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Journal of Nuclear Materials 352 (2006) 324–333

journal of nuclear materials

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New processing methods to produce silicon carbide and beryllium oxide inert matrix and enhanced thermal conductivity oxide fuels

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Abstract

For inert matrix fuels, SiC and BeO represent two possible matrix phase compounds that exhibit very high thermal conductivity, high melting points, low neutron absorption, and reasonably high radiation stability. BeO is chemically compatible with UO_2, PuO_2 and Zircaloy to very high temperatures, but SiC reacts with all three at somewhat lower temperatures. We have developed the *Polymer Impregnation and Pyrolysis* or *PIP* method, making use of a commercial SiC polymeric precursor, to consolidate both particulate fuels like ‘TRISO’ microsphere fuels, and to impregnate UO_2 fuels with pure stoichiometric SiC to improve their thermal conductivity. This method was employed to fabricate Enhanced Conductivity Oxide fuels, or ECO fuels with 5–10 vol.% of the high conductivity phase, and with 50 vol.% for TRISO dispersion fuels. For ECO fuels, a new ‘slug/bisque’ method of fabricating the UO_2 fuel granules was necessary to produce sintered fuel with open pore structures, allowing almost complete impregnation of the continuous SiC phase. The advantages of the PIP process are that it is a non-damaging consolidation process for particulates (TRU, UC or TRISO microspheres), forms a continuous, pure β-SiC phase at temperatures as low as 1573 K, and allows the maximum in fissile atom density. However, several PIP impregnation cycles and high crystallization temperatures are necessary to obtain high thermal conductivity SiC. For producing IMF fuels using the PIP process, the fissile PuC and/or TRU actinides can be added in small concentrations along with SiC ‘filler particles’ and consolidated with the SiC precursor for either open or closed fuel cycles. For BeO, a second approach was developed for ECO fuels that involves a ‘*co-sintering*’ route to produce high density fuels with a continuous BeO phase of 5–10 vol.%. Special granulation and mixing techniques were developed, but only one normal sintering cycle is required. For BeO matrix IMF fuels, PuO_2 granules and TRU actinides or YSZ granules containing actinides could be simply dry-mixed with BeO powder and co-sintered to produce a *dispersed* fuel form. Alternatively, the BeO could be granulated, and the PuO_2 and/or TRU oxides would fill the interstices forming a *continuous* minor phase that could be recycled in closed fuel cycles. Some advantages and disadvantages of these matrices are discussed.

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1. Introduction

High thermal conductivity matrix phases allow higher fissile atom loadings, and avoid many of the life-limiting phenomena associated with low conductivity matrices, providing they exhibit

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0022-3115/$ - see front matter © 2006 Published by Elsevier B.V.
doi:10.1016/j.jnucmat.2006.02.090

adequate chemical and radiation stability. We have studied various ways of incorporating a *minor*, *continuous* high conductivity phase in UO_2 fuels, which could result in significant benefits in power upgrading, and in severe accident scenarios like a LOCA. We call these Enhanced Conductivity Oxide or ECO, fuels. We show that these same techniques can be applied to the consolidation of dispersed particle fuels as well as TRU and Pu burning IMF fuels.

A review of high thermal conductivity compounds [1] with sufficient chemical compatibility with UO_2, stability in aqueous environments, compatibility with Zircaloy, neutronic properties, and irradiation performance, narrowed the choice to SiC and BeO. However, SiC is known to react with UO_2 in *open* systems at temperatures as low as 1643 K [2,3], but up to 2073 K in *closed* isothermal systems [4], and with Zircaloy at 1473 K [5]. The SiC matrix is stable to high temperatures with PuC as the fissile species, but SiC is very difficult to sinter below 2273 K without using large volume fractions of sintering aids [5]. BeO, on the other hand, can be sintered at typical fuel fabrication sintering temperatures, and is stable with UO_2 and PuO_2 up to their eutectic points of 2433 K [6] and 2408 K [7], respectively. BeO also forms a eutectic with ZrO_2 at 2418 K [6].

Ishimoto et al. [8] showed that a 25% increase in thermal conductivity of UO_2 could be obtained at 1100 K with an ‘almost’ continuous 4.2 vol.% of BeO phase at the grain boundaries. But to achieve this, they required processing above 2433 K, the eutectic temperature. Dispersed BeO in UO_2 that could be processed at lower temperatures did not result in the same improvements. For ECO fuels, we have found that 10 vol.% of continuous phase BeO in UO_2 increases the thermal conductivity ~50% over standard UO_2 fuel [9]. However, as opposed to the cubic β-SiC, BeO, has an hexagonal crystal structure and exhibits anisotropic crystal growth under energetic neutron bombardment that can lead to microcracking and even pulverization at high doses [10]. This tendency is related grain size, temperature and other factors not fully understood [10]. In addition, there is the production of helium by the $n, 2n$ and the n, α reactions on Be as well as the production of tritium by the (n, α) reactions on ^6Li, but these gas producing reactions are secondary to the irradiation growth problem [11]. Preliminary calculations show that the degradation in thermal conductivity from the formation of Pd_2Si [17] from fission fragment implanted Pd is negligible [18].

