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CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE - CEN, Mol

IRRADIATION DAMAGE IN BERYLLIUM OXIDE

by

A. BÜRKHOLZ

1966

Report prepared at the CEN
Centre d'Etude de l'Energie Nucléaire, Mol - Belgium
Association No. 006-60-5 BRAB

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The principal radiation effect is growth and fracturing of the BeO specimens. The higher the irradiation temperature the higher the dose required to start damage. Equally important is the fabrication history of the material, small grain size being favorable for radiation resistance. It is not yet possible to find experimentally an influence of the dose rate on radiation damage.

Provided that the right irradiation conditions are chosen, beryllium oxide should nevertheless withstand much higher doses than formerly expected.

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CONTENTS

SUMMARY

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1) PROPERTIES

BeO crystallizes in the hexagonal system. The O-atoms are arranged in a closest packing of spheres, the smaller Be-ions are located in interstices. BeO has a ionic-covalent bonding and is of Wurtzite type.

BeO is a polycristalline, ceramic material. Its density varies, depending on grain size and mode of fabrication, between 2.5 and 3.0 g/cm^3 . An extensive examination of the properties of unirradiated BeO has been made by the workers of General Electric. (8,17)

Apart from its excellent nuclear properties it is distinguished by its high point of fusion (2550°C), good thermal conductivity and corrosion resistance.

A BeO moderated reactor core can be made smaller than a graphite moderated core. Because of the good compatibility of BeO with CO_2 at high temperatures, CO_2 can be used as a coolant. In some installations, the neutron enhancement by the (n,2n) reaction can be a valuable contribution.

On the other hand, the high cost of the BeO and the radiation damage at high dosages are still a limiting factor to its application. (24)

2) UTILIZATION OF BERYLLIUM OXIDE IN NUCLEAR REACTORS

Like graphite, BeO is considered to be a good moderator and structure material for high temperature gas cooled reactors. With fine grains of UO_2 ThO_2 in dispersion, the BeO matrix could serve as a moderator and a cladding material at the same time. (25 to 31)

The great possibilities which one ascribes to the future uses of BeO in reactor technology has stimulated a good deal of research. As the fabrication of simple BeO compacts offers no major problems to the ceramics industries, investigation centered on the irradiation behaviour of BeO. So by 1960 systematic irradiation studies were in progress, especially in USA and Australia.

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The most serious damade to beryllium oxide was found to be extensive fracturing and powdering under certain irradiation conditions. However, results from different workers were not consistent and the emerging picture was therefore a rather confused one. (1)

More recent findings, especially those by the General Electric workers, helped much to enlighten the role of dose, temperature and flux on the damage mechanism. (8) The question of whether beryllium oxide will withstand extended irradiation remains still open. To limit damage, it should be kept at temperatures around $1000^{o}C$. However, even at these tempera - tures limited radiation resistance seems to preclude beryllium oxide from its use as a fixed moderator material. Greater confidence can be set in the use of BeO as a matrix material for dispersion fuels. Here difficulties might arise from the problem of finding feasible methods for reprocessing. (24)

The fact that a conference on beryllium oxide was organized (october 1963, Australia) stresses the rising importance of this material.

Remarks:

Experiments on radiation damage in beryllium oxide were mainly done by the workers of General Electric, ORNL and AAEC. To a somewhat minor extend radiation tests were done at General Dynamics, Harwell and Saclay. Most of the irradiations were done in the ETR and the Hifar reactors. Major test parameters were dose, temperature, grain-size and fabrication history; other parameters were density, additives, texture and dose rate. Exposures went up to about $5 \cdot 10^{21}$ nvt (all doses in this report refer to neutrons above 1 Mev), irradiation temperatures ranged from room temperature up to $1200^{o}C$. Post irradiation test methods are given in annex I.

3) EFFECTS OF FAST NEUTRON IRRADIATION

3.1 Nature of effects

Just as in beryllium metal, the two threshold reactions, Be(n,2n)2He and Be9(n,)He6, will eventually lead to the accumulation of He4 and H3 atoms within the lattice. For each $1o^{21}$nvt of fast neutrons, o.4 cm^3 of helium is formed per gramm of beryllium oxide. At temperatures higher than 6oo°C the He atoms diffuse and form bubbles. The formation of tritium is an order of magnitude lower than the production of helium.

