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МЕТОДИКА ФИЗИЧЕСКОГО ЭКСПЕРИМЕНТА

PRODUCTION OF INTENSE METAL ION BEAMS FROM ECR ION SOURCES USING THE MIVOC METHOD

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The production of metal ion beams by electron cyclotron resonance (ECR) ion sources using the MIVOC (Metal Ions from Volatile Compounds) method is described. The method is based on the use of metal compounds, which have high vapor pressure at room temperature, e.g., $C_2B_{10}H_{12}$, $Fe(C_5H_5)_2$, etc. Intense ion beams of B and Fe were produced using this method at the FLNR JINR cyclotrons. Experiments on the production of cobalt, chromium, vanadium, germanium, and hafnium ion beams were performed at the test bench of ECR ion sources.

Main efforts were put into production and acceleration of ^{50}Ti ion beams at the U400 cyclotron. The experiments on the production of ^{50}Ti ion beams were performed at the test bench using natural and enriched compounds of titanium $(CH_3)_5C_5Ti(CH_3)_3$. In these experiments, 80 μA $^{48}Ti^{5+}$ and 70 μA $^{48}Ti^{11+}$ beam currents were obtained at different settings of the source. Following successful tests, two three-week runs were performed with ^{50}Ti beams at the U400 cyclotron aimed to perform experiments on the spectroscopy of superheavy elements. The intensity of the injected $^{50}Ti^{5+}$ beam was $50-60$ μA. The source worked stably during the experiments. The compound consumption rate was determined at about 2.4 mg/h, which corresponded to the ^{50}Ti consumption of 0.6 mg/h.

Описано получение пучков ионов металлов из электронно-циклотронных резонансных (ЭЦР) источников методом MIVOC. Метод основан на использовании соединений металлов, имеющих высокое давление пара при комнатной температуре, например, $C_2B_{10}H_{12}$, $Fe(C_5H_5)_2$ и ряда других. С использованием этого метода на циклотронах ЛЯР ОИЯИ были получены интенсивные пучки ионов B и Fe.

Эксперименты по получению ионов кобальта, хрома, ванадия, германия и гафния были проведены на стенде ЭЦР-источников ионов.

Основные усилия были направлены на получение и ускорение пучков ионов ^{50}Ti на циклотроне У400. Эксперименты по получению ионов ^{50}Ti проводились на стенде с использованием естественного и обогащенного соединения титана $(CH_3)_5C_5Ti(CH_3)_3$. В стендовых экспериментах при различных настройках источника были получены пучки ионов $^{48}Ti^{5+}$ с интенсивностью 80 мкА и $^{48}Ti^{11+}$ с интенсивностью 70 мкА. После успешных стендовых испытаний были проведены два трехнедельных сеанса ускорения ионов ^{50}Ti на циклотроне У400 для экспериментов по спектроскопии сверхтяжелых элементов. Интенсивность инжектированного пучка ионов $^{50}Ti^{5+}$ составляла 50–60 мкА; в течение эксперимента источник работал стабильно. Расход соединения титана составил около 2,4 мг/ч, что соответствует расходу ^{50}Ti 0,6 мг/ч.

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INTRODUCTION

In recent years, the reactions of ^{48}Ca with ^{238}U, ^{242,244}Pu, ^{243}Am, ^{245}Cm, and ^{249}Cf have been used to synthesize new superheavy elements with $Z = 114-116$ and 118. In these experiments, a technique for the production of metallic ^{48}Ca was developed. The operation mode of the ECR ion source was set to optimize the intensity of $^{48}Ca^{5+}$ ions and attain maximum ionization efficiency [1]. Methods for collection and recovery of expensive isotopes were also developed. These studies allowed long-term experiments on synthesis of superheavy elements.

The heaviest target for experiments on synthesis of superheavy elements in heavy-ion reactions is ^{249}Cf, so further progress in the synthesis of elements with $Z > 118$ requires the production of intense beams of accelerated neutron-enriched isotopes, such as ^{50}Ti, ^{58}Fe, ^{64}Ni, etc. The use of new isotopes for the production of accelerated beams calls for searching for ways of optimization of the ECR source operation mode and the development of a material feeding technique.

