

**IMPROVEMENT OF AN ION SOURCE SAMPLE OVEN OF THE CERN  
HEAVY ION ACCELERATOR**

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*Abstract*

The subject of this paper is to propose improvements for an ion source sample oven and find new possibilities to introduce small quantities of solid materials into the plasma of an Electro Cyclotron Resonance Ion Source (ECRIS) by evaporation. At the present time CERN is operating a heavy ion acceleration facility, which will have to be prepared and adapted for the future Large Hadron Collider (LHC) project. For this the reliability of the existing sample oven for metallic ions has to be improved. Two methods for an improvement of the existing oven were elaborated which could meet the requirements. Both are electrically heated ovens, which are similar to the existing one, but differ in the material used as resistant element to heat the oven in order to provide higher reliability.

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## INTRODUCTION

The accelerators at CERN provide beams of high-energy particles to be used in a wide range of experiments. Since 1964, possibilities for the acceleration of heavy ions opened up. This has led to the development of the CERN heavy ion physics program. The CERN heavy ion facility provides a fully stripped lead ion beam ( $\text{Pb}^{82+}$ ) to various experiments. Ions are extracted from the ion source and pre-accelerated in a Linear Accelerator (LINAC). The heavy ion linac (Linac 3) accelerates the beam from 2.5 keV/u to 4.2 MeV/u before injecting into the Booster (PSB) for further acceleration up to 94 MeV/u. After passing the Proton Synchrotron (PS) and the Super Proton Synchrotron (SPS) the beam reaches a final energy of 157 GeV/u and is then directed to the different experiments [1]. To produce highly charged lead ions, a 14.5 GHz Electron Cyclotron Resonance (ECR) ion source is used. The ECR source at CERN, known as ECR4, is a new version of the CAPRICE design and was developed at GANIL (France) [2]. It produces a current of more than 140  $\mu\text{A}$  of  $\text{Pb}^{27+}$  during the afterglow is a sudden and brief increase of the beam intensity occurring just after the end of the microwave power pulse [3].

Lead vapor is produced by metal evaporation from a so-called 'micro-oven'. This oven which is introduced into the ion source, consists of a tungsten filament, a ceramic sample holder containing the solid material to be evaporated which is placed inside the filament. The metal vapor feed into the ion source plasma chamber can be controlled by the heating current. The lifetime of the present oven is 1-2 months. For future experiments the reliability has to be improved so the oven can be operated up to 6 months without unforeseen breaks. There is also the intention to run the ECR not only with lead, but also with other elements which have a higher melting point. The sample temperature, which is needed to get the desired vapor pressure from the sample material, could be up to 1200° C.

## METAL VAPOR FEED TECHNIQUES

Some nuclear physics experiments require ion beams of heavy elements. Most of the elements exist as solids at room temperature. Producing ion beams from gaseous materials (such as  $\text{N}_2$ ,  $\text{O}_2$ , and the noble gases) is generally easier and results in a more stable source operation than from the solids. A number of methods have been used as a means of introducing solids into the ECR plasma. The selection of the best method to feed solid material into the ion source depends on the operational mode, the variety of ion species needed, the temperature conditions inside the source, ion source reliability, operation time and ease of operation.

The most important methods for feeding solids into ion sources are:

### *Evaporation from an external furnace*

Externally heated ovens allow solids to be vaporized and introduced into an ECR plasma much like a gas:

Utilizing this method, the feed rate of the solid vapor is decoupled from the source plasma tuning and therefore allows the source to be optimized without affecting the vapor feed rate. Disadvantages of this method are their rather large material consumption rates which is a concern when using costly isotopically pure sample materials and the re-condensation of the vapor on colder parts of the source and the feed pipe before it reaches the plasma [7].

### *Evaporation in an internal electrically heated oven*

This method is used on the ECR of LINAC 3. The material consumption is low, small quantities of rare, expensive isotopes may be used and the evaporation is independent of the plasma characteristics [4,5,6].

### *Ablation by a laser beam*

A new technique using a pulsed laser for evaporation of solid material into the source plasma was tested in several laboratories. Pulsed KrF EXCIMER laser (UV radiation) and pulsed Nd:YAG laser (IR radiation) were used for this method. The laser beam was injected through the extraction hole of the ECRIS to ablate a rotating target. The particles ejected from the surface of the target are ionized in the plasma of the source which is fed with oxygen support gas. Potential advantages of this technique are the high efficiency of material usage, no plasma interaction with the bulk of the material and only a small access port to the plasma. It is also possible to produce beams from conductive and non-conductive solid materials. A demerit is the high cost, the laser lifetime, and long-term stability is not yet known. There is also a lack of control of neutrals in the plasma [4,7].

### *Ion sputtering*

This technique consists of placing a negatively biased metallic sample close to the ECR plasma. Positive ions from the plasma are accelerated toward the sample. After striking it, atoms are evaporated into the plasma and ionized. The beam is very stable for long periods and the exact position of the sample is not as critical as in the plasma heating method. A drawback is the high consumption of material, and a certain difficulty in controlling neutral pressure [4,8].

### *Plasma heating*

A rod of metal or oxide is introduced into the plasma near the resonance surface where energetic plasma electrons vaporize the sample. To compensate for the evaporation of the tip, the sample position must be continuously adjusted. This technique does not provide stable beams for long periods. Another disadvantage is that optimizing the source is difficult since changing the magnetic configuration or RF power changes the plasma and thus the vapor flow of the material [4].

### *Use of volatile chemical compounds*

In this method gaseous compounds of solid elements are used. Feeding the gas UF<sub>6</sub>, BF<sub>3</sub>, or SiH<sub>4</sub> into the source can produce beams of uranium, boron, and silicon. In this case special care has to be taken using aggressive compounds to prevent damage of some parts of the source [8].

### *The MIVOC-method*

It can be considered as a natural extension of the use of gaseous compounds and has been used with the JYFL-ECRIS since 1993. The MIVOC-technique (Metal Ions from Volatile Compounds) with an ECR ion source allows for the production of highly charged metal ion beams at room temperature conditions. In the MIVOC, vapors of volatile compounds that contain metal atoms in their molecular structure are used to release metallic elements. Compounds are placed in a separate vacuum chamber connected to the plasma stage of the ECR ion source. From there the vapor of the compound is allowed to diffuse into the source. Decomposition and ionization of the compound take then place in the plasma. Organic compounds (powders or liquids) with relatively high vapor pressure at room temperature ( $>10^{-3}$  mbar) are used to transform the metal into gaseous state. This allows production of ions in a similar manner as in the case of gaseous elements. Up to the present time, the MIVOC method has been used with at least 17 different elements, for example Mg, Cr, Fe, Ni, Mo, and W. The following are some apparent advantages of the MIVOC method: simplicity in construction, ease to use, production of stable long term ion beams and low consumption rate of material, but there could be risks of source pollution with aggressive ions [9].

### *Sample heating with infrared light beam*

This is a future oven design, which has not been tested, but it seems to be a promising alternative to the electrical heated ovens. This idea works on the principle to transfer an infrared light beam into the ion source in order to achieve the required power, which is needed to heat the oven sample. It should be possible to introduced heat from outside the source through an optical fiber or a highly reflecting tube. The light source can either be a powerful infrared lamp or a continuous wave infrared laser. Inside the ion source, an absorber is fixed that converts the light beam into heat and transfers it to the sample material. This method also has the benefit of independently controlled heating.

## **THE EXISTING OVEN**

The micro oven [4,5] consists of an aluminum oxide (also known as alumina) tube with a 0.5 mm tungsten filament inside. It is connected to the tantalum casing on the one side and to a molybdenum part on the other side. The alumina sample holder containing the solid material to be evaporated is placed inside the filament. Its cavity volume is about 30 mm<sup>3</sup> and contains approximately 250 mg of isotopically pure lead 208.

All these elements are placed inside a tantalum casing in order to reduce energy loss by heat radiation. This casing is clamped to a long copper push rod, which also carries the heating current. The rod allows the oven to be moved to an optimum position. A molybdenum part is connected to a coaxial molybdenum rod inside the copper tube. For a detailed drawing of the existing oven see Appendix.

The metal vapor feed can be controlled by the heating current together with the RF heating. Lead consumption is between 5 and 7 mg per day, so a normal charge of around 250 mg of lead lasts for approximately 700 hours, which is a favorable material consumption even for expensive isotopic pure samples (1g isotopic pure lead 208 costs around 10,000 US\$) [6].

