Processing and fabrication of mixed uranium/refractory metal carbide fuels with liquid-phase sintering

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Abstract

Optimization of powder processing techniques were sought for the fabrication of single-phase, solid-solution mixed uranium/refractory metal carbide nuclear fuels – namely (U, Zr, Nb)C. These advanced, ultra-high temperature nuclear fuels have great potential for improved performance over graphite matrix, dispersed fuels tested in the Rover/NERVA program of the 1960s and early 1970s. Hypostoichiometric fuel samples with carbon-to-metal ratios of 0.98, uranium metal mole fractions of 5% and 10%, and porosities less than 5% were fabricated. These qualities should provide for the longest life and highest performance capability for these fuels. Study and optimization of processing methods were necessary to provide the quality assurance of samples for meaningful testing and assessment of performance for nuclear thermal propulsion applications. The processing parameters and benefits of enhanced sintering by uranium carbide liquid-phase sintering were established for the rapid and effective consolidation and formation of a solid-solution mixed carbide nuclear fuel.
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1. Introduction

The design of a nuclear thermal rocket (NTR) presents a great challenge due to the chemically reactive nature of the proposed propellant, hydrogen, at very high temperatures of approximately 3000 K, high flow rates of more than 25 kg/s for a 223 kN (50 klbf.) thrust engine, and high power density around 5 GW/m^3 [1,2]. Efficient use of propellant is necessary to minimize the initial mass in low earth orbit (IMLEO) and associated launch costs since NTR assembly with propellant and other mission modules must occur in low earth orbit (LEO). One measure of efficiency for any rocket is the specific impulse (I_{sp}), or thrust divided by propellant mass flow rate, which is proportional to the square root of the propellant temperature divided by the molecular mass of the propellant with hydrogen being the lowest [3]. Accordingly, high temperatures are needed to minimize the propellant and IMLEO and hence cost of the mission. Notwithstanding the need for efficient use of propellant, high thrust is needed to accelerate quickly to reduce the required trip time and associated risks. To obtain this high thrust requires high propellant flow rates and a high heat flux. Therefore, due to the extreme conditions, the selection and qualification of fuel is central to the design of a NTR. For this reason, mixed uranium refractory metal carbides such as (U, Zr, Nb)C have been sought as a high performance space nuclear fuel.

2. Background

Advanced solid-solution tri-carbide fuels such as (U, Zr, Nb)C have been proposed for nuclear thermal propulsion (NTP) applications because of their expected

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longer life and higher operating temperature due to their high melting temperature, high thermal conductivity, and improved resistance to hot hydrogen corrosion [1,4–6]. Solid solution, mixed carbide and other advanced nuclear fuels have been studied in the US during the Rover/NERVA program and in the former Soviet Union for NTP applications [7–9]. Various fuel forms and microstructures were tested in the Rover/NERVA program including dispersed fuels with UO_2 or UC_2 particles in graphite and a composite solid-solution (U, Zr)C and graphite with both fuels protected by a NbC coating. The evolution toward an uncoated all carbide, solid-solution (U, Zr)C fuel was motivated by unacceptable mass losses from earlier designs due to the high reactivity of free carbon with the flowing hot hydrogen propellant and the missmatch in coefficient of thermal expansion between the graphite matrix and NbC or ZrC coatings.

At the time the US program was cancelled in 1973, insufficient test data was available for the solid-solution binary carbide fuels to judge their performance, but they were expected to operate for several hours at temperatures as high as 3100 K with acceptable mass losses [7]. Thermochemical analysis of the binary carbide systems (U, Zr)C and (U, Nb)C by Butt et al. [10] suggested that an optimized composition of the tri-carbide, (U, Zr, Nb)C, might exhibit the longest operating lifetime. An early study of this UC–ZrC–NbC system by Tosdale [11] also suggests possible improved performance from this system. In the mid-1990s, a joint study was performed by the Russian, Scientific – Industrial Association, LUTCH and the University of Florida's, Innovative Nuclear Space Power and Propulsion Institute (INSPI) to gain prior Soviet Era experience with tri-carbides of (U, Zr, Nb)C and (U, Zr, Ta)C [12]. This study included mass loss performance of these tri-carbides exposed to hot hydrogen at a 1 mg/s flow rate. However, these results were later reexamined in light of the fact that some samples contained higher amounts of porosity than desired and may not have been a solid solution of the mixed carbides.

