

Measurement of Hydrogen Generation from SNF Cladding Surrogates in a Lab-Scale “Mini-Canister”

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Abstract

Residual water in a spent nuclear fuel (SNF) canister, including both free water and water physisorbed or chemisorbed on surfaces, can break down under irradiation resulting in generation of hydrogen gas (H₂). SNF cladding surfaces provide large surface area for adsorbed water, and interactions between water and cladding with its attendant oxides are expected to strongly impact the H₂ generation rates and total yield. For example, aluminum research reactor fuel cladding can hold significant amounts of chemisorbed water in the form of aluminum (oxy)hydroxides, while zirconium cladding may accelerate radiolysis via energy transfer from ZrO₂ to physisorbed water.

Experimental data on radiolytic H₂ generation under conditions expected in dry storage is important for predicting the evolution of SNF-in-canister conditions to ensure safety during long-term dry storage. For this purpose, a lab-scale radiolysis testing apparatus was developed for repeated in-situ sampling of the gas inside a miniature steel canister (“mini-canister”) that fits into a ⁶⁰Co gamma irradiator. This system enables monitoring of radiolytic yield from a single sample and initial atmosphere over an extended irradiation.

Radiolysis testing of aluminum cladding surrogates with adherent aluminum (oxy)hydroxide films was conducted for three different drying conditions (one vacuum-only and two heated). The surrogate samples were exposed to gamma irradiation with absorbed doses up to 15 MGy. Testing included two “upset conditions”: the addition of a large “spike” of hydrogen from another source to one canister and post-irradiation heat treatments intended to release trapped hydrogen in the other two canisters. Similar testing of zirconium samples has begun recently to interrogate the radiolytic yield of H₂ from Zr surfaces with different oxide loadings, physisorbed water, and water vapor in the canister atmosphere. This data is expected to help in selection of drying approaches for aluminum-clad fuel and improve predictions of canister gas conditions over time for both SNF-relevant material systems.

1. Introduction

Radiolysis of residual water within spent nuclear fuel (SNF) dry storage canisters, whether in the form of free water, physisorbed water, or chemisorbed water (e.g., (oxy)hydroxides), can lead to build-up of hydrogen gas within sealed canisters. SNF dry storage canister configurations include a large surface area of SNF cladding, which can host adsorbed water. In particular, the cladding of aluminum-clad spent nuclear fuel (ASNF) can hold significant amounts of chemisorbed water as aluminum (oxy)hydroxides, which are not readily removed at low (<100°C) drying temperatures.

Safe storage of ASNF with its attendant chemisorbed water is of particular relevance for the U.S. Department of Energy (DOE), which currently has a large inventory of ASNF discharged from both foreign and domestic research reactors [1]. The current disposition path for this ASNF is chemical

processing at the Savannah River Site. Sealed, road-ready dry storage for ultimate disposition in a repository is being explored as a disposition alternative for the ASNF at SRS and as the primary disposition pathway of ASNF from the Advanced Test Reactor at the Idaho site. Joint research between Idaho National Laboratory and Savannah River National Laboratory has been investigating the corrosion, drying, and radiolysis of aluminum to establish a technical basis for safe, sealed dry storage of DOE ASNF [1].

Quantification of radiolytic gas generation from materials with surface conditions representative of SNF materials and under conditions similar to dry storage is important to establish expected H₂ generation rates and overall yield. This data facilitates the development of predictive models for SNF canister conditions over time during long-term dry storage. For this purpose, a laboratory-scale “mini-canister” radiolysis test system consisting of a small stainless-steel canister with an integrated gas sampling line was developed to enable repeated in-situ sampling of the gas space over samples under gamma irradiation. The extended in-situ monitoring enables tracking of the H₂ generation rate of each individual sample and sealed system from initial H₂ release through longer-term behavior.

The present paper discusses results from radiolysis testing of aluminum cladding surrogates with lab-grown aluminum (oxy)hydroxide films.