Contrary to the findings in Be metal, the main damage in BeO seems to be a result of the formation of point defects. At a dosage of $1o^{2o}$nvt about 13% of all the atoms were displaced once, but only 1% of them do not recombine with vacancies and remain interstitially. At higher temperatures they can diffuse and form clusters. This results in anisotropic lattice growth, which in turn leads to grain boundary cracking and volume growth.

This volume expansion is the most remarkable effect of neutron irradiation of BeO specimens. Other property changes are more or less a consequence of this expansion and can be described as a function of this variable. The most extensive radiation damage is found, as usual, after low temperature irradiations.

3.2 Volume expansion and cracking

3.2.1 Irradiations at about 1oo°C

Fig. 1 shows the volume expansion as a function of dosage. At low doses, beryllium oxide expands proportionally to dose until at about $2 \cdot 1o^{2o}$ nvt the expansion has reached about o.6%. The material remains coherent and retains its strength.

Above $2 \cdot 1o^{2o}$ nvt, volume expansion is still proportional to dose, but with a steeper slope. The grain boundaries begin to separate and the material looses more and more of its strength. At about $7 \cdot 1o^{2o}$ nvt the volume expansion reaches 5%. At still

higher doses, beryllium oxide begins to crumble and powder.(8)

Equally proportional to dose, one measures a growth of the lattice parameters, Δ a and Δ c for the a and c-axis respectively. Growth of the a parameter shows saturation at about $5 \cdot 10^{20}$ nvt. No saturation could be found for the growth of the c parameter. Under irradiations above 100°C the expansion of the lattice parameters is smaller and consequently not easily measured, so that different findings are published. At irradiations at temperatures higher than about 600°C, expansion is much smaller and predominantly in the c-axis.

As measured on crushed specimens and single crystals, the lattice expansion is equal to the expansion of the crystallites, see fig. 2.

Because the lattice expansion is anisotropic with $\Delta c/c$: $\Delta a/a$ greater than 1, considerable stresses are set up in the compact material. At doses higher than $1.6 \cdot 10^{20}$ nvt, stresses between grains of unlike orientation surpass grain boundary strength and the grains begin to separate. The resulting microcracking leads to volume expansion greater than that expected by lattice expansion alone. The weakening of the material goes on until at about 5% volume expansion it crumbles.

3.2.2 Irradiations at higher temperatures

Fig. 3 shows the volume expansion as a function of dosage for several temperatures. For temperatures of 700°C and higher expansion seems to reach a sort of saturation. (8) However, other investigators found even at very high temperatures an expansion proportional to dosage, so that the question must remain open. (3,22,24)

Elevated temperature irradiation not only reduces the amount of expansion for a given dose, but it also increases the amount of expansion at which grain boundary cracking occurs (e.g. from 0.6% volume expansion at 100°C to 1.2% at about 900°C). Expansion at higher temperatures consists again in microcracking and crystallite expansion. The latter can be measured on crushed samples. The lattice parameters remain almost unchanged, so the expansion must be attributed to defect

clusters and helium bubbles, which cause the lattice to expand without changing very much the lattice parameters. (24)

This is in concordance with the results of irradiations of single crystals. Irradiations at 650°C up to $4 \cdot 10^{21}$ nvt exhibited dark bandings parallel to the basal planes which are thought to be defect agglomerates. Expansion was as in crushed powders. Because lattice parameters are almost unchanged, expansion must again be ascribed to the formation of defect clusters. (24)

In sintered compacts, at 900 to 1000°C irradiations, only 8% of the volume expansion could be ascribed to the lattice expansion. Because the defect clusters are lying in the basal planes, they cause anisotropic expansion without markedly changing the lattice parameters.

Helium bubbles were observed in the grain boundaries of specimens irradiated at temperatures as low as 600°C. However, below 900°C they do not contribute much to volume expansion. The number and size of the bubbles increases with increasing grain size. After extensive microcracking fewer bubbles are formed and part of the gases escape.

