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Investigation of ITER candidate beryllium grades irradiated at high temperature

I.B. Kupriyanov [a,*], V.A. Gorokhov [a], R.R. Melder [b], Z.E. Ostrovsky [b], A.A. Gervash [c]

[a] *A.A. Bochvar All-Russia Scientific Research Institute of Inorganic Materials (VNIINM), Rogor Street 5, Box 369, Moscow 123060, Russian Federation*

[b] *Scientific Research Institute of Atomic Reactors, Dimitrovgrad, Russian Federation*

[c] *D.V. Efremov Scientific Research Institute, St. Petersburg, Russian Federation*

Abstract

Beryllium is one of the main candidate materials both for the neutron multiplier in a solid breeding blanket and for the plasma facing components. That is why the investigation of beryllium behaviour under the typical for fusion reactor loading, in particular under the neutron irradiation, is of a great importance. This paper presents some results of investigation of five beryllium grades (DshG-200, TR-30, TshG-56, TRR, TE-30, TIP-30) fabricated by VNIINM, Russia, and one (S-65) fabricated by Brush Wellman, USA. The average grain size of the investigated beryllium grades varied from 8 to 40 μm, beryllium oxide content was 0.7–3.2 wt.%, initial tensile strength 250–680 MPa. All the samples were irradiated in active zone of SM-3 reactor of 650–700°C up to the fast neutron fluence $(5.5-6.2) \times 10^{21}$ cm^{-2} (2.7–3.0 dpa, helium content up to 1150 appm), $E > 0.1$ MeV. Irradiation swelling of the materials was revealed to be in the range of 0.3–1.7%. Beryllium grades TR-30 and TRR having the smallest grain size and highest beryllium oxide content, demonstrated minimal swelling, which did not exceed 0.3% at 700°C and fluence 5.5×10^{21} cm^{-2}. Mechanical properties and microstructure parameters measured before and after irradiation are also presented.

1. Introduction

Beryllium is considered as a main candidate material for a fusion application. However, the efficiency of beryllium as a plasma-facing material and neutron multiplier for a breeding blanket strongly depends on its behaviour under neutron irradiation. In terms of fusion application the most important consequences of neutron irradiation on beryllium are helium induced swelling, embrittlement and tritium release. In spite of a rich variety of data on effect of irradiation on beryllium, which is available in the literature [1–4], not much is known about of the irradiation behaviour of main fusion-candidate beryllium grades [5].

This paper presents the results of the investigation on the effect of high-temperature irradiation on swelling, mechanical properties and microstructure of beryllium. The very first results of this work have been reported earlier [6].

2. Materials and experimental procedure

Seven grades of beryllium were investigated with the different characteristics of the initial powder and properties of the billets made of them. Among them six grades (DshG-200,TR-30,TshG-56, TRR, TE-30, TIP-30) were manufactured by VNIINM (Russia) and other one (S-65B)–by Brush Wellman (USA). Chemical composition and initial characteristics of the materials are presented in Tables 1 and 2.

All the materials were irradiated at 650–700°C in active zone of CM-3 reactor in leaktight capsules up to the fast neutron fluence $(5.5-6.2) \times 10^{21}$ cm^{-2} ($E > 0.1$ MeV). Irradiation parameters are presented in Table 3. Two types of the specimens have been tested. First type,

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[*] Corresponding author. Tel.: +7-95-190 8015; fax: +7-95-925 5972/925 2896; e-mail: kupr@vniinm402.msk.su.

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Table 1
Chemical composition of beryllium grades, (wt.%)

Material	Be	BeO	Fe	Al	Si	C	Mg
DshG-200	99.34	0.79	0.024	0.0045	0.013	0.077	0.003
TshG-56	99.10	0.95	0.10	0.013	0.014	0.077	
TR-30	97.64	3.2	0.12	0.015	0.013	0.05	0.006
TRR	97.64	3.2	0.12	0.015	0.013	0.05	0.006
S-65B	$\geqslant 99.0$	0.98	0.09	0.018	0.026	$\leqslant 0.1$	<0.003
TE-30	98.13	2.5	0.11	0.015	0.013	0.088	
TIP-30	98.27	2.1	0.11	0.014	0.013	0.075	0.006

