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The method design, manufacture and tests of the porous beryllium mock-ups for the ITER breeding blanket [1]

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Abstract

It is assumed that the porous beryllium will be used for the beryllium multiplier zones of the ITER breeding blanket. In the present report the preliminary results on fabrication technique and properties of porous beryllium are described. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

The fabrication technique comprises several successive steps, including a preparation of mixture of beryllium powder and beryllium hydride and manufacture of beryllium mock-up with 15% porosity.

Using the manufacturing procedure the porous beryllium mock-up in a SS shell with 2 mm wall thickness was fabricated.

Measurements and calculations have shown that the average porosity of the mock-up is near 15%. The results indicate that all the samples have the guaranteed open porosity.

The mechanical properties as averaged based on the results of two to three tests for each porosity are: for compression tests – 798 MPa and $\epsilon = 3.9\%$ (for the 12.36% sample porosity) and 412 MPa and $\epsilon = 2.4\%$ (for the 20.56% sample porosity); for tensile tests – 19 MPa and $\delta = 0$ (for the 18.36% sample porosity) and 72 MPa and $\delta = 0.8\%$ (for the 12.0% sample porosity).

The mean thermal expansion coefficient (TEC) for the two temperature ranges ($\alpha_{20\text{–}400^\circ\mathrm{C}}$ and $\alpha_{20\text{–}800^\circ\mathrm{C}}$ (1/°C) is: $(14.7 \pm 0.5 - 17.6 \pm 0.5) \times 10^{-6}$.

The measured thermal conductivity values were 80 ± 10 W/(m K) for the 18.69% sample porosity and 120 ± 10 W/(m K) for the 13.0% sample porosity.

1.1. Initial materials

For fabrication of the porous beryllium mock-up in the stainless steel (SS) shell a mixture of different percent ratio of commercial beryllium powder (CBP) of CBP-56 grade with the particles size – 56 μ, CBP-30 grade with the particles size – 30 μ and beryllium hydride (BeH_2) were used. For the fabrication of the mock-up with 15% porosity a hydride additive to beryllium powder was 8% wt. The beryllium content of the CBP-56 and CBP-30 grade powder is $\geqslant 98.5\%$ wt., while that of oxygen $\leqslant 1.0\%$ wt. The heavy metals impurity content in beryllium hydride is up to 0.1% wt., and content of oxygen – up to 0.8% wt. The characteristics of beryllium initial powders are listed in Table 1.

2. Brief description of the low temperature pressing (LTP) technique of beryllium mock-ups with 15% porosity

The technique comprises several successive steps:

1. Careful blending a mixture of beryllium powder and beryllium hydride to homogeneous condition in a single-line inductive rotator (SLIR) during 2 min.
2. Backfilling of the SS shell with the mixed powder and placing it into a heating die.
3. Placing of the heating die on the hydraulic press with the protective camera.
4. Heating of the die with the mixture in air at a heating rate up to 10°C/min to the temperature as high as 170°C.

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Table 1
Characteristics of initial beryllium powders

Element	Grades of beryllium powder	
	Chemical composition (wt. %)	
	CBP-56	CBP-30
Be	99.00	98.61
Si	0.014	0.014
Fe	0.15	0.18
Mn	0.012	0.0084
Mg	0.011	0.0093
Cr	0.036	0.064
Ni	0.014	0.017
Al	0.017	0.024
Cu	0.004	0.0071
C	0.077	0.07
O	0.66	1.0

5. Pressing of powder mixture at the pressure of 250–300 MPa to reach the additive density.
6. Heating of the pressed billet up to the temperature of 250–300°C at the heating rate of 3–5°C/min and keeping it under the pressure during 30–40 min.
7. Depressurization and cooling of the die down to room temperature.
8. Pressing out the powder billet from the die.

It is to be noted that in the resent experiments to produce porous mock-ups with slits the pressing force was reduced from 400 to 250–300 MPa as compared to the previous results [1].