For the required current of 100  $\mu$ A of Pb<sup>27+</sup> ions, a constant lead vapor pressure of about 10<sup>-5</sup> torr is needed. To provide this vapor feed, the sample material has to be heated up to around 460° C, which corresponds to an electrical power consumption of 2 Watts. For an oven temperature of 1000° C the dissipated power is estimated to be 15 Watts.

Sometimes (every few months) the filament inside the ceramic tube breaks where it is connected to the tantalum heat shield. In this case the ECR has to be shut down, the copper support rod which holds the oven on its front end has to be pulled out and the broken oven must be replaced. This part of the source where the oven introduction mechanism is located must be re-pumped. Although, there is a system of lock valves which allows the replacement of an old oven by a new one without breaking the whole source vacuum, this can cause an interruption of about 2 hours. During the start up time the ion beam which is delivered to the accelerators and to the experiments is either interrupted or unstable, sometimes up to 8 hours. The reason for the breaking of the filament in the contact area to the tantalum tube is not yet fully known. Possibly, the lead vapor and the low pressure oxygen atmosphere in combination with the high temperature cause an oxidation process on the wire surface, which leads to a hot spot. There the resistance increases and the wire melts.

## **CONDITIONS AND RESTRICTIONS**

A constant and well-regulated metal vapor feed is required for the stable and proper performance of the ion source and the vapor feed must be easy to control. Reliability has to be improved to avoid unforeseen failures which cause interruptions of the experiments. Removing and replacing of the heating element as well as the sample material should be feasible in an appropriate time and modifications on the present ECR should be minimized.

There are some restrictions and conditions for applied materials in an ECR ion source. It is important that the properties of the materials used fulfill these conditions.

The oven is used in a clean vacuum environment of mainly oxygen (about 10<sup>-5</sup> torr). That means only non out-gassing materials are suitable. The materials should

also be non-oxidizing, difficult to oxidize or at least form a stable oxide surface without major changes in material properties such as resistivity of the heating filament. Since compounds with corrosive elements like fluorine or chlorine could harm the plasma chamber and other parts of the source, they have to be avoided. In all high temperature applications, the compatibility of oven materials with the gas atmosphere, with the source materials and with the ion source sample material (e.g. lead) has to be considered. This is very important because undesired reactions at high temperatures may occur with resultant damage to the furnace and source

components. Even in high vacuum, reactions between heating elements and traces of residual gases may occur. These can come from out gassing of the material being heat-treated, leakage or improper operation of the vacuum equipment. It is also significant that the plasma stability must not be affected by vapor feed methods. The intention is to run the ECR not only with lead, but also with other elements, which may have a higher melting point. Possible elements include Calcium (Ca), Indium (In), Cesium (Cs), Lanthanum (La), Gold (Au), and Bismuth (Bi) (see Table 1).

| Symbol | Name      | Atomic number | Commonest isotope [ % ] | Melting point [° C ] | Boiling point [° C ] |
|--------|-----------|---------------|-------------------------|----------------------|----------------------|
| Ca     | Calcium   | 20            | 40 97%                  | 839                  | 1484                 |
| In     | Indium    | 49            | 115 96%                 | 157                  | 2080                 |
| Cs     | Cesium    | 55            | 133 100%                | 28.5                 | 705                  |
| La     | Lanthanum | 57            | 139 100%                | 921                  | 3457                 |
| Au     | Gold      | 79            | 197 100%                | 1064                 | 3080                 |
| Pb     | Lead      | 82            | 208 52%                 | 328                  | 1740                 |
| Bi     | Bismuth   | 83            | 209 100%                | 271                  | 1560                 |

Table 1: Possible sample materials for the ion source

The vapor pressure which is required for a good source operation with other sample materials is expected to be roughly the same as in the current case using lead:  $10^{-5}$  torr. If the oven method is maintained, the heating temperature which is recommended to get these vapor pressure must be higher than 1200°C. Figure 1 shows the curves of the vapor pressure versus temperature for some possible ion source sample materials.

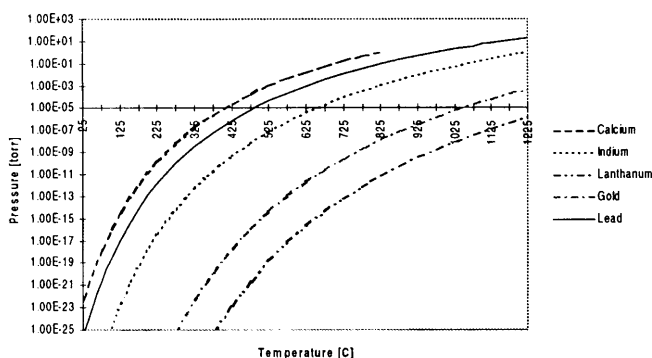


Figure 1: Vapor pressure of possible ion source sample materials [21]

The vapor pressure was calculated from the formula:

$$\log(p) = \frac{5.006 + A + B(T + 273)^{-1} + C \cdot \log(T + 273)}{133.3}$$

p is the vapor pressure in torr, T is the temperature in °C and A, B, and C are element specific coefficients taken from CRC Handbook of Chemistry and Physics [21]. This equation reproduces the observed vapor pressure to an accuracy of  $\pm 5\%$  or better.

## REFRACTORY LAYER AS OHMIC HEATER

The decision to maintain the electrically heated oven was confirmed by the following benefits of this method:

- Simple and independent heating of the ion source sample
- No requirements for major changes made to the source
- Flexible usage for different kind of ion source sample materials
- Simplicity of production
- A large range of resistance materials and different designs and shapes are possible.

The main advantage of electrical heating is ease of measurement and control as well as cleanliness.

### Basic Concept

The new heating element consists of a ceramic tube which is metallized on the outside by magnetron sputtering. Both ends of the tube are connected to the power supply. While the current flows from one end of the tube to the other, the metallized layer acts as an ohmic heater. The ceramic tube works not only as substrate material but also as a container for the sample material. It was expected that metallization would provide a better thermal conductance to the oven sample holder and gives a steadier heat distribution.

The primary requirements of materials used for heating elements are high melting point, low vapor pressure, high electrical resistivity, reproducible temperature coefficient of resistance, good oxidation resistance, absence of volatile compounds, and resistance to contamination. Other desired properties are good elevated-temperature creep strength, high emissivity, and low thermal expansion. The use of refractory materials (niobium, tantalum, molybdenum, tungsten and rhenium) to create the resistant layer seems to be obvious. Except for two of

the platinum group metals, osmium and iridium, refractory metals have the highest melting points and the lowest

vapor pressures of all metals.

| Symbol | Name       | Atomic number | Melting point [°C] | Boiling point [°C] | Electrical resistivity [ $\mu\Omega$ cm] | Temperature coefficient [1/K] | Linear expansion coefficient [1/K] | max. furnace operating temp. in air [°C] | max. furnace operating temp. (vacuum) [°C] |
|--------|------------|---------------|--------------------|--------------------|--|-------------------------------|------------------------------------|--|--|
| Nb     | Niobium    | 41            | 2468               | 4742               | 16                                       | 0.0026                        | 7.20E-06                           |  |  |
| Mo     | Molybdenum | 42            | 2617               | 4612               | 5.7                                      | 0.0044                        | 5.10E-06                           | 400                                      | 1650                                       |
| Ta     | Tantalum   | 73            | 2996               | 5425               | 13.5                                     | 0.0035                        | 6.50E-06                           | 500                                      | 2480                                       |
| W      | Tungsten   | 74            | 3410               | 5660               | 5.4                                      | 0.0048                        | 4.50E-06                           | 300                                      | 1650                                       |
| Re     | Rhenium    | 75            | 3180               | 5627               | 18.7                                     | 0.0045                        | 6.60E-06                           |  |  |
| Pt     | Platinum   | 78            | 1772               | 3827               | 10.58                                    | 0.00392                       | 9.0x10-6                           | 1500                                     |  |

Table 2: Properties of refractory metals [10]

The refractory metals are readily degraded by oxidizing environments at moderately low temperatures, a property that has restricted the applicability of the metals to low temperature or non-oxidizing high-temperature environments. Some properties of refractory metals are given in Table 2.

Since there are some problems in using tungsten to metallize ceramics by sputtering, as a good adherence is not guaranteed for the moment and rhenium, as a rare material has the drawback that the quantities needed to produce the sputtering target is extremely expensive, tantalum and niobium seem to be the materials for a first test, as they are easily available and niobium is well known for use with the magnetron sputtering technique.

