ELSEVIER Journal of Nuclear Materials 258–263 (1998) 566–570

journal of nuclear materials

Microstructure and mechanical properties of neutron irradiated beryllium

E. Ishitsuka [a,*], H. Kawamura [a], T. Terai [b], S. Tanaka [b]

[a] *Japan Atomic Energy Research Institute, Oarai Research Establishment, Oarai-Machi, Higashi Ibaraki-Gun, Ibaraki-Ken 311-13, Japan*

[b] *University of Tokyo, 7-3-1 Hongo, Bunkyo-Ku, Tokyo 113, Japan*

Abstract

Microstructure and mechanical properties of the neutron irradiated beryllium were studied. Specimens were produced by various methods and had different grain sizes and impurity contents, and neutron irradiation was carried out with total fast neutron fluences of 1.3–4.3 × 10^{21} n/cm^2 (E > 1 MeV) at 327–616°C. Swelling was increased by high irradiation temperature, high fluence, and by small grain size and high impurity content. Obvious decreasing of the fracture stress was observed in small grain specimens which had many helium bubbles on the grain boundary. Decreasing of the fracture stress for small grain specimens was presumably caused by crack propagation on the grain boundaries which were weakened by helium bubbles.

1. Introduction

More than 300 tons of beryllium are expected to be used as a neutron multiplier and plasma facing material in the International Thermonuclear Experimental Reactor (ITER). For a neutron multiplier in the blanket, beryllium pebble and/or low density beryllium are candidate materials because these materials are expected to give low swelling. As a plasma facing material, hot-pressed beryllium (S-65C) is a candidate material because of its high thermal shock resistance [1]. Recently, some studies for neutron irradiated beryllium were reported [2–4]. However, neutron irradiation data are poor in these materials, and discussion about grain size and/or impurity effects is insufficient. Therefore, the microstructure and mechanical properties of neutron irradiated beryllium produced by various methods with different grain size and impurity levels, were studied to get engineering data for fusion reactor design.

* Corresponding author. Tel.: +81-29 264 8368; fax: +81-29 264 8480; e-mail: ishi@jmtr.oarai.jaeri.go.jp.

2. Samples

Specimens were bending specimens produced by the hot-press or vacuum cast method, tensile specimens produced by hot-press or HIP (hot isostatic press) method and beryllium pebbles produced by the rotating electrode method. Each specimen had different grain size and impurity level. Information of each specimen is shown in Tables 1 and 2. Four sets of these specimens were irradiated with four inner capsules in the Japan Materials Testing Reactor (JMTR). Irradiation conditions are shown in Table 3. Helium and tritium production rate was calculated by HEINBE code [5].

3. Experimental

Swelling was measured with a density meter (Mettler SGM-3) using the echilarchale to prevent floating for pebble, or water for other specimens.

Fracture strength of two bending specimens, one tensile specimen and 5–10 compression specimens were tested at room temperature [6] for each of the inner capsules. Testing speed was 0.2 mm/min for each specimen. The fracture stress of the three points bending test was calculated by using the following equation [7].

PII: S0022-3115(98)00106-8

Table 1
Beryllium specimens

No.	Fabrication method	Grain size (mm)	Configuration
BHP (S-65C)	HP[a]	0.01	Bending
BVC (B-26D)	VC[b]	0.56	(4 × 3 × 40 mm)
THP (S-200F)	HP[b]	0.02	Tensile
THIP	HIP[b]	0.13	(∅3 × [t]56 mm)
P	REM[a]	0.53	Pebble ($^{\phi}$1 mm)

HP: Hot press, VC: Vacuum cast.
HIP: Hot isostatic press.
REM: Rotating electrode method.
[a]NGK Insulators Ltd.; [b]Brushwellman.