Table 2
Characteristics of beryllium

Material	Average grain size (μm)	Direction	Ultimate tensile strength (MPa)	Yield stress (MPa)	Total elongation (%)	Ultimate compressive strength (MPa)	Yield compressive stress (MPa)	Failure deformation	Compacting method
DshG-	22	Trans.	314	288	0.8	1606	497	31.0	VHP [b]+
200	25	Long.	240	–	0				Upsetting
TshG-56	22	Trans.	458	388	1.6	1653	561	29.5	VHP [b]+
	25	Long.	263	256	0.2				Upsetting
TR-30	9	Trans.	691	579	1.8	1927	609	30.1	VHP [b]
		Long.	589	580	0.2	2074	674	31.4	
TRR	9-8	Trans.	688	–	0	2100	688	33.4	HIP [a]
		Long.	690	–	0	1986	694	27.5	
S-65B	18-20	Long.	441	312	3.5	1752	563	37.8	VHP [b]
TE-30	16	Trans.	376	351	0.7	1296	504	23.4	VHP [b]+
	23	Long.	669	472	14	1807	398	35.8	Exstrusion
TIP-30	18	Trans.	487	416	1.8	1692	453	31.3	HIP [a]
	18	Long.	482	463	0.8	1677	463	30.5	

[a] Hot isostatic pressing.
[b] Vacuum hot pressing.

Table 3
Irradiation parameters of beryllium

Parameter	Material						
	DshG-200	TshG-56	S-65B	TRR	TE-30	TIP-30	TR-30
Neutron flux (E > 0.1 MeV) ($\mathrm{cm}^{-2}\ \mathrm{c}^{-1}$)				$(1.0–1.5) \times 10^{15}$			
Neutron flux (E < 0.68 eV) ($\mathrm{cm}^{-2}\ \mathrm{c}^{-1}$)				$(5.0–7.5) \times 10^{13}$			
F_s (E > 0.1 MeV) (cm^{-2})		$(4.2–6.2) \times 10^{21}$			$(3.9–5.5) \times 10^{21}$		$(3.9–5.8) \times 10^{21}$
F_{th} (E < 0.68 eV) (cm^{-2})		$(2.1–3.1) \times 10^{20}$			$(1.8–2.7) \times 10^{20}$		$(1.8–2.8) \times 10^{20}$
Radiation damage (dpa)		2.0–3.0			1.8–2.7		1.8–2.8
Irradiation temperature (°C)				650–700			
Helium content (appm)		770–1140			680–1010		680–1060

which was used for swelling and compressive characteristics measurements, represents the solid cylinders machined to close tolerances with 6 mm in diameter and 9 mm in length. The other type of the specimens with 3 mm in diameter and 28 mm in length were used for tensile testing and microstructure investigation. The testing was performed in air environment at room temperature (RT) and in vacuum at higher temperatures (>RT). The rate of straining was 1 mm min^{-1} both for tensile and compression testing. The errors were 5% and 10% for strength properties and elongation, accordingly. The error of the measurement of dimensions did not exceed 0.1–0.15%. Microstructure study was carried out by transmission electron microscope JEM 2000 FS-II.

3. Experimental results and discussion

3.1. Swelling

The results of swelling measurement are presented in Table 4. After irradiation swelling for all the samples was found to be in the range 0.1–2.1%. All the materials can be divided into two groups by the swelling values. The swelling value for the first group of materials (TR-30, TRR, TE-30, TIP-30) was rather small and did not exceed 0.4%, while that for the second group (S-6 5B, DshG-200, TshG-56) was significantly higher >1.3%. Data presented in Tables 2 and 4 confirm the conclusions of previous works [1–4], that fine-grained beryllium grades with increased beryllium oxide content are less susceptible to swelling at high temperature irradiation. In TR-30 and TRR grades, having the smallest grain size and the highest beryllium oxide content (3.2%), swelling did not exceed 0.1% after irradiation at 650–700°C with a fluence 5.5×10^{21} cm^{-2} ($E > 0.1$ MeV).

