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A High-Temperature, Short-Duration Method of Fabricating Surrogate Fuel Microkernels for Carbide-Based TRISO Nuclear Fuels

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Abstract

High-temperature gas-cooled reactor technology is a frontrunner among generation IV nuclear reactor designs. Among the advanced nuclear fuel forms proposed for these reactors, dispersion-type fuel consisting of microencapsulated uranium di-oxide kernels, popularly known as tri-structural isotropic (TRISO) fuel, has emerged as the fuel form of choice. Generation IV gas-cooled fast reactors offer the benefit of recycling nuclear waste with increased burn-ups in addition to producing the required power and hydrogen. Uranium carbide has shown great potential to replace uranium di-oxide for use in these fast spectrum reactors. Uranium carbide microkernels for fast reactor TRISO fuel have traditionally been fabricated by long-duration carbothermic reduction and sintering of precursor uranium dioxide microkernels produced using sol-gel techniques. These long-duration conversion processes are often plagued by issues such as final product purity and process parameters that are detrimental to minor actinide retention. In this context a relatively simple, high-temperature but relatively quick-rotating electrode arc melting method to fabricate microkernels directly from a feedstock electrode was investigated. The process was demonstrated using surrogate tungsten carbide on account of its easy availability, accessibility and the similarity of its melting point relative to uranium carbide and uranium di-oxide.

Keywords: Ceramic nuclear fuel, HTGR, fabrication, actinides, fast reactor

I. Introduction

Leading energy agencies around the world are proposing high-temperature gas-cooled reactors (HTGR) to satisfy their growing energy needs [1-7]. In the US, the Department of Energy has selected a wide variety of designs including the HTGR for development and deployment under the Generation IV nuclear reactor technology portfolio. Belonging to the same genera of HTGRs is the Gas-Cooled Fast Reactor (GFR) [1-4]. The GFR is proposed to address two crucial issues that are currently considered as major impediments to the full-fledged establishment of nuclear power plants around the globe [3-6]. First, the GFR is expected to aid in the production of hydrogen, which is promoted as the most important energy carrier of the future [3-7]. Second, GFRs can achieve very high burn-up, aiding in the recycling of minor actinide wastes from existing and future spent fuel stockpiles and thus reducing the radio-toxic loading on both temporary and permanent waste repositories. Since the GFR is designed to operate with a fast neutron spectrum, it is expected to achieve high burn-up by facilitating fission of both fertile (minor actinides) and fissile ($U_{235, 239}$, $Pu_{239, 241}$) materials [3,4,7-9]. However, the main challenge associated with high-temperature reactors in general, and the GFR in particular, is the qualification of fuel and structural materials that can withstand the proposed operating temperatures and fast

neutron fluence [3,4,7-9]. The GFR core temperatures are expected to reach 1400 °C during normal operation and up to 1600 °C during transients [6-8]. With such high temperatures, the use of metals and alloys for fuel and structural materials becomes impractical. Traditional nuclear alloys such as zircaloy undergo extensive temperature- and irradiation-induced creep deformation, which renders them useless [1-7]. Ceramics such as SiC and ZrC, on the other hand, have shown good promise at these operating temperatures on account of their high melting points, resistance to thermal shock and low activation while effectively containing radio toxic fission products [2-7].

Although urania (UO_2) has been the fuel composition of choice for reactors operating in the thermal spectrum, uranium carbide (UC) fuel is preferred for gas-cooled reactors, especially those operating in the fast spectrum, where a single atom of carbon is preferred to two atoms of oxygen [3-5,7,10]. This is thought to be beneficial in the context of reduced neutron moderation with one moderating carbon atom in UC as compared to two oxygen atoms in UO_2. A second advantage of UC fuel over UO_2 is the absence of oxygen released during the fission process, thus preventing the oxidative corrosion of the fission product barrier and the formation of carbon monoxide and carbon dioxide gases owing to the interaction of the released oxygen with the pyro-carbon layers. These gases accumulated over a period of time can potentially cause internal stress on the coatings. Another potential advantage of having

