

RECENT DEVELOPMENT IN METALLIC BEAM PRODUCTION WITH AN ECR ION SOURCES AT FLNR (JINR)*

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OVERVIEW OF THE FLNR (JINR) CYCLOTRONS WITH ECR ION SOURCES

U400 + ECR4M











FLNR's BASIC DIRECTIONS of RESEARCH

1. Heavy and superheavy nuclei:

- synthesis and study of properties of superheavy elements;
 chemistry of new elements;
- Fusion-fission and multi-nucleon transfer reactions;
- > nuclear- , mass-, & laser-spectrometry of SH nuclei.

2. Light exotic nuclei:

- > properties and structure of light exotic nuclei;
- reactions with exotic nuclei.

3. Radiation effects and physical groundwork of nanotechnology.

Many of the elements required for acceleration at the FLNR cyclotrons are available in the solid state form only.

Production of neutron reach light nuclei (6He, 8He,...)

Required beams -Li, B..

Synthesis of new super heavy nuclei

Required beams: ⁴⁸Ca-(0,19 %); ⁵⁰Ti - (5,2 %); ⁵⁸Fe-(0,3 %) ...

For the production of intense beams an expensive enriched isotopes are used



Efficiency of material consumption !!!

Production of the Li ion beam with hot screen



Yields (*eµ*A) for Li from the DECRIS-2 with UHF power in the range 300-400 W. 3+ Q 2+1+ 7Li 15 50 25 No screen 7Li 180 290 50 With hot screen



Li spectrum with hot screen

The same technique was used for production of ²⁶Mg, ⁴⁸Ca, Bi

Production of ⁴⁸Ca beam with ECR4M source



Since 1997 about 70% of the total beam time were used for acceleration of ⁴⁸Ca⁵⁺ ions for synthesis of new superheavy elements.



Reduction of calcium from calcium oxide

Spectra of Ca ions at different source settings

Measuring point	Beam int	Ion	Transmission factor				r	
ECR source, after separation	1·10 ¹⁴ pps	84 µAe	⁴⁸ Ca ⁵⁺	32%				
Cyclotron centre	3.5·10 ¹³ pps	27 μAe	48Ca5+		81%			
Extraction radius	2.8·10 ¹³ pps	22 µАе	⁴⁸ Ca ⁵⁺			40%		
Extracted beam (by charge exchange)	9.7·10 ¹² pps	28 µAe	⁴⁸ Ca ¹⁸⁺				82%	
Target	8-10 ¹² pps	23 μAe	⁴⁸ Ca ¹⁸⁺					8.5%



 ^{48}Ca consumption ~ 0.8 \div 1 mg/h

905 samples of metallic ⁴⁸Ca were used since November 1997 till the end of June 2015



isotopes, such as ⁵⁰Ti, ⁵⁸Fe, ⁶⁴Ni, etc.

Production of ions of solids

Vapor of metals (oven method) Of Volatile compounds (MIVOC method)





MIVOC-method (<u>Metal Lons from VO</u>latile <u>C</u>ompounds)

Requirements:

1) Chemical compound containing required element has to be found 2) The saturated vapour pressure (at room temperature) of the compound has to be high enough ($\geq 10^{-3}$ mbar)

- minimum usable vapour pressure of the compound is determined by the material consumption rate of the ion source and by the conductance between the MIVOC chamber and the ion source

 $(C_5H_5)_2$ Me – metallocene molecule (Fe, Ni,Cr..)

 $(CH_3)_4$ Me – tetrameethyl molecule (Sn, Ge)

(CO)₆Me – hexacarbonyl molecule (Cr, W)







 $Fe(C_5H_5)_2$

 $C_2B_{10}H_{12}$ - carboran

First MIVOC beams at FLNR

U-400M - 1998



U-400 - 2005

Acceleration of ⁵⁸Fe at the U-400 cyclotron

 ${}^{58}\text{Fe}(\text{C}_{5}\text{H}_{5})_{2}$ Injected ${}^{58}\text{Fe}^{7+}$ beam 40÷50 eµA (6 ÷ 7 pµA) ${}^{58}\text{Fe}^{23+}$ at the target 15 ÷ 17 eµA (~ 0.7 pµA). The consumption of ${}^{58}\text{Fe}$ 1,5 mg/h.



PRODUCTION OF TITANIUM ION BEAM

GSI – a resistor oven (temperature 1750 °C \div 1800 °C) ⁵⁰Ti⁸⁺ \ge 50 eµA. The oven lifetime \div 6 days.

ANL – induction oven (temperature 1600 °C) ${}^{50}\text{Ti}^{12+}$ – 5.5 eµA (7 days) 0.7 mg/h

JYFL – MIVOC method commercially available $(CH_3)_5C_5Ti(CH_3)_3$ compound was used as a working substance. ⁴⁸Ti¹¹⁺ion – 45 eµA. 0.22 mg/h. The ion beam was stable during a 282 hour period.

 $(CH_3)_5C_5Ti(CH_3)_3$ - sensitive to air, moisture, temperature and light

Synthesis of compound

 $TiO_2 + CCl_4 \longrightarrow TiCl_4 + (CH_3)_5C_5Si(CH_3)_3 \longrightarrow Cp^*TiCl_3 + 3CH_3Li \longrightarrow Cp^*Ti(CH_3)_3$ where Cp* - (CH_3)_5C_5

- All reactions should be performed under inert atmosphere or air-free conditions due to the reactivity of the transition metal species and other reagents.
- Extra care must be taken when handling CH₃Li due to the large quantities required and extreme reactivity of this compound.