The tantalum has found many applications because of its high melting point and the protective behavior of its oxide. In nature, tantalum is always accompanied by the element niobium that has a very similar chemical behavior. The working characteristics of tantalum are remarkably good for a body-centered cubic metal. However the incorporation of atoms with small radii (for instance, hydrogen, nitrogen or oxygen) into the crystal lattice, considerably reduces the ductility. Because of its high melting point (2996°C), tantalum has a very low vapor pressure and consequently a low evaporation rate at high temperatures. Tantalum parts are easy to machine and fabrication methods with appropriate joining techniques such as welding and brazing are possible. Its unique corrosion resistance is based on the natural formation of a dense oxide surface layer which protects the base metal from many corroding chemicals. Tantalum is inert to many inorganic compounds at room temperature, but with increasing temperature, the corrosion resistance falls. It is also inert to most of the liquid metals up to very high temperatures, but in contrast, oxygen and air react strongly with tantalum at elevated temperatures over 300°C. In high temperature furnaces, unexpected reactions may occur when tantalum is in contact with refractory oxides or graphite. There is a need to pay attention to the fact that even very stable oxides such as alumina, magnesia or beryllia are partially reduced at high temperatures when in contact with tantalum. Contact with graphite causes embrittlement due to carbide formation [11].

Niobium has similar properties to tantalum. It is ductile at ambient temperature, can easily be worked and is very

corrosion resistant. At elevated temperatures, the metal reacts with halogens, oxygen, carbon, hydrogen, and sulfur. It will oxidize in air above 200°C and form a volatile compound. The reaction, however, does not become rapid until it is above 500°C. Niobium forms high-melting point compounds with such elements as carbon, boron, silicon, and nitrogen [10].

Initial tests to metallize a ceramic piece with tantalum resulted in an insufficient adherence of the metal layer. A change of sputtering parameters (increase of voltage and providing higher vacuum) solved this problem so that the adherence was satisfactory.

Due to their use in microelectronics industry, sputtered tantalum films have been the subject of considerable studies in recent time. A high temperature polymorphic tantalum phase transition from the tetragonal beta phase to the cubic alpha phase takes place when tantalum thin films are heated up from 600°C to 800°C [19]. This phase transition causes a change in resistivity and stress behavior. The beta tantalum has an as-deposited resistivity of 170-210  $\mu\Omega$  cm. The resistivity of alpha tantalum is about 15-60  $\mu\Omega$  cm. Introduction of oxygen into the sputtering system also increases the resistivity of the film [20]. Upon heating, the resistivity of the sputter deposited tantalum film (0.1  $\mu\text{m}$  layer thickness) decreases linearly with temperature from about 225  $\mu\Omega$  cm at 25°C to 200  $\mu\Omega$  cm at 600°C. Then it gradually decreases from 600°C to 800°C to 60  $\mu\Omega$  cm [19]. The problem of the decreasing resistance due to the phase transition can be solved by a heat treatment at high temperatures to change the  $\beta$ -tantalum to the non-reversible state of  $\alpha$ -tantalum before using it as a heating element.

Alumina is used as a carrier material for the heating layer as it is easily metallized and provides good adherence. Equally, its high temperature resistance and the non-outgassing behavior in a vacuum environment are advantageous. Alumina is commercially available in a large range of form, quality and in various degrees of purity. Due to the strong chemical bond strength between the Al and O ions, alumina has an outstanding physical stability, such as a high melting point (2050°C), the highest hardness among oxides, and high mechanical strength. Highly pure alumina can be used up to 1700°C and it is gas tight up to 1300°C. Mechanical strength is

high at room temperature but is reduced above 1100°C. Thermal conductivity is relatively high among oxides. However, because the coefficient of thermal expansion is large, thermal shock resistance is small. Additional advantages are the similarity of the thermal expansion coefficient of alumina to that of tantalum and niobium. Alumina is a typical electrical insulator material. The volume resistivity of high-purity ceramics is  $>10^{21} \mu\Omega \text{ cm}$ . Values of elasticity and thermal conductivity increase as purity decreases. Mechanical strength and dielectric strength increase with increasing purity but are more dependent on density and ceramic microstructure. Alumina is relatively hard to machine and diamond tools are needed. It is also chemically very stable and has high corrosion resistance. It is insoluble in water and very slightly soluble in strong acid and alkaline solution. Vapor pressure is low, even at high temperatures. [15].

For connecting the metallized layer to the conducting parts of the oven, brazing seems to be a suitable solution for a reliable contact. It also protects the conducting area from oxidation and corrosion during operation. Problems could appear in finding a brazing filler metal which fulfills the given conditions. This brazing filler must be suitable for the material of the heating layer and the conducting material; it has to withstand high temperatures, show an excellent resistance against corrosion and should have the same coefficient of linear expansion as the joining partners. Furthermore, one has to pay attention during the brazing process to avoid resistance changes from wetting the resistance layer with brazing filler. The danger of contaminating the refractory layer at high temperatures by residual gases in the brazing oven has to be minimized. For potential brazing filler candidates see [18].

An alternative for brazing is to make an electrical contact by clamping metallic rings on each end of the metallized ceramic cylinder. Care must be taken because of the extreme fragility of the substrate ceramic tube. The relatively low thermal coefficients of expansion for refractory metals result in a probable mismatch with these of other metals and alloys. The thermal expansion coefficients of the joint members should match as closely as possible, ideally no more than 1 to  $1.5 \times 10^{-6}/\text{K}$  different. For the best compatibility, fasteners should be of the same material as the refractory metal or alloy joint elements and therefore the employment of tantalum or niobium to fabricate the rings is strongly recommended. These rings are then fixed to the outer heat shield and to the rear metal conductor either by clamping or by welding.

A heat shield made of a refractory metal seems to be necessary to reduce power consumption and when the oven is running at high temperatures, it protects other parts of the source from unwanted heating. It also helps to protect the heating layer from contamination by the evaporated sample material and provides additional mechanical strength to the oven. The suggested material for the heat shield is also tantalum or niobium in order to avoid problems caused by thermal stress.

## Experimental Set-up

A test environment producing similar conditions to those in the source was developed and to ensure the validity of the measured data. This set-up consists of a small vacuum chamber and a pumping station providing a pressure between  $10^{-5}$  and  $10^{-7}$  torr. The temperature is measured by a thermocouple (inside) and an infrared pyrometer (outside). For the pyrometer measurements, the vacuum chamber has a small view port made of Kodial-glass which is transparent for infrared wavelengths between 0.4 and  $2.5 \mu\text{m}$ . Electrical contacts through the vacuum chamber wall, which are able to carry a current of 20 A and an additional feed-through for voltage measurements were also installed. A schematic drawing of the experimental set-up is shown in Figure 2.

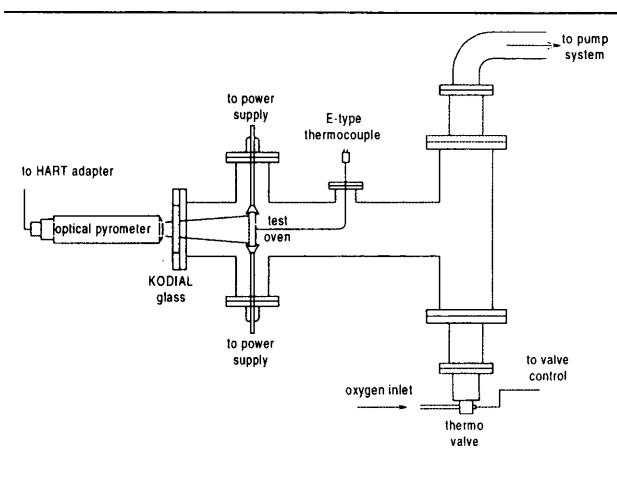


Figure 2: Schematic drawing of experimental set-up

Two pressure measurement units are installed on the experimental set-up. The first one is situated directly in front of the turbomolecular pump and is connected to the pump system control unit monitoring proper operation. The second unit is fixed next to the vacuum chamber to evaluate the actual pressure in the chamber and to provide the thermo valve control unit with an output signal to set up a closed control loop. For a controlled oxygen inlet, a thermomechanical gas inlet valve in conjunction with a valve control unit is used. In order to match different oven resistivities, two adjustable power supplies with different voltage and current ranges are used. The heating element resistance is evaluated by measuring the oven current and the applied voltage using the four wire method.

## Experimental Procedures

The first experiments were carried out to prove the principle of operation and to select a resistance layer material and its required thickness.

