

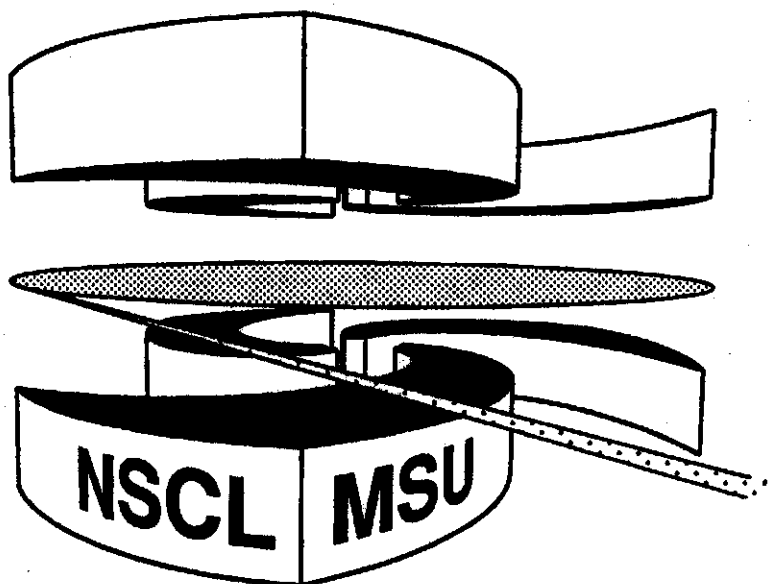


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**EFFICIENT PRODUCTION OF A ^{48}Ca BEAM FROM OXIDE
MATERIAL IN AN ECR ION SOURCE USING A LOW POWER
MINIATURE OVEN**

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Efficient Production of a ^{48}Ca Beam From Oxide Material in an ECR Ion Source Using a Low Power Miniature Oven

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A technique is described which allows the highly efficient production of a ^{48}Ca beam from oxide material in an ECR ion source at temperatures easily attainable with a low power miniature oven. Details on the design and fabrication of the miniature oven are also provided.

I. Introduction

A number of methods have been employed for the introduction of solid elements and compounds at standard temperature and pressure into an ECR ion source plasma. These include the use of externally heated **ovens**[1], plasma heated boats and **crucibles**[2], direct plasma evaporation of metallic wires and oxides of the elements, the use of gaseous compounds of solid elements (UF_6 , BF_3 , SiH_4), plasma interaction with sample material placed on the end of an insulating alumina rod (the "cigarette **method**") [3], and most recently direct ion plasma **sputtering**[4]. Laser ablation has also been investigated as a method of coupling solid material into an ECR **plasma**[5,6]. A concise summary of the advantages and disadvantages of these techniques has already been **presented**[5].

There is a strong demand from the experimental nuclear science community for the production and acceleration of rare naturally occurring very neutron-rich ion beams. Examples of these are ^{36}S , ^{58}Fe , ^{64}Ni and most notably ^{48}Ca . Adequate beam intensities require the use of isotopically enriched material (^{48}Ca comprises less than 0.2% of

natural calcium) and the high cost of the enriched material requires that the samples be small in size and utilized very efficiently. Calcium poses a particular challenge since the metal, which is the form most compatible with many of the above techniques, is chemically unstable in air and undergoes oxidation quite rapidly. This challenge becomes even more difficult when dealing with sample sizes of a few milligrams. In addition, vendors are very reluctant to supply enriched calcium isotopes in the metallic form, and then only at an additional monetary cost. While the oxide form is chemically stable in air and readily available from vendors, it is not best suited for solid beam development with the techniques described above. Calcium oxide cannot be used with the direct ion plasma sputtering method due to its insulating nature. Temperature requirements of over 1800° C make its use in externally heated ovens very difficult and even impossible. The "cigarette method" which, in the past, has been used to produce ⁴⁸Ca beams from oxide material at our laboratory[3] has a history of problems associated with beam instability, longevity and insufficient intensity, and has not proven very successful when attempted at other laboratories. Furthermore, this method makes optimizing the source extremely difficult since heating of the sample cannot be decoupled from source tuning parameters such as magnetic configuration, rf power and source pressure. Clearly, a method is needed which would allow the simple and efficient production of quality ⁴⁸Ca beams in an ECR ion source from oxide material.