3.3 The defect structure

Isolated point defects should not give rise to unsymmetric lattice expansion, nor should they cause line broadening. These two effects are ascribed to the formation of planar clusters of defects in the basal planes of the crystallites. The extensive broadening of the (hkl) lines for $l \neq 0$ in the absence of intergranular strain effects (powders), and the high temperatures needed to anneal the Δc parameter change, can best be interpreted in terms of clustered defects. (2,24)

The presence of defects of small dimensions (~ 10 Å) which anneal in the range of 100 to 800°C, was suggested by thermal conductivity measurements. These defects are thought to form the major portion of defects at small doses and saturate at about $5 \cdot 10^{20}$ nvt. These defects are responsible for the a-parameter change.

Above 10^{20} nvt most of the defects are formed by large

clusters in the basal plane. They need temperatures of 1100°C and more to anneal out. The clusters are interstitial and vacancy clusters, and are responsible for the c-parameter and anisotropic crystallite expansion. Their presence was first suggested by long wavelength neutron scattering. (18) By the method of electron transmission microscopy it was possible to establish the existence of small unresolvable dots at 100°C and planar vacancy and interstitial clusters at 1200°C. (8,24)

Some workers have found ß-phase BeO in crushed irradiated specimens and believe that irradiation could lead to the formation of this high temperature phase. (20)

3.4 Annealing of defects

3.4.1 In-pile annealing

The reduced expansion at higher temperature irradiations must be ascribed to the effect of damage annealing.

If one assumes a first order annealing process, the defect concentration can be described by

$$c = K/A \cdot (1 - \exp (- At))$$

where
t = irradiation time
K = rate of atomic displacement
A = annealing rate constant

In the absence of microcracking, the defect concentration can be regarded as proportional to the volume change $\Delta V/V$, and from the experimental expansion data follows (for irradiations between 600 and 1200°C) :

$$K = 3.3 \cdot 10^{-23} \cdot \Phi \qquad \Phi = \text{fast flux}$$
$$A = 6.1 \cdot 10^{-5} \exp (-1030o/RT)$$

Later work (8) refined this concept, so that one established a different A for interstitial and vacancy annealing. With this one is able to describe expansion down to temperatures of 100°C.

As there is no deterioration of the properties of BeO as long as microcracking is avoided, the above equation, which

predicts a saturation of volume expansion, sets a relation between flux and irradiation temperature. Because microcracking starts at about $\Delta V/V$ = o.5%, the ratio K/A must remain below this level, (see fig. 4)

There are other models for the kinetics of the annealing to account for the reduced radiation damage and expansion at higher temperatures. (24) They all assume that, provided temperatures are kept high enough, expansion will finally saturate before the onset of grain boundary separation. They further assume a certain proportionality of expansion to dose rate, fig5.

However, there are irradiations up to very high doses which failed to show this saturation of volume expansion. (24, 3,22) Equally, variations in dose rate of a factor of 5 to 1o were without any major effect on the amount and nature of the damage. (15)

So these two important predictions seem not to be supported strongly enough by experiment to be of practical value for the damage evaluation. The continued expansion at high temperatures could possibly be ascribed to the accumulation of the helium in bubbles. The independence of the damage on dose rate, however, seems to make a review of the annealing kinetics necessary.

3.4.2 Post irradiation annealing

As long as grain boundary separation is avoided, the volume expansion, along with the other parameter changes, can be annealed. Annealing temperature and annealing time depend on the extent of damage. With BeO irradiated below 1oo°C, annealing begins at 8oo°C for most property changes and is complete at 14oo°C. Recovery of Δa starts already at 2oo°C, whereas Δc does not anneal below 8oo°C; this might be further evidence that Δa is caused by isolated point defects and Δc by defect clusters. (2)

As annealing occurs over a wide range of temperatures, with a different temperature dependence for each parameter change, annealing seems to be a complicated process with no unique activation energy.

Annealing at 1400°C of samples irradiated to some 10^{21} nvt resulted in an expansion of the material. This must be ascribed to the formation of helium bubbles, which grow to the size of normal sintering pores. A similar expansion during high temperature irradiations could account for the continued volume growth with dosage. (8)

In samples with extensive microcracking, no expansion during annealing was found.

When annealed at 1300°C, recovery of the radiation induced expansion was found instead of expansion. (8)

3.5 The influence of material history on radiation damage

Contrary to general expectation, the influence of material properties on radiation damage does not seem to be a straightforward one. Correlations such as the dependence of preferred orientation on grain size are further complicating factors.

Porosity and chemical purity apparently do not affect radiation resistance markedly.

The rôle of density is not yet established, but low density seems preferable.