This study sought to investigate and optimize processing and fabrication techniques for net-shape processing of the solid-solution mixed carbide fuel, (U, Zr, Nb)C. In particular, processing and fabrication techniques were sought to provide samples with low porosity or greater than 95% theoretical density (TD) and a single-phase, solid solution of the mixed carbides. These criteria and associated fabrication techniques are necessary to ensure the quality of samples to permit meaningful testing and evaluation of their performance under NTP conditions [6].

3. Mixed carbide properties

The melting temperatures of several refractory monocarbides are listed in Table 1. Solid-solution mixed

Table 1
Temperature and carbon-to-metal ratio (C/M) of the congruent melting point and lattice parameter for some refractory monocarbides important to this study

Binary alloy	Melting point (C/M ratio) [13]	Lattice parameter (nm) [14]
NbC	3873 $\pm$ 25 K (0.79)	0.4469
TaC	4258 K (0.89)	0.4454
UC	2803 K (1.00)	0.49605 [8]
ZrC	3813 K (0.87)	0.4697

uranium/refractory metal carbide fuels exhibit high melting temperatures greater than 3700 K for uranium metal mole fractions of 10% or less. Higher uranium fractions lower the melting temperature and lead to greater uranium mass loss from either interactions with the flowing hot hydrogen propellant or vaporization from the fuel element surface especially near the bottom of the core where fuel surface and hydrogen exit temperatures might be expected to exceed 2800 K. The high solid-phase solubility of uranium carbide with the refractory metal carbides allows for a large degree of flexibility in fuel design. Therefore, to meet the requirements for space nuclear power applications such as compactness, high performance, and long life, high enrichments and low uranium metal mole fractions are preferred. Samples processed in this study contained 5% or 10% metal mole fraction of uranium.

One advantage of mixed carbide fuels over the more widely utilized UO_2 nuclear fuel is their high thermal conductivity approaching that of the metal (see Table 2). This leads to lower centerline fuel temperatures and increased safety margins. Alternatively, larger fuel elements with increased mechanical strength and lower manufacturing costs can be utilized at a greater heat flux.

Fuel microstructure has been shown to have a large impact on fuel performance both melting temperature and resistance to hot hydrogen corrosion. Studies of stoichiometric (U, Zr)C by Czechowicz et al. [15] revealed the development of a second phase, carbon, in equilibrium with the solid-solution (U, Zr)C_x. The melting temperature of these eutectic compositions, (U, Zr)C_x + C, was noted to be 100–700 K lower (depending on the uranium content) than the supposed single-phase, solid-solution (U, Zr)C from Tosdale [11]. Furthermore,

Table 2
Comparison of thermal conductivity of mixed carbide fuels with other nuclear fuels [8]

Fuel	Thermal conductivity (W/mK)
UO_2	3.5
UC	23
UN	25
$(U_{0.2}, Zr_{0.8})C_{0.99}$	30

the development of a free carbon phase is undesirable since it leads to greater mass loss due to the high reactivity of free carbon with flowing hot hydrogen. Except for UC, the refractory mono-carbides listed in Table 1 all exhibit a single phase mono-carbide region with an upper limit on C/M approaching one with congruent melting occurring at C/M significantly less than one [16]. Accordingly, during the Rover/NERVA program, to avoid eutectic formation at higher carbon content, the C/M was kept between 0.88 and 0.95 to maintain a single-phase, solid-solution carbide of (U, Zr)C [7]. A target C/M of 0.92 was established for samples processed in this study to ensure that samples have a single-phase microstructure for testing.

