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# **IRRADIATION DAMAGE IN BERYLLIUM**

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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#### IRRADIATION DAMAGE IN BERYLLIUM by A. BÜRKHOLZ

Association : European Atomic Energy Community - EURATOM Centre d'Etude de l'Energie Nucléaire - CEN, Mol Report prepared at the CEN - Centre d'Etude de l'Energie Nucléaire, Mol (Belgium) Association No. 006-60-5 BRAB Brussels, July 1966 - 24 Pages - 7 Figures - FB 40

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#### SUMMARY

A synthesis has been made from the most important of the available publications on irradiation damage in beryllium. The references of the articles are given in the bibliography. An irradiation effect characteristic to beryllium is the production of He-atoms by fast neutrons. Principal irradiation damage is the severe embrittlement of the material after exposure to high doses. At tempera-tures above 600° C, bubble formation at grain boundaries leads to exten-sive swelling of the beryllium.

#### 1) **PROPERTIES**

Favorable properties of beryllium as a reactor material:

- a low cross-section for absorption ( thermal = 0.01 barns) making beryllium an excellent material for structure, moderator and reflector.
- low density (1.85 g/cm<sup>3</sup>) and consequently little gamma heating
- 3) high point of fusion (about 1290°C).
- 4) high mechanical strength even at elevated temperatures (about 15 kg/mm<sup>2</sup> at 600°C)
- 5) excellent heat conductivity ( 0.35 cal/sec.cm<sup>2</sup> in 1<sup>o</sup>C/cm at room temperature ).

Unfavorable properties of beryllium:

- 1) poor ductility, especially at room temperature and at 600°C.
- 2) the resulting difficulties in the fabrication of beryllium pieces.
- 3) the anisotropy of beryllium.
- 4) the high cost of the raw material.
- 5) the toxicity of beryllium.
- 6) the easy reaction with other materials (metals) above  $500^{\circ}$ C.
- 7) the corrosion by  $CO_2$  above  $600^{\circ}C$ .
- 8) radiation damage starting at a dose of 10<sup>20</sup> nvt fast neutrons, resulting in an embrittlement at low temperatures and swelling above 700<sup>0</sup>C.

Manuscript received on March 10, 1966.

# 2) <u>RESEARCH ON BERYLLIUM AND USE OF BERYLLIUM IN THE</u> NUCLEAR FIELD

Beryllium was early recognized as an interesting reactor material, and its properties are treated in a great number of publications. All these works, however, are dealing almost exclusively with the mechanical properties and the fabrication methods of beryllium. Of the 100 references cited in "Reactor Handbook 1960", only 2 are on radiation damage. From 1960 on, systematic experiments on beryllium are done by irradiating specimens to high doses at various temperatures. The former neglect of the irradiation behaviour of beryllium might be due to the high dose required before property changes are showing up.

The actual great interest in beryllium is due to its promising properties as a space craft material. In the nuclear field, its use as a canning material was and as a reflector material is still considered.

As a canning material, the low absorption cross section of Be could balance the high neutron losses in the tube materials in pressure tube reactors. With heavy water (project EL-4) or graphite as a moderator, the use of natural  $UO_2$  would be feasible. In the AGR, the British are replacing metallic uranium by  $UO_2$  pellets as fuel. This requires either the use of slightly enriched uranium or the use of beryllium as canning material.

Some time ago, the British abandonned their beryllium program and decided for enriched fuel and stainless steel as canning. (21) At the moment, beryllium as a canning material seems out of the market. This state of affairs, however, must be attributed more to a general change in reactor philosophy than to really prohibitive properties of the beryllium. There are many difficulties in the use of beryllium, but there is no known serious reason that would exclude beryllium as a canning material.

As a reflector material, beryllium seems not to be as easily replaceable. Both for the ATR and the HFIR beryllium is to be used in the reflector. Instead of pure Be the use of Be-Al alloys seems now to be advantageous. (15)

Research on beryllium is going on to ameliorate the general properties of this metal:

a) work to improve the mechanical properties by changing the fabrication methods:

The mechanical properties, especially the ductility, depend to a great extent on the texture of the material. This texture, preferred orientation e.g., is a function of the mode of fabrication.

