Conical octopole ion guide: Design, focusing, and its application to the deposition of low energetic clusters

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A design of a radio-frequency (rf) octopole ion guide with truncated conical rods arranged in a conical geometry is presented. The performance is tested in a cluster deposition apparatus used for the soft-landing of size-selected clusters on well-characterized substrates used as a model system in heterogeneous catalysis in ultrahigh vacuum. This device allows us to focus 500 pA of a mass-selected Ni_{20}^{+} cluster ion beam from 9 mm down to a spot size of 2 mm in diameter. The transmittance is 70% ±5% at a rf voltage of 420 V_{pp} applied over an amateur radio transceiver with an interposed homemade amplifier-transformer circuit. An increase of the cluster density by a factor of 15 has been achieved. Three ion trajectories are simulated by using SIMION6, which are relevant for this focusing device: transmitted, reflected, and absorbed. The observed effects in the simulations can be successfully explained by the adiabatic approximation. The focusing behavior of the conical octopole lens is demonstrated by experiment and simulations to be a very useful technique for increasing molecule or cluster densities on a substrate and thus reducing deposition time. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2162439]

I. INTRODUCTION

A focusing ion-beam technique is able to increase the cluster flux density on a substrate. This is necessary for special analytical techniques which only probe small areas. Small sample areas are important in experimental setups such as in cluster depositions on cantilevers used as microcalorimeters (very small target area: $50\times500\ \mu m^2=2.5\times10^{-4}\ cm^2$),[1] in cavity ringdown spectroscopy,[2] as well as in guiding of molecular and cluster beams through differentially pumped vacuum chambers. Especially in electrospray ionization sources, the ion beam needs to be transported from its origin at atmospheric pressure down to (ultra)high vacuum for analysis or deposition.[3] For this application several differential pumping stages separated by small orifices are necessary. The effective guidance through these pumping stages without divergence of the molecular beam is important for this technique. In a cluster deposition apparatus used for the soft-landing of size-selected clusters the diameter of a typical cluster ion beam is 5–10 mm. All these applications require an efficient focusing device avoiding the loss of transmission.

In addition the desired mass range should cover several orders of magnitude (in our experiment for mass-selected clusters from 50 up to 4000 amu).[4] For these aims the presented radio-frequency (rf) octopole ion guide is a simple device to reach high cluster current even for broad mass or energy distributions.[5]

To achieve strong focusing of the cluster ion beam the parallel octopole geometry is modified to a conical shape. There are mainly electrostatic-conical octopole in the literature reviewed without using a rf device.[6–9] A different possibility to focus an ion beam is the so-called collisional focusing ion guides (CFIGs),[10,11] which usually consist of a quadrupole[12,13] or the sextupole ion-beam guide[14] (SPIG) besides the rf-driven ion funnels.[15,16] In all three cases the focusing behavior is due to collisions of the ions with an inert buffer gas at high pressure by reducing the axial kinetic energy in combination with a radial containment established by a rf field.[10–12] Simulations performed for the CFIG device can be found in Refs. 11, 15, 17, and 18. Conical-shaped rods have been already used partially in the CFIG case for changing the axial field along the rods in the direction of the z axis to haul the ions out of the rf device. In the cluster deposition the focusing device is situated under high- to ultrahigh-vacuum conditions ($<10^{-7}$ mbar). In this low-pressure regime the collisional method is inapplicable.

The presented experiments, with the latest designed conical octopole ion guide, demonstrate the expected increase of the cluster density on the substrate successfully. The transmitted cluster ions reach up to 70% ±5% of the overall cluster ions transported into the conical octopole at a rf voltage of 420 V_{pp}. It can also be shown that the beam diameter from around 9 mm (typical value in our experiment) decreases down to 2 mm.

