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Minimum Ag Addition to Capture Residuals and Trace Iodine from INL CH_3-I Loaded AgZ in GCM

Fuel Cycle Research & Development

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Separations Working Group

Prepared for
U.S. Department of Energy
Office of Nuclear Energy –
Separations Working Group
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Sandia National Laboratories
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U.S. DEPARTMENT OF ENERGY

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SUMMARY

The minimum amounts of silver flake required to prevent loss of I_2 during sintering in air for a SNL Glass Composite Material (GCM) Waste Forms containing three methyl iodide loaded AgI-MORs (INL, Test 5, Beds 1, 2 and 3) was determined to be 1.10 wt% Ag (Bed 1), 1.15 wt% Ag (Bed 2) and 1.2 wt% Ag (bed 3). The final GCM composition prior to sintering was 20 wt% AgI-MOR, 80 wt% Bi-Si oxide glass plus the additional Ag amounts. The amount of silver flake needed to suppress iodine loss was determined using thermo gravimetric analysis with mass spectroscopic off-gas analysis. These studies found that the ratio of silver to AgI-MOR required is lower in the presence of the glass than without it. Therefore an additional benefit of the GCM is that it serves to inhibit some iodine loss during processing. Alternatively, heating the AgI-MOR in inert atmosphere instead of air allowed the iodine to react with more of the excess silver originally present in the Ag-MOR, so overall a smaller amount of Ag flake addition is needed to fully suppress iodine evolution. The cause of this behavior is found to be related to the oxidation of the metallic silver when heated to above ~300°C in air. These results are similar to the results of previous studies on I_2-loaded AgI-MOR materials.

ACKNOWLEDGEMENTS

The authors thank J. Griego for his experimental help, Nick Soelberg for providing the INL CH_3-I-Ag-MOR samples, and both Stephanie Bruffey and Robert Jubin of ONRL for providing the Ag-MOR sample characterization information (samples used in INL CH_3-I sorption studies). SNL acknowledges funding from the DOE/NE Fuel Cycle R&D - Separations Working Group (Waste Forms Campaign). Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

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1. ABBREVIATIONS AND ACRONYMS

Ag	Silver
Ag^+	Silver ion
Ag°	Silver metal
AgI	Silver Iodine
Bi	Bismuth
°C	Degrees Celsius
FCRD	Fuel Cycle Research and Development
FY	Fiscal Year
g	Gram
GCM	Glass Composite Materials (waste form)
hr	Hour
I	Iodine
I_2	Iodine (gas)
INL	Idaho National Laboratory
K_{sp}	Solubility Product
L	Liter
min	Minute
MOR	Mordenite Zeolite
MS	Mass Spectroscopy
ORNL	Oak Ridge National Laboratory
Si	Silicon
SNL	Sandia National Laboratories
TGA	Thermo Gravimetric Analysis
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

2. INTRODUCTION

In the spent nuclear fuel reprocessing procedures under consideration by the US DOE, gas containing $^{129}I_2$ and CH_3-^{129}I is passed through a bed of a silver-exchanged Ag°-Mordenite (Ag°-MOR), that selectively captures the iodine to form AgI–MOR.[1,2] Most of the captured iodine reacts with the silver in the Ag°-MOR to form AgI while the rest is chemically adsorbed. Silver iodide (melting point = 558°C and density = 5.675 g/cm^3)[3] has a low solubility in water, 3 x 10^{-6} g/L at 20°C[3] (corresponding to a K_{sp} of $10^{-16.1}$), which makes silver a good candidate for the capture of ^{129}I. For safe storage, the AgI-MOR must be incorporated into a dense, durable solid waste form, so that the release of iodine is inhibited with time or environmental contact. Because of their flexibility of synthesis and chemical durability, low temperature sintering glasses are being examined for the encapsulation of AgI-MOR iodine capture materials to form Glass Composite Materials (GCMs), for subsequent use as waste forms.[4-7] They are easier and less expensive to prepare than conventional ceramics but also offer high durability, as shown in our previous work.[8]

In our previous reports[5-8] on I_2-loaded Ag°-MOR we identified a commercially available low-temperature sintering glass powder based on silicon and bismuth oxides that sinters to full density at 550°C and has excellent durability. To prevent surface/bulk chemisorbed I_2 vapor from escaping from the zeolite during GCM sintering, additional Ag flake was added to the GCM mixture to capture the desorbing I_2 vapor. The amount of Ag necessary to prevent loss of I_2 during GCM thermal processing of a GCM containing I_2 vapor loaded Ag°-MOR was studied in detail in FY14.[9] We have found that additional silver is necessary even when there is an excess of silver to iodine, on a molar basis. This process was successful as the excess Ag captured the adsorbed iodine and formed AgI.

The purpose of the present study is to optimize the amount Ag flake needed to suppress iodine loss in GCMs containing AgI-MOR that were loaded using CH_3-I. The AgI-MOR materials were supplied by Idaho National Laboratory (INL) and were labeled Test 5 Bed 1, Test 5 Bed 2 and Test 5 Bed 3.

3. EXPERIMENTAL METHODS

Starting Materials:

Three samples of CH_3-I loaded AgI-MOR were received from INL; due to the fact that CH3-I is not present in the final samples but AgI is present, these samples are labeled AgI-MOR. They were labeled Beds 1, 2 and 3 from Test 5. The silver mordenite (Ag-MOR) was originally supplied by Molecular Products to ORNL in an engineered pelletized form (Ionex-Type Ag 900 E16). It contains ~9.5% silver by weight and has a 1/16-in. pellet diameter. Before CH_3-I exposure, it was treated at ORNL by heating at 150°C under H_2 flow (3% in N_2) for ~12 hours to reduce the Ag^+ to the metallic state.[1]

At INL, the samples were prepared during Test CH_3I-5 (nomenclature for CH_3-I loaded Ag-MOR, Test 5). A dry air stream containing an average of 31 ppm CH_3I was passed through four fixed bed segments of AgZ sorbent at a temperature of 150°C, with cumulative adsorption time of 435 hours over all the beds; note, though this loading process was similar to that reported earlier,[10] the major difference is that these samples were prepared with no NO_x or H_2O added to the stream. Based on the total measured iodine decontamination factors (DFs) for each bed segment, the sorbent in beds 1-3 approached practical saturation levels for the test conditions, with DFs ranging between about 1.0 (no iodine capture) in Bed 1 to about 2.5 (low iodine capture) in Bed 3.[11] Bed 4, with DFs ranging about 40, was still capturing most of the iodine that exited Bed 3, and was not yet near saturation.