The selection of the best method to feed solids into ECR ion sources strongly depends on specific properties of materials.

Several methods for the production of ions from solid materials have been developed. Solid materials can be evaporated from a resistor or inductive oven inserted into a source chamber [2, 3]. Refractory metals can be sputtered by plasma ions [4] or inserted into plasma with subsequent heating by energetic plasma electrons ("insertion technique") [5, 6]. Another way of producing ions of solids is to feed plasma of an organometallic compound using the MIVOC method [7].

PRODUCTION OF METAL IONS USING THE MIVOC METHOD

Organometallic compounds with high vapor pressure at room temperature — $C_2B_{10}H_{12}$, $Fe(C_5H_5)_2$, etc. — were used for the MIVOC method.

The MIVOC method was first employed at the FLNR U400M cyclotron to produce an intense beam of $^{11}B^{3+}$ required for generation of secondary beams of ^{6}He and ^{8}He [8]. We used the $C_2B_{10}H_{12}$ compound, which has vapor pressure of about 1–2 Torr at room temperature. The compound was loaded into a glass reservoir and fed into the source through a standard piezoelectric valve. No additional support gas was needed; the ion source operated stably.

A maximum current of $^{11}B^{3+}$ up to 200 eμA was produced by the DECRIS-2 [9] ion source. A charge spectrum of boron ions is shown in Fig. 1. The material consumption value of 2.2–2.8 mg/h was estimated at the 100 eμA $^{11}B^{3+}$ current.

Subsequently, this method was successfully applied to produce iron ions. Figure 2 shows an iron spectrum produced at the test bench of a modified ECR4M [10] source using natural ferrocene as a working substance. The source settings were optimized for the production of $^{56}Fe^{10+}$ ions.

The ^{58}Fe beam was accelerated at the U400 cyclotron to conduct an experiment aimed to synthesize isotopes of element 120 using the ^{244}Pu(^{58}Fe, xn)$^{302-x}$120 reaction [11]. The intensity of the $^{56}Fe^{7+}$ beam injected in the cyclotron was 40–50 eμA (6–7 pμA), and the $^{58}Fe^{23+}$ beam intensity at the target constituted 15–17 eμA ($\sim$ 0.7 pμA). The consumption of ^{58}Fe equaled about 1.5 mg/h.

