OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U. S. ATOMIC ENERGY COMMISSION

ORNL - TM - 1011

COPY NO. - 2

DATE - December 1, 1964

Properties of BeO Compacts Irradiated to Fast-Neutron Doses Greater Than 10^{21} Neutrons/cm^2 at 110, 650, and 1100°C

G. W. Keilholtz
J. E. Lee, Jr.
R. E. Moore

Abstract

The behavior of BeO sintered compacts toward fast-neutron doses exceeding 10^{21} neutrons/cm^2 (> 1 Mev) was studied at 110, 650 and 1100°C over the flux range 1.3 to 5.7 x 10^{14} neutrons/cm^2 sec. Four types of specimens consisting of two densities (2.7 and 2.9 g/cm^3) and two grain sizes (~ 22 and 34-72 μ) were included.

At 110°C, there was severe fracturing of specimens irradiated to fast-neutron doses greater than 10^{21} neutrons/cm^2, and most samples disintegrated to powder after irradiation to doses exceeding 2 x 10^{21} neutrons/cm^2. Volume expansion, a reliable criterion of neutron damage to BeO compacts, increased from 2.5 to 6% over the dose range 0.7 to 2.2 x 10^{21} neutrons/cm^2. In comparison, crystal volume expansion calculated from lattice parameters increased only from 1.25 to 3.5% over the same dose range, indicating that grain-boundary separation accounts for about 50% of the volume increase of compacts. Anisotropic crystal expansion is the principal mechanism causing grain boundary separation in BeO compacts irradiated at low temperatures or to high doses at high temperatures.

In-pile annealing of fast-neutron damage to BeO occurs to some extent at 650°C and is most effective at 1100°C, as shown by smaller crystal volume increases and smaller volume expansion of compacts. There is much less gross damage and volume expansion of BeO irradiated at 1100°C than at 650°C, although some specimens irradiated at 1100°C disintegrated to powder after exposures to fast-neutron doses greater than 4 x 10^{21} neutrons/cm^2. By comparisons with crystal volume expansions it can be concluded that a major portion of the volume expansion of compacts irradiated to high doses is contributed by grain-boundary

separation. This has been confirmed by micrographic examinations.

No flux intensity effect on gross damage, volume expansion or lattice parameter expansion is apparent from comparisons of data for samples irradiated at 650°C for different time periods at equivalent dose values. In irradiations at 1100°C, there is no apparent flux intensity effect on gross damage. Surprisingly, however, volume expansion was greater in low fluxes than in high fluxes for equivalent doses, a result which is difficult to explain as a radiation effect. A proposed explanation is that thermal cycling of the reactor can produce grain boundary separation in compacts with anisotropically strained grain boundaries. Accordingly, there could be more grain-boundary separation and consequently greater volume expansion in a long-term, low-flux irradiation than in a short-term, high-flux irradiation for equivalent fast-neutron doses if the long-term irradiation experienced a greater number of thermal cycles.

INTRODUCTION

The BeO-irradiation program at ORNL[1-9], now of several years duration, has been directed primarily toward the determination of the effects of high fast-neutron doses ($> 10^{21}$ neutrons/cm^2) at elevated temperatures (up to 1100°C) on pure BeO compacts without additives. One of the major objectives has been to evaluate the mechanisms of fast-neutron damage under conditions to which BeO moderator bodies may be subjected in nuclear power reactors.

Much of the large amount of data on the neutron irradiation of beryllium oxide, accumulated by many different investigators, cannot be compared satisfactorily because the data were obtained from BeO compacts made from different starting materials and fabricated by different methods. Also important, there may be sizable discrepancies in the flux, dose, and temperature values in different experimental assemblies and reactor systems. For these reasons, a series of experiments was designed to permit comparisons of radiation damage to BeO compacts of different grain-sizes and densities as a function of the flux, dose, and temperature under controlled conditions. This report presents the results of these experiments.

EXPERIMENTAL DESIGN

There were two sizes of sintered cylindrical specimens (1/2 in. length x 1/2 in. dia. and 1/4 in. length x 1/4 in. dia.) for each of four types of sintered BeO (two densities and two grain sizes). The characteristics of the eight groups of samples irradiated in the experimental assemblies are given in Table I. All the samples were prepared by the ORNL Metals and Ceramics Division from the same batch of Brush Beryllium Company UOX Grade BeO powder by cold-pressing and sintering at 1750°C in a hydrogen

atmosphere.[10] The material of small grain-size was sintered for one hour, and the material of large grain-size was sintered for 60 hours. The low-density BeO samples were made by including epoxy resin in the pressed material and removing it by heating at 900°C in air before sintering.

The specimens were encapsulated in stainless steel and irradiated in the Idaho Engineering Test Reactor at 110, 650, and 1100° C. Temperatures were achieved in each case by gamma heat generated within the capsules.

The arrangement of the capsules of Experiments 41-8, 41-9, and 41-10 is shown in Fig. 1. Each narrow bar represents a capsule containing 30 1/4-in. specimens; each wide bar represents a capsule containing 15 1/2-in. specimens. All four types of BeO samples were included in each of the capsules. Each of the eight capsules of Experiment 41-10 was maintained during irradiation at approximately 110°C. In both Experiments 41-8 and 41-9, which were irradiated for different periods of time in order to achieve a separation of the flux and dose parameters, four capsules were maintained at 650°C and four were maintained at 1100°C.

IRRADIATION OF BERYLLIUM OXIDE AT 110°C

The four types of BeO compacts were irradiated at approximately 110°C over the fast-neutron dose range 0.5 to 2.23 x 10^{21} neutrons/cm^2 (> 1 Mev) in Experiment 41-10. The fast-flux ranged from 1.3 to 5.7 x 10^{14} neutrons/cm^2 sec (> 1 Mev). The gross damage to specimens irradiated in this experiment is summarized in Table II. An examination of the damage data revealed no significant differences between types of BeO. Although major fracturing of Type II began at a lower neutron dose than other types, a larger number of specimens of Type II survived intact up to a dose of 2 x 10^{21} neutrons/cm^2.

Consequently, this difference is not considered to be significant. Nearly all specimens disintegrated to powder at doses above 2 x 10^{21} neutrons/cm^2.

Volume expansion calculated from dimensional increases of the irradiated samples was found to increase nearly linearly from about 1.25% at a fast-neutron dose of 0.5 x 10^{21} neutrons/cm^2 to about 5.5% at 1.4 x 10^{21} neutrons/cm^2. Figure 2 shows the volume expansion of samples which survived without fracturing or powdering plotted against fast-neutron dose. It is apparent that there are no significant differences in volume expansion among the four types of BeO irradiated in this experiment. Also shown in the plot is the volume expansion calculated from density measurements of single crystals irradiated in the same experimental assembly and the volume expansion calculated from the lattice parameters from X-ray diffraction examinations of powdered BeO compacts. Self-constraint in the relatively very large single crystals may account for the lower values found for their volume increases as calculated from density changes compared with values calculated from lattice expansion of powdered specimens. Obviously, grain boundary separation contributes importantly to the expansion of compacts irradiated to doses above ~ 0.8 x 10^{21} neutrons/cm^2 (> 1 Mev).

Two mechanisms have been proposed to explain grain-boundary separation in BeO compacts exposed to fast-neutron irradiation: (1) anisotropic expansion of randomly oriented crystals, resulting in stresses at the grain boundaries which ultimately overcome the intergranular bonding forces and (2) breaking apart of grain boundaries by gas pressure of helium, which diffuses to the grain boundaries after its production by neutron reactions within the grains. In low temperature irradiations (less than 250°C, for example) the evidence indicates that the helium mechanism cannot be significant. First, diffusion

of helium to the grain boundaries should be very slow at low temperatures. Second, grain boundary separation has been observed in photomicrographs of BeO irradiated at very low fast-neutron doses where very little helium has been produced[11,12]. On the other hand, X-ray diffraction patterns of powdered BeO specimens irradiated at 110°C in Experiment 41-10 show lattice parameter increases large enough to produce grain boundary separation; the anisotropic expansion ratio $(\Delta c/c_o)/(\Delta a/a_o)$ averages about 20 in these patterns.

The lattice parameters of specimens irradiated at 110°C are given in Table III. These data were obtained from measurements of the 21·1 and 21·0 reflections using Ni-radiation, and represent the average c and a parameters including the agglomerations of defects between some of the planes[13]. Values of lattice parameters reported for specimens of previous experiments[6] were calculated from reflections observed as diffuse maxima, but which represent spacings between those planes which do not include defect agglomerates.

IRRADIATION OF BERYLLIUM OXIDE AT 650 AND 1100°C

In each of the high temperature experiments (41-8 and 41-9), the four types of BeO were irradiated at both 650 and 1100°C. The two experiments were similar except that the irradiation time of Experiment 41-8 was about twice that of Experiment 41-9. The purpose of this procedure was to permit comparisons of the radiation damage to specimens of the same types irradiated to the same fast-neutron doses for different fast-neutron flux values. In conjunction with the irradiation experiments, out-of-pile control tests were carried out to determine the effect of thermal treatment alone on BeO specimens of these types.

