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Monitoring Volatilization Products Using Residual Gas Analyzers

During MeV Ion Beam Irradiations

ABSTRACT

 The use of Residual Gas Analyzers (RGAs) during irradiation experiments can provide valuable information when incorporated into experimental end-stations. The instruments can track the volatilization products of beam-sensitive materials, which may ultimately aid researchers in selecting appropriate flux values for conducting experiments. Furthermore, the type of gaseous species released during an irradiation can be monitored directly, which may lead to new insights into the radiolysis and/or heating mechanisms responsible for gas evolution. A survey of several classes of materials exposed to extremes in particle flux is presented to show how RGA instrumentation can be incorporated to qualitatively assess ion-solid interactions in a variety of fields.

# INTRODUCTION

Common to many modern vacuum and accelerator systems are Residual Gas Analyzers (RGAs). RGAs are mass spectrometers used for monitoring vacuum conditions and are especially useful for identifying leaks. For instance, in a typical vacuum system operating in the 10-8 Torr range, an RGA readily measures the ~10-9 partial pressures of air, water, and oils, and is readily configured into a leak detection mode for helium measurement. However, if an RGA is appropriately designed into experimental end-stations, it can become a useful instrument for measuring gaseous species that may become volatilized due to ionization and beam heating during irradiation experiments. Two possible applications for RGAs in irradiation experiments include determining the proper beam flux for conducting accelerated irradiation experiments, and direct measurement of radiolysis (ionization) effects in materials.

Accelerated irradiation studies are experimental irradiations performed at dose rates in excess of the dose rates associated with the physical phenomena they are meant to simulate: such as in nuclear reactors, radioactive waste storage, and space weathering (Bennett et al., 2013; Mazey, 1990; Weber et al., 1991). Whereas the energy and fluence in irradiation experiments are typically predetermined, the flux is often not. Many experimentalists overlook this crucial variable, as the flux, or beam current, is oftentimes determined by what the equipment is capable of outputting on any given day. Researchers benefit from stable, high-current irradiations as they can save valuable time over the course of a long irradiation campaign, particularly when considering experiments lasting days and weeks. Furthermore, it is easy to reconcile the economics of an irradiation that could be performed over the course of eighty hours, or *tweaking* a couple of knobs in order to complete the experiment in one long working day.

Most ion irradiation experiments typically use keV or MeV beams with nanoamp to microamp beam currents, resulting in fractions of a watt-like energy deposition. Even these seemingly innocuous wattages can elevate the temperature in the irradiated region, as the volume of energy deposition is small; ion ranges are typically on the order of nanometers to tens of microns in depth for most materials. Limiting the temperature gain in the target is critical, as elevated temperatures may result in deleterious effects such as volatilization and undesired defect recombination or dynamic annealing (Goldberg et al., 1995; Kuznetsov et al., 2003; Pelaz et al., 2004). This limits the flux of particles which can be used without considering extensive mechanisms for heat removal such as liquid interfaces and other cooling schemes during irradiation (Naab et al., 2011).

In addition to tracking the role of beam flux during an irradiation, the RGA can be used to understand radiation degradation and radiolysis in materials. For instance, the technique has been used to simulate α-particle exposure in polymers that come into close contact with actinides (Fisher et al., 2006; Pugmire et al., 2009; Wetteland et al., 2006). A novel analogous example is understanding the effects of high-energy protons from solar flares on precursor planetary material. Stars such as our Sun were far more active proton emitters during their early evolution. It is possible that chemical alteration of primitive solar material could be simulated in experiments similar to those above, with chemicals released from the target material being tracked with the RGA.

In this report, we present several examples of ion irradiation experiments wherein we used an RGA to monitor radiolysis effects and target volatilization. Our examples involve multiple classes of materials.

# EXPERIMENTAL

 A series of ion beam irradiations were performed using 2 MeV H+ ions (University of Wisconsin-UW) and 5.5 MeV He++ ions (Los Alamos National Laboratory -LANL). Figure 1 shows a typical experimental setup. A Stanford Research Systems (SRS) 300 amu (atomic mass unit) RGA was connected in series with the end-station and the accelerator beam line for the LANL experiments. Twin 70 *l/s* turbo pumps are adjacent to the ionizing filament as can be observed in detail 10 of the figure. For the experiments conducted at UW, the RGA was mounted on the side of the end-station using a single 70 *l/s* pump adjacent to the filament. In both configurations, some portion of the volatilized products are pulled past the RGA filament due to the local pressure gradient created by the turbo pumps. The SRS RGA has manufacturer detection limits of 5x10-11 Torr using the instrument’s Faraday cup, and 5x10-14 when the optional electron multiplier is used. The maximum operating pressures for the Faraday cup and electron multiplier