Pincell neutronics depletion calculations have been made with the Helios Code [19], which show a very small difference in reactivity decrease with burn-up for ECO fuels containing 10 vol.% of either SiC or BeO phase compared to typical UO_2 fuels. To achieve approximately the same burn-up ($K_{\text{inf}} = 1$) would require a small increase in enrichment from 4.64% for normal UO_2 fuels to 5%.

We also performed depletion calculations [19] on inert matrix fuels with compatible combinations of PuC or UC in a SiC matrix, and PuO_2 or 20% enriched UO_2 in a BeO matrix. The reactivity decrease with time is expectedly more rapid than for typical UO_2 or MOX fuel for the same fissile atom content of $3.3\ \text{g cc}^{-1}$. However, the void and temperature reactivity coefficients are negative in all the above cases even for weapons grade Pu. The PIP–SiC matrix has the additional advantage that B can be incorporated into the SiC structure simply by changing slightly the precursor chemistry. Our depletion calculations show that the reactivity decrease with 0.05 wt% B matches well that of the UO_2 or MOX fuel.

The issue we immediately considered with our industrial hygienists before undertaking work with BeO was toxicity. Our review of 10CFR850 [20] indicates that the controls for enriched UO_2 should be adequate for BeO as well. We are systematically measuring BeO air concentration levels ($<0.2\ \mu\text{g m}^3$) from our processing activities done in a fume hood wearing masks, and doing bioassays. Both are well below 10CFR850 limits. Even under the previous 10 times higher Permissible Exposure Limit of $2\ \mu\text{g m}^3$, the Atomic Weapons Establishment beryllium facility in Cardiff, Wales had only one case of chronic beryllium disease stemming from a non-standard event in over 36 years of operation, testifying that consistent, rigorous operational controls can avoid this disease in a commercial facility [21].

2. Experimental

The polymeric precursor, allylhydridopolycarbosilane, now called ‘SMP-10’ by the manufacturer (Starfire Systems, Inc.), produces, according to the manufacturer, near stoichiometric SiC after high temperature pyrolysis and crystallization. No free carbon could be identified in our microstructures. As the polymer polymerizes and decomposes, hydrogen and methane are liberated which presumably eliminates the excess carbon, and the volume contracts leaving SiC solid that is only ~1/3 the volume of the starting organic precursor. We have observed the gas evolution process using small transparent tubes slowly heated in a furnace, and have seen channel formation in the polymer, and even polymer ejection from the porosity if done too rapidly. Therefore several impregnations are necessary to obtain the maximum pore filling.

The BeO powder (Lot No. UOX–1771B), courtesy of Brush Wellman, is reported by the vendor to have a specific surface area of 12 $m^2 g^{-1}$, and is furnished in granules of nominally 20 μm to avoid becoming airborne. A highly sinterable depleted UO_2, (DUO_2), powder, courtesy of the Siemens Corporation, was used for all experiments.

Fig. 1 shows the measured particle size distribution of the UO_2 and the BeO powders on a volume basis, using a Coulter LS 230 particle size analyzer with sonication during the measurement or between measurements with the same result. The UO_2 powder was also measured to have a specific surface area of 2 $m^2 g^{-1}$ by BET measurements with nitrogen. It would appear that some of the UO_2 agglomerates are not broken up by sonication used.

2.1. The ‘Slug-Bisque’ (SB) granulation process

To introduce the SiC in the UO_2 via the PIP route, it was necessary to develop a coarse open pore structure, but with a high density UO_2 phase to maximize the fissile atom density. Similarly, for the BeO ‘co-sintering process’, stable granules were needed. Therefore, a so-called ‘Slug-Bisque’ (SB) process was developed which involved pre-slugging the UO_2 at 600 MPa, granulating the compacts using a mortar and pestle, and sieving in the size range of 45–500 μm. A ‘self-milling’ process was then used to smooth the surfaces and spheroidize the granules using a ball-mill without grinding media as described more fully below.