Out-of-Pile Control Tests

Specimens of the four types of BeO were encapsulated in stainless steel and subjected to essentially the same thermal treatment out-of-pile that these types of specimens received during the irradiation of Experiments 41-8 and 41-9 at 650 and 1100°C. The total irradiation times (hours at 175 megawatts in the ETR) for Experiments 41-8 and 41-9 were 3573 and 1501 hours, respectively, and there were 45 thermal cycles of the ETR during the irradiation of Experiment 41-9 and 132 cycles during the irradiation of Experiment 41-8. The out-of-pile tests were planned to correspond with these times and to include the thermal cycling.

Examinations of the specimens after conclusion of the tests showed no gross fracturing or significant dimensional changes as a result of the thermal treatment at either 650 or 1100°C. Photomicrographs of the samples in the as-polished condition at 100X revealed no grain-boundary separation or transgranular fracture. The out-of-pile tests do not, of course, provide a temperature differential necessary for determining the effect of thermal stress, which may be important in irradiation studies.

Gross Damage to Irradiated Specimens

The experimental conditions and the gross damage data (fracturing and powdering) for the four types of half-inch BeO specimens irradiated in Experiments 41-8 and 41-9 are summarized in the bar graph of Fig. 3. Each of the bars covers the range of fast-neutron exposure of the specimens of the type represented. The boundaries of the damage regions shown in the bar graph are approximate. Some samples survived without visible gross damage, although in a greatly weakened condition, even in fast-neutron dose ranges

where most samples of the same type were severely fractured or powdered. Post-irradiation removal of the thick steel cladding required to achieve the necessary gamma heating of the capsules containing the quarter-inch samples proved to be very difficult. The gross damage data for these samples are not presented because many were probably damaged during the disassembly process.

Several conclusions may be drawn from the data summarized in Fig. 3:

1. The gross damage, which increases with increasing dose, is greater at 650 than at 1100°C for all four types of BeO.
2. Powdering of BeO compacts, which previously has been observed only in low temperature irradiations, can occur at temperatures as high as 1100°C after exposure to doses greater than 4×10^{21} neutrons/cm^2 (> 1 Mev).
3. In general, Type I BeO (low density-small grain size) withstood irradiation better than the other types in both experiments, while Type IV BeO (high density-large grain size) was generally damaged to a greater extent than the other types, particularly in irradiations at 650°C
4. Unexpectedly, there is no indication that damage is greater to samples irradiated in high fast-neutron fluxes than to samples irradiated in low fluxes at equivalent fast-neutron doses.

There is less damage to samples irradiated at 650°C in Experiment 41-9 than to samples irradiated at 110°C in Experiment 41-10 (see Table II). This indicates that in-pile annealing occurs even at temperatures as low as 650°C. Therefore, the absence of a flux intensity effect on gross damage must be related to reactor operational variables such as thermal cycling or thermal stress.

Volume Expansion of Irradiated Specimens

Volume expansion of irradiated BeO compacts has proved to be a good criterion of fast-neutron damage under a wide variety of experimental conditions.[1-9] The increase in volume of the half-inch BeO compacts which survived irradiation without severe fracturing in Experiments 41-9 and 41-8 are plotted against fast-neutron dose in Figs. 4 and 5, respectively. The volume expansion of the quarter-inch specimens is not included because the dimensional data for these specimens were not consistently reliable.

As can be seen in Fig. 4, the specimens irradiated at 650°C in the short term experiment (41-9) expanded less than specimens irradiated at 110°C in Experiment 41-10, but considerably more than the samples irradiated at 1100°C. There were no definite differences between BeO Type I, II, and III at 650°C; there was no survival of Type IV BeO. In irradiations at 1100°C, however, it is clear that Type I BeO (low density-small grain size) expanded much less than Type IV (high density-large grain size). Types II and III were intermediate in expansion.

There was no survival in irradiations at 650°C in the long term experiment (41-8). The volume expansion of the four types of samples irradiated at 1100°C, shown in Fig. 5, are in the same order as in Experiment 41-9, with Type I expanding the least, Type IV expanding the most, and Types II and III intermediate. As in the case for Experiment 41-9, the volume expansion increases with increasing fast-neutron dose.

If the volume expansions for irradiations at 1100°C in the two experiments are compared at equivalent dose values (see Table IV), it is apparent that there is greater expansion in Experiment 41-8 than in Experiment 41-9. The volume increases of Type I, Type III, and Type IV BeO in both

experiments are plotted against the fast-neutron dose in Figs. 6, 7, and 8, respectively. This unexpected finding of greater expansion in lower fluxes for equivalent doses can possibly be explained as an effect resulting from reactor operational variables such as thermal cycling. For instance, the long term experimental assembly (41-8) experienced three times the number of thermal cycles during irradiation as did the short term assembly (41-9). Thermal cycling of anisotropically strained grain boundaries in a BeO compact may result, therefore, in fracturing of some of the boundaries with each cycle. A greater number of thermal cycles during an irradiation could result, consequently, in greater expansion caused by grain boundary separation.

Micrographic Examinations

Photomicrographs of as-polished specimens clearly demonstrate that grain-boundary separation is the primary mode of fast-neutron damage in BeO compacts irradiated at low temperatures or to high doses at high temperatures. Transgranular fracture also occurs to some extent, especially in specimens of large grain size (~ 70μ). Grain boundary separation in irradiated specimens is illustrated in Figs. 9 and 10. Figure 9 shows photomicrographs comparing unirradiated BeO of high density (2.9 g/cm^3) and small grain-size (23 μ) with BeO of the same type which was irradiated at 1100°C to a fast-neutron dose of 3.92×10^{21} neutrons/cm^2. Extensive grain boundary separation can be seen. There is no significant reduction in apparent grain size from that of the unirradiated BeO, indicating that transgranular fracture is not severe. Figure 10 shows high density BeO of large grain size (70 μ) irradiated to a dose of 3.4×10^{21} neutrons/cm^2 (> 1 Mev) at 1100°C. Separation between grains appears to be wider than in the small-grained BeO, and there is evidence of a considerable amount of transgranular fracture. There is an apparent grain-size reduction of about 25% as a result of the transgranular fracture.

X-Ray Diffraction Examinations

Results of X-ray diffraction examinations[13] of selected BeO samples irradiated at 650 and 1100°C in Experiments 41-8 and 41-9 are presented in Table V. The values for the c parameter were calculated from measurements of the 21·1 and 21·0 reflections from Ni-radiation from BeO compacts ground to a fine powder.

Almost all the volume expansion calculated from the lattice parameters results from the increase in the c parameter; the a parameter increase is negligible in irradiations at 650 and 1100°C. The anisotropic volume increase calculated from the lattice parameters of samples irradiated at 650°C is considerably less than that of samples irradiated at 110°C (see Table III), but it is large enough (1.4 to 2.2%) to cause grain boundary separation observed in photomicrographs of these samples. The increased rate of in-pile annealing at 1100°C substantially reduces the c parameter increases from the values found for samples irradiated at 650°C. There is a question whether the small anisotropic volume expansion (0.02-0.34%) calculated from lattice parameters of samples irradiated at 1100°C can cause the grain boundary separation observed in photomicrographs.

Comparisons of different BeO types in Table V provide no indication, within the precision of the data, that density and grain-size have a bearing on lattice parameter increase. No flux intensity effect at 650°C is apparent from comparisons of data from the two experiments at comparable neutron doses. Because in-pile annealing at 1100°C should be very rapid, a marked flux intensity effect would be expected. The lattice parameter measurements on samples irradiated at 1100°C, however, are not precise enough to demonstrate this effect conclusively.

Single Crystal Studies[13]

Single crystals of beryllium oxide were irradiated in Experiments 41-8, 41-9, and 41-10 together with BeO compacts. The single crystals irradiated at 650°C in Experiment 41-9 exhibited a dark banding parallel to the basal planes which was not seen in crystals irradiated at 110°C in Experiment 41-10. The crystals irradiated at 650°C were found to be quite fragile, fracturing easily along the basal planes. Optical and X-ray diffraction data showed that the material in the dark banding is isotropic and amorphous, which strongly suggests that it consists of very large regions of BeO defect agglomerates. These regions are so large that they would not contribute to the values obtained for the c parameter through X-ray diffraction examinations. Single crystals irradiated at 1100°C show some striations, but very little as compared with crystals irradiated at 650°C. Apparently, at 1100°C in-pile annealing of point defects is so rapid that very little long-range agglomeration can occur.

In Fig. 11, No. 2 shows the dark banding in a crystal irradiated at 650°C to a fast-neutron dose of 1.1×10^{21} neutrons/cm^2; No. 4 is a crystal irradiated at 650°C to a dose of 4.1×10^{21} neutrons/cm^2 in which the dark banding appears to fill the crystal almost completely; No. 3 is a crystal irradiated at 54°C to 3.6×10^{21} neutrons/cm^2; and No. 1 is an unirradiated crystal.

