Research on Metallic Ion Beam Production at IMP^{*}

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Abstract Since 1998, many experiments for metallic ion production have been done on LECR2 (Lanzhou ECR ion source NO.2), LECR3 (Lanzhou ECR ion source NO.3) and SECRAL (Superconductiong ECR ion source Advanced design in Lanzhou) at Institute of Modern Physics. The very heavy metallic ion beams such as those of uranium were also produced by the plasma sputtering method, and supplied for HIRFL (Heavy Ion Research Facility in Lanzhou) accelerators successfully. During the test, $11.5 \mu AU^{28+}$, $9 \epsilon \mu AU^{24+}$ were obtained. Some ion beams of the metal having lower melting temperature such as Ni and Mg ion beams were produced by oven method on LECR3 too. The consumption rate was controlled to be lower for ²⁶Mg ion beams production, and the minimum consumption was about 0.3mg per hour. In this paper, the main experimental results are given. Some discussions are made for some experimental phenomena and results, and some conclusions are drawn.

Key words ECR, metallic ion, sputtering, consumption rate

1 Introduction

Electron Cyclotron Resonance (ECR) ion source^[1] is one of the most important instrument in the field of cyclotron accelerator and atomic physics. ECR ion source can almost provide all ion beams for the experiments of nuclear and atomic physics research. It is one gas discharging kind ion source, so it is very important to provide gas materials. But most of the metals and their compounds are solids, it is very hard to provide their gasifying elements for ECR ion source. There are several methods to provide gaseous metal for ECR ion source, such as inner oven heating, MIVOC (Metallic Ion from Volatile Compounds)^[2], plasma sputtering^[3], and so on.

Since 1997, we have researched the production of

metallic ion beams on LECR2^[4]. We had successfully developed a kind of small inner cavity micro-oven and a kind of big inner cavity micro-oven whose temperature can be increased up to more than 1800K. With the help of these ovens, many metallic ion beams were successfully extracted from LECR2, LECR3^[5] and SECRAL^[6]. For high melting point temperature metals, we have designed a kind of high temperature micro-oven. We have also obtained some ion beams of refractory metals by plasma sputtering method. Uranium ion beams were provided for HIFFL in a accelerator machine research experiment. In addition, some good results were also obtained by MIVOC method. Metallic ion beams obtained from the ECR ion sources at IMP are given in Table 1.

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Table 1.	Metallic ion	beams at	IMP.	Unit:	eμA.	

	40 Ca	^{26}Mg	$^{207}\mathrm{Pb}$	64 Zn	$^{63}\mathrm{Gu}$	⁵⁸ Ni	58 Ni [*]	Fe	Fe^*	$^{238}U^{\#}$	$^{181}\mathrm{Ta}^{\#}$
7+		50									
8 +		70									
10 +					26		64	74			
11 +	132 [287]			26	42			30	210		
12 +	76 [258]					30	74	25	175		
13 +	208			50	39		57	20.5	142		
14+	30 [162]				34	40					
15 +				30	10	59	31	19			
16 +	[75]					78	17		25		
17 +	[36]		40								
18 +											
19 +	[2.1]										
24 +			25							9	12
25 +			[180]							9.6	20
26 +											21
27 +			28							9.2	
			[173]								
28 +			21							11.5	10
29+			[143]								8.8
30+			18 [90]								
32 +			[32]							5	
33 +			13							-	2
35 +										1.5	
37 +			6.7								
40+			0.3 [0.7]								

Note: The data in "[]" were the experimental results on SECRAL by the big inner cavilty micro-oven, the data signed "*" were obtained by MOVIC method and the data signed "#" were obtained by ion plasma sputtering method.

2 Inner micro-oven technique

The inner micro-oven technique is the most common method to provide metallic vapor for ECR ion source. Crucible is usually made of Al_2O_3 , and the tantalum thread is winded around the ceramic crucible. When oven is inserted into the plasma chamber and placed at the area next to the plasma, the metal materials are evaporated and the metal vapor gas will diffuse to the main plasma and will be ionized there. A kind of small capacity oven was first designed and installed on LECR2 and LECR3 ion sources to produce many metallic ion beams^[7, 8]. But some shortcomings of the small oven were found in the experiments: the capacity of the oven was a little small; the maximum working temperature that this kind of oven can provide was less than 1600K; some electric connections were not good.

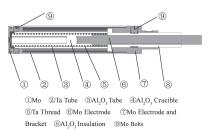


Fig. 1. The structure plot of the new big oven.