A series of ceramic bars were coated with either tantalum or niobium by magnetron sputtering. The ceramic bars were made of pure alumina and measured  $50 \times 10 \times 4.2 \text{ mm}$ . The layers, which were deposited on the upper side of the substrate, varied in their thickness from 1 to 6.5

$\mu\text{m}$ , and were calculated to have a resistance between 400 and 5600 m $\Omega$ . To obtain a proper positioning of the thermocouple, a small hole was drilled into the back of the alumina bar. Some of the test samples underwent a heat treatment. It turned out that the calculated value of resistance differed from the actual value of the metallized layer. This discrepancy occurred, because resistivity values given in literature refer to bulk materials. These values used to calculate the resistance of the heater element differ from the real resistivity values for evaporated and sputtered films. Thin film resistance depends also strongly on the grain size of the coated layer.

Each test oven was fixed in the vacuum chamber by two clamps. The resistance at room temperature was evaluated by measuring the voltage and the current, and also to verify whether the clamped contact was satisfactory. For this purpose, a very low voltage was applied to the test element to avoid a heating of the sample and a resulting resistance change. Then the vacuum chamber was closed and evacuated to its minimum pressure. At pressures below  $1 \times 10^{-5}$  torr, the measurements were started. For these series of tests, no oxygen was admitted to the vacuum chamber. The following pages describe the specific values and properties of each test oven and summarize the results of the measurements.

#### Test-oven 1

This oven had a layer of 1.3  $\mu\text{m}$  Tantalum. Its calculated resistance would be 500 m $\Omega$  unlike its actual resistance of 5.6  $\Omega$  measured at room temperature. For this sample, no special heat treatment was performed. The voltage was slowly increased up to 12 V, which corresponded to a temperature rise to 543 $^{\circ}\text{C}$ , where it was left for some time. As expected, the results showed a linear resistance/temperature behavior up to this temperature (Figure 3, oven 1a).

A change of the oven resistance of 1  $\Omega$  was caused by oxidation of the metallized layer by the incoming atmosphere gas at elevated temperatures due to the pumping system failure. By a further increase of the voltage up to 24 V leading to a temperature of 804 $^{\circ}\text{C}$ , the beta to alpha phase transition took place. It was observed by a decrease of resistivity of 2  $\Omega$  at increasing temperatures from 600 to 700 $^{\circ}\text{C}$ . In the range from 700 to 800 $^{\circ}\text{C}$ , the resistivity slowly increased again. At the temperature of 800 $^{\circ}\text{C}$ , the voltage was held constant for 1 hour, and a further increasing of resistivity was noticed. Slowly decreasing the voltage to 10 V and cooling the

oven to 450 $^{\circ}\text{C}$ , the resistance decreased linearly with falling temperature (Figure 3, oven 1b). During a further measurement cycle on the same oven, the results showed a nearly linear dependence of resistance with rising temperatures which suggests that a heat treatment can be useful to avoid changes in resistance during heating of the oven element (Figure 3, oven 1c).

Figure 3: Measurement results of test oven 1

The first test oven was destroyed by a power failure and the resulting venting of the vacuum chamber. The presence of an oxygen atmosphere at a raised temperature (760 $^{\circ}$ ) resulted in an oxidation and evaporation of the thin metallized layer.

#### Test oven 2

This was coated with a 1.3  $\mu\text{m}$  tantalum layer and underwent a heat treatment after sputtering. The metallized ceramic sample was slowly (+100 $^{\circ}\text{C}/\text{hour}$ ) heated up to 850 $^{\circ}\text{C}$  in a brazing furnace under an argon atmosphere; it was held at the top temperature for 1 hour before it was gradually cooled down (-100 $^{\circ}\text{C}/\text{hour}$ ). As proven by the following measurements, this process induced the irreversible phase transition of the tantalum film.

At room temperature, the calculated resistance of the metallized layer was 500 m $\Omega$ ; the measured is 1.92  $\Omega$ , which is nearly three times lower than the unannealed test oven 1. As expected, the annealing process had caused an alteration in resistance at room temperature. The sample oven was heated up to 1000 $^{\circ}\text{C}$  twice, when it is left for some time at constant voltage. In both cases, the resistance-temperature curve was linear; there was no change of resistance between 600 and 800 $^{\circ}\text{C}$ , indicating that the preceding heat treatment to force the beta-to-alpha transition was successful. During the first measurement cycle (Figure 4, oven 2a), when the oven temperature reached the 1000 $^{\circ}\text{C}$  mark and the voltage was subsequently held constant at 31 V for 1 hour, the resistance slowly increased ( $\Delta R=2 \Omega$ ). Cooling down and measuring the resistance again at room temperature indicated a rise of resistance by 300%. The second measurement cycle (Figure 4, oven 2a) brings similar results, although, the increase in resistance at 1000 $^{\circ}\text{C}$  is less pronounced.

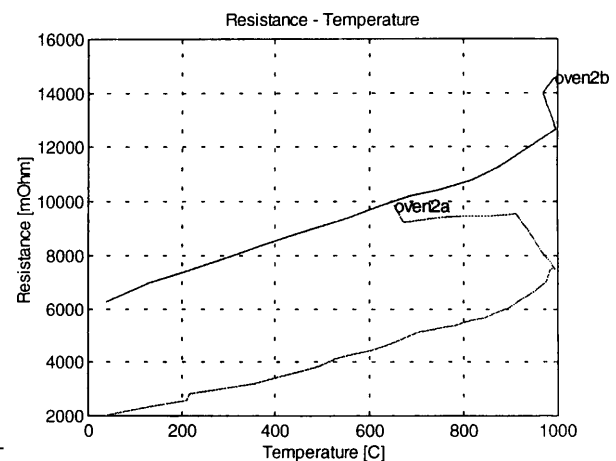
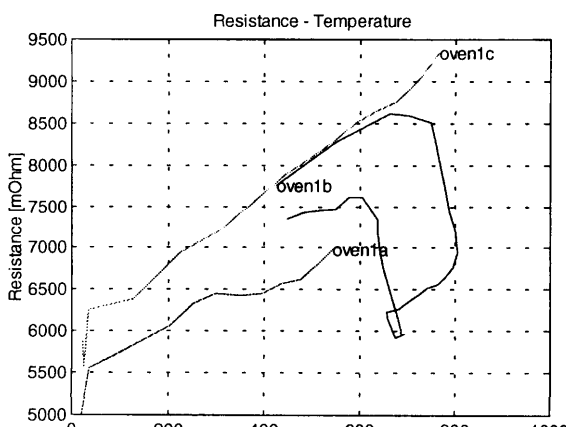


Figure 4: Measurement results of test oven 2

This increase of resistance during a constant applied power is caused by a change in grain size as well as recrystallization, which takes place at elevated temperatures.

Test oven 3

The ceramic substrate of test oven 3 was metallized with tantalum with a layer thickness of 6.5  $\mu\text{m}$ . The calculated and measured resistance was 100 m $\Omega$  and 410 m $\Omega$ , respectively. A heat treatment was not foreseen for this sample. Measurements showed a decrease of resistance between 600 and 800°C (phase transition), an increase of resistivity at 1000°C and constant voltage (alteration in grain size), and a linear behavior of the resistance-to-temperature curve during the second measurement cycle.

For test oven 4, 5, and 6, the alumina substrate was heat treated before metallizing, by slowly heating up to 1000°C step by step (100°C/h) under normal atmosphere pressure. At this temperature, the ceramic was left 2 hours in the furnace. Heat treatment under oxygen atmosphere helps to clean the substrate from carbon and lubricants that are used in manufacturing processes. Carbon particles react with oxygen under the influence of high temperature to produce carbon dioxide, which out-gases from the solid material. After the alumina samples were coated with either niobium or tantalum, another heat treatment was performed to accomplish the phase transition, to provide a better adherence to the substrate, and to increase the grain size of the metallized layer. The annealing temperature for these series was 1400°C for 3 hours.

Test oven 4

This oven (5  $\mu\text{m}$  tantalum layer, annealed, 420 m $\Omega$ ) showed an excellent linear behavior of the resistance versus temperature curve for both measurement cycles and also the increase of resistivity at constant temperatures at 1000°C was visible but less significant than in the experiments carried out earlier (Figure 5).