On the other hand, small grain size definitely favors the radiation resistance of beryllium oxide. It was also found, that cold-pressed sintered material shows a better radiation behaviour than hot-pressed BeO. (24)

For irradiations at 100°C, with material of about 1 μ grain size, onset of damage was between 1 to $1.4 \cdot 10^{21}$ nvt, whereas 10 to 15 μ material cracked already at $3 \cdot 10^{20}$ nvt. Figure 6 reflects this behaviour in the relaxation of x-ray line broadening, which is an indication of stress- release due to microcracking. (24)

Without microcracking, the expansion is the same for all materials, but the onset of microcracking occurs at higher doses for fine -grained material.

There are several theories to explain the higher resistance to microcracking of the fine grain-size materials. However, up to now this question can not be regarded as settled, because there are different models that can be envisaged. (11)

At 1000°C and 10^{21} nvt, bubbles were found to be nearly non-existent in materials of approximately 5 μ grain size. Bubbles increase in both size and number with increasing grain size and dose.

The experiments to investigate the influence of additives on the radiation resistance of BeO compacts are still in progress. (8) The favorable comportment of specimens of 1 $^{w}/_{o}$ bentonite in UOX compacts is already established. Irradiation test series of samples with glass additives are not yet terminated. The macroscopic expansion of the glass-phase compositions exceeds that of the AOX and UOX-MgO materials for comparable exposures. (8)

3.6 Mechanical and thermal property changes

Out of the multitude of more or less coherent results, a fairly clear pattern can be obtained if one plots the parameter changes as a function of volume expansion.

It follows that the elastic constants, strength, and thermal conductivity remain essentially unchanged as long as the material retains its compactness. As soon as grain boundary separation starts, however, these values deteriorate rapidly.(8)

In bigger specimens exposed to intensive gamma heating during irradiation, the decrease of thermal conductivity was thought to set up high high temperature gradients and high thermal stresses that led finally to extensive cracking and fracturing of the weakened material. (3)

However, another effect must be mentionned here. In irradiations at elevated temperatures, the thermally induced strain is reduced by radiation induced creep. On cooling, the thermally induced strain reappears, adds to the radiation strain and leads to cracking. (24)

3.7 Influence of energy spectrum on radiation damage

As usual with radiation damage, doses and fluxes given are those of neutrons above 1 Mev. Point defects, however, especially in light materials, are already produced by neutrons of much

less energy (about 1oo ev). Thus, caution should be exercised when comparing results obtained in different reactors (with different flux spectra). Nevertheless, fast flux (above 1Mev) seems to be a good measure of damage in beryllium oxide. (2,16)

4) DISPERSION FUEL ELEMENTS

4.1 Characterisation of the problem

For high temperature reactors, dispersion fuel elements seem to have most promising properties. The fuel is dispersed in a matrix, which serves simultaneously as a structure, moderator and cladding material. Besides good nuclear properties and resistance to radiation damage, the matrix must have good strength at high temperatures and low permeability for fission products.

There are two types of dispersion fuel elements that seem to meet this specifications: a graphite matrix with carbides of uranium and thorium as fuel and a matrix of beryllium oxide with the oxides of uranium and thorium as fuel.

In addition to radiation damage due to fast neutrons, damage could be expected by fission recoils and fuel particle swelling. In order to evaluate the effects of the two latter mechanisms, irradiation of dispersion fuel elements have been undertaken. These investigations started later than the experiments with pure BeO, but interest and work in BeO fuel elements seems to be growing rapidly. The different findings on radiation behaviour, however, are not yet clear or numerous enough to give an unequivocal picture.

4.2 (U,Th) O_2 - BeO dispersion fuels

Interest in dispersion type fuels is due to the possibility of :

a) high fissile material heat ratings
b) localisation of fission damage to a small volume fraction of the matrix (with fuel particle sizes not smaller than 15o μ)

c) retention of almost 9o% of the fission gases (with the same particle range as above).

To retain the fission products, high densities and low values of the interconnected porosity of the BeO matrix is required. The lattice diffusion in BeO is almost negligible for fission products.

The dispersion fuel elements are made of UO_2 or ThO_2 particles with diameters ranging from about 2o μ to several hundred μ, and which are embedded in a BeO matrix up to 5o $^{v}/o$. The fabrication of these dispersions has recently gained a considerable interest, and it is possible to fabricate elements with quite specific and homogeneous properties. One of the newer developments is the coating of fuel particles, or the whole dispersion fuel element, by BeO.