‘Bisquing’, or low temperature pre-sintering, was done at 1273 K for 2 h in flowing Ar–5%H_2. The SB process produces relatively robust granules that remained intact during subsequent pelletization at lower pressures (~300 MPa) yielding compacts of about 60% green density. Following compaction, sintering was carried out at 1873 K for 3–5 h under flowing Ar–5%H_2. The density of the UO_2 phase by quantitative image analysis was 98%TD. It was found that the final sintered density and open porosity values were insensitive to variations in the sintering time (above 3h), the final compaction pressures (above 240 MPa) or the initial compaction pressures (above 500 MPa). The open porosity could, however, be adjusted from 5% to 15% with varying SB granule size ranges. These variations are easily observed on the thermally etched microstructures, Fig. 2. Fine granule sizes led to fine structures and porosity, but the 45–500 μm size range yielded the desired coarse open pore structure that could be easily infiltrated. High temperature

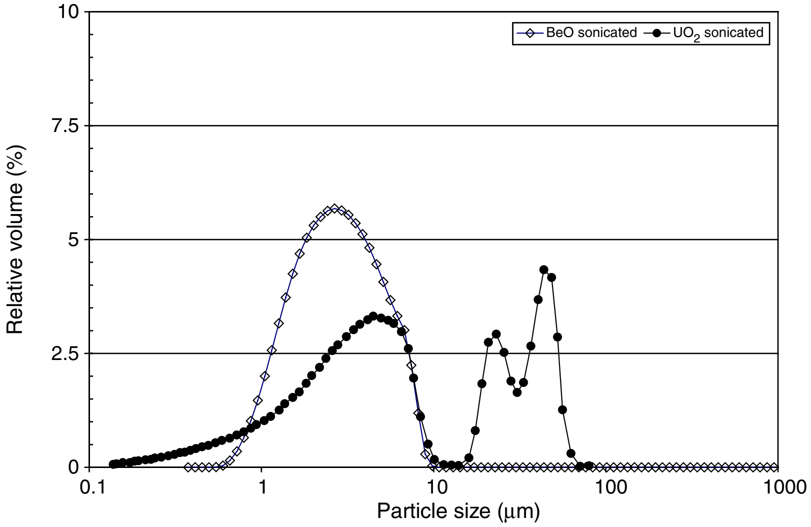

Fig. 1. Particle size distribution of the depleted UO_2 and the BeO powders measured with a laser scattering particle size analyzer with small volume module for dispersed powder with incorporated sonicator (Sonics Vibra Cell, Sonics & Materials Inc., Model VC70 T, frequency 20 kHz at a setting of 9W output power and 2 s on, 2 s off).

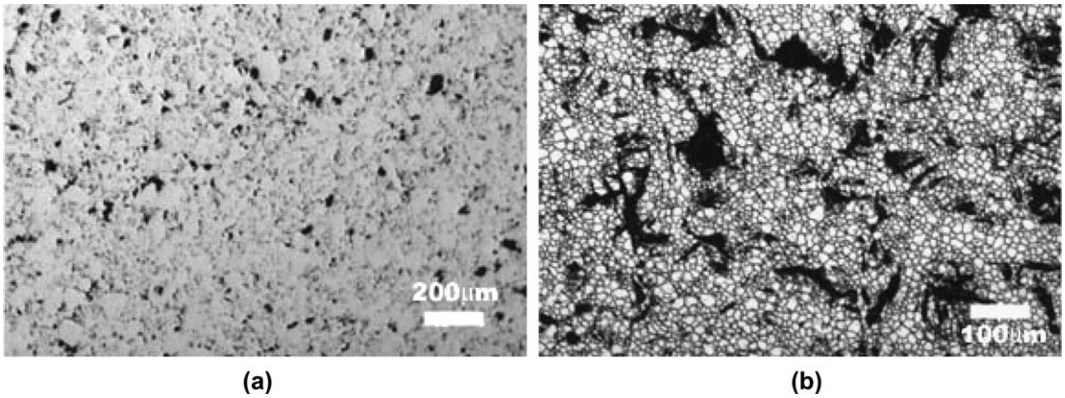

Fig. 2. (a) Polished section of a UO_2 pellet produced via the 'Slug Bisque' process, from granules <45 μm yielding 93%TD, 5%, and ~12 μm; (b) of a polished and thermally etched pellet fabricated from granules in the size range 45–500 μm yielding 90%TD, 8%, and 20 μm, and large interconnected porosity.

stability of these porous microstructures was established by resintering tests at 2023 K for time periods in excess of 6 h.

2.2. PIP processing

Once the sintered open pore structure is produced, it is progressively filled with SiC by the PIP process which involves (1) degassing the precursor and specimen, (2) vacuum/pressure impregnation, (3) pyrolysis and (4) crystallization. Because of the large volume decrease in the polymer upon pyrolysis, a number of cycles must be performed to fill the porosity to the maximum value possible.

