($-\Delta\rho/3\rho$) measurement was utilized instead. Because the linear growth, $\frac{\Delta L}{L}$, determined from the length or diameter is approximately the same as seen in Table III (average value normalized to fluence of 0.2122 vs 0.2055) the growth is isotropic and the utilization of the change in density values for the missing change in length values was deemed valid.

It is noted that the density change, Table III, is less than for the length change. This difference is believed due to the presence of a corrosion product observed on the surface of some of the specimens. The procedure for removal of this corrosion product was to give those specimens showing a film a chemical polish in $H_2SO_4 - H_3PO_4 - CrO_3$ until 2 - 8 mils of the diameter was removed. This is a standard etch procedure for preparation of tensile specimens. In some cases all the oxide on the etched specimens has evidently not been removed, Table III, as the normalized dimensional change (per 10^{22} n/cm²) is lower (0.1539) on those specimens requiring the treatment than it is for those that did not (.1612). Those specimens with a metallic luster upon removal from the capsules were assumed to not need the treatment. The higher density from the presence of the oxide after irradiation results in less density change, hence the

TABLE II
GROWTH MEASUREMENTS ON BERYLLIUM SPECIMENS
(Nominal 2% Be)

Specimen No.	Fluence Units of 10^{22}	$\frac{\Delta L}{L}$ %
6	2.03	.415
7	1.93	.394
8	1.85	.404
9	1.68	.373
10	1.55	.324
16	1.82	.387
17	1.92	.393
18	1.99	.442
19	2.08	.458
20	2.14	.450
133	1.79	.284
144	1.82	.259
128	1.86	.249
123	1.38	.185
173	1.95	.365
134	1.77	.298
124	1.37	.252
113	1.30	.281
69	1.37	.301
61	1.90	.350
101	1.47	.273
71	1.30	.221
111	1.36	.350
65	1.41	.288
158	1.77	.367
177	2.57	.478
176	2.49	.498
175	2.38	.438
172	2.26	.402
169	2.13	.407
31	1.69	.280
32	1.76	.342
33	1.81	.298
34	1.87	.323
21	2.52	.456
22	2.45	.392
24	2.26	.386
25	2.16	.413
11	1.76	.326
12	1.80	.296

TABLE II (Continued)
GROWTH MEASUREMENTS ON BERYLLIUM SPECIMENS
(Nominal 2% Be)

Specimen No.	Fluence Units of 10^{22}	$\frac{\Delta L}{L}$ %
13	1.85	.316
14	1.88	.313
15	1.90	.320
73	2.767	.573
72	2.849	.538
66	2.899	.529
55	2.949	.595
52	2.979	.502*
120	3.130	.490*
119	3.159	.496*
118	3.165	.474*
117	3.168	.750
115	3.151	.711
161	2.968	.694
139	3.003	.400*
103	3.005	.505*
95	3.005	.438*
106	3.045	.617
104	3.211	.679
93	3.322	.802
86	3.432	.730
81	3.516	.685
169	3.024	.686
172	3.192	.685
175	3.325	.701
21	3.441	.704

* $\Delta L/L$ % measurement was not available so
density measurement used $(-\Delta\rho/3\rho)\%$.