2. Background

Oxide types relevant to aluminum cladding include alumina (Al₂O₃), boehmite (aluminum oxyhydroxide, AlOOH or Al₂O₃·H₂O), and two aluminum trihydroxide polymorphs, bayerite and gibbsite (Al(OH)₃ or Al₂O₃·3H₂O). Other polymorphs of both Al(OH)₃ and AlOOH exist but are not commonly reported in studies of aqueous aluminum corrosion. The (oxy)hydroxides can be formed by direct reaction of water with aluminum metal (releasing H₂) or by hydration of existing oxide [2]. Multi-layered or mixed (oxy)hydroxide films have been reported in various studies, with boehmite-bayerite dual layers typically featuring bayerite on the outer surface and boehmite sandwiched between the bayerite and the metal [3].

The aluminum (oxy)hydroxides can be thermally dehydrated, with reaction paths and kinetics affected by factors such as particle size, rate of heating, and partial pressure of water vapor [2]. Drying experiments on bayerite films formed on aluminum substrates have demonstrated that it is feasible to at least partially decompose bayerite films by heating samples to >220°C by experimental methods including thermogravimetric analysis, heating under air in a furnace, or forced-helium dehydration (a drying process used for commercial SNF) [4, 5, 6]. The presence of boehmite after dehydration of bayerite has been confirmed in some of these studies [4, 5]. Thermal dehydration of boehmite requires significantly higher temperatures (e.g., ~400–500°C [2, 4]), making it impractical to remove during ASNF drying. The boehmite dehydration temperatures would be sufficient to cause phase changes in the aluminum metal [7] and exceed the design temperature range for the canister design intended to contain DOE SNF [8].

3. Experimental approach

3.1. Mini-canister system

The “mini-canister” system consists of a miniature stainless-steel canister (Figure 1) designed to fit inside the cavity of a ⁶⁰Co gamma irradiator and featuring a sampling tube through the lid to allow intermittent, in-situ sampling of the canister gas via connection to an instrumentation manifold. The canister is a commercially available vacuum vessel with internal diameter of 7.19 cm (2.83”), height

17.9 cm (7.06”), and copper gaskets sealing the bolted-flange endcaps. This system enables monitoring of the gas atmosphere within the mini-canister over the course of an irradiation period for a single sample and initial atmosphere, in contrast to previous glass-ampoule-based tests that yielded only a single data point per individual sample [9].

3.2. Aluminum cladding surrogates

Several aluminum sample assemblies were fabricated for radiolysis testing in the mini-canister system in order to investigate H₂ yields associated with aluminum (oxy)hydroxide films and to explore the impact of drying processes prior to helium backfilling and sealing [5, 9]. Each assembly consisted of 17 parallel, 1.3-mm (0.050-in) thick plates of aluminum 6061-T6 bolted together with aluminum hardware and separated by aluminum washers to provide a 2-mm gap between plates [5]. The plate height and widths were selected to conform closely to the cylindrical interior of the mini-canister in order to maximize the plate area in the test, resulting in an aluminum surface area of nearly 3800 cm² [5].

The assemblies were corroded to form an aluminum (oxy)hydroxide layer prior to irradiation testing to stand in for the (oxy)hydroxide layers found on reactor-exposed aluminum [10, 11]. Bayerite (Al(OH)₃) was chosen as the target oxide, allowing direct comparison of the radiolytic yield with and without thermal drying to dehydrate the oxide.

The plates were polished to 600 grit to remove existing oxide, assembled, and immediately immersed in room-temperature (22–23°C) distilled water to form a bayerite film over an immersion period of more than 30 days (Figure 2, left) [9]. The assemblies were weighed before immersion and after 7 days of air-drying post-immersion to determine the mass gain due to corrosion [5]. The



Figure 1. Sealed mini-canister for aluminum radiolysis campaign (with markings for a contaminated sample) showing sampling tube attached to lid.

