# Diagnostics of a low-pressure ICP discharge using a magnetic sector mass analyser

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**Abstract.** A simple magnetic sector mass analyser with an extraction system has been tested as a tool for monitoring ion contents in a low-pressure inductively coupled plasma in Ar, $N_2$, and their mixtures. The spectrometer demonstrated mass-resolution suitable for ion flux analyses in plasmas of various compositions. The capabilities of the mass analyser are discussed.

## 1. Introduction
Studies of surface modification processes in plasma facilities as well as improvement of their performance require knowledge and control of the plasma parameters. The results of plasma processing depend on plasma composition and degrees of dissociation and ionization. For example, plasma nitriding of metal alloys is very sensitive to the concentration of atomic particles and ions [1], and there are continuous debates about relative contribution of atoms and ions in the nitriding. Besides, the surface modification process can be strongly affected by small amounts of gas impurities and admixtures to the working gas [2, 3]. Therefore, investigation of plasma composition is crucial for technological applications.

Plasma composition is usually estimated using gas mass analysers. However, they are incapable of providing information about atoms and ions in plasma. Optical spectrometry is a useful tool, but it requires nontrivial knowledge how to convert the optical emission signals to real concentrations of atoms and ions [4]. Mass-spectrometry allows accurate determination of relative particle fluxes, but it is not often used since the equipment is usually expensive and complex. Nevertheless, the problem of ion contents measurements in plasma facilities for surface modification has been addressed in a number of papers [5–12], and various mass filters have been developed and applied.

We have recently investigated plasma parameters in an inductively-coupled plasma (ICP) set-up used for nitriding by means of Langmuir probes and optical emission spectroscopy (OES) [13, 14]. Here we describe a simple magnetic sector mass analyser and results of its experimental testing, with the aim of further investigation of plasma ion composition and comparison with OES and gas analyses data.
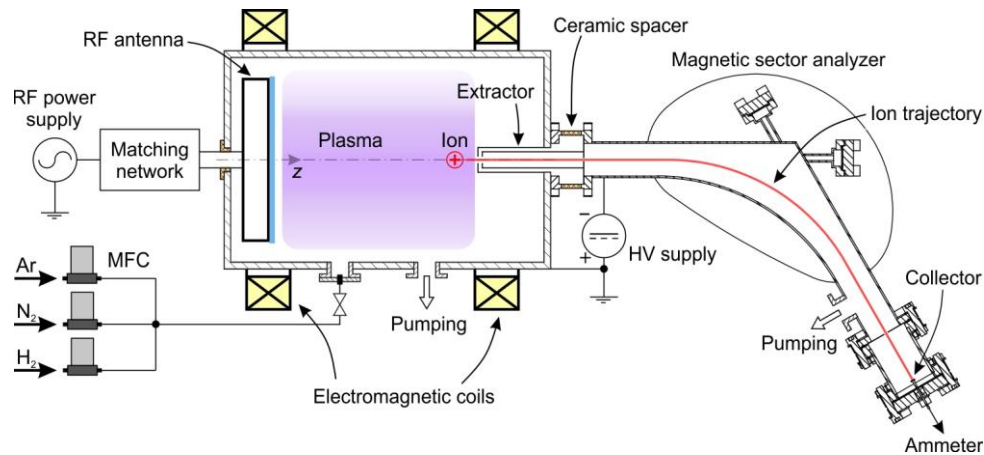
## 2. Experimental
The experiments were carried out in a plasma facility (figure 1) used for surface modification and described in [13]. The ICP discharge was excited by an internal flat circular antenna Beams&Plasmas

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RPG-250 cooled by water and protected by quartz shield. External magnetic field was produced by a pair of DC electromagnetic coils. The magnetic field at the axis $z$ was kept at 2 mT. The forward RF power was 1 kW, and the reflected power did not exceed 10%.



**Figure 1.** Experimental setup scheme.

The ICP vacuum chamber was evacuated to a base pressure of $1\times10^{-4}$ Pa with a turbomolecular pump backed by a dry scroll pump. Argon, nitrogen and their mixtures were used as working gases, and the total pressure was in the range of 0.1–1.5 Pa.

In order to mass-filter the plasma ions, a simple magnetic sector mass analyser (MA) with an electrostatic extractor was attached coaxially to the ICP chamber. The extractor, which was immersed into plasma, was comprised of two-electrode electrostatic lens with circular apertures. The entrance aperture and the extracting aperture were 2 mm and 3 mm in diameter, respectively. The entrance aperture was grounded, while the extracting one and the entire MA unit including the electronic equipment were under a high voltage (up to 5 kV). MA was individually evacuated with a turbomolecular pump backed by a diaphragm pump.

The ion beam was restricted by 2-mm wide slits located at both ends of the separator. The maximum magnetic field produced by the electromagnet was 0.265 T, while the main ion path had the radius of 26 cm. This filter design allowed detection of ions up to $E\times M = 210$, where $E$ is the ion energy in keV, and $M$ — its atomic mass. Therefore, argon ions with energies up to 5 keV could be detected.

The electromagnet was powered by a Matsusada RB5-6P supply. The collector current was recorded with a Keithley 6485 picoammeter. The measurement and control interface was based on NI LabVIEW. The mass analysing unit was fully automated, and the data were transmitted through a wireless protocol because of a high voltage applied.