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| Figure 1 An experimental end-station capable of performing both traditional ion beam analysis and RGA studies. Volatile products generated on the target located at (1) are pulled in an opposite direction to the beam towards the turbo pumps for the RGA and beamline; the aperture at (8) can be used to restrict loss to the beamline. To assure all gasses generated from exposure were measured by the RGA, a beam transparent window could be inserted at (8) to isolate the chamber completely from the remainder of the accelerator, increasing the sensitivity on the spectrometer.  |

are 10-4 and 10-6 Torr respectively. The instrument is capable of monitoring up to 10 different amu species simultaneously, with the data being output in multiple pressure-versus-time configurations. The data presented in this report typically entails only one or two of the volatilized products for a given sample, and is displayed with the partial pressure (Torr) on the ordinate, with time on the abscissa. It should be noted that the mass filter selects species based on their mass over charge ratio (*m/q*), and that data displayed represents a major element in the target. The targets were mechanically mounted on an electrically isolated, un-biased stainless steel stage, with charge collected using a Brookhaven Instruments Corporation (BIC) current integrator. The current measured on the isolated stage agreed well with a pre-chamber Faraday cup.

 Materials examined include: (1) a polished slab of Portland cement and (2) a zone refined nickel slab, each irradiated with un-scanned 2 MeV H+ ions to simulate neutron exposure in a reactor environment; (3) a Kapton® (polyimide) film irradiated with scanned 5.5 MeV He++ ions to simulate α-particle exposure from an actinide; and (4) a sulfide mineral irradiated with un-scanned 2 MeV H+ ions to simulate meteorite precursor material exposed to solar flare particles during stellar evolution. Post irradiation analysis techniques

employed in this study include scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDXS) on the sulfide mineral using a Phenom Pro-X SEM, and Thermal Gravimetric Analysis (TGA) on the cement using a TA Instruments Q500. Experimental irradiation conditions are summarized in Table 1.

Table 1 Target details and experimental irradiation conditions.

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| --- | --- | --- | --- | --- | --- | --- |
| **Material** | **Target Geometry** | **Ion & Energy** | **Facility** | **Irradiation** **Area** | **Beam** **Current** | **Flux/Fluence** |
| Portland Cement | Polished Slab 48 mm2 x 4 mm thick | 2 MeV H+ | UW | 0.04 cm2 | 30 nA60 nA | 4.7x1012 H+/cm2-s9.4x1012 H+/cm2-s |
|  |  |  |  |  |  |  |
| Nickel | Zone Refined Boule150 mm2 x 0.7 mm thick | 2 MeV H+ | UW | 0.1 cm2 | 1 µA6 µA8 µA10 µA | 6.2x1013 H+/cm2-s3.7x1014 H+/cm2-s5.0x1014 H+/cm2-s6.2x1014 H+/cm2-s |
| Kapton© HN | Film100 mm2 x 0.13 mm thick | 5.5 MeV He++ | LANL | 1 cm2 (0.09 cm2 Beam Spot) | 25 nA | (3) 2.7x1013 He++/cm2 |
|  |  |  |  |  |  |  |
| FeS2 | Irregular Mineral Slab ~ 64 mm2 x 2 mm thick | 2 MeV H+ | UW | 0.1 cm2 | 1 µA1.5 µA | 6.2x1013 H+/cm2-s9.5x1013 H+/cm2-s |
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# RESULTS and DISCUSSION

## III.a Cement Example

Figure 2 shows an RGA mass scan for microcrystalline cement irradiated with 2 MeV protons using two different ion fluxes. The initial background pressure of water for the cement is over two orders of magnitude higher than the other materials used in this study. Figure 2 shows that a flux of 4.7x1012 H+/cm2-s was applied at an elapsed time of 15 seconds and stopped after a minute of irradiation. A second irradiation was performed using a flux of 9.4x1012 H+/cm2-s and started at an elapsed time of 120 seconds, and stopped 140 seconds later. The irradiation was performed over a 0.04 cm2 area with respective beam currents of 30 and 60 nA.

A considerable difference in water loss is evident between the two flux values. Understanding and limiting water loss should be critical in performing accelerated aging studies in cement. Both radiolysis and heating effects during irradiation may result in water loss and subsequent mechanical property degradation of concrete structures (Field et al., 2015; Le Pape et al., 2015).