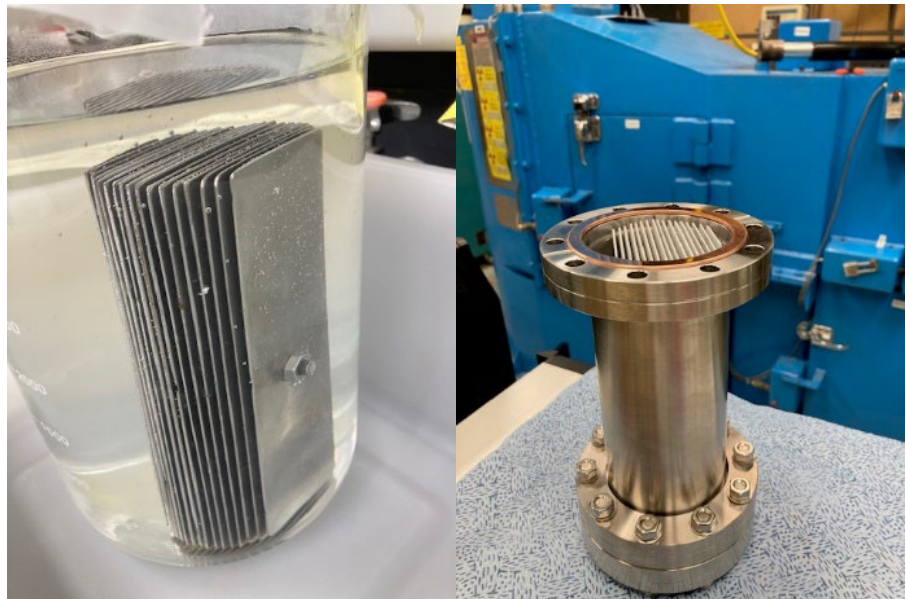


Figure 2. Surrogate assembly during immersion in room-temperature water (left) as well as loaded into the mini-canister (right) [9].

corrosion durations and associated weight gains for the three assemblies are summarized in Table 1.

Table 1: Weights of the sample assemblies before and after corrosion and of the characterization samples removed from them after corrosion. The weight gain provides a metric for estimating the amount of adherent oxide formed.

Assembly	Corrosion time (days)	Pre-corr. weight (g)	Post-corr. weight (g)	Weight gain (g)	% weight gain	Characterization sample weight (g)
As-Corroded	41	618.6	624.1	5.5	0.88%	7.4
220°C As-Dried	36	620.1	625.4	5.3	0.85%	8.6
150°C As-Dried	33	636.1	640.4	4.3	0.68%	N/A

A small section was cut from an external plate of two assemblies after corrosion for characterization of the oxide by X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD characterization of the as-formed oxide (“As-Corroded” assembly) showed prominent, sharp peaks for bayerite and for the aluminum metal of the substrate, as well as a small, broad peak corresponding to boehmite [5]. SEM showed a relatively consistent cross-section between 5 and 10 μm thick, and, in planview, a morphology consisting primarily of narrow ($\sim 0.1\text{--}0.5\ \mu\text{m}$ in width) rodlike structures, some of which appeared to form bundles of parallel rods [5].

The assemblies were then loaded into mini-canisters (Figure 2, right).

3.3. Drying

After the sample assemblies were loaded and sealed into the canisters, the three mini-canisters were subjected to different drying conditions: two different heated drying conditions and one unheated.

The heated drying consisted of placing the loaded and sealed mini-canister into a furnace at the target temperature (220°C or 150°C) with the sampling line open to lab air to allow moisture to escape [9]. Figure 3 shows water visibly dripping from the end of the open sampling line during drying at 220°C [9]. After 4 hours in the furnace, the sampling line was connected to a vacuum pump, the furnace was shut off, and vacuum was drawn for 1 hour while the canister remained in the residual furnace heat to remove remaining free water before it re-condensed [9].

All three mini-canisters were then exposed to vacuum for about 12 hours (at room temperature), followed by a residual air elimination process consisting of four alternating helium purges and short vacuum steps, and were finally backfilled with dry helium to ~ 24 psia [9].

The thermally dried mini-canisters are denoted as *220°C As-Dried* and *150°C As-Dried*, depending on the furnace temperature used during drying, while the canister with no heated drying is denoted as *As-Corroded*.