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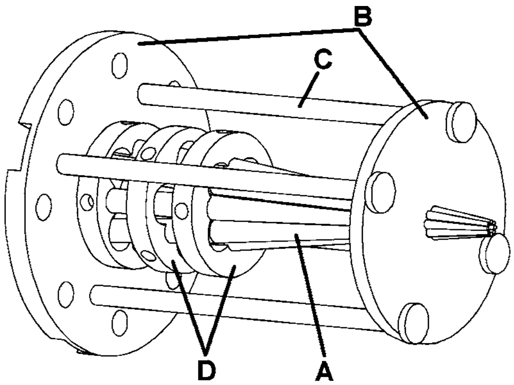

FIG. 1. Three-dimensional (3D) view of the conical rf-driven octopole ion guide. Eight truncated cone-shaped rods A (from ∅ 3 to ∅ 0.5 mm) are arranged in a conical geometry. Two Teflon plates B (one at each end) with four carriage bolts C and two metal collars D (in the cylindrical part) keep the conical geometry. The ion entrance orifice opens 9 mm in diameter and the exit focuses to around ∅ 2 mm (exit diameter is 1 mm).

Theoretical considerations on radio-frequency-driven ion optics can be carried out by different means. Although it may be impossible to solve the equation of motion for a charged particle in a complicated, time-dependent, inhomogeneous rf field, simulations and approximative methods are mostly used. A very popular software tool is the SIMION6 package,[19] which solves the Laplace equation for a given geometry of electrodes with fixed voltages to get a potential array. Furthermore, ion trajectories are simulated for the resulting potential. SIMION can treat electrical and magnetic fields, whereas only the first option is needed in this work.

Making the assumptions, that the electrical field in the rf device only changes slightly as a function of the position of the charged particle, and furthermore, that the radio frequency is high enough to keep the oscillating amplitude small, justifies the use of the adiabatic approximation.[5] The time-dependent potential is therefore replaced by a static effective potential and by a stability parameter called adiabaticity. Such calculations allow us to determine under which conditions an ion trajectory will be stable. The simulations for the conical-shaped rf octopole ion guide are able to reproduce the experimental results quite well, and it is possible to explain the principle of the conical rf device in dependence of the particle's kinetic energies.

II. EXPERIMENTAL SETUP AND THEORETICAL METHODS

A. Geometry of the conical ion guide

The conical octopole ion guide consists of eight conical circular rods made of stainless steel. The rods are hold by two Teflon plates at each end stabilizing the device (Fig. 1). The right Teflon plate is connected by four long carriage bolts, to the main Teflon plate, at the left side of the octopole drawing. Furthermore, two metal collars are attached to four alternating rods each for applying the rf/dc voltages.

For the realization of the conical conformation, the rods are arranged in a way that the inner diameter of the octopole rejuvenates from 9 mm at the entrance (left side of Fig. 1) down to 1 mm at the exit (right-hand side). At each end of the rod arrangement, two Teflon plates define the octopole

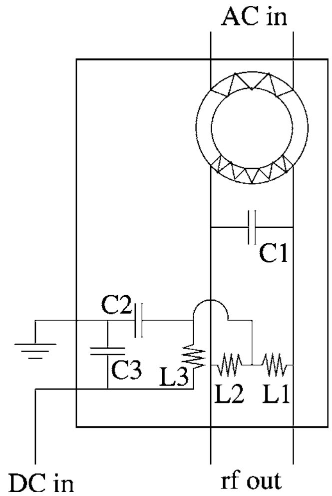

FIG. 2. Amplifier-transformer circuit for the rf output of the radio transceiver and the dc-floating input of a commercial power supply is drawn. L1, L2, and L3: 2 mH; C1: 50 pF; C2: 1000 pF; C3: 100 nF; and turn rate of transformer coil 3:12.

geometry. The big Teflon plate on the left side is also used to mount the conical octopole by using four screws in an ion guide assembly, for instance, onto a quadrupole mass spectrometer (in this case ABB Extrel Merlin, mass limit of 4000 amu).

The above-described geometry derives from an already established cylindrical octopole ion guide, with an inner diameter of 9 mm ($2r_0$) and rod diameters of 3 mm ($2R$). The important ratio R/r_0 amounts to 0.33 (R: rod radius and r_0: inner radius of the rod arrangement or the so-called trap radius), which fits exactly with Eq. (1) in the literature ($2n$: the number of rods).[5]

$$R = \frac{r_0}{(n-1)}. \qquad (1)$$

The target is to keep this ratio constant all along the conical octopole. Thus, the rods are tilted to the z axis of the conical octopole by an angle of 5° resulting in the following design.

