Microstructural Evolution and Radionuclide Release Behavior of SIMFUEL Under Coupled Effects of Burn-up, Sintering, and Leaching Temperature

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Abstract

Understanding the long-term behavior of spent nuclear fuel (SNF) is crucial for safe radioactive waste management. This study evaluates how burn-up, sintering temperature, and leaching temperature impact the leaching and microstructural stability of SIMulated FUEL (SIMFUEL) pellets designed to simulate SNF characteristics. Pellets with burn-up levels of 35 and 55 GWd/tU were sintered at 1550 °C and 1700 °C and subjected to leaching tests over one year at 25 °C, 60 °C, and 90 °C. Radionuclide release and microstructural changes were investigated using ICP-MS, SEM, XRD, and EDS. Results show that higher sintering temperatures enhance microstructural stability and reduce radionuclide release. Burn-up influences release behaviors in an element-dependent and time-sensitive manner. Leaching temperature generally accelerates release rates and surface corrosion. Despite subtle compositional and size changes at elevated temperatures, key metallic inclusions remain largely stable. These findings highlight the combined effects of fabrication and environmental factors on SIMFUEL durability and radionuclide mobility. This improved understanding aids in predicting long-term SNF performance and informs approaches for ensuring disposal safety.

Full Text

Preamble

Microstructural Evolution and Radionuclide Release Behavior of SIMFUEL Under Coupled Effects of Burn-up, Sintering, and Leaching Temperature

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Abstract

Understanding the long-term behavior of spent nuclear fuel (SNF) is crucial for safe radioactive waste management. This study evaluates how burn-up, sintering temperature, and leaching temperature impact the leaching and microstructural stability of SIMulated FUEL (SIMFUEL) pellets designed to simulate SNF characteristics. Pellets with burn-up levels of 35 and 55 GWd/tU were sintered at 1550 °C and 1700 °C and subjected to leaching tests over one year at 25 °C, 60 °C, and 90 °C. Radionuclide release and microstructural changes were investigated using ICP-MS, SEM, XRD, and EDS. Results show that higher sintering temperatures enhance microstructural stability and reduce radionuclide release. Burn-up influences release behaviors in an element-dependent and time-sensitive manner. Leaching temperature generally accelerates release rates and surface corrosion. Despite subtle compositional and size changes at elevated temperatures, key metallic inclusions remain largely stable. These findings highlight the combined effects of fabrication and environmental factors on SIMFUEL durability and radionuclide mobility. This improved understanding aids in predicting long-term SNF performance and informs approaches for ensuring disposal safety.

Keywords: SIMFUEL, Radionuclide Dissolution, Burn-up Effects, Sintering Temperature, Leaching Temperature

Introduction

As global energy demand rises and climate change concerns deepen, nuclear power continues to gain prominence as a low-carbon energy source. However, the safe and sustainable disposal of high-level radioactive waste, particularly spent nuclear fuel (SNF), remains a critical challenge. Ensuring the long-term stability of SNF is essential not only for environmental safety but also for energy policy, public trust, and international regulatory compliance [1, 2]. This necessity is further emphasized by continuous changes in regulations, ongoing shifts in nuclear fuel designs, and the need to secure long-term repository performance. Addressing these challenges requires reliable scientific approaches and a comprehensive understanding of SNF degradation under diverse environmental conditions, including thermal, chemical, and radiological conditions [3-5].

Of particular concern is high burn-up SNF, which exhibits altered microstructures after prolonged irradiation [6,7]. The situation becomes more complex when dealing with damaged fuel, where cladding failure or matrix degradation increases the risk of direct radionuclide release. Such fuel is generally unsuitable for direct disposal in its existing state because it negatively affects storage integrity and leaching resistance [8, 9]. Recognizing these challenges, countries such as France, Sweden, and Finland are actively investigating treatment methods aimed at stabilizing damaged fuel and preparing it for final disposal [10-13].

In this regard, dry treatment methods similar to the DUPIC (Direct Use of Spent PWR Fuel in CANDU reactors) process have gained attention [14]. These technologies can potentially transform damaged fuel into stable disposal forms. To validate such approaches, it is necessary to understand the leaching behavior and microstructural evolution of reprocessed fuel especially under simulated conditions relevant to deep geological repositories. Due to the complexities involved in directly handling SNF such as radiological hazards, cost, and regulatory restrictions, SIMulated FUEL (SIMFUEL) has been widely adopted as an experimental surrogate. This non-radioactive material is designed to resemble actual SNF in its morphological and chemical features by including stable fission product isotopes [15]. These additions are designed to replicate the formation of key secondary phases observed in irradiated fuel, such as metallic -particles and various oxide precipitates [16, 17].