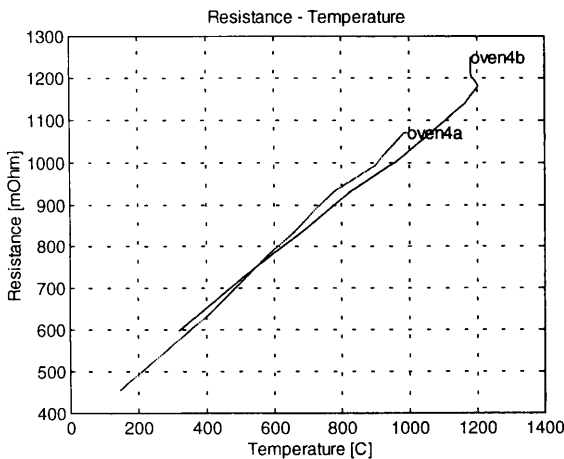


Figure 5: Measurement results of test oven 4

Up to 1000°C, the oven performance was stable (e.g. no noticeable change in resistance) for 20 hours. Upon further heating the oven to a constant temperature of 1180°C, the resistance increased slightly with time and after 11 hours, an area of the heating layer burnt out.

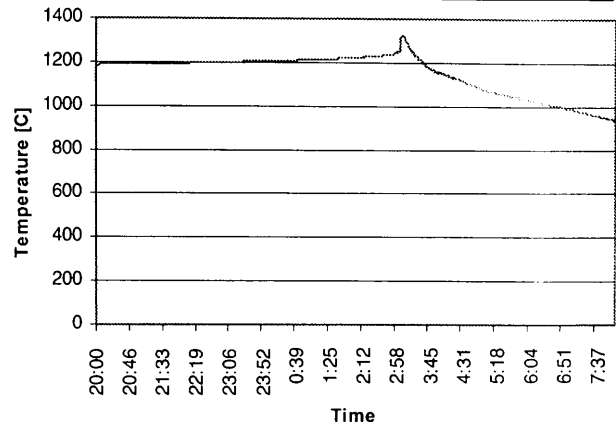


Figure 6: Oven temperature measurement data 4b

From measurement 2 of this series, a pyrometer was employed to evaluate temperatures higher than 1000°C and to ease achievements of long term tests and data recording with a computer aided measuring system. Figure 6 shows the recorded temperature data.

Test oven 5

Oven 5 (0.8  $\mu\text{m}$  niobium layer, annealed, 1.42  $\Omega$ ) was heated up to 1012°C very quickly during 30 minutes. This caused large thermal stresses, which became apparent by the formation of small cracks on the alumina substrate and lead to an oven failure after a short operation time.

Test oven 6

The best performance in high temperature resistance and long term reliability was given by test oven 6, which had a 2.8  $\mu\text{m}$  annealed niobium layer and a resistance of 520 m $\Omega$  at room temperature. The sample oven was tested in three measurement cycles. In the first cycle, the test oven did not show any remarkable changes in resistance when it was held at 970°C for 7 days

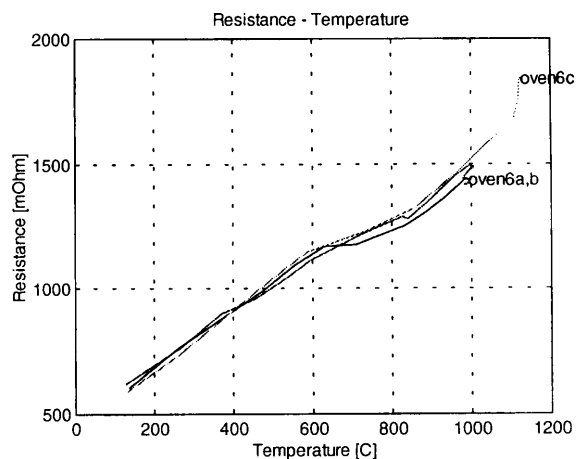




Figure 7: Measurement results of test oven 6

In the following test cycles, no major change in the resistance versus temperature curve could be seen. (6a,b). Finally, the oven was adjusted to a temperature of 1110°C, where a slow increase of the resistance (from 1.68 to 1.83 Ω) was noted. After 50 hours, an oven failure caused by destruction of the central area (hot spot!) of the metallized layer stopped the third measurement cycle (Figure 8).

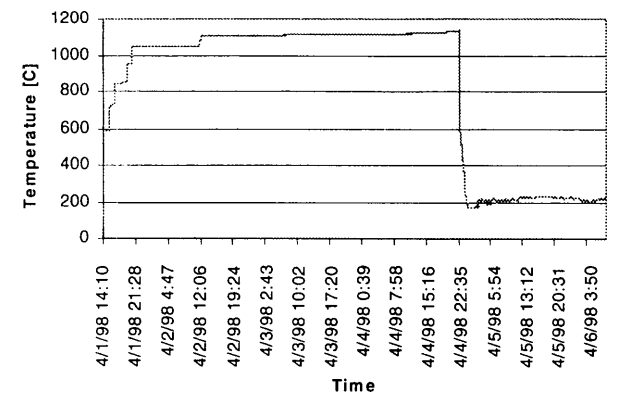


Figure 8: Oven temperature measurement data 6b

These experiments show that a reliable oven for temperatures lower than 1000°C in vacuum using an annealed niobium layer as an ohmic heater is possible. Stability and high temperature resistance is dependent on sputtered layer thickness, material, and heat treatment. As shown, niobium seems to be more suitable than tantalum for the high temperature ranges. The layer thickness should be as large as possible (it is only limited by the required minimum resistance) and annealing of the metallized layer is highly recommended.

The second part of this experimental series served to find out if brazing is possible method to join the heating layer to the oven casing. An alumina tube (1.2 x 2 x 50 mm) was coated with a 10 μm niobium film. This had to be carried out by coating one side of the tube, then turning it around, and metallizing the opposite site. Thus, there is no uniform coating, it is to be proven if such an irregular structure affects performance and lifetime of the heating element, as providing a uniform layer thickness costs a lot of additional effort during the sputtering process. Two niobium rings (2 x 5 x 5 mm) were machined and brazed on the ends of the coated ceramic tube using a gold filler. Gold was proposed for brazing molybdenum [26], but experiments have shown that it is also suitable for joining niobium parts together. Brazing fillers made of gold have a liquidus temperature of 1066°C, and it is expected that they provide an adequate contact even at oven temperatures exceeding 1000°C. Particularly, the ends which are in contact with the casing remain at lower temperatures. Gold was chosen as filler material as it is easy available and the maximum brazing furnace temperature at CERN is limited to 1200°C. As the

brazing process took place under similar conditions to the annealing procedure, an additional heat treatment after metallizing the ceramic tube was not necessary.

#### Test oven 7

It showed a linear increase of resistance with temperature up to 900°C. At this temperature no change in resistance was seen for a period 5 days. Neither end of the oven became glowing red which indicated that the temperature on the contact points were lower than 600°C even when the temperature in the middle of the tube exceeded 900°C. The power consumption to heat this test oven up to 900°C was less than 9 Watts. A failure on the main power and a following automatic venting of the vacuum system lead to a complete oven failure on the 6<sup>th</sup> days of operation due to a rapid oxidation of the metal layer at 900°C.

To avoid these failures in the future, an electro-pneumatic valve was installed between the pumping system and the vacuum chamber. This valve shuts automatically when the pumping system is switched off and the chamber stays under vacuum. Further, a latching relay enables that the heating current is switched off and stays switched off when a short main power interruption occurs.

#### Test oven 8

The measurements were continued on test oven 8, which has exactly the same properties as test oven 7. The first measurement cycle took place in vacuum at a pressure of  $4 \times 10^{-6}$  torr measured at the vacuum chamber. With rising voltage the resistance of the heating element increased linearly (see Figure 9, oven 8a).