Each rod is divided into two parts: a 20-mm-long cylindrical part (∅ 3.0 mm, $R/r_0=0.33$) for the mechanical and electrical connections and a 40 mm conical part, which is truncated at the smallest rod diameter (∅ 0.5 mm, $R/r_0=0.5$). The slight increase of the R/r_0 ratio along the conical octopole is the result of the technical limitations of dressing to size such rods. At the cylindrical part of the rods, two metal collars are used to stabilize their positions, being fixed with screws (M1.6) in a way that they are electrically isolated against each other. This is realized by alternately mounting the rods to the collars.

The conical octopole ion guide is driven by a superimposed dc on an ac voltage. The ac voltage is generated by an amateur radio transceiver (Kenwood TS–570D with FM unit), which is connected to both metal collars of the octopole. For the purpose of increasing the rf signal and floating the rf signal by a dc voltage independently, both voltages are applied to the ion guide via an interposed homemade amplifier-transformer circuit (Fig. 2). The frequency of the applied voltage is tuned automatically by the radio transceiver matching the resonance frequency of the entire octopole-amplifier-circuit system. Related to ground, typical values for the rf voltage are between 200 and 450 V_{pp} at 13.948 MHz. The voltage is set by tuning the applied rf power to the radio transceiver.

The amplifier circuit consists of a capacitor C1 (50 pF) between the transformer coil (coil ratio=3:12) and the octopole electrodes. Two homemade coils L1 and L2 (both 2 mH) are used to float symmetrically the rf signal by the dc voltage. The dc voltage is provided by a commercial power supply, with a range of 0 to ±300 V. The rf center voltage can be changed easily and thus the focusing behavior of the entrance of the conical octopole can be optimized. To ensure the decoupling between the rf and the dc circuit a combination of two capacitors C2 (1000 pF) and C3 (100 nF) and a coil L3 (2 mH) is used (left-hand side of Fig. 2).

B. Theoretical consideration

In order to analyze the presented device beyond the cluster current measurements, simulations with SIMION6, as well as theoretical considerations based on the adiabatic approximation, were performed.

The conical octopole was implemented in SIMION using ten points per millimeter resolution to reproduce the shape precisely. Randomized ion packages with starting parameters closely matching the experimental conditions were used for the simulations. The distance between the axis and the starting point was between 0 and 4 mm, corresponding to the radius of the upstream lens. The starting time was randomized between 0 and 50 ms and the angle between the axis and the ion direction between 0° and 5°. Like shown in the experiment Ni_{20}^{+} (m=1174 u), clusters were used under consideration of energies between 0.5 and 20 eV.

Though the simulations can reproduce the experimental results, they do not give an explanation for the observed effects. That is why for further investigations the effective potential

$$\Phi_{\mathrm{eff}} = \frac{q^2|\boldsymbol{E}(r,\theta,z)|^2}{4m(2\pi f)^2} \approx \frac{4}{k^8 z^8}\frac{q^2 V_{\mathrm{rf}}^2}{m(2\pi f)^2}\left[r^6 + \frac{r^8}{z^2}\cos^2(4\theta)\right] \tag{2}$$

and the adiabaticity

$$\eta = \frac{2q|\nabla \boldsymbol{E}(r,\theta,z)|}{m(2\pi f)^2} \approx \frac{24}{k^4 z^4}\frac{qV_{\mathrm{rf}}}{m(2\pi f)^2} r^2 \tag{3}$$

were calculated and used to interpret the data obtained from experiment and simulations. The static field $\boldsymbol{E}$ was approximated from the potential of an infinite long, parallel octopole:

$$\boldsymbol{E}(r,\theta,z) = \nabla\left[V_{\mathrm{rf}}\left(\frac{r}{kz}\right)^4 \cos(4\theta)\right], \tag{4}$$

where the constant $k=\tan\alpha = rz^{-1}$ is the geometrical parameter of the device with the opening angle α. To simplify the following considerations, the z axis has been rescaled to $z=0$ to be the tip of the conical octopole with an opening diameter of 1 mm.