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UC would be the formation of stable carbide-based compounds of fission products. These carbides are thought to be much more thermally stable compared to oxides at fuel operating temperatures [11, 12].
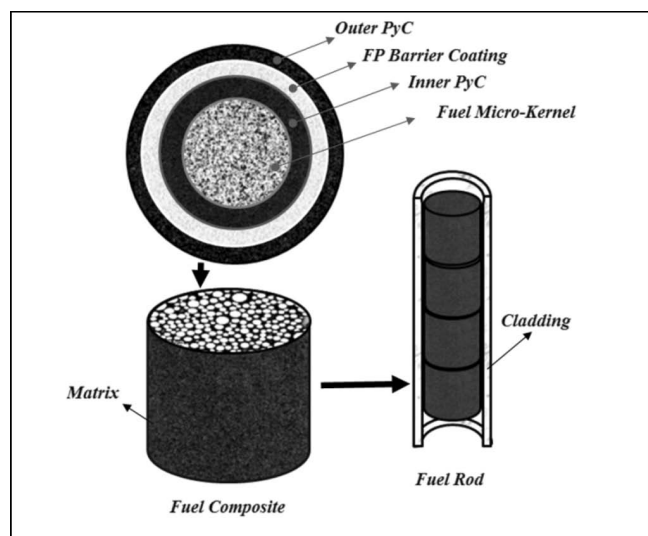

Fig. 1: Advanced nuclear fuel structure.

The GFR fuel pellet is expected to resemble the 'blueberry muffin' structure (Fig. 1) where encapsulated UC/UO_2 microkernels (200 – 900 μm diameter) are dispersed in an inert material matrix [7 – 10]. These encapsulated fuel kernels are popularly referred to as the Tri-structural Isotropic (TRISO) fuel particles on account of isotropic nature of the fuel geometry and the thickness of the encapsulating layers [2 – 12]. The TRISO fuel particle consists of a central UC/UO_2 microkernel followed by three distinct encapsulating layers as shown in Fig. 1. The first layer consists of an inner pyro-carbon (PyC) buffer layer to allow for fuel swelling during operation while simultaneously functioning as a plenum for the collection of fission gases. This layer is surrounded by the fission product barrier layer, which is fabricated out of either SiC or ZrC [7 – 10]. An outer protective layer of medium density PyC then surrounds the fission product barrier layer, acting as a protective packing layer. These TRISO fuel particles are then dispersed in a matrix such as graphite to resemble a cylindrical pellet (~ 1.27 cm diameter and ~ 3.81 cm height).

Traditionally UC microkernels have been fabricated on a laboratory scale by subjecting precursor UO_2 microkernels to high-temperature, long-duration carbothermic reduction, where the UO_2 is converted into UC in a substitution reaction [9 – 15]. The precursor UO_2 microkernels are produced with the sol-gel process [9 – 12]. The sol-gel process, which itself involves the use of organic and inorganic precursors, often generates mixed radioactive wastes which potentially turn into high-level mixed wastes in the context of minor-actinide-bearing fuels, thus making large-scale production of fuel using this method less attractive [11 – 15]. Additionally, the carbothermic process is inefficient owing to the formation of highly stable intermediate oxy-carbides which generally pose a formidable challenge to the final product purity [11]. Another crucial disadvantage of the sol-gel fabrication route is the time-consuming, high-temperature pyro-metallurgical pro-

cesses involved in the conversion of UO_2 to UC and negative effects of the process parameters (time and temperature) on minor actinide losses [10, 11, 16 – 21]. It is anticipated that such long-duration carbo-thermic reduction processes applied to minor-actinide-bearing nuclear fuel may potentially lead to relatively high losses in minor actinides such as americium [16 – 21]. Additionally, the radioactive wastes generated by the sol-gel process may pose handling and disposal issues. These disadvantages called for the development of a new process that could potentially be extended to minor-actinide-bearing advanced UC microkernels suitable for GFR TRISO fuel.