Immediately following the adsorption test period, dry air without any CH_3I was passed through the beds for 37 hours to desorb any weakly physisorbed species. During this purge period, the concentrations of CH_3I and I_2 in the gas stream exiting Bed 1 (the most saturated of the beds) decreased by about one order-of-magnitude, from about 0.1-0.2 ppmv each to about 0.01-0.02 ppmv each, and the cumulative loss of total iodine asymptotically approached about 0.6% of the total iodine loaded on that bed. *INL samples sent to SNL were from the most-nearly-saturated test bed segments 1-3 from Test CH_3I-5.*

The low-sintering temperature glass used in this work was a Bi–Si-oxide glass that is available commercially (3 µm average particle size, a density of 5.8 g/ cm^3, coefficient of thermal expansion of 7.8 x 10^{-6}/°C, from Ferro Corp., Cleveland, OH).[6] The glass has a composition of: 7.8 wt% ZnO, 63.4 wt% Bi_2O_3, 5.4 wt% Al_2O_3, 23.4 wt% SiO_2. The glass sinters to essentially full density after only 15 min at 550°C and does not crystallize during the

sintering process. The silver material that was added was <10 μm Ag flake (Aldrich, Milwaukee, WI).

GCM Waste Form Preparation:

A portion of the as-received INL AgI-MOR samples, respectively, were ground to <150 μm using a mortar and pestle to form a homogeneous powder. These materials were characterized as-received and with varying amounts of Ag flake. The Ag flake was mixed with the ground AgI-MOR and DI water and allowed to air dry. Some of this material was used to prepare GCM waste forms by mixing the AgI-MOR and Ag flake with the glass powder in DI water. The mixtures were 20 wt% AgI-MOR and 80 wt% glass.[5,6,8] After air drying, the mixtures were pressed in a steel pressing die at 5000 psi to form small pellets ("preforms").

Characterization:

The as-received materials were examined using powder X-ray diffraction (XRD, Siemens Kristalloflex D 500 diffractometer, Bruker-AXS Inc., Madison, WI) and X-ray fluorescence (XRF, Bruker Micro-XRF,). Post-TGA AgI-MOR was also analyzed using XRF.

Simultaneous thermo gravimetric analysis and differential scanning calorimetry (TGA/DSC, SDTQ600, TA Instruments, Newcastle, DE) with mass spectroscopic (MS, Thermo Star™, Pheiffer Vacuum, Inc., Asslar, Germany) off-gas analysis was performed on powder samples of the as-received AgI-MOR materials, the powdered as-received AgI-MOR materials with added Ag flake, and on small portions of the pressed GCM samples with various amounts of added Ag flake. The experiments designed to determine the minimum Ag additions were performed on small pieces of the preforms that were heated in air at 5°C/min heating rate up to 550°C for a 1 hr soak, since this is the thermal treatment used to densify the GCMs. Follow-on heating studies of the preforms in inert atmosphere (N_2) were also performed.

4. RESULTS AND DISCUSSION

4.1 Characterization of As-Received Materials

The powder XRD patterns of the three as-received AgI-MOR materials after grinding are shown in Fig. 1. All three materials had diffraction peaks for mordenite and AgI. Bed 3 also had small Ag metal peaks. When the peak heights of the two most intense peaks for AgI and for MOR are ratioed, the results are similar for Beds 1 and 2 while Bed 3 has a significantly lower

ratio. Based on the XRD data therefore, Beds 1 and 2 are similar in their crystalline AgI contents and do not contain any detectable crystalline Ag, whereas Bed 3 has less crystalline AgI and a small amount of metallic Ag. This AgI concentration change between beds 1 and 2, versus 3 is confirmed by preliminary Reitveld refinement of the XRD powder patterns; as peak heights correlate to concentration in XRD, the reduction in AgI relative to MOR in Bed 3 compared to the other beds confirms a smaller iodine loading in that sample bed.

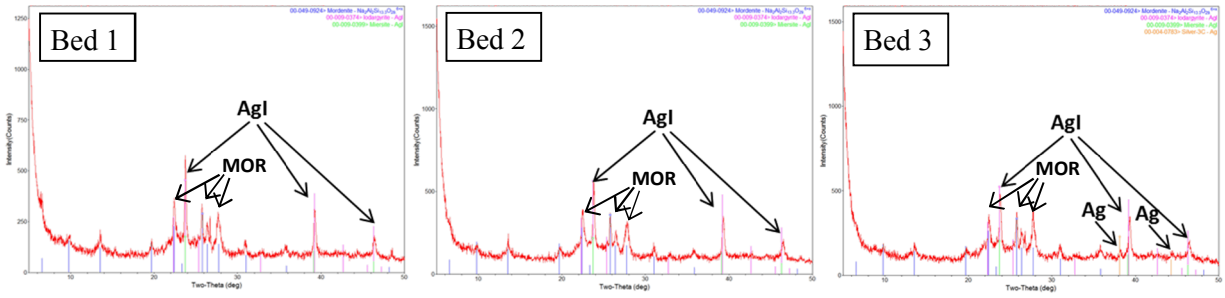

Figure 1. Powder XRD patterns for the three AgI-MOR samples received from INL (CH_3-I loaded Ag-MOR).

The XRF results for the three as-received materials after grinding indicated that Beds 1 and 2 had the same iodine content (13.32 wt% I) whereas Bed 3 had 12.05 wt% I (~9.8 wt% less iodine) than Beds 1 and 2. The Ag contents found by XRF were 37.7 wt% for Bed 1, 36.5 wt% for Bed 2 and 35.3 wt% for Bed 3. The XRF data is semi-quantitative as no calibration standards were used but is useful for comparison studies between samples. Also, the XRF analysis did not include oxygen or other light elements so the actual iodine weight percent content is expected to be less than the values reported. In comparison, SNL received the results of EDS elemental analyses performed at INL on these materials from Nick Solberg; these results reported iodine contents for Beds 1, 2 and 3 to be 6.94 wt%, 8.15 wt% and 7.27 wt%, respectively (including oxygen content in the analysis). The Ag content found by EDS was 11.6 wt% for Beds 1 and 3 and 12.05 wt% for Bed 2. While both the XRF and EDS analyses indicate that all three samples had relatively similar levels of I and Ag and that the Ag content was significantly higher than the I content on a molar basis, the discrepancies in the data suggests that more work is needed to determine the most accurate analysis technique – XRF, EDS or possibly some other technique.