Table 2
Chemical composition

No.	Element (wt.%)					
	Be	BeO	Fe	C	Al	Mg
BHP	99.1	0.9	0.10	0.14	0.04	<0.01
BVC	99.8	0.02	0.03	0.08	0.04	0.04
THP	98.9	1.2	0.10	0.14	0.05	0.02
THIP	99.72	0.18	0.047	0.093	0.026	0.0115
P	98.2	1.51	0.11	0.023	0.078	0.017

Table 3
Neutron irradiation condition

Inner capsule	T_{irr} (°C)	Fluence[a] × 10^{21} (n/cm^2)	Helium[b] × 10^3 (appm)	Tritium[b] × 10^1 (GBq/cm^3)
1	327	1.3	0.3	0.5
2	445	3.0	0.7	1.8
3	616	4.3	1.0	3.0
4	524	4.1	1.0	2.8

[a]Fast neutron energy: $E > 1$ MeV.
[b]Production rates were calculated by HEINBE code.

$$\sigma_b = \frac{3PL}{2wt^2}, \quad (1)$$

where σ_b is the fracture stress in the bending test, P the fracture load, L the span width of the lower support (30 mm), w the specimen width, t the specimen thickness. For evaluation of the fracture strength of the pebble, we used the following equation.

$$\sigma_c = \frac{L_c}{r^2}, \quad (2)$$

where σ_c is the compression fracture stress factor, L_c is fracture load, and r the radius of pebble.

Scanning electron micrograph (SEM) observation of the fracture surface for each specimen was carried out in the JMTR hot-laboratory. Metallographical observation was also carried out after chemical etching [dipping in a mixture of ethyl alcohol (45 cm^3) and hydrogen fluoride (5 cm^3) for 15–30 s]. Then, the micro vickers hardness was measured under the conditions of 200 g and 30 s.

4. Results and discussion

4.1. Swelling

Measured swelling is shown in Fig. 1. Swelling increased with increasing irradiation temperature and fluence, and with decreasing grain size and increasing impurity level. These results are reasonable because the single crystal specimen (with large grains and low impurity level) showed low swelling [8]. Swelling of the pebble specimen showed negative values. The reason is not clear. The swelling equations by Beeston [9] and Sernyaev [8] are also shown in the same figure. The formula by Beeston predicted a rather low value. This difference was caused by the difference in neutron spectrum. The formula by Sernyaev gave good agreement with the results using different structure-sensitive factors [8] ("Max" and "Min" mean the maximum and the minimum of structure-sensitive factor). However, further studies will be needed to understand the swelling

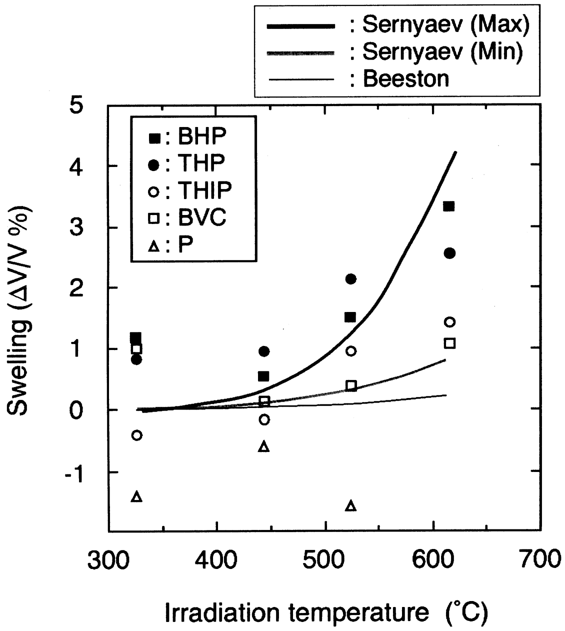

Fig. 1. Swelling as a function of irradiation temperature.

mechanism, for example, structure-sensitive factor and separation of effects of the grain size and impurities.

4.2. Microstructure and mechanical properties

Results of the bending and tensile test are shown in Figs. 2 and 3. By increasing irradiation temperature, the fracture stress of small-grained specimens (BHP, THP) rapidly decreased. These results are attributed to helium bubbles because many helium bubbles were observed on the grain boundaries. Rapid decreasing of the fracture stress for the larger-grained specimens was not observed, though larger grain specimens retained larger helium bubbles.