TABLE III

GROWTH MEASUREMENTS AND NORMALIZED COMPARISONS

Specimen No.	Fluence (n/cm ²) x 10 ²² E > 1 MeV	$\frac{\Delta L}{L}$ %	$\frac{\Delta L}{L}$ % 10 ²²	$\frac{\Delta D}{D}$ %	$\frac{\Delta D}{D}$ % 10 ²²	Unetched		Etched		Specimen No.
						$\frac{1}{3} \left(\frac{-\Delta \rho}{\rho} \right)$ %	$\frac{1}{3} \left(\frac{-\Delta \rho}{\rho} \right)$ % 10 ²²	$\left(\frac{-\Delta \rho}{3\rho} \right)$	$\left(\frac{-\Delta \rho}{3\rho} \right)$ % 10 ²²	
73	2.767	.573	.207	.577	.2085	.437	.1579	.502	.1685	52
72	2.849	.538	.1888	.567	.1990	.455	.1597	.490	.1565	120
66	2.899	.529	.1824	.604	.2083	.463	.1597	.496	.1570	119
55	2.949	.595	.2017	.560	.1898	.450	.1526	.474	.1498	118
106	3.045	.617	.2026	.765	.2512	.546	.1793	.495	.1571	117
104	3.211	.679	.2114	.842	.2622	.533	.1660	.487	.1546	115
93	3.322	.802	.2414	.712	.2143	.540	.1626	.436	.1469	161
86	3.432	.730	.2127	.705	.2054	.565	.1646	.400	.1332	139
81	3.5156	.685	.1948	.680	.1934	.558	.1587	.505	.1681	103
169	3.024	.686	.2268	.549	.1815	.499	.1650	.438	.1458	95
172	3.192	.685	.2145	.337	.1682	.484	.1516			
175	3.325	.701	.2108	.614	.1846	.524	.1576			
117	3.168	.750	.2367							
115	3.151	.711	.2256							
161	2.962	.694	.2338							
21	2.441	.704	.2046							
AVERAGE	(16) .2122 ± .0170		(12) .2055 ± .0273		(12) .1612 ± .0072		(10) .1539 ± .0105			

dimensional change measured from density tends to be low. However, the larger volume of the oxide formed from the volume of metal tends to make the length measurements too high. The combination of the length and density data will produce a wider scatter as seen in Table II and Figure 2.

The geometric regression curve for the growth data extends the fluence to $3 \times 10^{22} \text{ n/cm}^2$ and gives a dimensional change, $\frac{\Delta L}{L} \%$, as

$$\frac{\Delta L}{L} \% = .1829 (\phi t)^{1.035} \quad \text{where } \phi t \text{ is in units of } 10^{22} \text{ n/cm}^2$$

The correlation coefficient for the data is 0.897 and the absolute standard error of estimate ($\sim \sigma$) is 0.066. The confidence interval for the unknown true $\frac{\Delta L}{L} \%$ values is illustrated, Figure 2, for the 99% confidence level. Thus we are 99% sure that the unknown true value is not greater or less than illustrated.

Extrapolation beyond the range of the data may be subject to error from a change in the growth mechanism, although none is apparent at this fluence. Because the expansion is isotropic as seen by the length and diameter measurements, the volume expansion is approximately given by

$$\frac{\Delta V}{V} \% = 3 \frac{\Delta L}{L} \% = .549 (\phi t)^{1.035}$$

4.0 COMPRESSION STRENGTH

The compression strength of six irradiated specimens was measured on a compression jig and die set as recommended in ASTM E9. The tests were made on an Instron at a crosshead speed of .005"/min. (strain rate of $\sim .004 \text{ in/in min.}$). The data are added to that given previously⁽⁵⁾ in Table IV. The data are plotted in Figure 3, and represented by the parabolic regression curve $S = 67.8 + 196.6 (\phi t) - 55.2 (\phi t)^2$, where ϕt is in units of 10^{22} n/cm^2 .

Figure 2. Growth Measurements on Beryllium Specimens

Figure 3. Compression Yield or Fracture Strength vs Fluence

Twenty-three points give a correlation coefficient of 0.896 with a standard error of estimate ($\sim$ sigma value) of 26.5 ksi. The 99% confidence level and standard error of estimate are illustrated in Figure 3.

The function gives a maximum strength of 242.9 ksi at a fluence of 1.78×10^{22} n/cm² $E > 1$ MeV.

It is not advised to extrapolate the data beyond a fluence of 3.0×10^{22} n/cm², since the two low values at a fluence of $3.3 - 3.5 \times 10^{22}$ n/cm² may be due to specimen alignment which becomes more critical as the fluence increases.

The fracture mode in compression was generally along planes parallel with the loading direction with a complex stress pattern at the platens. Failure occurred with the release of considerable energy, and the shards produced were quite fine and elongated confirming that planes of tensile stress are produced perpendicular to the axis of compression loading.

The finite element method of stress analysis has been used⁽¹¹⁾ to describe the stresses produced in cylindrical compression ceramic creep specimens. The tensile stresses are produced in the radial and hoop directions. Cavities and cracks were formed during creep testing which were associated with these tensile stresses. The cavities formed on boundaries parallel to the compression axis. The stress distribution is not uniform due to end constraints. The values of these radial and hoop tensile stresses are low compared with the compressive axial and shear stresses -- approximately 10 percent of the shear and 5 percent of the axial compressive stress.