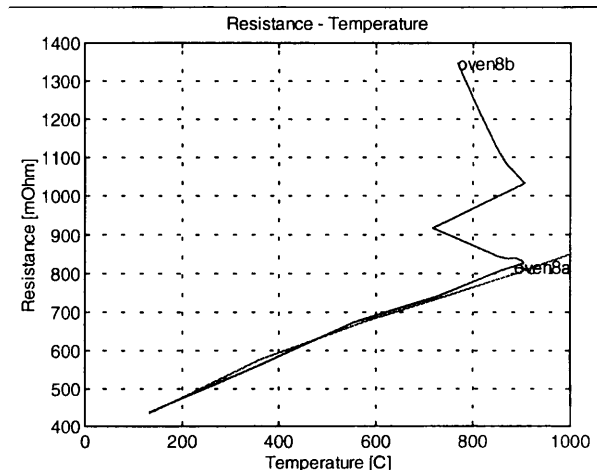


Figure 9: Measurement result of test oven 8

The power consumption at 1000°C was 10.5 Watts. The oven was left on at the voltage of 2.6 Volts for 2 days. After this period an increase of the resistance and a resulting 3% decrease of the heating temperature was noted

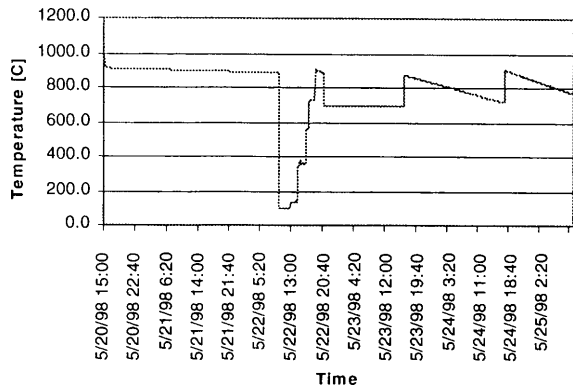


Figure 10: Oven temperature measurement data 8

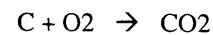
The second measurement cycle was carried out under oxygen atmosphere. After the first cycle, the oven was cooled and heated again in vacuum better than  $2 \times 10^{-6}$  torr. When the temperature of  $904^{\circ}\text{C}$  was reached, the thermo valve was opened to maintain a constant oxygen atmosphere at  $9 \times 10^{-6}$  torr. During 2 hours the resistance increased and the temperature decreased from 904 to  $885^{\circ}\text{C}$ . The oven was left at lower temperature ( $696^{\circ}\text{C}$ ) without an additional oxygen atmosphere for the night and no changes in resistance occurred. The following measuring period of 24 hours was carried out under  $5 \times 10^{-5}$  torr oxygen pressure with a starting oven temperature of  $873^{\circ}\text{C}$ . At a constant voltage of 2.7 V the results showed a resistance increase of approximately 100 m $\Omega$  and a decrease of temperature of  $157^{\circ}\text{C}$ . Then the voltage was adjusted to 3.75 V where it was left constant for another 18 hours. After this period a temperature decrease from 905 to  $767^{\circ}\text{C}$  and an increase of the resistance of 300 m $\Omega$  appeared (see Figure 10).

These experiments indicated that the major problems are caused by oxidation of the heating element at elevated temperatures. Precautions to avoid or minimize a direct contact of oxygen or the metal vapor with the heating element are indispensable. A protective coating, which requires further investigations, could be a suitable solution for this type of oven.

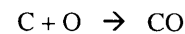
## GRAPHITE AS OHMIC HEATER

### Basic Concept

Since graphite has a very high volume resistivity, an extremely low vapor pressure and supports high temperatures, it is an alternative to use it as a resistance material for an ohmic heater. Even at 4000K the vapor pressure of the three gaseous species of carbon ( $\text{C}$ ,  $\text{C}_2$ , and  $\text{C}_3$ ) is less than atmospheric pressure. Graphite remains solid over this entire temperature range. A disadvantage is its readiness to react chemically with ceramics and oxygen. Oxygen, which is used as the support gas in the source, can cause severe problems for the use of graphite as it is present in the plasma chamber at low pressure. The reaction equations:



and



show the production of carbon monoxide and carbon dioxide, consuming and reducing the oven material. The resulting gas is pumped out of the plasma chamber, but it can also contaminate the extracted ion beam.

The resistivity of graphite is between 500 and 3000  $\mu\Omega$  cm depending on the type of graphite used. The resistance of graphite is a function of temperature. Initially it decreases with temperature, reaches a minimum around  $500^{\circ}\text{C}$  and then rapidly increases. This requires that the power supply should be capable of varying voltage over a wide range in order to maintain the required power as the temperature changes. This is especially true at low temperatures where the resistance decreases with increasing temperature.

As noted above, graphite reacts readily with oxygen at temperatures above  $400^{\circ}\text{C}$  to form carbon monoxide (CO), making it imperative that oxygen is excluded from the furnace atmosphere. The graphite heating elements can also be protected by some high temperature protection coatings such as there are silicon carbide (SiC), tantalum carbide (TaC), niobium carbide (NbC), zirconium carbide (ZrC), or pyrolytic boron nitride (PBN). Silicon carbide coatings are made by two processes: Chemical Vapor Deposition (CVD) or Chemical Vapor Reaction (CVR). These coatings (see Table 3) not only protect graphite from wear and chemical attack, but also prevents gas desorption and loss of graphite to the surroundings.

| Name                    | Symbol    | Density [g/cm <sup>3</sup> ] | Coating Temperature [ $^{\circ}\text{C}$ ] | Melting Point [ $^{\circ}\text{C}$ ] | max. Service Temperature in air [ $^{\circ}\text{C}$ ] | Electrical Resistivity [ $\mu\Omega$ cm] | Linear Expansion Coefficient [1/K] | Thermal Conductivity [W/mK] |
|-------------------------|-----------|------------------------------|--|--------------------------------------|--|--|------------------------------------|-----------------------------|
| Silicon carbide         | SiC (CVD) | 3.2                          | 1200-1400                                  |                                      | 1000   | 1000                                     | $4.8 \times 10^{-6}$               | 110                         |
| Silicon carbide         | SiC (CVR) | 3.17                         | 1800-2000                                  |                                      | 800  | 1-100                                    | $5.6 \times 10^{-6}$               | 30                          |
| Tantalum carbide        | TaC       | 15                           | 2100                                       | 3880                                 | 700  | 30                                       | $6.3 \times 10^{-6}$               | 22                          |
| Niobium carbide         | NbC       | 7.9                          | 2100                                       | 3760                                 | 800  | 30                                       | $7.0 \times 10^{-6}$               | 13                          |
| Zirconium carbide       | ZrC       | 6.7                          | 2100                                       | 3540                                 | 800  | 50                                       | $6.7 \times 10^{-6}$               | 21                          |
| Pyrolytic boron nitride | PBN       | 1.95-2.22                    | 1600-2000                                  | 2500                                 | 800  | $10^{+16}$                               | $2.0 \times 10^{-6}$ (a)           | 20-120 (a)                  |

Table 3: Properties of protective graphite coatings [13]  
(a) parallel to the layer planes

Graphite is particularly difficult to join. Probably mechanical fastening is the most common method of joining graphite, particularly to other materials and especially to metals. Three problems must be dealt with when mechanically fastening graphite to one another or to other materials:

- Physical damage to the carbonaceous substrate by fastening loads
- Thermal stresses from mismatch of the thermal expansion coefficient
- Corrosion or oxidation of the fastener

To prevent galvanic corrosion and high-temperature oxidation, fasteners are usually fabricated from titanium alloys. Oxidation-resistant coatings are also frequently used.

The two major difficulties to cope with in brazing carbonaceous materials are achieving wetting and dealing with thermal expansion. Graphite is inherently more difficult to wet with the more common brazing filler metals. Two techniques are used to overcome this wetting difficulty. First the graphite is coated with a more readily wettable layer, such as a 0.2 to 6  $\mu\text{m}$  thick layer of molybdenum or tungsten applied by chemical vapor deposition. This coated or metallized material can then be brazed. Second, brazing filler metals containing strong carbide forming elements, such as chromium, titanium, or zirconium, can be employed. Such elements react with carbon to form carbides, and bonding is achieved. Several brazing filler metals have been developed for brazing carbonaceous materials [18].

The heater element would consist of a cylindrical graphite tube. A rough calculation of a tube with dimensions 2 x 3.5 x 40 mm shows that the resistance would be less than 100 m $\Omega$ . To avoid too high currents, the resistance of the oven should be increased. To do this without affecting the cylinder wall thickness, the tube could have grooves machined into each side of the tube. Since alumina reacts less with carbon than other oxides, it is suggested as a ceramic insulator and as a basis for the sample material container. Materials for the connection to the graphite tube must be chosen carefully, so as not to form unstable carbides. Possibilities are tungsten or molybdenum with the preference on molybdenum as it is easier to machine.

### **Experimental Procedures**

For this series of experiments, the same experimental set-up was used as for the ovens with refractory layers. The only required modification was a change of the sample holders to provide a good electrical contact to the graphite tubes.