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| Figure 2. Water (18 amu) release from microcrystalline Portland cement detailing the effect of beam current on the overall change in partial pressure. An approximate 5% change in water vapor is detected by the RGA when the slab is irradiated using 30 nA, while a ~70% increase is detected when twice the beam current is used.  |

 Gas generation in the case of cement via radiolysis is accomplished by the dissociation of water and the subsequent formation of liberated molecular species. Limited literature exists which describes the radiolysis of cement. However, a specific concern in these scenarios is the buildup and explosion hazard of H2 gas generated from radioactive materials sealed in cement casks. Siskind (1992) determined that H2 and H2O are generated by the radiolysis of trapped pore water in the cement structure. Bibler and Orebaugh (1976) examined beta particle irradiation in concrete from tritiated water and found that a steady state H2 production was reached which is directly related to the intensity of irradiation.

Common to these studies is the quantification of gas generation though use of the *G value*. The *G* *value* is defined as the number of molecules generated when a substance absorbs 100 eV of energy. In the above studies, the gasses were confined and measured from a closed system. As only a portion of gasses generated in this study were measured by the RGA, it is not possible to report actual *G* values.

Water loss can additionally occur during radiation induced heating of the cement. TGA was used to establish how weight loss in cement varies with increasing temperature. Figure 3 shows weight loss in Portland cement between 24 and 950 °C as measured with TGA. Data shows a relatively small decrease in weight occurs between room temperature and 105 °C, which correlates to the loss of adsorbed water. Beyond 105 °C, weight loss is from the decomposition of chemically bound water from the main hydration product of Portland cement: calcium-silicate-hydrate or C-S-H (Bazant and Kaplan, 1996).

 It is likely that both ionization and thermal mechanisms maybe responsible for water loss in cement/concrete systems. Discerning the relevant contribution of each mechanism is beyond the scope of this work. However, limited heating and ionization is expected in the case of neutron radiation damage in a reactor environment. Therefore, selecting a beam current that minimizes water loss as identified by the RGA may result in data that best reflects conditions required to perform accurate accelerated aging studies in concrete systems.

## III.b Metal Example

In contrast to cement, metals can be exposed to significantly higher fluxes of protons before measureable effects can be observed with the RGA. Figure 4 shows the partial pressures of masses (*q* equal to 1) 58 and 56 for a zone purified nickel target irradiated with 2 MeV H+ ions. The target was sequentially subjected to fluxes of 6.2x1013, 3.7x1014, 5.0x1014, and 6.2x1014 H+/cm2-s at elapsed times of 360, 660, 920, 1030 seconds. Figure 4 shows distinct changes in the mass 56 signal with changing flux, while minimal changes were detected from mass 58. The fluxes applied are equivalent to a 1, 6, 8, and 10 µA beam over a 0.1 cm2 area. The series of irradiations resulted in over an order of magnitude change in vapor pressure in the mass 56 signal during the course of the experiment. It should be noted that during the irradiation, the target transitioned from spectral emission in the orange to emission in the white. Visible emission in the orange of metals is associated with temperatures of approximately 900°C, while the white is approximately 1200°C.

Thin film techniques for vapor deposition of metallic elements use temperature-pressure curves (Honig and Kramer, 1969) to determine optimal conditions for vaporizing metallic targets. From such curves, it can be determined that the temperature predicted by the color change in nickel would result in a pressure change from ~7x10-8 to ~7x10-5 Torr for conditions similar to Honig and Kramer (1969). However, the data from Fig. 4 indicates that minimal detection of evolved nickel vapor (*m/q*=58) was observed over the course of the irradiation. What is directly correlated, is a regular change in the *m/q* equal to 56. It is plausible that the rise in 56 signal is from iron in the stainless steel holder that the nickel target was mounted on. Per the vapor curves, iron does exhibit a greater degree of volatility as compared to nickel. However, it is more likely that this experiment demonstrates the dangers of contamination and mass interferences in identifying liberated species during the irradiation event.

Several prominent hydrocarbon species exist in the ~50 amu range, one type being alkenes. Alkenes can be formed from the cracking of long-chained oil compounds ubiquitous in vacuum systems, with possible direct mass interference coming from the hydrocarbon C4H8. It is probable that the signal generated for mass 56 represents the fragmentation of oils on the target, target holder, and end-station as the temperature substantially increases over the irradiation. This particular mass interference would make it exceedingly difficult to measure iron volatilization in a similar measurement as performed here for nickel. Overall, the results indicate that the sensitivity of the instrument is unable to detect gaseous nickel over the range of experimental conditions investigated.

