



Evaluation of thermophysical properties of UO₂-10 Vol% Mo nuclear fuel pellets

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ABSTRACT

Uranium dioxide (UO₂)-molybdenum (Mo) composites have been proposed as accident tolerant fuel candidates due to their high thermal conductivity, high melting point, high boiling point, and ability to have low thermal neutron absorption cross-section. It is therefore essential to evaluate the method of fabrication and thermophysical properties of this system. In this work, UO₂-Mo samples with a Mo fraction of 10 Vol% and different densities were fabricated using spark plasma sintering (SPS). This method allows sintering at relatively low temperatures and within a short time. Experimental analysis was performed on UO₂-Mo specimens to characterize thermal diffusivity and thermal conductivity as a function of temperature up to 900 °C. Thermophysical properties were analyzed for various densities, and grain sizes as a function of temperature. The grain size and microstructural differences related to porosity were also analyzed using the scanning electron microscopic (SEM) and electron backscatter diffraction (EBSD).

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1. Introduction

The improvements in thermophysical properties of UO₂ have been an active area of research after the Fukushima disaster. The nuclear reactor safety studies showed that thermal conductivity is an important property of the fuel because it controls the fuel operating temperature and therefore has an influence on the safety of reactor operation. Low thermal conductivity leads to the meltdown at the core of the reactor during the loss of coolant scenario. The thermal conductivity is therefore a critical parameter in the reactor design that governs the transformation of heat produced from fission to electricity [1,2]. The composite fuel research is focused on improving the thermophysical properties of UO₂, with special emphasis on the improvement of the thermal conductivity [3–7]. In such composite fuels, excellent thermal properties, good irradiation resistance, control of fissile material density, and good mechanical properties can be obtained through the microstructural design and optimizing the concentration of the added metal [8,9].

Molybdenum (Mo) is a potential additive material because it has high thermal conductivity, high boiling temperature, high melting point, and low thermal neutron absorption cross-section [10]. Other, non-metal, materials such as BeO, SiC, and diamond,

have been previously evaluated for such applications [10,11]. Kim et al. [10] fabricated the micro-cell UO₂-Mo pellets with uniform Mo cell-walls using the compatible conventional process. These studies demonstrated that continuous Mo channels increase the bulk thermal conductivity more efficiently than randomly distributed Mo particles up to 1000 °C.

Buckley et al. [12] attempted to produce UO₂-Mo through the SPS technique and improved the thermal conductivity of the matrix by optimizing the concentration of dopant up to 800 °C. Similarly, Finkeldei et al. [13] developed the UO₂-Mo composite fuel from the sol-gel feedstock. The fabrication of this composite from Mo metal powder feedstock and microspherical UO₃ feedstock was explored for UO₂-Mo pellet fabrication. This study showed that the thermal conductivity was increased in UO₂ fuel candidates with the addition of a secondary Mo phase.

In our study, we used the SPS method to fabricate UO₂-Mo composite using a wide range of processing parameters. Compared to all the UO₂-Mo composites reported to date, we obtained very high densities (above 96% TD) at relatively low sintering temperatures. The density of the pellet plays an important role in the performance of nuclear fuel. The nuclear fuel with the desired density is expected to accommodate fission gas release and avoid possible in-reactor shrinkage [14]. In the present work, the effect of processing conditions such as sintering temperature, holding time, and heating ramp rate on the density and structure of pellets will

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Table 1
Summary of dopant powders.

Dopant #	Material	Supplier	Particle Size	Purity (%)
Mo Powder		Alfa Aesar	150 μm	99.9
Mo Powder		Sigma-Aldrich	100 nm	99.8
Mo Powder		Alfa Aesar	60–80 nm	99
Mo Powder		US Research Nanomaterials Inc.	35–45 nm	99.9