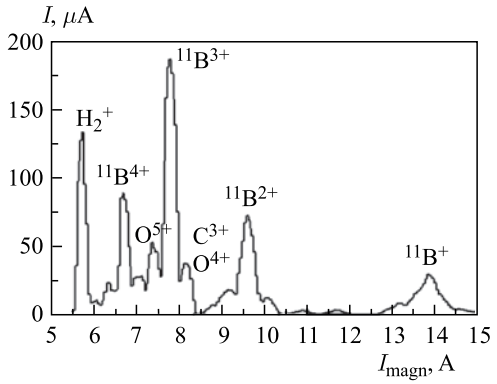

Fig. 1. Charge spectrum of boron ions produced by the DECRIS-2 ion source

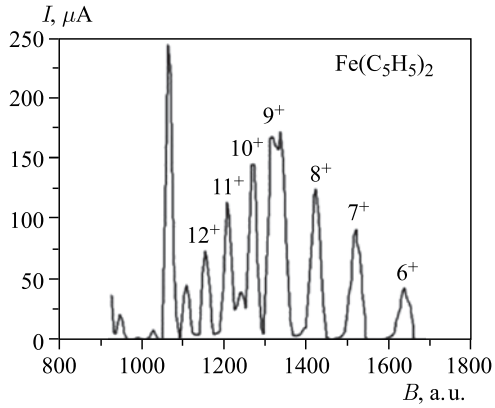

Fig. 2. Charge spectrum of iron ions produced by the ECR4M ion source

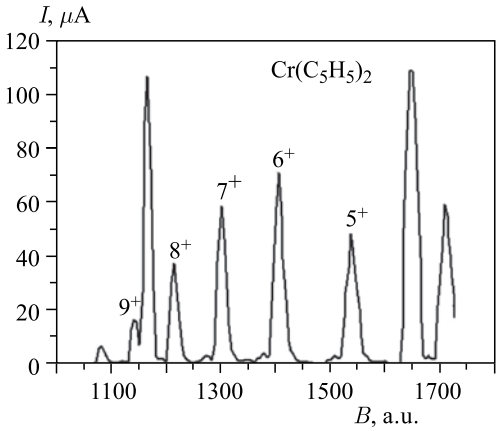

Fig. 3. Charge spectrum of chromium ions produced by the DECRIS-2M ion source

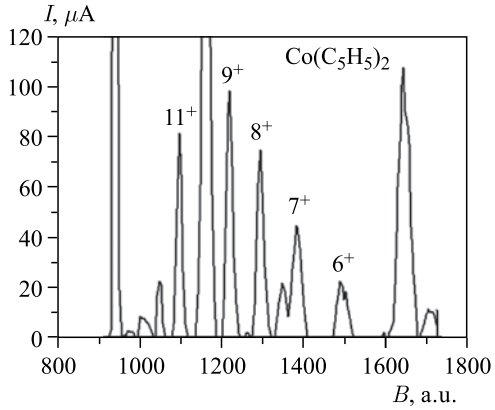

Fig. 4. Charge spectrum of cobalt ions produced by the DECRIS-2M ion source

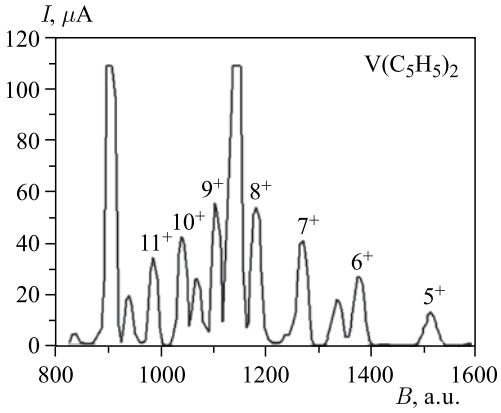

Fig. 5. Charge spectrum of vanadium ions produced by the DECRIS-2M ion source

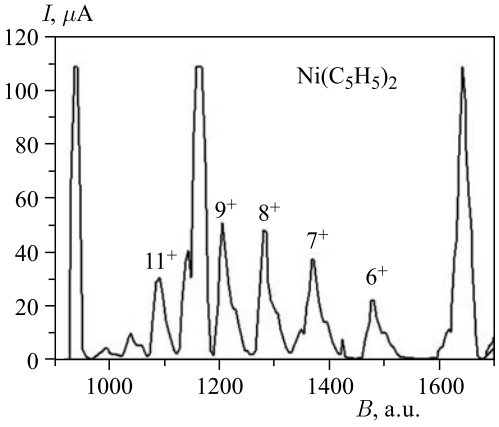

Fig. 6. Charge spectrum of nickel ions produced by the DECRIS-2M ion source

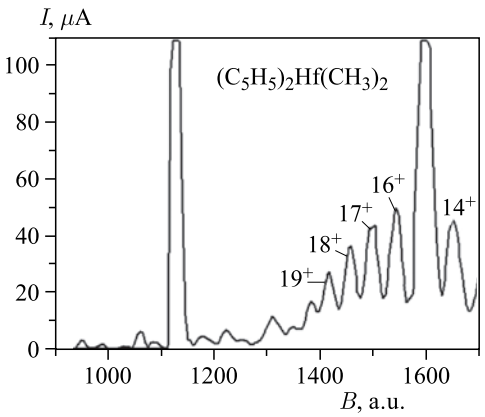

Fig. 7. Charge spectrum of hafnium ions produced by the DECRIS-2M ion source

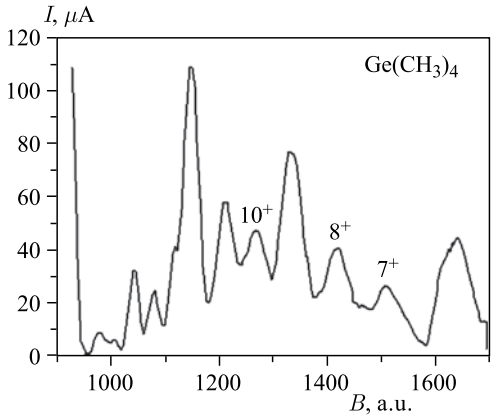

Fig. 8. Charge spectrum of germanium ions produced by the DECRIS-2M ion source

Table 1. **The intensity (eμA) of metal ion beams produced at the test bench using the MIVOC method**

Z	Fe	Co	Cr	V	Ni	Ge	Hf
5^+			50	75*			
6^+	43	57	70*	54	45*		
7^+	93	80	60	41	43	43*	
8^+	125	86	37	54	48	54	
9^+	172	98	17	55.5*	53*		
10^+	145*		7	43		47*	
11^+	114	82*		34	30		
12^+	73	25		19.5	10		
13^+	45						31
14^+							45
16^+							50*
17^+							45*
18^+							36
19^+							27
20^+							17

* Intensity optimization.

The experiments on the production of chromium, cobalt, vanadium, nickel, and hafnium ion beams were performed using the DECRIS-2M (Dubna ECR ion source) source [12] installed at the test bench. Natural compounds $Cr(C_5H_5)_2$, $Co(C_5H_5)_2$, $V(C_5H_5)_2$, $Ni(C_5H_5)_2$, and $(C_5H_5)_2Hf(CH_3)_2$ were used as working substances. Figures 3–7 show spectra of chromium, cobalt, vanadium, nickel, and hafnium ion beams, source tuning being optimized for maximum production efficiency of Cr^{6+}, Co^{11+}, V^{9+}, Ni^{9+}, and Hf^{17+} ions, respectively. For the production of germanium ions, two compounds were tested, i.e., tetraethylgermane $Ge(CH_2CH_3)_4$ and tetramethylgermanium $Ge(CH_3)_4$. Experiments with tetramethylgermanium yielded better results. Figure 8 shows a spectrum of germanium ions, source tuning being optimized to reach maximum production efficiency of Ge^{10+}. The results obtained at the test bench are presented in Table 1.