## III.c Polymer Example

 Kapton® is an aromatic polyimide film with a long history of use in radiation and vacuum environments due to its thermal stability and radiation tolerance (Hanks and Hamman, 1971; Megusar, 1997). A Kapton® film was exposed to three successive 2.7x1013 He++/cm2 irradiations (~5 MGy) using 5.5 MeV particles. Figure 5 shows that over the elapsed time of the irradiation, a reduction in evolved CO2 gas as a function of dose is observed. The oscillatory pattern of evolved CO2 gasis due to scanning the 25 nA, 0.09 cm2 beam, over a 1 cm2 area in order to achieve a uniform irradiation and to minimize target heating. The dip in the pattern represents a period during the scan where the beam was not hitting the polymer target, while the peak represents the entire beam on the film. Figure 5 indicates that during the first seconds of the irradiation, an approximate 40x increase in the partial pressure of mass 44 (CO2) was detected by the RGA. Over the course of the first irradiation, the partial pressure of CO2 was observed to decrease. This trend continues for the second dose, while the pressure during the third remains relatively unchanged over the course of the irradiation. The data also indicates that only ~2 minutes were required between each irradiation for the system to return to the background partial pressure of CO2.

The rapid detection of CO2, direct correlation between CO2 generation and scan pattern, and relatively low beam current scanned over a wide area, are all indicators that gas generation in the polymer is likely related to ionization effects rather than thermal heating. Literature results for the gaseous species generated in KAPTON® support this, and vary for the type of incident radiation. Hegazy et al. (1992) found that when using gamma-rays the dominant evolved species in decreasing order were CO2, CO, H2, and CH4. Lewis and Lee (1991) found that CO, H2, and CO2 were the dominant species when using 200 keV Si ions. In the case of the Si irradiation, the authors developed separate mechanisms for the formation of CO and CO2. Double scission of a carbonyl-amidic bonds results in the spontaneous formation of CO, while the formation of CO2 is morecomplex. Ionization results in the formation of an O-C=N radical; this is followed by double scission which leads to decarboxylation forming the CO2 molecule. The decrease of CO2 generated over the course of the irradiation described by Lewis and Lee is similar to the trend observed in Fig. 5. Overall, the results indicate that the RGA is sensitive to volatilization products formed during scenarios which simulate α-particle radiolysis in polymers.

## III.d Sulfide Example

Understanding mechanisms responsible for the formation of planets and the distribution of elements in the solar system can be investigated by studying meteorites. Meteorites can contain primitive assemblages of minerals, metals and amorphous phases, which represent various stages of cooling and condensation from a hot nebular gas. These may include high-temperature refractory minerals such as corundum (Al2O3) and forsterite (Mg2SiO4), coexisting with a suite of medium and low-temperature condensation minerals including iron/nickel metal and sulfides (FeS, and Fe1-xS). It may be that protons discharged in exaggerated solar flare events during stellar evolution could chemically alter these phases. Whereas the above experiments on nickel may be relevant to investigating some metallic phases in meteorites, additional 2 MeV proton irradiations were conducted on an FeS analog, pyrite (FeS2).

Figure 6 shows an RGA scan of mass 64, S2, from an irradiated polished slab of FeS2. At an elapsed time of 360 seconds, a flux 6.2x1013 H+/cm2-s (1 µA over 0.1 cm2) was applied to the target; no change in the RGA spectrum was observed. However, at 720 seconds the flux was increased to 9.5x1013 H+/cm2-s, and a significant change in the S2 signal was detected. The scan in Fig. 6 shows a steady increase in the S2 signal for approximately 720 seconds before it begins to level out. At approximately 1500 seconds, the signal exhibited a drastic increase again; in this case, there was no change in the beam current. At approximately 1600 seconds, the beam was turned off and the S2 signal decreased rapidly, then slowly decayed toward background over 1000 seconds before the measurement was stopped.

The sulfide example has similar behavior to the cement, as a threshold flux was required to generate volatilization products. Furthermore, the rapid approximately two order of magnitude change in S2 could be another indicator ionization is the dominant mechanism for gas generation. The sharp increase at 1500 secondswas likely the exposure of fresh FeS2 material to the beam due to a small portion of the surface spalling off. This was observed post irradiation with optical and backscattered electron SEM images as shown in Figs. 7a and 7b. An EDS line scan in Fig. 7c across the surface shows the relative concentrations of Fe and S. The irradiated area indicates sulfur loss, while the inner region reveals the FeS2 stoichiometry. It is expected that S2 generation would have reached a steady value and eventually fallen off, had the surface remained intact.