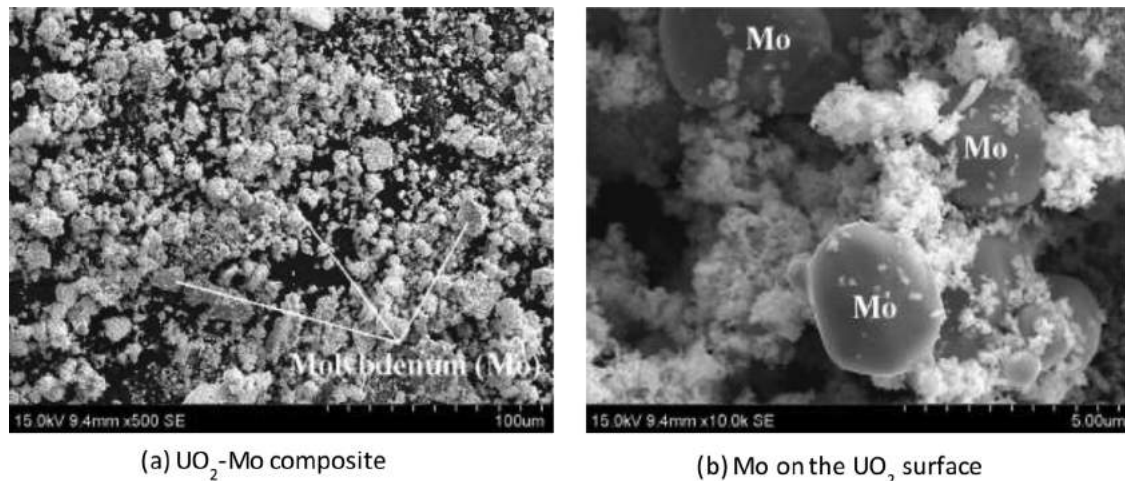


Fig. 1. Micrographs of UO_2 -Mo composite after mixing in the ball milling. The highlighted are some Mo particles but many of them can also be observed on this image.

be presented. The microstructural analysis of UO_2 -10vol% Mo powder and pellets was carried out to study the impact of Mo particle size on the development of homogeneous pellets and the evaluation of the porosity. The porous samples are expected to have lower thermal conductivity than high-density pellets, and therefore, high-density composite fuel pellets are desirable in the fabrication of enhanced thermal conductivity fuel pellets. The thermal conductivity changes as a function of temperature will be measured and the effect of microstructure on thermomechanical properties will be analyzed.

2. Materials and methods

2.1. Precursor material

Homogeneous uranium dioxide (UO_2)-molybdenum (Mo) powder mixtures with following Mo mean diameters of 150 μm , 100 nm, 60–80 nm, and 35–45 nm were prepared. The commercial uranium dioxide (UO_2) powder was purchased from Cameco Corporation (Saskatoon, SK, Canada). The dopant constant composition of 10 vol% Mo in UO_2 was taken. Table 1 summarizes the dopant material, supplier, and purity of the samples. Weighted powders with different mean diameters Mo in UO_2 were blended in a planetary ball mill (Torrey Hills-ND2L) for powder preparation.

2.2. Powder preparation and sintering

Five powder batches were prepared: UO_2 , UO_2 with 150 μm Mo, UO_2 with 100 nm Mo, UO_2 with 60–80 nm Mo, and UO_2 with 35–45 nm Mo with a constant dopant of 10 vol% Mo. Each batch of 30 g of the UO_2 and Mo mixture was placed in stainless steel 285 ml vessel with a ball to powder ratio of 8:1 for 2 h. Isopropanol was used as the process control agent (PCA), and the six wt% of it was mixed with UO_2 -Mo powder before milling. The milling speed was maintained at 200 rpm. After the powder

blending, there is no significant change in the particle size due to the optimized milling parameters. The SEM observation of UO_2 -Mo mixed powders shows that Mo particles are spongy aggregates, as shown in Fig. 1. EDS analysis of composite powders showed that Mo is uniformly distributed on the surface of UO_2 particles, as shown in Fig. 2. It is also evident from the EDS Spectrum that no reaction product or intermetallic was formed after the whole milling process.

The pellets were fabricated from each batch in an argon atmosphere using a Thermal Technologies 10-3 spark plasma sintering (SPS) system at the University of British Columbia (Kelowna, BC). The powder was loaded into a graphite die with an internal diameter of 12.7 mm. A thin graphite foil (0.125 mm) was used to separate the powder to avoid chemical reaction and eliminate friction between the UO_2 -Mo powder and the graphite tool. An optical pyrometer monitored the die temperature during the experiment. The temperature, chamber pressure, voltage, and current were observed as a function of time. The sintering temperature of pure UO_2 pellets was between 1000 and 1300 $^{\circ}\text{C}$. In the UO_2 -Mo-10 vol% composite, the sintering temperature had increased to 1300–1800 $^{\circ}\text{C}$ to achieve comparable density levels. The different heating ramp rate of 50–125 $^{\circ}\text{C}/\text{min}$ was applied. The sintering pressure was held constant at 60 MPa and a holding time of 0.5 min at the highest sintering temperature was used. After the sintering process, the pellets were ground using 500 grid silicon carbide (SiC) paper. The pellets were then polished by grinding them with SiC papers starting with 300 grit and gradually polishing up to 4500 grit to remove the residual graphite foil.