3.2. Mechanical properties

3.2.1. Tensile test results

The data on mechanical properties of beryllium under tensile testing before and under irradiation are presented in Table 5. Testing was performed both at RT and at 650°C. Irradiation at both temperatures results in a significant change of beryllium properties.

After irradiation, ultimate tensile strength (σ_b), yield stress ($\sigma_{0.2}$) and total elongation (δ) of beryllium decreased by 21–49%, 7–44% and 86–100%, respectively, when testing was performed at RT. Under testing at 650°C, σ_b, $\sigma_{0.2}$ vary from +10% to −47%, from +42% to −40% and δ decreases by 94–100% respectively. When testing both at RT and 650°C, the strength parameters (σ_b and $\sigma_{0.2}$) for the first group of materials (TR-30, TRR, TE-30, TIP-30) were significantly higher than that for the second group (S-65, DshG-200, TshG-56). Elongation for the first group of materials is identical to that for the second group or higher.

3.2.2. Compression test results

Table 6 presents the data on effect of irradiation on mechanical properties of beryllium under compression testing. Testing was performed at RT and in the range of 450–650°C.

Table 4
Swelling of beryllium samples

Material	DshG-200	TshG-56	S-65B	TRR	TE-30	TIP-30	TR-30
Swelling(%)	1.36 ± 1.1	2.1 ± 1.0	1.77 ± 1.0	0.1 ± 0.1	0.36 ± 0.3	0.38 ± 0.1	0.1 ± 0.05
Helium content (appm)		1060–1140			940–1010		

Table 5
Tensile test results of beryllium samples before and after irradiation with fluence up to $5.1–5.8 \times 10^{21}$ cm^{-2} (up to 1.8–2.8 dpa)

Material	Condition	T_{irr} (°C)	Test temperature (°C)					
			20			650		
			σ_b (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)	σ_b (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)
DshG-200	Unirr	–	314	288	0.8	161	144	17.2
	Irr	650–700	161	–	0	86	–	0
TshG-56	Unirr	–	458	388	1.6	131	121	24.4
	Irr	650–700	298	–	0	74	73	0.2
TR-30	Unirr	–	691	579	1.9	202	188	13.3
	Irr	650–700	461	–	0	160	153	0.8
TRR	Unirr	–	688	–	0	235	213	17.9
		650–700	511	–	0	174	170	1.1
TE-30	Unirr	–	669	472	14.0	103	79	34
	Irr	650–700	457	427	2.0	113	112	0.9
S-65	Unirr	–	441	312	3.5	105	96	19.0
	Irr	650–700	287	–	0	83	83	0.5
TIP-30	Unirr	–	487	416	1.8	182	167	11.2
	Irr	650–700	386	–	0	130	127	0.6

The rate of straining – 1 mm min^{-1}.

Table 6
Compressive test results of beryllium samples before and after irradiation with fluence up to 5.5–6.2 × 10^{21} cm^{-2} (up to 2.7–3.0 dpa)

Material	Condition	T_{irr} (°C)	Test temperature (°C)						
			20			450	500	550	650
			σ^c_b (MPa)	$\sigma^c_{0.2}$ (MPa)	ε (%)	$\sigma^c_{o.2}$ (MPa)	$\sigma^c_{0.2}$ (MPa)	$\sigma^c_{0.2}$ (MPa)	$\sigma^c_{0.2}$ (MPa)
DshG-200	Unirr	–	1606	497	31.0	464	351	318	316
	Irr	650–700	846	766	2.2	411		267	
TshG-56	Unirr	–	1653	561	29.5	286	279	255	
	Irr	650–700	856	540	1.0	290		284	
TR-30	Unirr	–	1927	607	30.1	439	426	370	361
	Irr	650–700	1234	1078	3.5		546		234
TRR	Unirr	–	2100	688	33.4	503	475		317
	Irr	650–700	1333	840	11.1		615		391
TE-30	Unirr	–	1807	398	35.8	383	286	257	
	Irr	650–700	963	604	10.5		349		
S-65	Unirr	–	1752	563	37.8	268		242	
	Irr	650–700	899	617	6.2	336		247	
TIP-30	Unirr	–	1692	454	31.3	380	364	344	249
	Irr	650–700	1187	670	13.9		380		335

The rate of straining – 1 mm min^{-1}.