When conditions permit their use, externally heated ovens can offer very impressive beam production for solid materials (based on stability, intensity, ion charge state and longevity) in an ECR ion source[1,7]. This is because the feed rate of the solid vapor can be decoupled from the source parameters, much like gas feed, allowing the source to be tuned and optimized without affecting the solid vapor feed rate. An undesirable feature of externally heated ovens, however, is that their physical size may have to be so large (especially true of high temperature, high power ovens) that coupling the oven solid vapor output into the ECR plasma can be very inefficient in terms of sample material usage. Vapor is lost as a result of condensation on the oven

exit hardware and plasma chamber entry ports before reaching the plasma.

We describe below a technique which allows the highly efficient production of a ^{48}Ca beam from oxide material in an ECR ion source at temperatures easily attainable with a low power miniature oven.

II. Design of the Low Power Miniature Oven

The 6.4GHz room temperature electron cyclotron resonance (RTECR) ion source[8] at The Michigan State University National Superconducting Cyclotron Laboratory is used to inject the laboratory's K1200 superconducting cyclotron. It should be noted that, unlike many other ECR ion sources which are "horizontal" in their spatial orientation, the RTECR is positioned vertically. The ion source has three large radial ports, each measuring 2.3cm wide by 12.7cm long, which allow access into the source plasma chamber. The ports have been used to develop beams from solid materials employing the cigarette method, direct plasma insertion, and direct ion plasma sputtering techniques referred to earlier. Taking advantage of these large radial ports, we set out to implement a miniature oven which could be mounted directly inside one of these ports. Such a design would allow excellent coupling of the oven's vapor output into the ECR plasma and is shown in Figure 1. In addition, the upward angle of the oven assures that sample materials which liquify before reaching evaporation temperatures, such as gold and lead, are adequately contained and cannot spill out into the plasma chamber.

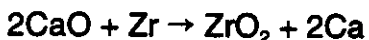
The design of the miniature oven itself, shown in Figure 2, is an adaptation of the very successful micro-oven designed and used by the Grenoble group in their Caprice ECR ion source[7,9]. Referring to Figure 2, the oven is comprised of an alumina (Al_2O_3) tube (oven) with a thread machined in its outer diameter so that multiple turns of resistive heater wire (0.05cm diameter 75% W/25% Re) can be

wrapped around the alumina. A larger alumina tube (sheath) is placed over the oven tube to ensure that the heater wire remains intact. The alumina parts were fabricated by the Coors Ceramics Company, Golden, Colorado. Multiple layers of 0.003cm thick dimpled tantalum foil are wrapped around the alumina parts to provide a heat shield and then the entire assembly is enclosed in a tantalum tube (0.05cm wall thickness). A crucible containing the sample material is inserted into the oven. Depending on the sample material, either an alumina or molybdenum crucible is used in order to avoid undesired chemical reactions.

The oven is heated using an 18V/10A regulated DC voltage supply. Initial calibrations performed off-line using a high temperature thermocouple demonstrated that temperatures of over 1400° C were attainable using relatively low power (12.0V/4.3A). The oven was tested at 1400° C over a five day period demonstrating temperature stability within 2° C and no signs of failure.

III. Production of a ⁴⁸Ca Beam From Oxide Material

The 1400° C capability of the miniature oven still was not adequate to attain the more than 1800° C required to evaporate calcium oxide. A method employed by target makers[10] to fabricate very thin ⁴⁸Ca foils from oxide material involves the use of a chemical reducing agent, such as zirconium, according to the reaction:



Using this technique, ⁴⁸Ca foils can be made at temperatures of about 1325° C. Such a temperature can be attainable in our miniature oven, so a similar reduction method was investigated and is described below.