As an alternative to the currently used sol-gel method followed by the long-duration carbo-thermal reduction process for the fabrication of UC fuel kernels, a unique and quick process involving arc melting a rotating feedstock (electrode) was evaluated to produce surrogate microkernels. The current method drew heavily on the experiences of fabricating micro- and nano-sized ceramic powders using the electro discharge machining method [22 – 29]. For the evaluation of the method, WC was chosen as a surrogate to UC on account of its economical availability, nil-contamination risks, and the similarities of its melting point with uranium carbide (UC: 2623 K; WC: 3143 K [30]).

II. Experimental

The experimental set-up consists of a custom-designed rotating electrode stand as shown in Fig. 2. The design of this particular stand was based on the principle that a melt piece dislodged from a feedstock electrode rotating at high speeds would condense into spherical microkernels owing to the centrifugal force acting on the melt combined with the forces of surface tension. The electric arc was generated using a non-consumable 1.7145-cm diameter tungsten electrode to which current was supplied from a high-current DC source. The bottom tungsten arcing electrode assembly was designed to closely resemble a tungsten-inert gas (TIG) welding electrode. Microkernel fabrication runs were performed using 1.27-cm-diameter, 5.08-cm-long cobalt-stabilized WC blanks (~96 % dense) as feedstock electrodes. Ultrahigh-purity argon gas was constantly purged into the tank to expel all the atmospheric air, thus reducing chances of abnormal breakdowns and oxygen contamination. Argon also acted as a dielectric medium and coolant, preventing the arc-generating tungsten electrode from experiencing localized melting.

Experimental runs to produce microkernels were conducted by varying the rotational speed of the electrode to evaluate the effect of rotational speed on the size distribution of the microkernels [22 – 28]. Three values of rotational speed were used: 500 rpm, 1000 rpm and 1500 rpm for the experimental runs. The temperature of the electrode was expected to reach values greater than 3200 K at full power (similar to MIG welding temperatures). The gap voltage was fixed at 31.2 V for the experimental runs. This gap voltage was found to supply an arc current of 130 A, which was sufficient to produce the range of kernel sizes required for the proposed application. The duration of the runs was set to 10 minutes. At the end of each run, the produced microkernels were sieved using ASTM sieves. Microkernels were separated into four size groups:

$150-355\ \mu m$, $355-600\ \mu m$, $600-850\ \mu m$, $850-1000\ \mu m$. Microkernels with diameters smaller than 150 μm and larger than 1000 μm were rejected. Microkernels in each group were collectively weighed and the results were expressed as weight percentages with respect to the total weight of all microkernels collected. Microstructures of representative microkernels were analyzed with a JOEL electron microscope after rough metallographic preparation. Electron energy dispersive spectroscopy (EDS) was used to qualitatively analyze the elemental compositions of representative specimens. Small-sized random microkernels were selected and subject to x-ray diffraction analysis using a PANalytical X'Pert x-ray diffractometer to ascertain the different phases that were present in the final product.
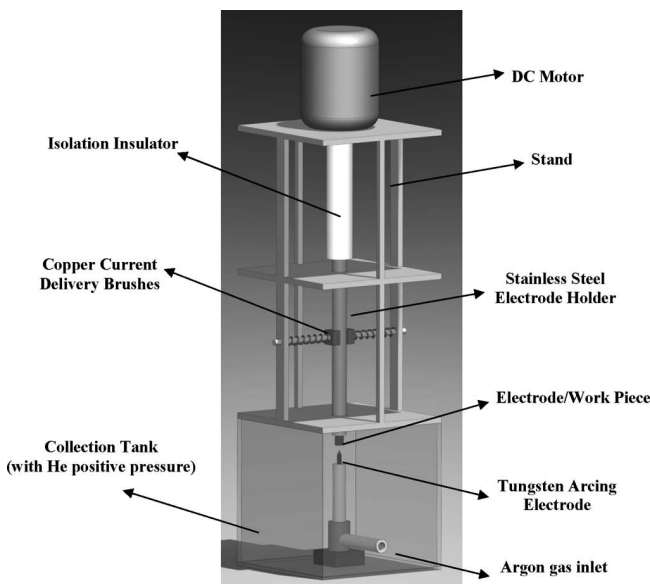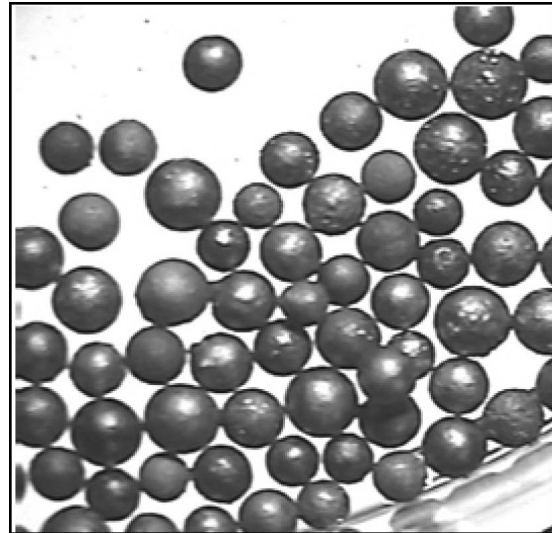