The ground (<150 µm) AgI-MOR materials were analyzed using simultaneous TGA/DSC with MS off-gas analysis. The thermal cycle used for the TGA runs was the same as that typically used to thermally process the GCMs: 5°C/min to 550°C for 1 hr in air. The purpose of the TGA/MS analysis is to determine the amount of iodine that escapes during heating from the

mass loss data along with the MS signal for iodine species. Fig. 2 shows the TGA (in black) and iodine-species MS signals (I^- with ME = 127 in pink and I_2^- with ME = 254 in blue) for Bed 1 material. In Fig. 2a the data are plotted with temperature along the x-axis and in Fig. 2b with time along the x-axis so that the data during the 1 hr hold can more clearly be seen. The MS signal for water, OH, and CO_2 are also shown. Iodine-species were detected in the off-gas starting at 353°C, where an increase in the slope of the TGA curve also occurred corresponding to the loss of iodine. This occurs in spite of the fact that the material contains excess Ag able to react with all of the I present. The peak in the MS iodine signals occurs at 470°C corresponding to the maximum TGA slope. By the end of the hold at 550°C, the iodine signals are essentially back to baseline levels and the TGA curve is flat indicating that no more I_2 is evolving. The mass lost below 353°C is due to water and carbon dioxide from the methyl iodide. The total mass lost was 11.6%, of which 7.5% occurred before 353°C. Therefore, the mass loss due to the evolution of iodine was 4.1% at most, since some of the loss above 353°C could be due to water or other species.

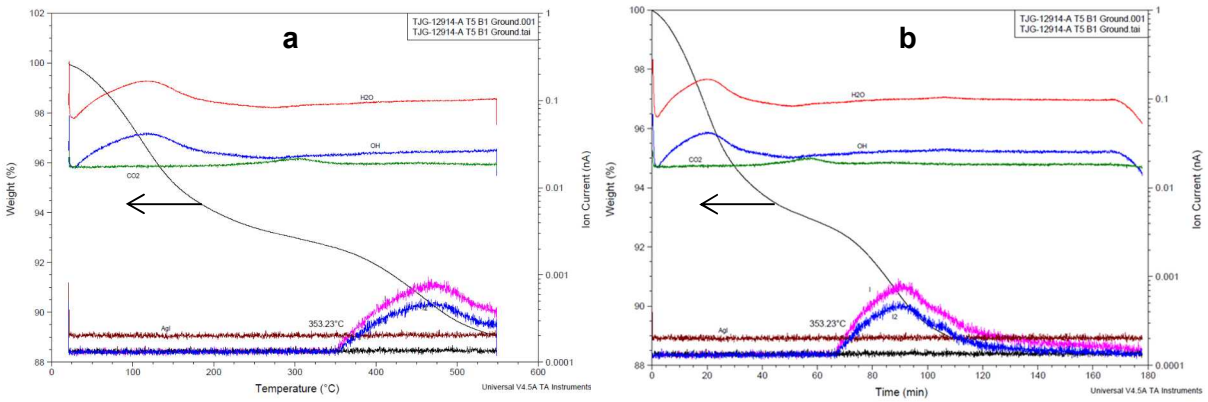

Figure 2. TGA with off-gas MS for as-received ground INL (Test 5 Bed 1) AgI-MOR as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

Fig. 3 shows the TGA-MS data for Test 5 Bed 2 material. The results are very similar to Bed 1. In this case, the iodine is first detected at 333°C and the amount of mass loss attributable to iodine is about 4.3%. For Bed 3, as shown in Fig. 4, the results are also similar but with the first iodine coming off at the higher temperature of 370°C and with a slightly smaller mass attributable to iodine of 3.3%. XRF on post-TGA Bed 3 powder confirmed that the I content was much lower than in the starting material (6.6 wt% as opposed to 12.05 wt% indicating about 45% of the iodine left).

For comparison, the data versus temperature for all three samples are plotted together in Fig. 5. In summary, these data indicate that ~50% of the iodine present escapes during heating in air to 550°C for 1 hr starting around 350°C and that Bed 3 loses 25% less iodine than Beds 1 and 2, respectively. *This information will be used to estimate the amount of additional silver necessary to keep any iodine from escaping the GCMs during thermal treatment.*

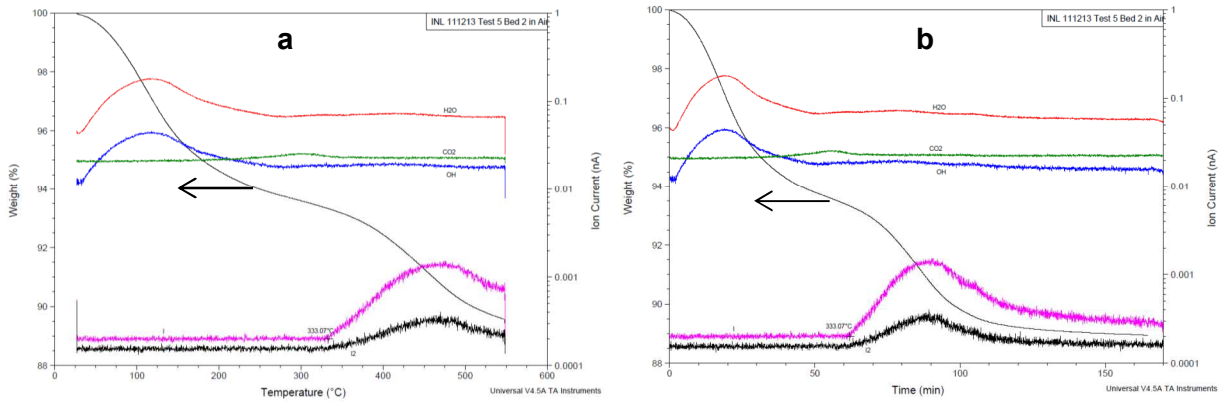

Figure 3. TGA with off-gas MS for as-received ground INL (Test 5 Bed 2 AgI-MOR) as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