A mixture of natural calcium oxide powder and fresh zirconium powder (made by

filing a zirconium rod) was prepared according to the correct stoichiometric ratio (a mass ratio of about 1.25 parts calcium oxide to 1.0 part zirconium) and pressed into a pellet. The pellet was placed in a molybdenum crucible and the entire oven assembly was run at a temperature of about 350° C for five hours in an off-line vacuum chamber to allow for outgassing of water and volatiles. The oven and sample were then transferred to the RTECR ion source.

The ion source was brought up on an N₂ plasma and the temperature of the oven was slowly increased. Very surprisingly, calcium (over 5eμA of ⁴⁰Ca¹²⁺ and 2eμA of ⁴⁰Ca¹³⁺) was clearly noticeable in our mass-to-charge spectrum at a corresponding temperature of only 900° C. It should be noted that no noticeable zirconium or oxygen was detected from the reduction reaction. The calcium beam was very stable over a 12 hour period and the ⁴⁰Ca peaks were not present in a mass spectrum taken 20 minutes after the oven was turned off. Having demonstrated the feasibility of this technique, a similar sample was made using calcium oxide isotopically enriched to the level of 93% ⁴⁸Ca (the sample contained 24.2mg of ⁴⁸CaO after baking at 350° C for 5 hours). The sample was then loaded into the RTECR ion source in preparation for a 144 hour experimental run which required the K1200 cyclotron to accelerate ⁴⁸Ca¹³⁺ to E/A = 80MeV.

In order to minimize the possibility that the sample might be totally consumed before the experiment concluded and so as not to waste any of the costly ⁴⁸Ca, the ion source was operated in a mode that produced only the beam current necessary for the success of the experiment and no more. Typically, 1% to 3% of the beam current measured immediately after the ion source analysis magnet is extracted from the K1200 cyclotron. During the first half of the experiment about 16eA of beam current was needed out of the cyclotron, requiring an oven temperature of approximately 780° C. The second half of the experiment was run to conclusion (total run time of 140 hours) with over 65eA of very stable beam current out of the cyclotron, requiring an

oven temperature of approximately 875° C. At the conclusion of the experiment a mass spectrum was taken (at the exit of the ion source) and, although many of the mass 48 charge state peaks were obscured by nitrogen and contaminant peaks that have similar mass to charge ratios, both $^{48}\text{Ca}^{11+}$ and $^{48}\text{Ca}^{13+}$ were clearly visible each with a beam current of about 4eμA. A spectrum taken minutes after the oven was shut off clearly showed these peaks disappear. In addition, the $^{48}\text{Ca}^{13+}$ beam extracted from the cyclotron was monitored showing that the beam current dropped more than a factor of 100 within a few minutes after the oven was turned off. Reweighing the sample showed, very surprisingly, that only 2.4mg of ^{48}Ca was consumed during the entire 140 hours of running time.

IV. Discussion and Conclusions

The present measurements reveal that a much lower temperature is required than originally thought (875° C as opposed to 1325° C) for the adequate production of calcium from oxide material in an ECR ion source using the reduction technique described above. This is understandable if one considers that target makers generally choose to fabricate their thin foils on a time scale of minutes, not days, and a much higher rate of evaporation is needed.

The 2.4mg of ^{48}Ca consumed during the entire 140 hour run, although surprisingly small, is not unreasonable. If it is assumed that 4eμA of $^{48}\text{Ca}^{11+}$ was produced during the entire 140 hours (a conservative overassumption) and that this charge state accounted for 20% of the total calcium beam current, this amounts to approximately 5×10^{18} atoms. The 2.4mg sample contained 3×10^{19} atoms indicating a total overall ion source efficiency of approximately 17%. The RTECR ion source has a history of very efficient use of sample material[3].