The fraction of intergranular fracture for each specimen is shown in Fig. 4. It is clear that the fracture mechanism of small-grained specimen changed to intergranular fracture by increasing of irradiation temperature. The grain boundaries of BVC specimen were

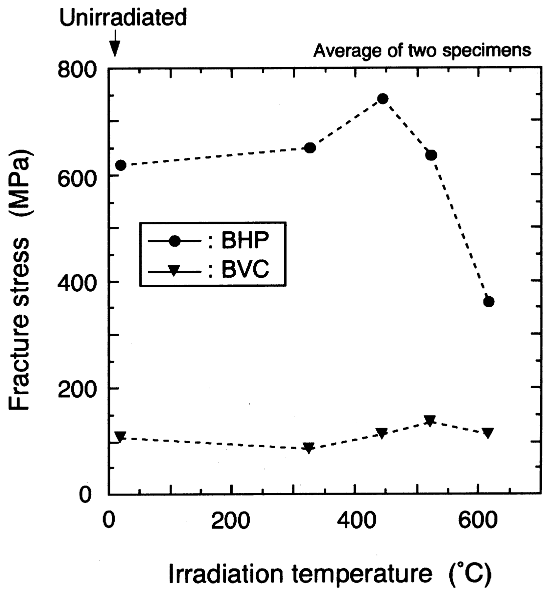

Fig. 2. Results of the bending test.

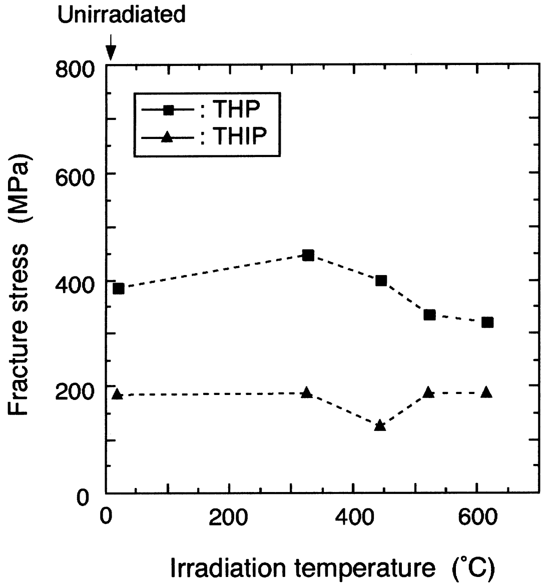

Fig. 3. Results of the tensile test.

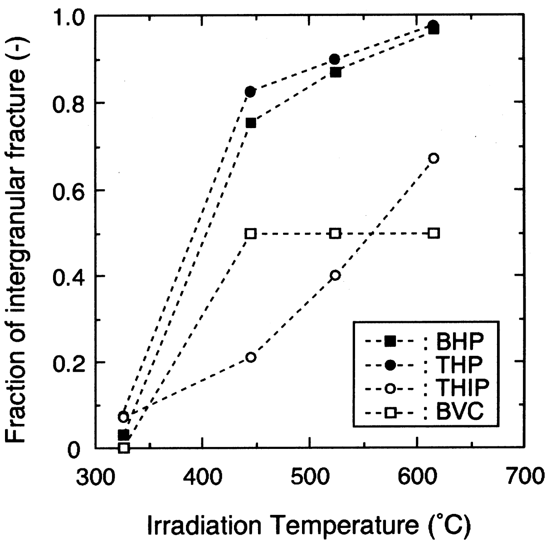

Fig. 4. Fraction of intergranular fracture for the each specimen.

almost completely covered by helium bubbles. However, obvious decreasing of the fracture stress was not observed. These results show that weakening of the grain boundaries by helium bubbles did not affect the fracture stress, directly. From these results, the rapid decreasing of the fracture stress for the small grain size specimens was presumed to be caused by the propagation of cracks on the grain boundaries because the connection of cracks may be easier for the small grain size.

Generally, small-grained specimen show high fracture stress (Hall–Petch relation [10]). However, we should pay attention to the selection of grain size in case neutron irradiation, and should find the most suitable grain size for each neutron irradiation condition.