5.0 TENSILE TESTS

The tensile properties of unirradiated nominal 2% BeO hot pressed beryllium are usually 30-40 ksi yield strength, 40-55 ksi ultimate strength, with 1.0 - 4.8 percent elongation. The tensile properties of irradiated

TABLE IV
COMPRESSION YIELD OR FRACTURE STRENGTH

Specimen No.	Fluence n/cm^2 $E > 1$ MeV	Yield or Fracture Strength (ksi)
Unirr.	0	38
166	0.24×10^{22}	127
160	0.23	131
153	0.50	159
3	0.45	159
4	0.50	165
151	0.47	174
82	0.51	126
57	0.81	178
148	1.02	198
90	1.05	178
77	1.13	223
109	1.13	238.1
69	1.37	249.0
124	1.37	213.4
133	1.79	242.5
25	2.16	200.2
72	2.85	218.0
73	2.77	198.4
86	3.43	78.2
93	3.32	58.3
117	3.17	158.0
139	3.00	202.2

hot pressed beryllium at high fluence are difficult to measure due to the embrittlement and notch sensitivity that occurs. At irradiation temperatures less than 450°C and fluences of $1-4 \times 10^{21}$ n/cm² E > 1 MeV, room temperature testing shows (12, 13, 14) that the yield strength is equal to the ultimate strength of about 50 ksi. Direct tensile values beyond this fluence have yet to be determined.

Methods for measuring the tensile strengths of brittle materials have involved direct and indirect tests. The indirect tests are conducted by flexure and by splitting tensile tests. An extension of these tests to higher fluence is made in this section, and all the data are analyzed by statistical methods to arrive at the most representative curve for the tensile behavior of highly irradiated beryllium.

5.1 Flexure Tests

The flexure strength of seven specimens was obtained by loading the cylindrical specimens in a three-point bend self-aligning-rolling fixture. The fixture was the same as described in ASTM E-399-72. The outer fiber tensile strength was calculated from the equation

$$\sigma_o = \frac{8PL}{\pi D^3}$$

where

P = applied load

L = span length = 1.125 inches

D = specimen diameter = 0.403 inches

Those specimens that showed the presence of a corrosion film were chemically polished prior to testing with the standard solution (H₂SO₄ - H₃PO₄ - CrO₃) used for machined tensile specimens. Although previous results⁽⁵⁾ showed no effect of the chemical polish, in these tests with fluences above

$2.5 \times 10^{22} \text{ n/cm}^2$ the chemical polish gave higher flexure strengths (three specimens), Table V. The data are plotted in Figure 4.

The linear regression curve for the twenty data points is given by

$$T = 76.8 - 13.8 (\phi t) \text{ where } \phi t \text{ is in units of } 10^{22} \text{ n/cm}^2$$

where ϕt is the fluence in units of 10^{22} n/cm^2 $E > 1 \text{ MeV}$. The correlation coefficient is 0.781 and the standard error of estimate is 7.78 ksi.

5.2 Splitting Tensile Tests

The splitting tensile or Brazilian test^(15, 16) induces a zone of tensile stress perpendicular to the plane of diameters in the test disc. If failure occurs in the tensile zone as the compression load is increased, then these tests can be regarded as indirect methods of measuring the tensile strength. It was shown that there are two regions in the specimen which are severely stressed: (a) a complex stress field directly below the loading area, (b) a zone of tension along most of the central plane of the vertical diameters. Failure can initiate in either area depending on loading conditions and specimen geometry. These effects are attributed to: (a) crushing at the line of application of the load, (b) misalignment and lack of uniform application of the load, and (c) crack initiation at other than the plane of maximum tensile stress. Ten tests were conducted and the data were added in Table VI to that obtained previously. The mode of fracture indicated that generally the tensile zone was formed since the specimens failed along the plane of diameters as indicated in Figure 5.

