10.4 IRRADIATION STUDY OF ADVANCED CERAMICS: STATUS OF IRRADIATION EXPERIMENTS

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OBJECTIVE

This report describes the technical planning and progress of neutron irradiation experiments on advanced ceramics materials for potential use in fusion structural components.

SUMMARY

HFIR Rabbit capsules were designed for irradiation of various advanced ceramics. The materials include isotopically-controlled ultra-high temperature ceramics such as TiB_2 and ZrB_2, MAX phases such as Ti_2AlC, Ti_3AlC_2, and Ti_3SiC_2, SiC with various qualities, AlN, TiC, YAG, glassy carbon, and Si. The first phase of the irradiation has been completed.

PROGRESS AND STATUS

Introduction

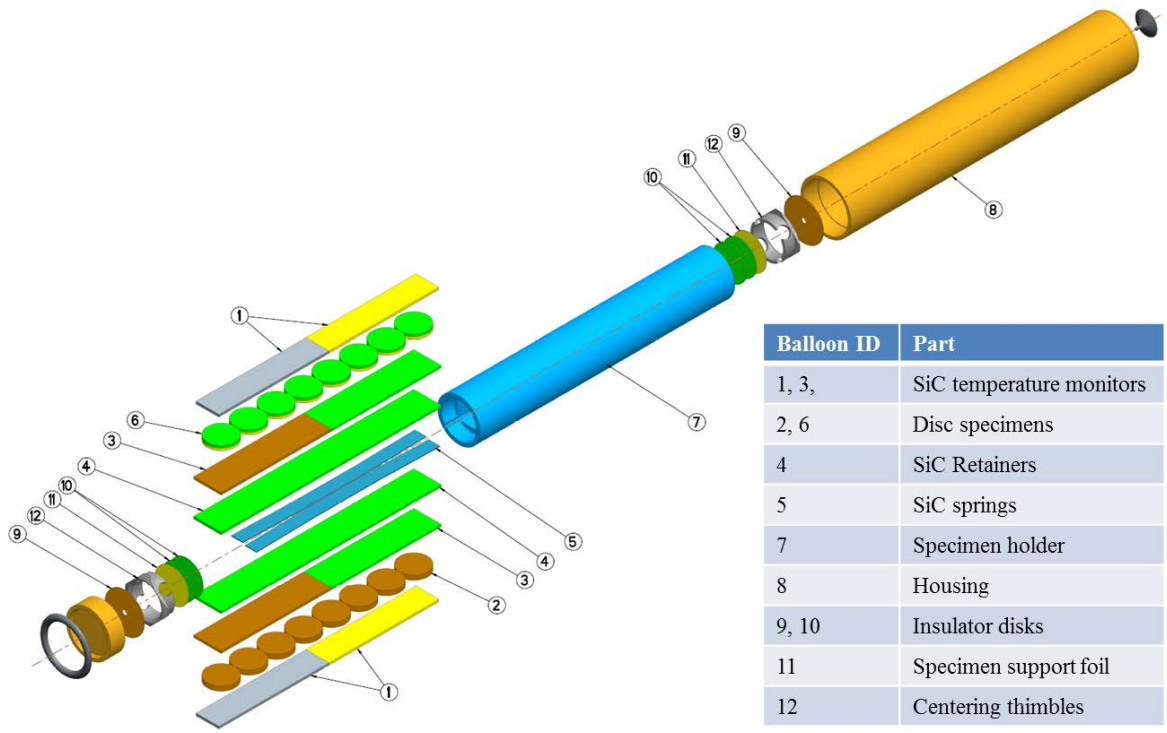
The novel high-temperature ceramics including ultra-high-temperature ceramics (UHTCs) and a family of layered ternary compounds (MAX phases) are recently recognized as attractive materials for use in nuclear fusion applications [1]. UHTCs are generally capable of surviving temperatures of ~2000°C because of their extremely high melting temperatures (above ~3000°C). There are potential fusion applications for these materials including tokamak divertor and plasma facing materials [1]. MAX ($M_{n+1}AX_n$) phase materials, where M is an early transition metal, A is an A-group element, and X is either C or N, exhibit interesting mechanical properties. More than a few percent of failure strain was achieved for a Ti_3SiC_2 material at 1200°C [2], which is significantly larger than common ceramics and beneficial for structural applications. Though UHTCs and MAX phase materials are attractive materials used in extreme environments, the major concern for nuclear fusion applications is irradiation effects on the mechanical and physical properties. Very limited information regarding neutron irradiation effects on these materials is available.

