NEUTRON IRRADIATION OF BERYLLIUM PEBBLES — D. S. Gelles, H. Tsai[1], and R. M. Ermi (Pacific Northwest National Laboratory)* and (Argonne National Laboratory)[1]

OBJECTIVE

The purpose of this work was to provide quantitative stepped helium release response from prototypic irradiated beryllium pebbles. Such pebbles are under consideration as the neutron multiplier medium in the European Fusion Technology Program Helium Cooled Pebble Bed (HCPB) Blanket.

SUMMARY

Seven subcapsules from the FFTF/MOTA 2B irradiation experiment containing 97 or 100% dense sintered beryllium cylindrical specimens in depleted lithium have been opened and the specimens retrieved for postirradiation examination. Irradiation conditions included 370°C to 1.6×10^{22} n/cm^2, 425°C to 4.8×10^{22} n/cm^2, and 550°C to 5.0×10^{22} n/cm^2. TEM specimens contained in these capsules were also retrieved, but many were broken. Density measurements of the cylindrical specimens showed as much as 1.59% swelling following irradiation at 500°C in 100% dense beryllium. Beryllium at 97% density generally gave slightly lower swelling values.

PROGRESS AND STATUS

Introduction

Twenty-two subcapsules containing beryllium specimens have been irradiated in U.S. DOE Office of Fusion Energy Sciences (OFS) experiments in the Fast Flux Test Facility (FFTF) in Hanford, WA. With the increased difficulty for performing irradiation experiments, due in part to the shutdown of FFTF and EBR-II and other reactors worldwide, these specimens are becoming very valuable. The German fusion materials development program centered at Forschungszentrum Karlsruhe INR has been concentrating efforts on design of a fusion blanket using beryllium as a neutron multiplier. Therefore, Forschnungszentrum Karlsruhe INR has concluded that it is efficient use of their available funds to invest in the testing of some of these specimens. This report is intended to provide details concerning FFTF experimental design, subcapsule specimen loading, specimen removal operations from seven of the capsules, and density change measurements on selected specimens from those capsules.

Experimental Details

It was possible to obtain permission to include beryllium specimens in the U.S./DOE OFS irradiations of fusion reactor materials in FFTF Materials Open Test Assembly (MOTA) irradiations 2A and 2B performed in 1989 and 1991, respectively. The MOTA design placed specimens or their containers in direct contact with flowing reactor sodium, and therefore it was decided to irradiate the beryllium samples in subcapsules made from the molybdenum alloy TZM and in direct contact with depleted lithium in order to minimize impurity pickup. Two specimen geometries were irradiated: right solid cylinders of pressed and sintered beryllium provided by

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G.L Longhurst of INEEL, Idaho Falls, ID, and transmission electron microscopy (TEM) disks. The sintered beryllium specimens were prepared at EG&G Rocky Flats with manufacturing details previously described by Beeston etal [1]. Sintered densities were either 80, 85, 97 or 100%. Specimens were all 0.300 inches in diameter by either 0.25, 0.80 or 0.1.20 inches in height. TEM specimens were 3mm (0.125 inch) disks of pure beryllium punched from a sheet 0.010 inches thick made from A65B powder and called PF60 foil from Brush Wellman or of niobium beryllide ($Be_{12}Nb$) obtained from S.M. Bruemmer (PNNL) for subcapsules containing 1.20 inch specimens. Punching was done at room temperature, with the understanding that microcracking might result. Specimen loading details are provided in Table 1 and dosimetery estimates are provided in a companion report beginning on page 64.

It was decided to attempt to open seven of the TZM capsules at Argonne National Laboratory using facilities previously used to open similar TZM capsules containing vanadium alloys in lithium. The seven capsules are defined in Table 2. These capsules were selected because they either contained 97 or 100% dense beryllium cylinders as well as TEM specimens. Note that the TEM disks in V491 are of $Be_{12}Nb$ whereas all other TEM specimens are of sintered beryllium at the same density as the larger specimens in the subcapsule. The disassembly was conducted in an alpha-free air cell. Subcapsule V491 was opened first, as a test case, and then the remaining subcapsules were opened. Capsules were opened with a tube cutter and the contents with subcapsule remnants, in a wire basket, were transferred to a beaker containing liquid ammonia. After dissolution of the lithium, the wire basket was transferred into an alcohol bath for cleaning. After drying, an inventory of the contents of the wire basket was made. Specimens were then placed in vials and transferred to PNNL.