2.3. Characterization

The pellets' densities were measured using the Archimedes principle [15]. The densities can be calculated by measuring the weight in air and the weight in water. The density measurement TORBAL (AGCN200) equipped with a resolution of 0.0001 g for

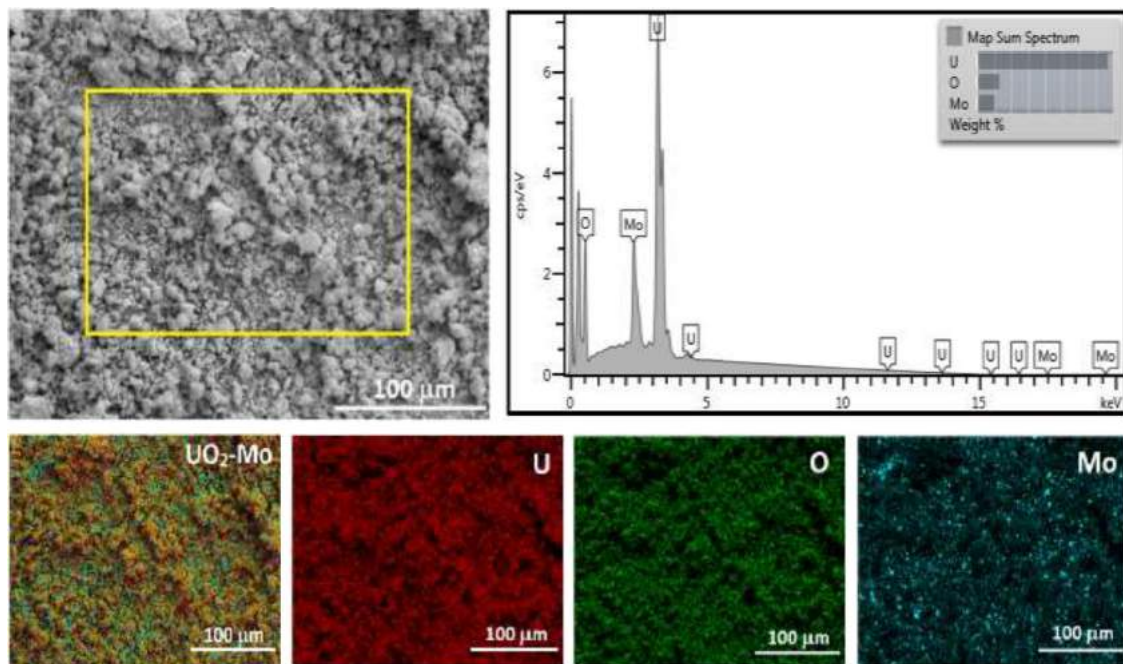


Fig. 2. EDS analysis of the UO_2 -Mo mixture showed the elemental composition; Mo is uniformly distributed on the surface of UO_2 structures.

accuracy in determining each pellets' densities was analyzed by immersing the pellets into the distilled water. The three readings were recorded for each specimen, and the average data with the error bar was reported. The theoretical density of the UO_2 -Mo composite pellet was calculated using the volume fractions for UO_2 and Mo, 90 and 10 vol% and the theoretical densities for UO_2 and Mo, 10.97 g/cm³ and 10.28 g/cm³. The theoretical density of the UO_2 -Mo-10vol% composite pellet was 10.901 g/cm³, respectively. The thermal diffusivity measurements of the sintered pellets were carried out using a laser flash TA instrument (DLF-1/EM-1300). In this technique, a high-power laser with (450 μs pulse width) of a solid-state Nd: glass laser pulse is used to illuminate the pellet's front face. Simultaneously, the resulting temperature rise is observed on the rear face with a liquid nitrogen-cooled InSb infrared detector. The specimen is coated with graphite spray to increase the emissivity and absorptivity of the laser pulse. The measurements were performed in the argon atmosphere, starting from room temperature to 900 °C. By measuring the time taken for the temperature rise in the rear face, the laser flash technique records the thermal diffusivity (α) of the samples using the Parkers relations [16] given in Eq. (1):

$$\alpha = 0.1388 \times \frac{L^2}{t_{1/2}} \quad (1)$$

Where L is the thickness of the sintered sample, and $t_{1/2}$ is half of the maximum time for the signal to reach the detector. From the measured thermal diffusivity (α), the thermal conductivity as a function of temperature ($k(T)$) was calculated using the relation (2):

$$k(T) = \alpha(T) \times \rho(T) \times C_p(T) \quad (2)$$

Where $\rho(T)$ is the density, and $C_p(T)$ is the heat capacity at a constant pressure as a function of temperature. The heat capacity was calculated using the model derived for thermal linear expansion data by Bakker et al. [17].

Pellets used in this analysis were disk-shaped and diameter about 12 to 12.7 mm in diameter and the thickness of samples in the range of 2 to 3 mm. The specimen thickness was measured using a calibrated micrometer and the average value was used.