The apparatus for performing the vacuum impregnations is shown schematically in Fig. 3. The specimens are first evacuated and the liquid precursor is degassed under vacuum while heating to 333 K while stirring to remove the most volatile species. Then the precursor is allowed to surround the specimens at 333 K to reduce its measured viscosity from about 0.08 to 0.02 Pa s. This enhances both the degassing process and the rate of impregnation. Finally, pressures up to 2.76 MPa are applied to the surrounding polymer in a pressure vessel to increase the rate of polymer penetration. Ultra high purity argon is used for the pressurization to reduce oxygen and water vapour absorption which degrades the polymer. The pressurization step becomes progressively more important as the pores become filled with SiC and their diameters become smaller. Eventually the interconnected pore channels close preventing further impregnation.

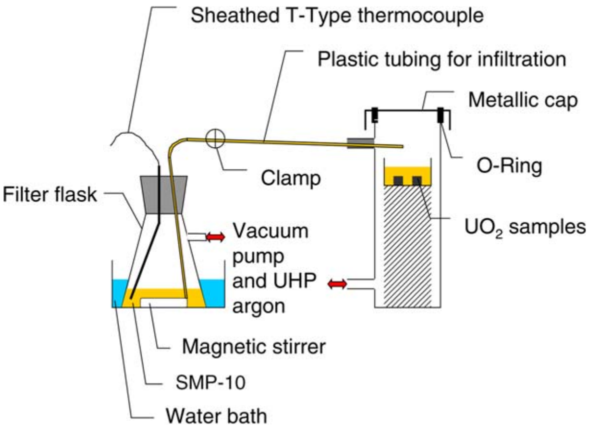

Fig. 3. Impregnation system for performing PIP. Not shown is the pressurization step in the impregnation process performed in a pressure vessel.

Following the impregnation step, pyrolysis was carried out at 1273 K for 2 h under flowing high purity argon to produce amorphous SiC. Typically, after three such impregnation-pyrolysis cycles, we crystallize at 1573 K for 15 h to avoid the UO_2–SiC reaction. As shown in the flow chart of Fig. 4, this impregnation, pyrolysis and crystallization cycle is generally repeated twice. However, a third cycle is sometime necessary if the volume to fill is larger than 10 vol.%.

An example of the microstructure of the SiC and UO_2 phases, Fig. 5, shows that there is generally good physical contact between the UO_2 and SiC phases, but some cracking in the SiC phase is observed due to the high shrinkage during the pyrolysis. The maximum density of the SiC phase was 75%TD, defined as the volume of the SiC divided by the *total* volume of the original open porosity. This is only slightly lower than the density of

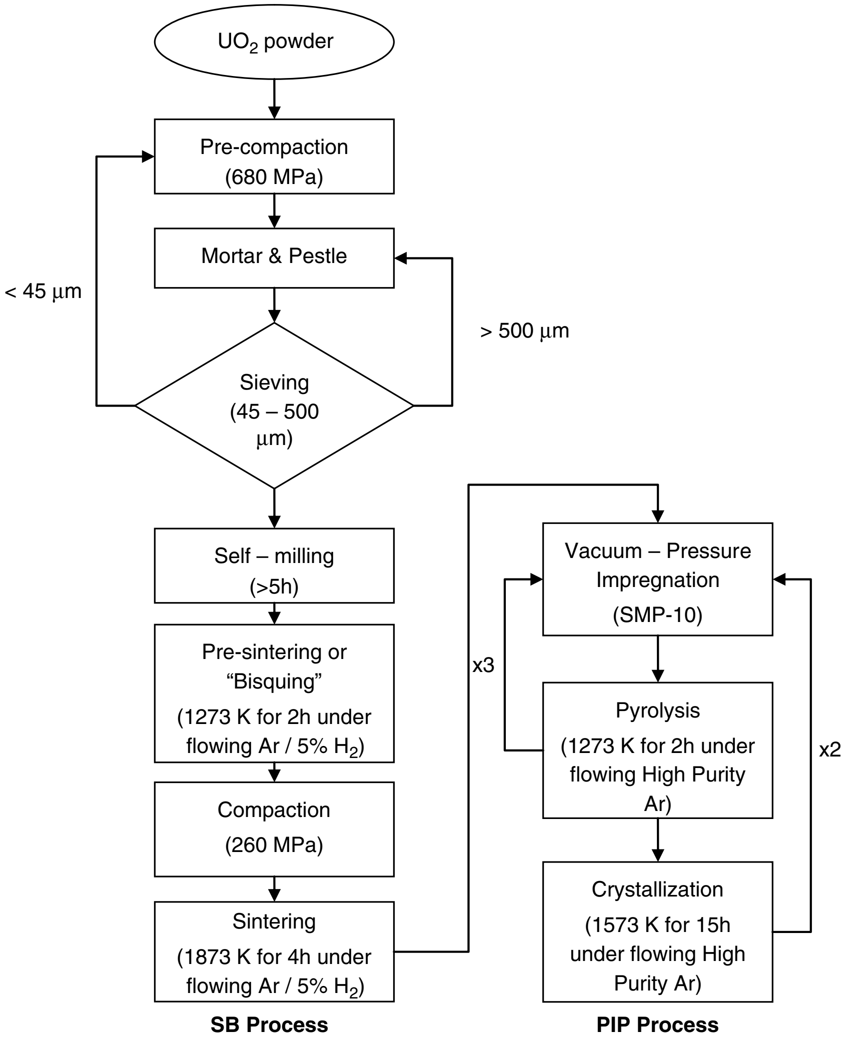

Fig. 4. PIP processing flow chart.

free-standing SiC of 80%TD, crystallized at 1573 K. Higher crystallization temperatures would no doubt increase the density still further.