Therefore, this study aims to investigate the long-term leaching and microstructural behavior of SIMFUEL under repository-relevant conditions. Specifically, we examine the effects of burn-up level (35 and 55 GWd/tU), sintering temperature (1550 °C and 1700 °C), and leaching temperature (25 °C, 60 °C, and 90 °C). By analyzing the relationships among these parameters, the study seeks to clarify how fabrication conditions and burn-up level influence leaching resistance and phase stability. The academic contribution of this work lies in its detailed analysis of the relationships between synthesis parameters, fuel microstructure, and leaching performance. Practically, the findings offer data essential for evaluating repository safety and for developing techniques to stabilize high burn-up or damaged SNF prior to disposal.

2.1. SIMFUEL Pellet Preparation and Characterization

SIMFUEL pellets were fabricated to simulate spent nuclear fuel (SNF) with burn-up levels of 35 GWd/tU and 55 GWd/tU. The compositions of fission product simulants were determined using the ORIGEN (Oak Ridge Isotope Generation and Depletion) code [18] as shown in Table 1 . High-purity UO_2 powder was dry-blended with stable oxide surrogates (e.g., MoO_3, Y_2O_3, $BaCO_3$, ZrO_2, and rare-earth oxides) designed to replicate major post-irradiation elements, excluding volatile elements. The mixture was homogenized using a planetary ball mill and mixed with 0.2 wt% Acrawax as a lubricant. Powder mixtures were pressed under 250 MPa into cylindrical green pellets.

Table 1. Oxide surrogates and elemental compositions in SIMFUEL pellets corresponding to 35 and 55 GWd/tU burn-up levels.

Sintering was performed at either 1550 °C or 1700 °C under a reducing atmosphere (Ar-4% H_2) for 6 hours with a heating and cooling rate of 5 °C/min. The dimension of each pellet was approximately 8.5 mm in diameter and 6.5 mm in height. Pellet densities were measured using the Archimedes method with a precision densimeter (MDS-3000), and theoretical densities (TD) were calculated individually per composition. Resulting values were: 96.3% TD (35

GWd/tU, 1700 °C), 88.9% TD (35 GWd/tU, 1550 °C), 90.8% TD (55 GWd/tU, 1700 °C), and 86.2% TD (55 GWd/tU, 1550 °C).

Microstructural features were characterized via scanning electron microscopy (SEM; Hitachi SU-8010). Grain size was determined from backscattered images using ImageJ software. Over 200 grains per sample were measured for statistical reliability. Crystalline phases and structural changes were identified by X-ray diffraction (XRD; Bruker D8 Advance). Compositional mapping and point analyses were conducted with energy-dispersive X-ray spectroscopy (EDS; Horiba X-Max), particularly focusing on -phase inclusions such as Mo–Ru-rich particles.

2.2 Leaching Experiments and Analytical Procedures

Leaching behavior was evaluated under semi-dynamic conditions using a modified ASTM C1308 method [19]. Each SIMFUEL pellet was immersed in 35 mL of deionized water in PTFE vessels, with a solution-to-surface-area ratio of approximately 11.7 mL/cm^2. Experiments were conducted at 25 °C, 60 °C, or 90 °C in temperature-controlled ovens. Periodic sampling was carried out over 365 days at selected intervals (3 h, 1, 2, 3, 7, 14, 28, 56, 112, 182, and 365 days) with leachant replacement to maintain sink conditions. Leaching temperature effects were examined primarily using 55 GWd/tU pellets sintered at 1700 °C, representing a well-sintered microstructure with low intrinsic release. Burn-up and sintering temperature effects were also analyzed consistently at 60 °C.

Collected leachate samples were filtered through 0.45 m PTFE filters and acidified to 1% HNO_3 prior to elemental analysis. Analysis was performed using inductively coupled plasma–mass spectrometry (ICP–MS; Analytik Jena PlasmaQuant MS 9100) at KAERI's accredited laboratory. A total of 17 elements were targeted: U and 16 key fission product analogues, including Mo, Sr, Ba, Zr, Ru, Rh, Pd, Te, La, Ce, Pr, Nd, Sm, Eu, and Gd. While detection limits varied by element (typically <1 ppb), quantifiable concentrations were primarily observed for U, Mo, Sr, and Ba which were consistently above detection thresholds across all conditions.

To evaluate the extent of elemental release during leaching, results were interpreted using two key metrics defined by ASTM C1308-21. The Incremental Fraction Leached (IFL) represents the mass of an element released into solution during each sampling interval. The Cumulative Fraction Leached (CFL) reflects the total fraction of a given element released over time, calculated by summing all preceding IFL values up to the specific time point.