Testing at RT show, that after irradiation at 650–700°C compressive strength (σ^c_b) and failure deformation (ε) decreased by 30–49% and 56–97%, accordingly, while compressive yield stress ($\sigma^c_{0.2}$) increased by 10–78%, that is a characteristic of the irradiated beryllium. Failure deformation for the materials of the first group was in the range of 3.5–4%, while that for the second group was 1.0–6.2%. By testing at 450–650°C it was shown that $\sigma^c_{0.2}$ increased by 10–78%. Unfortunately, attempts to carry a test to failure at 450–650°C have not met with success due to a good plasticity of initial beryllium at higher temperatures (>50–60%) and also because of the restrict force of the testing machine, used for testing at higher temperatures. At the maximal stress of 900 MPa, the deformation of beryllium after irradiation was 10.3–22.3%.

These results show that the materials of the first group, having higher mechanical properties in the initial condition, demonstrate less degradation of the mechanical properties under irradiation. The advantage in elongation of the second group of materials (S-65B) disappears under irradiation.

The tendency for a degradation of mechanical properties revealed at this work, allows to anticipate, that under the neutron irradiation a resistance of the second group of materials against a thermal fatigue will reduce faster than that of the first group. It can lead to a loss of the advantage in the resistance against thermal shock/fatigue, reported in [7,8] for S-65B and DshG-200 grades in unirradiated condition. Therefore, it seems improper to choose beryllium grade taking into account either thermal fatigue or neutron irradiation behaviour separately.

To validate the choice for fusion application there is a need to summarise the data on beryllium behaviour under combined exposure to thermal loading and neutron irradiation. In addition, it seems reasonable to say that the different approaches should be used, when choosing beryllium for the application in the different fusion elements.

3.3. Microstructure of irradiated beryllium

Microstructures of the beryllium irradiated at different temperatures are shown on Fig. 1. It was found that in the samples investigated the bubble formation takes place on the grain boundaries, inside the grains of a matrix and on the interface of matrix-second phase. The bubbles have a specific crystallographic form. In addition, the linear aggregates of pores have been brought out in matrix, whose formation took place on dislocations occurred in material before the irradiation annealing.

The main features of the helium bubble formation in the samples are as follows:

1. The largest bubbles are found at the matrix-second phase interface. The bubble dimension is comparable with the dimension of the inclusion.
2. The largest bubbles in the matrix decorate the initial dislocations dissociated by irradiation.
3. Among the matrix bubbles the disk-like bubbles are generally revealed with a thickness of 2–2.5 nm and a diameter of 15–20 nm. The bubbles lie in the basal plane (0 0 2) of hexagonal close-packed lattice of the beryllium.
4. The size of grain boundary bubbles depends on the grains orientation. They have a lens-like form with axis ratio 2:1. Grain boundary bubbles as well as