Uranium mass loss is an issue even in solid-solution carbides where uranium will be lost from the outer surface, which has the effect of raising the melting temperature of the surface layers of the fuel [10]. However, a low uranium diffusion rate in the solid-solution carbide would result in a steep gradient in the uranium concentration profile that is limited to about the outer 40 μm [17]. Subsequent losses of uranium are predicted to be smaller and only in relative proportion to zirconium losses. Storms [17] concluded that loss of uranium due to vaporization would be the life-limiting phenomenon for solid-solution carbide NTP systems. Porosity in the carbide fuel either from fabrication or created by irradiation, serves to increase uranium diffusion to the surface leading to higher mass loss rates. Also, open porosity further increases uranium mass losses by increasing the surface area exposed to the propellant gas stream. Because of the deleterious effects of porosity, a minimum density of 95% TD was set for samples processed in this study.

4. Methods

This study was divided into two parts in which the first phase examined a stoichiometric composition, while the second phase examined several hypostoichiometric

compositions with a C/M of 0.92 (see Table 3). To calculate the TD of the mixed carbides in Table 3, Vegard's Law, which assumes a linear relationship in lattice parameter with composition, was invoked to estimate the lattice parameter of the mixed carbides by weighting the lattice parameter of the individual carbides by their corresponding proportions.

The Phase I composition was processed from powders of the constituent carbides, namely – UC/UC_2, $ZrC_{0.94}$ and $NbC_{0.92}$ – to produce the mixed carbide, (U, Zr, Nb)C. These powders were mixed for 24 h in a ball mill in the desired proportions with a binder, 3 wt% stearic acid. The powders were uniaxially cold pressed in stainless steel dies at pressures of 120–140 MPa to form the green compact. This compact was transferred to a graphite susceptor for sintering in a 450 kHz, 20 kW induction furnace (see Fig. 1). A graphite punch attached to a linear motion feedthrough contacted the compact and made it possible to monitor compaction with a dial indicator and also to apply a small amount of pressure (approximately 3 MPa) to the compact during sintering (see Fig. 1). For Phase I (stoichiometric) samples, the coil and susceptor configuration allowed for a peak temperature near 2700 K for only several minutes before longer sintering times of up to 2 h at temperatures between 2500 and 2600 K.

Measurement of sintering temperature was done by using a Maxline™ temperature acquisition and control system. The control unit was a Maxline™ model MX-MR04 with infrared thermometers, which span a temperature range from 977 to 3866 K. These so-called 'two color' sensors operate by measuring the ratio of energy emitted by the target at two infrared wavelengths of 0.7 and 1.07 μm. Following sintering, samples were polished through 15 μm and examined by SEM including imaging for compositional contrast using back scattered electrons. Also, small pieces of the processed samples were ground and mounted on slides for analysis by X-ray diffraction (XRD). Wafers of each sample weighing about 0.25 g were cut using a slow speed diamond-wafering saw. These wafers were weighed and then

Table 3
Compositions of the tri-carbides studied

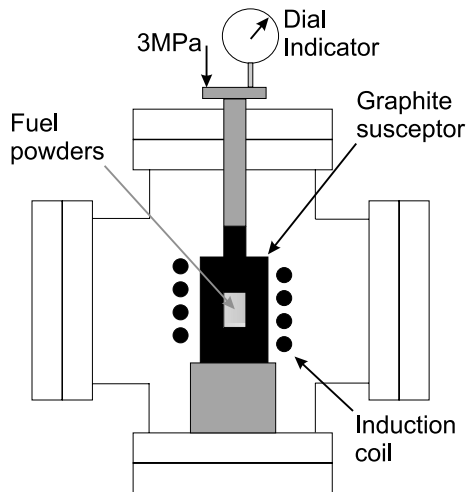| Nominal composition	U density (kgU/m^3)	TD (kg/m^3)	C/M	U/M
Phase I				
$(U_{0.1}, Zr_{0.45}, Nb_{0.45})C$	1838	8115	1.0	0.1
Phase II				
$(U_{0.1}, Zr_{0.58}, Nb_{0.32})C_{0.92}$	1563	7773	0.92	0.1
$(U_{0.1}, Zr_{0.68}, Nb_{0.22})C_{0.92}$	1539	7660	0.92	0.1
$(U_{0.1}, Zr_{0.77}, Nb_{0.13})C_{0.92}$	1522	7551	0.92	0.1
$(U_{0.05}, Zr_{0.62}, Nb_{0.33})C_{0.92}$	801	7379	0.92	0.05
$(U_{0.05}, Zr_{0.71}, Nb_{0.24})C_{0.92}$	788	7265	0.92	0.05
$(U_{0.05}, Zr_{0.81}, Nb_{0.14})C_{0.92}$	776	7153	0.92	0.05