Single crystals irradiated at 650°C to fast-neutron doses of 1.1 and 4.1 neutrons/cm^2 expanded in volume by 0.9 and 3.8%, respectively, as determined from density measurements. Irradiations of single crystals at 1100°C to fast-neutron doses of 1.5 and 5.7×10^{21} neutrons/cm^2 produced

volume increases of 0.6 and 0.9%, respectively. These volume increases are substantially greater than those calculated from the lattice parameter increases of powdered compacts irradiated to comparable doses. The differences may be attributed to contributions of defect agglomerates in planes perpendicular to the c axis which are much too large to be included in the c parameter expansions as measured by X-ray diffraction. These agglomerates, nevertheless, would produce an additional anisotropic strain at the grain boundaries.

DISCUSSION AND SUMMARY

Irradiation of sintered beryllium oxide compacts in high fast-neutron fluxes produces crystal damage through production of point defects, as indicated by lattice parameter expansion, and leads ultimately to grain boundary separation and fracturing and powdering of the material. In irradiations at ~ 100°C, a temperature at which in-pile annealing of point defects is insignificant, the anisotropic crystal volume expansion increases from about 1.25% to about 3.5% over the fast-neutron dose range 0.7 to 2.2 x 10^{21} neutrons/cm^2, which produces grain boundary separation in the compacts. The grain-boundary separation produces an additional volume expansion of irradiated compacts. The total volume expansion of compacts irradiated over this dose range increases from 2.5 to 6%. Many specimens irradiated to doses greater than 10^{21} neutrons/cm^2 fractured, and most samples disintegrated to powder above 2 x 10^{21} neutrons/cm^2.

In-pile annealing of point defects occurs to some extent in irradiations at 650°C, as evidenced by a reduction in the lattice parameter expansion compared with irradiations at 110°C. There is less total volume increase and gross damage at 650 than at 110°C. Although

gross damage data for irradiations at 110°C showed no significant differences among the four grain size-density combinations, in irradiations at 650°C, BeO compacts of low density and small grain-size withstood irradiation better than other types, and BeO of high density and large grain-size was damaged to a greater extent than the other types. No indications of a flux intensity effect on gross damage can be found in comparisons of results of the long term experiment with those of the short-term experiment.

The gross damage and total volume expansion of BeO compacts irradiated at 1100°C is less than that at 650°C. The beryllium oxide compacts of low density and small grain-size are damaged less and expand less than the other types, and the compacts of high density and large grain-size are damaged to the greatest extent and expand the most. Very small crystal volume increases were found in samples irradiated at 1100°C where in-pile annealing must be quite effective. The anisotropic volume expansion at 1100°C calculated from lattice parameters ranges from 0.02 to 0.34%. Density measurements of single crystals irradiated at 1100°C, however, give values for volume expansion as high as 0.9%. The additional expansion is probably a result of defect agglomeration. There is a question as to whether crystal volume expansion even as great as 0.9% can produce the amount of grain boundary separation and volume increase observed in the compacts irradiated at 1100°C. For example, crystal volume expansion greater than 1% must occur before grain boundary separation begins in irradiations at ~ 100°C.

If the gross damage to samples irradiated at 1100°C in the long term experiment and the short-term experiment are compared at equivalent

fast-neutron dose values, no indication of a flux intensity effect can be seen. If anything, there is greater damage in low fluxes than in high fluxes. The data for volume expansion clearly show, however, that there is greater expansion in low fluxes. This unexpected result is difficult to explain as a radiation effect. The explanation may be that reactor operational variables such as thermal cycling cause grain-boundary separation in samples irradiated at high temperatures. There may be more grain-boundary separation in long-term experiments with a greater number of reactor thermal cycles than in short-term experiments. This explanation could also account for the unexpectedly large amount of grain-boundary separation in samples irradiated at 1100°C relative to the small amount of crystal expansion observed. If there is a flux intensity effect on crystal expansion at 1100°C, it is obscured by the grain-boundary effect.

ACKNOWLEDGEMENTS

The authors are grateful to G. M. Watson for his encouragement and helpful discussions covering all phases of this work. The authors gratefully acknowledge the contributions of the following individuals at the Oak Ridge National Laboratory: A. F. Zulliger and G. H. Llewellyn, engineering design of experimental assemblies; H. L. Yakel, G. W. Clark and R. M. Steele, X-ray diffraction examinations and single crystal studies; E. L. Long, Jr. and E. J. Manthos, micrographic examinations.

References

1. R. P. Shields, J. E. Lee, Jr., and W. E. Browning, Jr., "Effects of Fast-Neutron Irradiation and High Temperature on Beryllium Oxide", Oak Ridge National Laboratory Report ORNL-3164, March 16, 1962.

2. R. P. Shields, J. E. Lee, Jr., and W. E. Browning, Jr., Trans. Am. Nucl. Soc. 4 (2) 338 (November 1961).

3. G. W. Keilholtz, J. E. Lee, Jr., R. P. Shields, and W. E. Browning, Jr., "Radiation Damage in BeO", Proc. Symp. Radiation Damage in Solids and Reactor Materials, Venice, May 7-11, 1962, Vol. II (Vienna, IAEA, 1962).

4. G. W. Keilholtz, J. E. Lee, Jr., and R. E. Moore, "The Effect of Fast-Neutron Irradiation on Beryllium Oxide Compacts at High Temperatures", Oak Ridge National Laboratory Report ORNL-TM-741, December 11, 1963.

5. G. W. Keilholtz, J. E. Lee, Jr., R. E. Moore, and R. L. Hamner, "Behavior of BeO under Neutron Irradiation", Oak Ridge National Laboratory Report ORNL-TM-742, December 11, 1963.

6. G. W. Keilholtz, J. E. Lee, Jr., and R. E. Moore, "The Effect of Fast-Neutron Irradiation on Beryllium Oxide Compacts at High Temperatures", J. Nuclear Materials 11 (3) 253-264 (1964).

7. G. W. Keilholtz, J. E. Lee, Jr., R. E. Moore, and R. L. Hamner, "Behavior of BeO under Neutron Irradiation", J. Nuclear Materials, Proceedings of BeO Meeting, October 1963, Sydney, Australia (in press).

8. G. W. Keilholtz, J. E. Lee, Jr., and R. E. Moore, "Behavior of Beryllium Oxide Compacts under Fast-Neutron Irradiation", pp. 264-275, GCRP Semiannual Prog. Report, September 30, 1963, USAEC Report

ORNL-3523, Oak Ridge National Laboratory.

9. G. W. Keilholtz, J. E. Lee, Jr., and R. E. Moore, "Behavior of Beryllium Oxide Compacts under Fast-Neutron Irradiation", pp. 196-200, GCRP Semiannual Prog. Report March 31, 1964, USAEC Report ORNL-3619, Oak Ridge National Laboratory.

10. R. L. Hamner, "Fabrication and Characterization of High-Purity Beryllium Oxide Specimens for Irradiation Testing", Oak Ridge National Laboratory Report ORNL-TM-767, March 1964.

11. J. Elston, "Radiation Damage in Solids", Proc. Symp. Radiation Damage in Solids and Reactor Materials, Venice, May 7-11, 1962, Vol. II (Vienna, IAEA, 1962).

12. High Temperature Materials Program Progress Report, No. 12, Part A, Nuclear Materials and Propulsion Operation, Flight Propulsion Laboratory Department, General Electric, Cincinnati (USA) Report GEMP-21A, June 15, 1962.

13. H. L. Yakel and co-workers, Metals and Ceramics Division, Oak Ridge National Laboratory, personal communications.

Table I. Characteristics of Beryllium Oxide Specimens Irradiated in Experiments 41-8, 41-9, and 41-10

BeO Type		Batch Number	Specimen Size (in.)	Average Bulk Density (g/cm^3)	Average Grain Size (μ)
I.	(Low density, small grain size)	A11	0.25	2.7	24
		A18	0.5	2.7	17
II.	(Low density, large grain size)	A13	0.25	2.7	60
		A19	0.5	2.7	34
III.	(High density, small grain size)	A10	0.25	2.9	23
		A16	0.5	2.9	25
IV.	(High density, large grain size)	A15	0.25	2.9	71
		A17	0.5	2.95	74

Table II. Gross Damage to Half-inch BeO Specimens Irradiated in Experiment 41-10 (~ 110°C)

BeO Type		Fast-Neutron Dose Range (neutrons/cm^2)	Fast-Neutron Dose for Minor Fracturing (neutrons/cm^2)	Fast-Neutron Dose for Major Fracturing (neutrons/cm^2)	Fast-Neutron Dose for Major Fracturing with Powdering (neutrons/cm^2)
		x 10^{21}	x 10^{21}	x 10^{21}	x 10^{21}
I.	(Low density, small grain size)	0.61-2.23	1.1	1.6	2.0
II.	Low density, large grain size)	0.56-2.23	-	0.56	2.0
III.	(High density, small grain size)	0.67-2.2	-	1.3	2.0
IV.	(High density, large grain size)	0.5-2.22	-	1.5	2.1

Table III. Results of X-ray Diffraction Examination of BeO Irradiated at 1100°C in Experiment 41-10*

BeO Type	Fast-Neutron Dose (> 1 Mev) (neutrons/cm^2)	Fast-Neutron Flux (> 1 Mev) (neutrons/cm^2 sec)	$\Delta a/a_o$	$\Delta c/c_o$	$\Delta V/V_o$ **
	x 10^{21}	x 10^{14}			
IV	0.7	1.8	0.0010	0.0100	0.0120
I	1.67	4.3	0.0012	0.0256	0.0280
IV	2.22	5.7	0.0013	0.0298	0.0324
I	2.23	5.7	0.0013	0.0326	0.0352

* Lattice parameters were calculated from measurements of the 21·1 and 21·0 reflections from Ni-radiation from BeO compacts which were ground to a fine powder.