The integrated flux between 1 keV and 2 MeV in solar flares should be greater than the flux of 2 MeV protons used in this experiment. This is due to a predicted 105 increase in proton flux from developing stars (Feigelson et al., 2002), as compared to present day values. However, the distribution of particles in a solar flare would favor a lower-energy distribution of protons, as an exponential relationship between energy and flux exists (Reedy and Arnold, 1972). Based on these results, sulfides in the early solar system may have limited stability in an irradiation environment. Over the course of a prolonged irradiation, it may be possible to completely reduce a sulfide to a metal.

## III.d Lessons Learned and Recommendations

The results presented here are strictly qualitative, but steps could be taken to both quantitatively determine volatilization products and increase the overall sensitivity. For best results, the end-station should be completely isolated from the beamline; this would involve passing the beam through a thin-window such as Havar© or Si3N4. In this experimental configuration, all pumping would be accomplished through a high-vacuum pump in series with the RGA, where all evolved gasses would pass by the filament of the RGA. While this configuration may be the simplest, there exists a limitation for the total beam current which can pass through the thin film. Thin film windows could degrade and fail under the presence of prolonged high-current beams.

A second option for quantifying the technique and increasing the sensitivity, could be the combination of a differentially pumped end-station and a calibrated leak. A differentially pumped vacuum system, where the end-station pressure was below that of the beamline, would favor the collection of the majority of volatilized species. This could be accomplished by increasing the pumping speed in the end-station and installing a restrictive orifice before the chamber. A calibrated leak could additionally be used to determine what percentage of the gas is lost to the beam-line vacuum system. A correction factor based on the gas lost could then be used to quantify the data.

To best eliminate mass inferences due to contaminates, dedicated small irradiation chambers which have been sufficiently cleaned and baked should be used. Furthermore, irradiating a standard target which is unlikely to evolve any gasses over a range of temperatures could be used to set background values of contaminants in the system prior to actual irradiations. The RGA in this experiment came with a channel electron multiplier which can increase the sensitivity of the instrument, but its functionality was not examined here. However, it has a maximum operating pressure that depending on the volatility of the target, may limit its usage.

The degree of volatilization in any target is determined by the compounds present, beam current used, and the ability of the target to conduct heat from the near surface. Heat removal is dependent on the electronic properties of the material, size of the target, mounting mechanisms, beam size, and additional steps taken to remove heat (air, chilled water, etc.). Materials possessing metallic bonding resist volatilization from ionization, and can conduct heat away from the beam spot to limit local temperature increases. However, metals with lower vapor pressure, poor thermal conductivity, or those which have dramatic changes in the conductivity as the temperature increases may begin to exhibit volatilization. Non-metallic materials are more difficult to determine the nature of volatile product formation, as gasses can evolve from both ionization and heating. Separating these effects is difficult, but could be resolved by cooling the target and using a sequence of low-flux irradiations with a more sensitive experimental geometry.

The cement and metal examples share a similar experimental context, as both materials are the subject of accelerated aging studies associated with neutron damage in nuclear reactors. The flux of these two H+ ion irradiation experiments represent two extreme values. The relatively lengthy time (1 hour) required for the initial pump down of the cement target to 1x10-5 Torr, as compared to the nickel (20 minutes) can be a pre-irradiation indicator of the relative sensitivity of the targets to flux.

In the case of the sulfide experiments, currents above 3 µA resulted in pressures in the 10-4 range at the RGA. These high pressures are beyond the upper operating limit of the RGA, and care should be taken during testing to determine maximum beam currents to be investigated. Precautions should also be taken to isolate the RGA from the end-station during sample exchange in order to avoid filament exposure to low-vacuum or atmospheric conditions. The filaments are extremely sensitive and costly to replace if damaged.

1. CONCLUSIONS

Ion irradiation experiments were conducted to demonstrate how a Residual Gas Analyzer (RGA) can be used to monitor the evolution of volatilization products as a function of irradiation conditions. In many cases, volatilization of primary elemental or molecular constituents can be directly identified by the corresponding atomic mass as measured by the RGA. However, care should be taken to account for compounds that may mass interfere; this includes diatomic molecules, multiple charge state ions, and other vacuum contaminates such as hydrocarbons. While a major use for collecting RGA spectra during an irradiation could be to limit deleterious heating and ionization effects from high-beam currents, it is also ideal for tracking volatilization products from radiolysis and in scenarios that may mimic early solar system processes.

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