PRODUCTION OF TITANIUM ION BEAM



PRODUCTION OF TITANIUM ION BEAM





TiCl₄ (5.2% of ⁵⁰Ti)

The intensity of the ⁵⁰Ti ion beam extracted from the cyclotron was about 200 enA.

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Titanium isopropoxide (Ti{OCH(CH_3)_2}_4)
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Cyclopentadienyl cycloheptatrienyl titanium (C₅H₅TiC₇H₇)

 $\frac{\text{Commercial compound}}{\text{Sigma-Aldrich (Germany)}}$ DALCHEM (Russia)

ECR sources: DECRIS-2, DECRIS-4, ECR4M



Ti ion spectrum produced from DECRIS-4. The source settings are optimized for ⁴⁸Ti⁵⁺

Development of ⁵⁰Ti beam using MIVOC method

Collaboration between IPHC (Strasbourg, France) and FLNR JINR.

Synthesis of compound (two steps)

 $TiCl_4 + (CH_3)_5C_5Si(CH_3)_3 \rightarrow Cp*TiCl_3 + 3CH_3Li \rightarrow Cp*Ti(CH_3)_3$

where $Cp^* - (CH_3)_5C_5$

Starting material: ${}^{50}\text{TiCl}_4$ enrichment > 90% - available from Trace Sciences International Inc.

The first step of synthesis was performed at IPHC, the final step at FLNR. The efficiency of synthesis is more than 90%.





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 ⁴⁸Ti 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).



Following these extremely promising results, a 92.57 % enriched compound was synthesized and tested at the ECR4M test bench. Under similar conditions, up to $80 \text{ e}\mu\text{A}$ of the ${}^{50}\text{Ti}{}^{5+}$ beam was extracted.

Acceleration at the U-400 cyclotron

After successful tests at the test bench the accelerated beam of ${}^{50}\text{Ti}{}^{5+}$ was produced at the U-400 cyclotron for the experiment on spectroscopy of super heavy elements.

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The intensity of the injected beam of ^{50}\text{Ti}^{5+} \geq 50~\text{e}\mu\text{A}
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The intensity at the target ~ 10 $e\mu A$ (~ 0.5 $p\mu A$)

The compound consumption rate of 2.4 mg/h (⁵⁰Ti consumption of 0.52 mg/h)

During the experiment (two three week runs) the source have shown stable operation.



ECR4M source



MIVOC chamber with EVR116 regulating valve

Recovery of used material

 $cp*Ti (CH_3)_3 + nHCl + (4-n)H_2O \longrightarrow cp*H + 3CH_4 + Ti(OH)_{(4-n)}Cl_n$



X-ray fluorescence spectrum of dried sediment

Operation time \div 370 hours ⁵⁰Ti consumption ~ 190 mg Collected ~ 22 mg (~ 11%)

Intensity (eµA) of Titanium Ion Beams Produced at Different Laboratories by MIVOC (a) and Oven (b) Methods

	JYFL ^a	GANIL ^a	FLNR ^a	GSI ^b	ANL ^b	IMP ^b
⁴⁸ Ti ⁵⁺			79			
⁴⁸ Ti ¹⁰⁺		20				
⁴⁸ Ti ¹¹⁺	45		70			24
⁵⁰ Ti ⁵⁺			82			
⁵⁰ Ti ⁸⁺				50		
⁵⁰ Ti ¹¹⁺	20					
⁵⁰ Ti ¹²⁺					5.5	

Set of experiments on production of metal ion beams by MIVOC method was performed at the ECR test bench

0.

I, μA

¥ 150





Cr(C,H)2

L, μΑ

The intensity (eµA) of metal ion beams produced at the ECR test bench using the MIVOC method

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

(* - intensity optimisation)

CONCLUSION

The MIVOC method was successfully used for producing and accelerating titanium-50 ion beam at the U-400 cyclotron. This method helps produce intense ion beams, provides long-term stability and is promising for experiments on synthesis of superheavy elements.



DECRIS-5

Room temperature source, 18 GHz

Ion	Ar ⁸⁺	Ar ⁹⁺	Ar ¹¹⁺	Kr ¹⁵⁺	Kr ¹⁷⁺	Kr ¹⁸⁺	Kr ¹⁹⁺	Kr ²⁰⁺	Xe ²⁰⁺
I, (eµA)	1200	750	300	325	230	182	120	70	220



DECRIS-SC2 Hybrid type source, 14 GHz

Ion	O ⁵⁺	O ⁶⁺	S ⁹⁺	S ¹¹⁺	Ar ⁸⁺	Ar ⁹⁺	Ar ¹¹⁺	Ar ¹²⁺	Kr ¹⁵⁺	Kr ¹⁷⁺	Xe ³⁰⁺
I, (еµА	920	820	265	90	880	680	250	120	250	150	~ 1

DC-280 Cyclotron



Parameters of DECRIS-PM.

Frequency	14 GHz
B _{inj}	≥ 1.3 T
B _{min}	0.4 T
B _{extr}	1.0 ÷1.1 T
B _r	1.05÷1.15 T
Plasma chamber internal diameter	70 mm



DECRIS ion sources



















