USE OF AN ECR ION SOURCE FOR MASS SPECTROMETRY

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Introduction

At ANSTO we have been developing an Isotopic Ratio Mass Spectrometer (IRMS) system utilising an Electron Cyclotron Resonance Ion Source (ECRIS). The ECRIS has less than advantages over traditional systems. Efficient ionization of sample gas up to 2 orders of magnitude greater than conventional electron impact ionization, and the brake up of molecules in most cases once ionized greater than +1, removing molecular interferences. For this reason our ECRIS system typically make measurement of the 2+ charge state ions, thus the name adopted for our system is the RMIS++. Initial testing of the system have proven the ability of the ion source to generate appropriate charge states, and efficient utilization of the sample. This testing has also found 2 main undesirable characteristics of the ECRIS. These are high backgrounds as shown in Figure 5, and long response time and decay times of samples in the source as demonstrated in Figure 6. Much of our recent work has been focused on suppressing these characteristics, while still maintaining suitable capabilities for IRMS.

Experimental Arrangement

The configuration of the RMIS++ instrument developed at ANSTO is shown in Figure 1, including an electrostatic analyser which has been installed since the work was reported [1]. The ECRIS was developed in house to produce low to medium charge state ions with minimal cost. The source is described briefly below; a more detailed description of it can be found in a recent paper [2]. The source operates at 19.25 GHz at a frequency of 6.8 - 7.15GHz. The magnetic field is provided by permanent magnets, constructed from 2 ring magnets to form the axial field, and a hexapole to from the radial field. The axial field characteristics can be seen in Figure 7. The plasma chamber is formed by a closed end quartz plasma bottle, and a gold plated plasma facing electrode with a 2.5mm aperture which can also be seen in Figure 7. Gas is delivered via silica capillaries, allowing transfer of gases from the sample chambers at pressures ranging from 5-1000 Torr.

High Backgrounds & Contamination

Figure 2: The top images show residual surface contamination on the plasma electrode and on the plasma bottle. The lower images show the ion source after atmospheric baking at 1000°C for 2 hours. Note that the plasma electrode, and the rear of the plasma bottle both display a clean region which is in line with the loss zone of the plasma and is surrounded by the contaminatees.

Source of Contamination:
The following process were found to contribute the overall background of the ion source.

Oxygen or water vapor contamination in the helium carrier gas.

The contaminants in the helium carrier gas were trapped out of the gas stream in a liquid nitrogen trap.

Outgassing of ferrules used to seal the capillaries

An alternative all metal sealing product for the capillaries namely Silitek® [5] ferrules were installed in place of the Vespel system. Baking of these fittings showed a distinctive outgassing cycle with no sign of an ongoing leak, or permeation.

Prevent the process the stainless steel fitting was modified and adhered to the quartz with Torr Seal [6] epoxy.

This gave an improved performance over the alumina seeded and can also be baked to 122°C. After initial baking of the Torr Seal bond and fitting, there is no need to re-baking the join and fitting.

Surface cleaning of the ion source prior to running the containments in the plasma.

Baking up the plasma delivers the contaminant ions and the rear plasma is vacuumed for 30 minutes. The green plot showing the pre-modified results, and in blue the modified ion source rise and delay times, or memory effects impacted on the cycle time. The plasma was turned off and back on during different parts of the sample cycle, as well as extended periods of time after reaching maximum beam current across the plasma.

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The process of retaining sample, and releasing sample is performed over a period of 10 hours after the introduction and removal of a nitrogen gas sample. The nitrogen was introduced for 10 hours for the remaining 30 minute cycle. The green plot showing the pre-modified results, and in blue the modified ion source rise and delay times, or memory effects impacted on the cycle time.

Figure 7: The above is a plot of the axial field of the ECRIS magnet array. The relative positions of the various elements of our ion source are can be seen with respect to the field structure. Note that the plasma facing electrode, and end of the quartz plasma bottle are located on the ECRIS zone side of their respective B. The benefit of these modifications can be seen comparably in Figure 8, showing the current of each of the charge states of nitrogen produced by the ion source with the original and compacted ion source geometries.

The Problem: The IRMS++ system is intended for the measurement of isotopic ratios of oxygen, carbon, and nitrogen in substances such as water, carbon dioxide, nitrogen gas, etc. It is imperative that the residual species of these species be minimized in the system.

Oxygen peaks of the order of hundreds of nanoamps compared to just over 10µA of helium beam. The blue plot shows the resulting spectrum of the original position, once again the plasma chamber was displaced, moving the ECRIS region as demonstrated in Figure 8. Much of our recent work has been focused on suppressing these characteristics, while still maintaining suitable capabilities for IRMS.

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The problem: long rise and decay times, or memory effects impacted on the cycle time between samples, and potentially effected the validity of the measurements of isotopic ratios. Figure 6 shows the rise and delay of the 16+ beam from the introduction and removal of a nitrogen gas sample. The nitrogen was introduced for 10 hours for the remaining 30 minute cycle. The green plot showing the pre-modified results, and in blue the modified ion source rise and delay times, or memory effects impacted on the cycle time.

Source Retention Processes: Our investigations into the rise and decay effects has established the following characteristics.

1. The rise & delay effect is not a vacuum conductance effect. This was proven by the variation of the length of the capacitive that supply the plasma ion source, and by changing the position of the gas into the source with the plasma extinguished.

2. Inert gases are not retained as strongly as reactive species. The same process used in Figure 6 was performed with Helium, Neon, and Argon. Inert ion sources have much lower rise and decay rates.

3. Surface reactivity of the plasma bottle does not play a strong role in the rise and decay process. The introduction of different species into the plasma bottle was followed by the contamination being removed by the emission of the ion source. This showed no impact on the rise or decay rate.

4. The process of retaining sample, and releasing sample is performed over a period of 10 hours after the introduction and removal of a nitrogen gas sample. The nitrogen was introduced for 10 hours for the remaining 30 minute cycle. The green plot showing the pre-modified results, and in blue the modified ion source rise and delay times, or memory effects impacted on the cycle time. The plasma was turned off and back on during different parts of the sample cycle, as well as extended periods of time after reaching maximum beam current across the plasma.

5. The process of retaining sample, and releasing sample is performed over a period of 10 hours after the introduction and removal of a nitrogen gas sample. The nitrogen was introduced for 10 hours for the remaining 30 minute cycle. The green plot showing the pre-modified results, and in blue the modified ion source rise and delay times, or memory effects impacted on the cycle time. The plasma was turned off and back on during different parts of the sample cycle, as well as extended periods of time after reaching maximum beam current across the plasma.

Post extraction, the system incorporates an einzel lens, followed by X and Y deflectors. Beam energy analysis with the electrostatic analyzer is performed after the mass to charge ratio in the analyzing magnet. Multiple beams can be measured simultaneously by the Faraday cup array. The UHV system is oil-free, and regularly achieve base pressures <4×10^-9 Pa.

References

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