In the resulting expressions, the effective potential is proportional to r^6 and the adiabaticity to r^2. Both contain the rf amplitude V_{rf} as to be the only parameter that is tunable in the experiment, while the frequency is determined by the octopole-amplifier circuit. Along the z axis the effective potential and the adiabaticity drop very fast ($\eta \propto 1/z^4$, $\Phi_{\mathrm{eff}} \propto 1/z^8$).

The calculation of the effective potential and of the adiabaticity allows us to consider in which way the ions are affected by the rf field. As a typical value the effective potential from 80% of the octopole radius is considered to be the limitation for a particle's transversal kinetic energy. Ions with higher energies will get over this barrier and hit the rods or leave the guided beam. The adiabaticity is a parameter to consider the energy conservation of the system. It is a measure for the energy transfer between the rf field and the ion. If the particle gains kinetic energy, its oscillation amplitude in the rf device might rise, which corresponds to a heating of the beam. If the value of the adiabaticity is lower than 0.3, ions do not gain kinetic energy from the rf field.[5] Their trajectories will remain stable in the case that they fulfill the condition of the transmission given by the effective potential.

III. RESULTS AND DISCUSSION

In order to evaluate the performance of the conical octopole ion guide, the spatial beam profile as well as the transmittance of a mass-selected Ni_{20}^{+} cluster beam were studied. For both purposes a Faraday plate was used to measure the cluster ion current. The plate was mounted 1 mm downstream to the exit of the octopole ion guide, movable by a linear drive in the plane perpendicular to the beam axis. This geometric arrangement mimics the actual configuration in which clusters are deposited on a substrate.

A. Beam profile

For establishing the beam profile the mass-selected Ni_{20}^{+} cluster ion current was measured as a function of the plate position according to the center of the cluster beam. The spatial beam profile was calculated from the derivative of the measured cluster current with respect to the plate position. Figure 3 shows normalized experimental (open circles) and theoretical values of the ion current density (dashed curve).

The cluster ion spot will be approximately 2 mm in diameter [full width at half maximum (FWHM) of 1.3 mm]. Although the exit orifice of the conical octopole constitutes only of 1 mm in the diameter, the ion beam already expands almost twice in size at 1 mm downstream. It should therefore be emphasized that a substrate for cluster deposition must be placed as close as possible to the octopole exit in order to prevent from an expansion of the beam and thus from a decreasing cluster density. Indeed when the Faraday plate is placed at 10 mm downstream from the octopole exit, the ion beam spreads to about 8 mm in the diameter. A reason for this beam divergency downstream to the conical octopole is the particle's energy conservation, which is shown in the simulations (see below). Summarizing, the introduced collision-free device is not a so-called phase-space compres-

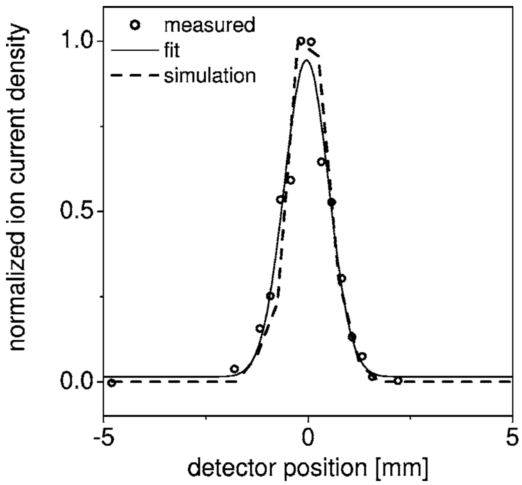

FIG. 3. Spatial cluster ion-beam profile 1 mm downstream from the exit of the conical octopole ion guide. It exhibits a spot size about 2 mm in diameter and a full width at half maximum (FWHM) of 1.3 mm. The same values can be obtained using SIMION simulations (dashed line).

