A High Efficiency Thermal Ionization Source Adapted To Mass Spectrometers

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Introduction

The high temperature ion source used on the isotope separators at Los Alamos was originally developed almost simultaneously by Beyer in Dubna [1] and Johnson at Livermore [2]. Some contemporaries of the authors claim that the concept for the source arose from a discussion between two of the authors at a conference and that credit for the work should be shared jointly. During the past ten years we have learned how to make the source produce reliably and with high throughput. Experience seems to be an important factor in determining what not to do. If reliable operation of the ion source were as simple as the authors of the two papers implied, this type of ion source might be in wider use today.

There are three requirements for repeatable results with high throughput: a well-engineered physical assembly that will operate at the required high temperatures, contaminant-free chemical preparation of the samples, and a well-understood operating procedure. A new user is typically faced with all three of these hurdles simultaneously, and the challenges can prove daunting. We were fortunate to have fabricated sources on hand similar to that shown in [2]. The chemistry and operational procedures followed with time.

Figure 1 indicates the elements for which the source is suitable. Those marginally suitable are shaded. Both the first ionization potential (through the Saha-Langmuir equation) and the operating temperature (through the vapor pressure of contaminants) affect the ionization efficiency [3,4,5,6]. Using a tungsten crucible that had been pre-baked to a temperature where thorium could be seen as a contaminant of the tungsten, we achieved an europium ionization efficiency of 72%. Using unbaked crucibles, 40% efficiencies were the norm. This reflects the results reported in [2]. Lutetium has a lower first ionization potential but its vaporization temperature is twice that of europium. This causes increased vaporization of contaminants including tungsten, lowering throughputs to 25%. Zirconium is an element having both high ionization potential and low vapor pressure and our ionization efficiencies for it range between 2 and 3%.

While experimental and theoretical efforts over the past twenty years have proved fruitful on isotope separators, no one has adapted electron bombardment, cavity type sources to the field of mass spectrometry. The mass spectrometry source most

resembling the source discussed here was the tube source described by Delmore, Appelhans and Peterson [7]. That arrangement, however, is incapable of reaching the temperatures required for efficient ionization of refractory materials such as zirconium, hafnium and thorium.

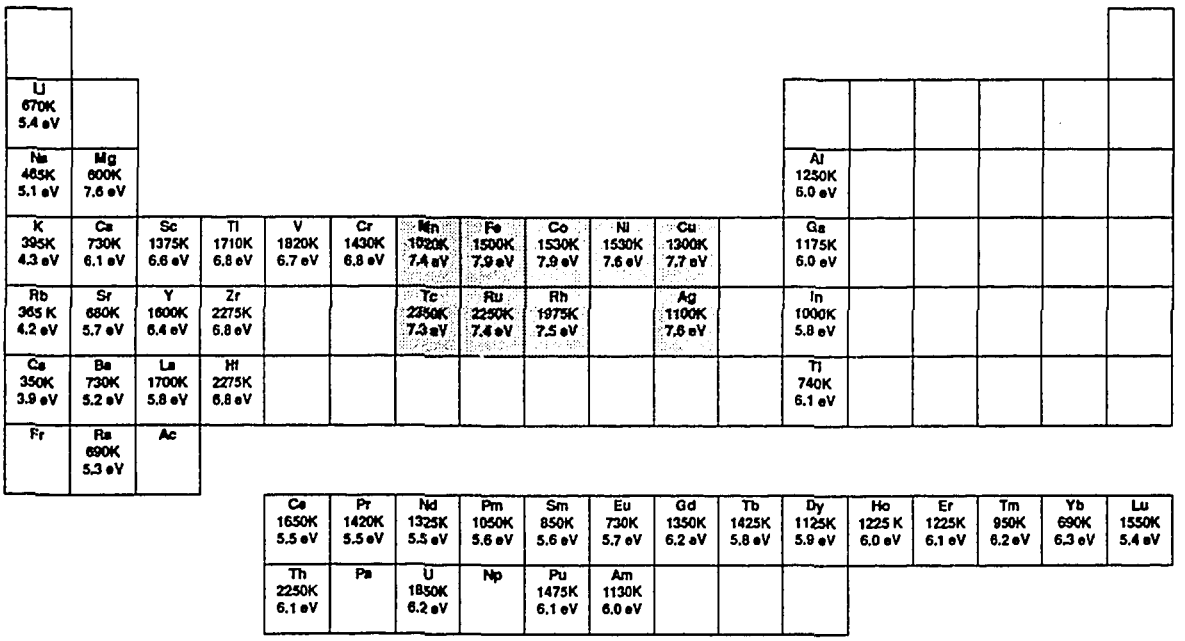

Figure 1: Elements Ionizable Using High Temperature Techniques
(Listed are temperature to achieve 10^{-4} torr and first ionization potential)