The aim of this task is understanding radiation resistance of the advanced ceramics. The materials investigated are UHTCs such as TiB_2 and ZrB_2, MAX phases such as Ti_2AlC, Ti_3AlC_2, and Ti_3SiC_2, and additional high-temperature ceramics including SiC with various qualities, AlN, TiC, YAG, glassy carbon, and silicon. Note that the diboride samples were isotopically controlled; >99 wt% purity of ^{11}B powder was used for fabrication instead of ^{10}B powder to avoid producing transmutant helium during irradiation. Irradiation experiments using rabbit capsules will be conducted. This report presents the capsule design, the current status of HFIR irradiation, and plans for the post-irradiation examination.

Capsule design and test specimens

The overall capsule assembly is shown in Figure 1. This rabbit has 32 disk specimens that are nominally 0.5 mm thick and 6.0 mm diameter. There are two sets of 16 specimens located on either side of the center retainer spring. Each set has 2 layers of 8 specimens stacked between SiC thermometry pieces. The arrangement of the disk specimens changes from one capsule to another. Although the loading order is unique for each capsule, the total volume that the specimens occupy is the same for all capsules. There are SiC thermometry bars and SiC retainer specimens pressed against the disk specimens. Thermometry and specimens are pushed to the inside surfaces of the holder with SiC springs. There are insulator discs at the top and bottom of the assembly to reduce axial heat loss.

A summary of the irradiation conditions and test materials is given in Table 1. A total of 8 rabbit capsules are to be irradiated. The target irradiation temperatures range from 200 to 1000°C. The number of HFIR cycles is up to 5, and the neutron fluence will be up to ~1×10^{26} (n/m^2, E > 0.1 MeV).



Balloon ID	Part
1, 3,	SiC temperature monitors
2, 6	Disc specimens
4	SiC Retainers
5	SiC springs
7	Specimen holder
8	Housing
9, 10	Insulator disks
11	Specimen support foil
12	Centering thimbles

Figure 1. Exploded view of the rabbit capsule assembly. The table shows the explanation of each capsule part.

Table 1. Irradiation and specimen matrix.

Capsule ID	Target Irradiation Temperature [°C]	Number of HFIR Cycles	Materials Investigated
IMR 3	200	1	SX 3C SiC, SX 6H SiC, Faulted SiC, Si, AlN, YAG, TiC,
IMR 4	200	1	TiB_2, ZrB_2, Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon
IMR 6	600	1	TiB_2, ZrB_2, Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon, Si, AlN, YAG, TiC
IMR 7	600	3	TiB_2, ZrB_2, Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon, YAG, TiC
IMR 8	600	5	TiB_2, ZrB_2, Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon, Si, YAG, TiC
IMR 9	1000	1	Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon, AlN, YAG, TiC
IMR 10	1000	5	Ti_3SiC_2, Ti_3AlC_2, Ti_2AlC, Sintered SiC, Glassy carbon, YAG, TiC
IMR 11	1000	5	TiB_2, ZrB_2, Sintered SiC