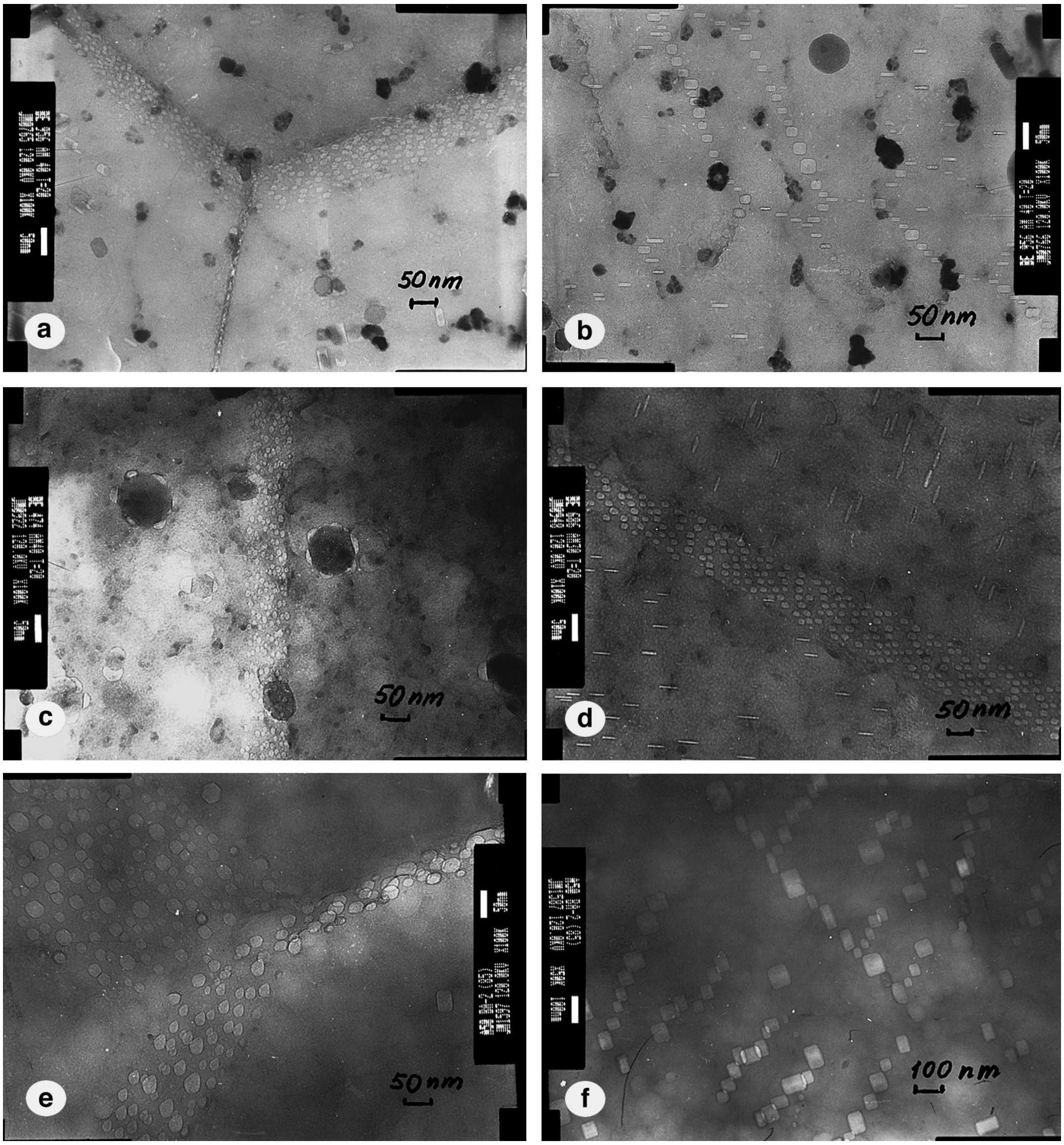

Fig. 1. Microstructure of beryllium irradiated at 650–700°C: (a,b) TE-30 grade; (c,d) TR-30 grade; (e,f) S-65 grade; (a–e) × 100 000; (f) × 50 000.

the bubbles inside the grains result in the distortion of crystal lattice.

The average size of the bubbles was found in S-65B beryllium grade. It is 20.6 nm in matrix, although the bubbles formed earlier at dislocations are larger by a factor of 2. The average size of the bubbles, which formed on the boundaries, varies from 17.5 to 20 nm depending, on the orientation of the grains. The size of matrix and boundary bubbles in DshG-200 and TshG-56 grades was 15 nm and 7–12 nm (depending on the grain orientation), accordingly. In TR-30 grade the size of grain-boundary pores was 3–10 nm.

4. Conclusion

The study on swelling, mechanical properties and microstructure of seven grades of beryllium has been carried out. It has been confirmed, that fine-grained beryllium grades with high beryllium oxide content are less susceptible to swelling at high temperature irradiation. Beryllium grades TR-30 and TRR having the smallest grain size and the highest beryllium oxide content (3.2%), demonstrate the minimal swelling, which does not exceed 0.1% after the irradiation with fluence 5.5×10^{21} cm^{-2} ($E > 0.1$ MeV) at 650–700°C.

Irradiation results in a significant change of mechanical properties of beryllium and its embrittlement. Beryllium grades having the higher level of strength properties in initial condition and less susceptibility to swelling (group I), demonstrate less degradation of strength properties under irradiation. The advantage in initial elongation for the second group of materials (S-65) disappeared under irradiation.

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