- b) work to find out, less costly fabrication methods: At room temperature and near 600<sup>°</sup>C beryllium has a minimum of ductility. This brittleness requires costly procedures to form and shape the metal.
- c) work to improve the ductility of the metal by high chemical purification:

Experiments with monocrystals have shown that the brittleness of beryllium is caused by different cleavage mechanisms along crystal planes. After uttermost purification of the starting material, it was possible to grow monocrystals that did not show this inherent brittleness but were as ductile as other metals. Remembering that the current commercial beryllium has a far higher content of chemical impurities than other pure metals, it is believed that brittleness in beryllium is due to precipitation hardening.

d) work to improve corrosion resistance and compatibility with other metals:

At temperatures of  $600^{\circ}$ C beryllium is reacting with a number of other metals (fuel and structural), and above  $650^{\circ}$ C it undergoes a break-away corrosion with  $CO_2$ , if there are traces of water vapour present. All these deficiencies can be more or less met by a proper surface treatment. Against  $CO_2$  corrosion, a surface oxidation of the Be powder during fabrication has shown good results. e) work to study the extent and the mechanism of radiation damage:

Systematic study of the behaviour of beryllium under neutron irradiation started some years ago, especially in Great Britain, USA and Australia. A number of beryllium specimens have been irradiated in capsule experiments and studied in post-irradiation measurements. The influence of dose, irradiation temperature and annealing temperature on the properties of beryllium can now be assessed to a satiafactory extent. The role of fabrication history on irradiation and the mechanism of radiation damage can still not be considered to be fully established.

With the loss of interest in beryllium metal at both the AAEC and UKAEA, the two most important programs to study the radiation damage in beryllium are terminated. This is inasmuch regrettable as just the more recent irradiation results showed that radiation effects are rather strongly dependent on material properties.

A practical interest to continue to study the possible radiation damage in beryllium exists at MTR, because there some highly irradiated parts of the lattice and reflector beryllium were found to have partially fractured and bowed. At MTR a program is under way to determine the total dose and the time of exposure of those pieces that were found to have broken off a considerable time ago. (see section 3.2.1) (15)(16a)

f) It shall be mentionned here that the highly irradiated parts of the MTR beryllium seem to be interesting as a possible source of the isotope Be 10, which is formed from Be 9 by thermal neutrons with a cross section of 0.01 barns. (15)

#### 3) RADIATION DAMAGE IN BERYLIIUM

# 3.1) The causes

Of the different types of high energy radiation that exist in a reactor, it is exclusively the flux of fast neutrons that effects the properties of beryllium. These fast neutrons are acting on the crystal lattice by two different ways: displacement of Be atoms by nuclear collisions and atom transmutation by nuclear reactions.

#### 3.1.1) Nuclear collisions

The immediate result of displacement of atoms by energetic neutrons is the introduction of point defects in the material, i.e. interstitial atoms and an equal number of vacant lattice sites (about 1000 per colliding fast neutron).

The point defects can annihilate each other by recombining, or they can combine to form clusters of interstitial atoms or clusters of vacancies. These processes are diffusion controlled and consequently a function of temperature. Foint defects as well as clusters embrittle the material in hindering the movement of dislocations. These radiation effects are general and not specific for beryllium. In fact, they are considered as to be only of minor importance in beryllium.

3.1.2) Nuclear transmutations

#### a) Reaction mechanism

Depending on the absorption spectrum, the neutrons can react with the nuclei of the material. This results in the production of impurity atoms that are characteristic for the material considered. Together with boron, beryllium is the only light material that undergoes a sort of fission reaction by neutrons: I Be 9 (n,2n) 2  $\propto$ II Be 9 (n, $\alpha$ ) He 6 He 6  $\rightarrow$  Li 6 Li 6 (n<sub>th</sub>, $\alpha$ ) H 3 H 3  $\rightarrow$  He 3

Cross sections are as follows:

energy range (mev)	barns	
	(n <b>,X</b> )	(n,2n)
4-10	0.010	0.58
2-4	0.085	0.20
1-2	0.020	0.0