4.3 Irradiation experiments

A certain difficulty in the interpretation of the irradiation tests lies in the fact that sometimes it is not clear whether the observed effects are due to fission fragment or fast neutron bombardment.

Investigations on the irradiation behaviour of fuelled beryllium oxide are reported from a great number of laboratories. The exposures went up to an average fission density of $1o^{21}$ fissions per cm^3 of specimen. (3o)

The results can be stated as follows:

1) The smaller the fuel particle diameter, the higher the fraction of the BeO matrix that is damaged by fission product recoils. Thus, fuel particles smaller than 1oo μ seem not to be indicated.
2) With coarse fuel particles of UO_2, dimensional increases were usually less than 1% at fission doses up to about $5 \cdot 1o^{2o}$ fissions per cm^3. At $1o^{21}$ fissions per cm^3, dimensional changes of 5 to 1o% were found.
   The causes of swelling could be
   a) lattice expansion due to displacement type damage by fission fragments

b) lattice expansion due to accomodation of fission fragments

c) formation and growth of fission gas bubbles

3) Up to $6 \cdot 10^{20}$ fissions per cm^3, fission gas releases seem to be less than 1%. At 10^{21} fissions per cm^3, however, releases of 20% were found.

4) Changes in microstructure consist in a weakening of the matrix and pull-out of grains during polishing. The fuel particles were found to swell, which also affects the matrix by setting up strains. At higher doses, a complete loss of diffraction pattern of the BeO of fine dispersions was found. (30)

5) Although the irradiation experiments covered a temperature range from 450 to 1650°C, the influence of temperature could not be clearly established.

ANNEX I

Test methods

1) Volume change:

Volume expansion can be determined by
a) measuring dimensional change
b) measuring liquid displacement in alcohol or water

2) Mechanical properties:

Properties tested are
a) modulus of rupture
b) dynamic elastic constants (Young's modulus and shear modulus) - calculated from resonant frequencies
c) inner friction
d) creep strength

3) X-ray diffraction studies:

The reflection lines (of Cu K radiation) obtained on polycrystalline BeO samples are compared with those from unirradiated samples. Irradiation causes line displacement and line broadening.
Line displacement indicates lattice parameter changes. In the hexagonal BeO crystal, irradiation causes anisotropic growth of the c and a parameter with $\Delta c/c : \Delta a/a > 1$.
Line broadening reveals that the material is under stress. In randomly oriented, polycrystalline BeO the anisotropic lattice expansion causes considerable stresses, hence (hkl)-line broadening.
Results obtained on powders and solid material are not identical. Crushing to powder relieves some of the stresses, so that broadening of (hko)-lines disappears. The ratio $\Delta c/c : \Delta a/a$ is about 8 for powders. For solid material,

this ratio is smaller, because intergranular stresses tend to restrain anisotropy in lattice parameter changes. Line displacement is often too small and too much blurred by line broadening to be measured exactly. So results given by different workers are not always in accordance. Recently, some lines were found ~~to~~ unaffected by irradiation broadening. (24)
Comparison of intensities of reflection lines is also used to determine preferred orientation in BeO specimens.

4) Long wavelength neutron scattering:

Neutron wavelengths beyond the Bragg cut-off, where coherent elastic scattering is eliminated, are scattered incoherently by lattice defects. By comparing the scattering in irradiated and unirradiated specimens, one obtains some information on the nature, number and distribution of the defects. (18)

5) Thermal conductivity:

a) Comparative method
The temperatur drop along part of the specimen is compared with temperature drop in a piece of pure iron.
b) Heat pulse method
The time of flight of a heat pulse along the specimen is measured.

6) Electrical resistivity:

Resistivity decreases after irradiation, but this decrease is about a factor of 10^6 smaller than would be expected if all the Be interstitials which were produced were available as current carriers.

7) Stored energy:

The build-up of stored energy in BeO could be of the same

order of magnitude as in graphite. Energy release can be measured by plunging an irradiated and an unirradiated specimen each in a calorimeter, held at the appropriate temperatures, and comparing the temperature evolution. The few results reported do not yet give a clear picture.

