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MICROSTRUCTURAL CHARACTERIZATION OF BURNABLE ABSORBER MATERIALS BEING EVALUATED FOR APPLICATION IN LEU U-MO FUEL PLATES

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ABSTRACT

The starting microstructure of a fuel plate will impact how it performs during irradiation. This includes any burnable absorber materials that may be added to a dispersion or monolithic fuel plate to compensate for excess reactivity and/or to flatten the radial power profile. The borated compounds B_4C, ZrB_2, and Al-B alloys have been selected for testing in the ATR as part of the RERTR-13 test, and to support completion of this test, microstructural characterization has been performed on the samples in the as-fabricated condition. This paper will discuss the results of optical metallography, scanning electron microscopy, and transmission electron microscopy analyses that have been performed. Overall, there was a fairly significant difference in how the boron-containing phases were distributed in the various samples. The ZrB_2 and B_4C samples had the most uniform distribution of the boron-containing phases. The HIP process used to fabricate the final plate samples effectively closes porosity that was present in some of the foil samples in the as-rolled condition.

1. Introduction

Burnable absorbers (BA) are used in a reactor fuel plate when it is necessary to suppress excess initial reactivity in a fuel and/or to flatten the radial power profile within a fuel element. In ATR and HFIR fuel elements, B_4C is currently used as the burnable absorber [1,2]. As part of the development of low-enriched U-Mo fuel, it is of interest to identify BA material(s) that can be employed for the reactors where the use of such a material is required and also to identify the best methodology of incorporating the material into the fuel plate (particularly for the monolithic fuel) such that there will be no impact of the BA material on the mechanical integrity of the fuel plate during irradiation.

The RERTR-13 reactor experiment being performed in the Advance Test Reactor (ATR) (see Table 1) will test BA-containing samples where the phases that contain boron (B), are B_4C, ZrB_2, or AlB_2. The test is comprised of 28 plates (8 non-fueled material test plates, 8 dispersion plates, and 12 monolithic plates). The B-10 concentrations employed in the materials are prototypic of what would be employed in LEU ATR plates [1]. The prime phenomena of interest in these plates are helium behavior, fuel swelling, blister anneal failure temperature and interaction layer behavior. In order to understand the effects of irradiation, it is necessary to perform detailed pre-test characterization on as-fabricated samples. The pre-test characterization was comprised of optical metallography (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) analyses. This paper will describe the

microstructures that were observed and will focus on the morphology and distribution of the phases that contain the boron.

Table 1. RERTR-13 Experiment Matrix.

ID	Capsule	Column 1	Column 2	Column 3	Column 4
13-A	Top	A-1	A-2	A-3	A-4
		U-7Mo + B_4C dispersion in Al-4 wt% Si, U235-25%	U-7Mo + B_4C dispersion in Al-4 wt% Si, U235-69%	U-7Mo + ZrB_2 dispersion in Al-4 wt% Si, U235-69%	U-7Mo + ZrB_2 dispersion in Al-4 wt% Si, U235-25%
	Bottom	A-5	A-6	A-7	A-8
		Aluminum Dummy	Aluminum Dummy	U-10Mo monolithic + B_4C-Al dispersion, U235-66%	U-10Mo monolithic + Al-1.5B alloy U235-66%
13-B	Top	B-1	B-2	B-3	B-4
		U-10Mo monolithic + ZrB_2-Al dispersion, U235-19.7%	U-10Mo monolithic + ZrB_2-Al dispersion, U235-66%	U-10Mo monolithic + B_4C-Al dispersion, U235-66%	U-10Mo monolithic + B_4C-Al dispersion, U235-19.7%
	Bottom	B-5	B-6	B-7	B-8
		Al-4.5B dispersion	Al-1.5B dispersion	ZrB_2-Al dispersion (Enriched B)	B_4C-Al dispersion
13-C	Top	C-1	C-2	C-3	C-4
		U-10Mo monolithic + ZrB_2-Al dispersion, U235-19.7%	U-10Mo monolithic + ZrB_2-Al dispersion, U235-66%	U-10Mo monolithic + B_4C-Al dispersion, U235-66%	U-10Mo monolithic + B_4C-Al dispersion, U235-19.7%
	Bottom	C-5	C-6	C-7	C-8
		Aluminum Dummy	Aluminum Dummy	U-10Mo monolithic + B_4C-Al dispersion, U235-66%	U-10Mo monolithic + Al-4.5B alloy, U235-66%
13-D	Top	D-1	D-2	D-3	D-4
		U-7Mo + B_4C dispersion in Al-4 wt% Si, U235-25%	U-7Mo + B_4C dispersion in Al-4 wt% Si, U235-69%	U-7Mo + ZrB_2 dispersion in Al-4 wt% Si, U235-69%	U-7Mo + ZrB_2 dispersion in Al-4 wt% Si, U235-25%
	Bottom	D-5	D-6	D-7	D-8
		Al-4.5B dispersion	Al-1.5B dispersion	ZrB_2-Al dispersion	B_4C-Al dispersion