Fig. 3: Optical micrograph of tungsten carbide microkernels produced by rotating electrode mechanism at 1500 rpm.

The surfaces of the microkernels, as observed from the electron micrographs, were relatively smooth with some patches of roughness and the same was confirmed with a scanning electron microscope (Fig. 4). It is hypothesized here that this roughness in actual nuclear fuel kernels will perhaps be beneficial by providing a higher degree of adhesion of the IPyC layer resulting in stronger interlayer stitching. An EDS spectrum of the spheres are shown in Fig. 5. However careful observation of the spectrum revealed an anomalous phenomenon. No cobalt peaks were observed, considering the fact that it is traditionally used as an additive to produce high-density tungsten carbide blanks, like the ones used in this work.

Fig. 2: Rotating electrode assembly.

III. Results and Discussion

The designed assembly was successful in producing significant quantities (tens of grams) of spherical kernels of tungsten carbide in a very short duration of time. Fig. 3 shows an optical microscope image of the microkernels obtained during a typical run. Initial direct visual observations using an optical microscope revealed microkernels with good qualitative sphericity and the same was confirmed later by means of electron microscopy. Sphericity of the microkernels becomes important in the context of applying barrier coatings, as significant deviations can result in highly localized stresses on the coatings during operation, especially with the TRISO fuel microkernel known to exhibit the 'amoeba effect'[31]. The sphericity of the 25 random representative microkernels produced from each run was estimated by measuring the aspect ratios (the quotient of the shortest maximum chord and the maximum feret diameter) using a calibrated optical microscope and ImageJ image analysis software[32]. The highest and lowest aspect ratios measured were associated with microkernels produced at speeds of 500, 1000 and 1500 rpm and were: 1.0 and 0.9, 0.98 and 0.78 and 1.0 and 0.79 respectively, indicating no significant dependence of sphericity on the rotating speed.
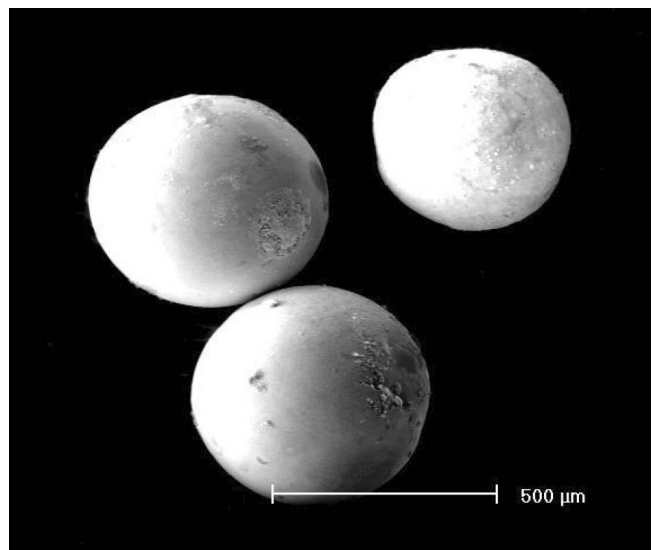

Fig. 4: Scanning electron micrographs of representative microkernels produced at 500 rpm.