** The fractional volume increase, $\Delta V/V_o$, was calculated from the equation $\Delta V/V_o = 2(\Delta a/a_o) + (\Delta c/c_o)$.

Table IV. Volume Expansion of Half-Inch BeO Specimens Irradiated in Experiments 41-8 and 41-9 at 1100°C*

BeO Type	Experiment	Percent Volume Increase at Fast-Neutron Dose		
		2.0 x 10^{21} neutrons/cm^2	4.0 x 10^{21} neutrons/cm^2	7.2 x 10^{21} neutrons/cm^2
I	41-8	2.0	2.2	2.4
I	41-9	0.7	1.4	-
III	41-8	2.8	3.1	3.5
III	41-9	1.3	2.6	-
IV	41-8	4.0	4.3	4.7
IV	41-9	1.8	3.6	-

* The values of volume increase at the three neutron doses were interpolated from linear data plots. Type II was omitted because there were too little data available, but the expansion appears to lie between Types I and IV.

Table V. Results of X-ray Diffraction Examination of BeO Irradiated at 650 and 1100°C*

Experiment	BeO Type	Fast-Neutron Dose (> 1 Mev) (neutrons/cm^2) x 10^{21}	Fast-Neutron Flux (> 1 Mev) (neutrons/cm^2 sec) x 10^{14}	Temp. (°C)	$\Delta a/a_o$	$\Delta c/c_o$	$\Delta V/V_o$ **
41-9	IV	1.4	1.9	650	0	0.0150	0.0150
41-9	I	1.65	2.25	650	0	0.0158	0.0158
41-9	I	3.65	5.0	650	0.0001	0.0140	0.0142
41-9	IV	3.7	5.1	650	0	0.0226	0.0226
41-8	IV	2.2	1.6	650	0.0001	0.0152	0.0154
41-8	II	2.85	2.05	650	0	0.0114	0.0114
41-8	II	3.1	2.2	650	0	0.0205	0.0205
41-8	II	3.3	2.35	650	0	0.0191	0.0191
41-8	II	3.6	2.6	650	0	0.0194	0.0194
41-8	I	5.1	3.65	650	0	0.0212	0.0212
41-8	IV	7.95	5.7	650	0.0004	0.0209	0.0217
41-8	I	8.1	5.8	650	0.0005	0.0204	0.0214
41-9	I	1.65	2.25	1100	0	0	0
41-9	IV	1.8	2.45	1100	0	0	0
41-9	IV	4.0	5.5	1100	0	0.0019	0.0019
41-9	I	4.0	5.5	1100	0	0.0034	0.0034
41-8	II	1.65	1.2	1100	0.0001	0.0016	0.0018
41-8	IV	3.2	2.3	1100	0.0001	0	0.0002
41-8	IV	5.5	3.9	1100	0	0.0028	0.0028
41-8	I	5.9	4.2	1100	0.0001	0	0.0002

* Lattice parameters were calculated from measurements of the 21·1 and 21·0 reflections from Ni-radiation from BeO compacts irradiated in Experiments 41-8 and 41-9 which were ground to a fine powder.

** The fractional volume increase, $\Delta V/V_o$, was calculated from the equation

$$\Delta V/V_o = 2\,\Delta a/a_o + \Delta c/c_o.$$

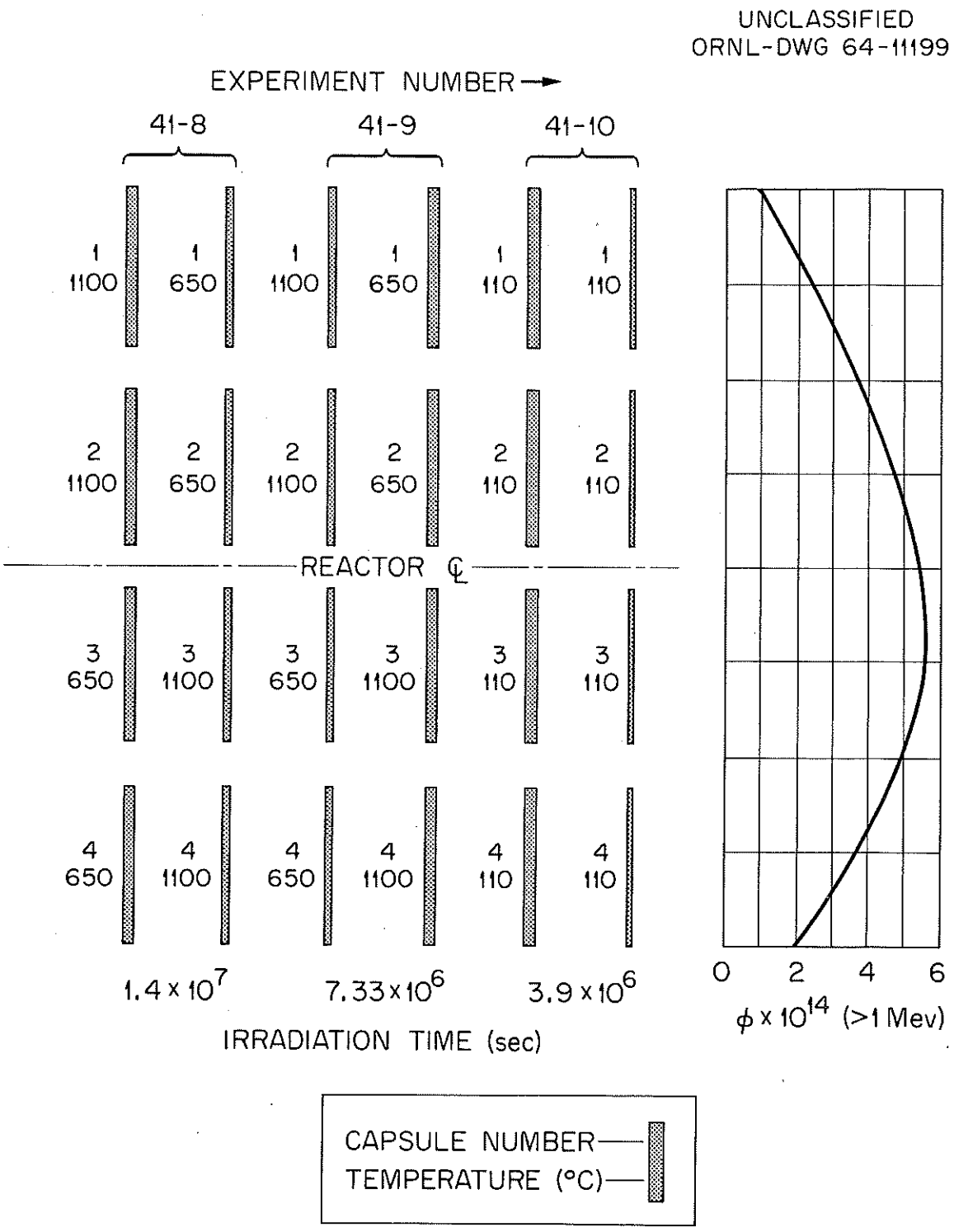


Fig. 1. ORNL BeO Irradiation Experiments 41-8, 41-9, and 41-10.