The strength was determined from the equation $S_o = \frac{2P}{\pi DL}$, where P is the load, D is the diameter, and L is the specimen length. The data are plotted in Figure 6. The strength and fluence were examined by linear regression analysis, which for 24 data points gives the equation

$$S_o = 39.3 - 4.73 (\phi t) \text{ where } \phi t \text{ is in units of } 10^{22} \text{ n/cm}^2$$

Figure 4. Flexure Test Model

Figure 5. Failure Mode in Splitting
Tensile Tests

TABLE V

FLEXURE TESTS

Specimen No.	Fluence 10^{22} n/cm^2 $E > 1$ MeV	Surface Condition*	Flexure Strength ksi	Normalized Flexure Strength $ksi-n/cm^2$ Units 10^{22}
8	1.85	U	48.0	88.8
9	1.68	U	43.4	72.9
111	1.36	U	68.0	92.5
6	2.03	U	54.8	111.2
101	1.47	U	58.2	85.6
144	1.82	U	56.6	103.0
66	2.90	U	29.3	85.0
55	2.95	U	25.8	76.1
106	3.05	U	33.0	100.7
104	3.21	U	31.1	<u>99.8</u>
AVERAGE				91.6
10	1.55	P	58.6	83.1
17	1.92	P	54.1	103.9
20	2.14	P	47.7	102.1
33	1.81	P	46.5	84.2
158	1.77	P	42.3	74.9
125	1.21	P	71.6	86.6
34	1.87	P	38.8	72.6
161	2.97	P	44.9	133.4
115	3.15	P	40.7	128.2
118	3.17	P	42.0	<u>133.1</u>
AVERAGE				100.2

*U means unpolished

P means chemically polished

with correlation coefficient of 0.686 and standard error of estimate of 4.63 ksi. The 99% confidence level is also shown in Figure 6.

5.3 Analysis of Tensile Data

The combination of the flexure and splitting tensile data for a proposed tensile strength curve are expected to give a larger variance than was seen in the data for flexure or splitting tensile taken separately. In the flexure test the outer fibers of the specimen are subjected to the higher breaking stress, consequently the defect distribution, the deformation due to plastic flow, and alignment and friction in the test fixture bearing edges will affect the measured strength which, because of the first two effects, will tend to be higher than in the direct tensile test. Since irradiated beryllium is very brittle the effect of plastic flow should be negligible and a self-aligning-rolling-fixture was used to reduce this effect. In the splitting tensile test, the measured strength tends to be less than the direct tensile strength due to crushing, alignment, and crack initiation effects. Any weighting factor for the incorporation of the indirect tests other than to give all the tests equal weight to produce a proposed curve is uncertain. It appears that to combine the data directly would give the best representation. Accordingly, the linear regression curve extends the tensile data to a fluence of $3 \times 10^{22} \text{ n/cm}^2$ and was determined as

$$S = 53.3 - 7.49 (\phi t)$$

where ϕt is in units of 10^{22} n/cm^2 with R of 0.47 and standard error of estimate of 11.4 ksi. The 99% confidence level is also shown in Figure 7. It is noted that this proposed curve does give equivalent values to the irradiated tensile strength at a fluence of $1 - 4 \times 10^{21} \text{ n/cm}^2$ of about 50 ksi.

Figure 6. Splitting Tensile Test
Model

Figure 7. Tensile Strength as
Function of Fluence

TABLE VI
SPLITTING TENSILE TESTS

Specimen No.	Fluence 10^{22} n/cm ²	Diameter D	Length L	Load P	Strength So (ksi)
100A	0.80	.3993	.2930	6380	34.7
B	0.80	.3990	.2890	6100	33.7
83A	1.02	.3999	.2925	4830	26.3
B	1.02	.3990	.2833	6050	34.1
114C	1.21	.4013	.2892	5320	29.5
D	1.21	.3995	.3234	6940	34.2
11A	1.76	.3972	.3892	8750	36.0
B	1.76	.3974	.4102	8750	34.2
32A	1.76	.4006	.288	5870	32.4
B	1.76	.4001	.2825	5800	32.7
61A	1.90	.3998	.2903	6830	37.5
B	1.90	.3997	.2868	5930	32.9
7A	1.93	.3985	.2960	6040	32.6
B	1.93	.3978	.2893	5600	31.0
52A	2.98	.4015	.277	3800	21.8
B	2.98	.4015	.272	5200	30.3
81A	3.52	.401	.351	4650	21.0
C	3.52	.401	.281	4100	23.2
95A	3.01	.3955	.292	3875	21.4
C	3.01	.396	.378	5000	21.3
103A	3.01	.398	.412	8350	32.4
B	3.01	.3988	.282	2575	14.6
120A	3.13	.402	.294	5475	29.5
B	3.13	.402	.382	4800	19.9

6.0 ANNEALED DENSITY AND ELECTRON MICROSCOPY

The immersion density was measured after annealing specimens at temperatures between 200 and 500°C for one hour. The irradiated specimens were cut in half and one half was used for the pre-anneal control for density and electron microscopy. The results of the density measurements are given in Table VII and Figure 8, where it can be seen that a temperature of 300°C is required before measureable swelling has occurred in the one hour anneal.