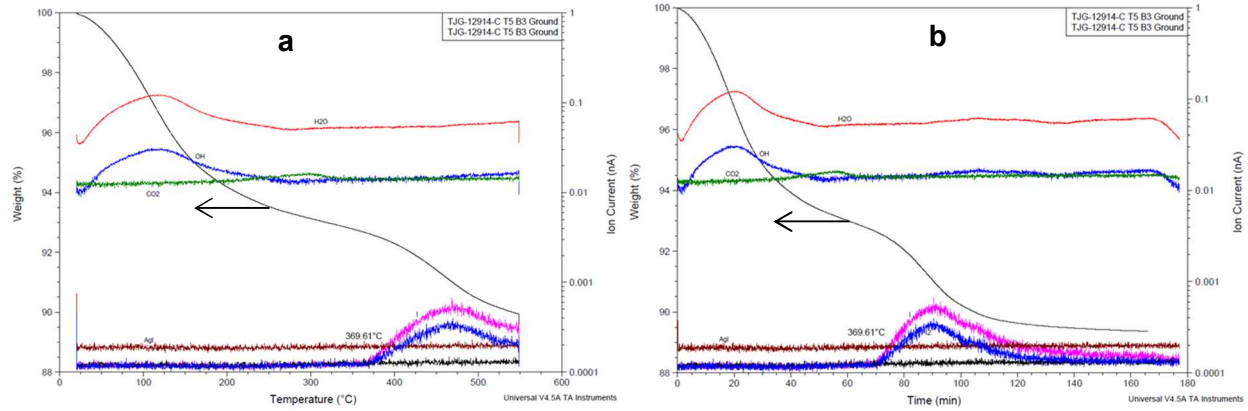

Figure 4. TGA with off-gas MS for as-received ground INL (Test 5 Bed 3) AgI-MOR as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

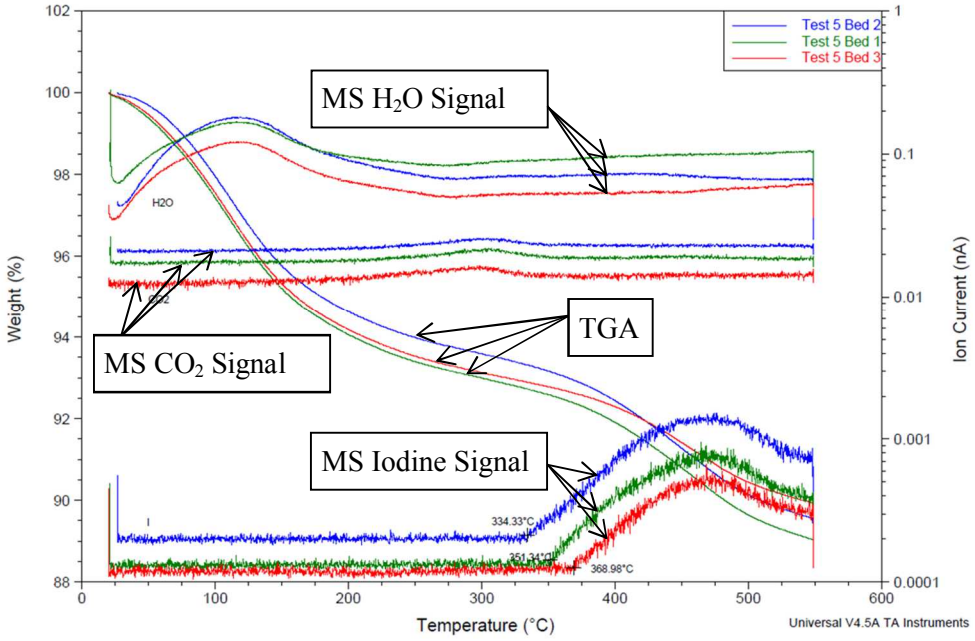

Figure 5. Comparison of the TGA and off-gas MS data for the three INL AgI-MOR samples (Test 5 Bed 1 is green, Test 5 Bed 2 is blue and Test 5 Bed 3 is red).

4.2 Silver Addition Experiments

Calculations based on the amount of iodine lost from the original/as-received AgI-MOR materials give a starting point for the amount of Ag flake that will need to be added to the GCM to suppress iodine loss during thermal processing. The mass of iodine lost is simply converted to moles of iodine and then the mass of an equal number of moles of silver is calculated. For Bed 2, ~4.3% of the mass of the original AgI-MOR sample was lost as iodine. Therefore, based on the atomic masses of I and Ag, 3.6 wt% of Ag needs to be added to react with the escaping iodine. This assumes that all of the added Ag will be utilized.

Fig. 6 shows the TGA/MS results for a GCM preform with 20 wt% of Bed 2 AgI-MOR and 80 wt% glass with additional Ag added at the level of 4.25 wt% of the mass of the AgI-MOR or 0.85 wt% of the mass of the GCM. As shown in Fig. 6a, some iodine is released from the sample, although starting at a higher temperature of 525°C and a much smaller amount (the green line is the I^- signal in Fig. 6a and the pink line in Fig. 6b). This indicates that not all of the added Ag is being utilized, as this level of added Ag is above the 3.6% calculated above based on the iodine mass loss from the AgI-MOR. Silver utilization could potentially be improved by

modifying the size and/or shape of the Ag particles or the heating schedule. These are areas for future research.

Additional GCM preform compositions were then prepared and ran in the TGA in order to bracket the amount of Ag needed to prevent iodine loss for each of the three INL Test 5 samples.