3.1. Burn-up and Sintering Effects on SIMFUEL Stability

The initial microstructure of SIMFUEL pellets was evaluated to assess the effect of intrinsic fabrication parameters (sintering temperature and burn-up level) on matrix stability and leaching behavior. Figure 1 shows representative SEM images and grain size distributions for all pellet groups. Sintering at 1700 °C resulted in consistently larger grain sizes compared to 1550 °C, confirming grain coarsening due to enhanced material diffusion [20, 21]. Burn-up effects on grain size were less pronounced but present.

Figure 1 [Figure 1: see original paper]. SEM images and grain size distributions of as-sintered SIMFUEL pellets. (a) 35 GWd/tU at 1700 °C; (b) 55 GWd/tU at 1700 °C; (c) 35 GWd/tU at 1550 °C; and (d) 55 GWd/tU at 1550 °C. Histograms show grain size distributions fitted to Gaussian functions to determine average grain size and standard deviation.

Lower sintering temperatures led to increased porosity and grain boundary density, which are known to enhance dissolution kinetics and promote radionuclide mobility [22]. This behavior is reflected in Figure 2, showing cumulative fraction leached (CFL) curves for representative radionuclides over 365 days at 60 °C. Pellets sintered at 1700 °C generally exhibited lower radionuclide release than those sintered at 1550 °C, confirming the microstructural stabilization effect of higher sintering temperature.

Burn-up effects varied by element and sintering temperature. For U-238, 55 GWd/tU pellets sintered at 1700 °C released more uranium than their 35 GWd/tU counterparts, whereas this trend reversed at 1550 °C. U-238 release rates increased with time but generally plateaued after about 56 days, indicative of surface depletion and matrix stabilization [23]. Mo-95 release steadily increased over time for all samples, suggesting gradual dissolution or surface mobilization of Mo from -particles distributed within the matrix [24]. Sr-88 and Ba-137 exhibited more complex patterns. Under 1700 °C sintering, Sr-88 release was initially higher in 55 GWd/tU pellets but later surpassed by the 35 GWd/tU samples, implying differential contributions of soluble and secondary phases over time [25]. At 1550 °C, higher burn-up correlated with consistently higher Sr-88 release, likely due to increased grain boundary connectivity. Ba-137 showed similar trends, influenced by both burn-up and sintering temperature, reflecting phase-specific dissolution kinetics [26].

These results underscore that sintering temperature is the dominant factor reducing radionuclide release by stabilizing the microstructure, while burn-up influences are element- and time-dependent, mediated by complex interactions of phase stability and microstructural evolution. These findings emphasize the importance of considering both fabrication parameters and burn-up conditions for long-term leaching assessments.

Figure 2 [Figure 2: see original paper]. Cumulative Fraction Leached (CFL) of selected radionuclides (U-238, Mo-95, Sr-88, and Ba-137) as a function of time. Open symbols: 1550 °C; closed: 1700 °C. Blue: 35 GWd/tU; Red: 55 GWd/tU.

3.2. Leaching Temperature Effects on SIMFUEL Stability

The effect of leaching temperature was examined using 55 GWd/tU SIMFUEL pellets sintered at 1700 °C. As shown in Figure 3 [Figure 3: see original paper], temperature significantly influenced radionuclide release kinetics and magnitudes, although each element responded differently. For U-238, higher temperature increased dissolution rate and cumulative release, with a plateau reached more rapidly at elevated temperatures within ~7 days at 90 °C, ~28 days at 60 °C, and slowly approaching plateau near 365 days at 25 °C. This trend is attributed to fast initial matrix dissolution followed by passivation or surface saturation, consistent with oxidative dissolution processes [27]. Mo-95 exhibited steady, continuous release at all temperatures with no plateau, implying ongoing corrosion or partial dissolution of -particles. Sr-88 and Ba-137 displayed temperature-dependent release patterns with an initial plateau or minimal increase during the first ~14 days, followed by accelerated release at higher temperatures. This suggests a delayed onset of more aggressive corrosion or dissolution of Sr- and Ba-containing secondary phases [28].

Hence, leaching temperature intensifies radionuclide release by both accelerating kinetics and altering phase stability in SIMFUEL. The distinct release profiles reflect differing dissolution mechanisms for U-238 (matrix-dominated), Mo-95 (metallic phase corrosion), and Sr-88/Ba-137 (soluble secondary phases). Understanding these detailed temporal and thermal dependencies is critical for reliable prediction and modeling of SIMFUEL's long-term behavior.

Figure 3 [Figure 3: see original paper]. Cumulative Fraction Leached (CFL) of U-238, Mo-95, Sr-88, and Ba-137 from 55 GWd/tU SIMFUEL pellets sintered at 1700°C at different leaching temperatures (25°C, 60°C, and 90°C).