The density of representative specimens was measured using a density measurement kit associated with a microbalance using the simple liquid displacement method. The measured density was $\sim 15.1\ g/cm^3$ with a standard deviation of ± 0.52. This value is well within the range of the cemented WC blank ($\sim 14.9\ g/cm^3 \pm 0.33$) that was

used to fabricate the particles, indicating that the particles did not have much porosity.
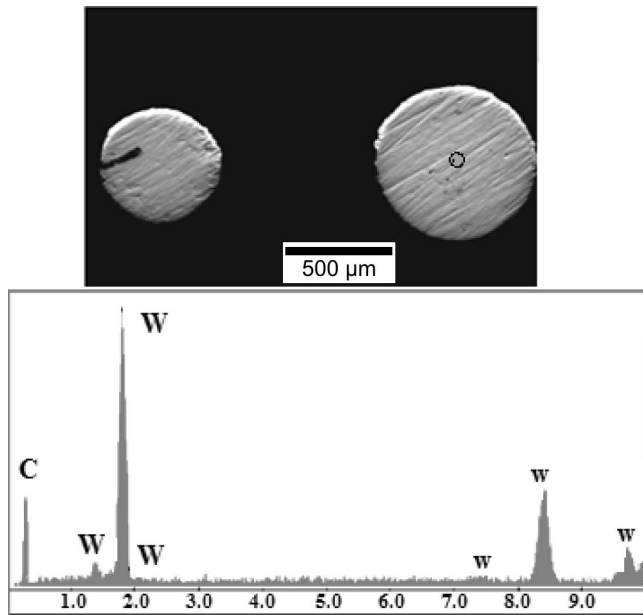

Fig. 5: EDS spectrum and the approximate location where the spectrum was sampled on microkernels produced at 1000 rpm.

Representative specimens were roughly polished to observe the cross-section and ensure that the kernels were completely solid. It is noted here that, as described in the literature [23–26], similar electro-discharge process adopted in the past have led to the formation of hollow particles. However, no such phenomenon was observed in the current work.

Electron Dispersive Spectroscopy (EDS) was performed to ensure that no oxidative reactions had occurred, resulting in the formation of oxides of tungsten and to confirm the elemental compositions of the final product. The purging of high-purity argon gas had largely inhibited any noticeable oxidation of the spheres. To test the veracity of the EDS spectrum and to confirm the absence of significant quantities of cobalt from the final product spheres, the samples were subject to x-ray powder diffraction. Contrary to the EDS spectrum, the resulting diffraction pattern showed the presence of some cobalt in the samples, confirming the assumption of localized evaporation (Fig. 6). A possible explanation for this anomaly was the fact that significant quantities of cobalt had evaporated owing to the high temperatures that the melt reached during the runs. It is noted here that the melting and boiling points of cobalt are approximately 1495 and 2870 °C respectively [30], and the process as discussed in the previous section was expected to reach temperatures in excess of 3000 °C. Another possible explanation was the fact that the EDS spectra obtained, as expected, were highly localized. Nevertheless, the observation of some cobalt peaks was encouraging as it is indicative of the ability of the process to retain certain low-melting-point compounds and metals which could prove beneficial if the process were to be extended to fabricate minor-actinide-bearing nuclear fuel. A detailed compositional analysis using x-ray mapping and followed by electron microprobe analysis to charac-