Fig. 1. An illustration of the induction furnace chamber showing major components.

combusted in a LECO carbon determinator to measure the amount of CO_2 and hence the amount of carbon in each sample. Measurement of carbon is made using an infrared cell that measures the amount of absorption of the infrared energy by the CO_2. This same method was used to characterize the batches of powders comprising each sample.

As noted earlier, hypostoichiometric fuels are required to maintain a single-phase microstructure and a target C/M of 0.92 was selected for this study. In Phase II, constituent refractory carbide powders of $NbC_{0.92}$ and $ZrC_{0.99}$ were mixed with uranium hydride (UH_3) and graphite in proportions to produce this target C/M. The UH_3 was produced by flowing Ar–7%H over uranium metal rod heated to 473 K. The hydrogen reacts with the surface uranium metal producing UH_3, which is swept from the surface forming a fine UH_3 powder for mixing with the other powders [18]. Based on Gibb's free energy calculations using the FACT computer code [19] for the decomposition of UH_3, the hydride should decompose above 675 K evolving hydrogen and leaving uranium metal together with the powders of graphite and refractory carbides to form a mixed carbide. These samples were processed in the same manner as the earlier samples by cold pressing and sintering. The only exception was a modification to the coil and susceptor configuration to permit some Phase II samples to be processed for short periods of a few minutes at peak temperatures above 2800 K instead of 2700 K as with earlier samples.

5. Results

Table 3 lists the composition, TD and uranium density for samples processed in this study. The initial

processing of mixed carbides in Phase I from the constituent carbide powders showed that pre-compaction was necessary to achieve densities greater than 70% TD for sintering times of up to 2 h. Since low porosity samples (less than 5% porosity) were needed for testing, this was unacceptable and all later samples processed were first cold pressed prior to sintering as described above.

All of the Phase I samples exhibited good consolidation and produced mechanically robust samples (see Figs. 2 and 3). These samples were cold pressed at 120–140 MPa and sintered at peak temperatures approaching 2700 K for short periods of a few minutes before longer sintering times of up to 2 h at 2500–2600 K. XRD analysis showed these samples to be solid solutions of the mixed carbides (see Fig. 4).

The initial results of Phase II (hypostoichiometric) samples processed from $ZrC_{0.99}$, $NbC_{0.92}$, UH_3 and graphite were markedly different. These samples did not produce dense, well-consolidated and sturdy samples, as did those processed from the constituent carbides under the same conditions. Instead, they were weak and would not stand up to polishing but were ground down. SEM
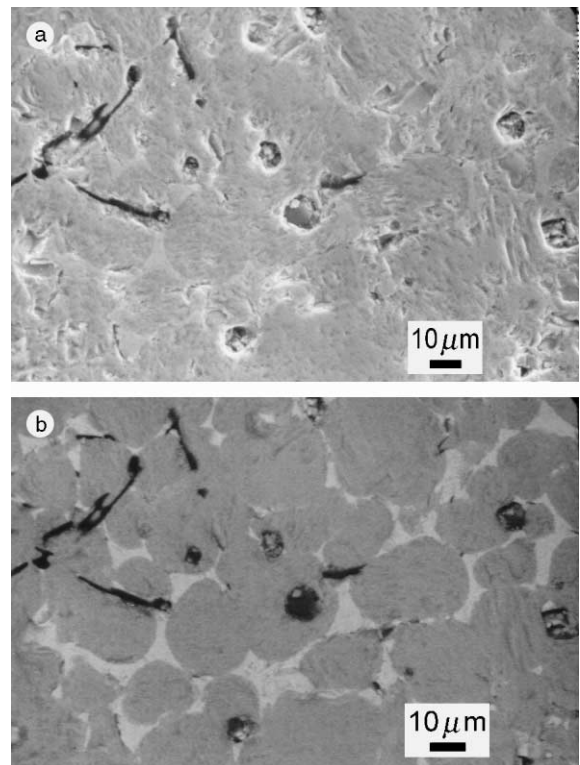