#### *Graphite test oven 1*

This oven was made of a 44 mm long graphite tube with an interior diameter of 2.1 mm, to hold a small ceramic tube, and an outer diameter of 3.5 mm. For electrical connections, a molybdenum ring was pressed on each tube end. The resistance of this oven was 140 m $\Omega$ . For the first test, the graphite oven was heated very slowly to a temperature of 740°C where the current limit of the power supply was reached. The results indicated a completely stable operation during a period of 13 days.

To increase the resistance and stay at the required wall thickness, 7 grooves were machined into the tube. This increased the resistance by more than 20% to 170 m $\Omega$ . The second measurement cycle gave the results that stable working performance at 870°C is possible for 7 days.

A third measurement cycle, which was carried out in an oxygen atmosphere of  $6 \times 10^{-4}$  torr, ended in an oven failure after 5 days of unstable operation. In the central hot zone a large amount of graphite was consumed by the reaction with oxygen which led to a visible shrinkage of the cylindrical wall. Consequently, the resistance, and the temperature, increased around the hot zone which accelerated the oxidation process until the oven failed.

#### *Graphite test oven 2*

It measured 48 mm in length, 2.1 mm in inner diameter and 3 mm in outer diameter. No slits were machined into the tube, since these grooves make the oven too fragile for the use in the experimental set-up. To achieve higher temperatures despite limited current output of the available power supply, these oven dimensions have been calculated to provide a resistance of 170 m $\Omega$ .

Graphite oven 2 was tested in a atmosphere of  $2 \times 10^{-5}$  torr oxygen pressure where it shows good performance for 14 days at temperatures around 800°C (see Figure 11, oven 10).

During a test period of 340 hours no decrease or increase of the resistance could be measured and the temperature of 805°C maintained stable ( $\pm 0.3\%$ ) at the constant voltage of 2.2 Volts. The required power was 47 Watts. The molybdenum rings provided a good electrical contact to the graphite tube and no sign of unreliability occurred.

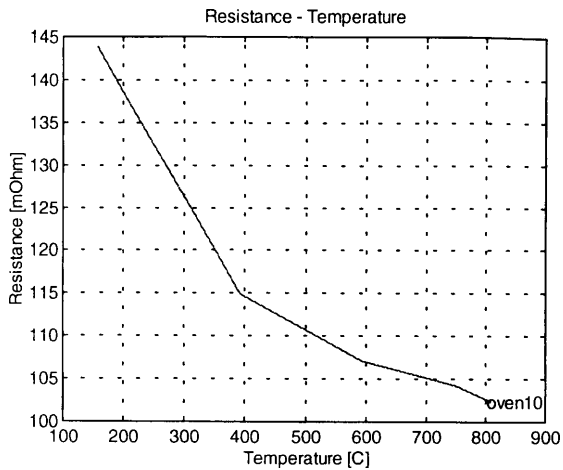


Figure 11: Measurement results of 'Graphite oven 2'

The graphite oven seems to be a suitable solution to achieve elevated temperature in an oxygen atmosphere up to  $2 \times 10^{-5}$  torr as it is found in the plasma chamber of the ECR ion source at CERN. To attain higher temperatures than 800°C or improve long term reliability a protective coating is suggested to prevent the oven from unwanted oxidation and to avoid reducing the oven wall thickness by evaporation.

### Proposed Prototype

A graphite tube, measuring 40 mm in length, 2.1 mm in interior diameter and 3 mm in outer diameter is the essential part of this oven design. The tube cavity is designed to carry a smaller alumina sample holder (37 x 1.2 x 2 mm). This inner tube can carry an amount of 480 mg lead, filling which can last for an uninterrupted source run of 1600 hours or two month.

As mentioned above, 12 machined microgrooves result in an increase of the resistance so it is possible to maintain the length and the wall thickness of the oven and increasing the resistance.

At the front, the tube is connected to the outer metal heat shielding tube with a molybdenum ring. The other electrical connection is made by a cylindrical molybdenum part which is pressed into the bore of the graphite cylinder. A small alumina ring on the back end holds the graphite part in its correct position. For this type of oven, there is no need to modify the actual source construction. For a detailed drawing of the proposed oven see Appendix

The price for one prototype oven is estimated to be around 500 CHF, (the working hours for machining the graphite element, for manufacturing the contacts, and for assembling the oven are included).

The power to attain a temperature of 500°C falls into the 20 Watts range. To get to 1000°C, an 80 Watts power supply will be required. Because of the relatively low resistance of the graphite heater, a power supply with low output voltage (<10 Volts) but a high output current (>30 Ampere) is necessary.

### OUTLOOK

There exist also some other ideas to cope with the oven problem. Pyrolytic graphite (heating element) in combination with pyrolytic boron nitride (substrate material) as it is provided by Advanced Ceramics Corp. could be an interesting material for the employment in a micro-oven. These materials are already used in a number of high temperature furnace applications, since they have extraordinary characteristics at elevated temperatures.

Beside normal graphite types there is a special kind of graphite, called pyrolytic graphite (PG). This material is a unique form of graphite manufactured by decomposition of a hydrocarbon gas at very high temperature in a vacuum furnace. The result is a highly pure product which is near theoretical density and extremely anisotropic. This anisotropy results from a layered structure. Most properties of PG in the direction parallel to the planes are different from that perpendicular to the planes. Mechanical, thermal, and electrical properties are generally far superior to conventional graphite. PG is highly chemical inert, has a high purity, is stable to 3000°C, and is non-dusting. Its application is found in sputtering targets, ion beam grids, thermal isolators, crucibles for ultra high vacuum, and heater elements [13].

Hexagonal boron nitride exhibits a strong anisotropy. Thermal and electrical conductivity such as other properties parallel to and perpendicular to the planes are substantially different. Boron nitride is an opaque white, non-toxic, non-porous compound of boron and nitrogen. The electrical resistivity is high, even at temperatures to 1600°C where it exceeds  $10^{11} \mu\Omega \text{ cm}$ . Boron nitride is a refractory material and its thermal shock resistance is also very good. It is inert to almost all reagents at room temperature and to a large number of reagents, metals, and compounds at temperatures from 1000 to 2000°C. It does not react with graphite at 2000°C and only slightly with tungsten at 2000°C. Boron nitride can be exposed to oxygen indefinitely at temperature up to 750°C. Oxidation rates at higher temperatures are limited by the formation and slow volatilization of  $\text{B}_2\text{O}_3$  from exposed surfaces [26].

For further investigations on materials for a new oven, low resistance ceramics, particularly silicon carbide and molybdenum disilicide which can be used as an ohmic heater should be mentioned as well. Properties of ceramics often vary in a wide range depending on their composition, purity, and manufacturing process. Values for ceramic properties that are given in this article are only approximate values from the referenced literature.

Silicon carbide is commercially available in numerous shapes. It has a negative temperature coefficient of resistance at temperatures ranging from 500 to 750°C. The resistance drops by a factor of two, then increases in a near linear fashion to twice its room-temperature value at 1500°C. Because the resistivity of silicon carbide is two orders of magnitudes higher than graphite, higher

voltages and lower currents can be used. Silicon carbide is extremely brittle, but since its resistivity is so high, silicon carbide elements can be made in large cross sections in order to reduce resistance and increase strength. Silicon carbide however has an excellent oxidation resistance and can be used at temperatures up to 1600°C. The elements undergo an aging phenomenon time caused by intergranular oxidation in which their resistance slowly increases with [12].

The other common non-oxide ceramic-heating element is molybdenum disilicide ( $\text{MoSi}_2$ ). It has a maximum operating temperature of 1800°C. Pure molybdenum disilicide is too brittle for reasonable use. The addition of metallic and ceramic binding agents reduces this brittleness to a practical level. The resistivity of molybdenum disilicide is between 27 and 37  $\mu\Omega$  cm at room temperature and increases nearly linearly to 400  $\mu\Omega$  cm at 1700°C. Molybdenum disilicide has a high resistance to thermal shock. Its excellent high-temperature performance is brought about by the chemical reaction that takes place on the surface: above 980°C, the material reacts with oxygen to form a silicon dioxide (quartz glass) coating which protects the base material against chemical attack, including further oxidation. This film is "self-healing": surface cracks developed by mechanical damage are covered by a new coating of quartz glass when the element is again heated above 980°C in an oxygen atmosphere. The breakdown of this coating limits the maximum operation temperature, which is highest in air and lowest in reducing atmospheres. Molybdenum disilicide is usually not used in vacuum. Their maximum operating temperatures are 1200°C at  $10^{-3}$  torr and 1450°C at 1 torr [12].

