Controllable evaporation of cesium from a dispenser oven

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The instrumentation allows for controllable evaporation of the alkali metal cesium with a wide range of evaporation rates from an easy-to-use alkali metal reservoir adaptable in the stored amount of cesium. The oven comprises three elementary components: (i) the alkali metal reservoir, a dispenser, allowing for ultra pure metal vapor of adjustable amount via the dispenser current; (ii) the full metal valve and full metal body reducing contaminations of the activated dispenser and for operation at elevated temperatures (up to 300 °C) with small temperatures variations (5°C) avoiding the build-up of uncontrolled cold spots; (iii) the surface ionization detector (SID) for measuring and controlling the evaporation rate. The dispenser oven can be easily transferred to the other alkali-metals.

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I. INTRODUCTON

Controlled evaporation of alkali metals such as cesium is required for many industrial applications in vacuum and thin film technology, e.g. photo cathode fabrication, OLED devices, as well as for many basic research experiments, ranging from ion sources to Bose-Einstein-condensation experiments and magneto-optical traps [1,2,3,4]. The requirements on the evaporation rate, the stability and reliability depend of course on the application and also on the vacuum conditions since alkali metals not only getter oxygen and water but also easily form compounds due to their reactivity. Thus, several concepts with individual solutions for the purpose are in use. One example is evaporation of cesium in ion sources for negative hydrogen ions [3]. Such ion sources are widely used as front-end source in accelerators and as ion source for generating neutral beams for fusion experiments. As the formation of negative ions depends on the thickness and quality of the cesium layer at the extraction surface, the performance of these ion sources depends on the cesium control. For the latter, improved oven concepts are mandatory to fulfill the demands of the next generation of ion sources.

The negative ion source development for accelerators[5] and for the neutral beam injection systems of the international fusion experiment ITER[6] requires adjustable, controlled and reliable evaporation of cesium into the ion source. Evaporation rates of the order of 10 mg/h are necessary and evaporation has to take place in vacuum (background pressure in the range of 10^{-5} mbar to 10^{-7} mbar) and in plasma operation (hydrogen plasma at a pressure of 0.3 Pa to 1 Pa) during an operational campaign.

At present several cesium oven concepts are in use[7,8], all of them working with liquid cesium reservoirs with the main drawbacks of contamination of the pure cesium reservoir by impurities and of laborious cesium handling. The evaporation rate is governed solely by the coldest spot of the oven and thus not adjustable independently from the reservoir. A detection system for the evaporation rate is missing, which means the actual evaporation rate is

unknown and not quantitatively adjustable. In addition, the cesium reservoir will be depleted after a certain time which means replacement is not an easy task keeping the safety issues in mind. In order to improve the cesium distribution in large ion sources, first investigations started to distribute cesium from a multi nozzle system[9].

In some ion sources for accelerators cesium cartridges are used as reservoir[10]. These dispensers contain cesium compounds embedded in getter materials. Cesium is evaporated by a temperature activated chemical reaction. The dispensers are directly implemented in the source and heated by the plasma load which in turn requires an experienced procedure for reproducible evaporation. Very small sizes with low cesium contents are in use being in the order of one mg per dispenser.

In order to ensure a reliable evaporation at variable evaporation rates during vacuum and plasma operation a new cesium oven concept is introduced in the following. Focus is laid on the usage of the dispenser as cesium reservoir with variable cesium amount and the measuring device at the oven nozzle allowing for monitoring and feedback control of the evaporation rate. First measurements of the evaporation in vacuum and in plasma operation will be shown proving also the functionality of the oven concept. Operation in negative hydrogen ion sources is envisaged.

II. OVEN CONCEPT

A. General layout

The full system basically consist of three key elements: (i) a dispenser which serves as alkali metal reservoir, (ii) a valve between reservoir and evaporation pipe to separate the two volumes and (iii) the surface ionization detector at the oven nozzle for measuring the actual evaporation rate under vacuum conditions. Figure 1 shows a drawing of the instrumentation.
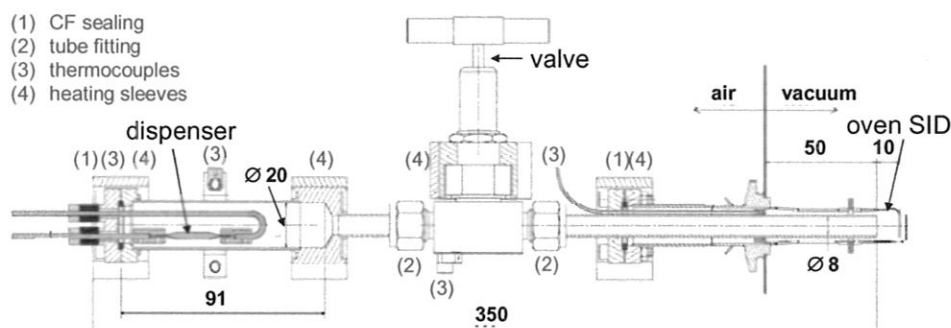
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FIG. 1. Drawing of the full system with dimensions in mm.