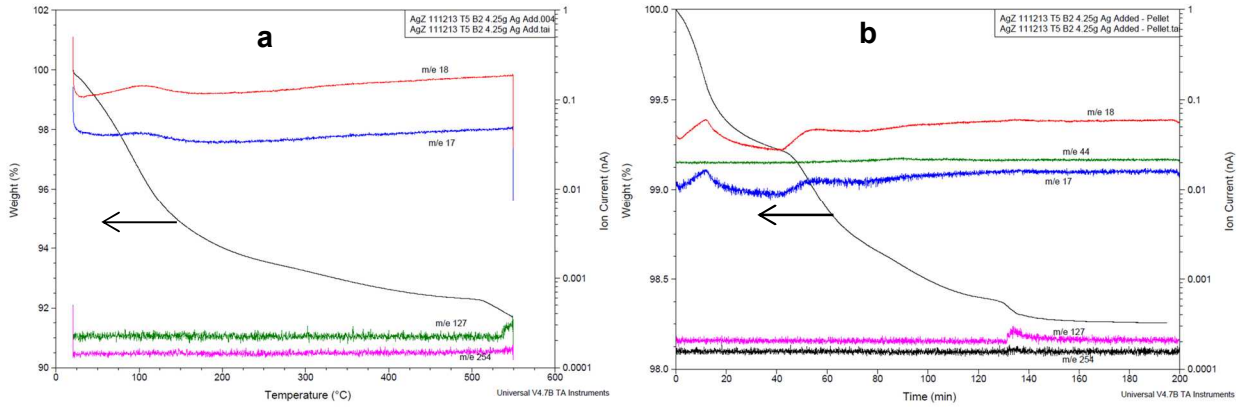

Figure 6. TGA with off-gas MS for a 20 wt% INL Test 5 Bed 2 AgI-MOR GCM with 0.85 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

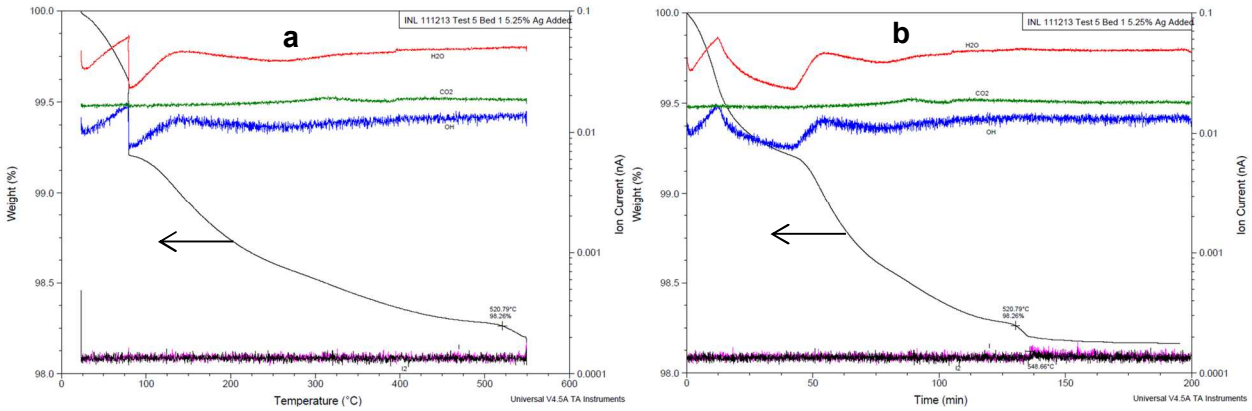

Figure 7. TGA with off-gas MS for ground a 20 wt% INL Test 5 Bed 1 AgI-MOR GCM with 1.05 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

Fig 7 shows the TGA/MS results for a GCM preform made with 20 wt% of Bed 1 AgI-MOR with 1.05 wt% of added Ag using the total GCM mass as a basis (or 5.25% of the mass of the AgI-MOR). Fig. 7a shows that no iodine species are detected in the off-gas during the ramp to 550°C. However, Fig. 7b shows that a small amount of iodine comes off starting at the beginning of the isothermal hold (the pink line). Fig. 8 shows that for a sample with 1.10 wt%

added Ag flake to a 20 wt% Bed 1 GCM, no iodine is detected in the off-gas during both the temperature ramp and the isothermal hold. *Therefore, 1.1 wt% of Ag (based on the total GCM mass) must be added for GCMs with 20 wt% AgI-MOR from Test 5 Bed 1 to prevent any iodine loss during thermal processing.*

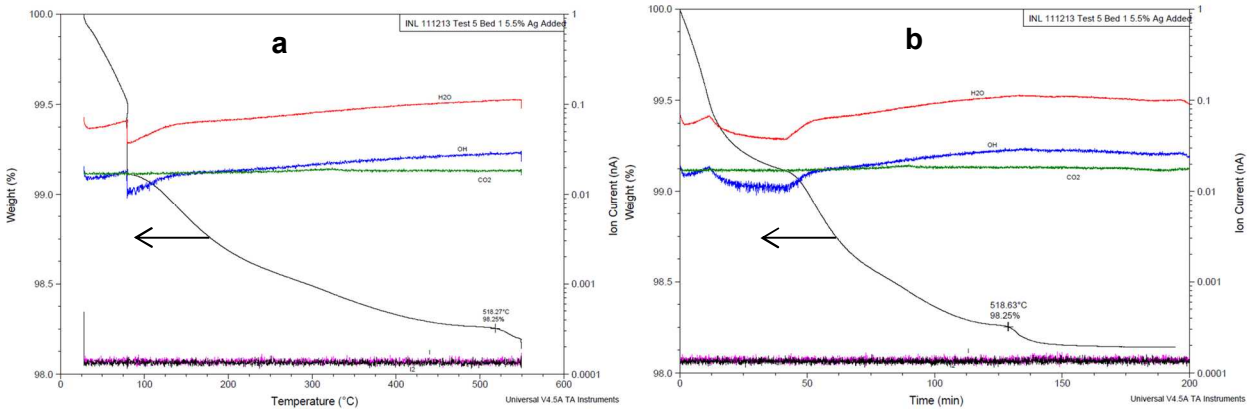

Figure 8. TGA with off-gas MS for a 20 wt% INL Test 5 Bed 1 AgI-MOR GCM with 1.1 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

The same procedure was used to determine the minimum amount of Ag needed for Beds 2 and 3. Fig. 9 shows that for a Bed 2 GCM with 1.1 wt% added Ag flake, a small amount of iodine comes off during the hold whereas for a 1.15 wt added Ag GCM, no iodine comes off as shown in Fig. 10. Fig. 11 shows that for a Bed 3 GCM with 1.15 wt% added Ag flake, a very small amount of iodine comes off during the hold whereas for a 1.20 wt% added Ag GCM, no iodine comes off as shown in Fig. 12.