Another type of non-oxide ceramics which are attractive for the application as heating elements are borides. Boron reacts with many elements to form a wide variety of compounds. The strong covalent bonding of most borides is responsible for their high melting points, moduli, and hardness values. The coefficients of thermal expansion for borides are in the moderate to high range for ceramics. A number of borides have thermal expansion coefficients in the 6 to 8  $\times 10^{-6}$  / K range at temperatures between 20 and 1000°C. The generally high thermal conductivity of borides gives many of them a high thermal shock resistance. Most borides are excellent electrical conductors with resistivities in the range of 5 to 80  $\mu\Omega$  cm. The chemical resistance of borides is superior to most ceramics, and the oxidation resistance of borides, especially of  $\text{HfB}_2$ -SiC composites, is excellent at elevated temperatures [14].

Joining ceramic to an electrical contact can be realised by mechanical fastening. The same material considerations have to be respected as mentioned above concerning the mechanical fastening of refractory metals. Ceramics are also brazeable. A number of brazing processes for ceramics apply a metal layer to the ceramic, referred to as metallizing, with subsequent brazing to join the metallized ceramic to another

metallized ceramic or to a metal. By metallizing the surface of the ceramic by depositing or embedding metal by electroplating, sputtering, ion-implanting, or some other means, brazing with a metal filler can be accomplished as it normally is with metal substrates, that is, by selecting a filler that is compatible with the metallized surface material. Another method of brazing ceramics is called metal brazing, which uses metallic materials as filler without employing a metallized intermediate layer on the ceramic [17,18].

Last but not least, heating the ion source sample material with an infrared light beam seems to be a promising and reliable idea for future improvements and should therefore be followed.

## CONCLUSIONS

The main purpose of this paper is to give an overview of the wide range of possible improvements for an ion source sample oven and it was decided to maintain the micro-oven method. A newly designed oven was proposed and the theoretical and experimental work has demonstrated that an improvement of the existing oven is possible, although some modifications of the specifications and the desired demands were needed.

It appears to be rather difficult to satisfy all necessary conditions, but good results were achieved using a machined graphite tube, both working as an ohmic heater. With that proposal, it seems to be possible not only to improve the reliability of the ion source sample oven but also provide a high temperature operation to vaporize elements with lower vapor pressure than the presently used lead.

Experiments to prove the principle of operation and to select the resistant material for a heating layer made of refractory metals were carried out. A heat treatment of the substrate before (to clean the ceramic) and after coating (to force a phase transition) is required for tantalum as well as for niobium. For the high temperature ranges, niobium is more suitable than tantalum. The niobium coated heating element can be used to reach temperatures up to 1000°C in vacuum ( $6 \times 10^{-6}$  torr). For higher temperatures and for the use in an oxygen atmosphere as in the ion source plasma chamber ( $2 \times 10^{-5}$  torr) a coating of the metal layer is required to protect it from oxidation.

A better performance at elevated temperature was shown by the graphite test ovens. In vacuum at the pressure of  $6 \times 10^{-6}$  torr there appears no change of resistance up to a temperature of 900°C. In a  $2 \times 10^{-5}$  torr oxygen atmosphere, a graphite heating element can be used to achieve temperatures up to 800°C reliably, but at a pressure of  $6 \times 10^{-4}$  torr it becomes very unstable and cannot be used as a ohmic heater without protection from oxidation.

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**APPENDIX**

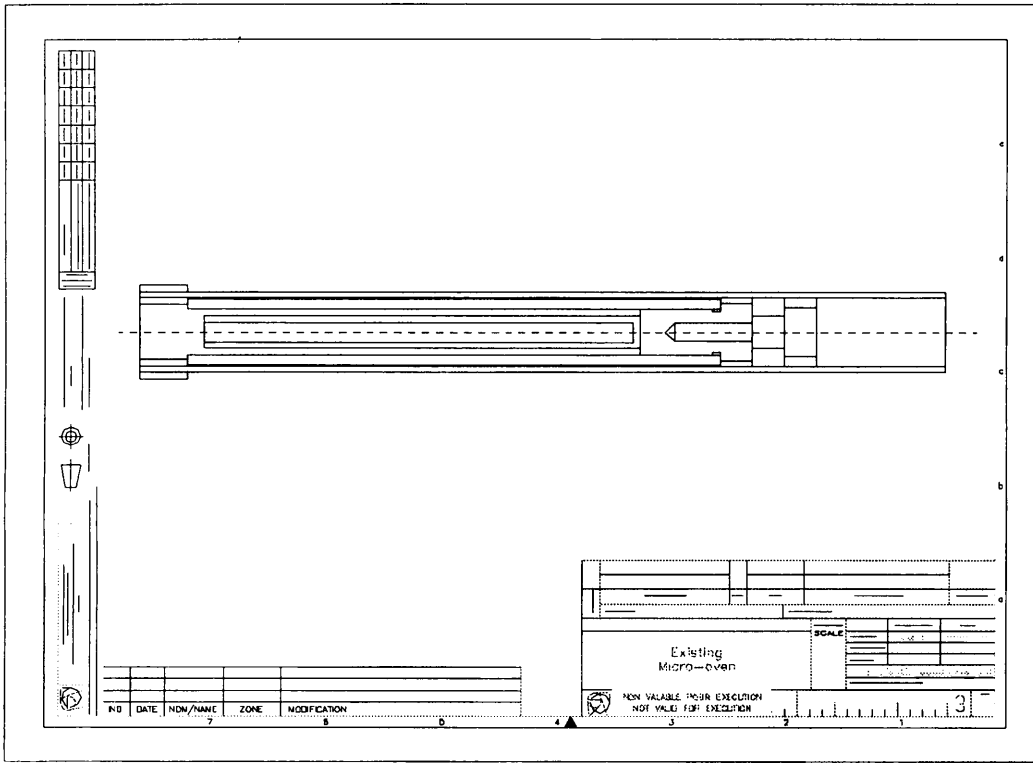


Figure 12: Existing oven

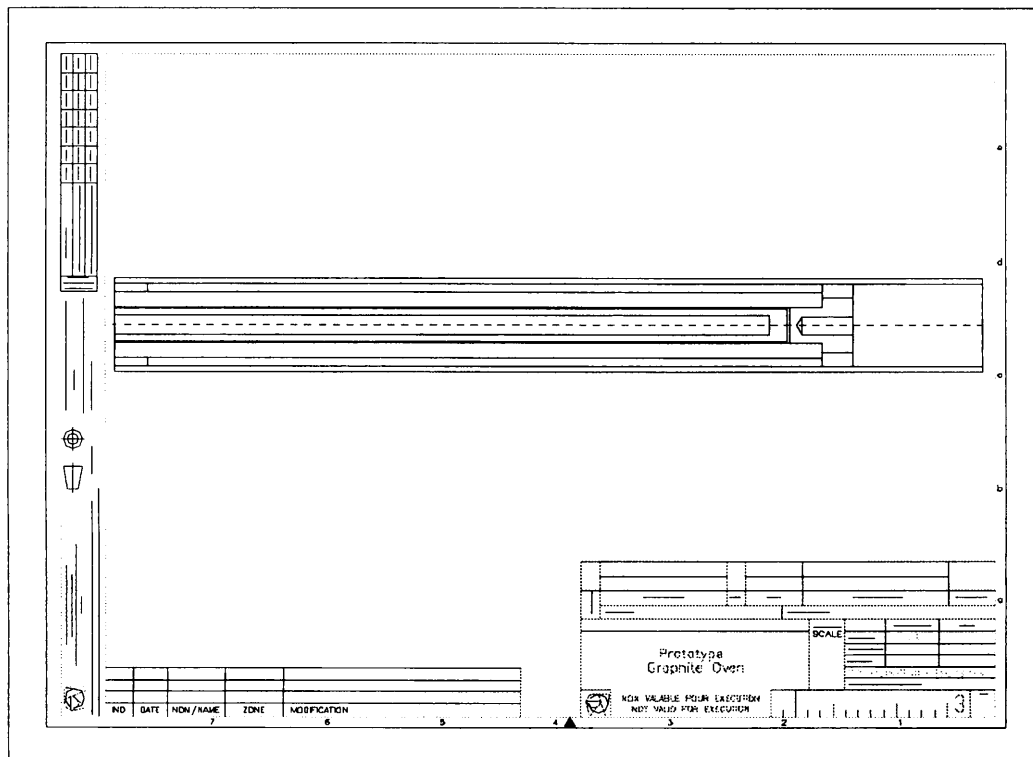


Figure 13: Proposed Graphite-Oven