The MIVOC method was also applied for the production of accelerated metal ion beams to carry out applied research at the IC-100 cyclotron [13]. The accelerated beams of $^{56}Fe^{10+}$ (0.5 eμA), $^{127}I^{22+}$ (0.25 eμA), $^{184}W^{31+}$ (0.035 eμA), and $^{184}W^{32+}$ (0.017 eμA) were produced at the target.

PRODUCTION OF TITANIUM ION BEAMS

Experiments on the production of Ti ion beams using various methods were carried out at numerous laboratories worldwide.

The production of Ti ion beams by evaporation from a resistor oven was studied at the GSI Helmholtz Centre for Heavy Ion Research, Germany [14]. The evaporation of pure titanium can occur at a temperature between 1750 and 1800 °C. In these experiments, $^{50}Ti^{8+}$ beams of over 50 eμA were produced at a high level of beam stability. The oven lifetime was estimated at six days.

Experiments on the production of Ti ion beams by evaporation from the induction oven were carried out at ANL (Argonne National Laboratory, USA) [15]. A $^{50}Ti^{12+}$ beam with the intensity of 5.5 eμA had been kept during seven days.

The MIVOC method was first employed for the production of Ti ion beams by the JYFL (University of Jyväskylä Institute of Physics, Finland) group [16]. A commercially available $(CH_3)_5C_5Ti(CH_3)_3$ compound was used as a working substance. The intensity of the $^{48}Ti^{11+}$ion beam reached 45 eμA. The value of the titanium consumption was 0.22 mg/h. The ion beam was stable during a 282 h period.

Therefore, the MIVOC method seems to be extremely promising in terms of the beam intensity, stability, reliability, and material consumption. The method is efficient in producing ^{50}Ti ion beams for long-time continuous (up to several months) experiments on synthesis of superheavy elements.