(according to ORNL information)

For a fission spectrum effective cross sections can be assumed as:

(n <b>, X</b> )	0.025	barns		
(n,2n)	0.100	barns		
	(accor	ding to	AAEC	information)

b) The amount of impurity atoms produced

The high threshold energies necessary for these nuclear reactions mean, that it is the fast flux that determines the radiation damage in beryllium. Helium is produced at a rate proportional to  $\emptyset_{\text{fast}}$ , while the rate of production of H3 is proportional to  $\emptyset_{\text{fast}}$ .  $\emptyset_{\text{thermal}}$ , that is the square of the flux. Reaction I is the important one and the amount of the He produced is at least one order of magnitude higher than for H3. So, radiation damage is ascribed to the presence of He only.

For each transmuted Be atom, 2 atoms of He are produced, and exposed to a fast dose of  $10^{22}$  nvt, per cm<sup>3</sup> of beryllium an amount of 20 cm<sup>3</sup> gas at NTP, equivalent to an atomic concentration of about 1%, is trapped.

Because of the formation of Li6 and He3 there is a buildup of poison. While the Li6 will soon reach an equilibrium (high thermal cross section), He3 will increase with time. The decrease in reactivity is small but might have an influence in special cases. The concentration of Li6 will be proportional to the ratio of fast flux/ thermal flux.

#### 3.2) Experimental results

3.2.1) Methods and parameters

Irradiation of beryllium samples covered the exposure range from some  $10^{19}$ nvt (where effects begin to be measurable) up to almost  $10^{23}$ nvt (reactor beryllium of the MTR). Temperatures ranged from room temperature to about  $700^{\circ}$ C.

Table 1 shows the parameters investigated by the different groups.

a) Gas contents:

The beryllium specimen is melted under vacuum and the released gas is measured and analysed, e.g. by gas chromatography or mass spectrometry. The same is done with a control sample for reference. It was found that appreciable gas losses are only to expect when the sample was irradiated or annealed at temperatures considerably above the swelling temperature. So the quantity of the He4 released on melting the sample is a good measure of the total fast dose the specimen received. MTR workers found a rather good agreement with values derived from theoretical considerations of flux distribution. Most of the gas is He4, the portion of H3 and He3 is rather small. The determination of the ratio of the latter two gases was used successfully by workers of MTR to assess the time of fracture of some beryllium pieces which fell to the bottom of the reactor. Because of the high (n,p) cross section of He3, during irradiation the ratio of He3 / H3 $_2$  reaches very soon a stable value ( $\lambda / \sigma \cdot \phi_{\text{th}} = 0.5\%$  in this case;  $\lambda =$ decay constant of H3,  $\sigma$  = cross section of He3). Fig. 1 shows the correlation of  $H_{3_2}$  / He3 with time. (15) b) Swelling:

Swelling is measured by simple geometric measurement or by water displacement methods.

c) Electron microscopy:

Mostly, fracture surfaces were examined by replica techniques to study growth and distribution of gas bubbles. The resolving power is about 100 to 150 Å. Bubbles of somewhat smaller diameter can be observed in thin foils and flakes by the transmission technique.

- d) Electrical resistivity:
   Measurements of electrical resistivity are easily
   made and can be done even during irradiation.
- e) Mechanical tests:

Mechanical tests are done following well established standard methods. One measures the properties of irradiated and unirradiated specimens subjected to identical heat treatments. In comparing results care must be taken of the fact that tested properties are a function of test temperature.