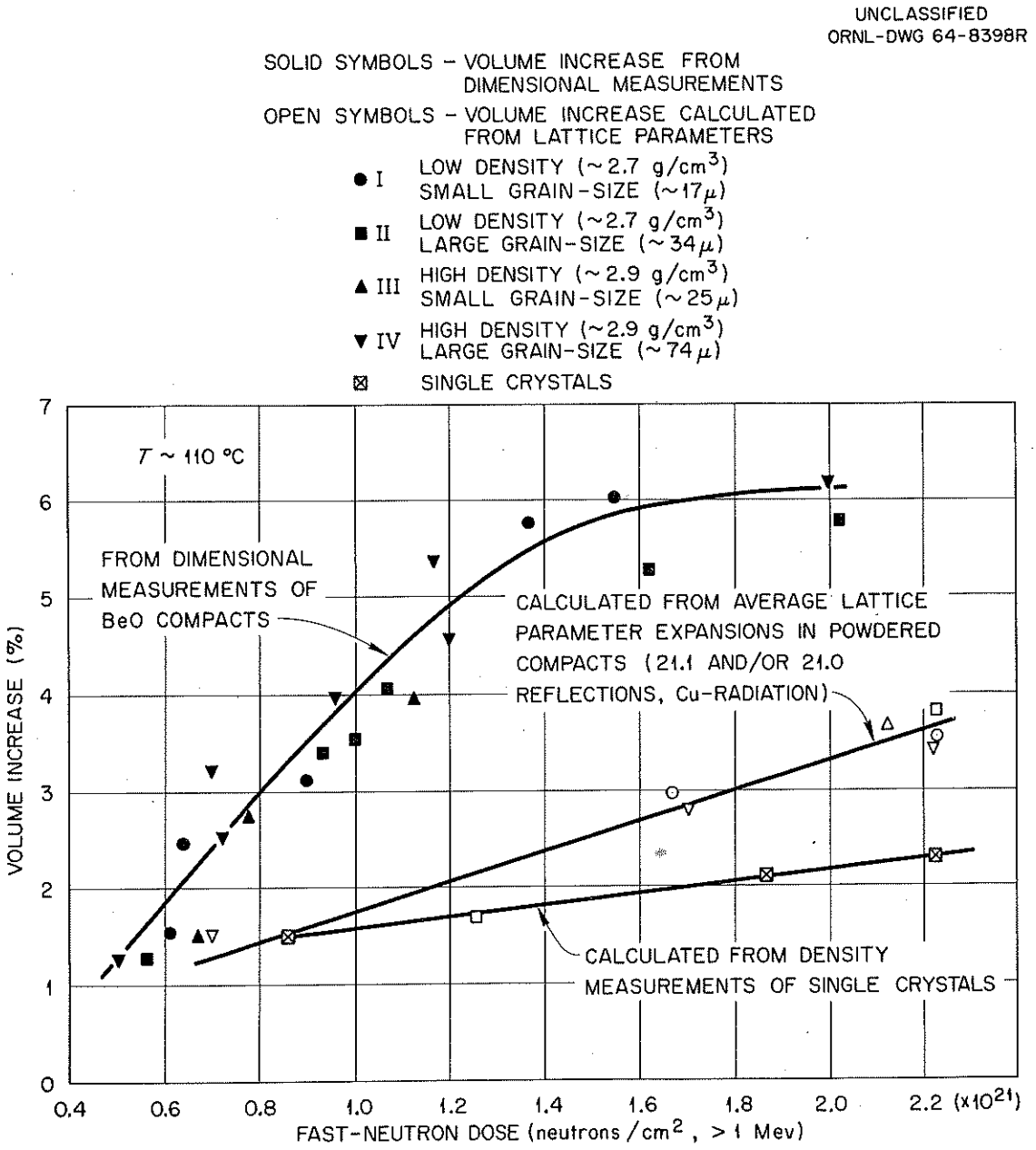


Fig. 2. Volume Increase of 1/2-in. BeO Compacts and Single Crystals vs Fast-Neutron Dose in Experiment 41-10.

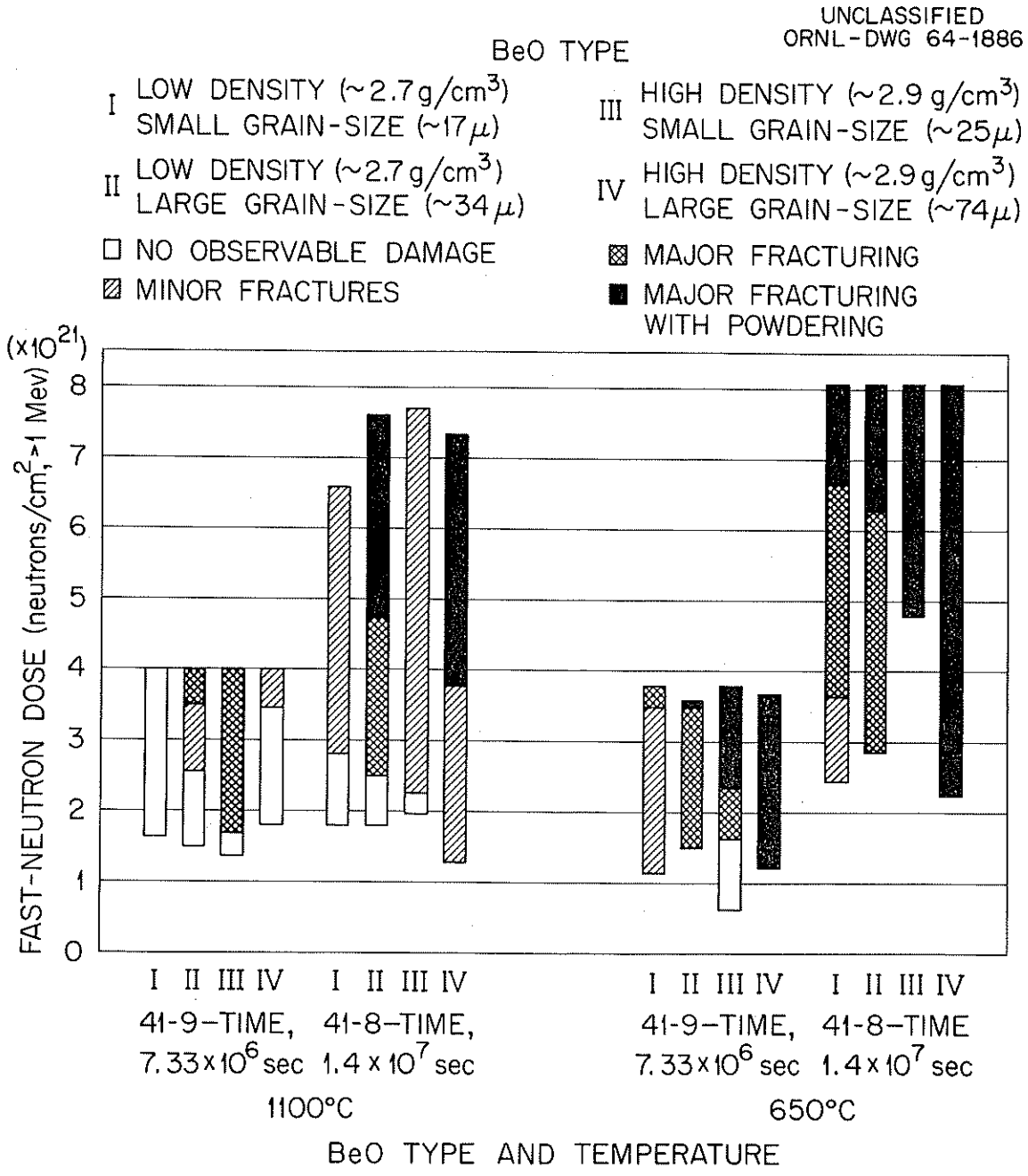


Fig. 3. Gross Damage to BeO Specimens in Experiments 41-8 and 41-9 as a Function of the Fast-Neutron Dose and Temperature.

