REFRACTORY OVENS FOR ECR ION SOURCES AND THEIR SCALING

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Abstract

The radiofrequency (rf) oven can be used as a metal vapour injector for Electron Cyclotron Resonance ion sources; the application to high temperature boiling metals (like Cr, Ti and V) was recently demonstrated. Duration and reusability of oven parts were good, since only crucible needs to be maintained at a high temperature T_s. For vanadium case, achieved T_s was over 2300 K with about 280 W of rf power, with the present design and size, tailored to our 14.4 GHz ECRIS. Optimization for higher temperatures is also discussed. Materials, more than rf power coupling, emerge as ultimate limits. Comparisons with sputter probes and with different metals are briefly reported.

INTRODUCTION

ECR ion sources (Electron Cyclotron Resonance Ion Sources [1]) are a typical component of nuclear physics accelerators, since they may ionize any heavy element X to high charge state i, if the mass flow F_X of X atoms trapped inside ECRIS plasma is stable and moderate: say $F_X/A_X \cong 1$ ng/s with A_X the mass number, for a high performance source giving a total of 50 particle μA of X ions. For metallic element, miniaturized ovens or some other accessories are needed to optimally inject the element vapor in the source plasma. A series of radiofrequency heated ovens was developed[2] from a sample temperature $T_s = 1300$ K of the first copper coil oven (used for silver beams) to 1500 K (tested with Sn and Pr) to recent 2400 K with molybdenum coil (used for V, Ti and Cr up to now) [3], Many technical issues were solved, in order to maintain run duration over one week. Some experiments with resistive heated oven (the usual choice for simplicity) were also performed with silver. Comparison with sputter probes was studied elsewhere in detail[3].

Advantages of rf oven techniques (over most ohmic ovens) are: a) only crucible and few nearby parts need to be exposed to temperature as high as T_s and to be built in refractory materials; b) coupling between heater and crucible is not subject to uncertainties of conduction between contacting parts. Use of rf furnaces is well documented in atomic physics[4] and semiconductors industry, for which modern 3D simulations are now developed[5].

Two schemes of rf oven are shown in fig 1. The tantalum crucible with radius r_s (containing up to 50 mm^3 of X sample) is thermally insulated from the coil (of radius r_c and with temperature T_c), even if efficiency of rf heating decrease with r_c/r_s increasing. A proper set of simulation tools (in a two dimensional geometry r, z) was developed to

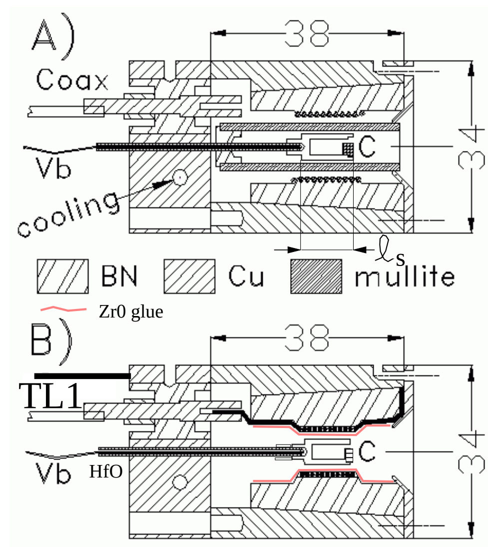

Figure 1: Scheme of rf oven: A) copper coil; B) Mo coil Dimensions in mm.

compute η_s, defined as the ratio of the power dissipated in the crucible to the power P_o dissipated in the whole oven; η_s depends on the coil conductivity σ_c and crucible conductivity σ_s, increasing with $(\sigma_c/\sigma_s)^{1/2}$. For this reason, Cu and Mo are good material for coil, while iron, tantalum and vanadium are good materials for a crucible. We considered $\eta_s > 0.4$ an acceptable design compromise. Coil needs strong cooling, and by the way, η_s increases when T_c decreases.

