MIVOC METHOD AT THE mVINIS ION SOURCE

by

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Based on the metal-ions-from-volatile-compounds (MIVOC) method with the mVINIS ion source, we have produced multiply charged ion beams from solid substances. Highly intense, stable multiply charged ion beams of several solid substances with high melting points were extracted by using this method. The spectrum of multiply charged ion beams obtained from the element hafnium is presented here. For the first time ever, hafnium ion beam spectra were recorded at an electron cyclotron resonance ion source. Multiply charged ion beams from solid substances were used to irradiate the polymer, fullerene and glassy carbon samples at the channel for the modification of materials.

Key words: electron cyclotron resonance, heavy ion source, multiply charged ions, ion beam irradiation, modification of materials, fullerenes

INTRODUCTION

The mVINIS ion source, a part of the TESLA Accelerator Installation at the Vinča Institute of Nuclear Sciences, Belgrade, is a source of highly charged heavy ions based on the micro-wave heating of the plasma on electron cyclotron resonance (ECR) frequencies. The mVINIS has enabled us to extract versatile ion beams from various standard gases (He, N, O, Ne, Ar), isotopic enriched gases (Kr, Xe), and many solid substances (Zn, Pb, B, Fe, *etc.*) [1-3]. The solid substance ion beam production is mainly realized by two different methods: (a) the evaporation of metals by using the miniature oven technique [4-6], and (b) the MIVOC (Metal-Ions-from-Volatile-Compounds) method by using the modified gas inlet system [7-13]. Both experimental methods of solid substance loading into the plasma of the ECR ion source have been successfully developed at the TESLA Accelerator Installation. The MIVOC method has enabled us to obtain highly intense multiply charged ions from different solid substances with high melting points [14]. Continuing with the further improvement of the MIVOC technique, we have recently obtained significant results in the extraction of hafnium (Hf) ion beams, presented here.

In irradiation experiments at the channel for the modification of materials (L3A) [15, 16], ion beams from solid substances have been used. The L3A channel is a low energy experimental channel directly connected to the ECR ion source injector. Multiply charged ions obtained from the mVINIS are utilized to modify the surface, electrical, mechanical, and optical properties of different materials. Some of the results of the heavy ion beam bombardment of fullerene thin films are shown here.

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THE MIVOC METHOD

The physical and chemical properties of materials determine the way solid substances are loaded into the ECR plasma chamber. Metal evaporation and plasma sputtering resulting from the miniature oven technique are appropriate for materials with low melting points (below 1000 ºC). The MIVOC method is a more adequate method for high melting point materials (over 2000 ºC). The production and extraction of solid substance ion beams through the miniature oven technique were realized by using the main gas inlet system at the injection stage of the source. Based on the calibration method of two manometers in the standard ion source working regime, the main and support gas flows are in the region of 10^{-5} and 10^{-3} mbarl/s, respectively [17]. In the case of the MIVOC method, the compound vapour flows depend on the conductivities of gas feed line parts attached to the metal organic compound vessel. For example, the saturated vapour flows of the ferrocene compound ($Fe(CH_3)_5$) are in the region of 3 10^{-4} mbarl/s [18]. A detailed description of gas flow calculation is presented in ref. [19].

Using the MIVOC method, we have recently obtained the production and extraction of hafnium (Hf) ion beams. The redesigned container, closed with a manual, right angle vacuum valve, was replete with the metal organic compound Bis (cyclopentadienyl)dimethyl-hafnium(IV) (chemical formula $(C_5H_5)_2Hf(CH_3)_2$) (see fig. 1).

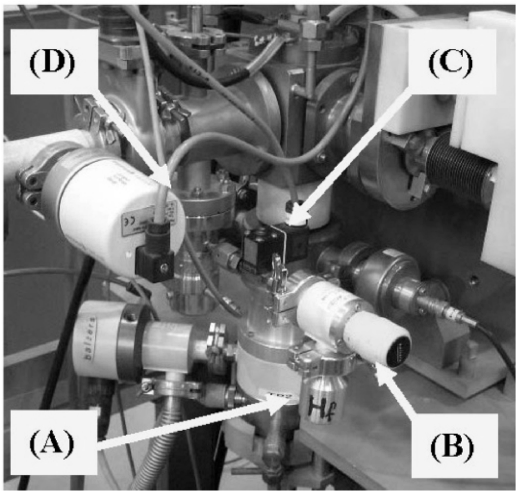

Figure 1. The modified main gas inlet system of the MIVOC method at the injection stage of mVINIS
The stainless steel vessel (V = 6 cm³) with a metal organic compound of hafnium (A) and a right angle valve (B) is connected to the isolation valve (C) prior to the main gas dosing valve (D). Because the hafnium compound is strongly sensitive to air and moisture, the handling and storage were done under an argon atmosphere

The said compound was successfully used for the first time during the summer of 2006, at the TESLA Accelerator Installation. The obtained hafnium ion beam spectrum is shown in fig. 2.