From the electron microscopy, Figures 9 and 10, it is seen that this swelling behavior can be correlated with the resolution of gas bubbles. At specimen capsule temperatures (~ 100°C) and after 1 hour anneal at 200°C the gas bubbles are not resolved, Figure 9. After annealing at 300°C the gas bubbles start to be resolved at about 30 angstroms, Figure 10, while after the 400°C anneal for one hour the diameter of the gas bubbles has increased to 60 Å, and after 500°C the diameter increased to 90 Å.

7.0 SUMMARY

Five sets of data from specimens irradiated at fluences between 2.7 and 3.5×10^{22} n/cm² were added to those previously analyzed to extend the relationship of fluence to the various strength and growth properties of irradiated beryllium. The relationships determined are as follows:

1. Growth measurement or change in length as a function of fluence, $\frac{\Delta L}{L} \% = 0.1829 (\phi t)^{1.035}$, standard deviation = $\pm .066\%$.
2. Flexure strength as a function of fluence, $T = 76.8 - 13.8 (\phi t)$, standard deviation = ± 7.8 ksi.

TABLE VII
ANNEALING TESTS

Spec. No.	Annealing Temp. °C	Fluence n/cm ² E > 1 MeV x 10 ²²	Pre-Irrad. Density g/cm ³	Pre-Annealed Density g/cm ³	Annealed Density g/cm ³	Density Change After Irrad. $-\frac{\Delta\rho}{\rho}\%$	Density Change After Anneal $-\frac{\Delta\rho}{\rho}\%$	Change From Anneal Density	Change Norm. To Fluence $-\frac{\Delta\rho}{\rho}\%$ 10 ²²
169	200	3.024	1.8508	1.8222	1.8223	1.55	1.54	None	0
172	300	3.192	1.8512	1.8235	1.8206	1.50	1.65	.15	.05
175	400	3.325	1.8511	1.8209	1.7914	1.63	3.23	1.6	.48
21	500	3.441	1.8523	1.8260	1.7507	- 1.42	- 5.49	- 4.07	- 1.18

Figure 8. Annealed Density Changes

A Specimen 120, Fluence of $3.1 \times 10^{22} \text{ n/cm}^2$
at $T \sim 100^\circ\text{C}$

B Specimen 169-A, Fluence $3.0 \times 10^{22} \text{ n/cm}^2$
at $T \sim 100^\circ\text{C}$

C Specimen 169-B, Fluence $3.0 \times 10^{22} \text{ n/cm}^2$
at $T = 200^\circ\text{C}$ for 1-hour

Figure 9. Bubbles Not Resolved

A Specimen 172-B Fluence of 3.2×10^{22} n/cm²
at T = 300°C for 1-hour Bubbles about
30 Angstroms Diameter

B Specimen 175-B, Fluence of 3.3×10^{22} n/cm²
at T = 400°C for 1-hour Bubbles about
60 Angstrom Diameter

C Specimen 21, Fluence of 3.4×10^{22} n/cm²
at T = 500°C. Bubbles about 90 Angstroms Diameter

Figure 10. Bubbles Resolved

3. Splitting tensile strength as a function of fluence, $T = 39.3 - 4.7 (\theta t)$, Standard deviation = ± 4.6 ksi.
4. Tensile strength as a function of fluence, $S = 53.3 - 7.5 (\theta t)$ standard deviation = ± 11.4 ksi.
5. Compression yield or fracture strength as a function of fluence, $S = 67.8 + 196.6 (\theta t) - 55.2 (\theta t)^2$, standard deviation = ± 26.5 ksi.

From one-hour annealing tests a threshold temperature of 300°C is required before measureable swelling occurs, and 425°C is required before 1% swelling occurs.

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