Results of compression testing are shown in Fig. 5. The vertical axis is normalized by the average of the σ_c [see Eq. (2)] for the unirradiated sample. Mechanical properties of the beryllium pebble specimen show the feature of larger-grained specimens like BVC (grain size of pebble is almost same as BVC, see Table 1), and rapid decreasing was not observed. The dispersion at low temperature was presumed to be caused by the anisotropy in strength [11], and anisotropy became small with the growth of helium bubbles. Fractured surfaces of BVC and pebble are shown in Fig. 6. Helium bubbles on the grain boundaries of the pebble specimen were smaller than those of BVC specimen although both specimens had a similar grain size. This was presumed to be caused by the size effect of specimen (Most helium was released because of the short diffusion length in the pebbles) and/or the initial defects introduced in production process; pebble specimen had many initial defects because of rapid cooling, and helium will be trapped by these defects. For these reasons, beryllium pebbles have some advantages compared with small grained specimens.

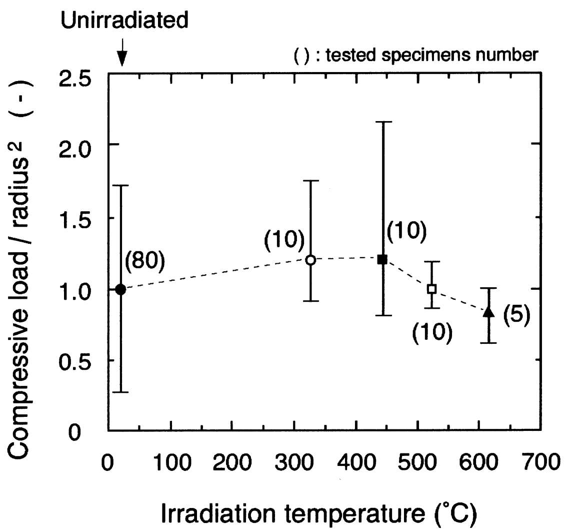

Fig. 5. Results of compression test.

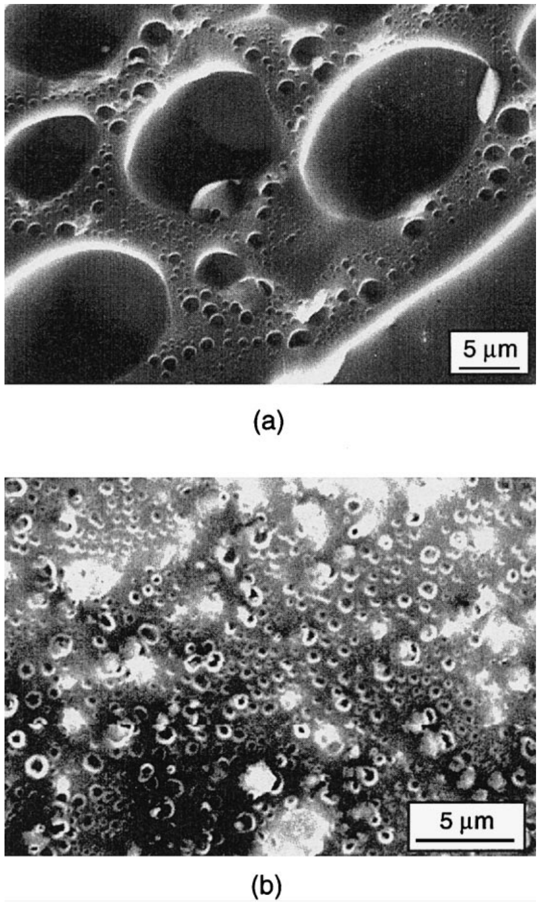

Fig. 6. Fracture surface of BVC and pebble specimen. (a) Inner capsule No.3, BVC specimen. (b) Inner capsule No.3, pebble specimen.

Decreasing hardness was observed on the smaller-grained specimens, while no obvious tendency to decrease was observed on the larger-grained specimens. This tendency is similar to the case of fracture stress.

4.3. Impurity effects on mechanical properties

Re-evaluation of the existing data was attempted because the discussion on the impurity effects was insufficient by using only the data obtained in this experiment.