Objectives

The isotope separator ion source is unsuitable for mass spectrometry use. It is bulky, designed for single sample operation, expensive to fabricate, requires great care in assembly, requires high heating power and, as a result, significant cooling water flow. Before design of the modified source began, we established a set of objectives:

- Reduce the number of parts to minimize fabrication costs and allow disposal rather than cleanup.
- Minimize complexity of parts; design to commercially available shapes and components.
- Have an inexpensive, one piece crucible.
- Have a modular system.
- *Operate with little or no water cooling.*
- Use existing power supplies and control system.

The Crucible and Beam Formation

The most uncertain of these objectives was the ability to achieve a reasonable ion beam emittance while using a one piece crucible. Figure 2 (top) shows the crucible and cap used in isotope separator operations. The cap surface is shaped to minimize the overall magnification of the beam. The ionizing channel diameter is about 1/3 millimeter. Figure 2 (bottom) shows the design for mass spectrometer operation. The ionizing channel diameter is slightly less than 1/2 millimeter and is over a centimeter deep. In both cases, the ionizing channel opening presents a small apparent object to the optic system but is large enough to ensure that a quasi-neutral plasma can be maintained [4]. The mass spectrometer crucible can be mass produced at reasonable cost and can be baked at high temperature prior to loading. Actual background measurement of contaminants can be performed prior to sample loading if required. Early on, test samples of $Th(NO_3)_4$ were run using this crucible in the isotope separator ion source to quantify changes in beam focussing. The results were encouraging in both ionization efficiency and focal plane spot size.

The electrostatic field code SIMION [8] was then used to model the ion source, comparing relative values of extracted beam emittance while varying crucible position, extractor position and the potential of the electron bombardment filament relative to the crucible. The position of the crucible relative to the electron reflector/heat shield and extractor position had little effect on the beam emittance. The electron bombardment energy (filament and reflector/heat shield) potential had the most effect on the emittance.

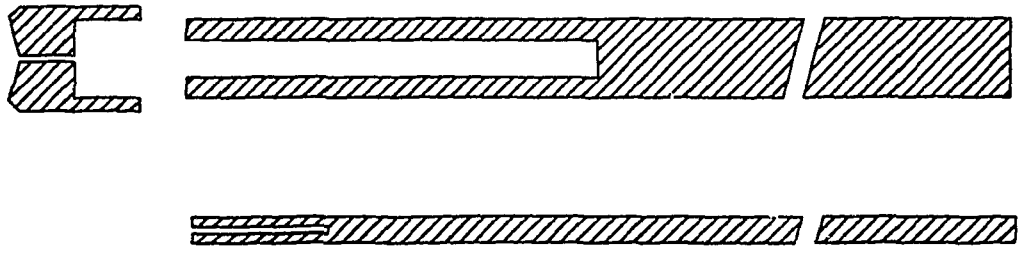

Figure 2: Isotope Separator Crucible (top) and Mass Spectrometer Crucible (bottom)

The Electron Bombardment Assembly

The ion source is shown in Figure 3 in two views. On the left is a transverse section view showing the source mounted on an RF-quadrupole mass spectrometer. On the right is the axial view seen by ions leaving the source. The transverse section view of

the source shows that it consists of three major assemblies: the quadrupole/source mounting adapter, the electron bombardment assembly and the crucible holder. The mounting adapter will take a form appropriate to each application. The crucible holder shown is a purely functional item assembled from Conflat vacuum parts together with one machined rod and two set-screws.