The mVINIS ion source was optimized for the Nd-like ion (ion charge state $q = +12$) and achieved a beam current of over 40 A (or 3 p A). The compound was loaded through the thermal dosing valve as a standard main gas. The vapor pressure of the compound at room temperature was in the range of 1 10^{-3} mbar. In order to increase the hafnium ion

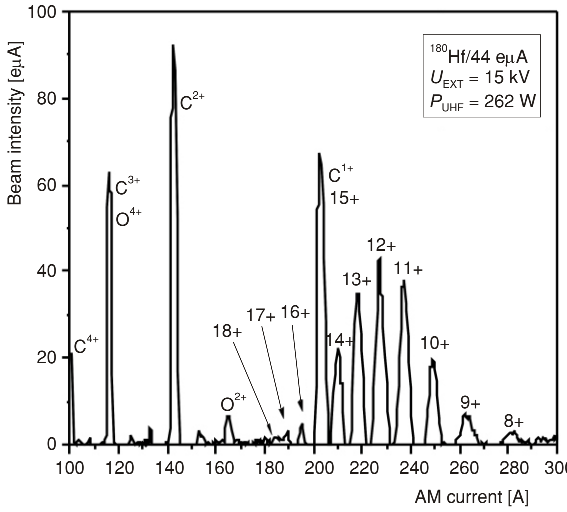

Figure 2. Spectrum of ¹⁸⁰Hf ions obtained by the MIVOC method
The Hf^{12+} ion beams were extracted up to currents of 43 eµA and kinetic energy of 180 keV. U_{EXT} was the applied extraction potential and P_{UHF} was the microwave heating power. Natural hafnium isotopic abundance is ^{176}Hf = 5.2%, ^{177}Hf = 18.6%, ^{178}Hf = 27.3%, ^{179}Hf = = 13.6%, and ^{180}Hf = 35.1%

beam yields, the container was heated up by a hot air stream to 60 °C so as to raise the partial compound vapor pressure. The ion source working regime was stable, without any need for loading support gases. The vapor flow of the metal organic compound was in the region of 10^{-3} to 10^{-4} mbarl/s.

The basic parameters of the multiparticle ECR plasma, as well as the main ionic optic parameters, are estimated from the ion beam current intensities of the recorded spectrum [17]. The density and the velocity of extracted hafnium ions Hf^{12+}, for the extraction potential of 15.0 kV, are 2.3 10^9 cm^{-3} and 4.4 10^{-5} m/s, respectively [17].

Several plasma parameters of the hafnium ions Hf^{12+} are listed in tab. 1. The presented parameters are the electron-ion collision frequency (ν_{ei}), the mean free-path length (λ_i), the Debye radius (R_D), the plasma frequency (f_{pi}), and the ion cyclotron frequency (f_{ci}). They were calculated under following as-

Table 1. Basic plasma parameters of Nd-like hafnium ions ($q = +12$)

Parameter	Value
ν_{ei} [Hz]	1.2 10^3
λ_i [cm]	1.8 10^6
R_D [cm]	1.9 10^{-6}
f_{pi} [kHz]	18.9
f_{ei} [MHz]	0.51

sumptions: ion density of 1 10^{10} cm^{-3}, warm electrons temperature of 1 keV and the magnetic field of 0.5 T coresponding to the magnetic field of the electron cyclotron resonce heating in the plasma chamber.

Obtained hafnium ion beams will be applied in irradiation experiments of special stainless steel samples for the development of new materials in the next generation of nuclear plants, purposes such as the packing of nuclear waste and the construction of experimental fusion reactors.