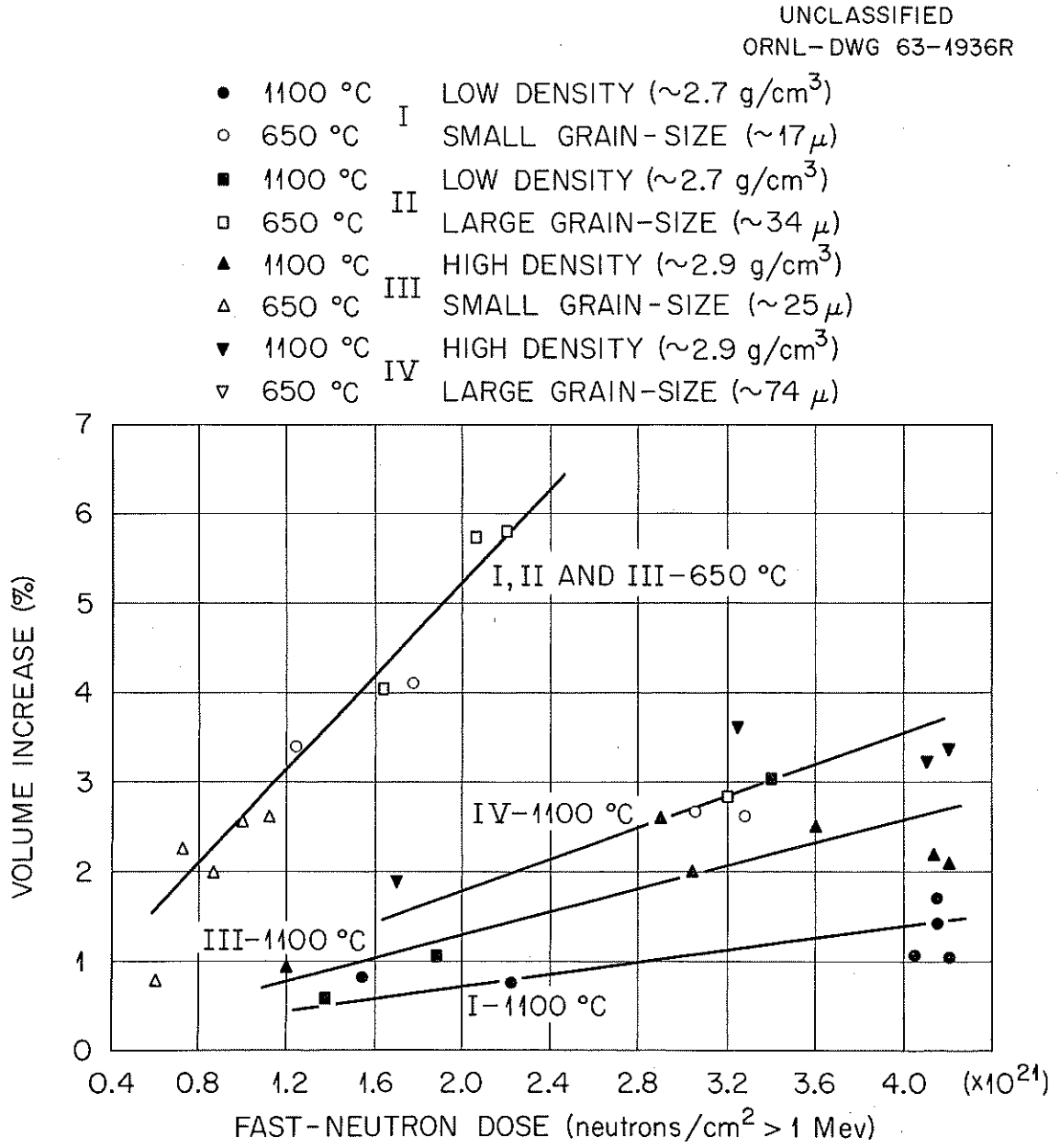


Fig. 4. Volume Increase of 1/2-in. BeO Specimens vs Fast-Neutron Dose in Experiment 41-9.

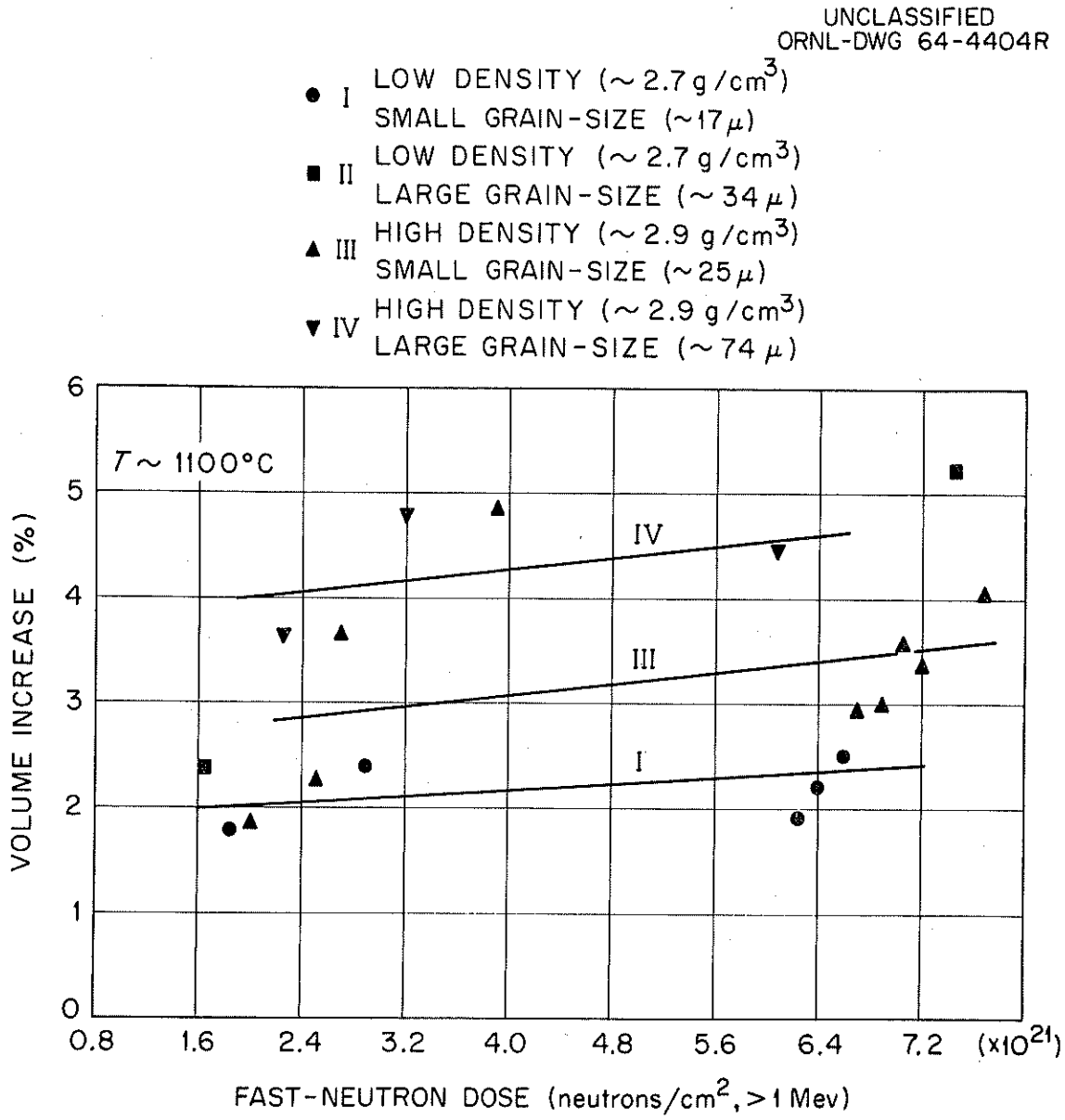


Fig. 5. Volume Increase of 1/2-in. BeO Specimens vs Fast-Neutron Dose in Experiment 41-8.

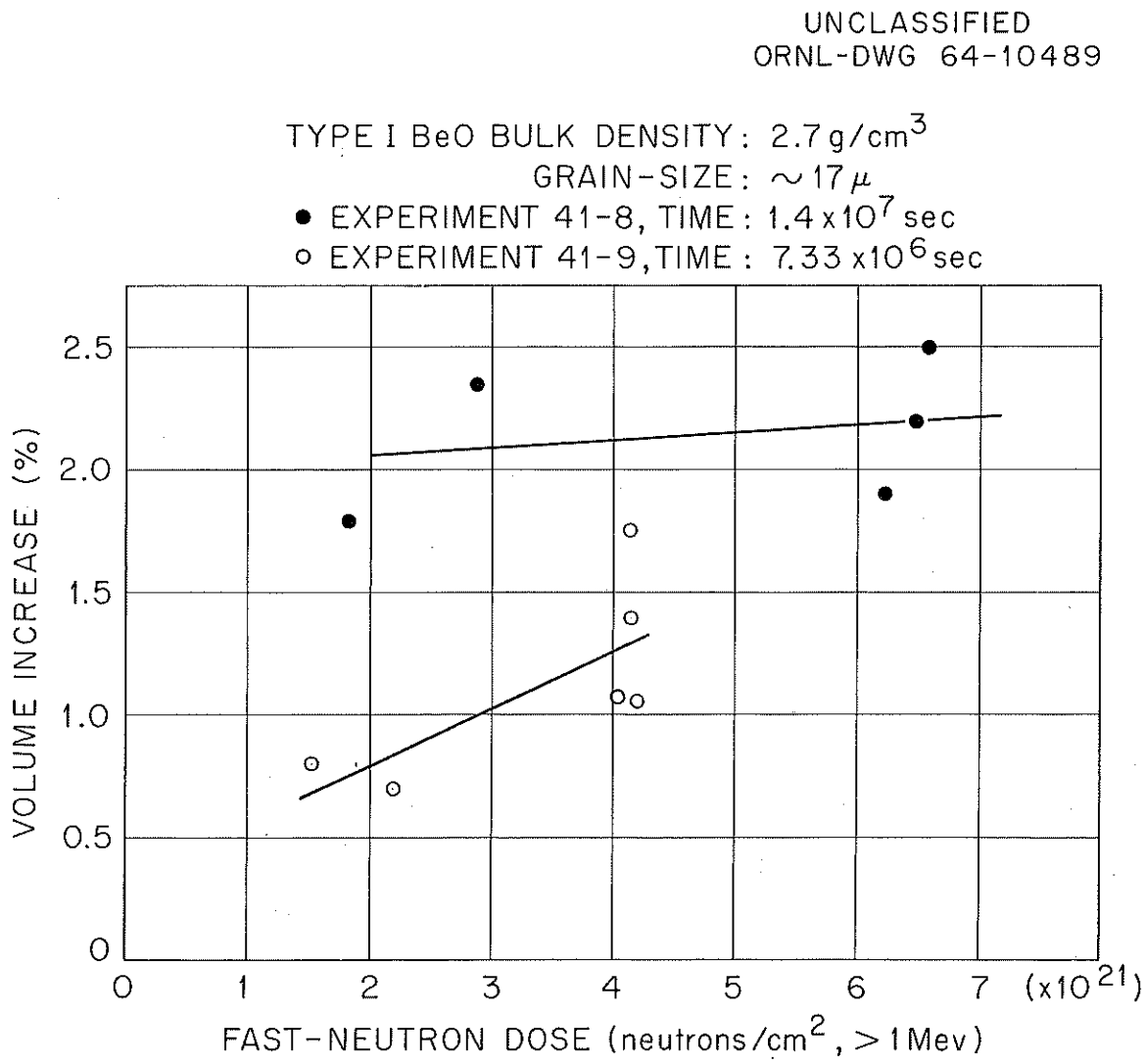


Fig. 6. Volume Increase of Type I 1/2-in. BeO Cylinders Irradiated at 1100°C as a Function of the Fast-Neutron Dose at Two Flux Levels.