8) Microstructure:

Optical and electron micrographs of irradiated and unirradiated BeO specimens are compared. For electron micrographs, replica and transmission techniques are used.

9) Gas contents:

After degassing in vacuum (to release adsorbed gases), the BeO specimens are fused in salt melts. The evolved gas is captured in vacuum and analyzed (with gas chromatographs e.g.).
At 600°C helium begins to migrate to pores. The existence of the helium in the gas state can be proved by annealing the specimens at rising temperatures followed by measuring the specific heat around 2.16 °K. At this temperature He gas (better: liquid helium) shows an anomaly in specific heat, caused by the so-called λ transformation. (10,13)

Note: Test methods, results and interpretation of results were discussed in great detail during the Sydney conference on beryllium oxide. (24)
A thourough presentation of the tested properties, especially of unirradiated BeO, can be found in report (8).

ANNEX II

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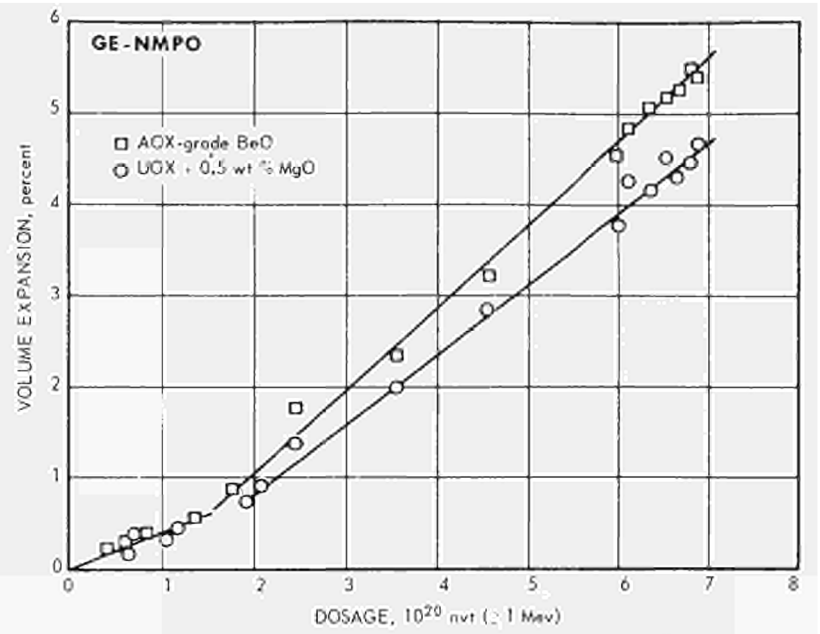

Fig. 1 – Expansion of 20-micron grain size BeO, 2.9 g/cm^3 density, irradiated at approximately 100°C

From third annual report high-temperature materials and reactor component development programs - Advanced Technology Services of Genral Electric - (GEMP - 270 A) - p. 114