In summary, the amount of Ag flake needed to prevent iodine loss in GMCs with 20 wt% of the Test 5 AgI-MOR materials is 1.10 wt% for Bed 1, 1.15 wt% for Bed 2 and 1.20 wt% for Bed 3. Expressed as a percent of the mass of just the AgI-MOR the values are 5.50 wt% for Bed 1, 5.75 wt% for Bed 2 and 6.00 wt% for Bed 3. Interestingly, even though Bed 3 had less mass loss attributable to iodine than the others based on the TGA/MS results on the as-received materials, it required the most additional Ag to prevent iodine loss from a GCM preform, albeit by only a small amount. This implies that Bed 3 AgI-MOR did not utilized the added Ag to the same extent as did the others. More work is needed to further understand this result.

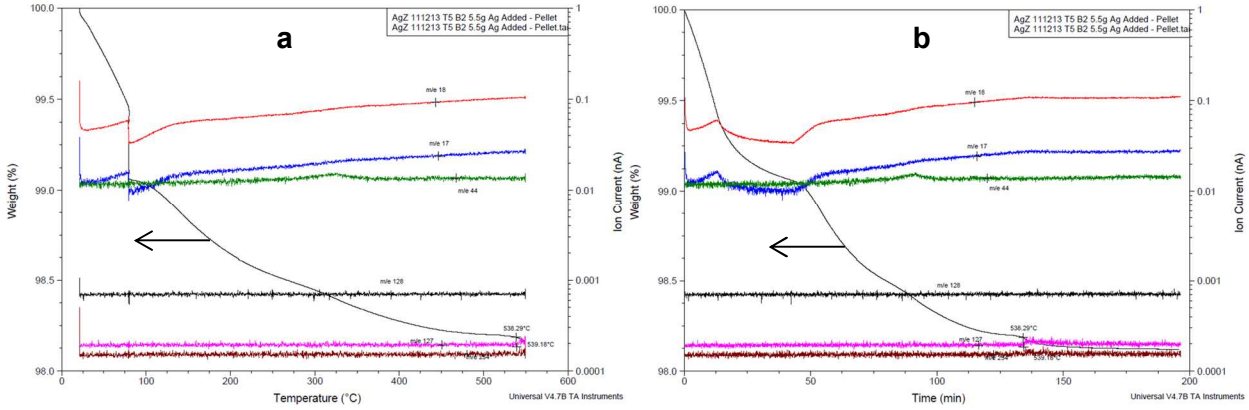

Figure 9. TGA with off-gas MS for a 20 wt% INL Test 5 Bed 2 AgI-MOR GCM with 1.1 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

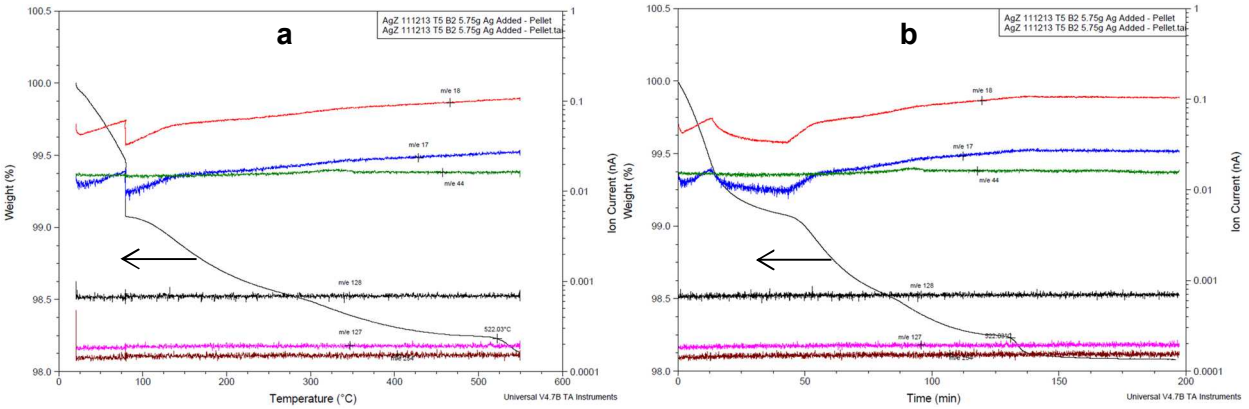

Figure 10. TGA with off-gas MS for a 20 wt% INL Test 5 Bed 2 AgI-MOR GCM with 1.15 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

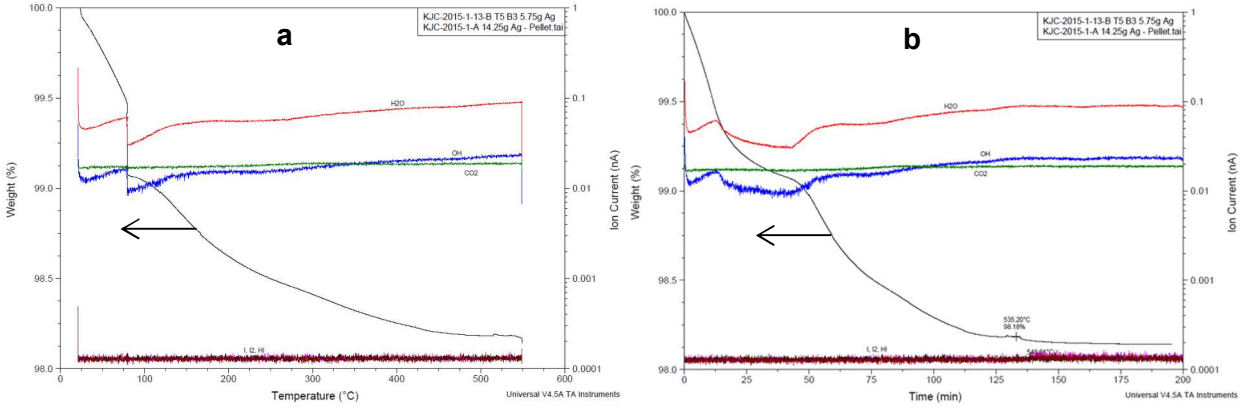

Figure 11. TGA with off-gas MS a 20 wt% INL Test 5 Bed 3 AgI-MOR GCM with 1.15 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