Right cylinder specimens 0.250 inches in height from the capsules describe in Table 2 were selected for density measurements. (Larger specimens were not expected to fit into the density cell.) Density measurements were performed in a cell designed for larger specimens using water as a buoyancy medium. Calibration was based on measurements using laboratory standards.

Results

Specimen retrieval from FFTF subcapsules was very successful. There were two types of specimens in each capsule: cylinders and TEM disks. The condition of the retrieved cylinders appeared to be good, i.e., without obvious cracks or deformation as seen through the hot cell window with a surveyor's telescope. The condition of the TEM disks was mixed: some were intact but some were fractured. The fractures appeared to have occurred during irradiation as the broken disks could be seen immediately after the lithium dissolution before any handling. As many of the broken pieces were collected as possible in case good uses could be found for them. Very small pieces, i.e., less than ≈¼ the size of a full disk, had a tendency to fall through the mesh screen used to contain the specimens. These very small pieces were therefore impossible to retrieve. An inventory of specimens recovered from the capsules is provided in Table 2. Therefore, right cylinders appeared in excellent shape, and at least one whole TEM specimen was obtained from each capsule, with better success retrieving TEM specimens from capsules irradiated at 370°C, indicating that irradiation was probably responsible for TEM specimen degradation.

Results of density measurements on right cylinder specimens 0.250 inches in height are provided in Table 3. The results shown in Table 3 demonstrate that swelling is highest following irradiation at 550°C, with values between 1.23 and 1.59%, the higher value corresponding to the fully dense condition. Following irradiation at 425, the swelling is found to be lower, between 0.23 and 0.51%, again with the higher value corresponding to the fully dense condition. The lowest swelling observed was for specimens irradiated at 370°C to approximately one third the fluence, where swelling was between 0.13 and 0.29%, but this time the 97% dense specimen showed higher swelling. It can be noted that differences on successive measurements were less than 0.003%.

Table 1. Beryllium containing subcapsules in fusion reactor materials irradiation tests

Irrad. Vehicle	Packet ID	Dimensions (in.)	Contents including Li mass (in gms.)
MOTA 2A	V460	.375 OD x 2.5	2 0.30" Be cylinders & 3 TEMs/Li (.19)
"	V461	.375 OD x 2.5	2 0.30" Be cylinders & 3 TEMs/Li (.19)
"	V462	.375 OD x 2.5	2 0.30" Be cylinders & 3 TEMs/Li (.18)
"	V463	.375 OD x 2.5	2 0.30" Be cylinders & 3 TEMs/Li (.18)
"	V464	.375 OD x 2.5	2 0.30" Be cylinders & 3 TEMs/Li (.19)
"	V458	.375 OD x 1.8	0.30" Be cylinder/Li (.14)
MOTA 2B	V308	.375 OD x 2.5	4 0.30" Be cylinders/Li (.22)
"	V309	.375 OD x 2.5	4 0.30" Be cylinders/Li (.21)
"	V310	.375 OD x 2.5	2 0.30" Be cylinders & 12 TEMs/Li (.21)
"	V311	.375 OD x 2.5	2 0.30" Be cylinders & 7 TEMs/Li (.21)
"	V312	.375 OD x 2.5	0.30" Be cylinder & 7 TEMs/Li (.23)
"	V487	.375 OD x 2.5	4 0.30" Be cylinders/Li (.21)
"	V488	.375 OD x 2.5	4 0.30" Be cylinders/Li (.24)
"	V489	.375 OD x 2.5	2 0.30" Be cylinders & 12 TEMs/Li (.21)
"	V490	.375 OD x 2.5	2 0.30" Be cylinders & 7 TEMs/Li (.21)
"	V491	.375 OD x 2.5	0.30" Be cylinder & 3 TEMs/Li (.24)
"	V566	.375 OD x 2.5	4 0.30" Be cylinders/Li (.23)
"	V567	.375 OD x 2.5	3 0.30" Be cylinders & 2 Discs/Li (.22)
"	V568	.375 OD x 2.5	2 0.30" Be cylinders & 11 TEMs/Li (.23)
"	V569	.375 OD x 2.5	3 0.30" Be cylinders & 6 TEMs/Li (.20)
"	V570	.375 OD x 2.5	0.30" Be cylinder & 3 TEMs/Li (.23)
"	V571	.375 OD x 2.5	0.30" Be cylinder/Li (.24)