Simulation of irradiation temperatures

Irradiation capsules were designed to control the specimen temperatures using three-dimensional thermal analysis performed with the ANSYS software package. The simulations consider the arrangement of the specimens and the influence of <1at% of B^{10} content in the diborides on heat generation in the capsules. A calculation showed that the heat generation of the diborides changes significantly during irradiation, indicating changes in specimen temperatures [3]. At the initial stage, high heat generation is due to neutron absorbance by B^{10}. After ~13 days of irradiation, all the B^{10} burns out, and the heating rate largely drops by ≥70%. Figures 2A & B shows the simulation results for the irradiation temperatures at the end of the irradiations. The specimen temperatures at the end of irradiation are generally within 50-60°C of the design temperature for all design cases, and they are within ~30°C of the 200°C design case. In addition, the temperature differences at the beginning and the end of irradiations are as large ~300°C for the diborides and ~50–~150°C for the other materials in the capsules containing diborides specimens. The magnitude of these temperature differences depends on the specific design (capsule materials, fill gas, etc.), specimen matrix, and specimen location within the capsule. The actual irradiation temperatures of the specimens will be experimentally investigated by dilatometry of 8 SiC temperature monitors which are designed to be in contact with the disc specimens.

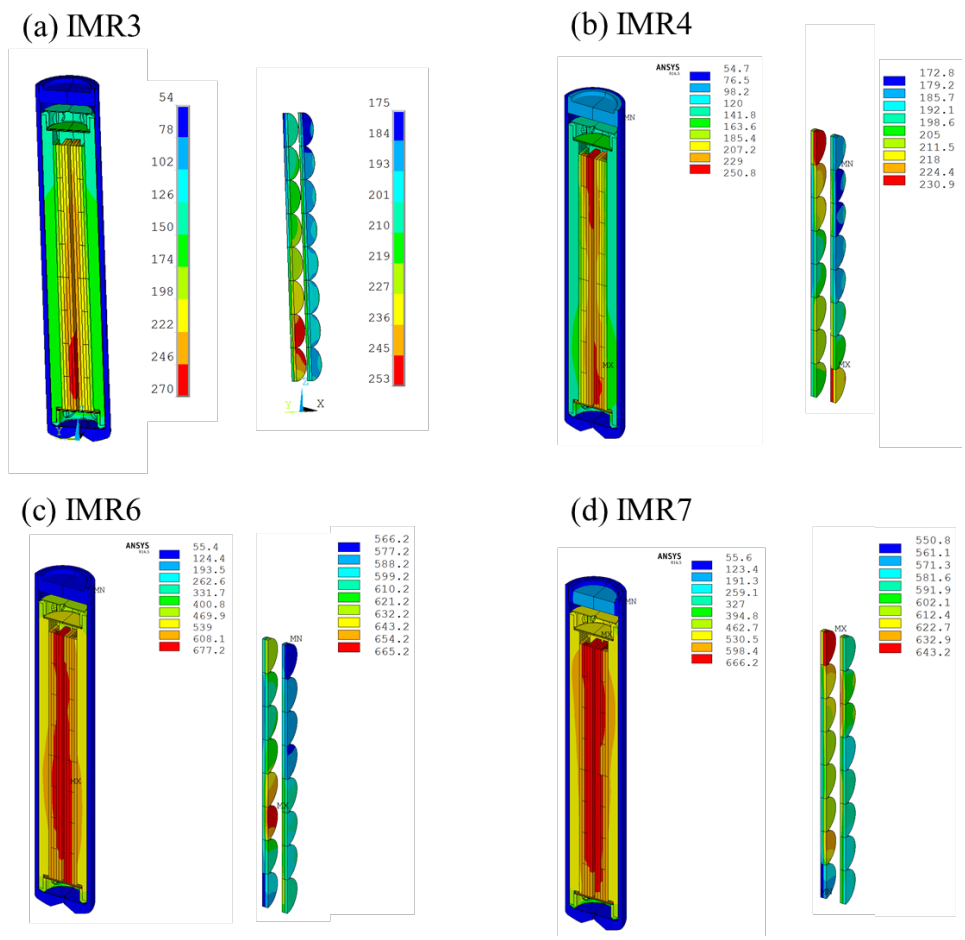


Figure 2A. Calculated temperature distributions expected at end of irradiation for ceramic irradiation capsules designed for 200C (IMR 3 & 4) or 600C (IMR 6 & 7).