The sample cut from the 220°C As-Dried sample assembly was dried in the furnace alongside the corresponding mini-canister and characterized post-drying. XRD characterization showed that distinct, sharp boehmite peaks were present after 220°C



Figure 3. Water bubble forming on at the exhaust of the vessel during drying at 220°C [9].

drying and the height of the bayerite peaks relative to the aluminum peaks was smaller than for the As-Corroded oxide, although the bayerite peaks remained the most prominent (oxy)hydroxide signal [5]. This suggests that the 220°C drying process partially dehydrated the bayerite to well-crystallized boehmite. SEM showed that the post-drying oxide retained nearly identical morphology to the As-Corroded, except for the appearance of some narrow cracks [5]. The appearance of cracking with minimal visible change to other morphology is consistent with previous observations of bayerite films dried to >220°C and contraction of the oxide film from bayerite to the denser boehmite [2, 4].

3.4.Irradiation

The mini-canisters were sized to fit inside the cavity of a ^{60}Co gamma irradiator. The As-Corroded and 220°C As-Dried mini-canisters were initially irradiated in a JLS Model 484 irradiator with relatively low dose rate. Subsequently, an FTS Model 812 self-contained ^{60}Co gamma irradiator with a higher dose rate was used, and both the As-Corroded and 220°C As-Dried canisters were moved to the Model 812. The 150°C As-Dried canister irradiation was entirely completed in the Model 812.

Fricke dosimetry was performed inside the Model 812 irradiator in May 2022 using a large volume of solution evenly distributed throughout an otherwise empty mini-canister, indicating an average dose rate of 124 Gy/min [12]. Fricke dosimetry in a mini-canister inside the Model 484 irradiator indicated a decay-corrected dose rate of 6.3 Gy/min at the start of irradiation for the first (As-Corroded) mini-canister [13]. Reported cumulative doses account for the predictable falloff of the dose rate of the ^{60}Co due to decay with a 5.27-year half-life, although the impact is relatively small over the time scales of these experiments. Previous “round-robin” studies showed that radiolytic yields appeared to be independent of differences in dose rate between 8.3 and 175 Gy/min [14].

3.5.Gas sampling

A gas sampling manifold was used to collect 10-mL gas samples intermittently over the course of the irradiations. The pressure was recorded after each sample, and the gas sample was analyzed by a gas chromatograph (GC) to measure the H_2 , O_2 , and N_2 concentrations [12]. Two GC columns were used: the primary column used to measure H_2 was a 20-meter molecular sieve column with argon carrier gas; the secondary column, used primarily for O_2 and N_2 , was a 10-meter molecular-sieve column using helium cover gas [12]. Several gas samples are drawn from the canister for each data point, and the initial sample is discarded to avoid errors from possible gas contamination in the tubing, while the others are averaged. A vacuum pump evacuates the sampling manifold between samples [12].

Because each gas sample was permanently removed from the gas inventory remaining in the vessel, the cumulative radiolytic H_2 generated was calculated based on the current gas composition, the moles of H_2 removed in previous samples, and the changing vessel pressure.

3.6.Upset conditions

When the As-Corroded canister was moved from the Model 484 to the Model 812 irradiator, the valve in the gas sampling line was inadvertently positioned inside the radiation field, resulting in a “spike” of H_2 production attributed to irradiation of the valve. The H_2 concentration inside the mini-canister reached 13 vol% due to this H_2 spike [13]. This disruption terminated the initial experiment, but provided an opportunity to observe the system’s response to an increase in gas space H_2 concentration from a source external to the sample. Therefore, the experimental setup was

adjusted to irradiate the mini-canister with the valve positioned outside the radiation field, and additional samples were taken to see whether the sample's post-H₂-spike generation rate was changed by the increase in H₂.