We also anticipate a further reduction in the pressing forces via the optimization of the process parameters.

3. Description of preparing technique for porous beryllium samples

Using the technology, described in Section 2, eight porous beryllium mock-ups were fabricated in a SS shell with 2 mm thick with and without radial slits.

The fabricated briquettes of porous beryllium tightly adjoined the SS shell without visible clearances.

The bottom of the shell was removed by machining. The bottom part of the porous briquette was a compact porous structure. No diffusion bonding between the SS bottom and the porous beryllium was noted.

Then the initial porous mock-up was cut into two parts: the lower and upper ones. From both the parts 16 cylinders of different sizes were machined which were cut out of different parts and in different directions of the mock-up. This approach allowed an unbiased estimation of the structure, mechanical properties and, particularly, porosity and inhomogeneity of porosity within the mock-up volume.

4. Techniques and results of porous beryllium mock-up studies

4.1. Structure

Microsections in two mutually perpendicular planes for upper and lower segments of the mock-up were prepared. The microsection size for the planes parallel to pressing axis had: for the upper segment – 45 mm × 12 mm; for the lower segment – 45 mm × 25 mm. It have been found that the mode of the structure for the mutually perpendicular planes is the same, the pores have different shapes from round to elongated ones.

Fractured surfaces of tensile-tested porous beryllium were examined by SEM Fig. 1(a) and (b).

The examination evidenced individual large pores which are quite uniformly distributed. Also, it revealed the porosity as irregular channels at the powder particle interface. In the plane of fracture equiaxial pores were 6 μm in size while elongated ones could be as big as 30 μm.

In parallel with this, fine-mesh porosity was detected. It was characterized by aggregation of 1–2.5 μm pores separated with walls less than 0.2 μm thick.

To detect macroporosity, pores in microsections made from mutually perpendicular planes were filled with zinc oxide. With the filler, contrast helped to identify the shape, size and location of pores. Their spatial arrangement was evident from layer-by-layer removal of beryllium.

Micrographs demonstrate:

- quite uniform pore distribution throughout the volume;
- instead of individual pores an extensive system of interfacial channels which assure an open porosity;
- fine-mesh (1–2.5 μm) porosity (see Fig. 1(a) and (b)).

4.2. Average porosity, open porosity and inhomogeneity of porosity into mock-ups volume

To determine the specified features two techniques were used [2].

For the more unbiased evaluation of porosity and inhomogeneity of porosity 16 cylinders were prepared. Each cylinder was weighed, its geometric sizes were measured to the accuracy ± 0.05 mm and the average density, inhomogeneity of density (porosity, inhomogeneity of porosity) were determined within the mock-up volume.

Measurements and calculations for 16 cylinders have shown that the average porosity of the mock-up is 14.85%. However, the inhomogeneity of the porosity along the mock-up height was observed, that varied from the average value of 12.44% in the upper part of the mock-up to 18.58% in the lower part of the mock-up. Density of dense beryllium is assumed equal to 1.847 g/cm^3 [3].