2. Experimental

Table 2 enumerates the six samples that were characterized, their form, the specific analyses that have been performed to date, and the B-containing phase in the material. The laminate samples were fabricated by blending 400 mesh (< 37 μm) powders of B_4C and ZrB_2 powders (supplied by Ceradyne, Inc.) and aluminum powders, followed by compacting of the powders, and then rolling of the material to a desired thickness inside of an aluminum can. The edges of the can were then trimmed, leaving a layer of boron-bearing material sandwiched between two thin layers of AA6061 cladding. The single layer samples were fabricated by commercially procuring boron-bearing ingots from Ceradyne, Inc. and then rolling them to the desired thickness. The Al-4.5B (nominal wt% composition) ingot was manufactured by adding a boron halide to molten aluminum, and the Al-1.5B ingot was manufactured by adding a mixture of boron-halide and titanium-halide compounds to molten aluminum [1]. The single layer and laminate materials were inserted into fuel plates that were fabricated using the hot isostatic pressing (HIP) method [3]. All four samples (Al-1.5B, Al-4.5B, B_4C, and ZrB_2) were characterized in the unassembled (free foil) condition, and two samples (Al-1.5B and ZrB_2) were also analyzed in the as-HIPed condition.

The SEM analyses were conducted using a JEOL 7000F FEG SEM with energy-dispersive and wavelength-dispersive spectrometers (EDS/WDS) and a Zeiss Ultra-55 field emission SEM with X-ray energy dispersive spectroscopy (XEDS). Backscattered electron (BSE) imaging and X-ray mapping was performed on transverse cross sections of the samples to identify the microstructures and partitioning of the sample constituents amongst different phases. Specimens for TEM were prepared with a focused ion beam (FIB) in-situ (INLO) technique using a FEI™ TEM 200 FIB. The TEM analyses were performed using a FEI/Tecnai F30 300keV TEM/STEM equipped with a Fischione high-angle annular dark field (HAADF) detector. Electron diffraction was performed in the TEM to accurately identify the constituent phases based on crystal structure.

Table 1. Samples Employed for Microstructural Characterization

Label	Sample Form	Nominal B Loading (wt%)	Percentage of B that is B-10	Analysis Performed	B-Containing Phase
Al-1.5B	Single Layer	1.5	95.90	OM, SEM	AlB_2
Al-4.5B	Single Layer	4.5	98.50	OM, SEM	AlB_2
ZrB_2	Laminate	9.6	75.17	OM, SEM, TEM	ZrB_2
B_4C	Laminate	1.9	97.58	OM, SEM	B_4C
Al-1.5B HIP	HIP plate	1.5	95.90	OM, SEM, TEM	AlB_2
ZrB_2 HIP	HIP plate	9.6	75.17	OM, SEM	ZrB_2

3. Results

3.1 Al-1.5B and Al-1.5B HIP Sample

Figure 1 shows a BSE image of the microstructure observed for a section of the Al-1.5B sample. A heterogeneous distribution of precipitates was observed in the sample, along with relatively large areas of porosity. X-ray mapping of the sample demonstrated that the dark-contrast precipitates are enriched in Al, and the bright-phase precipitates are enriched in Ti. The Al-1.5B HIP sample had a similar microstructure without relatively large regions of porosity.

TEM analysis was performed on the Al-1.5B HIP sample. The bright contrast phases in the microstructure were comprised of Al_3Ti (larger particles) and $AlTi_3$ and Al_3Fe (smaller particles). The dark precipitates were AlB_2.

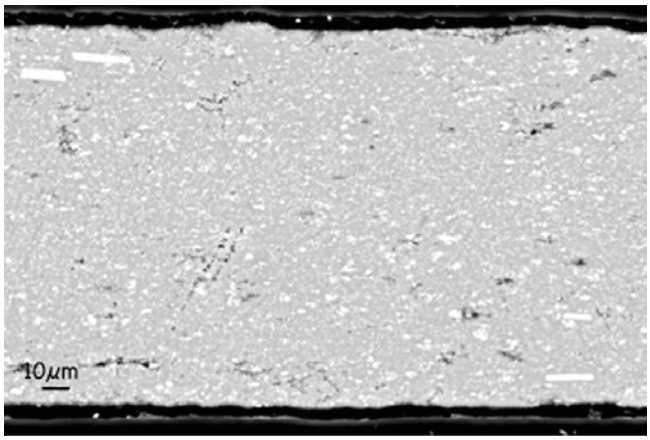

Fig. 1. BSE images of the microstructure observed for the Al-1.5B sample.

3.2 Al-4.5B Sample

Figure 2 shows a BSE image of the microstructure observed for a transverse cross section of the Al-4.5B sample. A heterogeneous distribution of precipitates were observed in the sample, along with relatively large areas of porosity. X-ray mapping of the microstructure demonstrated that the dark-contrast phases are enriched in Al and B.