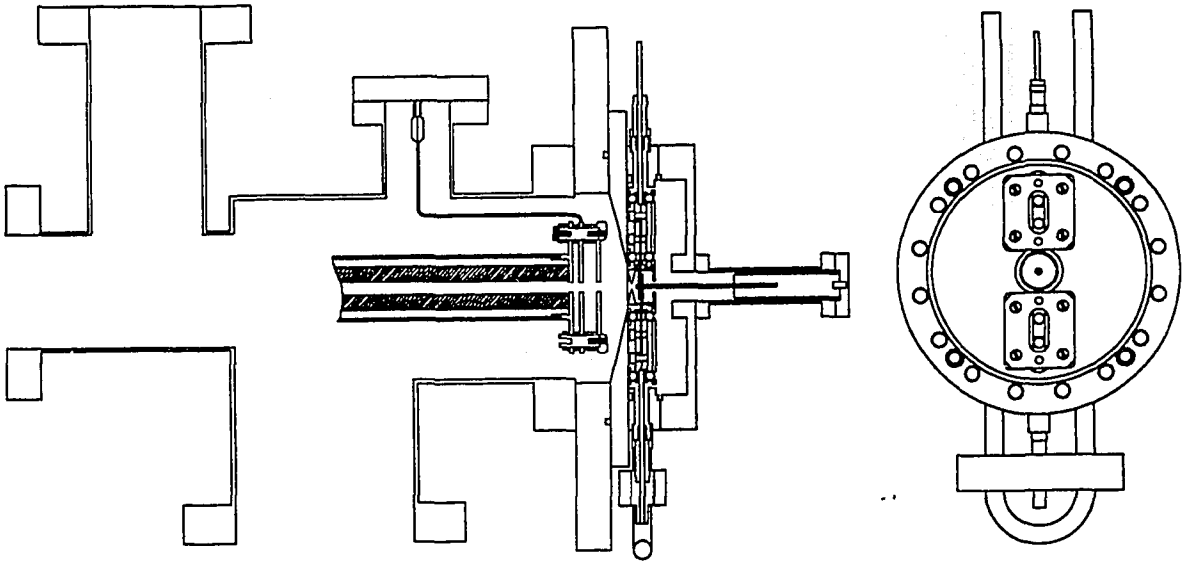

Figure 3: (Left) The ion source mounted on a quadrupole mass spectrometer. (Right) A coaxial view of the source.

The electron bombardment assembly was designed around a 6 inch diameter, double-sided Conflat blankoff flange. Precise machining operations are minimal and are intended for a numerically-controlled milling machine. Interior parts consist of or are fabricated from easily available commercial components. An alignment jig for filament fabrication and a necessary welding operation are a part of the design. The design was carried out using AutoCAD-12, making modifications simple when adapting to specific applications. Cooling water channels have been designed into the system as shown in the axial view. The flow tubes are arranged laterally on both sides of each filament-current electrical feedthrough. These feedthroughs are operated at the filament bias voltage (up to 2.5 kV) and are a hazard to personnel if not covered. The water tubes provide a firm foundation for a clamp-block arrangement which provides personnel safety protection and a securing post so that the electrical feedthrough does not bear the weight of conductors. Without cooling water flowing, the ion source body may reach temperatures which could damage adjoining equipment or personnel. A fan directing air onto the source body usually provides sufficient cooling under these conditions.

Obviously a design which required the operator to break vacuum to introduce each sample would be less than satisfactory, so there is a multiple crucible system in the initial design stages.

Matching The Ion Beam Into a Sector Type Spectrometer

Figure 4 illustrates a GIOS [9,10], first-order calculation of the proposed matching of the ion beam into a 12-inch radius, 90-degree sector type mass spectrometer. The top portion of the figure illustrates the beam in the non-dispersive plane and the bottom shows the mass-dispersive plane. The path shown through the system is 1.79 meters long. The transverse dimensions shown are ±2 cm. Our objective is to match the ion beam into the entrance slit in the dispersive plane and to have a beam