The PIP/SiC process is highly adaptable to consolidating numerous chemically compatible species. (Other organic precursors exist to produce different compounds.) An example is the consolidation of a TRISO type fuel. In Fig. 6, we show an SEM image of simulant TRISO spheres (courtesy of CEA-Grenoble) that have been consolidated using the PIP process. The β-SiC outer layer on the spheres was left without the usual final carbon coating so as to be completely compatible with the β-SiC produced from PIP, and thus allows 'normal' PIP/SiC crystallization at 1873 K for 4 h. The sphere loading was 50 vol.% for these monodispersed spheres of ~0.001 m diameter that were poured into a mould. In such a case, 20 µm 'filler' particles of α-SiC are necessary to consolidate high volume fractions of SiC up to 100% SiC. These are the particles visible in Fig. 6 in the matrix phase. The spheres were made visible following sectioning with a diamond wire saw. It should be noted that the SiC diffusion barrier layer is not damaged by the 'soft' PIP process, and some spheres exposed by the diamond saw have simply fallen out of their sites. Nevertheless, mercury intrusion porosimetry showed that the consolidated body had essentially no open porosity after 6 impregnation cycles.

This application of the PIP process shows that matrix fractions as high as desired can be obtained for inert matrix fuels by mixing filler particles with

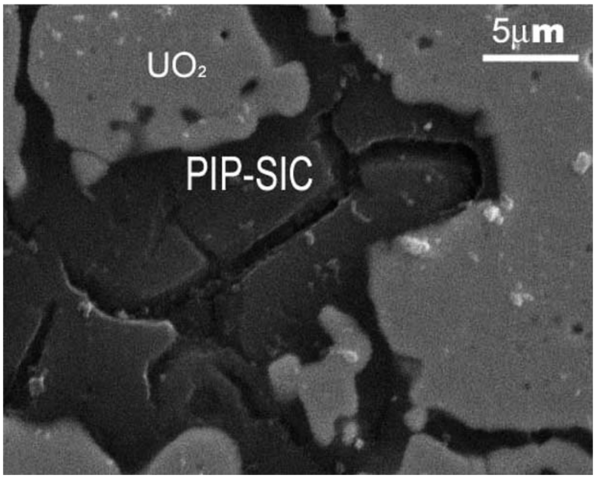

Fig. 5. Morphology of the PIP/SiC phase in UO_2. Cracking is observed in the SiC phase.

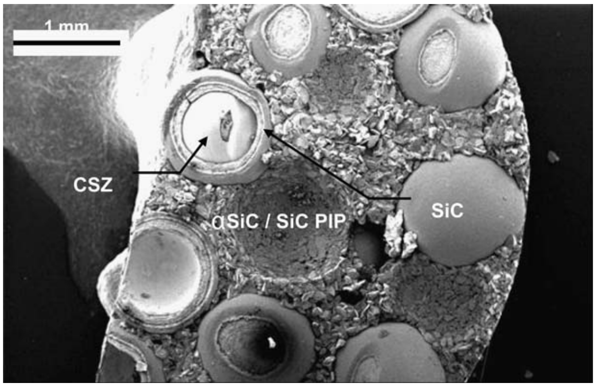

Fig. 6. PIP/SiC-consolidated simulant TRISO fuel microspheres with a SiC outer layer, and a CSZ simulant fuel kernel. The matrix phase is α-SiC surrounded by the β-SiC from the PIP process. No damage to the SiC outer layer is observed with this 'soft' consolidation process. Work performed by A.A. Solomon at CEA Cadarache, 2002, unpublished.

the Pu or TRU wastes and then consolidating the mixture with the PIP process.

2.3. The BeO/UO_2 'co-sintering' process

The second processing method that was developed to produce a continuous high conductivity phase of BeO in UO_2 was called the 'co-sintering' process.

To uniformly distribute the BeO powder amongst the UO_2 granules, either green or SB UO_2 granules, were first 'self-milled' as shown in Fig. 7(a). After 12 h, the granules became significantly more spherical which aided the uniform distribution of the BeO powder. No sieving was done after this step to remove any loose UO_2 powders. The BeO powder was then added to the UO_2 spheres and mixed for another 20 min. The optical image on the right shows that the BeO powder uniformly coated the spheres and, in fact, the coated spheres are indistinguishable from uncoated spheres. No free BeO powder was observed.

