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Post irradiation characterization of beryllium and beryllides after high temperature irradiation up to 3000 appm helium production in HIDOBE-01

A.V. Fedorov^{a,*}, S. van Til^a, M.P. Stijkel^a, M. Nakamichi^b, M. Zmitko^c

^a Nuclear Research and Consultancy Group, Westerduinweg 3, Postbus 25, Petten, 1755 ZG, The Netherlands ^b Japan Atomic Energy Agency, Rokkasho, Japan ^c The European Joint Undertaking for ITER and the Development of Fusion Energy, c/ Josep Pla, n° 2, Torres Diagonal Litoral, Edificio B3,

Barcelona 08019, Spain

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ABSTRACT

Titanium beryllides are considered as advanced candidate material for neutron multiplier for the helium cooled pebble bed (HCPB) and/or the water cooled ceramic breeder (WCCB) breeder blankets. In the HIDOBE-01 (*HIgh DOse irradiation of BEryllium*) experiment, beryllium and beryllide pellets with 5 at% and 7 at% Ti are irradiated at four different target temperatures (T_{irr}): 425 °C, 525 °C, 650 °C and 750 °C up to the dose corresponding to 3000 appm He production in beryllium. The pellets were supplied by JAEA.

During post irradiation examinations the critical properties of volumetric swelling and tritium retention were studied. Both titanium beryllide grades show significantly less swelling than the beryllium grade, with the difference increasing with the irradiation temperature.

The irradiation induced swelling was studied by using direct dimensions. Both beryllide grades showed much less swelling as compare to the reference beryllium grade. Densities of the grades were studied by Archimedean immersion and by He-pycnometry, giving indications of porosity formation. While both beryllide grades show no significant reduction in density at all irradiation temperatures, the beryllium density falls steeply at higher $T_{\rm irr}$.

Finally, the tritium release and retention were studied by temperature programmed desorption (TPD). Beryllium shows the same strong tritium retention as earlier observed in studies on beryllium pebbles, while the tritium inventory of the beryllides is significantly less, already at the lowest T_{irr} of 425 °C.

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1. Introduction

Titanium beryllide (BeTi) is considered as promising neutron multiplier for the helium cooled pebble bed (HCPB) and/or the water cooled ceramic breeder (WCCB) breeder blankets [1]. As compared to pure beryllium, BeTi is characterized by higher melting temperature, lower chemical reactivity with stainless steel and water vapour, lower swelling and lower retention of tritium [1,2].

HIDOBE-01 and HIDOBE-02 are two long-duration irradiation campaigns conducted at High Flux Reactor in Petten, aimed to investigate behaviour of candidate grades of beryllium and beryllides under the DEMO blanket relevant neutron irradiations conditions in terms of dpa/He ratio's and temperatures. The HIDOBE-01 and HIDOBE-02 irradiations targeted the helium

* Corresponding author. E-mail address: fedorov@nrg.eu (A.V. Fedorov).

http://dx.doi.org/10.1016/j.fusengdes.2015.10.024 0920-3796/© 2015 Elsevier B.V. All rights reserved. production in beryllium at levels of 3000 and 6000 appm, respectively. The latter corresponds to 30% of the DEMO End-Of-Life helium production [3]. The irradiation set temperatures were 425, 525, 650 and 750 °C.

The material matrix irradiated in both HIDOBE experiments is identical and presents various grades of European, Russian and Japanese beryllium, and titanium beryllides provided by Japan.

Both irradiation campaigns have started in June 2005, and were successfully completed in October 2007 (HIDOBE-01) and in August 2011 (HIDOBE-02), respectively.

The HIDOBE-01 Post Irradiation Examination (PIE) programme was conducted in 2009–2012 under EFDA contract TW2-TTBB-004b and F4E grant-030 Action 3, in collaboration with KIT (Karlsruhe Institute of Technology), UL (University of Latvia), and ITN (Instituto Tecnológico e Nuclear, Lisbon). The PIE comprised the following studies: dimension measurements, density measurements using helium pycnometry and immersion, tritium





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Table 1-1	
HIDOBE-01 and HIDOBE-02 irra	adiation campaigns.