In order to operate the device at elevated temperatures, i.e. above 200°C, the system is a full metal device. A stainless steel bellow valve is used (Swagelok SS-10BG-MM) as well as standard CF sealings and metal tube fittings. The main body is manufactured from stainless steel. A good thermal conductivity is provided by the copper tube which is nickel-plated on the inner side to prevent the formation of oxycuprates with the alkali metal during aeration times of the system as it is the case for Cs forming $CsCuO_2$ on Cu in ambient air. Heating sleeves (JEKA Piroslim PRSL000003) are used to heat all components to uniform temperature which in turn is measured and controlled by thermocouples (type K) at four locations. A temperature control system is connected to achieve stable temperatures via feedback control, adjusted to allow for deviations of maximum 5°C. As the evaporation rate is controlled by the dispenser current, the absolute temperature of the oven is not critical. Typically the temperature is set to 260°C; the system allows temperatures up to 300°C. A high temperature is particularly important for avoiding cesium accumulation whereas small variations are particularly important for avoiding uncontrolled changes in the evaporation.

B. Dispenser as reservoir

The alternative to the usage of liquid cesium reservoirs filled in a glove box or the

usage of pure cesium filled glass ampoules which are cracked on demand is the usage of alkali metal dispensers in which the alkali metal is chemically bound at room temperature. Such an alkali metal vapor source is commercially available from different companies, in different sizes and different alkali metal contents[11,12]. Typically, the dispenser is sealed and thus chemically stable in ambient air at room temperature. By thermal activation, done by ohmic heating via an electrical current flowing through the dispenser, the sealing melts. With increasing the heating current the pure alkali metal is released and its evaporation can be controlled and readjusted by the current flow[13]. The dispenser type based on chromate compounds[11] requires reducing agents as Al and Zr to getter the oxygen. Much less additives are embedded in the chromate-free dispenser type[12] to generate ultra pure metal vapor in a very controllable manner. For the type of oven presented here, the chromate-free dispenser (Bi_2Cs) with a slotted cylindrical shell is chosen whose diameter and length basically determine the amount of cesium in the reservoir. The diameter is a few mm, the length is a few cm, being 3 mm and 2 cm respectively for the 100 mg reservoir chosen here. For activation currents of 3 – 4.5 A are needed, for evaporation 5 – 10 A are used. For this type of dispenser, the available alkali metal amount in a dispenser ranges from milligrams up to the several grams at similar required heating currents being favorable for an easy exchange.

C. Surface Ionization Detector (SID) for in-situ measurements

For determination and control of the actual evaporation the concept of the surface ionization detector (SID), also known as Langmuir-Taylor detector, [14] is used. The detector is directly mounted at the oven opening as shown in figure 2. The alkali metal is ionized at the hot tungsten wire which is the ionization filament. The ions are accelerated by a bias voltage between the ionization filament and the second filament, the ion collector. Since the method relies on measuring the ion current, the SID is used in vacuum only. For plasma operation the

bias has to be switched off due to the high electron current which would be drawn In contrast to the known usage of SIDs, the ion collector is also heated to prevent an enrichment of cesium at high evaporation rates on the wire and thus guarantee reliable measurements. In addition, the geometry is chosen such that the oven SID is capable of measuring the full range of accessible evaporation rates without being covered by the alkali metal. The tungsten wires have a diameter of 0.3 mm. The heating currents are typically 2-3 A; for acceleration a voltage of 40-50 V is appropriate. The measured cesium ion current, the SID signal, varies from nA to mA. The background current is typically below 10 nA.
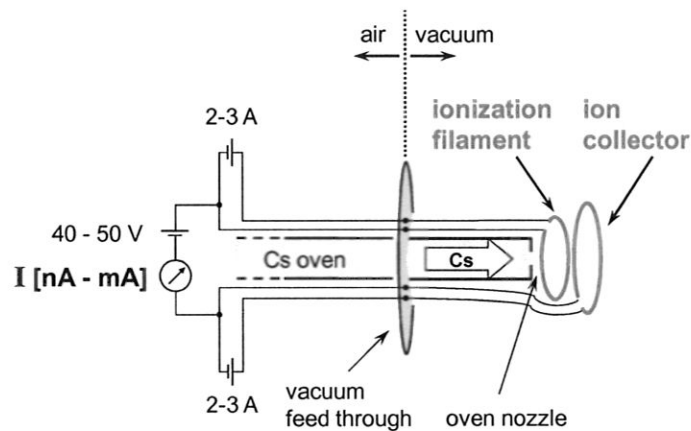

FIG. 2. Conceptual drawing of the SID at the oven opening.

