

An experimental setup for studying the core-excited atoms and molecules by electron impact using energy analysed electron–ion coincidence technique

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Abstract: Operation and performance of an apparatus for studying the decay dynamics relevant to core–hole decay processes in atoms and molecules excited by energetic electrons using an energy analysed electron–ion coincidence technique are described in some detail. The setup consists of a time- and position sensitive double-field linear TOF mass spectrometer coupled with a dual MCP detector and a single-pass CMA to select the energy of detected electrons. Details of different components involved in the setup are presented and discussed. To demonstrate the performance and capability of the apparatus, we present some typical results extracted from the TOF argon ion-mass spectra observed in coincidence with 18-energy selected electrons emitted from interaction of a continuous beam of 3.5 keV electrons with a dilute gaseous target of argon atoms. Specifically, the variation of relative correlation probability for the final ion-charge states Ar^{1+} to Ar^{4+} produced in the considered collision reactions as a function of energy of emitted electrons is determined and discussed.

Keywords: Core–hole decay processes; TOF mass spectrometer; Relative correlation probability; Energy selected electron–ion coincidence technique

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1. Introduction

Multiple-ionization of core-excited or ionized atoms and fragmentation pathways of doubly or highly ionized molecules have been investigated extensively in the last two decades, in particular by photo-ionization [1–5] and by electron impact ionisation [6–11]. The multiply charged ions of atoms and molecules are produced through different processes, for instance, direct double Auger (DDA) process, auto-ionization, Coster–Kronig (CK) transitions followed by Auger cascade and shake processes. Such studies have been made mostly by mono-energetic X-rays and synchrotron excitation sources. The multiply charged molecular ions formed as a result of core ionization followed by Auger decay rapidly dissociate in charged fragments due to Coulomb explosion. Among different techniques, the (Auger)

electron–ion coincidence is well suited to study the multiply charged atomic and molecular ions formed by impact of photons or by charged particles.

In an ionisation process, electrons and ions of different energies are produced. With the help of a coincidence technique it is possible to examine the particular ions that are formed together with the electrons with specified energies. The electrons and ions produced from one event arrive at their respective detectors at different timings after their formation and although the signals at the detectors are not simultaneous they do have a definite temporal relationship. Ionic species arising due to the interaction of excitation source can be correlated with a particular electronic state of the molecular ions as their precursors by observing mass spectra in coincidence with Auger electrons, which have specific kinetic energy and are emitted in the formation of a specific electronic state of the molecular ion. In TOF mass spectrometry the kinetic energy of the ions is determined so one can link the excitation energy of a molecular ion

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state to the internal energy of the fragments [12]. Multiply charged molecular ions produced due to inner-shell ionisation following Auger or cascade Auger decay processes are meta-stable and due to Coulomb explosion, these ions fragment into two or more than two ion products.

A large majority of Auger electron–ion coincidence techniques reported in the literature is based on photo-excitation [13–17]. However electron impact excitation of atoms and molecules has been also performed and reported in the literature [18–23]. It is known that the nature of the electron energy spectrum is independent of the source of excitation. So, the electron spectra resulting from photo-excitation is identical to that from charge particle excitation. In photo-excitation, the complete energy of photon is transferred to the atoms or molecules while in the case of a charge particle impact, only a fraction of the kinetic energy of the projectile is transferred to the target. We have performed the (Auger) electron–ion coincidence experiments by making use of a continuous beam of electrons of a chosen energy.

In this paper we report on the operation and performance of an apparatus initially developed for studying the ion-surface interactions under bombardment of energetic charged particles [24]. The characteristic feature of the present experimental set up is an employment of a continuous electron beam as an excitation source along with a single pass cylindrical mirror analyser (CMA) for energy analysis of the ejected electrons from the collision reaction. If one uses on contrary a pulsed electron beam, then the removal of false electron–ion coincidences becomes a problematic issue causing a severe difficulty to analyze weak structures present in the ion-spectra. The novelty of the present set-up is its potential use for analytical mass spectroscopy, since atomic core-holes usually decay through Auger transitions which decompose the contributions related to the specific final ionic charge states of the atoms under consideration. Such studies have shown different decay schemes which lead to different structures in the non-coincidence Auger spectra. Furthermore, interesting studies using this set up can be made to provide detailed information about the fragmentation dynamics of highly charged molecules excited by energetic charged particles, for instance, the kinetic energy release of the fragment ions, their angular distributions and the molecular ionic states involved in the fragmentation process. As an example of the performance of the apparatus, some experimental results on decay of L-shell hole created in argon atoms by impact of a continuous beam of 3.5 keV electrons are presented. The resulting different argon ion charge states are observed in coincidence with 18-selected energy of ejected electrons.