After irradiating the 150°C and 220°C As-Dried assemblies to absorbed dose levels of >15 MGy, post-irradiation heat treatments were performed at 100°C, 150°C, and 200°C to see whether any potentially trapped H₂ could be liberated from the crystalline structure of the oxide and (oxy)hydroxide layers [12]. The still-sealed mini-canisters were exposed for 1-h in an oven heated to the target temperature, allowed to cool to room temperature, and then the gas was sampled [12].

4. Results and discussion

4.1. Radiolytic hydrogen yield under normal irradiation conditions

The As-Corroded mini-canister was irradiated to a dose of 1.45 MGy and the two As-Dried mini-canisters were irradiated to 15.6 MGy prior to their upset conditions. The cumulative radiolytic H₂ yields for all three are shown as a function of the absorbed dose in Figure 4.

First, the H₂ generation rate for the As-Corroded canister, which experienced only unheated vacuum drying, was significantly larger than that of the two thermally dried mini-canisters.

Second, the two As-Dried mini-canisters showed relatively similar H₂ generation over the course of their irradiations, despite the difference in drying temperatures. The 150°C As-Dried sample is expected to retain all of its chemisorbed water due to its drying temperature being below the threshold for decomposition of bayerite, while the 220°C drying was confirmed to partially decompose bayerite to boehmite. The 150°C As-Dried yield is somewhat lower than that of the 220°C As-Dried by the end of

the irradiation; however, the 150°C plate assembly experienced ~19% less mass gain during its corrosion step, and its cumulative H₂ yield at 15.6 MGy is about 17% lower, so it is likely this difference is attributable to variation in the amount of oxide grown rather than different radiolysis behavior. The similarity between the two As-Dried canisters and reduction compared to the As-Corroded suggest that much of the H₂ yield from the As-Corroded sample arises from physisorbed water that requires elevated temperatures, not just vacuum, for effective removal.

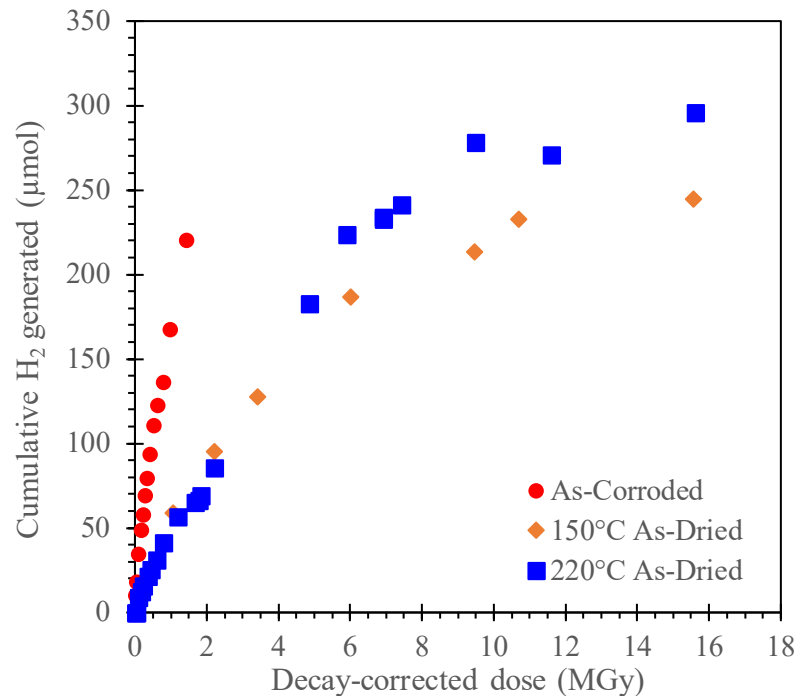


Figure 4. Cumulative radiolytic H₂ yields from all three mini-canisters under normal irradiation conditions.