D. Features of the device

Basically, the introduced concept is suitable for monitored evaporation of elements with ionization energies below the work function of tungsten (4.54 – 4.8 eV) since surface ionization takes place at the ionization filament. This requirement is fulfilled for potassium, rubidium and cesium, with ionization energies of 4.34 eV, 4.17 eV and 3.89 eV, respectively. Francium is excluded because of being radioactive. For these three candidates dispensers are commercially available. Due to the wide use of cesium for different applications and the

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initial trigger given for the development of ion sources for negative hydrogen ions, cesium is used in the following.

The full metal valve has two functionalities in the closed position. First, it prevents the already activated dispenser from contamination by impurities from the vacuum chamber, e.g. during aeration times, and thus ensures fast and reliable reactivation of the evaporation process. Secondly it allows for an easy handling of the reservoir and an easy exchange of the dispenser without breaking the vacuum and the conditioning in the vacuum chamber.

The dispenser acts as a controlled reservoir since the evaporation is controlled by an electric current. The combination of the valve with the dispenser allows for a fast response time of evaporation including the option to stop the evaporation in a short time.

With the heating circuit and the full metal construction the device can be heated up to 300°C. Heating circuits, feedback controlled by thermocouples at four positions (see figure 1), allow for a uniform temperature in the whole system within 5°C. This is mandatory due to the steep temperature dependence of the alkali metal vapor pressure.

With the combination of the individual components the device is suitable for a wide range of evaporation rates (0.1 – 50 mg/h) and the SID offers online monitoring of the evaporation with fast response time (in-situ).

III. DEMONSTRATION OF FUNCTIONALITY

The dispenser oven has been tested in a vacuum chamber at background pressures of typically 5×10^{-6} mbar achieved by a turbo pump system. The cylindrical chamber (stainless steel with a quartz plate on top) has a diameter of 15 cm and a height of 10 cm. Hydrogen plasmas at typically 10 Pa are generated by using inductively coupling with a planar coil. The generator frequency is 27.12 MHz and a maximum power of 600 W is available. The experiment is often used for fundamental investigation on cesium in hydrogen plasmas[15] with

the purpose to support the negative hydrogen ion source development for ITER.

The oven is mounted at a bottom port of the chamber. All measurements introduced in the following are taken with the same dispenser with a cesium reservoir of 100 mg. Activation took place at 4.5 A. The dispenser was already in use for 3 months. Due to the decrease of the evaporation rate with the depletion of the cesium reservoir the dispenser current has to be adjusted from time to time; typically after several hours of operation. The SID signal is measured in-situ by an automatic data acquisition system with a time resolution of about 3 seconds. The signal of the SID, i.e. the SID current, was calibrated after the campaign by integrating the signal over the whole time in which the 100 mg dispenser has been emptied. The latter has been checked by weight loss measurements. The oven has been operated at a typical evaporation rate of 1 mg/h.
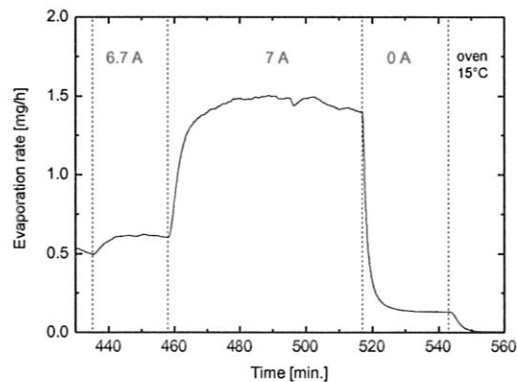

FIG. 3. Stable evaporation and variation of the dispenser current at an oven temperature of 260 °C.

Figure 3 shows stable evaporation of the oven at a dispenser current of 6.7 A (example 1) and secondly the increase when changing to 7 A (example 2). The evaporation rate is very sensitive to the dispenser current: low variations of the current result in huge changes of the evaporation rate. Thus, a high dynamic and flexibility of the evaporation rate is possible. After a certain time the dispenser current has been switched off, demonstrating that the major

source of evaporation is the dispenser and not reservoirs formed at inner surfaces of the device. Finally, the oven temperature has been set to 15°C and the signal goes back to zero, proving the reliability of the SID.