The coated granules are then pelletized at 200 MPa (to ∼55%TD) and sintered at 1973 K for 5 h under flowing Ar + 5% H_2.

Table 1 lists the densities and porosities measured by immersion in ethanol of several pellets with PIP SiC and with varying amounts of co-sintered BeO. For the two phase materials, the theoretical densities assumed for UO_2, SiC and BeO were 10.96, 3.21 and 3.01 g cc^{-1}, respectively, and the relative density of phase mixtures is obtained by dividing the measured bulk density by the volume-weighted theoretical density of each phase. The values in this table are precise to ±1%. High density is achieved with all UO_2/BeO ratios from 0% to 100% BeO, with the help of filler particles. An optical image of a polished and thermally etched section of a pellet fabricated from green UO_2 spheres is shown in Fig. 8. The light regions in the image correspond to the UO_2 phase, the darker regions are the BeO phase, and the black regions are porosity or pull-out. The BeO phase in this case contains ∼24 vol.% of UO_2 by quantitative image analysis. As can be seen in the macro view (Fig. 8(a)), the high conductivity BeO phase occupies all of the available phase space between the UO_2 granules.

The high UO_2 'contamination' of the BeO phase probably comes from the 'erosion' of the relatively soft UO_2 green granules during the mixing step. One obvious way to reduce this is to use 'harder' SB granules. Fig. 9 shows optical images of a polished and thermally etched section of a pellet made from green granules on the left and SB granules on the right. As expected, a significant reduction in the amount of UO_2 occurred for the SB granules. However, the UO_2 'contamination' has the desirable effect as a grain growth inhibitor which can affect the onset of cracking in BeO matrices due to anisotropic radiation growth [11].

2.4. Continuity of the high conductivity phase

From Ishimoto et al.'s results [8], it is highly desirable that the high conductivity phase in ECO

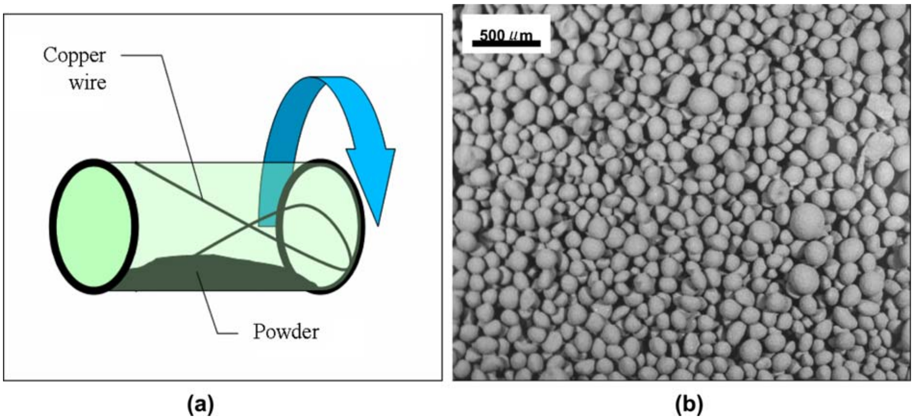

Fig. 7. (a) Schematic of the ‘self milling’ process, and (b) an optical image of UO_2 granules mixed and uniformly coated with BeO. No free BeO was observed after mixing.

Table 1
Density of pellets containing PIP/SiC and varying amounts of BeO

Description	BeO or SiC (vol.%)	Final relative density[a] (%)	Open porosity (%)
UO_2	0	98	<1
BeO	100	99	<1
UO_2–PIP/SiC[b]	13	96	2
UO_2 (green granules)–BeO	8	94	5
UO_2 (green granules)–BeO	11	98	1
UO_2 (SB granules)–BeO	11	95	4
UO_2 (green granules)–BeO	20	95	4
UO_2 (powder)–BeO	50	96	2
UO_2 (powder)–BeO	74	98	1

In the description of the specimens, ‘green’ or ‘SB’ granules refers to the starting condition of the granules.

[a] Theoretical density of the materials are calculated by considering the relative volume of each phase.