The standard deviation of the average sample thickness was less than 0.02 mm. The cross-sections of powders and pellet samples were prepared for microstructural characterization using a Field Emission Scanning Electron Microscope (SEM) Hitachi Su 6600, equipped with Energy Dispersive Spectrometry (EDS). The SEM and EDS scans were measured with an acceleration voltage of 15 kV and a working distance of 9–10 mm. The crystallographic structure was analyzed through X-ray diffraction (XRD) using a BRUKER D8 Discover system with chromium K_α radiation. XRD patterns did not show the formation of any intermetallic products during the SPS process.

3. Results

3.1. Composite density

The densities of the fabricated UO_2 and UO_2 -10vol% Mo composite pellets were measured after different processing conditions. Fig. 3(a) shows the theoretical density (TD) of UO_2 -10 vol% Mo pellets at different sintering temperatures. The peak of sintering temperature was varied between 1300 and 1800 °C while all the other sintering conditions were the same. The density of sintered pellets increased with the increase in the sintering temperature. The results showed that the choice of sintering temperature had a significant effect on the pellets' final density and varying the sintering temperature from 1300 to 1500 °C resulted in average density increasing from 94 to 98% TD. Increasing the sintering temperature further to 1800 °C did not cause any considerable difference in the density, indicating that the maximum compaction of UO_2 -Mo occurs at a temperature of 1500 °C. However, it was also observed that the pellets with a density of ~97%TD could be obtained at a sintering temperature as low as 1300 °C. With other processing parameters constant; like applied pressure of 60 MPa, heating ramp rate of 125 °C/min, and a time of 0.5 min. Under the higher testing temperatures, compaction of the pellets was ~98% TD and complete closure of pores in the UO_2 -Mo composite was observed. The densities obtained at 1300, 1500, and 1800 °C sintering temperature with 100 °C/min heating ramp rate at a constant pressure of 60 MPa and a constant holding time of 0.5 min are shown in

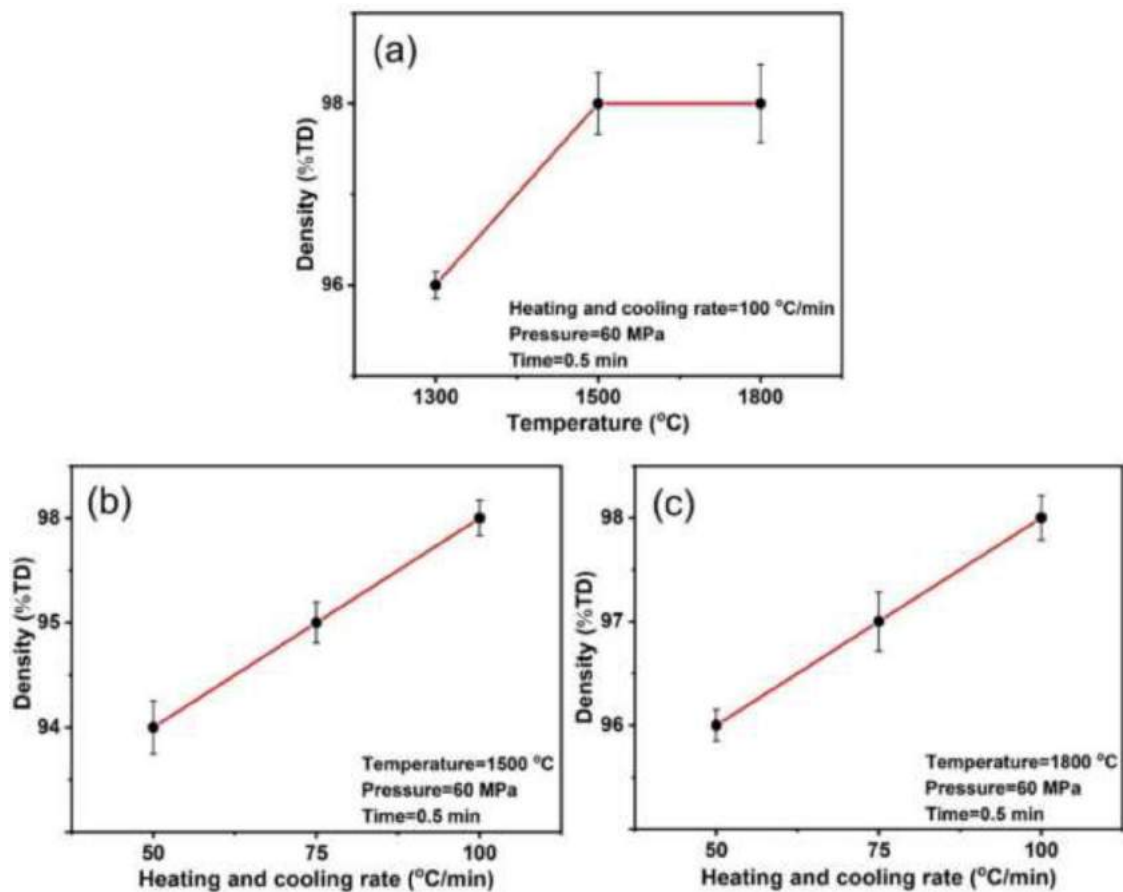


Fig. 3. The influence of (a) sintering temperature (b,c) heating ramp rate °C/min on the densification of the sintered pellets at 1500 and 1800 °C sintering temperature with an applied pressure of 60 MPa and constant sintering time of 0.5 min.