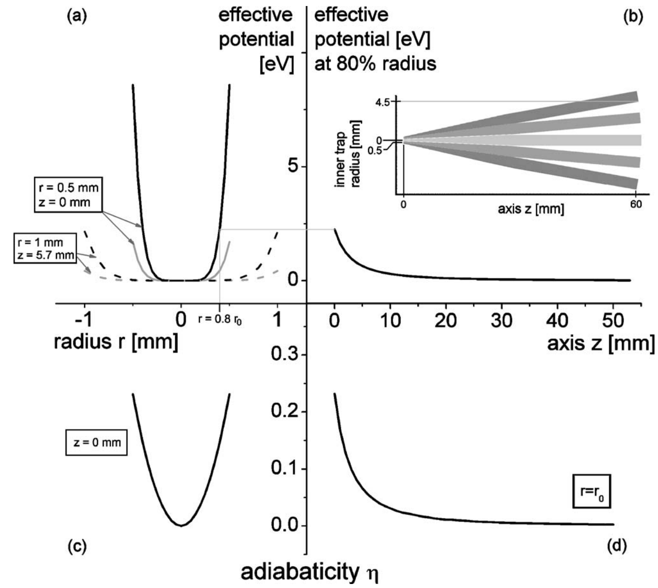

FIG. 4. Effective potential [(a) and (b)] and adiabaticity [(c) and (d)] perpendicular and along the beam axis of the conical octopole. Two cases are displayed in (a): V_{rf}=100 V (gray) and V_{rf}=225 V (black); in (b) and (c) only the last case. (a) The effective potential shows the typical potential wall $\propto r^6$. The typical value, the potential wall at 80% of the trap radius, is indicated for z=0 mm. For the cut at z=5.7 mm resulting with an inner radius r=1 mm a widening of the effective potential along the octopole rods is shown (dashed lines, gray and black). (b) The effective potential drops very fast ($\propto z^{-8}$) along the z axis. The inset shows the geometry of the octopole for a better visualization. (c) and (d) The adiabaticity never exhibits values high enough (η>0.3) to affect the cluster ion beam, although rising $\propto z^{-4}$ along the z axis.

sor, such as the CFIG devices,[12] thus it must be divergent and it is optimized on the entire setup of the cluster deposition apparatus.[4]

The behavior observed in the simulations is similar to the experimental results. The current distribution of the target shows an agreement with the experiment (see Fig. 3). Calculated from the spot size, the beam spread angle estimates to 26° in the experiment. The initial beam spread was set to 5° in the simulations, widening up to 22°, after passing the octopole lens.

The calculation of the adiabaticity [Figs. 4(c) and 4(d)] shows values quite below the critical value of 0.3 for the highest possible amplitudes. This means that an ion beam is not heated by energy transfer from the rf field in the conical octopole. Furthermore, the energy distribution obtained from the simulations shows that the particle energy is almost conserved. The transfer of energy only occurs from axial into transversal motion.

A detailed analysis of the particle trajectories reveals that the increased beam spread occurs due to the conical geometry. Besides stable trajectories, which allow the ion to pass the octopole (transmitted) and unstable trajectories (absorbed), there are stable but reflected trajectories (reflected). Those three kinds of ion paths are shown in Fig. 5 (right side). Their occurrence can be understood by means of the adiabatic approximation mentioned before. Both effective potential and adiabaticity show low values in the wide part and high values in the narrow part of the conical octopole. This denotes that the ions can move nearly free in the first, wide part, but they are affected by the rf field in the second, narrow part.

Along the full length of the octopole, there is a large area with low potential values close to the axis, where the ions can pass the octopole lens [Figs. 4(a) and 4(b)]. The ion beam is confined to the inner part of the octopole lens, by the effective potential, generated through the rf voltage. The higher the rf power is, the higher the effective potential is. A particle hitting this barrier will be either reflected there when the radial part of the energy

$$E_r = E - E_a = E - mv_z^2/2 \propto p_r^2 \quad (5)$$

is lower than the barrier height. It will leave the guided beam in case of a higher radial energy E_r compared with the potential wall (E_a: axial energy and p_r: radial momentum). Hence, beams exhibiting a high spread angle also show high values of E_r for their particles.