[b] Relative volume of SiC is obtained from the volume difference between initial and final open porosity (before and after PIP).

fuels be continuous for appreciable enhancement in thermal conductivity. Since the inter-granular BeO phase appeared to exhibit various levels of contamination by the dispersed UO_2 phase, the 3D continuity of the BeO phase needed confirmation. Bulk dissolution of the UO_2 phase was carried out by immersing the sintered UO_2/BeO pellets containing ~10 vol.% BeO in boiling, 50% concentrated HNO_3 for ~15 min. Fig. 10(a) shows a UO_2/BeO specimen and its corresponding BeO structure after dissolution of the UO_2. In Fig. 10(b) is shown a macroscopic picture of the BeO ‘skeleton’. Immersion density measurement indicated the ‘skeleton’ was 85% dense, which is in agreement with the density and porosity measurement of the composite bodies in Table 1. The ‘skeleton’ was mechanically robust, allowing geometric measurements and positioning for photographs. Dissolution experiments have been additionally conducted on specimens with only

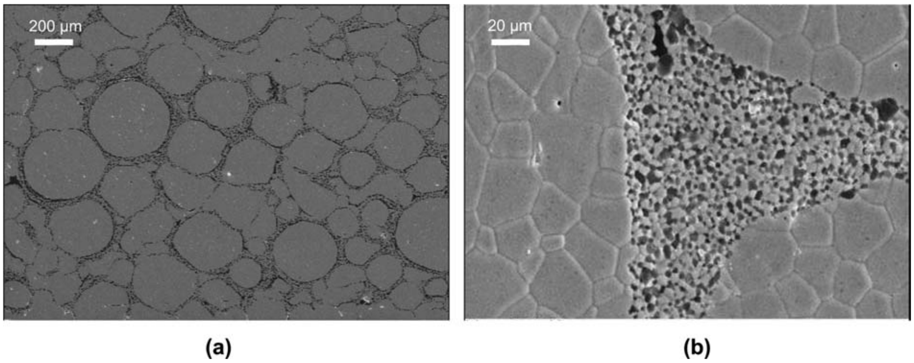

Fig. 8. (a) Polished section of co-sintered UO_2/BeO using green granules, showing that the high conductivity phase fills all of the available phase space between the UO_2 granules. (b) SEM image of a polished and thermally etched section (93% overall density) showing that the BeO phase contains UO_2 ‘contamination’ and porosity, which cannot be distinguished from the dark BeO phase.

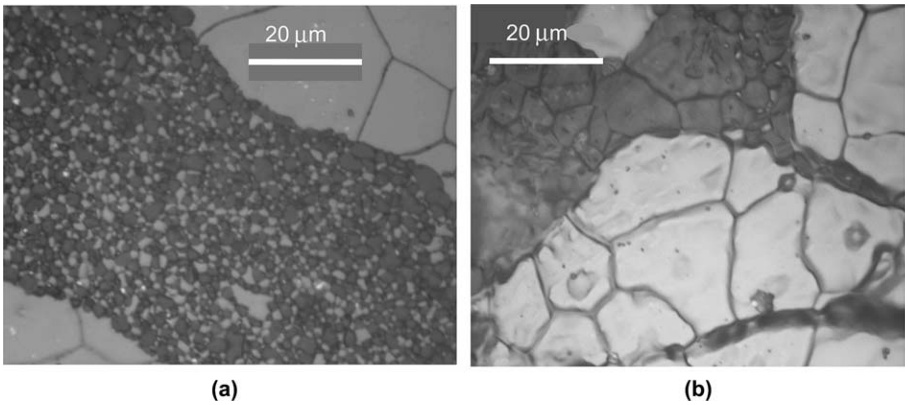

Fig. 9. (a) Polished and thermally etched section of a co-sintered UO_2/BeO pellet fabricated from green, self-milled granules, and (b), from 'self-milled' bisqued granules. Light regions are UO_2, while dark regions are BeO. Reduction in the amount of dispersed UO_2 phase within the BeO phase using bisqued granules is observed.

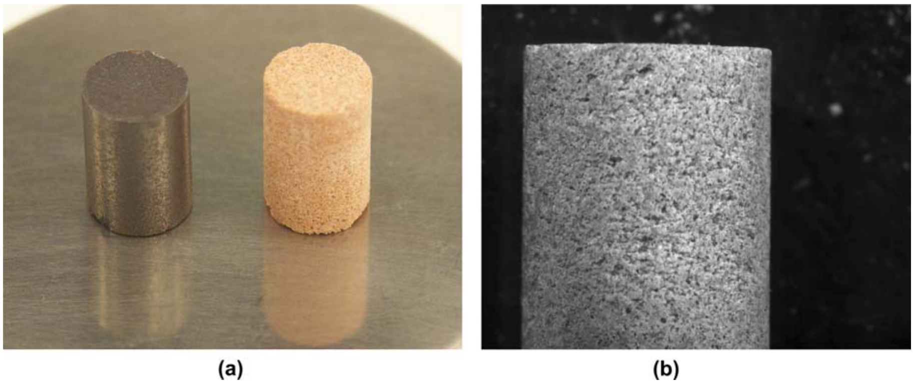

Fig. 10. (a) A co-sintered UO_2/BeO pellet of 8 mm diameter (dark), and the remaining BeO 'skeleton' (light) after dissolution of the UO_2 and (b) shows the BeO skeleton at higher magnification.

