TWO-CHAMBER CONFIGURATION OF THE BIO-NANO ECRIS

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Abstract

We are studying the application of the electron cyclotron resonance ion source (ECRIS) for the new materials production on nano-scale. Our main target is the endohedral fullerenes. There are several promising approaches to produce the endohedral fullerenes using an ECRIS. One of them is the ion-ion collision reaction of fullerenes and aliens ions to be encapsulated in the mixture plasma of them. Another way is the shooting of ion beam into a pre-prepared fullerene layer. In this study, the new device configuration of the Bio-Nano ECRIS is reported which allows the application of both methods. The basic concept and the preliminary results using Ar gas and fullerene plasmas are described.

INTRODUCTION

The Bio-Nano ECRIS was designed for new materials production on nano-scale [1,2]. Our main target is the endohedral fullerene, where endohedral means some alien atoms are encapsulated inside the fullerene cage. The endohedral fullerenes have much potential in medical care, biotechnology and nanotechnology. In particular, endohedral Fe-fullerene can be applied as a contrast material for magnetic resonance imaging or microwave heat therapy. There are several promising approaches to produce the endohedral fullerenes using an ECRIS. One of them is the ion-ion or molecule-ion collision reaction of fullerenes and aliens ions to be encapsulated in the mixture plasma of them. Here we call it the plasma method. Another way is the shooting of ion beam into a pre-prepared fullerene layer. Here we call it the ion implantation method.

One of our team has been successfully synthesized the endohedral N-fullerene using the plasma method [3]. Also the endohedral N-fullerene were synthesized using ion implantation method by S. Abe et al. [4]. In addition, several kinds of atomic species, such as Li, Na, K, Rb, and Xe were encapsulated into the fullerenes by ion implantation method [5,6,7]. However, the number of the atomic species, which are successfully encapsulated into fullerene cage, is limited. Also the yield of endohedral fullerenes is not enough to develop them for above-mentioned practical applications.

In this study, the new device configuration of the Bio-Nano ECRIS is reported which allows the application of both the plasma and the ion implantation methods. The ions of the synthesized materials can be checked by in-situ extraction and analysis. This device configuration will make it possible to produce the endohedral-Fe fullerene and improve the yield of the endohedral fullerenes.

DEVICE CONFIGURATION

Since 2006, we have developed and studied the Bio-Nano ECRIS with min-B configuration (see Fig. 1) [1,2,8]. Since 2008, the new device configuration, which we report in this paper, has been manufactured and tested.

Fig. 2 shows the schematic of the new device configuration. In contrast to the normal min-B ECRIS configuration, the plasma chamber is divided into two chambers by installing mesh electrodes and by installing an optional, second chamber (Fig. 3) which is also called processing chamber. Therefore we call this new device configuration the two-chamber configuration. The processing chamber is installed between the normal plasma chamber and the extraction box, and has 8 vacuum ports to host several connections like wave-guide, boat heater, substrate (plasma electrode) bias, substrate cooling, vacuum gauge, etc. In the gas injection side, a 2.45 GHz microwave can be introduced into the 1st chamber via coaxial waveguide. Also a 10 GHz microwave can be introduced in this chamber. There are several gas sources such as a resistance heating oven for fullerenes evaporation and an induction heating oven for iron evaporation, and a gas inlet for N_2, Ar, He, O_2, etc. Thus, we can make several plasmas in the 1st chamber. In the extraction-side 2nd chamber, a 10 GHz microwave can be introduced using a rectangular waveguide (WR75). We note that this frequency combination (2.45 GHz in the 1st chamber and 10 GHz in the 2nd chamber) was decided by some technical reasons and we do not consider them as the final solution. Later other frequencies are planned to be tested in both chambers.

An evaporation boat for fullerene is also installed in the processing chamber. The mesh electrode between the chambers and the plasma electrode can individually be biased to any DC voltage. The plasma electrode (frequently called also as collector or substrate holder) has a hole in its center to allow in-situ beam extraction.