We have demonstrated a technique which allows the highly efficient production

of a ^{48}Ca beam from oxide material in an ECR ion source at temperatures easily attainable with a low power miniature oven. In addition, this method yields superior beam quality in terms of stability, intensity and longevity compared to methods which have been used in the past.

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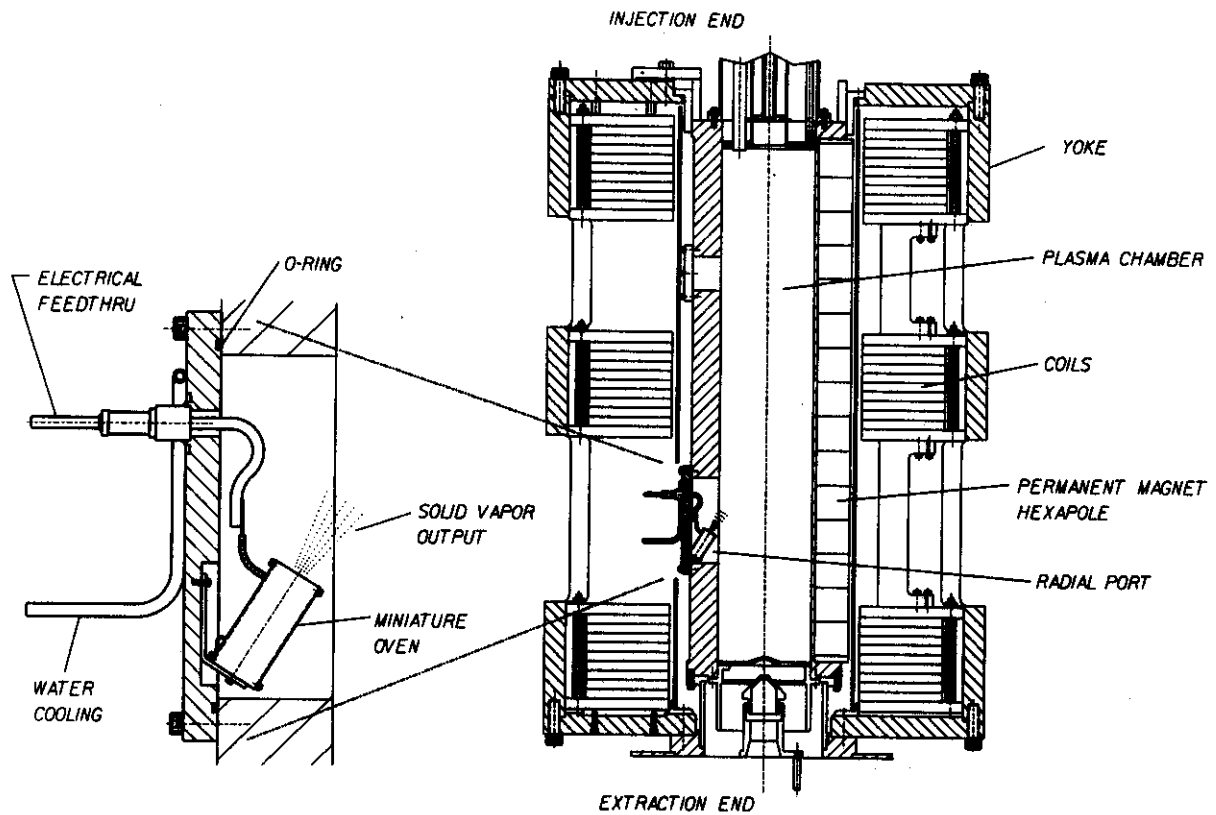


Figure 1 Cross-sectional view of the MSUNSCL RTECR ion source showing the miniature oven mounted within one of the source's radial ports.