5 vol.% BeO, which still show continuity of the BeO phase. XRD on the 'skeleton' showed no detectable UO_2 remained.

3. Results and discussion

The PIP method of forming a continuous SiC phase within UO_2 has been successfully developed and employed to produce ECO fuels. The maximum density of the SiC was 75%TD based on the original open porosity, and had some conductivity limiting cracks, but good apparent contact was observed between the phases. This limiting density appears to be due to pore blockage by the SiC during impregnation as well as the low crystallization temperature used. Moreover, the low crystallization temperature also leads to 30 nm grain sizes in the SiC as determined by X-ray line broadening. The measured thermal conductivity of the UO_2/PIP–SiC samples crystallized at 1573 K showed *no* enhancement in thermal conductivity over typical reactor density UO_2, as opposed to the 50% enhancement for the UO_2/BeO samples. We have shown, by isolating the SiC matrix phase, that this is primarily due the low crystallization temperatures that were used to avoid reactions with UO_2 [9]. However, there is no reason to believe that if carbide fuels were used in inert matrix fuels, the crystallization temperatures could be much higher, and the matrix thermal conductivity would be high [9].

The PIP method was shown to be a very good method of consolidating other dispersed phases like TRISO fuel particles with compatible SiC outer layers. Therefore, this methodology could also be used as an inert matrix fuel or target for other chemically compatible particles for Pu or actinide burning or gas-cooled reactor fuels with the aid of β-SiC filler particles.

A large range of BeO contents in UO_2 were also successfully produced by the 'co-sintering' route to high density. This is likely due to the similarity of the sintering rates of the two powders under the reducing gas conditions used [13–16]. Moreover, the compatible sintering rates for *mixtures* of BeO and UO_2 phases suggest that the *interphase* boundaries have approximately the same atomic transport mobility as the pure materials. The self-milled granules led to higher densities which may be due to improved sphere packing. The self-milling may also lead to a more uniform coating with the BeO powder, as well as aiding the BeO redistribution to fill the spaces between the UO_2 granules during pressing.

Contamination of the inter-granular BeO phase by finely dispersed UO_2 was observed and reduced by using harder bisqued granules. However, the bisqued granules do not compact as readily during pressing, which leads to slightly lower density, as seen in Table 1. Our modeling calculations [12] show that it is better to have the UO_2 contamination than to have more porosity in the BeO phase to achieve high thermal conductivity. Moreover, a side benefit of the UO_2 contamination is the reduction in BeO grain size. The well-known anisotropic growth of BeO under irradiation that can cause microcracking is delayed with finer grain sizes and higher temperature irradiations [10]. The anisotropic growth and cracking of BeO is probably its major drawback for inert matrix fuels [11], but it can be delayed with finer grain sizes and higher temperatures or more thermalized neutron spectra.

For closed fuel cycles, the ease of dissolving the *major* phase UO_2 in hot nitric acid from the BeO 'skeleton' suggests that fuel recovery and recycling will be very simple in that case. For BeO matrices in inert matrix fuels, it may be possible to create a *continuous* fissile phase by reversing the roles of major and minor phases in ECO fuel fabrication. In that case, the BeO would be granulated, and the Pu or actinides would coat the granules and be sintered to form a continuous phase that can be dissolved. However, for *dispersed* fissile phases in large volume fractions of BeO or SiC, it will normally be necessary to crush and leach to recover the Pu. It may be possible to recycle the matrix phase in fuel production.

4. Summary

Two processing routes: Polymer Impregnation and Pyrolysis or PIP and 'co-sintering' were successfully developed to produce high density UO_2 specimens with a continuous minor matrix phase of SiC and BeO, respectively. The methodology is flexible so that much larger matrix fractions can be used for consolidating particulates like TRISO microspheres, and for inert matrix fuels. The PIP/SiC process involves several cycles of impregnation to achieve high density, and required relatively low processing temperatures to avoid reactions with UO_2 which impairs its thermal conductivity. Therefore, UC or PuC would be preferable as the fissile phase in SiC. Also, since there is negligible shrinkage in the process, it yields 'near net shape' pellets that would require very little if any grinding. In the BeO co-sintering process, only one mixing step followed by normal sintering is necessary, and the fuel or target is compatible with UO_2 and PuO_2 to very high temperatures. However, the issue of anisotropic radiation growth in BeO at high damage doses would need to be addressed for particular applications.

Acknowledgements

The authors would like to thank Mitchell Meyer, Rory Kennedy and Andrew Madison of the Idaho National Laboratory for their help in conducting the thermal property measurements, and the US-DOE for their financial support of this work, under the DOE – NERI program. (Project: No. 02-180 Award DE-FG07-02ID22613.)

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