Here let us explain the experimental procedure to produce the endohedral fullerenes using the two-chamber configuration. The gas ions to be encapsulated and the fullerene molecules or ions are generated in the 1st and the 2nd chambers, respectively. The injection of microwave into the second chamber and its power are
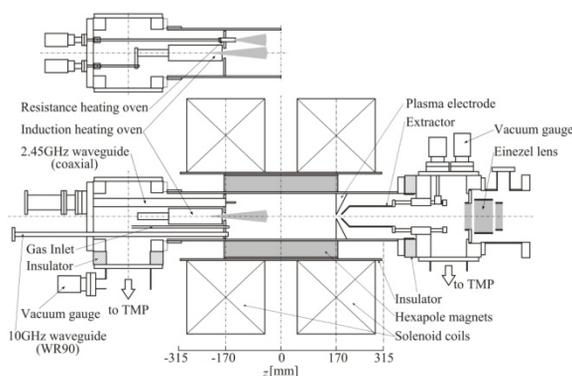
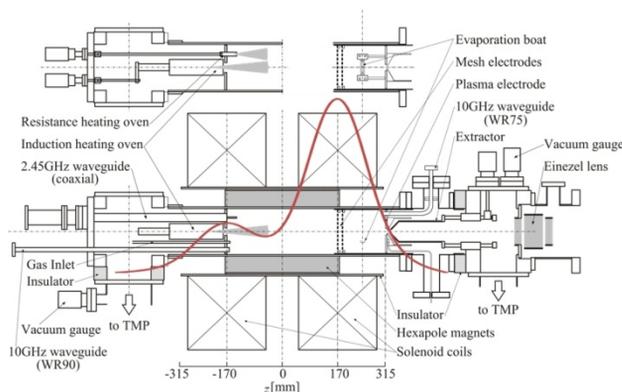Figure 1: Schematic of the normal Bio-Nano ECRIS with min-B configuration.

Figure 2: Schematic of the two-chamber Bio-Nano ECRIS with the optimized axial magnetic field distribution.
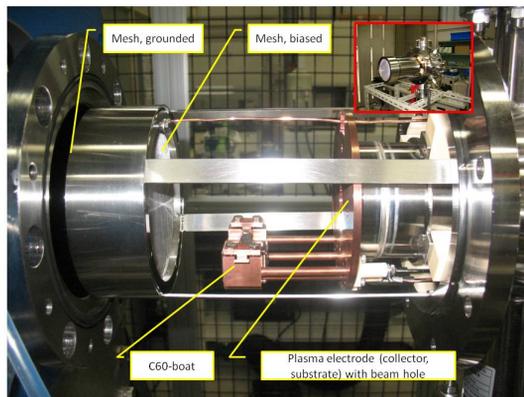

Figure 3: The second (processing) chamber before installation

optional. Thus C_{60} particles with any q charge can be produced in this chamber where q can be 1 (positive fullerenes) or 0 (neutrals) or -1 (negative ions). Furthermore, partly damaged positive fullerene ions can also be produced as C_{58}, C_{56}, C_{54}, etc. ions. The ions produced in the 1st chamber are extracted to the 2nd chamber through the mesh. This mesh actually is a double mesh. The one facing to the 1st chamber is grounded to the chamber and operates as many small plasma

electrodes. The other mesh facing to the 2nd chamber is biased to some negative voltage and operates as many small puller electrodes. In the 2nd chamber vapor-phase ion-ion collision reaction can occur both in the plasma and on the surface of the collector plasma electrode. In the latter case ion implantation or ion beam deposition on the plasma electrode can be done. Thus, we expect that the endohedral fullerenes can be synthesized by ion-ion collision reaction or by ion implantation in the 2nd chamber. The ions in the 2nd chamber are in-situ extracted and analyzed. Also the ions of the synthesized materials can be collected by ion beam deposition and can be examined off-line using different materials analysis tools and methods.