- f) Measurements of long wavelength neutron scattering and stored energy has also been tried on specimens irradiated to a dose of 6.10<sup>20</sup> nvt, but failed to give results, indicating that damage is too low to show up in these tests. (8)
- 3.2.2) Radiation damage in as-irradiated beryllium

The main parameters for radiation damage in beryllium are

fast dose
irradiation temperature
materials history

The principal radiation effects in beryllium are

embrittlement bubble growth swelling Properties affected by irradiation:

Mechanical properties:

- a) Increase in hardness up to 100% in specimens with exposures between  $10^{21}$  and  $10^{22}$  nvt. (1 to 14)
- b) Increase in yield strength also up to 100% depending on dose, material and irradiation temperature.
   Fig.3 gives an example. (1 to 15)
- c) Fracture stress seems less affected by irradiation, see fig. 4. (12)
- d) Reduction of elongation to practically zero above 10<sup>21</sup> nvt. Fig. 5. (1 to 15)
- e) Increase in bending strength and loss in bending ductility. Beryllium rods with exposures of 10<sup>21</sup>nvt fractured immediately in bending tests. (1,2)
- f) The development of yield points and multiple yielding has also been observed in irradiated beryllium.(12)
- g) Where extensive bubble growth occurred at grain boundaries, the bubbles can initiate premature grain boundary fracture under stress at higher temperatures (above 400<sup>o</sup>C). (4,5)
- h) Beryllium irradiated to very high doses (more than 10<sup>22</sup> nvt) at room temperature becomes so brittle that it fractures immediately under mechanical strain. (6,15)

The mechanical properties of beryllium depend heavily on test temperature (see fig. 3,4,5). Beryllium irradiated at low temperatures and tested at higher temperature shows at the same time annealing effects.

Electrical properties:

Electrical resistivity increases.

Structure properties:

Bubbles are appearing at irradiation temperatures between 450 and  $650^{\circ}$ C, depending on material history. The bubbles grow preferentially at grain boundaries, but sometimes they can also be found within the grains.

The fracture mode of beryllium seems to be unaffected by irradiation unless extensive bubble formation at grain boundaries has occurred. (12)

#### Density:

With rising temperature (above  $650^{\circ}$ C), irradiated beryllium begins to swell.

The dependence of swelling on dose and irradiation and annealing temperature seems now to be well established. Fig. 6 shows swelling of irradiated samples as a function of annealing temperature and heating time. Fig.7 shows the threshold temperature for the onset of swelling as a function of fast dose.

So at room temperatures even with exposures in excess to  $10^{22}$  nvt no detrimental volume changes must be feared. However, if larger pieces of beryllium are exposed to high flux gradients, the generation of internal stresses can lead to bowing, as was found at the MTR lattice beryllium, (15)(16a).

Variables influencing the nature and severeness of radiation damage in beryllium:

### Dose:

Radiation effects increase with dose. At irradiation temperatures below  $100^{\circ}$ C a linear dependence of property changes (electrical and mechanical) could be found.(5) Below a dose of  $10^{20}$  nvt radiation damage in beryllium is negligible.

#### Irradiation temperature:

Table 2 presents a summary of the context of irradiation temperature and hardness, bubble growth and swelling of a typical material.

#### Material history:

The evolution of mechanical properties and the bubble formation outlined in table  $2^{i}$  not followed by each material.

The irradiation behaviour of beryllium is also a function of its fabrication history and is different for hot-pressed, extruded and for heat treated and quenched material. Research in this field has begun recently, but because of the termination of the beryllium programs studies could not be carried on. The material history determines the temperature for bubble growth, their distribution at grain boundaries and within the grain, and the mechanical properties. So bubble growth within the grain and hardening was suppressed in a material that had no second phase precipitates nuclei due to a heat treatment prior to irradiation. (5, 8 to 11)

#### 3.2.3) Radiation damage in annealed beryllium

Because heat treatment of unirradiated samples sometimes yield considerable property changes, it is not always easy to separate damage annealing from normal heat treatment effects.

In a rough approximation, irradiation at a higher temperature is equivalent to an irradiation at a low temperature and subsequent annealing at temperatures somewhat higher than the above mentionned irradiation temperature. So table 2 describes also the annealing behaviour of a beryllium sample irradiated at a low temperature, if one replaces irradiation temperature by annealing temperature.

Up to  $550^{\circ}$ C swelling does not exceed 1%. At higher temperatures the specimens undergo considerable swelling (up to 30% and more), with large voids appearing between the grains. (6,10,16) At about  $1100^{\circ}$ C degassing sets in. Fig.6 shows the decrease of density on annealing.