4.2. Radiolytic hydrogen yield under upset conditions

4.2.1. H₂ spike

After its initial irradiation in the Model 484 irradiator, resulting in the data plotted in Figure 4, the As-Corroded mini-canister was moved to the Model 812 irradiator to continue irradiation at a higher dose rate [13]. Samples taken after the start of irradiation in the Model 812 showed anomalously high H₂ concentrations, corresponding to a H₂ generation rate nearly 15x higher than that seen during the initial irradiation, and the H₂ in the canister atmosphere reached 13 vol% [13]. Inspection of the setup revealed that the sample line's ball valve, which includes a polyether ether ketone surface, had been inadvertently positioned within the radiation field [13]. Adjustment of the apparatus to remove the ball valve from the radiation field resulted in the H₂ generation rate dropping again, confirming that irradiation of the valve was responsible for the H₂ spike. (No O₂ generation was observed as a result of the valve irradiation.)

Due to the discontinuity in the measured H₂ concentration, the As-Corroded mini-canister was repurposed to assess whether the addition of H₂ in the canister gas altered the radiolysis behavior of the sample [13]. It was hypothesized that back-reactions or competing reactions involving gaseous H₂ could potentially account for the gradual decrease in H₂ generation rate with increasing absorbed dose; if so, an increase in the H₂ concentration would be expected to inhibit further H₂ generation [13].

The cumulative H₂ generated before the H₂ spike (same data as in Figure 4) and after the removal of the valve from the radiation field are plotted together in Figure 5. The post-spike data includes only the calculated H₂ generation *after* re-starting the irradiation, i.e., the zero point for the "Post-Spike" data corresponds to the 13 vol% H₂ concentration (~3200 μmol) present at the beginning of this stage. The H₂ generation rate observed was very similar to that observed pre-spike. This suggests that the increased concentration of H₂ in the gas phase does not notably inhibit further radiolytic H₂ release from the sample, and consequently that any apparent "roll-over" effect observed in the radiolytic H₂ yield is not attributable to the elevated H₂ in the gas.

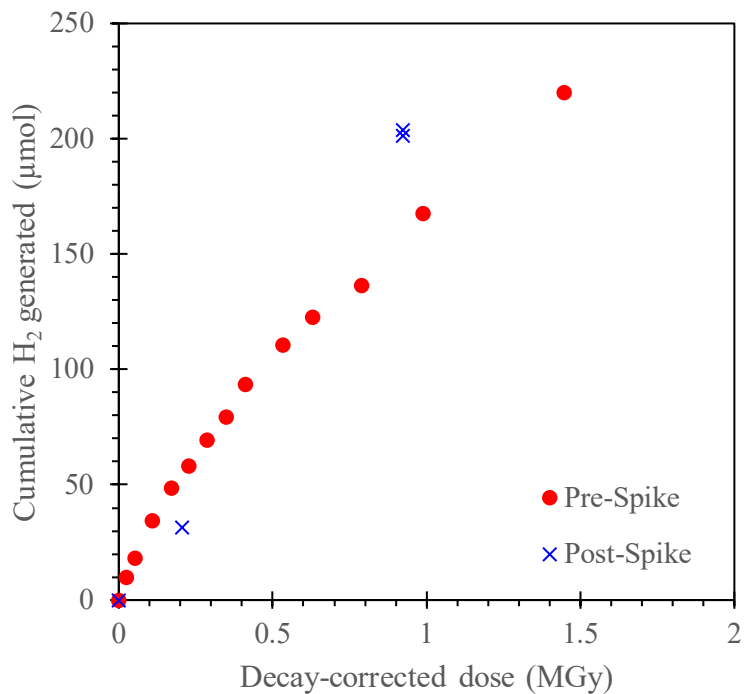


Figure 5. Cumulative radiolytic H₂ yields from As-Corroded mini-canister during the irradiation period before ("Pre-Spike") and after ("Post-Spike") the rapid addition of H₂ due to irradiation of a valve.

4.2.2. Heat treatments

The post-irradiation heat treatment of both As-Dried mini-canisters was performed after irradiation to over 15 MGy absorbed dose apiece. Table 2 gives the cumulative H₂ generation for each canister after the end of the irradiation (“Room Temp.”) and after 1-hour exposures of the sealed canisters to air temperatures of 100°C, 150°C, and 200°C [12].

Table 2: H₂ yields following post-irradiation heat treatments of 150°C and 220°C As-Dried mini-canisters [12].