Nevertheless, the compound's major drawback is that it is fairly difficult to handle due to its sensitivity to air, moisture, temperature, and light. Moreover, the synthesis of the compound is rather complicated, especially when using enriched titanium that is available only in scarce amounts (just up to several grams).

Test Experiments. The ^{50}Ti ion beam was accelerated at the U400 cyclotron in 2005. The objective was to produce a 30 enA ^{50}Ti beam at the target intended for experiments on fission physics [17]. Since the intensity requirements were low, we used $TiCl_4$, which has vapor pressure of about 10 Torr at room temperature and is sufficient for feeding the ECR source with a working substance. A glass ampule containing natural $TiCl_4$ (5.2% of ^{50}Ti) was connected to a standard piezoelectric leak valve, which is used when the source operates with gases. The intensity of the ^{50}Ti ion beam extracted from the cyclotron was about 200 enA. The source was running stably for two weeks.

The major drawback of using $TiCl_4$ is that it does not provide the intensity required for experiments on synthesis of superheavy elements.

Taking into account problems related to the synthesis and handling of $(CH_3)_5C_5Ti(CH_3)_3$, we decided to try to find other compounds suitable for the MIVOC method. Therefore, we tested the titanium isopropoxide ($Ti\{OCH(CH_3)_2\}_4$) and cyclopentadienyl cycloheptatrienyl titanium ($C_5H_5TiC_7H_7$). With the exception of a few microamperes of Ti^{5+}, no noticeable titanium currents were observed. The ($C_5H_5TiC_7H_7$) compound was subsequently used at the Lanzhou All Permanent ECR Ion Source No. 2 (LAPECR2) of the Institute of Modern Physics (IMP), China [18] to produce titanium ions employing the oven technique. In this test, 24 eμA of the Ti^{11+} ion beam was obtained using a 14.5 GHz microwave power of 250 W.

Subsequently, experiments on the production of titanium beams using the oven method were performed with the DECRIS-2 source. The titanium tetrafluoride (TiF_4) was used as a working substance. TiF_4 is a colorless crystal compound with a melting point of 426 °C. The temperature of about 50–80 °C is required to provide the vapor pressure sufficient for the source operation. In this temperature range, it is difficult to control the oven temperature, because the oven is also heated by UHF and plasma [19]. To decrease the material flux into the source chamber, we used an oven with a thin long channel, 15 mm in length and 1 mm

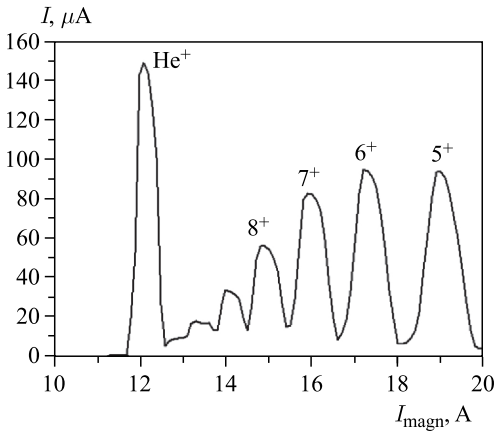

Fig. 9. Charge spectrum of titanium ions produced in the DECRIS-2 ion source by the insertion technique

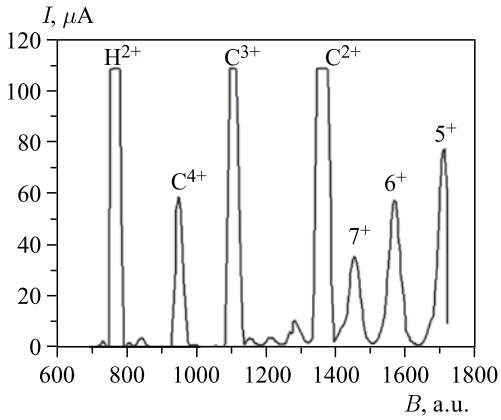

Fig. 10. Charge spectrum of titanium ions produced in the ECR4M source using the MIVOC method at the microwave power of 20 W

in diameter. The oven was axially inserted into the source, and its position was adjusted remotely. Fairly stable source operation was achieved at the intensity level of $^{48}Ti^{6+}$ of about 10–20 eμA. As the material feed increased, the discharge became unstable.

