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Multiply charged ions from solid substances with the mVINIS Ion Source

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Abstract. We have used the well known metal-ions-from-volatile-compounds (MIVOC) method at the mVINIS Ion Source to produce the multiply charged ion beams from solid substances. Based on this method the very intense and stable multiply charged ion beams of several solid substances having the high melting points were extracted. The ion yields and the spectra of multiply charged ion beams obtained from solid materials like Fe and Hf will be presented. We have utilized the multiply charged ion beams from solid substances to irradiate the polymers, fullerenes and glassy carbon at the low energy channel for modification of materials.

1. Introduction
The mVINIS Ion Source, which is an electron cyclotron resonance ion source, can produce the highly charged ions [1]. The versatile ion beams of the standard and enriched gases as well as of solid substances are extracted until now [2] [3]. Ion beams are obtained from solid substances using the two well-known methods: (a) the metal evaporation method, in which an inlet system based on a mini-oven is used [4], [5], [6], and (b) the MIVOC (metal-ions-from-volatile-compounds) method [7-13], in which a new design of gas inlet system is used. We have used the latter method and produced the intense multiply charged ion beams of several solid substances having the high melting points (over 2000 °C) [14]. The spectra of multiply charged ion beams obtained from solids are represented in this work.

In addition, we have used the multiply charged ion beams from solid substances generated with the mVINIS Ion Source to modify materials at the channel for modification of materials (L3A) [15] of the TESLA Accelerator Installation [16]. These ions have been implanted into different materials to modify their electrical, optical, mechanical and other properties. Some of the results obtained with the irradiation of fullerenes will be shown.

2. MIVOC method
The large ion implantation doses require high intensity ion beams for the experiments in modification of materials at the L3A channel. Our main aim was to produce intensive multiply charged ion beams of metals and other solid substances. The techniques of solid material loading into an ECR ion source basically depend on physical and chemical characteristics of materials. At the mVINIS Ion Source the
mini-oven technique is adequate for the solid material with low melting point (the maximum oven temperature of 900 °C) and the MIVOC method is appropriate for the materials with higher melting point. Both techniques of solid substance loading into the mVINIS are successfully developed. The production of multiply charged ion beams from solid substances with high current intensity is described in detail in ref [14].

The recent test of the MIVOC method was the production and extraction of hafnium ion beams. The new designed container, closed with manual right angle vacuum valve (see figure 1), was replete with the compound Bis(cyclopentadienyl)dimethyl-hafnium (IV) (chemical formula (C₅H₅)₂Hf(CH₃)₂), which was successfully used during summer 2006, in the TESLA Accelerator Installation in Belgrade. An obtained hafnium spectrum is shown in figure 2a. The mVINIS Ion Source was optimized for the Nd-like ion (q=12+) and achieved beam current over 40 eμA (or 3 pμA). The compound was loaded as standard main gas into ion source through the thermal dosing valve. The vapor pressure of the compound at room temperature is in the range 1x10⁻³ mbar. In order to increase the hafnium ion beam yields the container was heated up by a hot air stream to 60 °C to raise the partial compound vapor pressure. The ion source working regime was stable without loading of support gases. The hafnium ion beam will be used in the irradiation experiments of special stainless steels for the development of new material for the nuclear plants, nuclear waste and experimental fusion reactors.

The production of the iron ion beams were achieved earlier also by the MIVOC method. Based on the well-known ferrocene powder Fe(C₅H₅)₂ [7, 8] the multiple charged iron ion beam spectrum was extracted (figure 2(b)).

The advantages of the new system are the following: its production is cheap (for every compound can be fabricated its own vessel), the use is quite simple, the construction is space-saving and convenient at high voltage side, the produced ion beams are stable in a long period, and the rate of consumption of the compounds is very low (approximately 5 mg/hour) making it possible to use expensive, rare isotopes. Some disadvantages should be also remarked: often the compounds are toxic and inflammable, the plasma chamber can be contaminated with carbon, and the influences of water vapor pressure can disable the ion source optimization.

**Figure 1.** The new experimental setup for MIVOC method based on the modified main gas inlet system at the injection stage of the mVINIS. The stainless steel vessel of V=6 cm³ with the metal organic compound of hafnium (A) is connected to the right angle ventil (Balzers DN 16) (B) and the isolation valve USV (C) before the gas dosing valve UDV140, Balzers (D). Because the hafnium compound is strongly air and moisture sensitive, the handling and storage were done under argon atmosphere.
Figure 2. (A) Typical hafnium ion beam spectrum obtained by MIVOC technique. Natural hafnium isotopic abundance of $^{176}\text{Hf}=5.2\%$, $^{177}\text{Hf}=18.6\%$, $^{178}\text{Hf}=27.3\%$, $^{179}\text{Hf}=13.6\%$ and $^{180}\text{Hf}=35.1\%$ broadens the width of recorded ion beam peaks. The Hf$^{12+}$ ion beams were extracted up to currents of 43 eμA and energy 180 keV; (B) Characteristic spectrum of the iron ion beams extracted from the mVINIS, optimized for the Fe$^{7+}$ beam. The beam energy and current are 105 keV and 74 eμA (10 μA). $U_{\text{ext}}$ is the extraction voltage and $P_{\text{shf}}$ is the microwave power, at the frequency of 14.5 GHz.

3. Irradiation experiments with solid substances ion beams

The ECR ion source was connected to an experimental channel for the modification of materials (L3A) in 1998 [15] providing the large number of experiments with the different low energy ion beams. The important request for many modification of materials were the high ion implantation doses (up to $D=10^{18}$ cm$^{-2}$). In order to recalculate the desired particle doses the multiply charged ion beam currents have to be divided by the beam ionization state. In such situation we have the task to produce as the highest possible intensities of ion beams (usually over 200 eμA) with the mVINIS Ion Source obtaining maximal irradiation doses in a shorter time period (from few hours to several days). The transported ion beams in front of target chamber were additionally reduced with a low beam transmission of the L3A channel (30%) [3].

Since summer 2005, the conventional implantation by solid substances multiply charged ion beams were performed at the L3A channel for the modification of materials. By using ion implantation technique different physical properties of materials (structural, electrical, optical, mechanical etc) expected to be altered to a great extent.

The following irradiation experiments were performed with the new extracted ion beams:

a) Surface and structural modification of fullerenes films by multiply charged nitrogen, boron, and iron ions bombardment.

b) Multiply charged ion beam induced modification of physical properties of the Fe-polymer nano-composite thin films.

c) Modification of surface and structural properties of glassy carbon by boron intensive ion beams.

The studies are based on the Raman spectroscopy, the X photon electron spectroscopy (XPES), the grazing incidence X ray diffraction (GIXD), Fourier Transform Infra Red (FTIR) spectroscopy and the atomic force microscope (AFM) analysis. A recent result obtained by AFM characterization is presented in figure 3. Ion bombardment caused the strong disruption of surface order. AFM analysis has shown that size of sp$^{2}$ clusters had been reduced at the highest implantation doses (in range from 200 to 500 nm). After irradiation experiments by using multiple charged nitrogen ions (N$^{2+}$, N$^{5+}$) and boron ions (B$^{12+}$) the formation of new bonds in fullerenes films have been founded by the FTIR characterization [17].
Figure 3. AFM 3D images of implanted fullerenes thin films (C$_{60}$) by nitrogen, boron and iron ions with different charge states (q), energies (E) and irradiation doses (D). Scan sizes were 1.5x1.5 $\mu$m.

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