Alignment of the crucible was a problem for a long time, but was recently solved by increasing fitting length ℓ_f in the crucible, shown in fig 2. For particular elements (with high vapor pressure before melting point, like iron) a consumable crucible may be used as a sample [6]. We propose a reentrant shape, not yet tested, for a consumable crucible (of Fe or V) in Fig. 2.B. Since the HfO stem sustaining the crucible will probably fail over $T_s > 2500K$, stronger constructions are envisioned for samples of Nb, Ta (and so on) in Fig 2.C and 2.D. Another important technical issue is the matching of the rf generator to the oven (depending on T_s), for which a satisfactory solutions were developed[2, 7].

Total oven size is typically CF35 compatible, even if smaller versions, down to a 25 mm diameter seems possible [8]. In fig 1 note also that oven is enclosed in shell, intended originally to shield plasma from oven rf. That shell proved most useful to shield source from oven thermal radiation (also necessary in high T_s ohmic oven [9]) and to shield rf coil from being sputtered from the source plasma.

In the next section, we report the final result of oven test

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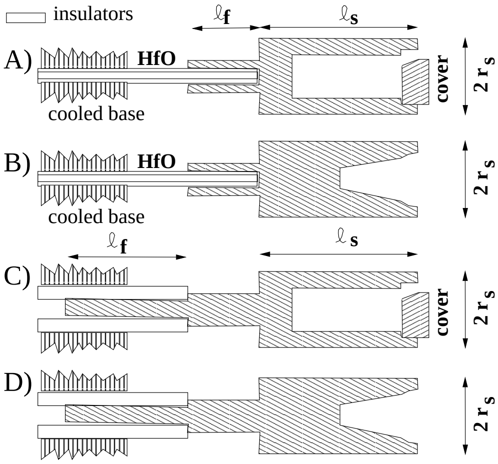

Figure 2: Scheme of crucible: A) general purpose; up to $T_s = 2400$ K with Hf0 stem; B) consumable; C) and D) general purpose and consumable for $T_s > 2500$ K concept. In the general purpose crucible, note the sustaining Hf0 rod; $\ell_s = 10$ mm and $r_s = 2.6$ mm; fitting length ℓ_f is variable. For good efficiency ℓ_s should be a little shorter than coil.

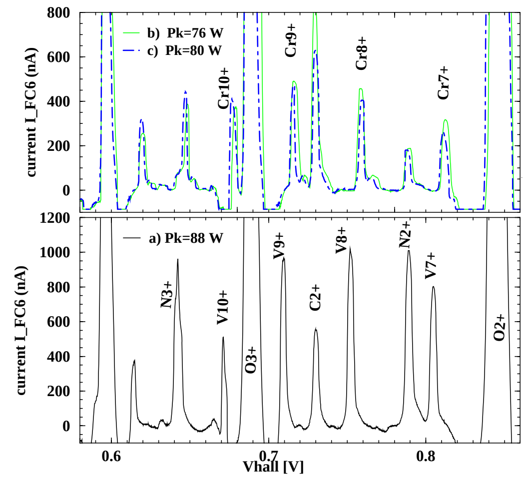

Figure 3: Spectra of extracted currents for plasma elements: a) Oxygen/vanadium $P_o = 260$ W and $P_k = 88$ W; note carbon and large nitrogen impurities. b) Oxygen/chromium $P_o = 80$ W and $P_k = 76$ W. c) Oxygen/chromium $P_o = 80$ W and $P_k = 80$ W. Hall probe voltage is proportional to magnetic field 1 V for 1 kG; its offset drift was compensated by aligning the C^{2+} peak.

with Cr, Ti, V in detail. Perspective of improving oven design and hardware for very refractory metals (from Nb) and brief comparison of oven concept with sputter probes are discussed in the last section.