Fig. 2. Not fully sintered Phase I tri-carbide, ($U_{0.1}$, $Zr_{0.45}$, $Nb_{0.45}$)C, sintered for 20 min above 2600 K (peak sintering temperature near 2700 K): (a) SEM; (b) SEM with compositional contrast.
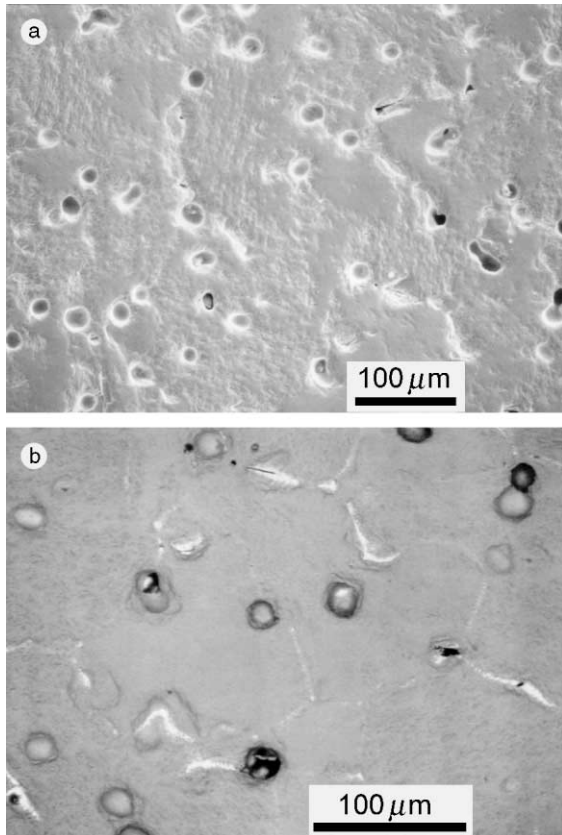
Fig. 3. Phase I tri-carbide, $(U_{0.1}, Zr_{0.45}, Nb_{0.45})C$, sintered for 20 min above 2600 K and 142 min above 2500 K (peak sintering temperature near 2700 K): (a) SEM; (b) SEM with compositional contrast.
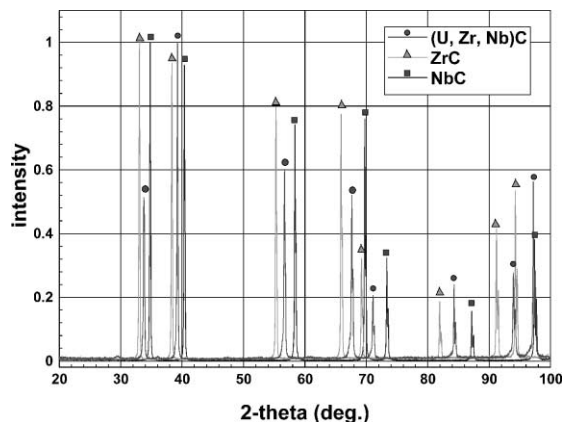

Fig. 4. Comparison of XRD patterns of a Phase I, solid-solution tri-carbide with the original powders of ZrC and NbC (UC/UC$_2$ omitted for clarity).