RESULT

We checked the production of plasma in each chamber. The presence of the plasma was confirmed by extracting and analyzing the ions from each chamber. We measured the total extracted ion beam current in the straight beamline using a faraday cup FC1 (I_{FC1}), and the ion beam current for each ion in the analyzed beamline using a faraday cup FC2 (I_{FC2}). The extraction voltage throughout the whole experiment was 5.0 kV.

First, we checked the plasma production in the 1st chamber using Ar gas. Ar gas was supplied from the gas inlet at 0.3 sccm. Then the gate valve of the injection-side turbo molecular pump was closed in order to increase the chamber pressure to get the highest possible intensity of singly charged Ar plasma and beam. The pressure at the injection-side (P_{inj}) is 6.2×10^{-2} Pa, and that at the extraction-side (P_{ext}) is 9.1×10^{-4} Pa. The ion source was tuned for Ar^+ beam. The ions were extracted through the second chamber as described. The mirror coil currents for both injection-side (I_{inj}) and extraction-side coils (I_{ext}) were 100 A and 340 A, respectively (see Fig 4). As will be shown later, we used a very unusual and asymmetrical magnetic field distribution in order to get resonance zones in both chambers. The 2.45 GHz microwave power of 40 W was fed (the forward power is 70W, and the reflected one is 30 W.). Without the bias voltages to the mesh and the plasma electrodes (V_{mesh} and V_{plasma}, respectively), we observed negative current on both electrodes (electron flow dominant). These currents on the two electrodes (I_{mesh} and I_{plasma}) increased toward zero with increasing the negative bias voltages. The I_{mesh} and I_{plasma} became zero at certain negative bias voltages. Then, we observed positive values of I_{mesh} and I_{plasma} at higher negative bias voltages (ion flow dominant). However we must always consider the contribution of both electrons and ions. Finally, the optimized V_{mesh} and V_{plasma} were -1 and -40 V, respectively which resulted in a near zero (-0.6 mA) I_{mesh} and 0.27 mA of I_{plasma}, respectively. The I_{FC1} is 12 μA. We could observe the I_{FC2} of 3.5 μA for Ar^+, I_{FC2} of 0.15 μA for Ar^{++}, and the ion currents due to the impurities such as H_2O^+, OH^+, O^+. If the ECRIS is optimized for the highest plasma electrode current then the axial magnetic field distribution changed (I_{inj} of 160 A, I_{ext} of 130 A) and the

resulted I_{plasma} was 0.7 mA. While this value is higher than the one optimized for FC1, is still not enough for macroscopic material collection. It means we need denser plasma in stage 1.
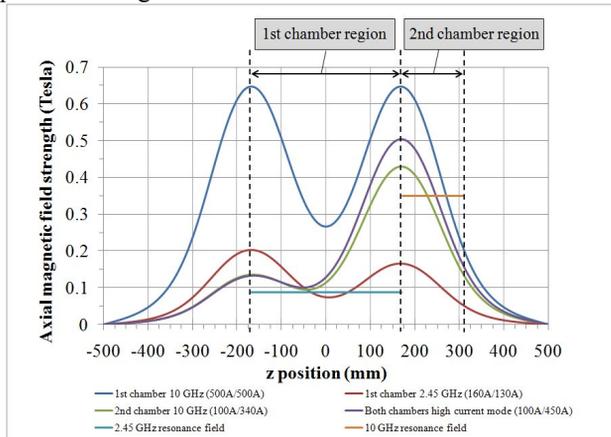

Figure 4: Magnetic field variations of the mirror coils.