EXPERIMENTAL RESULTS

For test of rf ovens two facilities were available: 1) an uncooled plasma chamber in the ECR ion source Alice (a small ECR ion source described elsewhere [2]), so that ECRIS microwave power P_k is then limited to about 100 W (instead of 350 W); 2) the MetAlice teststand, where oven temperature and evaporation rate can be measured (without an ECRIS plasma).

Standard sinusoidal rf generators can be used, with some special consideration. Oven coil is part of an LCR series circuit, where a fixed capacitance C is mounted on the vacuum flanges, and a 40 cm vacuum transmission line TL1 connects coil and capacitances. Rf generator is connected to this LCR by an insulating transformer (11 to 13 turns in our case). Since the LCR impedance Z_o depends on σ_s and thus on T_s, we find that Z_o decreases when T_s increases, and resonant frequency f slightly changes. In the case of Fig1.B, oven Z_o went from 180 Ohm at cold to 70 Ohm when $T_s = 2350K$; f went from 1290 kHz at cold to 1260 kHz at 2350 K, where resonance is very broad. Presently, rf is supplied by a 350 W linear amplifier (1-60 MHz range) capable of operating with a load range between 50 and 150 Ohm. Custom built rf generators are used elsewhere[6, 8], also using square waves. Moreover, industrial rf generators can be used[7].

Up to $P_o = 230$ W, TL1 was a coaxial line, with quartz beads as insulators and a 1.5 mm spacing between conductors. The larger power requires a larger spacing in TL1, now about 5 mm, with a 50 KHz decrease of oven resonant frequency f. New crucibles (in Ta) have a longer fitting length $\ell_f \geq 5$ mm to the sustaining stem (in HfO), so loss of alignment during operation is prevented. Moreover, ECRIS conditions are very sensitive to vapor mass flow stability, so we stabilize the AC input voltage of the rf generator within 1 % to stabilize P_o and thus F_X.

Number and duration of experimental runs was limited, due to the beam schedule of the ion source Alice. Some runs with Ti samples shows reasonable beams, and helped in improving crucibles and stems. In a vanadium run with the Fig 1.B oven, total vanadium fill was 0.24 g, almost maximum capacity, considering interstitial space in the powder used. A vanadium beam was maintained for

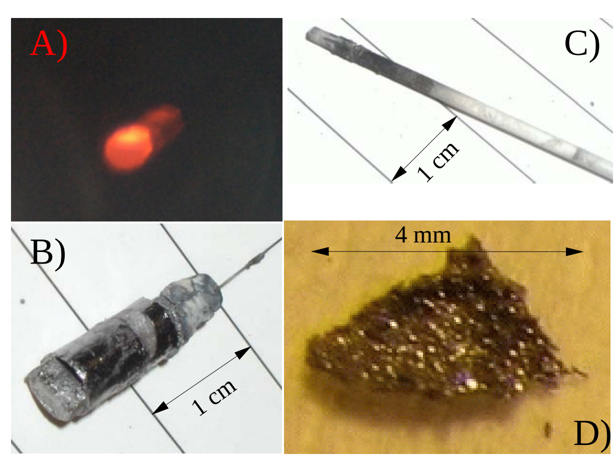

Figure 4: Deteriorated parts after more than 250 h near $T_s = 2400$ K: a) crucible warm; b) crucible cold; c) HfO stem; d) glue cover fragment

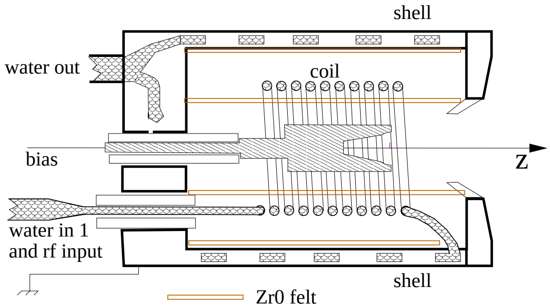

Figure 5: Concept of a $T_s = 3000$ K rf ECRIS oven. Note cooling of the coil and of the outer shell. Coil spacers not shown.