revealed these samples to have a large amount of open space in the microstructure and XRD analysis showed
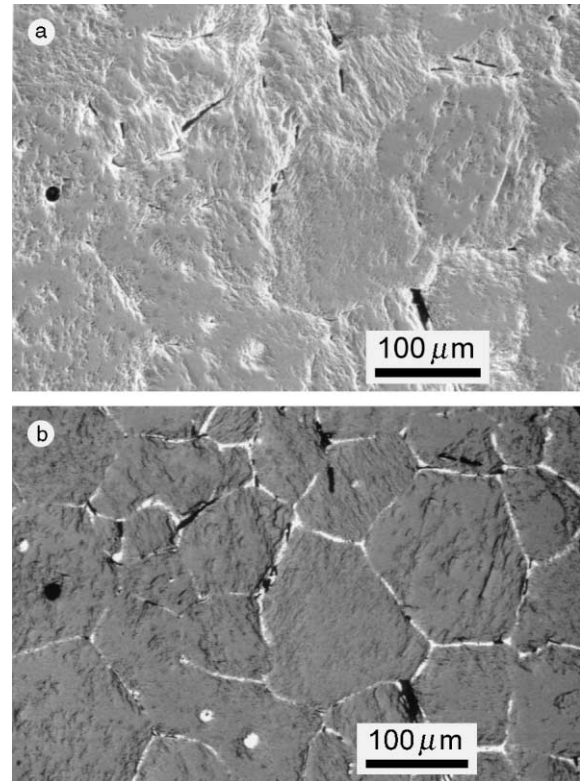

Fig. 5. Hypostoichiometric (Phase II) tri-carbide, $(U_{0.1}, Zr_{0.77}, Nb_{0.13})C_{0.98}$ sintered for 128 min above 2500 K (peak sintering temperature of 2800 K): (a) SEM; (b) SEM with compositional contrast.

that these samples were not solid solutions. However, this was remedied by increasing the peak sintering temperature above 2800 K to produce well-consolidated samples with low porosity (see Fig. 5).

Analysis of the carbon content of as processed samples showed no change for Phase I (stoichiometric) samples from their as mixed compositions. However, Phase II (hypostoichiometric) compositions with the original target of C/M equal to 0.92 showed an increase of 6.5% to 0.98% for the as processed samples. This significant increase in carbon content can likely be attributed to carbon 'pickup' from the graphite susceptor with which samples are in contact with during sintering at temperatures above 2500 K and for times of 2 h or more.

6. Discussion

As noted above, all the Phase I samples exhibited good consolidation and produced a mechanically robust sample. SEM of representative samples are shown in Figs. 2 and 3 along with backscattered electron images

showing compositional contrast. In these images, the lighter colored areas indicate regions of largely UC, which are located in the grain boundaries and between regions of the refractory carbides. Comparing the compositional contrast images, less UC is observed in the grain boundaries for longer sintering times, which provides for greater volume diffusion and produce a more homogeneous sample. Also, samples with less porosity are achieved as the material is allowed to sinter for longer periods of time (see Figs. 2 and 3). A sintering time of approximately 2 h was required to produce single-phase samples with less than 5% porosity.

The expansion and contraction of the sample was monitored during sintering by the linear motion feedthrough and dial indicator (graduated in thousandths of an inch) that contacted the graphite punch and sample (see Fig. 1). An initial expansion of the sample was noted as it was heated to the sintering temperature followed by a period of compaction (consolidation) upon approaching the peak sintering temperature. The compaction of Phase I samples was noted to be rather fast in the early minutes before reaching a much slower compaction rate. This is further evidenced by some samples that were sintered for only 5 min but achieved almost 90% TD. This combined with the large amount of UC in the grain boundaries of samples sintered for less than 2 h indicates liquid-phase sintering with the UC/UC_2 present in the compact. The rapid consolidation upon approaching the peak sintering temperature near 2700 K can be explained by the melting of the UC/UC_2 and the rearrangement of the refractory carbide particles together with the liquid UC filling the open spaces of the microstructure between particles.