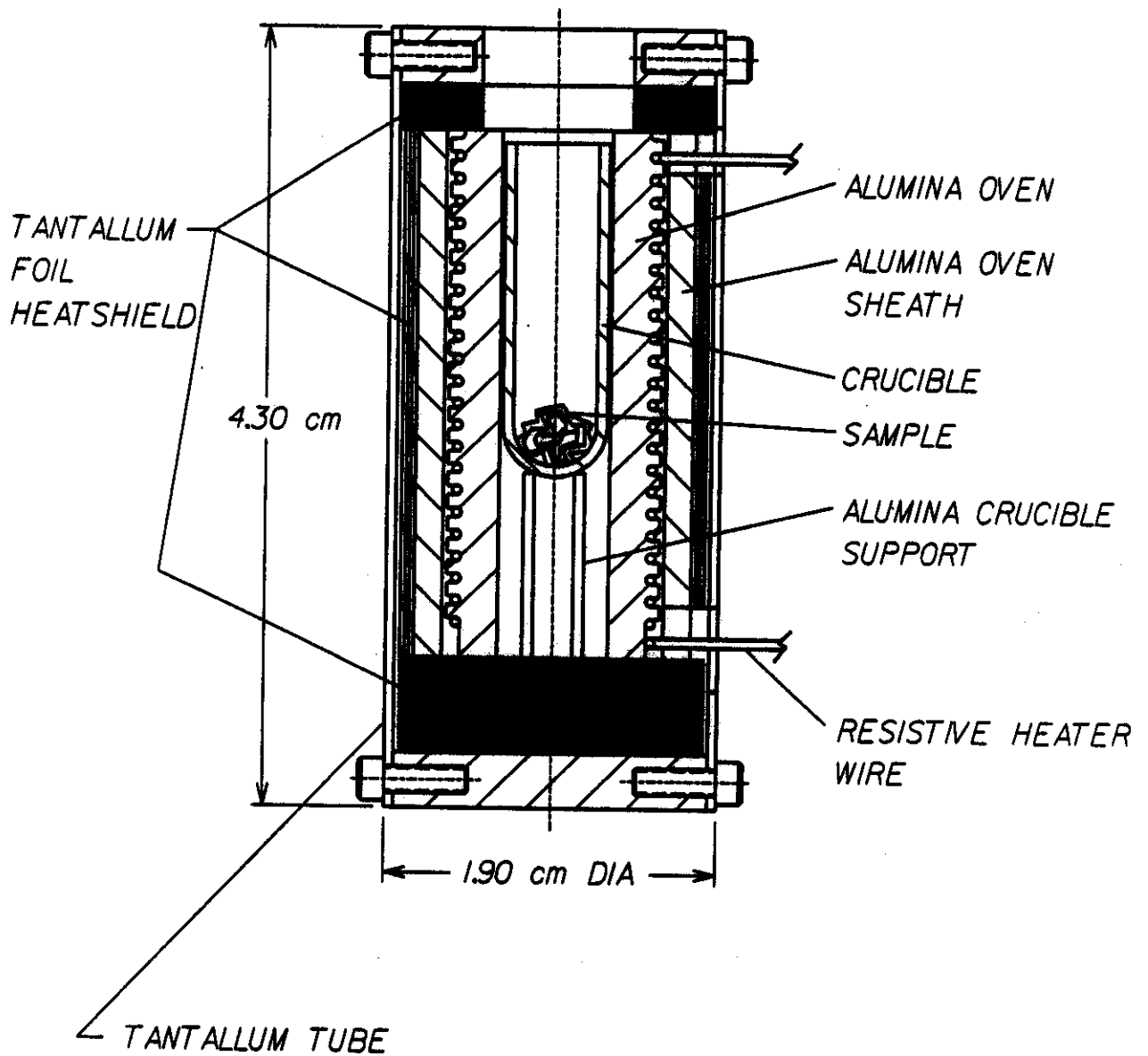


Figure 2 Detailed cross-sectional view of the miniature oven. The oven is an adaptation of the micro-oven designed and used by the Grenoble group in their Caprice ECR ion source.