	HIDOBE-01	HIDOBE-02
Fluence ($E_n > 0.1 \text{ MeV}, \times 10^{26} \text{ n/m}^2$)	1.5	3
Fluence ($E_n > 1 \text{ MeV}, \times 10^{26} \text{ n/m}^2$)	0.7	1.4
Helium production in Be (appm)	3000	6000
Tritium production in Be (appm)	250	700
Neutron damage in Be (dpa)	17.9	35.8
Helium production in Be ₁₂ Ti (appm)	2740	5480
Tritium production in Be ₁₂ Ti (appm)	235	562
Neutron damage in Be ₁₂ Ti (dpa)	19.5	38.9
Irradiation target temperature (°C)	425, 525, 650, 750	425, 525, 650, 750

desorption and X-ray diffraction. The PIE of the HIDOBE-02 is currently underway.

This paper presents the HIDOBE-01 PIE results performed on pure beryllium pellets and two grades of titanium beryllide pellets (Table 1-1).

2. Experimental

2.1. Materials

The beryllium and titanium beryllide specimens were provided by NGK INSULATORS, Ltd, Japan. Two grades of beryllides were prepared by arc melting 5 at% Ti and with 7 at% Ti. According to the pre-irradiation characterization the Be–5at%Ti contained Be and Be₁₂Ti grains. The microstructure of Be–7%Ti contained Be₁₀Ti, Be₁₂Ti and Be phases. Further in the text the two grades are referred as 'Be5Ti' and 'Be7Ti'. The chemical composition of the specimens is presented in Table 2-1.

The samples presented \emptyset 8 mm disks with 2 mm in thickness.

2.2. HIDOBE-01 irradiation experiment

The beryllium specimens, pebbles and pellets, were placed in the EUROFER-97 drums that are positioned inside the sample holder. Geometry of each drum is designed to maintain a specific temperature and specific set of specimens. In total 13 drums of three different types, A, B and C, were stacked vertically as shown in Fig. 2-1, covering eight temperature zones with four well defined set temperatures, 425, 525, 650 and 750 °C. The drums were enclosed inside a double wall irradiation assembly. The irradiation assembly was instrumented with 24 thermocouples located in different temperature zones, and 5 fluence detectors. During the irradiation all drums were purged with the reference gas, He + 0.1%H₂. Deviations from the target temperatures are small during all 25 irradiation

Table 2-1

Chemical composition of the beryllium and beryllides (provided by JAEA).

	Be Content, wt%	Be–5at%Ti Content, wt%	Be–7at%Ti Content, wt%
Ве	99.5	78.0	71.2
BeO	0.02	0.26	0.27
Ti		21.7	28.5
V		< 0.01	< 0.01
W		< 0.05	< 0.05
Mg	0.05	< 0.001	< 0.001
Al	0.07	0.064	0.060
Si	0.03	0.020	0.033
Cr	<0.01	0.006	0.005
Fe	0.10	0.034	0.031
Со	0.0003	< 0.001	< 0.001
Ni	0.01	0.002	0.003
Cu	<0.01	0.006	0.004
Mn	0.007	0.008	0.007
Sc		< 0.001	< 0.001
U	0.0068	0.0059	0.0041



Fig. 2-1. Temperatures zones in HIDOBE-01 experiment.

 Table 2-2

 The HIDOBE-01 irradiation parameters.

425	525	650	750
0.41	0.51	0.6	0.65
0.57	0.69	0.81	0.89
11.3	13.9	16.3	18.1
1890	2300	2680	2950
176	213	252	285
	425 0.41 0.57 11.3 1890 176	425 525 0.41 0.51 0.57 0.69 11.3 13.9 1890 2300 176 213	425 525 650 0.41 0.51 0.6 0.57 0.69 0.81 11.3 13.9 16.3 1890 2300 2680 176 213 252

cycles. For the central thermocouples the deviations do not exceed 3%, for the rest of the thermocouples the difference is within 5-10% and for few 10-15%.

The HIDOBE-01 irradiation was completed after 25 reactor cycles, or 649 days at full reactor power (>40 MW). The neutron fluences, helium and tritium production levels, and neutron damage in beryllium were obtained from the MCNP/FISPACT calculations and validated with the data from the on-board fluence detectors. The results are presented in Table 2-2. The set irradiation temperature within each zone was maintained by using a specific geometry of the drum. Due to the axial buckling of the neutron flux there is a correlation between the irradiation temperature and the neutron fluence, and consequently helium and tritium production rates, observed in Table 2-2.

The beryllium and beryllides pellets presented in this work were irradiated inside the central channel of the drums type C at all four set temperatures. The top view of the drum type C is shown in Fig. 2-2. From four drums of C type only two, drum 5 and drum 7, were instrumented with thermocouples, which were located at the edge of the drum. The thermo-mechanical calculations showed a significant radial gradient present in the drum. Thus the central temperature 525 °C in drum 5 corresponds to an edge temperature

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