The advantages of the new gas inlet system for the MIVOC method are the following: (1) the handling of the experimental setup is simple, (2) the design is space-saving, (3) the construction is convenient at the high voltage side of the source, (4) the extracted ion beams are stable over a long period (from several hours to a few days), and (5) the low consumption rate (approximately 5 mg/h) offers better possibilities of producing ion beams of expensive and rare isotopes. The disadvantages of the MIVOC method imply the following: (1) the compounds are often toxic, corrosive, and inflammable, (2) the plasma chamber can be contaminated with carbon, and (3) the influences of water vapour pressure can disable the ion source optimization.

MODIFICATION OF MATERIALS WITH SOLID SUBSTANCES ION BEAMS

Numerous experimental programs with versatile ion beams are performed at the channel for the modification of materials (L3A) [20]. Ion beam irradiation has been carried out since 1998, when the channel was linked to the mVINIS ion source [16]. The main aim of many of these experiments was to accomplish high implantation fluences (up to $D = 10^{18}$ cm^{-2}). In order to recalculate the desired particle dose, the multiply charged ion beam current has to be divided by the ion beam charge state (I/q). Based on this fact, our task was to obtain table ion beams with highest possible intensities (over 200 eμA), while reaching maximal irradiation doses in a reasonably short time scale (from a few hours to several days). The multiply charged ion beams extracted from the ECR ion source are separated by charge to mass ratio (q/m) and transported to the target chamber for the modification of materials. The low beam transmission of the channel (about 30%) additionally reduces the intensity of the transported ion beams [3]. By applying ion implantation techniques, the different properties of materials (structural, electrical, optical, mechanical, *etc.*) can be altered to a great extent.

Experiments concerning the irradiation of fullerenes, polymers, and glassy carbon by multiply charged ions of solid substances were initiated in the summer of 2005.

Over the last two years, some of the following experimental programs were performed with multiply charged ion beams:

modification of surface and structural properties of fullerenes thin films by multiply charged nitrogen (N^{2+}, N^{5+}), boron (B^{1+}, B^{3+}), and iron (Fe^{6+}) ions bombardment,

surface and structural modification of glassy carbon by intensive boron ions implantation (B^{1+}, B^{2+}, B^{3+}),

argon (Ar^{8+}) and iron (Fe^{6+}) ion beams induced modification of the physical properties of the polymers and Fe-polymer nano-composite thin films, and

implantation of xenon ion beams (Xe^{17+}) into nuclear glasses and ceramic composites for research purposes involving new materials to be used in nuclear plants.

The following techniques are used for investigating the characteristics of irradiated samples: (a) Raman Spectroscopy, (b) Fourier Transform Infrared (FTIR) Spectroscopy, (c) Atomic Force Microscope (AFM) Analysis, (d) X Photon Electron Spectroscopy (XPES), and (e) Grazing Incidence X-Ray Diffraction (GIXD) [21]. AFM 3D images of fullerenes thin films implanted by nitrogen, boron, and iron ions with different charged states (q), ion beam energies (E), and irradiation doses (D) are given in figs. 3-6. The figures show destroyed surface ordering due to the multiply charged ions bombardment. Sp^2 clusters were formed, within the range of 50 to 500 nm, depending on the ion implantation dose (cluster size decreases with the increase of the implantation dose) [21-23]. An amorphous layer of boron-carbide (B_xC) was formed at the surface of fullerenes thin films irradiated by triple charged boron ions [23].

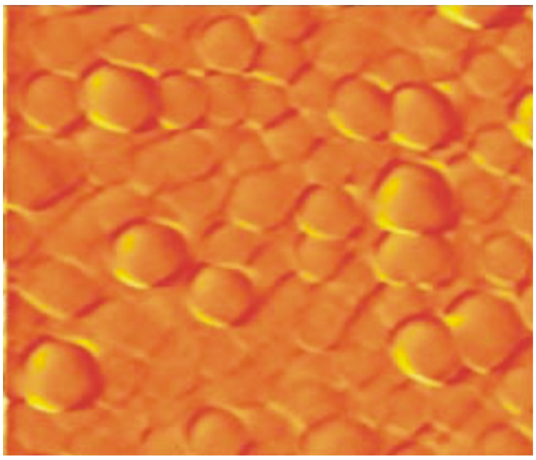

Figure 3. AFM scan of implanted fullerenes thin films (^{60}C) by nitrogen ions N^{2+} with an irradiation dose of D = 2 10^{17} cm^{-2} and kinetic energy E = 30 keV. Image size: 1.5 1.5 μm^2