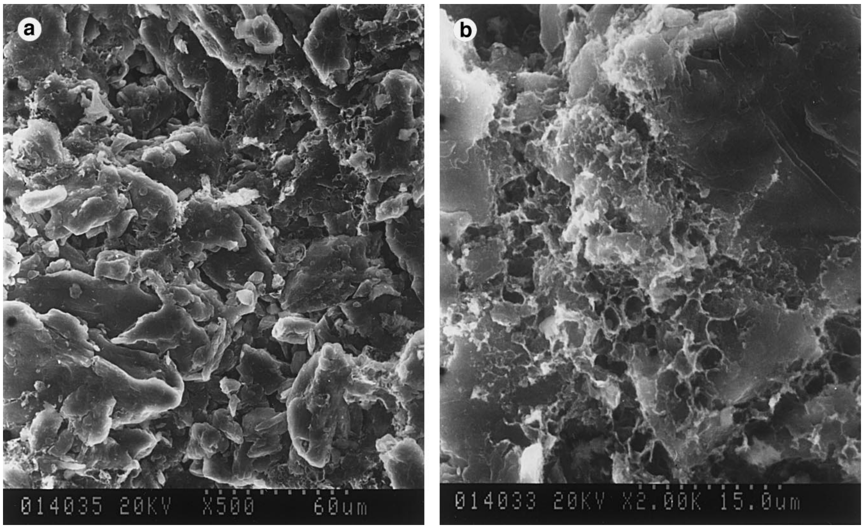

Fig. 1. SEM micrographs of the porous beryllium sample (a,b); tensile-tested fractured sample surfaces.

The per cent open and closed porosity was determined by hydrostatic weighing in high purity decane according to [2]. The open porosity was found from the formula

$$P = ((m_2 - m_1)/(m_2 - m_3)) \times 100[\%],$$

where m_1 is the mass of a dry sample, m_2 the mass of decane saturated sample and m_3 the hydrostatic mass of a sample in decane.

According to [2] the measurements were carried out using samples cut out of the upper and lower parts of the mock-ups. The measured results are given in Table 2.

The results indicate that all the samples have the guaranteed open porosity. The fact that the experimentally found porosity values are somewhat higher than the calculated ones can be explained by the following:

- deviation (to the side of increasing) of the actual density of the compact material from the handbook density of beryllium metal which results in too low values of the calculated porosity;
- an error in determining the value of m_2 due to the availability of the working liquid at the sample surface.

Thus, as evidenced by SEM and hydrostatic suspension the fabrication technique developed is adequate to assure open porosity in beryllium.

4.3. Mechanical properties

The cylinders of the mock-up lower part were used to prepare samples to be tensile and compressive tested.

The tensile samples had a total length of 28 mm and the gauge dia of 3 mm. The compressive samples had a

Table 2
The measured results for total and open porosity of the beryllium samples

Samples number	Some characteristics of the porous beryllium samples					
	Diameter (mm)	Height (mm)	Weight (g)	Density (g/cm^3)	Porosity (%)	
					Total	Open
1	9.96	29.37	3.4107	1.492	19.23	19.22
2	6.98	24.64	1.4153	1.502	18.69	20.47
3	9.02	23.54	2.2792	1.515	17.97	18.87
4	7.94	31.22	2.2947	1.484	19.63	20.73
5	9.95	29.57	3.4173	1.488	19.45	20.68
6	10.00	28.79	3.3911	1.500	18.80	20.05
7	8.00	10.85	0.8885	1.610	12.86	14.48
8	9.97	14.52	1.8279	1.612	12.71	12.67
9	9.95	10.68	1.3502	1.628	11.88	13.04
10	9.96	10.51	1.3659	1.668	9.69	10.71
11	9.96	12.24	1.5847	1.662	10.03	10.46
12	7.99	11.24	0.9340	1.657	10.27	12.95

Table 3
Compression mechanical properties of beryllium with open porosity

Test temperature (°C)	Density (g/cm^3)	Open porosity (%)	Mechanical properties		
			σu (MPa)	σy (MPa)	εfr (%)
20	1.68	9.2	890	837	9.6
	1.64	11.2	794	744	3.9
	1.56	15.6	575	–	3.2
	1.548	16.3	528	528	4.1
	1.47	20.56	412	–	2.4
400	1.667	9.87	>575	447	>11.1
	1.617	12.6	>587	443	>11.5
	1.55	16.2	>520	330	>22.9
	1.427	22.86	370	322	12.5