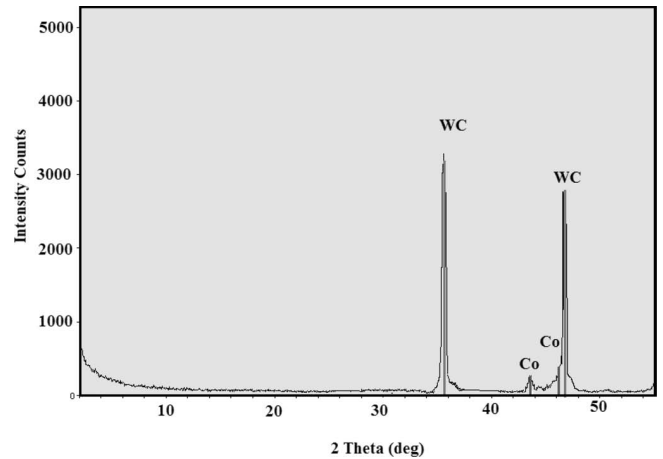terize the potential loss in cobalt is currently underway at the high-temperature materials laboratory.

Fig. 6: X-ray diffraction peaks indicating chemical composition of powders sampled from a mixture of microkernels produced at 500, 1000 and 1500 rpm.

The particles collected from each of the runs were sieved and separated into size groups based on their diameters. Their collective weights were expressed as weight percentages in each of the four chosen size (diameter) intervals. The plot of the size distribution (weight % versus median diameter of the group) with changing rotational speed is shown in Fig. 7. Trends indicated that the size distribution evened out with increasing rotational speeds.
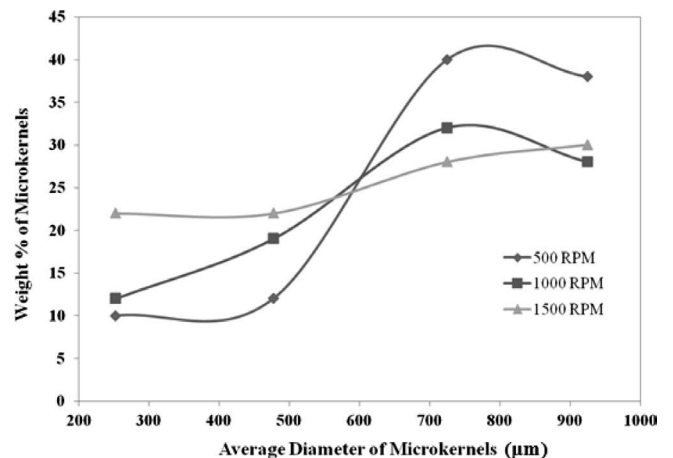

Fig. 7: Size distribution of micro-kernels with changing rotational speeds.

The wide range in the resultant microkernel sizes from this method is in stark contrast to the traditionally used sol-gel method which allows fine control of the particle size. From the perspective of increasing the burn-up (MWd/MTHM) of a reactor while optimizing the core size, it is necessary to achieve a reasonably high fuel density (kilograms of heavy metal/cm^3) [33]. This issue becomes crucial for dispersed composite fuels that are a mixture of two distinct phases: the inert matrix and the TRISO-coated fuel particle. It is a well-known fact that in composite fabrication a wider size distribution of microkernels results in a higher packing fraction (number of fuel microkernels per unit volume of the composite) [34]. Hence an optimal packing fraction is required to make the fuel more efficient in terms of increased burn-up. However, currently

no standard optimal packing fraction of advanced fuel is prescribed. Some studies in the recent past have suggested that a size of 600 μm may be an optimal particle fuel diameter for a GFR inert matrix fuel for both fabrication and irradiation performance [35]. On the other hand studies especially evaluating spherepac fuel, similar in concept and design, but different in geometry, suggest the explicit use of at least two different microkernel sizes for achieving optimal fuel loading conditions [36]. However, it is suggested that the particle fuel diameters in the range of 400 – 600 μm may be more suitable in the context of the mechanical integrity of the coated UO_2 particles during in-core operation [37]. However, no such benchmarking studies are currently available for UC-based fuels. In case the same applies to UC-based nuclear fuels, data from the current experiments (Fig. 7) suggest that the application of lower speeds (~500 rpm) where the yield of particles with diameters greater than 400 μm increases (size distribution narrows towards the larger diameters) will be perhaps more suitable.