Due to the conical shape of the octopole, an ion moving parallel to the beam axis will change its direction by approxi-

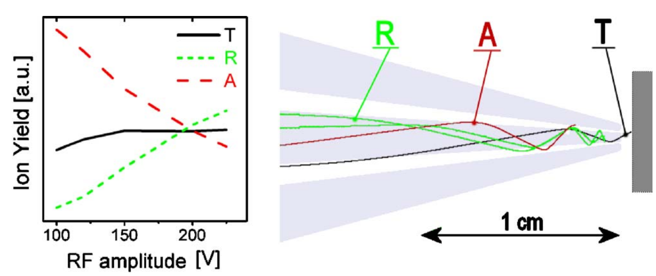

FIG. 5. (Color online) Three possible ion trajectories in the conical octopole are depicted. R: reflected Ni_{20}^{+} cluster ion trajectory after several encounters with the effective potential wall, changing the direction by approximately 10° each time with the 5° tilted rods. A: absorbed ion trajectory by hitting an electrode. T: transmitted particle trajectory. Left-hand side: Normalized reflected, absorbed (unstable), and transmitted ion currents dependent on the rf voltage obtained by simulations. For rising amplitude the effective potential increases. Therefore, fewer ions are able to leave the guided ion beam. The transmitted current on the other hand does not rise in such a strong way because more ions became reflected instead of getting transmitted or absorbed. Right-hand side: The tip of the conical octopole with the simulated cluster ion trajectories is sketched (simulation with 10° tilted rods is shown for easier visualization).

mately 10° in the case of reflection at the effective potential wall. Because of the rising radial energy component, this change in direction can be directly related to a heating of the beam, according to Eq. (5). After at least ten of such reflections at the potential wall, the particles direction will have been turned around, leaving the octopole lens towards the entrance (reflected particle). This effect is illustrated in Fig. 5 (right graph), with simulated trajectories for each transmitted (T), absorbed (A), and reflected (R) particles. Ions, reflected fewer times at the effective potential, may be transmitted or absorbed. These ions, leaving the octopole in the original direction, are detected. Due to the reflections at the potential wall, they show a high radial energy and hence a high beam spread angle. The mechanism of the beam heating in the case of the conical octopole is therefore clearly related to the geometrical shape, while the ions do not gain energy from the rf field.

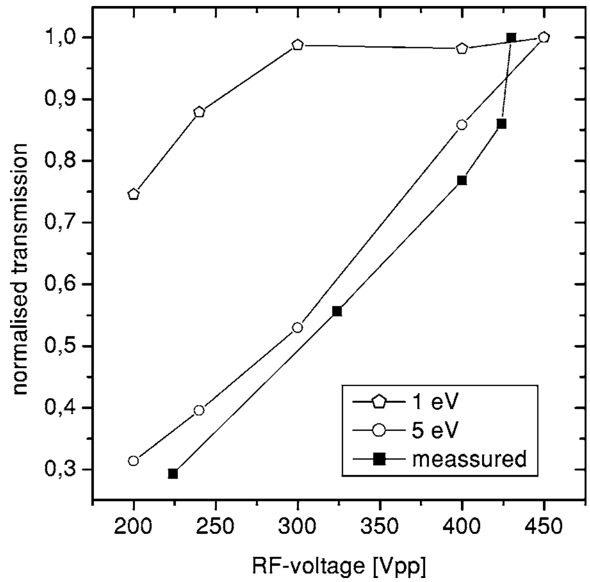

FIG. 6. Normalized transmission of 500 pA Ni_{20}^{+} cluster ion beam through the conical octopole ion guide, compared with the simulations as function of the applied rf voltage (V_{pp}). The transmission of the 5 eV cluster ions shows a quite good agreement with the measured cluster currents.