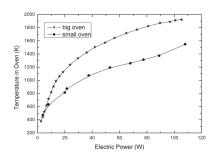


Fig. 2. The contrast of the temperature curves.

A new kind of big capacity oven was developed and the typical parameters were improved. The structure plot is shown in Fig. 1, and the temperature curves of both the big and small cavity ovens are shown in Fig. 2. The experimental results were very good on LECR3 and SECRAL with the big cavity oven installed.

2.1 Operation of the oven

To produce metallic ion beam, enough metallic vapor must be produced by heating with the microoven. In many experiments, we found the optimized working temperature of the oven didn't reach the melting point of some metals, while for other metals the working temperature should be increased much higher than their melting points.

For those metals that need the oven working temperature higher than the melting points, the typical examples are calcium and magnesium. When ion source is under baking, the oven's temperature usually is kept as lower as possible than the melting points of the metal. There are two important demands to handle the metal material. One is that the metal material must be shaped as granules or slices. The diameter of the granules are usually from 0.5to 1.5mm, and the metal slices are usually with the thickness of 0.5 to 1.0mm and the length of 1 to 3mm. If the metal granules are too small, their surfaces are easy to be oxidated. The melting points of the oxides are much higher than the metal, so the evaporation will be minished and intense metallic ions production will be suppressed. The other important demand is that the loaded metal material quantity must be suitable. The oven must not be filled with too much metal, otherwise the outer metal surface of the filled cavity will be too tight and the vapor gas will be suppressed and can not flow freely in the oven.

For the metals that need an oven working temperature lower than the melting points, they are lead, copper and zinc, etc. Their metallic granules may be very small or just in power. It is worth mentioning that the temperature distribution inside the oven cavity is uneven and the temperature at the mouth of the oven is normally lower that that inside of the oven. When the oven is filled with the metal material, the temperature at the mouth might be lower than the melting points while the inner temperature is higher than the melting point of the metal which can result in coagulation and the exit of the oven might be blocked. As a result, the metallic vapor can't flow out of the oven, and therefore the ion source can not obtain enough vapor gas to strip and produce the metallic ion beams. So, the oven should not be fully loaded with the metal material, and some space must be left next to the mouth of the oven.

2.2 Metal consumption rate

Metal consumption rate is one of the most important aspects in metallic ion beam production research. It is tighly related with the ion beam intensity that can be delivered to the accelerators, especially for some expensive isotope metals. The metal material consumption rate is generally determined by the operation state of the oven and the condition of the ion source. When the oven is carefully tuned and the working condition of the ion source is very stable, the smallest consumption can be possibly realized. In 2006, ²⁶Mg⁸⁺ ion beam was supplied for HIRFL, and the minimal consumption rate obtained was 0.3mg/h, and the ion source kept working in this state for HIRFL accelerators for about 102.5 hours. Table 2 gives all the typical consumption rates of the metal material which had been used to produce metallic ion beams for HIRFL accelerators these years.

Table 2. Consumption rate of some typical metals.

metal	consumption rate/(mg/h)
Ca	0.5 - 0.7
^{24}Mg , ^{26}Mg	0.3 - 1.9
Pb	0.7 - 1.5
Ni	3.5

3 Plasma sputtering method

The experimental equipment includes a negatively biased metal sample positioned close to the ECR plasma. The sketch of the experimental equipment is shown in Fig. 3. A metallic sample is inserted into the plasma chamber from the injection side of ECR ion source. The positive ions from the plasma are accelerated by the negatively biased high voltage toward the sample and impact upon its surface and some atoms are bombarded into the plasma. These atoms can be ionized to high charge state by the step by step stripping. Finally, part of these produced metallic ions are extracted from ion source, and we will get the ion beam with the charge state we want. The holder of the sample is water cooled to prevent the stainless steel rod from being melted.

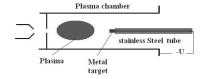


Fig. 3. The sketch plot of the plasma sputter equipment.