As the next step, titanium ions were produced using the insertion technique. The experiments were also performed with the DECRIS-2 ion source. A 3 mm diameter titanium rod was axially inserted into the source chamber through the bias tube. The position of the rod could be remotely adjusted. Helium was used as a support gas. The evaporation rate of titanium depended on the microwave power, helium pressure, and the position of the rod.

Figure 9 shows a typical spectrum of titanium ions produced at the microwave power of about 140 W with the source tuning optimized for the production of Ti^{5+}. The intensity was sufficient enough, but the long-term stability of the beam was not achieved. Without source tuning done within one hour, the beam intensity varied by 30%, discharge became uncontrollable, and the beam intensity dropped to zero.

The next step was to produce titanium ion beams using the MIVOC method and the (trimethyl)pentamethyl-cyclopentadienyl titanium compound. The commercially available compound produced by the Sigma–Aldrich company [20] was tested with the DECRIS-4 [21] and DECRIS-2 sources. The sources were optimized for the production of Ti^{5+}. Similar results were achieved using both sources, of about 50 eμA of the Ti^{5+} ion beam was obtained.

As the next step, the compound produced by the DALCHEM company was to be tested [22]. The main advantage was that the compound was provided in welded glass ampules, which made it much easier to handle. We did not need to use an argon-filled glove box; the ampule could be destroyed under vacuum in a specially designed MIVOC chamber. Tests were performed at the test bench of the modified ECR4M source. Figure 10 shows a spectrum of titanium ions; the source was tuned for Ti^{5+}. In all the experiments, no support gas was used. The MIVOC chamber temperature was not controlled either. The operation of the sources was stable and reproducible.

Production of ^{50}Ti Ion Beams. Major progress in the ^{50}Ti beam production was achieved through collaboration between IPHC (Strasbourg, France) and FLNR JINR.

Following several years of developments in chemistry carried out at IPHC, the $C_5(CH_3)_5Ti(CH_3)_3$ compound was synthesized using 92.57% enriched ^{50}Ti. Two-step chemistry was done with quite a high efficiency from $TiCl_4$ to $C_5(CH_3)_5Ti(CH_3)_3$ through an intermediate $C_5(CH_3)_5TiCl_3$ organic compound.

The first MIVOC isotopically enriched beam was developed and tested in 2011 at the University of Jyväskylä. Following the optimization, up to 19.4 eμA of $^{50}Ti^{11+}$ could be extracted from the JYFL 14 GHz ECRIS2 ion source [23, 24].

The natural titanium compound synthesized at IPHC was also tested at the GANIL (Grand Accélérateur National d'Ions Lourds) laboratory, France [25]. An intensity of 20 eμA was maintained for $^{48}Ti^{10+}$ for four days, while regulating the temperature of the MIVOC chamber. A consumption of 1.5 mg/h has been deduced for the MIVOC compound, i.e., 0.23 mg/h for ^{48}Ti.

In 2012–2013, several samples of the $(CH_3)_5C_5Ti(CH_3)_3$ compound synthesized at IPHC were tested at the test bench at FLNR. The major challenge was the long-time transportation of samples from IPHC to JINR, which caused the compound destruction and resulted in the production of poor currents of titanium. Therefore, we decided to perform the final step of synthesis at the FLNR chemistry laboratory.

First natural material synthesized by the IPHC group at FLNR was tested in October 2013 using the ECR4M ion source test bench. After optimization, stable ^{48}Ti beams were produced with the intensities up to 70 eμA for the 11^+ charge state (6.2 pμA) and 75 eμA for the 5^+ charge state (15.0 pμA). Figure 11 shows charge state distribution of the ^{48}Ti ion beam, source settings being optimized for the production of $^{48}Ti^{11+}$.