Fig.2 gives an example of the partial recovery of mechanical properties due to isochronal annealing. The elongation anneals only to a fraction of its unirradiated value, the ductility and hardness recoveramore or less completely in a temperature range just below the onset of swelling.

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### 3.3) The mechanism of the radiation damage

### 3.3.1) Point defects

While copper exhibits radiation damage at a dome as low as  $10^{17}-10^{18}$  nvt, practically no damage can be found in beryllium after exposures less than  $10^{19}-10^{20}$  nvt. Consequently, radiation effects in beryllium are supposed not to be caused by point defects (interstitial atoms and vacancies) but by the helium atoms.

In fact beryllium contains, contrary to copper, chemical impurities in such a level that their influence on material properties is much greater than that of point defects. (26)

## 3.3.2) He-atoms in enforced solid solution

At low temperatures the helium atoms remain in enforced solid solution distributed in the lattice just where they were produced. In sufficient number (exposure  $10^{20}$  nvt) they provoke solution hardening, and property changes are directly proportional to dose. (5)

## 3.3.3) Bubble growth

At higher temperatures, helium atoms as well as vacancies can migrate. As a noble gas, the helium atoms can not react chemically with the lattice atoms but will precipitate as bubbles at grain boundaries, dislocation lines and second phase precipitates. The kinetics of this mechanism is a problem of solid state physics that is not yet solved. Important parameters are temperature, diffusion coefficients of both He atoms and vacancies, diffusion length and scale of nucleation sites (as are grain boundaries, dislocation lines, second phase precipitates).

As a result one observes the appearance of a great number of very small bubbles. (16) In the case that the bubbles appear within the grains, they embrittle t/ne and harden the material in pinning down the dislocation lines and hindering their movement, (precipitation hardening). (3) With rising temperature the big bubbles grow at the expense of the smaller ones, thus decreasing the number of the bubbles but increasing their size. It has been demonstrated that the helium bubbles in copper can migrate and coalesce. It is possible that the second phase precipitates do not direct-ly act as nucleation sites but that they det trap moving dislocation lines. The bubbles attached to the latters are thus prevented from coalescing and growing.

The coarsening of the bubbles decreases the number of the pinning sites of dislocation lines and consequently removes hardening and restores ductility.

Finally, at still higher temperatures, bubbles are growing considerably, and variations in swelling from grain to grain set up considerable stresses that will separate grains, causing the appearance of large holes at the grain boundaries that account for the majority of the swelling in beryllium.

#### ANNEX

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Table I

Research on radiation damage in beryllium (numbers indicate references given in annex)

Organisation tested parameters	UKAEA	AAEC	ORNL	Canada	MTR-ETR
gas contents	1,2,3,7	8,9,10		6	15
swelling	1,2,3,7		13,14	6, 12	15
microscopy	1,2,3,4	5,16	9	6	
hardness	1,3,7	5,8 to 1	1	6 <b>, 1</b> 2	
tensile strength	1,2,3,4	5 <b>,</b> 8tto11		12	
ductility	1,2,3,4	5,8 to11		12	
bending strength	1				
stress-rupture strength		8 to 11	9	12	

Table II

Bubble formation, hardness and swelling as a function of irradiation temperature (ref.5)

Irradiation temperature	Bubbles	Hardness	Swelling
<b>∢</b> 300 <sup>0</sup> C	none	moderate to large increase	0
300 - 550 <sup>0</sup> C	many very small ones	large increase	< 1%
550 - 750 <sup>0</sup> C	fewer and bigger ones	decrease to almost norm <b>al</b>	<b>≼</b> 10 <b>56</b>
> 750 <sup>0</sup> C	few very big ones at grain boundaries		>10%

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Fig. 1

From Beryllium Gases Experiment by R.L. Tromp - MTR-ETR Technical Branches Quarterly Report, January 1 - March 31, 1963 - IDO-16898 - P18



Fig. 2 From The mechanical properties of some highly irradiated beryllium by J.B. Rich, G.P. Walters and R.S. Barnes - AERE - R 3449



(g. ) Yield stress for S-200-B beryllium in heat-treated and irradiated conditions.



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Alfred Nobel

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