The plasma sputtering technique has been tested successfully with Ta and U. The sputtering ions energy is usually several thousand electron volts, so the physical mechanism is the linear cascade collision model^[9]. In the theory, the sputtering yield is enhanced when the ionic energy is higher. Usually, the sputtering yield reaches maximum when the ionic energy is close to 10keV. The phenomenon was observed during the experiments. When lower charge state ion beams were optimized, the highest ion energy was reached with the sample negatively biased to two thousand volts. In the experiments, we optimized tantalum and uranium ion beam production with oxygen and argon as sputtering gas respectively. In principle, the sputtering efficiency of argon is higher than that of oxygen, so more metallic atoms can be sputtered into the plasma and the experiment results is better^[10]. But, actually, the results with oxygen as sputtering gas were much better than the results with argon as sputtering gas. We thought that the sputtering gas oxygen has better mixing gas effect as well as sputtering effect. When oxygen was used as sputtering gas, the best results were $20\mu A \text{ Ta}^{25+}$, $8.8\mu A$ Ta²⁹⁺, $9\mu A^{238}U^{24+}$, $11\mu A^{238}U^{28+}$. The spectra when optimizing the production of Ta^{29+} and $^{238}U^{28+}$ are shown in Fig. 4 and Fig. 5 respectively.

The experiments were done in which the sputtering target was placed at different distances from the position of the maximum axial field at the injection side. But no obvious effect was observed. Actually, metallic sputtering yield is mostly decided by sputtering ionic energy. The change of the sputtering target position may only affect the loss of the sputtering yield a little. If the sputtering target was placed at too deep position into the plasma, the plasma could be affected. It was found in the experiment that the plasma became unstable when the inserted position was beyond 30mm behinde the injection magnetic peak.

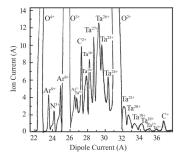


Fig. 4. The ion beam spectrum, optimized for Ta^{29+} .

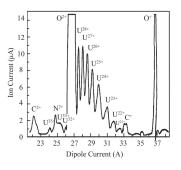


Fig. 5. The ion beam spectrum, optimized for U^{28+} .

4 MIVOC technique

The MIVOC technique was first developed in Finland as early as 1994^[2]. In the MIVOC technique, solid state volatile metallic compounds can produce vapor gas having metallic atoms even at room temperature. Compounds are placed in a separate vacuum chamber connected to the plasma stage of the ECR ion source. When the vapor of the compound diffuses into the plasma from the vacuum chamber, decomposition and ionization of the compound then takes place in the gas-mixed plasma. Thus the metallic ion beams are achievable.

Since 1998, we have done a lot of experiments producing metallic ion beams by the MOVIC technique. Good results were obtained on LECR3 ion source, and Fe and Ni ion beams were extracted with ferrocene $[Fe(C_5H_5)_2]$ and nickelocene $[Ni(C_5H_5)_2]^{[11]}$. The sketch of the experimental equipment is shown in Fig. 6.

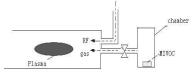


Fig. 6. The sketch of the experimental equipment.

In our experiments, we found it was very important that the vacuum chamber should be pumped before it was connected to the ion source. Usually, a small vacuum system was connected with the vacuum chamber which was loaded with the volatile compounds. Some vapor (composed of some air, some moisture, some metallic gas coumpound and also some decomposed components) was pumped out of the chamber and normally about 10^{-5} mbar vacuum could be obtained. Then, stop the pumping, stable and dense metallic compound vapor was produced, and intense metallic ion beams could be extracted. Several experiments done with nickelocene without using the process failed to produce intense nicel ion beams in our lab. Sometimes, to enhance the vapor of the volatile compounds, it was very necessary to heat the chamber with a heating system.

Additionally, nickelocene was not very stable, and it was easy to decompose into other components which might not be volatile. So, the volatile sample should not be placed in the vacuum chamber for too long time when the experiment was over. Furthermore, the vapor of nickelocene was less than that of ferrocene under the same temperature. In our experiments, the results of nicklelocene were far worse than those of ferrocene. It is noticeable that nickelocene has stronger toxicity, so we must be very careful while operating it. Aeration is also very important.

5 Conclusion

Some good metallic ion beam production results were obtained by several special techniques at IMP. The MOVIC technique is very simple and convenient, and higher current metallic ion beams can be obtained. Yet, it is very evident that the micro-oven technique is suitable for producing higher charge state metallic ion beams than the MOVIC technique. For most metals, the oven technique is the best method. If the oven is optimized, metal material consumption tate can be very small. It is especially important for precious isotope metals. For several kinds of metals with much higher melting point, the plasma sputtering technique might be a good choice. But the obtained ion beam current might be comparably weak.

However, it is not enough with the three techniques, and we need to research new techniques to produce intense and stable metallic ion beams. A higher temperature micro-oven is also unser developing in our lab. Some experiments on laser technique and electron bombardment technique are also under researching.

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