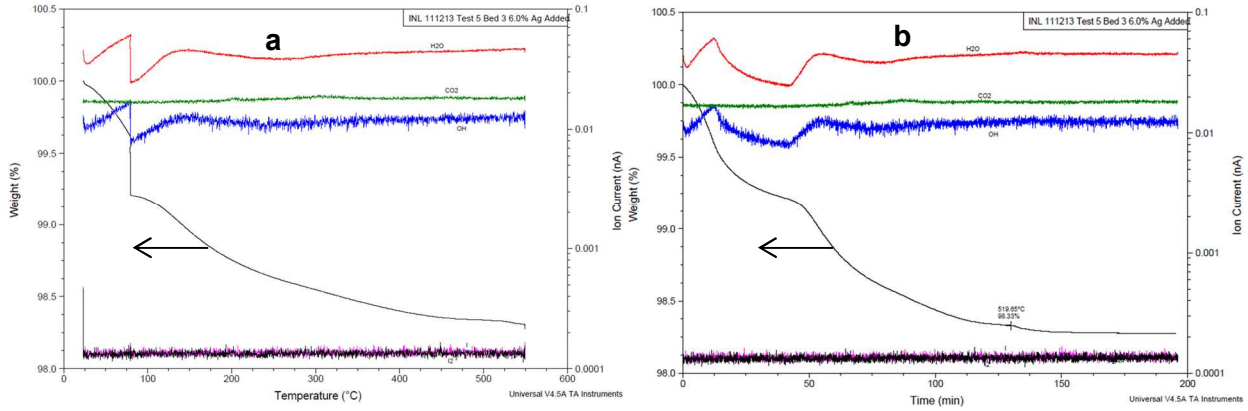

Figure 12. TGA with off-gas MS for a 20 wt% INL Test 5 Bed 3 AgI-MOR GCM with 1.2 wt% of added Ag flake as a function of (a) temperature and (b) time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

Fig. 13 shows the TGA/MS results with time on the x-axis for a sample of ground Bed 2 powder that was mixed with 6.25 wt% Ag flake, an amount greater than the 5.75 wt% (based on the AgI-MOR mass) needed to prevent iodine loss in the GCM. As the figure shows, a small amount of iodine is detected leaving the sample at the start of the isothermal hold. This suggests that the evolving iodine reacts to a greater extent with the added Ag when in a GCM compacted preform as opposed to a loose powder leading to a slightly higher Ag utilization. Therefore the experiments to determine the *amount of Ag needed must be conducted on GCM preforms* and not just on the AgI-MOR powders in order to find the correct value.

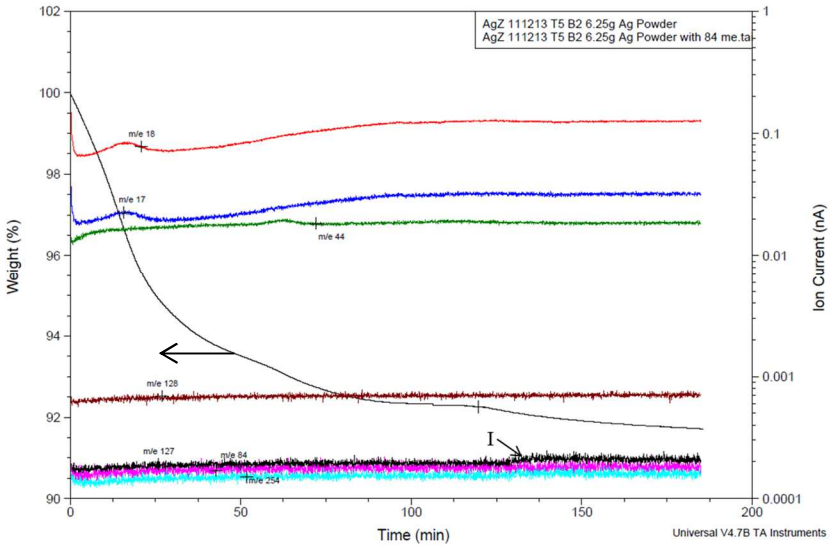

Figure 13. TGA with off-gas MS for as-received ground INL Test 5 Bed 2 AgI-MOR with 6.25 wt% of added Ag flake as a function of time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold.

4.3 Effect of Atmosphere on Iodine Loss

In previous work on I_2-loaded AgI-MOR materials,[9] we found that the amount of iodine loss was less (or none) when the thermal processing was done under an inert gas as opposed to air. We theorized that heating in air oxidized the un-reacted silver in the MOR that was previously prevented from reacting with the I_2; by contrast heating in inert gas avoided silver oxidation and therefore the evolving I_2 was able to react to form AgI instead of off-gassing. Therefore we ran TGA/MS on the Test 5 samples in nitrogen (ground powders with no added Ag, not GCM preforms). The results are shown in Fig. 14 with Bed 1 in Fig. 14a, Bed 2 in Fig 14b and Bed 3 in Fig 14c.