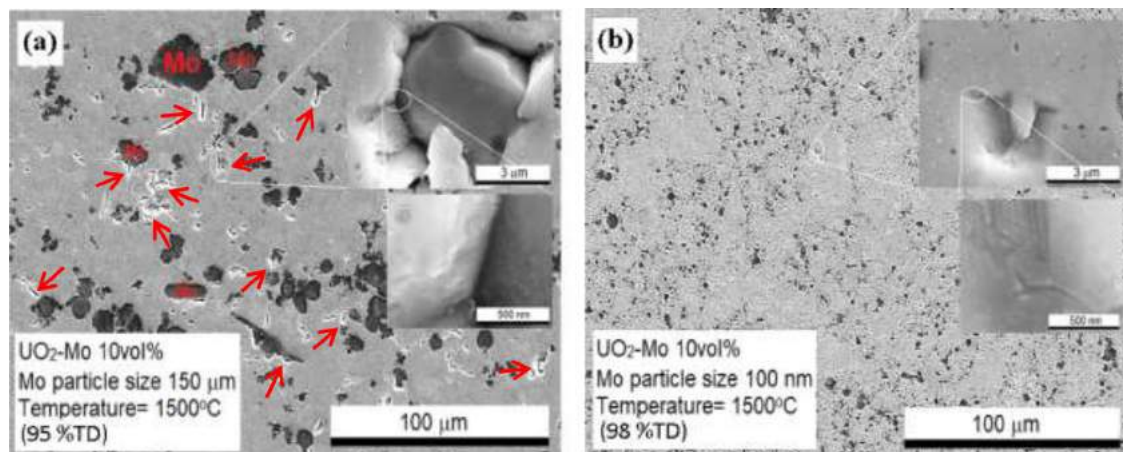


Fig. 4. The microstructure of UO₂-10vol% Mo composite pellets with two different size particles. (a) 150 μm. (b) 100 nm. Note micro-cracks are originating from large size Mo particles in a.

Fig. 3. The effect of heating ramp rate (°C/min) on the density of the composites was analyzed by changing the heating ramp rate between 50 °C/min, 75 °C/min, 100 °C/min at 1500 and 1800 °C sintering temperature, keeping the sintering pressure at 60 MPa and holding time for 0.5 min. The performed experiments show that the heating ramp rate of 100 °C/min is sufficient to get a pellet of UO₂-Mo with the desired density. Consequently, the density measurement clearly shows that using SPS we can lower the sintering temperature to 1300 °C and the sintering time 0.5 min.

3.2. Composite microstructure

Fig. 4 shows the microstructure and the porosity of the two UO₂-10Mo pellets fabricated at the same sintering conditions by varying for different sizes of dopant particles. The Mo particles appear black, and the brighter area represents the UO₂ matrix. The uniformity of Mo distribution in UO₂ was observed in the composite with small-sized Mo particles, as shown in **Fig. 4b**. The larger particle size induces higher compressive stress due to the lower