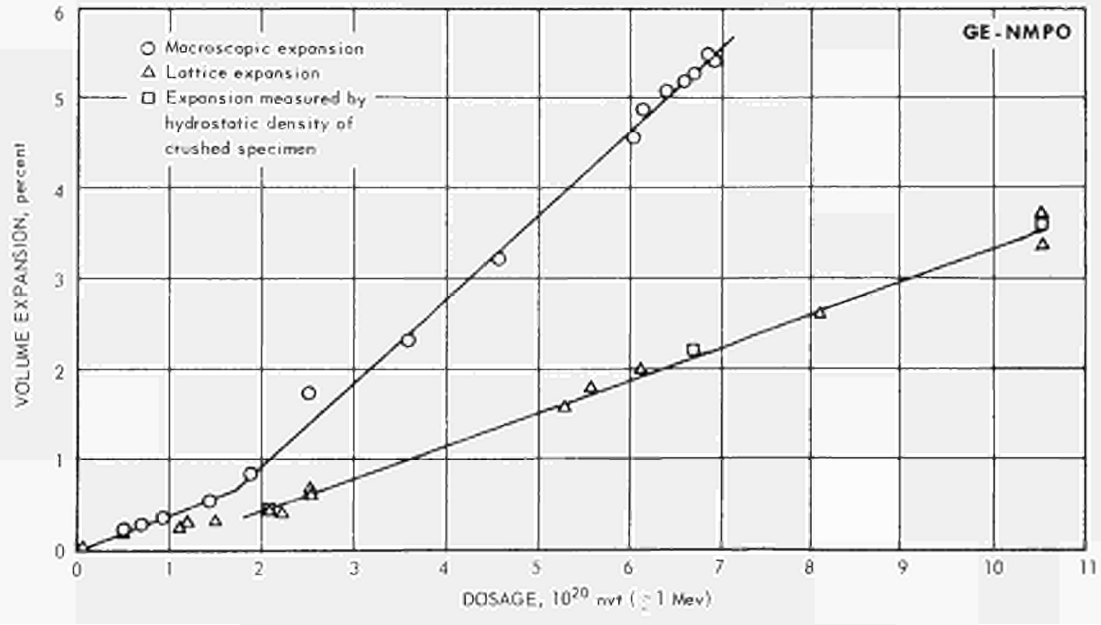

Fig. 2 – Components of volume expansion in BeO irradiated at 100°C

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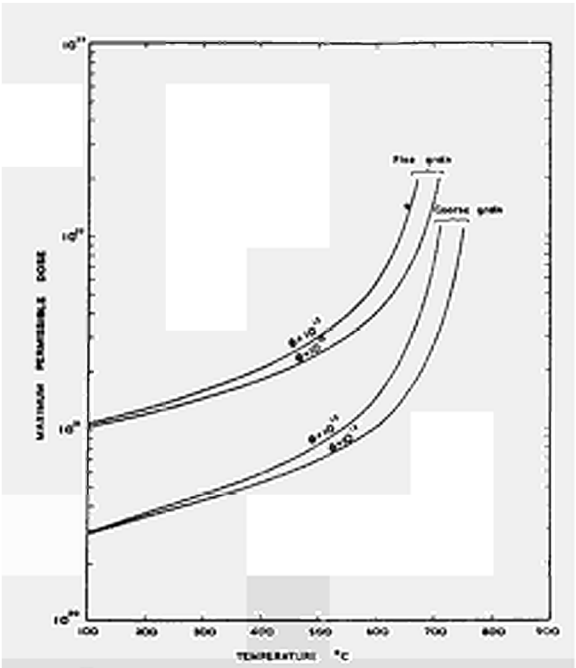

Fig. 5 Suggested values for the maximum permissible dose which cold pressed and sintered material could be subjected to without microcracking as a function of temperature, dose rate, and grain size.

From the effect of neutron irradiation on beryllium oxide by B.S. Hickman and A.W. Pryor - Journal of Nuclear Materials 14 (1964) 96-110

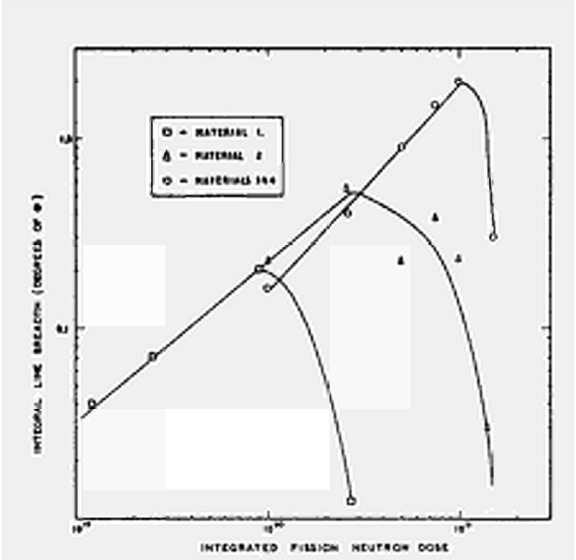

Fig. 6 Integral breadth of (300) Cu K_{α_1} reflection as a function of neutron dose for various materials.