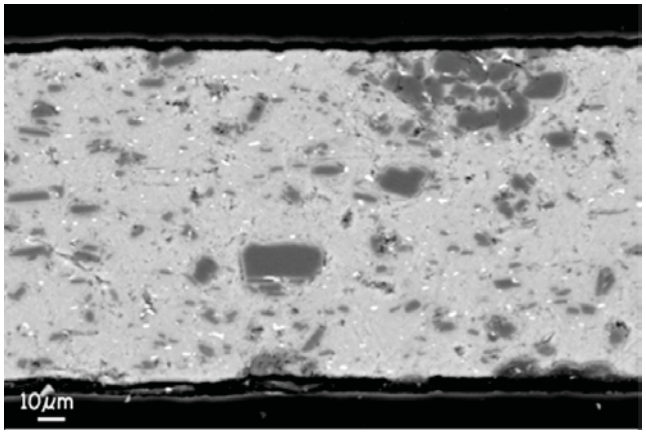

Fig. 2. BSE image of the microstructure observed for the Al-4.5B sample.

3.3 ZrB_2 and ZrB_2 HIP Sample

BSE images of the ZrB_2 sample microstructure are presented in Fig. 3. A generally uniform microstructure was observed with some areas of porosity. In the ZrB_2 HIP sample a similar microstructure was observed without any relatively large regions of porosity. TEM characterization of the ZrB_2 sample confirmed the presence of the ZrB_2 precipitate (bright contrast) and identified a minor $(Al,Si)_{12}Mg_{17}$ precipitate (dark contrast).

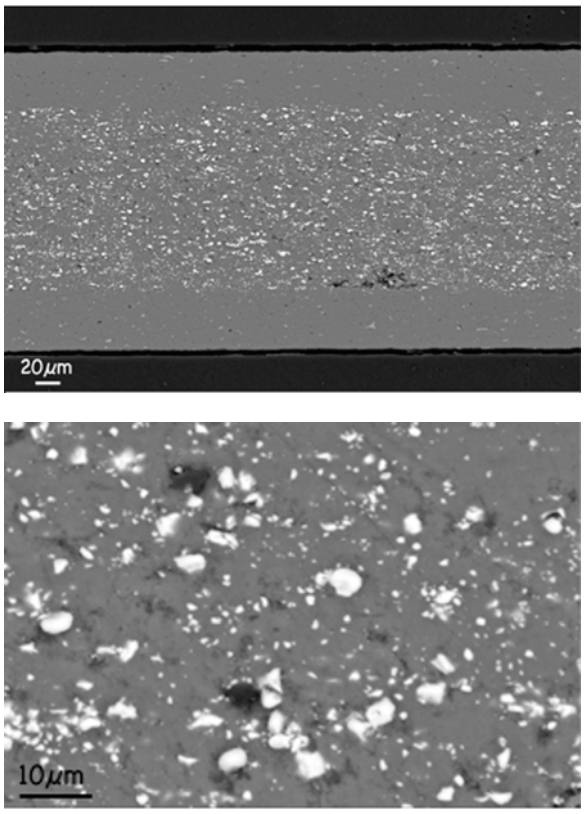

Fig. 3. BSE images of the microstructure observed for the ZrB_2 sample.

3.4 B_4C Sample

In Fig.4, the BSE image indicates that a fairly uniform microstructure was observed for the B_4C sample. The dark-contrast precipitates are the B_4C phase.

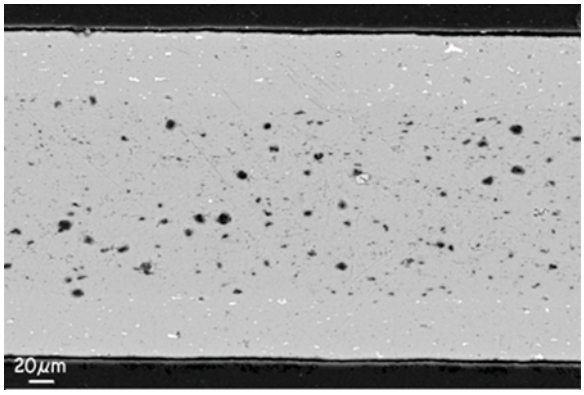

Fig. 4. BSE image of the microstructure observed for the B_4C sample.

4. Discussion

Microstructural characterization of Al-1.5B, Al-4.5B, ZrB_2, B_4C samples indicated that each sample has a very unique microstructure. The Al-1.5B sample has a significant fraction of Ti-containing precipitates and a minority AlB_2 phase that contains the boron. The Al-4.5B sample microstructure displays the AlB_2 phase as the majority phase, but there is a wide range in the size of the precipitates. The ZrB_2 sample has fairly uniform distribution of the ZrB_2 phase, but there is a fairly significant variation in the size of the precipitates. The B_4C sample also has a fairly uniform distribution of the B_4C phase and a farily significant variation in the size of the precipitates.

When microstructural analysis is performed on the as-irradiated samples, it will be of particular interest to track the development of the porosity in the microstructure due to the generation of He from nuclear reactions with the boron contained in the different phases.

5. Conclusions

Based on the microstructural analyses performed on the BA samples that will be tested in the RERTR-13 experiment, there was a fairly significant difference in how the boron-containing phases were distributed in the various samples. The ZrB_2 and B_4C samples had the most uniform distribution of the boron-containing phases. The HIP process used to fabricate the final plate samples effectively closes porosity that was present in some of the foil samples in the as-rolled condition.

Acknowledgments

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