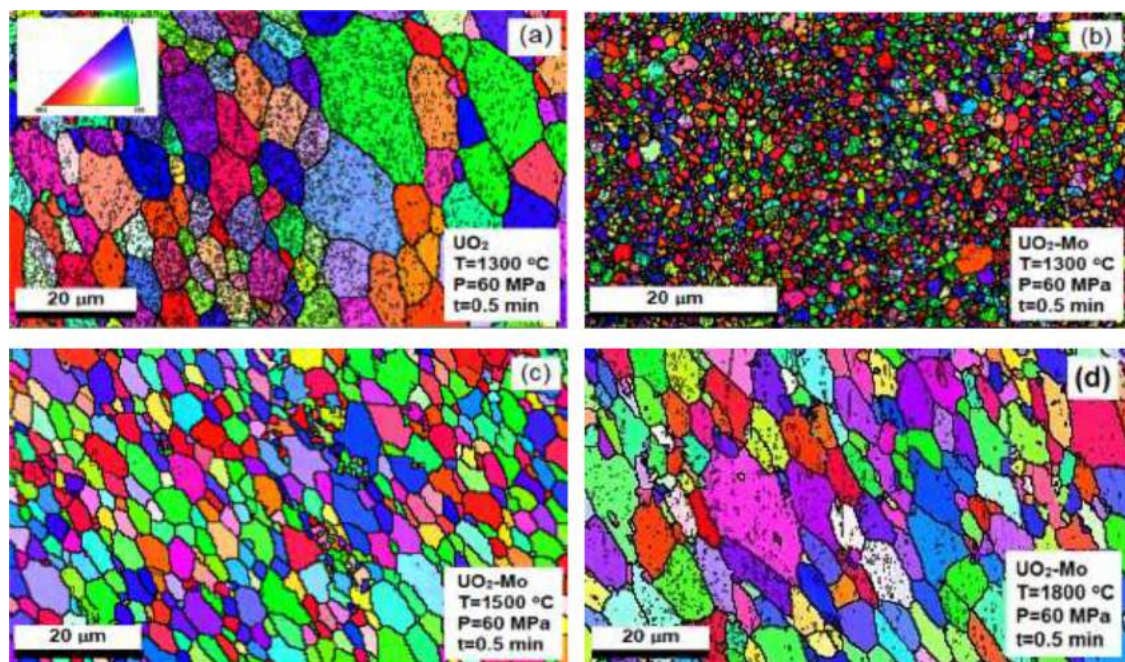


Fig. 5. EBSD maps (a) UO_2 at 1300 °C with an average grain size of (5.5 μm), (b) $\text{UO}_2\text{-Mo}$ at 1300 °C with an average grain size of (0.9 μm), (c) $\text{UO}_2\text{-Mo}$ at 1500 °C with an average grain size of size (2.4 μm), (d) $\text{UO}_2\text{-Mo}$ at 1800 °C with an average grain size of size (5.6 μm).

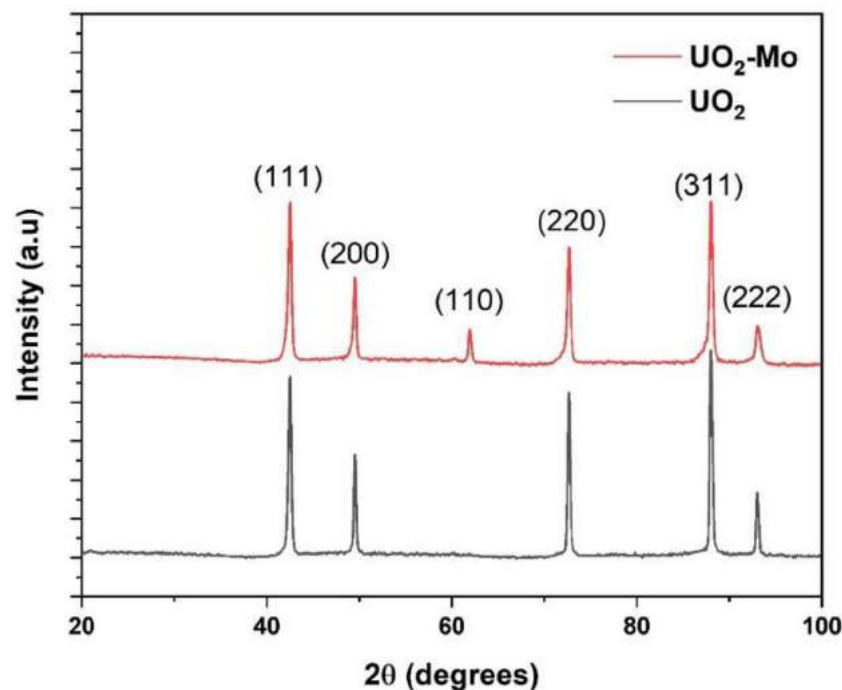


Fig. 6. Comparison of the X-ray diffraction (XRD) patterns of the sintered UO_2 and $\text{UO}_2\text{-10vol\% Mo}$ composite pellet.

surface-area-to-volume ratio [18]. However, micro-cracking and interfacial debonding exist in many sintered composites and are a consequence of different coefficients of thermal expansion (CTE) of the matrix and the particles [18–22]. Lu et al. [20] observed the thermal micro-cracking in several intermetallic composites and noted that an optimum particle size exists at which the micro-cracking is suppressed. Fig. 4a shows the micro-cracks in the composite, which can create open porosities, and a large amount of fission products can be released. Once the gas is released to free space in the fuel pin, the pressure inside the fuel pin increases,

which may lead to cladding rupture. Also, the porous samples are expected to have lower thermal conductivity than high-density pellets.

3.3. Composite grain size

The grain size of the pellet plays an important role in fission gas release and swelling [7,23]. It is essential to have control over the grain size of the composite fuel pellets. The EBSD analysis quantitatively and visually represents the grain size of the pellets.