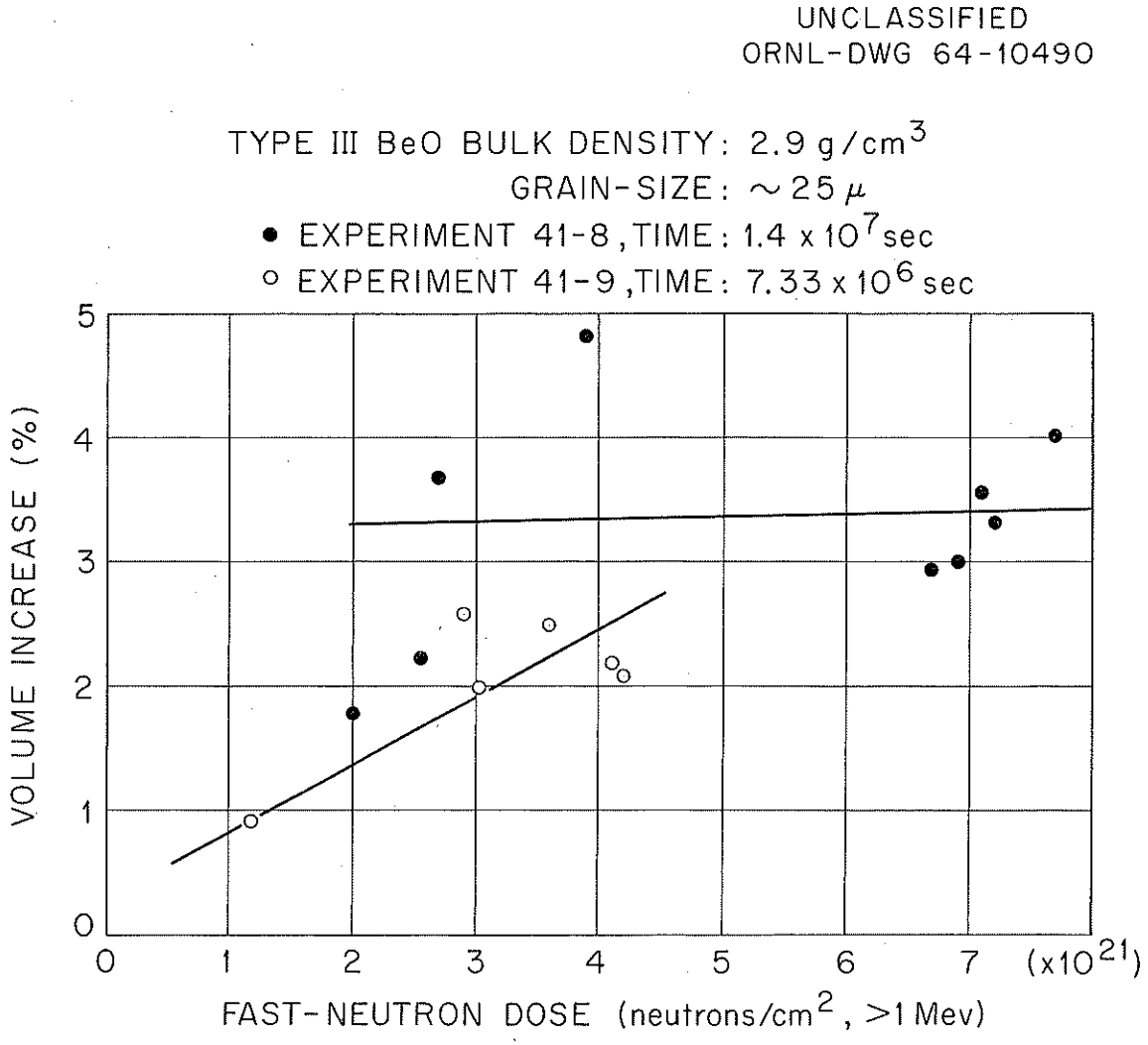


Fig. 7. Volume Increase of Type III 1/2-in. BeO Cylinders Irradiated at 1100°C as a Function of the Fast-Neutron Dose at Two Flux Levels.

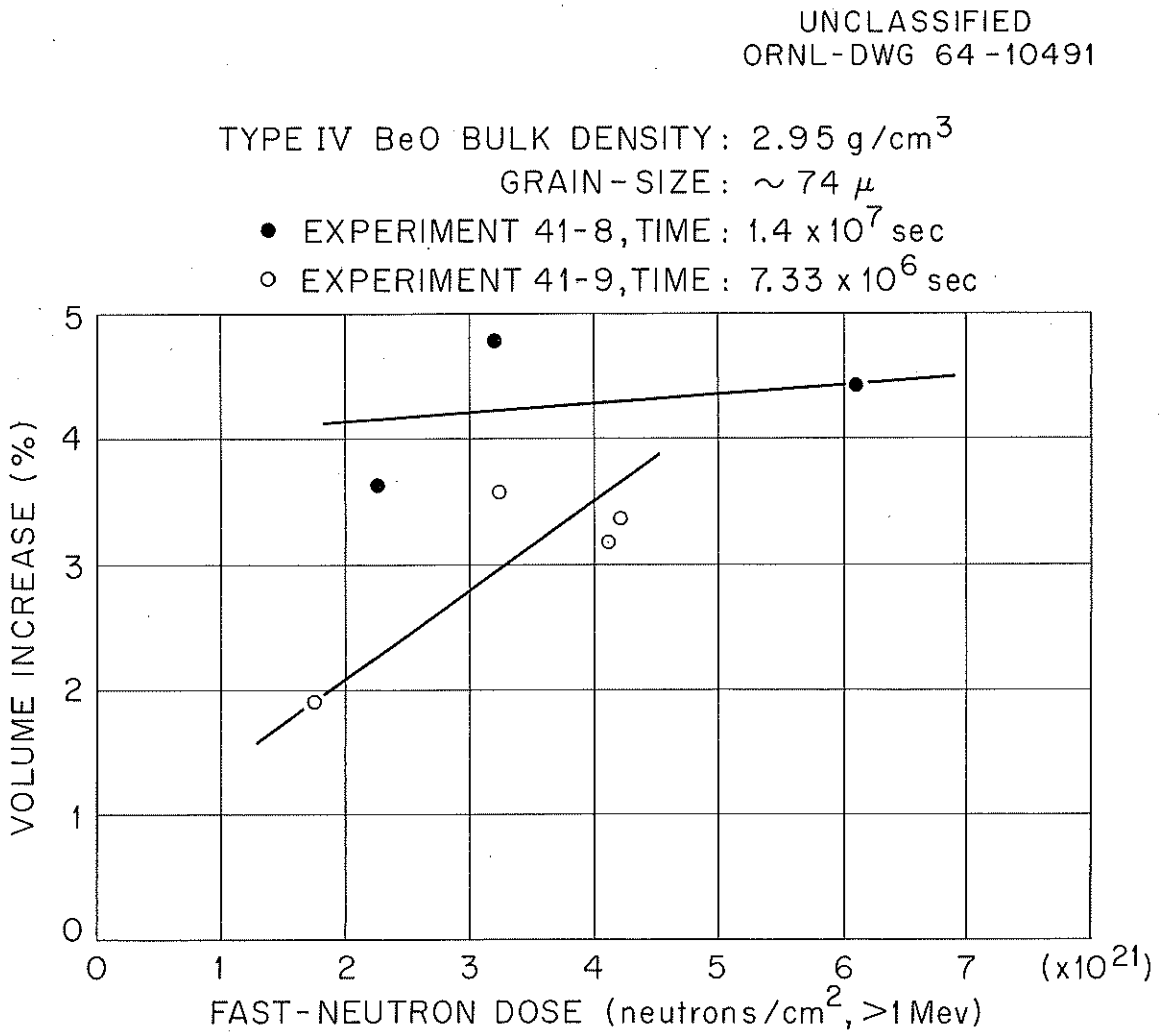
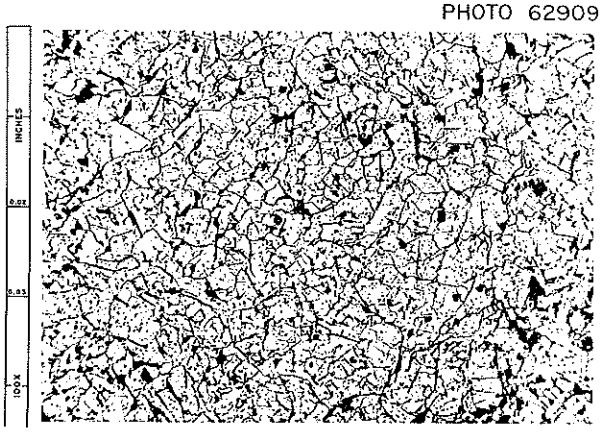


Fig. 8. Volume Increase of Type IV 1/2-in. BeO Cylinders Irradiated at 1100°C as a Function of the Fast-Neutron Dose at Two Flux Levels.