Table 4
Thermal expansion coefficient of porous beryllium

Open porosity (%)	$\alpha_{20\text{–}400^\circ\mathrm{C}}$ (1/°C)	$\alpha_{20\text{–}800^\circ\mathrm{C}}$ (1/°C)
18.26	$14.6 \pm 0.5 \times 10^{-6}$	$18.2 \pm 0.5 \times 10^{-6}$
	$14.9 \pm 0.5 \times 10^{-6}$	$17.6 \pm 0.5 \times 10^{-6}$
	$14.7 \pm 0.5 \times 10^{-6}$	$17.0 \pm 0.5 \times 10^{-6}$
13.0	$16.5 \pm 0.5 \times 10^{-6}$	$17.2 \pm 0.5 \times 10^{-6}$
	$15.5 \pm 0.5 \times 10^{-6}$	$18.0 \pm 0.5 \times 10^{-6}$
	$16.0 \pm 0.5 \times 10^{-6}$	$17.6 \pm 0.5 \times 10^{-6}$

Table 5
Thermal conductivity vs. open porosity of beryllium

Beryllium grade	Open porosity (%)	Thermal conductivity at 320 K, W/m K
S65B	1.0 [a]	190 ± 10
LTP	13.0	120 ± 10
LTP	17.3	95 ± 10
LTP	18.69	80 ± 10

[a] close porosity.

6 mm dia and 9 mm height.The test was carried on at room temperature at the crosshead speed of 2 mm/min.

Results of tension and compression tests of porous beryllium at room temperature and 400°C are given in Table 3.

It is to be noted that during testing porous beryllium behaves like a typical sintered material. A good correlation is observed between the level of beryllium properties and the amount of its open porosity. At 400°C the samples demonstrated a high strength and ductility and did not fail in the tests, therefore, the actual strength and ultimate compressive strain of the samples are much higher.

4.4. Thermal expansion coefficient

The thermal expension coefficient (TEC) was measured with a high temperature dilatometer of the 'Adamel' (France), DHT-60 model, with the CT-60 furnace at 1500°C in helium. During the measurements the heating rate was 5–6°C/min. The accuracy of the measurements was ± 1–3%. The investigations were implemented in the temperature range of 20–820°C.

To measure the TEC two cylindrical samples were taken from the mutually normal planes: from the mock-up top of 13% porosity and the central sample from the mock-up bottom of 18.26% porosity.

In the specified temperature range three measurements for each sample were carried out.

The mean TEC for the two temperature ranges is given in Table 4.

It is to be noted that the TEC value for porous beryllium is close to ones for dense beryllium [3–5].

4.5. Thermal conductivity

A technique similar to standard method of measurement for the longitudinal bars has been expressly developed for this work. Briefly the technique is as follows. A specimen bar 10 mm in diameter and ranging in length from 30 to 47 mm is heated in vacuum 10^{-4} Pa by electron bombardment at one end. At another end the bar is clamped between lead plates. Temperature gradient along the specimen is evaluated by measuring temperature at two points of the bar separated $L = 10$–16 mm. For this purpose, Cr–Al thermocouple electrodes 0.2 mm in diameter are spot-welded at the points through 0.1 mm nickel plate welded to the bar before. Thermocouple signals are registered by 'Philips' PM 8120 *x-y* recorder. Normally after 5 min heating the steady-state difference in temperature of the points ΔT is established. Ignoring radiant losses the magnitude of thermal conductivity K can be derived as follows: $K = WL/F\Delta T$, where W-input power, F-cross section area of the bar.

All runs were performed at the same $W = 1.15 \times U \times I$, where $U = 380$ V-electron energy, $I = 5$ mA-emission current. The electrical power is corrected by factor of 1.15 to include tungsten filament radiation. The accuracy of the K derivation is mainly limited both by low L available and edge effects.

As shown in Table 5, thermal conductivity depends strongly on the porosity of beryllium.

5. Conclusion

1. The scrap-free low temperature (250–300°C) pressing technique has been designed for porous beryllium items at the specified porosity level (15%) with the pore inhomogeneity within the volume not exceeding ±5%.

2. The porosity formed in beryllium is described by not only individual pores but also by a relatively developed system of intersectional tunnels promoting formation of inherently open porosity.

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