The orientation maps could also provide information regarding the porosity, microstructure, and grain orientation. The maps of the sintered UO_2 and UO_2 -10Mo pellets as a function of fabrication temperature ranging from 1300 to 1800 °C represents an average grain size of 5.5, 0.9, 2.4 and 5.6 μm with heating and cooling rate 50 to 75 °C/min, constantly applied pressure of 60 MPa, and constant hold time of 0.5 min are shown in Fig. 5. The orientation maps indicate that increasing the fabrication temperature can produce fuel pellets with different grain sizes.

3.4. Structural analysis

Fig. 6 compares the XRD patterns of the UO_2 pellet sintered at 1300 °C and the UO_2 -10vol% Mo composite pellet sintered at 1500 °C. The XRD peaks of the sintered pellets matched with the joint committee on powder diffraction standards (JCPDS) database [24], the peak at 62° corresponds to (110) plane of Mo. The other peaks at 42°, 49°, 73°, 88°, and 94° correspond to the (111), (200), (220) (311), and (222) planes of UO_2 . The XRD result in Fig. 6 on the UO_2 -10vol% Mo composite, also indicates that there is no formation of any by-products and no intermetallic reactions during the sintering process.

3.5. Thermal diffusivity and thermal conductivity

The thermal diffusivity and the thermal conductivity measurement of UO_2 and UO_2 -Mo as a function of densities are presented in Fig. 7. The measurements are made three times at each temperature, and an average value is plotted from room temperature to 900 °C. The average thermal conductivity values of UO_2 are in good agreement with the literature data [7,25]. The UO_2 -Mo composite pellets were made for two different samples sintered at 1300, and 1500 °C, and the measured values of densities are ~96% TD and ~98% TD. Whereas the UO_2 pellet sintered at 1300 °C has a density of ~98% TD as shown in Fig. 7b. The UO_2 at 1300 °C had an average grain size of 5.5 μm , and the UO_2 -Mo at 1300 °C and 1500 °C had an average grain size of 0.9 and 2.4 μm , respectively. The UO_2 -Mo sample sintered at a temperature of 1500 °C had a higher thermal conductivity than the pellet sintered at 1300 °C. The reason for this was the enhanced microstructure, including the density and grain size.

The effect of densities of the UO_2 and UO_2 -Mo composite pellets on the thermal diffusivity measurements, as shown in Fig. 7a. In this analysis, the samples were heated in a graphite resistive furnace, and the measurements were carried out under vacuum at a temperature from 25 to 900 °C. The laser flash apparatus is based on the standard propagation of error results from the nature of the thermal conductivity measurements. The relative error estimated for the whole temperature range is about 10% by considering the relative uncertainties on the thermal diffusivity ($\pm 4.5\%$), heat capacity (2%), and density 3% [26].

4. Discussion

The UO_2 -Mo composite shows a similar tendency to UO_2 , a gradual reduction of thermal conductivity with an increase in temperature. This trend in thermal conductivity changes is due to the phonon-phonon scattering mechanism that dominates in ceramics and composite materials [7,27]. It is significant for the presented results that SPS provides denser UO_2 -Mo composite pellets without any intermetallic products in a much shorter sintering time, compare to traditional sintering. Beside, the structure of the composite is highly homogeneous, and thermal conductivity can be controlled. However, it was noted that the specimens with a smaller grain size have lower thermal conductivity [28,29]. The grain growth is mainly affected by the sintering temperature,