B. Transmittance

For establishing the transmittance of the ion guide, a 500 pA mass-selected Ni_{20}^{+} cluster ion beam was transported and focused by the octopole, with various rf-power outputs, that can be associated with the applied rf voltages, at specific resonance frequency. The dc voltage was manually adjusted in a range of +5 to −5 V in order to optimize the ion current. The Faraday plate 1 mm downstream of the octopole exit was used to measure the net transmission current.

The transmittance of the ion guide increases with enlarging rf voltage. At 420 V_{pp} a transmittance of about 70% ±5% of the cluster ions can be obtained. It must be emphasized that the conical device works under high to ultrahigh vacuum, focusing an ion current of 500 pA at only 420 V_{pp} of rf voltage. Compared with "gassy" focusing devices such as CFIG (Ref. 10) and SPIG,[14] gaining a transmission efficiency up to 90% at high pressure, the presented conical octopole has a quite good performance excluding an absolutely confined cluster beam beyond the octopole exit such as in the CFIG case.[15]

To avoid overheating of the amplifier-transformer circuit, and thus damaging the radio transceiver, the rf-power outputs were limited to 25 W, which corresponds to 420 V_{pp} at the given resonance frequency.

Figure 6 shows the experimental results compared with the data of the simulations. They were carried out for several particle energies between 1 and 10 eV, whereas the results for energies around 5 eV show the best matching of the simulated curves with the experiment. For the unknown experimental energy distribution of the cluster ion beam, a distribution between 0.5 and 20 eV was taken. The agreement of the experiment and theory also justifies the adiabaticity approximation.

The analysis of the simulation data shows a rise of the reflectance and a decreasing absorbance with increasing rf voltage (see left graph of Fig. 5). Also a difference between high and low energetic particles is observed (Fig. 6). The reflection of particles only plays a role for low energies. For high energies, the only mechanism that determines the ion current is the absorption one. In both cases a stronger decrease of the absorbance, compared to the rise of the reflectance with the rf voltage, is observed.

Which type of mechanism, reflection or absorption, determines the loss of ions in the octopole depends strongly on the starting condition. An ion, injected under a high angle or with high energy, will easily overcome the low barrier in the wide part of the octopole lens [see Fig. 4(b)], while an ion of low energy will rather be reflected on the potential wall. In the second case the energy might be low enough, that the particle is reflected sufficiently on the potential wall, to change its direction. Therefore, in the case of high energy or big angle starting conditions, absorption dominates the ion loss and in the case of low energy, reflection does.

IV. SUMMARY

A design for focusing a cluster ion beam by a conical octopole ion guide onto a target is presented. A higher focusing behavior is achieved by this construction, compared with standard cylindrical octopole ion guides or electrostatic lenses. Compared with focusing devices such as CFIG and SPIG, respectively, nearly the same transmission efficiencies are obtained but under high-vacuum conditions in the presented case. 500 pA of a Ni_{20}^{+} cluster ion beam was successfully focused from 9 onto 2 mm spot size in diameter, with a transmittance up to 70% ±5% at 420 V_{pp} of rf voltage. Thus, the cluster density increases by a factor of about 15, compared to the octopole entrance orifice. Simulations are able to reproduce the experimental beam profile quite well.

It is shown that there are three major particle pathways in the conical octopole: transmitted, absorbed, and reflected. Theoretical considerations using the adiabatic approximation successfully explain the observed effects. With both simulation and theoretical consideration, it is possible to explain the focusing behavior and the current-rf-voltage characteristics of the octopole lens. The heating of the cluster ion beam occurs due to the conical shape of the focusing octopole ion guide and not due to the rf field. According to the adiabaticity parameter η lower than the critical value of 0.3, the ion beam is almost conserved. The ion trajectories are only affected by the rf field in the narrow part of the conical octopole by transferring axial into transversal kinetic energy. The transmitted cluster does not gain axial kinetic energy. With a radial energy E_r higher than the potential wall, the cluster ions get absorbed. For lower radial energies ions can be transmitted or reflected. In the latter case, the ion trajectories have to be reflected approximately ten times at the potential wall before they change their direction leaving the conical octopole through the entrance orifice.

The conical octopole lens proved to be a very useful tool for the focusing of ion beams for a wide range of parameters, such as energy or spatial distribution.

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