Fracture stress was measured by the JMTR surveillance test more than ten years ago [12]. Two kinds of beryllium specimens [hot-pressed tensile specimens produced by NGK and KBI (the present name is NGK Metals Corp.)] were used in this test, and it became clear by the reevaluation that these specimens had different levels of Fe impurity caused by different production processes (other impurities were in the same level). The

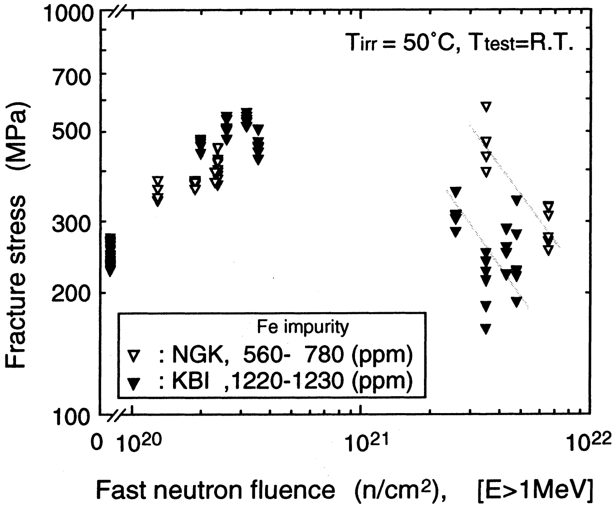

Fig. 7. Effects of Fe impurity on fracture stress.

replotted figure for the specimens with different Fe impurity levels is shown in Fig. 7. Fracture stress for the specimen with more Fe impurity decreased in the high neutron fluence range. This result shows that the Fe impurity affects the fracture stress in the high fluence range. An explanation of this result could be that, Fe impurity such as $Be_{11}Fe$ traps or nucleates helium bubbles [13], and helium bubbles weaken the mechanical properties.

5. Conclusion

Microstructure and mechanical properties of the neutron irradiated beryllium produced by different methods were discussed, and the following results were obtained.

- Swelling increased with increasing irradiation temperature, and fluence, and with decreasing grain size and increasing impurity level.
- Fracture stress of the small-grained specimens rapidly decreased at high irradiation temperature. This was attributed to the propagation of cracks on the grain boundaries. This result means that we should pay attention to the selection of grain size in case of neutron irradiation.
- Beryllium pebble has some advantages due to larger grains, smaller size and more initial defects in comparison with the other small grained specimens.
- Fe impurity affects the fracture stress in the high fluence range.

Some results were obtained from this study. However, these data are insufficient to discuss the grain size and effects of impurities in detail. Further studies are required to use beryllium for the fusion application.

Acknowledgements

The authors greatly appreciate the helpful comments on this paper by Dr. O. Baba (Director, Department of JMTR, JAERI).

References

[1] R.D. Watson, D.L. Youchison, D.E. Dombrowski, R.N. Guiniatouline, I.B. Kupriynov, CONF – 9509218 INEL, 1995, pp. 7.
[2] F. Moons, L. Sannen, A. Rahn, J.V. De Velde, J. Nucl. Mater. 233–237 (1996) 823.
[3] A.S. Pokrovsky, S.A. Fabritsiev, R.M. Bagautdinov, Yu.D. Goncharenko, J. Nucl. Mater. 233–237 (1996) 841.
[4] G.A. Sernyaev, A.S. Pokrovskiy, R.M. Bagautdinov, S.A. Fabritsiev, I.V. Mazul, V.R. Barabash, J. Nucl. Mater. 233–237 (1996) 891.
[5] S. Shimakawa, E. Ishitsuka, M. Saito, JAERI-M 92-174, 1992.
[6] E. Ishitsuka, H. Kawamura, Beryllium neutron irradiation study with JMTR, in: Fourth International Symposium on Fusion Nuclear Technology, Meiji Kinenkan, Tokyo, Japan, April 6–11 1997.
[7] Japanesse Indstrial Standard, JIS R 1601, 1981.
[8] D.S. Gelles, G.A. Sernyaev, M. Dalle Donne, H. Kawamura, J. Nucl. Mater. 212–215 (1994) 29.
[9] J.M. Beeston, L.G. Miller, E.L. Wood Jr., R.W. Moir, J. Nucl. Mater. 122&123 (1984) 802.
[10] S. Koda, Introduction to Metal Physics, Corona Publishing, Tokyo, Japan, 1980, p. 297.
[11] E. Ishitsuka, H. Kawamura, Fusion Eng. Des. 27 (1995) 263.
[12] T. Takeda, H. Amezawa, K. Tobita, JAERI-M 86-007, 1986.
[13] B.S. Hickman, J.H. Chute, J. Australian Inst. Metals 8 (3) (August 1963).