Following the temperature-dependent leaching behavior detailed in Figure 3, the surface microstructural changes of the SIMFUEL pellets after 365 days of leaching were examined via SEM and XRD, as presented in Figure 4 [Figure 4: see original paper]. As leaching temperature increased, the SIMFUEL pellet surface exhibited worsened degradation—in particular, the 90 °C sample showed extensive corrosion pits and surface roughness compared to smoother surfaces at 25 °C and 60 °C (Figures 4a–c). The XRD patterns further confirmed significant structural alterations correlated with temperature. While all samples maintained the primary fluorite UO_2 phase, the peaks in the 90 °C sample showed noticeably reduced peak intensities and a distinct shift toward higher 2 angles (Figure 4d). This peak shift indicates lattice contraction typically resulting from oxidation-driven formation of hyperstoichiometric UO_2 [29].

These microstructural changes support the release behaviors observed in Figure 3, where higher temperatures not only accelerated the rate and amount of radionuclide release but also caused increased oxidation and surface damage of the SIMFUEL pellets. Overall, the SEM and XRD analyses show that temperature strongly affects SIMFUEL stability by promoting oxidation-driven degradation, which in turn can lead to enhanced radionuclide release under conditions relevant to long-term storage.

Figure 4 [Figure 4: see original paper]. (a-c) SEM images showing the surface morphology of 55 GWd/tU SIMFUEL pellets sintered at 1700°C after 1 year of leaching at (a) 25°C, (b) 60°C, and (c) 90°C, respectively. (d) X-ray Diffraction patterns of the same SIMFUEL pellets, including pristine (as-sintered) and leached samples at various temperatures, along with a UO_2 cubic Fm-3m reference.

Further insight was gained from EDS mapping and point analysis (Figure 5 [Figure 5: see original paper]). The presence and persistence of -particles (Ru, Mo, Rh-rich metallic inclusions) at grain boundaries were confirmed under all leaching conditions (Figures 5a–c). Despite the continuous Mo release observed in leachate (Figure 3), these inclusions largely retained their spatial integrity, implying dissolution primarily occurs via surface mechanisms or alternative mobile Mo reservoirs within the microstructure rather than wholesale particle breakdown.

Quantitative point analyses (Figure 5d) showed that at 25 °C and 60 °C, -particles consistently had high Ru and Mo contents, exceeding uranium levels, confirming their identity as metallic phases. At 90 °C, an apparent compositional shift was observed with increased uranium and decreased Ru/Mo, attributed to particle size reduction and embedment in the UO_2 matrix that affects EDS measurement volume rather than true stoichiometric change.

These observations highlight a strong temperature dependence of SIMFUEL degradation, with -particles showing macroscopic stability despite subtle surface or size modifications at elevated temperatures. This behavior complements the leaching kinetics and microstructural changes discussed earlier, underscoring the critical influence of temperature on long-term radionuclide dissolution dynamics.

Figure 5 [Figure 5: see original paper]. EDS analysis results of 55 GWd/tU SIMFUEL pellets (sintered at 1700°C) after 1 year of leaching at various temperatures. Panels (a), (b), and (c) show the results for 25°C, 60°C, and 90°C leaching conditions, respectively. Each panel includes a composite EDS layered image, individual elemental maps for U, Ru, Mo, and Rh, illustrating their spatial distribution. Additionally, average quantitative compositional data from 2-3 -particle analyses for each respective temperature are presented as bar graphs in (d).

4. Conclusion

This study highlights how the long-term stability and radionuclide release behavior of SIMFUEL pellets are governed by an interplay of burn-up level, sintering temperature, and leaching temperature. Higher sintering temperatures promote the formation of coarser grains and denser microstructures, which confer greater resistance to degradation and mitigate radionuclide mobilization.

The influence of burn-up on leaching behavior is complex and element-specific, interacting with sintering conditions to yield varying release patterns over time. Elevated leaching temperatures consistently accelerate dissolution kinetics and enhance surface corrosion, directly impacting fuel stability and radionuclide mobility. Despite some evidence of surface modifications and minor changes in metallic inclusion size or composition at higher temperatures, key fission product -particles show remarkable persistence, underscoring their relative stability in long-term aqueous environments.

These microstructural and chemical changes align with observed leaching trends, demonstrating the critical role of fuel fabrication parameters and environmental exposure in dictating SIMFUEL performance. Overall, the findings emphasize the necessity of a comprehensive perspective when evaluating spent fuel models for disposal, one that considers coupled effects of burn-up history, material fabrication, and thermal conditions.

Such understanding improves the predictability of radionuclide dissolution models, supporting more reliable safety assessments and informing strategies for the stabilization and management of high burn-up or compromised nuclear fuels in geological repositories. This work contributes valuable insights toward enhancing the safety and effectiveness of long-term SNF disposal solutions.

Conflicts of interest

There are no conflicts of interest to declare.

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Note: Figure translations are in progress. See original paper for figures.