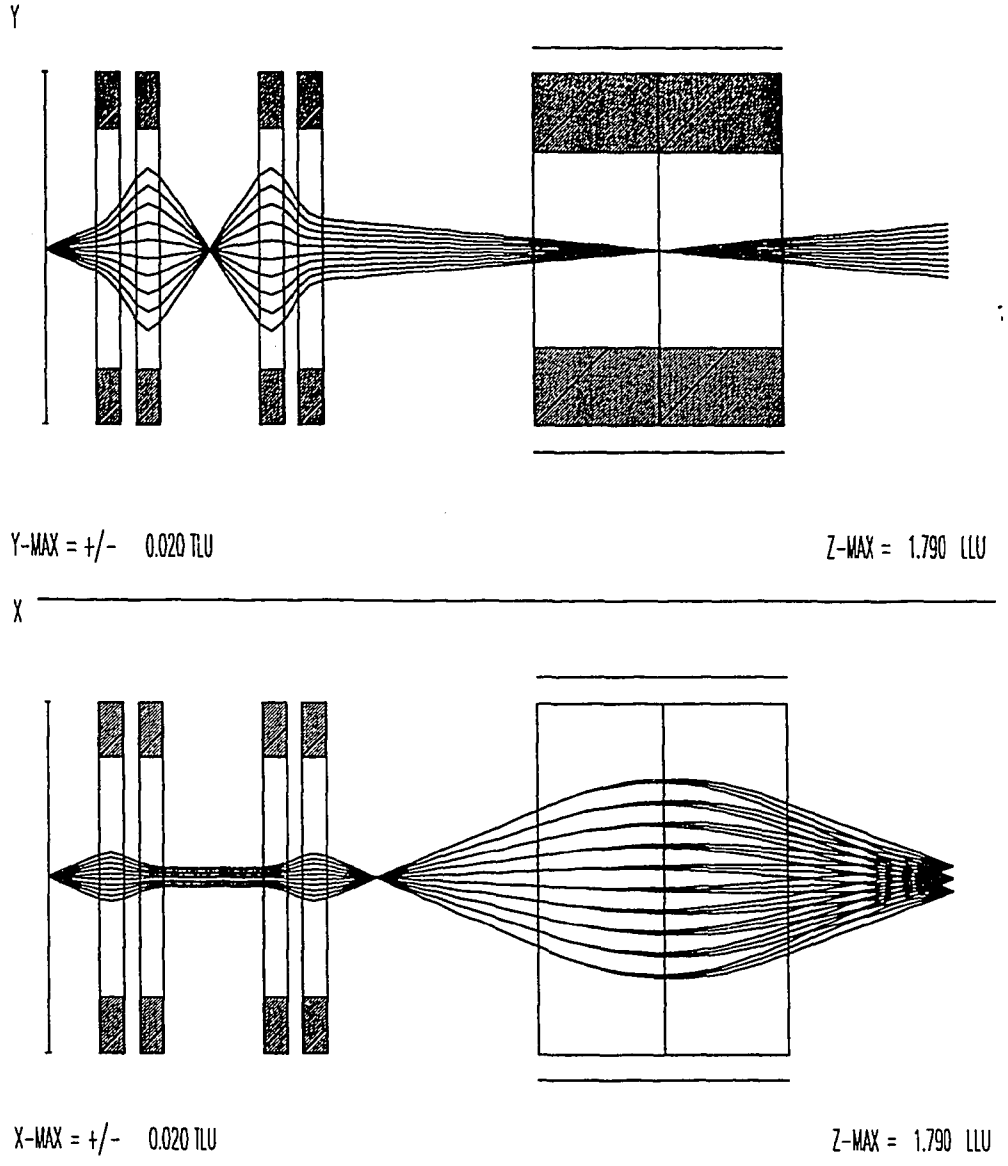

Figure 4: Calculated ion trajectories from the ion source to the focal plane for 231, 232, and 233 amu ions. The top/bottom illustrations are for the non-dispersive and dispersive planes, respectively.

crossover midway through the magnet in the non-dispersive plane. This approach minimizes some aberrations associated with the normal-entrance/normal-exit pole pieces of the homogeneous magnet and provides a smaller magnification in the non-dispersive plane so that the pulse counting apparatus at the focal plane can have a smaller entrance aperture. By not disturbing the entrance slit, exit slit or flight tube of the existing spectrometers, it should prove expedient to switch between this source/lens system and the standard thermal ionization arrangement.

The matching system consists of two electrostatic quadrupole doublets arranged in a FD-DF (dispersive plane) geometry. The drift space between the doublets allows for addition of a pump, emittance defining slits and diagnostic apparatus. The pump will be required only if a gas-discharge-type source is added at some time in the future. It is our intent to minimize any increase in the length of the beam matching system so that the overall length of the system does not become excessive. However, initial second and third-order calculations show that too short a drift space between the first and second quadrupole doublets causes increased aberrations in the non-dispersive plane. We do not propose achieving short physical length at the cost of beam quality. Therefore, we have deferred finalizing the design of the matching unit until we have experimental emittance data in hand.

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