From X-Ray diffraction studies of irradiated beryllium oxide by D.G. Walker, R.R. Mayer and B.S. Hickman - Journal of Nuclear Materials 14 (1964) 147-158

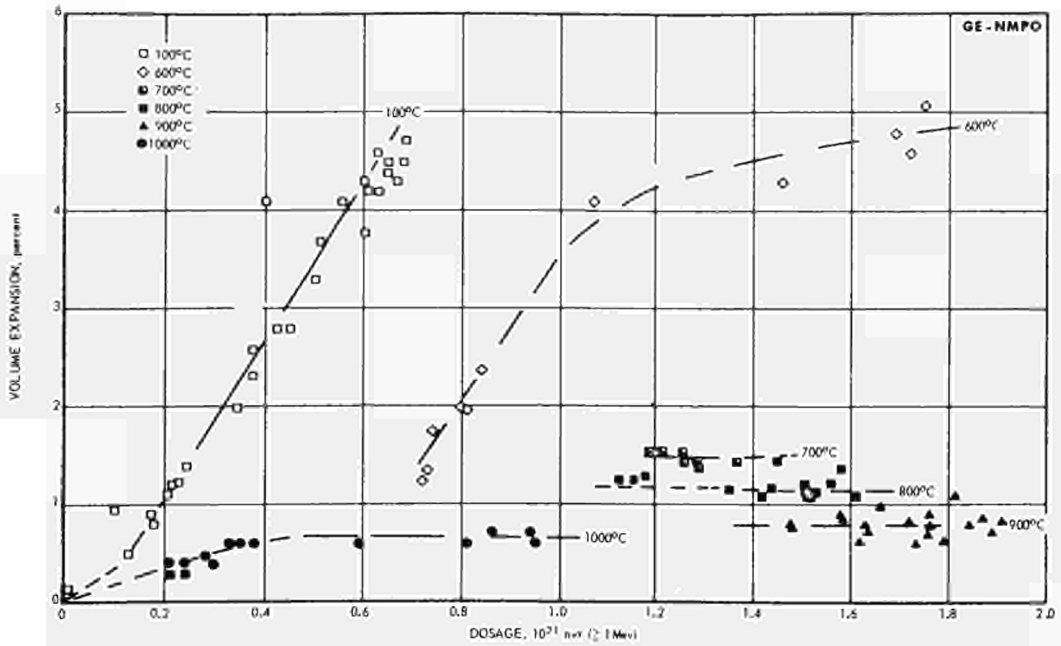

Fig. 3. – Volume expansion of UOX-grade BeO specimens containing 0.5 percent MgO, 20-micron grain size and 2.9 g/cm^3 density, as a function of irradiation temperature and dosage

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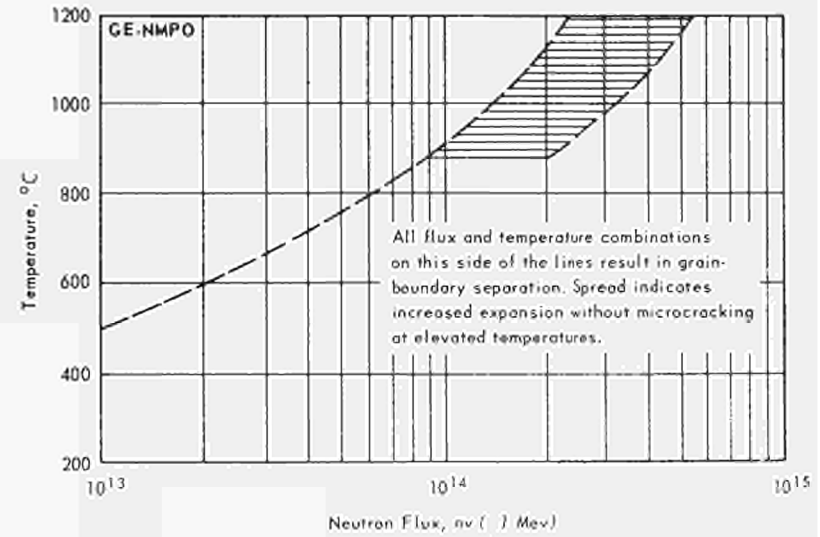

Fig. 4 – Estimated minimum irradiation temperature to avoid grain-boundary separation in BeO of 20-micron grain size and 2.9 g/cm^3 density

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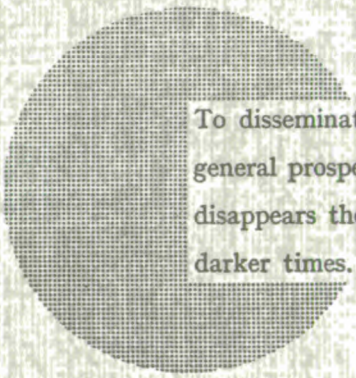

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Alfred Nobel

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