Table 2. Inventory of specimens retrieved

Capsule No.	Irradiation Conditions	Loading	Retrieved
V310	370°C, $1.59x10^{22}$	2 cylinders (97%) 12 TEM disks	2 cylinders 11 whole TEM disks
V311	370°C, $1.59x10^{22}$	2 cylinders (100%) 7 TEM disks	2 cylinders 7 whole TEM disks
V489	425°C, $4.76x10^{22}$	2 cylinders (97%) 12 TEM disks	2 cylinders 4 whole TEM disks 3 ≈50% TEM pieces 2 <50% TEM pieces
V490	425°C, $4.76x10^{22}$	2 cylinders (100%) 7 TEM disks	2 cylinders 2 whole TEM disks 4 ≈50% TEM pieces 2 <50% TEM pieces
V491	425°C, $4.76x10^{22}$	1 cylinder (100%) 3 TEM disks ($Be_{12}Nb$)	1 cylinder 1 whole TEM disk 2 ≈50% TEM pieces
V568	550°C, $4.95x10^{22}$	2 cylinders (97%) 11 TEM disks	2 cylinders 6 whole TEM disks 5 ≈70% TEM pieces 1 ≈50% TEM pieces
V569	550°C, $4.95x10^{22}$	2 cylinders (100%) 6 TEM disks	2 cylinders 2 whole TEM disks 3 ≈70% TEM pieces 3 <50% TEM pieces

CONCLUSIONS

Seven subcapsules from the FFTF/MOTA 2B irradiation experiment containing 97 or 100% dense sintered beryllium cylindrical specimens in contact with depleted lithium have been opened and the specimens retrieved for post-irradiation examination. Irradiation conditions included 370°C to $1.6x10^{22}$ n/cm^2, 425°C to $4.8x10^{22}$ n/cm^2 and 550°C to $5.0x10^{22}$ n/cm^2.

TEM specimens contained in these capsules were also retrieved, but many were broken. Higher irradiation temperatures resulted in more breakage indicating that irradiation was probably responsible for specimen degradation.

Density measurements of the cylindrical specimens showed as much as 1.59% swelling following irradiation at 550°C in 100% dense beryllium. Beryllium at 97% density generally gave slightly lower swelling values. A limited inventory of irradiated beryllium specimens for microstructural examination is now available, including 97% dense and 100% dense pure beryllium as well as $Be_{12}Nb$.

Table 3. Results of density measurements on 0.200 inch right cylinders either unirradiated or irradiated in selected FFTF/MOTA 2B subcapsules. Results for two successive measurements on the same sample are shown.

Subcapsule	Reactor Conditions (°C, n/cm^2 E>0.1 MeV)	Form	weight in air (mg)	weight in liquid (mg)	Density (gm/cm^3)	Result
Unirr.		100% Be	535.789 535.792	247.62 248.08	1.869674 1.868958	Average 1.869316
Unirr.		97% Be	532.899 532.961	245.019 245.334	1.861462 1.859638	Average 1.86055
V310	370°C, 1.59x10^{22}	97% Be	527.59252 7.548	241.895 241.88	1.857009 1.853378	Swelling 0.29%
V311	370°C, 1.59x10^{22}	100% Be	511.399 511.305	236.164 236.184	1.868426 1.865169	Swelling 0.13%
V489	425°C, 4.76x10^{22}	97% Be	525.949 525.877	241.432 241.172	1.858902 1.853756	Swelling 0.23%
V490	425°C, 4.76x10^{22}	100% Be	531.479 531.192	244.235 244.429	1.860613 1.859051	Swelling 0.51%
V568	550°C, 4.95x10^{22}	97% Be	523.434 523.27	237.339 237.182	1.839821 1.835650	Swelling 1.23%
V569	550°C, 4.95x10^{22}	100% Be	516.718 516.655	234.394 234.663	1.840474 1.838780	Swelling 1.59%

FUTURE WORK

This work will be continued when further funding is available.

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REFERENCES

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