However, liquid-phase sintering at these temperatures requires further explanation. The uranium carbide used in this initial study was a mixture of UC/UC_2 as confirmed through XRD analysis. At the high temperatures encountered during sintering, these uranium carbide powders with excess carbon, $UC_{1.65}$, are part of a homogeneous cubic phase, $UC_{1\pm x}$, extending to the dicarbide [20]. While stoichiometric UC melts at approximately 2800 K, this carbon rich composition exhibits a minimum in the melting temperature of 2677 K at 60 at.% C [16,20]. The peak sintering temperature experienced by these samples exceeded this minimum for short periods of a few minutes allowing for enhanced sintering by UC_{1+x} liquid-phase sintering.

XRD analysis of samples processed for times longer than 2 h showed them to be solid solutions of the mixed uranium/refractory metal carbides with sharp, well defined peaks sharing the same cubic structure of the constituent carbides (see Fig. 4). Therefore, one would expect that crystal spacing the solid solution would be intermediate between that of the predominant monocarbides. Indeed that is what is shown in Fig. 4 with peaks lying at expected values of 2-theta (crystal spacing

d) between those of the major components, the refractory carbides – namely ZrC and NbC.

Early hypostoichiometric samples of Phase II processed in this same manner except with different starting powders of $ZrC_{0.99}$, $NbC_{0.92}$, UH_3 and graphite exhibited a general lack of consolidation and a high degree of porosity. XRD analysis showed that they were not solid solutions but largely the individual refractory carbides. From their SEM images, they appeared much as binary carbide samples processed without uranium and exhibiting a high degree of porosity. Either of these cases can be understood in the absence of enhanced UC liquid-phase sintering since the UC formed in these Phase II samples from the uranium metal and graphite would melt at a higher temperature nearer the congruent melting point of 2803 K, which was not achieved during sintering of these early Phase II samples.

Indeed after modifications were made to the coil and susceptor configuration to reach peak sintering temperatures in excess of 2800 K, Phase II samples processed at this peak temperature did exhibit the same good consolidation and higher densities of the earlier samples produced from the constituent carbides with UC_{1+x} liquid-phase sintering (see Fig. 5). Continued sintering of these samples at lower temperatures of 2500–2600 K for times of 2 h or greater produced homogeneous solid solutions of these mixed uranium/refractory metal carbides, which was confirmed with XRD analysis. Furthermore, samples processed with 5% metal mole fraction of uranium all showed larger amounts of porosity than samples processed with 10% uranium under the same conditions, which can be understood since less of the liquid phase, UC or UC_{1+x} would be available to enhance the sintering process. This observation points to the importance of liquid-phase sintering to achieve low porosity samples. However, as discussed earlier, the amount of uranium should be limited to about 10% since larger fractions would further lower the melting point of the fuel and reduce its service temperature.

7. Conclusions

A processing and fabrication procedure was developed requiring pre-compaction such as cold pressing of samples prior to sintering to achieve the required density. Peak sintering temperatures above 2800 K are needed to achieve liquid-phase sintering and the associated rapid consolidation of the fuel material. Longer sintering times up to 2 h at high temperatures of 2500–2600 K are needed to achieve densities greater than 95% TD and to allow for sufficient volume diffusion and homogenization to form a solid-solution mixed carbide fuel. To achieve the greatest performance from

advanced tri-carbide fuels but still provide adequate UC liquid-phase for enhanced sintering, a uranium metal mole fraction of 10% was adopted. A C/M ratio of 0.92 was sought to maintain a single-phase microstructure, however, carbon pickup during sintering raised this to 0.98. Any greater pickup of carbon from the graphite susceptor wall than that observed in this study could pose a problem in future work if it was sufficient to form a second, carbon, phase. This can be remedied by sintering in a tungsten susceptor to prevent any increase in carbon content.

The techniques demonstrated in this study permit the fabrication of quality mixed carbide fuels for testing and characterizing their performance for NTP applications. Ongoing research is focused on testing and characterization of these advanced fuels including melting point determination and flowing hot hydrogen testing. Further plans include additional systems such as (U, Zr, Ta)C and (U, Zr, Hf)C, which in addition to their expected high performance may prove useful to ensure that the system remains sub-critical in any water submerged accident scenario.

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