In the meantime, in view of a lack of strong experimentally fortified consensus in favor of using a uni-sized or multi-sized fuel microkernels for GFR composite fuels, it can be argued that at present, employing a multi-size microkernel design seems to be potentially beneficial in terms of achieving a higher fuel loading density (kilograms of heavy metal /cm^3). Nevertheless a comprehensive investigation of the performance of fuel composites using both uni-sized and multi-sized microkernels needs to be conducted to establish the feasibility and reliability of either design.

In line with the above argument, in a scenario in which a multi-size microkernel-based composite fuel design is chosen in the future, the proposed method has a potential advantage over the sol-gel method on account of its ability to produce microkernels that are diverse in terms of diameters. A second potential advantage of this method is in the simplicity of microkernel fabrication compared to the multi-step, long-duration sol-gel processes. The method is also clearly capable of producing significant quantities of microkernels in a short period of time without requiring laborious drying, sintering, calcining or carbo-thermic reduction (in case of UC microkernel production) steps. A third foreseen advantage of this method is the potential reduction in hazards associated with the generation, handling and disposal of mixed wastes which are at present a part and parcel of the sol-gel routes. For example, the proposed method as applicable to the fabrication of UC microkernels could use sintered UC feedstock electrodes which can be produced by simple and quick methods such as induction hot pressing of uranium hydride and graphite powders facilitating the formation of UC by self-heating synthesis. Experiments for the fabrication of such UC feedstock electrodes are currently underway at the High-Temperature Materials Laboratory. A fourth potential advantage of this method lies with its ability to reuse/recycle the microkernels that do not find use in composite fuel fabrication on account of their sizes. All such unused microkernels can potentially be re-fabricated into new feedstock electrodes by means of well-established methods such as

pulverization and homogenization in a ball mill, followed by induction hot pressing and sintering.

IV. Conclusions

A unique arc melting, rotating-electrode device was built and tested to fabricate spherical tungsten carbide microkernels with diameters ranging from 150 – 1000 μm. Tungsten carbide was used as a surrogate for UC, which is the TRISO fuel of choice for advanced gas-cooled fast spectrum nuclear reactors owing to similarities in melting point temperatures. The designed assembly was successful in producing spherical microkernels with a solid core. The quality of microkernels was found to be satisfactory on visual inspection. The size distribution of the microkernels was evaluated against the speed of the rotating electrode. It was found that the distribution evened out as the rotating speed increased. The process was successful in producing microkernels with a wide range of diameters, a feature that may potentially prove beneficial to achieving optimal fuel loading densities to increase the intended fuel performance in case future composite fuel designs tend to lean towards a multi-sized microkernel based design, purely from the perspective of achieving higher burn-ups. The potential advantages of this method over the currently used sol-gel methods are associated with the duration of the fabrication process, the quantity of fuel microkernels that can be fabricated and its simplicity without involving liquid precursors and the generation of mixed wastes. However, the process lacks the ability to produce microkernels only of any one specific size as opposed to the sol-gel method. The process did not seem to alter the microkernel chemical composition, although further detailed investigations are currently underway to ascertain the chemical compositions of the final product. The process reached temperatures which facilitated melting of tungsten carbide (> 3100 K) and hence could potentially be extended to the fabrication of UO_2 microkernels (melting point ~ 3138 K) for use in TRISO fuel for thermal spectrum reactors, although potential fabrication process issues need to be addressed in detail as urania being an electrical insulator will pose a challenge with the generation of an electric arc. Considering all the results, the method showed promising potential to be adopted for the production of actual uranium carbide and uranium di-oxide fuel microkernels with perhaps future extensions to minor-actinide-bearing TRISO fuels albeit it is expected that the method will require suitable design and process modifications.

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