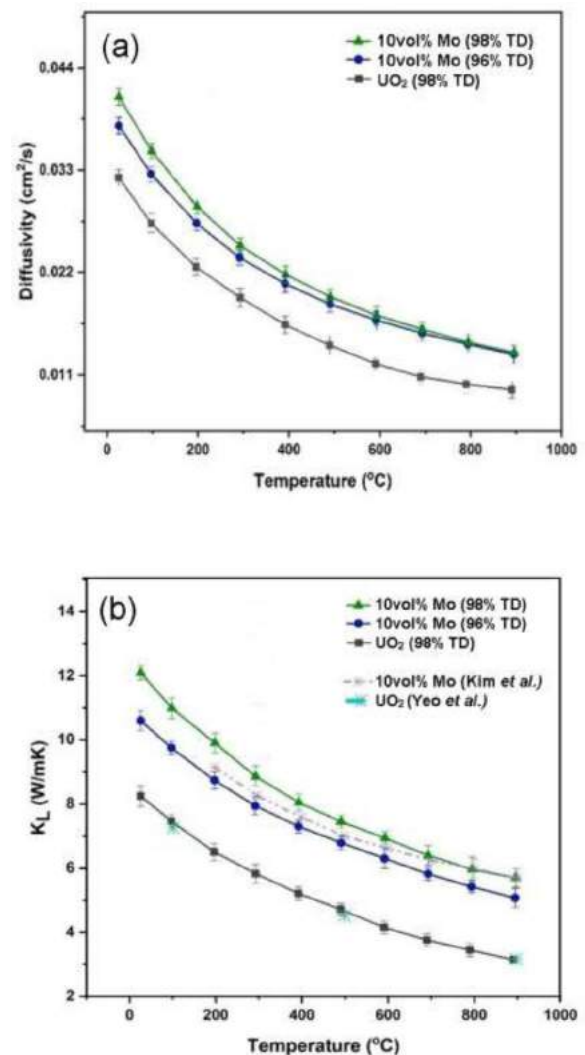


Fig. 7. (a) The thermal diffusivity of UO_2 and UO_2 -Mo as a function of temperature for samples sintered at 1300 and 1500 °C (densities are ~96% TD and ~98% TD). The uncertainty in the data points from the laser flash method is $\pm 4.5\%$. (b) The thermal conductivity of the UO_2 at 1300 °C (average grain size of 5.5 μm), and the UO_2 -Mo at 1300 and 1500 °C (average grain size of 0.9 and 2.4 μm).

and the grain size of these pellets sintered at 1300, 1500, and 1800 °C was 0.9, 2.4, and 5.6 μm . Whereas the UO_2 pellet sintered at 1300 °C have a grain size of 5.5 μm , as shown in Fig. 5.

The UO_2 -Mo composite showed an enhanced thermal conductivity compared to UO_2 pellets. Noticeably, the higher density, sintering temperature, and the larger grains, the higher the measured thermal conductivity. A maximum thermal conductivity improvement was observed in the UO_2 -Mo composite sintered at 1500 °C, with a density of ~98% TD, respectively, compared to the UO_2 thermal conductivity, as shown in Fig. 7b. Similarly, Yeo et al. [7] noted that the SPS sintering temperature had a significant effect on thermal conductivity than the oxidative sintering. The pellet density and grain size were different in the UO_2 pellet than the UO_2 -Mo pellet sintered at the same conditions. That differences contribute to the enhanced thermal conductivity of the UO_2 pellet.

5. Conclusion

The densification process is an important stage in the composite fuel manufacturing cycle; however, it is difficult to obtain dense fuel pellets using traditional sintering techniques. The con-

ventional sintering of nuclear fuels required a maximum sintering temperature higher than 2000 °C, and long holding time, and needs for various sintering aids. The UO₂-Mo composites produced by SPS can have high-density pellets at a low sintering temperature within a short time. The UO₂-Mo pellet with the desired density of ~97%TD can be obtained at a temperature as low as 1300 °C. An investigation of the influence of processing conditions on the UO₂ and UO₂-Mo composite pellets' density was explored. The range of sintering temperature and heating ramp rate (°C/min) has been systematically varied from 1300 – 1800 °C, and 50 – 125 (°C/min), respectively. Also, this work demonstrated that the densification of the UO₂-Mo composite fuel could be controlled by the choice of the processing parameters.

EDS observations showed that the Mo particles are homogeneously distributed in the UO₂ matrix. To understand the influence of Mo particle size and distribution of the second phase particles in the UO₂-10vol% Mo composite. The 10vol% of different Mo (micro and nanoparticles) was incorporated in the UO₂ matrix. The uniformity of Mo distribution in UO₂ was observed in small size Mo for particles of different sizes 35–45 nm, 60–80 nm, 100 nm, respectively. SEM studies demonstrated that by varying the Mo particle size, it is possible to tune the microstructural characteristics of UO₂-Mo composite fuel. EBSD studies demonstrated that higher SPS temperatures resulted in larger grain sizes. The homogeneous distribution of Mo particles influences morphology, by minimizing the porosity and reducing the visible cracks in the sintered pellets. Also, non-homogeneous large-size Mo particles support the hypothesis that cracking and interfacial debonding are responsible for the reduction in thermal conductivity of UO₂-Mo composite. The thermal conductivity measurement reveals their dependence on the densification and grain size of the specimens. Understanding the effect of the processing parameters on the pellets' density, and thermal conductivity enables us to tailor the fabrication parameters to manufacture a highly customized composite fuel with preferred properties. Combined experimental investigations demonstrated that the UO₂-Mo composite pellet has significantly improved the thermal conductivity of the UO₂ pellet.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Murali Krishna Tummalapalli: Conceptualization, Methodology, Investigation, Writing – original draft. **Jerzy A. Szpunar:** Supervision, Funding acquisition, Writing – review & editing. **Anil Prasad:** Formal analysis, Visualization. **Linu Malakkal:** Writing – review & editing. **Lukas Bichler:** Supervision, Writing – review & editing.

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