Following these extremely promising results, a 92.57% enriched compound was synthesized and tested at the ECR4M test bench. Under similar conditions, up to 80 eμA of the $^{50}Ti^{5+}$ beam was extracted, which corresponded to 16.0 pμA. Figure 12 shows charge state distribution of the ^{50}Ti ion beam, source settings being optimized for the production of $^{50}Ti^{5+}$.

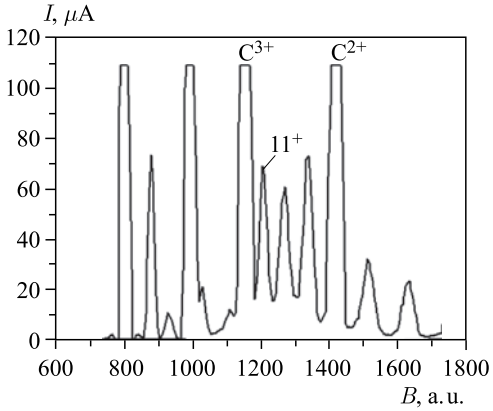

Fig. 11. Charge spectrum of titanium ions produced in the ECR4M source using the MIVOC method at the microwave power of 300 W

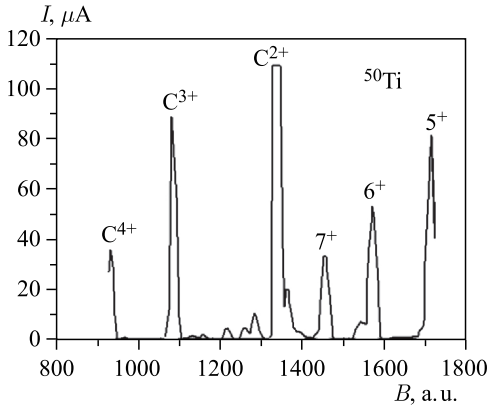

Fig. 12. Charge spectrum of ^{50}Ti ions produced in the ECR4M source using the MIVOC method at the microwave power of 30 W

Table 2. **The intensity (eμA) of titanium ion beams produced at different laboratories using the MIVOC** (a) **and oven** (b) **methods**

Ion beam	Laboratory					
	JYFL (a)	GANIL (a)	FLNR (a)	GSI (b)	ANL (b)	IMP (b)
$^{48}Ti^{5+}$			79			
$^{48}Ti^{10+}$		20				
$^{48}Ti^{11+}$	45		68			24
$^{50}Ti^{5+}$			82			
$^{50}Ti^{8+}$				50		
$^{50}Ti^{11+}$	20					
$^{50}Ti^{12+}$					5.5	

This beam was then produced using the DECRIS-2M source and accelerated at the U400 cyclotron to carry out experiments on the spectroscopy of superheavy elements [26]. A stable and intense 55 eμA beam was injected into the cyclotron at the 5^+ charge state (11.0 pμA). The beam intensity of 0.49 pμA was maintained at the target for several weeks in October–November 2013. This beam was stable, and the titanium consumption was quite low, i.e., 0.6 mg/h.

The next run with the ^{50}Ti ion beam was performed at the U400 cyclotron in April–May 2014. The DECRIS-2M source of the U400 cyclotron was at the time replaced by the ECR4M source. The intensity of the $^{50}Ti^{5+}$ beam was maintained at the level of 55–62 eμA for three weeks. The material consumption was similar to the previous run when the DECRIS-2M ion source was used.

Table 2 summarizes the results of the titanium ion beam production at different laboratories using the MIVOC method (JYFL, GANIL, FLNR) and oven technique (GSI, ANL, IMP).

CONCLUSION

Over the past few years, notable results and significant progress have been achieved in the production of intense multiply charged metal ion beams in ECR ion sources using the MIVOC method.

The MIVOC method was successfully used for producing and accelerating ^{50}Ti and ^{58}Fe ion beams at the U400 cyclotron. This method helps to produce intense ion beams, provides the long-term stability and is promising for experiments on synthesis of superheavy elements.

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