UNCLASSIFIED
PHOTO 62909

Y-45349

R-15768

AS POLISHED, 100X
DENSITY: 2.9 g/cm^3
GRAIN SIZE: 23 μ

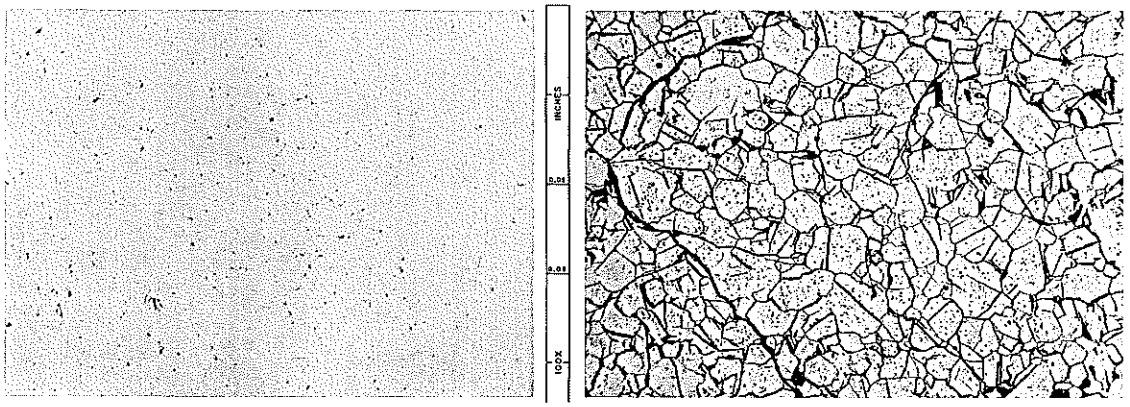
CONTROL

AS POLISHED, 100X
3.92 x 10^{21} *nvt*
> 1 Mev
1100 °C

IRRADIATED SPECIMEN A10-63

Fig. 9. Comparison of the Unirradiated Cold-Pressed and Sintered BeO of High Density (2.9 g/cm^3) and Small Grain Size (23 μ) with BeO of the Same Type Irradiated to 3.92 x 10^{21} neutrons cm^{-2} (> 1 Mev) at 1100°C in Experiment 41-9 Showing Extensive Grain-Boundary Separation (As Polished, 100X).

UNCLASSIFIED
PHOTO 62940R



Y-45352

AS POLISHED, 100X
DENSITY: 2.9 g/cm^3
GRAIN SIZE: 70μ

CONTROL SPECIMEN

R-15762

AS POLISHED, 100X
3.4 x 10^{21} *nvt*
> 1 Mev
1100 °C

IRRADIATED SPECIMEN

Fig. 10. Comparison of Unirradiated Cold-Pressed and Sintered BeO of High Density (2.9 g/cm^3) and Large Grain-Size (70 μ) with BeO of the Same Type Irradiated to 3.4 x 10^{21} neutrons cm^{-2} (> 1 Mev) at 1100°C in Experiment 41-9 Showing Grain-Boundary Separation and Transgranular Fracture.

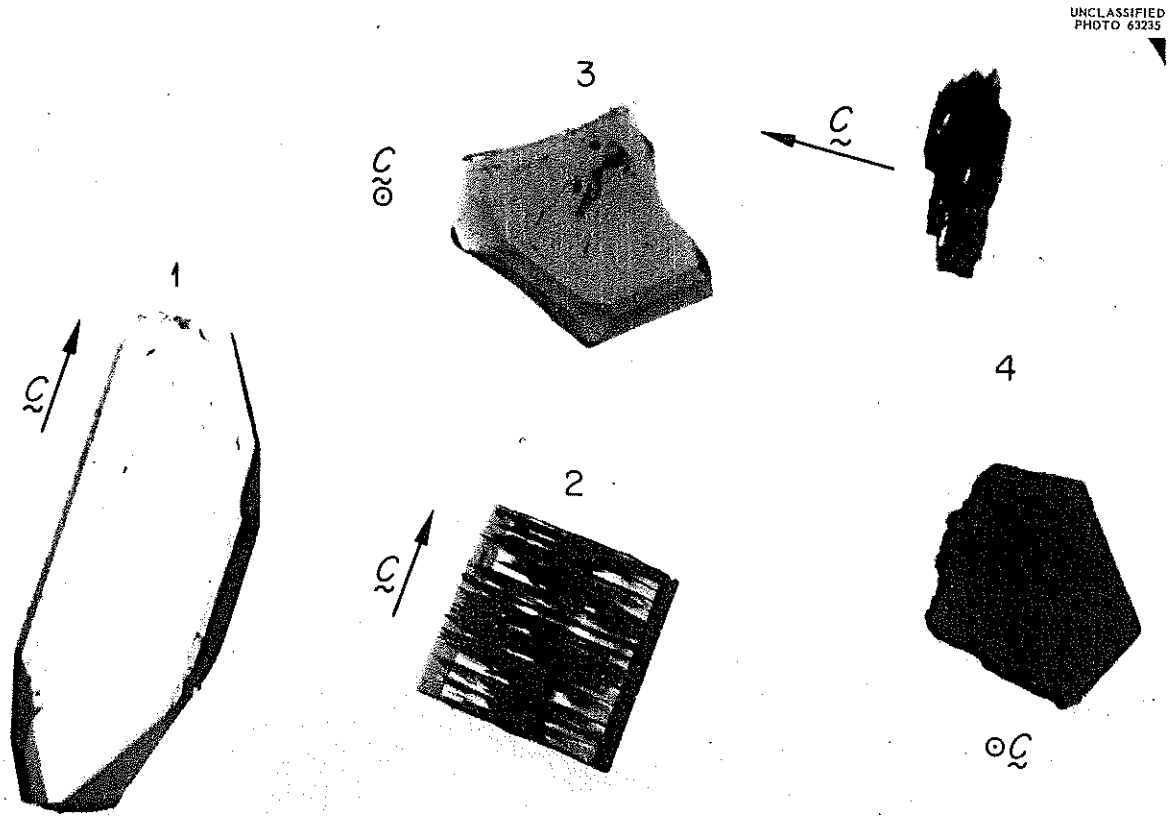


Fig. 11. Irradiated BeO Single Crystals.

Internal Distribution

1. Biology Library
2-3. Central Research Library
4. Reactor Division Library
5. ORNL Y-12 Technical Library
6-10. Laboratory Records Department
11. Laboratory Records, ORNL R. C.
12. C. J. Barton
13. D. S. Billington
14. F. F. Blankenship
15. A. L. Boch
16. E. G. Bohlmann
17. G. E. Boyd
18. R. B. Briggs
19. W. E. Browning, Jr.
20. R. H. Chapman
21. W. E. Clark
22. J. H. Coobs
23. F. L. Culler
24. A. P. Fraas
25. J. H. Frye, Jr.
26. R. J. Gray
27. W. R. Grimes
28. R. L. Hamner
29. W. O. Harms
30. T. Hikido
31. R. G. Jordan (Y-12)
32-51. G. W. Keilholtz
52. M. T. Kelley
53. A. Larson
54. C. E. Larson
55. J. A. Lane
56-76. J. E. Lee, Jr.
77. G. H. Llewellyn
78. R. A. Lorenz
79. H. G. MacPherson
80. R. E. MacPherson
81. H. F. McDuffie
82. D. L. McDonald
83. A. S. Meyer
84. A. J. Miller
85. C. E. Miller, Jr.
86-105. R. E. Moore
106. J. P. Murray (K-25)
107. M. W. Rosenthal
108. G. Samuels
109. J. H. Shaffer
110. R. P. Shields
111. C. H. Secoy
112. M. J. Skinner
113. C. D. Susano
114. D. B. Trauger
115. E. H. Taylor
116. G. M. Watson
117. J. C. White
118. A. M. Weinberg
119. M. C. Wittels
120. H. L. Yakel
121. A. F. Zulliger
122. F. Daniels (Consultant)
123. F. T. Gucker (Consultant)
124. Leo Brewer (Consultant)

External Distribution

125-139. Division of Technical Information Extension (DTIE)
140-143. Nathaniel Stetson (SRO)
144. H. M. Roth (ORO)