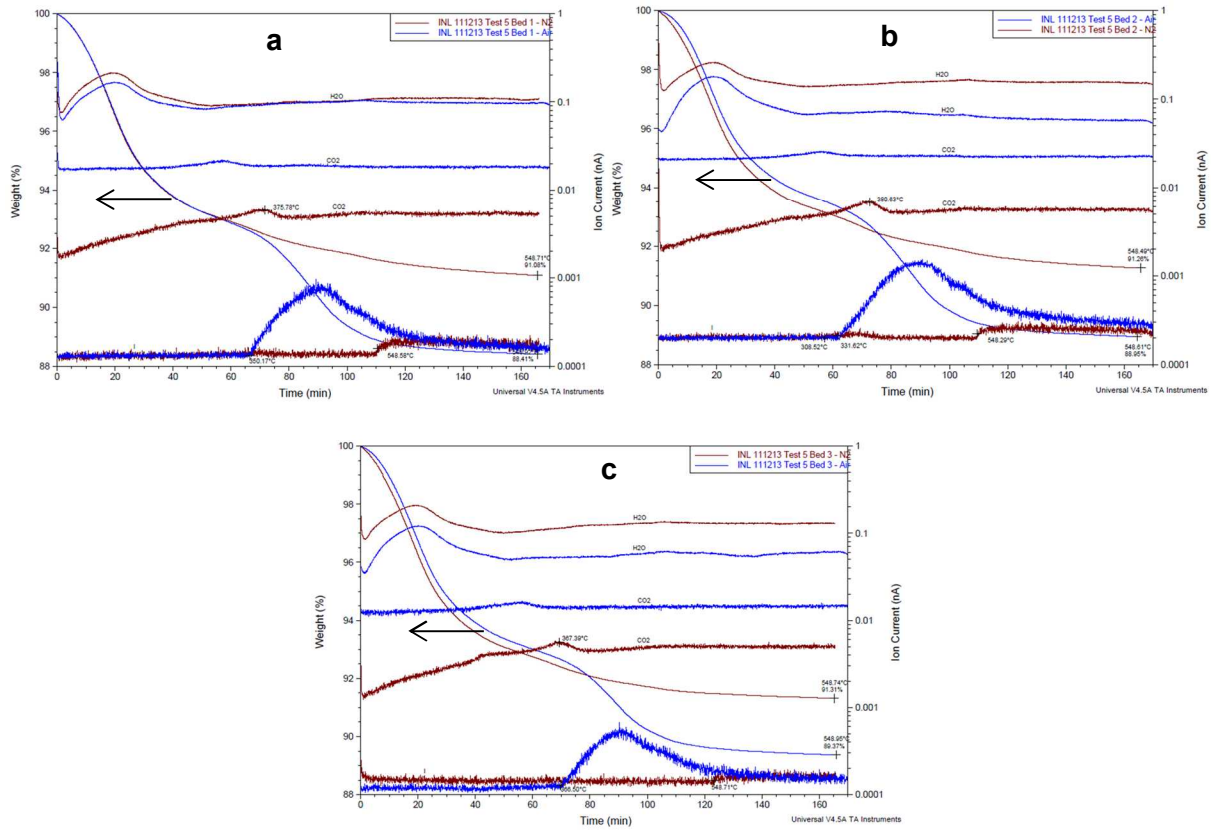

Figure 14. TGA with off-gas MS for as-received ground INL Test 5 (a) Bed 1, (b) Bed 2 and (c) Bed 3 AgI-MOR of time during a heating cycle in air of 5°C/min to 550°C followed by a 1 hr hold in N_2 (brown) and air (blue).

The results for the runs in N_2 are in brown; the results in air (seen in blue) are shown for comparison. For all three samples, the mass loss and the amount of iodine detected in the off-gas

were significantly less for the N_2 runs than for the air runs. The decrease in mass loss, which is attributable to less iodine loss, is 2.67% for Bed 1, 2.31% for Bed 2 and 1.94% for Bed 3. Even though significantly more of the silver in the mordenite is utilized when heated in nitrogen, some must remain unreacted since there is more Ag (atomic basis) present in the material than I, yet some iodine escapes for all three materials. XRF analysis on Bed 3 material after heating in nitrogen showed the iodine content was 10.5 wt%, which is a loss of ~13% of the iodine originally present, yet much less than the 45% lost during heating in air. *Overall, the results indicate that if the GCMs were to be processed in inert gas, a significantly lower amount of silver would need to be added than was found here for when they were processed in air.*

5. CONCLUSIONS

Sandia-developed low temperature sintering glasses are being examined for the encapsulation of a variety of fission gas loaded getter materials. In particular, we are focusing on the incorporation of silver mordenite iodine capture materials (AgI-MOR) to form Glass Composite Materials (GCMs), for subsequent use as waste forms. The AgI-MOR materials used in this study were methyl iodide loaded Ag-MOR, supplied by INL, Test 5 (Beds 1, 2 and 3). The optimization of Ag flake amounts needed in GCMs were determined for all three materials.

Experiments utilizing simultaneous TGA and DSC with MS off-gas analysis found that for Bed 1, 1.10 wt% of additional Ag flake must be added to the GCM (with 20 wt% AgI-MOR and 80 wt% glass) to suppress iodine loss during sintering (when processing in air) by heating at 5°C/min to 550°C for 1 hr. The amounts for Beds 2 and 3 are 1.15 and 1.20 % Ag, respectively. These amounts are slightly higher than the amounts predicted from the mass loss of iodine when the AgI-MORs are heated alone in air. These results indicate one of two things: (1) a portion of both the added Ag flake and the excess crystalline surface Ag are being oxidized to Ag^+ and migrating into the MOR pores facilitated by the presence of oxygen during heating, and/or (2) the excess Ag is being oxidized to AgO, a non-iodine-reactive nanoparticle both inside MOR pores or on bulk surface.[12]

In contrast, when the GCM is sintered in inert atmosphere these initially present unreacted Ag particles do not reoxidize to the same extent as when the GCM is sintered in air. Therefore, much of the Ag metal is able to react with the desorbing iodine vapor to form AgI. As a result, a

GCM sintered in inert atmosphere requires a much lower amount of Ag flake to suppress iodine loss than when air sintered.

This study also showed that the desification process in the formation of the GCM (in air, where Ag flake addition is necessary) results in the trappling of a small amount of the iodine. This means that less Ag flake addition is required in the GCM waste form than if the glass were not present. Both of these results are similar to what we found previously for I_2 loaded AgI-MOR.[9]

A number of areas for ongoing and future studies have been identified. The first area is determine the most accurate method of (a) elemental analysis of the AgI-MOR materials for I and Ag content and (b) spectroscopy, possibly Raman, to identify that will determine how the iodine is partitioned between the AgI and adsorbed material. The second area of study is to test the effects of silver morphology on the amount of additional silver added. The third area identified is related to inert atmosphere sintering of the GMCs, as well as determining the maximum level of oxygen allowable in the gas so as to prevent Ag metal oxidation.

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