The functionality of the valve is shown in figure 4. In addition, the signal from a standard SID mounted in the bottom plate of the vacuum chamber[15], the local SID, is plotted. In this case, data acquisition is done manually. Since this SID is several cm away from the oven nozzle, it is not directly in the line of sight of oven opening. As can be seen, the oven SID is more sensitive to changes. The response time of closing and opening the valve is within a minute scale. Again, a decrease to the background level is obtained, meaning that formation of cesium spots did not take place.
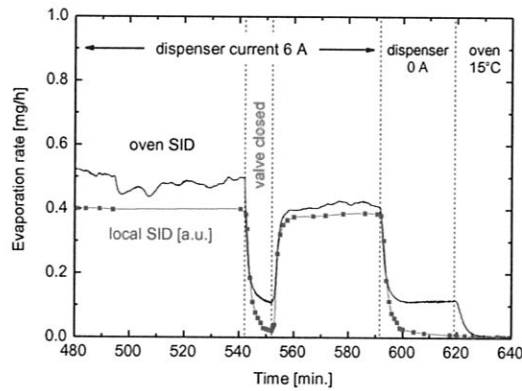

FIG. 4. Functionality of the valve and response time at an oven temperature of 260 °C.

The fast response time and the sensitivity of the measuring device are shown in figure 5 for which the evaporation was changed by purpose. Again, the local SID is much less sensitive. Thus minor variations of the evaporation rate are not detectable with such a setup highlighting the relevance of a measuring device at the oven opening. In order to prove the functionality of the oven SID, the signal is also compared to a line of sight integrated cesium density measurement close to the nozzle. For this purpose the white-light absorption spectroscopy (W-ABS) of the resonance line at 852 nm is been utilized[15,16]. As soon as the

absorption signal is above its detection limit a very good agreement with the oven SID is obtained. It should be noted that the absorption signal is recorded manually, explaining that fast peaks are missed. As also demonstrated in figure 5, the high sensitivity of the oven SID remains even after a long period of evaporation. This is achieved only by heating the ion collector also.
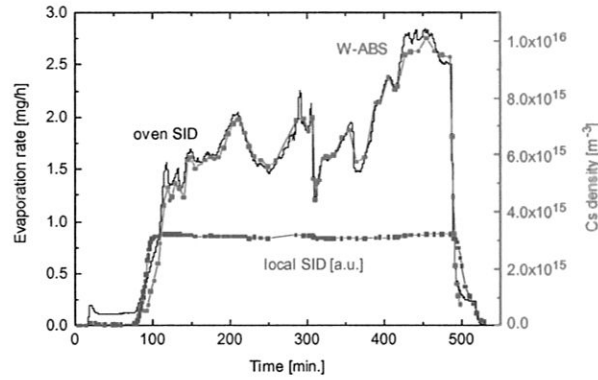

FIG. 5. Comparison of the oven SID with a local SID and absorption spectroscopy (W-ABS) during active variations of the evaporation rate over a long period of time.
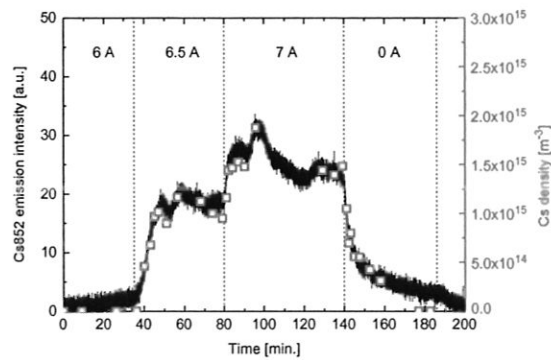

FIG. 6. Variation of the dispenser current at an oven temperature of 260°C in a hydrogen plasma at 10 Pa. The cesium amount is recorded by emission and absorption spectroscopy (left and right axis respectively).

Evaporation during plasma operation is demonstrated in figure 6. In this case, however, the oven SID cannot be used for measurements. Thus, the absorption technique is applied again as well as emission spectroscopy monitoring the cesium line emission at 852 nm[15].

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Similar cesium densities as under vacuum evaporation can be achieved. The response time on the dispenser current is not influenced by the plasma, proving the suitability for plasma operation.

As a result of the reliable performance of this oven concept, the prototype ion source for ITER will be equipped in the next campaign with such a device, replacing the liquid reservoir oven concept used so far[17].

IV. CONCLUSIONS

In order to fulfill the requirements on reliable evaporation of alkali metals for several applications a new oven concept has been introduced with focus on application in ion sources where ultrahigh vacuum conditions are not available. Beside evaporation at a pressure of 10^{-6} mbar, plasma operation is also mandatory. The device makes use of an adequate dispenser as cesium reservoir with variable amount (1 mg to 1 g), a valve between the reservoir and the oven nozzle and a measuring device for the evaporation rate in vacuum operation at the oven outlet (Surface Ionization Detector - SID). For stable and reliable operation all components are temperature controlled and capable to work at temperatures up to 300°C avoiding evaporation from uncontrolled cold spots. The functionality of the oven has been demonstrated in vacuum and plasma conditions relevant for ion sources. As other alkali metals have similar properties and are available as dispensers the presented invention can also be used for reliable evaporation of these elements.

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