Plasma density $n$ and electron temperature $T_e$ in the low-pressure ICP discharge used here are nearly uniform along the $z$-direction as our previous studies [13, 14] demonstrated. Therefore, regardless of the extractor immersion depth, the measured ion contents should satisfactorily represent those in the bulk plasma where the samples are usually processed.
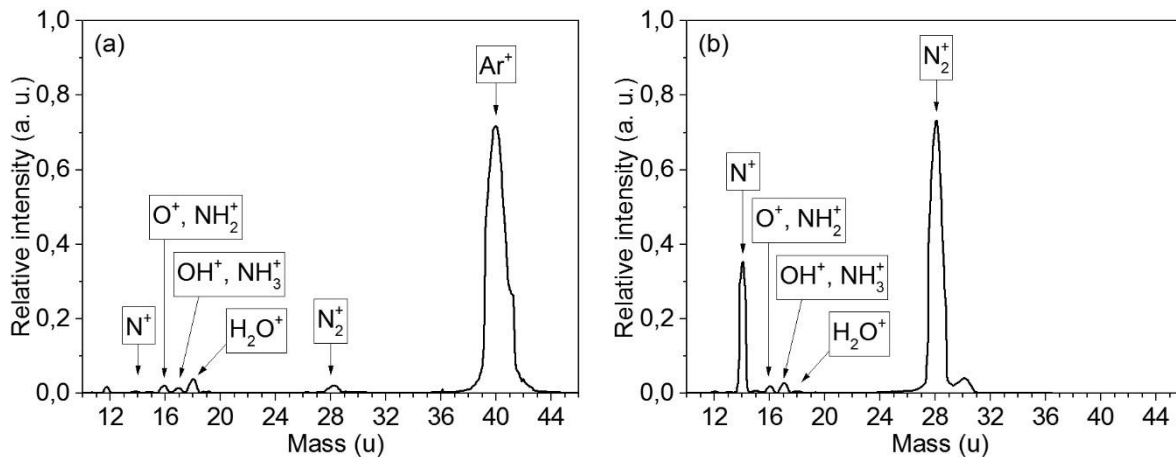
One must note that the extractor in plasma becomes surrounded by a multicomponent double layer, and the extracted fluxes of various species, which have different masses and mobilities, would therefore be different even if their concentrations are equal. This makes the quantitative determination of ion concentrations in plasma a challenging task. Nevertheless, the extractor is in about the same surrounding as the sample embedded in plasma for plasma treatment. Therefore, the mass analyser gives reasonable information about ion fluxes incident on the sample during plasma processing.

The energy distribution of ions in plasma is characterized by their temperature $T_i$, which we assume to be $T_i < 0.1$ eV. Hence, the energies of ions in plasma are much less than the energy they receive
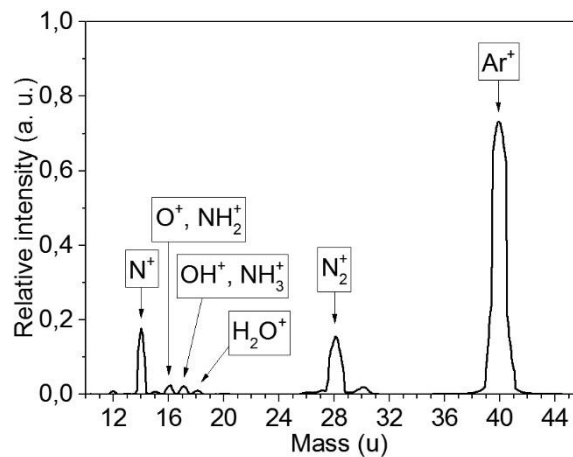
when accelerating to about 1–5 keV. Therefore, one can consider monoenergetic energy distribution of ions entering the magnetic field to be a good approximation.

## 3. Results and discussion

The test measurements were performed with the ICP discharge operated in Ar, $N_2$ and their mixtures at RF power of 1 kW and total pressure of 1 Pa. The relative mass spectra are presented in figures 2 and 3. Note that the intensities in figures are normalized to different peak currents.



**Figure 2.** Mass spectra of ions extracted from the ICP discharge operated in (a) Ar and (b) $N_2$ at RF power 1 kW and total pressure 1 Pa.



**Figure 3.** Mass spectrum of ions extracted from the ICP discharge operated in an Ar–$N_2$ mixture at RF power 1 kW and total pressure 1 Pa.

One can see that the dominant peaks in figure 2 are ions of the working gas. Besides, one can see masses $M = 16$–19 in all spectra. In the case of argon plasma, the relative intensities of these masses are similar to those observed by gas analysers. In the case of nitrogen plasma, however, the intensity of $M = 17$ exceeds that of $M = 18$, and this can be explained by formation of $NH_x$ molecules due to interaction of nitrogen with residual or impurity hydrogen. Formation of $NH_x$ molecules was discussed in many publications [15].

If the discharge takes place in the gas mixture Ar–$N_2$, relative intensities of $N_2^+$ and $N^+$ ions change: more atomic ions are produced, and this may be due to more intense dissociation of nitrogen

molecules in presence of argon. The effect of correlation between the $N^+$ content and Ar flow will be examined in future.

## 4. Conclusion

A magnetic ion mass analyser with an extraction system was developed and tested in a low-pressure RF plasma in Ar, $N_2$, and Ar–$N_2$ mixtures. The mass resolution is sufficient to control ion spectra in technological plasma. The obtained mass spectra correspond to ion fluxes that can be expected on samples embedded in plasma. Main ion species are ions of working gas. Formation of $NH_x$ molecules in nitrogen discharge was suggested to explain the behaviour of ions with $M = 17$. Change of relative intensities of molecular and atomic nitrogen ions was observed if Ar was added to $N_2$ in the discharge.

Since the ions entering the magnetic sector are extracted from different equipotential surfaces, their energies must be mass-dependent. To take this effect into account, an energy analyser is planned to be developed for this mass spectrometer.

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