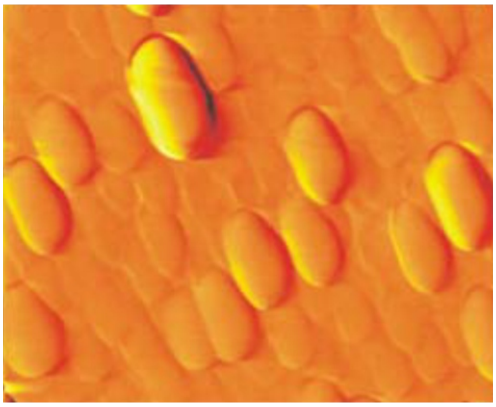

Figure 4. AFM scan of implanted fullerenes thin films (^{60}C) by nitrogen ions N^{5+} with an irradiation dose of $D = 2\ 10^{17}$ cm^{-2} and kinetic energy E = 75 keV. Image size: 1.5 1.5 μm^2

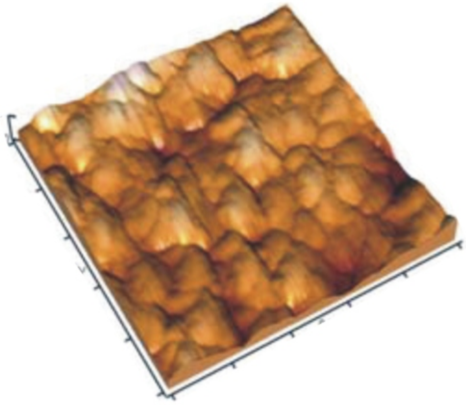

Figure 5. AFM scan of implanted fullerenes thin films (^{60}C) by boron ions B^{3+} with an irradiation dose of $D = 2\ 10^{16}$ cm^{-2} and kinetic energy E = 45 keV. Image size: 1.0 1.0 μm^2

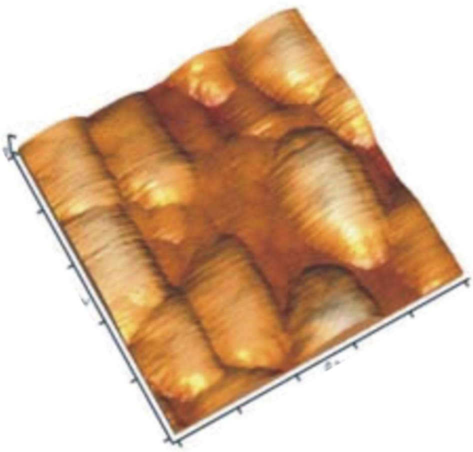

Figure 6. AFM scan of implanted fullerenes thin films (^{60}C) by iron ions Fe^{6+} with an irradiation dose of $D = 2\ 10^{16}$ cm^{-2} and kinetic energy E = 90 keV. Image size: 1.0 1.0 μm^2

CONCLUSION

The mVINIS ion source has enabled us to produce multiply charged ions of solid substances. Both methods of solid substance loading into the ion source, the miniature oven technique and the MIVOC mode, successfully developed at the Laboratory of Physics of the Vinča Institute of Nuclear Sciences, have provided excellent results in the extraction of intensive multiply charged ion beams.

During the summer of 2006, based on the MIVOC method, a hafnium ion beam spectrum was obtained with the maximal value of an Hf^{12+} ion beam current of 43 μA. This was the first instance ever of a hafnium ion beam spectra being recorded at an ECR ion source.

At the experimental channel L3A, beams of solid substances are used for the irradiation of fullerenes, polymers, and glassy carbon.

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МИВОК МЕТОДА НА ЈОНСКОМ ИЗВОРУ мВИНИС

Вишеструко наелектрисани снопови јона из чврстих супстанци произведени су коришћењем МИВОК (MIVOC – Metal Ions from VOlatile Compound) методе, на јонском извору мВИНИС. Употребом ове методе добијени су високоинтензивни стабилни вишеструко наелектрисани јонски снопови из чврстих супстанци са високом тачком топљења. У раду је представљен спектар вишеструко наелектрисаних снопова јона хафнијума. Спектри јонских снопова хафнијума по први пут су снимљени на једном ЕЦР извору. Коришћењем вишеструко наелектрисаних јонских снопова из чврстих супстанци озрачиване су различите врсте полимера, фулерена и стакластог графита на каналу за модификацију материјала.

Кључне речи: електронска циклотронска резонанца, извор тешких јона, вишеструко наелектрисани јони, озрачивање јонским сноповима, модификација материјала, фулерени