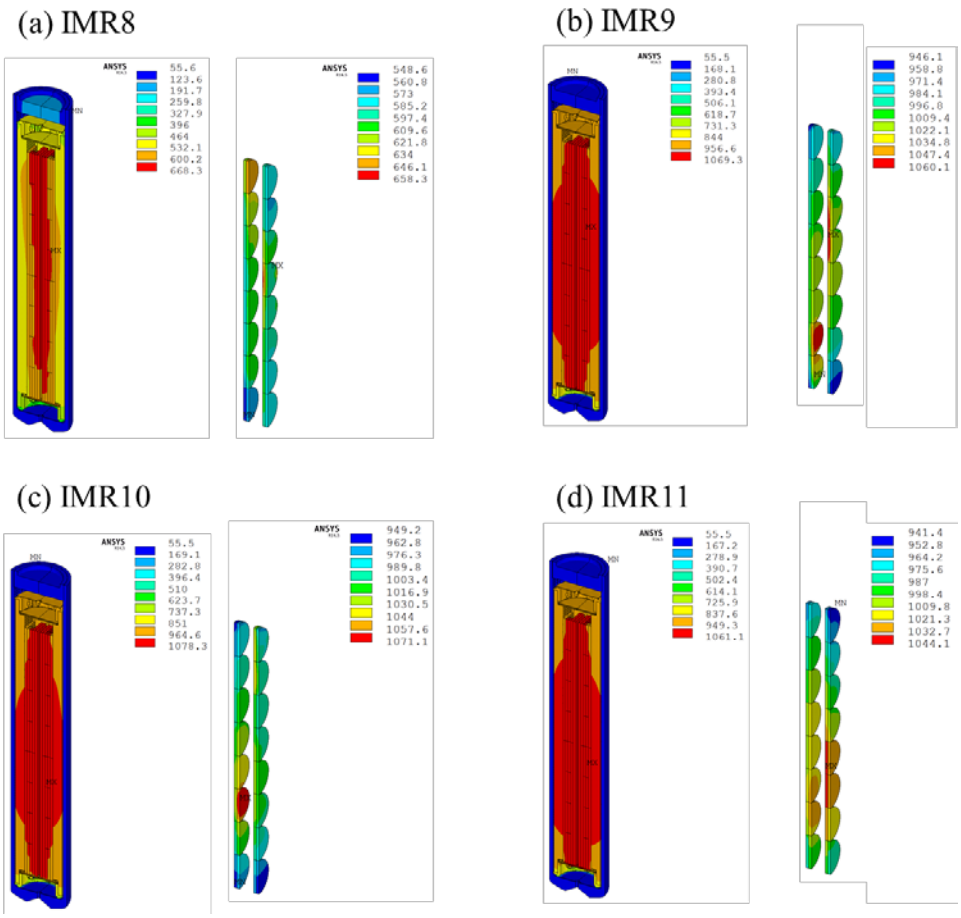


Figure 2B. Calculated temperature distributions expected at end of irradiation for ceramic irradiation capsules designed for 600C (IMR 8) or 1000C (IMR 9, 10 & 11).

Plan for post irradiation experiments

Disk specimens with 6 mm diameter and 0.5 mm thickness will be used. Post-irradiation examination will include investigations of swelling, thermal properties, electrical properties, equibiaxial flexural strength at room temperature and high temperature, microstructure, and positron annihilation lifetime. In addition, dilatometry of SiC samples placed in the irradiation capsules will be conducted to measure actual irradiation temperatures.

Progress of irradiation and future work

The irradiation of the IMR3 capsule has been completed and the other capsules are under fabrication for the irradiation. The next step is characterization of unirradiated specimens to obtain reference information to compare with results obtained from the irradiated materials.

References

[1] W.E. Lee, M. Gilbert, S.T. Murphy, and R.W. Grimes, J. Am. Ceram. Soc., 96 [7] (2013) 2005–2030.
[2] M.W. Barsoum and M. Radovic, Annual review of materials research 41 (2011) 195–227.
[3] ORNL internal document, C-HFIR-2014-022 Rev.1