about 3 days (curve 'a' in fig. 3), with a reasonably clean spectrum, after which the experiment had to be suspended. Later, evaporation from the same crucible continued for an additional 200 hours (fractioned in 3 weeks) on the Met-Alice teststand at progressively increasing power level (to maintain a given deposition rate of 4×10^{-12} m/s, equivalent to an emitted flow of $F_V = 200$ ng/s, judged to be suitable for keeping vanadium beam levels) up to $P_o \geq 300$ W, corresponding to $T_s = 2400$ K. After this prolonged heating, some deterioration of the crucible, of the ZrO glue covering the coil and on the HfO stem were observed; $r_s = 2.6$ mm becomes $r_s = 2.5$ mm, tiny droplets appeared on the glue cover, and the HfO rod thinned (see fig 4).

Operation with chromium ($T_s = 1600$ K) has a neglible effect on these oven parts. After other 3 days of operation with chromium (curve 'b' and 'c' in fig 3) at a quiet 500 nA level, we raised the oven temperature very rapidly (T_s from about 1600 K to 2200 K in 6 hours) to terminate the experiment. Peaks of 900 nA at $^{52}Cr^{9+}$ position and of 1.3 μA at V^{8+} (from leftover of previous experiments) were observable, but with large impurities.

In summary, beam operation of about one week are well possible (and a few days was demonstrated) also for refractory rf oven for several elements. On the other side, operation conditioning requires at least 24 hours for the large quantity of BN used inside oven, and changes of operation temperature are unsuggested.

TOWARDS HIGHER TEMPERATURES

Osmium which is considered a very interesting element for nuclear physics experiments has a vapor pressure $p = 0.3$ Pa at $T_s = 3000$ K, that we can consider as an ultimate design limit. Tantalum is more adequate for intermediate tests for manufacturing reasons. Since material evaporated from crucible sides is lost for ECRIS operation and deteriorates oven operation, crucible geometry like in Fig 2.D must be considered for Ta, to increase the temperature on axis and the evaporation surface facing the plasma; moreover fig 2.D (or 2.B) will partially collimate vapor forward. The need of 3D simulations including non uniformity of the crucible temperature is apparent.

To reach a T_s near 3000 K, a power $P_o = 750$ W is needed with Fig 1B geometry, which is not adequate to sustain it (even if some further progress over $T_s > 2400$ K seems possible). A typical rf generator has $P_o = 2500$ W on a 50 Ohm output with adjustable frequency $f = 2\pm0.2$ MHz. We conclude that these power generators may easily tolerate the frequency changes of a rf oven, while impedance matching will require more studies. The problem is similar to rf antenna matching for inductively coupled plasma negative ion sources (with typical frequency f from 1 MHz to 13.56 MHz), also actively studied in our group. Assuming that available power is somewhat reduced by matching conditions, reasonable design values are $P_o = 1.5$ kW and oven voltage $V_o \cong 300$ Vrms.

Strong cooling of coil is then necessary, as used elsewhere[7], even if technically difficult. A provisional geometry is shown in fig 5, incorporating also a cooling of the shell. Note that two Zr0 felt layers are used to repair coil from radiated heat, so going back to the intermediate shield of Fig1.A. Another concept shown in fig 5 is a small quantity of insulators, for hopefully reducing outgassing.

Other methods available to ECRIS include the MIVOC method[8], the insertion method[10], the laser ablation[11], the evaporation by an electron beam, and the sputter probes[3], as described elsewhere. Sputter probes are appealing for simplicity, but we had to note some issues: electrode must be cooled[12]; a fraction of vapor is ionized and accelerated by sputter voltages, so that it escape from ECRIS plasma[3]; the large sputter current may perturb ECRIS operation.

Further investigation on the induction oven concept of fig 5 and adequate modelling are therefore necessary. As in the case of Fig 1B oven, solving the problem of operation at large T_s will also improve reliability for operation at lower T_s for other elements.

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