Post-Irradiation Heat Treatment	Total H ₂ Produced Over Test (μmol)	
	150°C As-Dried	220°C As-Dried
Room Temp.	245	296
100°C for 1 hr	249	311
150°C for 1 hr	246	306
200°C for 1 hr	252	307

The heat treatments did not produce an appreciable change in the cumulative H₂ release into the headspace of either canister [12]. No more than a 5% difference in calculated cumulative H₂ generation was observed across the post-irradiation heat treatments of an individual mini-canister, and this degree of fluctuation could be caused by instrument uncertainty or variations in the internal container temperatures across different samples, despite allowing the external canister surface to cool to ambient room temperature (22°C–24°C) before each sample.

4.2.3. Oxygen

Molecular oxygen was also measured at each gas sample throughout the irradiation and after each post-irradiation heat treatment. No evidence of radiolytic generation of O₂ with increasing dose was observed during irradiation, nor additional release of O₂ during the heat treatments [12].

The lowest concentration of O₂ for which calibration was performed was 510 ppm, and calculated O₂ concentrations never exceeded this value for any of the gas samples for the three mini-canisters [12]. There were detectable levels of O₂ observed in GC samples below this concentration. However, these measurements fluctuated independently of dose and have been attributed to remaining O₂ impurities in the sampling manifold following extended periods between manifold purges. Same-day sequential sampling consistently resulted in O₂ concentrations estimated to be near 100 ppm at all dose and heat treatment conditions; instrument calibration using certified gas mixtures with lower O₂ concentrations would be needed to confirm the exact value.

5. Conclusions

Radiolysis experiments in helium-backfilled steel “mini-canisters” were able to intermittently measure radiolytic gas yields from ASNf cladding surrogates over the course of extended irradiations. The preparation conditions were chosen to investigate the impact of potential drying processes on the H₂ yield in storage: vacuum drying only (“As-Corroded”) versus two heated drying conditions at different temperatures (“150°C As-Dried” and “220°C As-Dried”). Two upset

conditions, an influx of H₂ from a source external to the sample (for the vacuum-dried sample) and post-irradiation heat treatments (for thermally dried samples), were also investigated.

The baseline irradiation data for the three mini-canisters showed the highest H₂ generation rate from the As-Corroded mini-canister (irradiated up to ~1.5 MGy), while the two thermal drying conditions yielded very similar H₂ yields over the entire irradiation to >15 MGy. The highest H₂ generation rates for all three occurred at the beginning of the irradiation, with some reduction in rate with increasing dose. The 220°C drying partially dehydrated the initial bayerite layer to boehmite, while 150°C is well below the temperature threshold where bayerite decomposition is expected. Therefore, the similarity between the yields of the two heated drying conditions and their substantial reduction compared to unheated vacuum drying suggests that the main contributor to the H₂ yield in this range is physisorbed water that requires elevated temperatures for effective removal.

A large “spike” of H₂ to ~13 vol% in the gas space of the As-Corroded canister occurred due to irradiation of a ball valve, but the elevated H₂ had no evident impact on the continued H₂ generation rate once the valve was removed from the radiation field. The unchanged H₂ generation rate suggests that the H₂ concentration in the cover gas has minimal effect on the radiolytic generation.

Post-irradiation heat treatment of the two As-Dried plate assemblies did not release any additional H₂ or O₂ beyond what was already available in the headspace, suggesting that no significant quantities of radiolysis products are trapped in the (oxy)hydroxide layers in a form likely to be released through ASNf self-heating or temperature excursions.

The mini-canister experimental setup continues to be used for additional radiolysis testing. Current test campaigns are measuring radiolytic yield from reactor-formed aluminum (oxy)hydroxide on a Missouri University Research Reactor ASNf fuel cropping [13] and from Zr surrogate samples to investigate yields relevant to commercial SNF [15]. Similar testing of a new As-Corroded sample assembly without extended vacuum drying is planned to provide a true bounding case for yield from physisorbed water.

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