Second, we checked the Ar plasma production in the 2nd chamber. First most conditions (mirror coil currents, gas flow rate, pressure, and two bias voltages) are set to the same values as the 1st chamber plasma. A 2.45GHz microwave was not fed in the first chamber. A 50 W 10 GHz microwave power was fed in the 2nd chamber. However, the plasma could not be ignited in the 2nd chamber. This is because the magnetic configuration is not B-minimum type in the 2nd chamber and there is no radial confinement (no hexapole here). If there is plasma in the 1st chamber, the plasma was ignited in the 2nd chamber easily, provided if the magnetic field distribution is suitable for the ECR condition in the 2nd chamber. The optimized I_{inj} and I_{ext} for plasma production in the 2nd chamber were 100 A and 340 A, respectively. This is a very unusual and asymmetrical magnetic field distribution. The high extraction peak however is necessary to get a resonance surface in the 2nd (processing) chamber. The resonance value of 10 GHz is 0.35 Tesla. The low injection peak was necessary to get a resonance zone (or at least a resonance point) in the 1st chamber where the resonance value of the 2.45 GHz frequency is only 0.09 Tesla. The magnetic field distribution in Fig. 4 thus is a result of several compromises. After setting this special magnetic field distribution we generated Ar plasma in the 1st chamber using 2.45GHz, under the above-mentioned conditions. Then, the 50W 10GHz microwave power was fed in the 2nd chamber. We observed the I_{FC1} of 10.8 µA. After that, the 2.45GHz microwave power could be turned off and we could still observe the extracted ion beam current. The I_{FC1} was 0.45 µA. This value is much smaller than that for the 1st chamber plasma. In order to increase the extracted beam current, we checked the effect of the 10GHz microwave power and the extraction-side mirror coil current. We could observe the increase of the I_{FC1} from 0.45 µA to 0.86 µA by increasing the microwave power from 50 W to 100 W. Also we observed the increase of I_{FC1} from 0.86 µA to 40 µA by increasing the I_{ext} from 340

A to 447 A. The I_{plasma} meanwhile was also increased from 0.36 mA upto 19 mA! We could confirm that the magnetic system is very effective to obtain high extracted beam current from the 2nd chamber but, of course, strongly prevents ions from the 1st chamber.

Finally, we tested the production of fullerene plasma in the 2nd chamber. We generated the plasma in the 2nd chamber using Ar mixing gas. Then we increased the evaporation boat current gradually. We used pure C_{60} powder (99.5% purity, nanom purple SU, Frontier Carbon Corporation) as fullerene source. We could observe the I_{FC2} of 100 pA for C_{60}^{+} and the I_{FC1} of 0.4 µA with the boat current of 37 A. The source conditions are as follows: Ar gas flow rate of 0.338 sccm, the P_{inj} of 8.7×10^{-2} Pa, the P_{ext} of 1.4×10^{-3} Pa, the I_{inj} of 139 A, the I_{ext} of 340 A, 10 GHz microwave power of 22 W, the V_{mesh} of -1 V, the V_{plasma} of -20 V. In order to increase the I_{FC2} for C_{60}^{+}, we examined the effect of the 10GHz microwave power and the I_{ext}. The I_{FC2} for C_{60}^{+} did not change with increasing the microwave power up to 40 W. We could observed the increase of the I_{FC2} for C_{60}^{+} from 100 pA to 45 nA by increasing the I_{ext} from 340 A to 400 A.

SUMMARY

The conclusions of the Ar-plasma and C_{60}-evaporation experiments in the two-chamber Bio-Nano ECRIS are summarized. The first and most important result is that the two-chamber configuration ECRIS works in each tested modes. The 1st chamber only operation mode works as traditional B-minimum ECRIS. In the 2nd chamber operation mode only a not-closed ECR-surface exists and both gaseous and C_{60} plasmas and ion beams can be produced. In the two chambers together mode however the configuration is strongly limited by the requirements for the 2nd chamber. A strongly asymmetrical magnetic field distribution is necessary where the extraction peak is much higher than the injection peak. The next technical steps in the two-chamber configuration experiments logically are the testing of other frequencies in the chambers. It can be the application of the same 10 GHz in both chambers or simply the exchange of the present two frequencies.

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