2. Experimental details

The measurements of at least two products “in coincidence” require that both the products to be detected occur on a time scale which is short compared with the resolving time of the coincidence counting device. Often the counting rates in coincidence experiments are extremely low and rather long times are needed to accumulate data with sufficient statistics. Hence a careful optimization of the experimental setup is required.

A photograph of the top-view of the presently modified experimental setup is shown in Fig. 1. The setup consists of a scattering chamber equipped with: Faraday cup, vacuum pumping system, electron gun and gas assembly, spectrometers (TOF mass spectrometer and cylindrical mirror analyzer (CMA) and data acquisition system and associated electronics. Different components of the set up are summarised in the following paragraphs.

A scattering chamber employed in the present experiments has been already described in detail [24]. One of the fifteen CF-35 ports adopts an electron gun and the three CF-150 ports are used to mount a CMA, a TOF mass spectrometer and two turbo molecular pumps (TMPs). A Faraday cup is mounted in front of the electron gun at a distance of about 150 mm to collect the transmitted electrons from the interaction zone. The interior surface of the

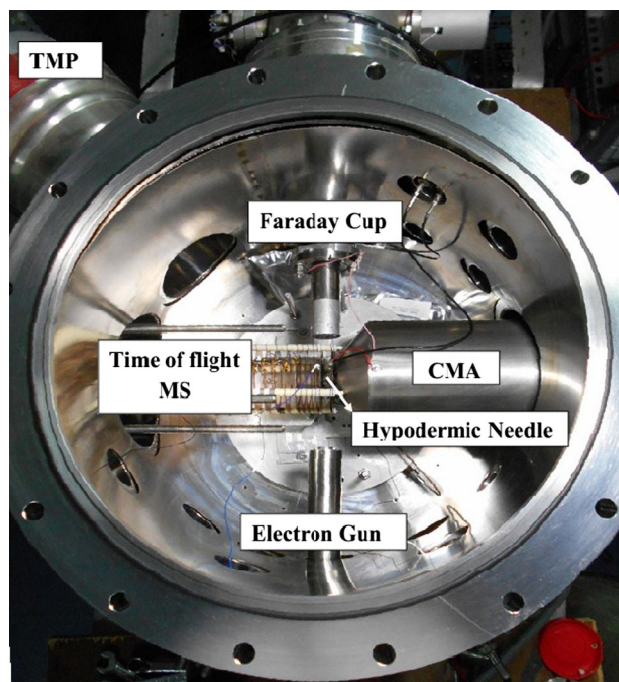


Fig. 1 Top-view of the scattering chamber showing electron gun, Faraday cup, double-field TOF mass spectrometer, single-pass cylindrical mirror analyzer (CMA) and gas target assembly to study core-hole decay processes in atoms and molecules using energy resolved electron–ion coincidence technique

chamber has been electro-polished to reduce out-gassing and covered with a 0.25 mm μ -metal shielding sheet to minimize the effect of external magnetic fields. The top flange of the chamber is sealed with a viton-ring with which the base pressure of 8.5×10^{-8} Torr is routinely achieved without baking the scattering chamber.

We have used a Faraday cup (Model FC-1, Beam imaging solutions, USA) for monitoring the transmitted beam current through the target. It is mounted in front of the electron gun and perpendicular to the axes of TOF mass spectrometer and CMA. The output of the Faraday cup is connected through a BNC cable to a pico-ammeter.

The scattering chamber is maintained at a base pressure of 9×10^{-7} Torr and at 5×10^{-5} Torr with a gas load. This high vacuum (HV) reduces the adsorption of background gases into the chamber wall. The pressure of the scattering chamber is monitored by a dual pressure gauge (Pfeiffer Vacuum, Model: IKR 251). The HV in the scattering chamber is achieved by two TMPs [each 700l/s (Model: HiPACE-700, Pfeiffer Vacuum)], mounted one at the bottom of the chamber and the other near TOF mass spectrometer; they are backed by their respective rotary pumps (Model: DUO-10M, Pfeiffer Vacuum).

The electron gun used in the setup provides a mono-energetic electron beam with energy from 100 to 5 keV (Model: ELS-5000, PSP, UK). The gun is mounted on one of the CF-35 flanges and its axis is perpendicular to the axes of CMA and TOF mass spectrometers (see, Fig. 1). The size of the electron beam spot at the collision centre is monitored on a fluorescent screen which is about $1 \text{ mm} \times 1 \text{ mm}$ for a beam of 3.5 keV electrons. The sample gas is introduced in the chamber through a hypodermic needle which is fitted with copper tubing having inner diameter and length 1 and 30 mm respectively. The needle is mounted in such a way that its tip is about 2 mm below the electron beam axis at the collision centre.

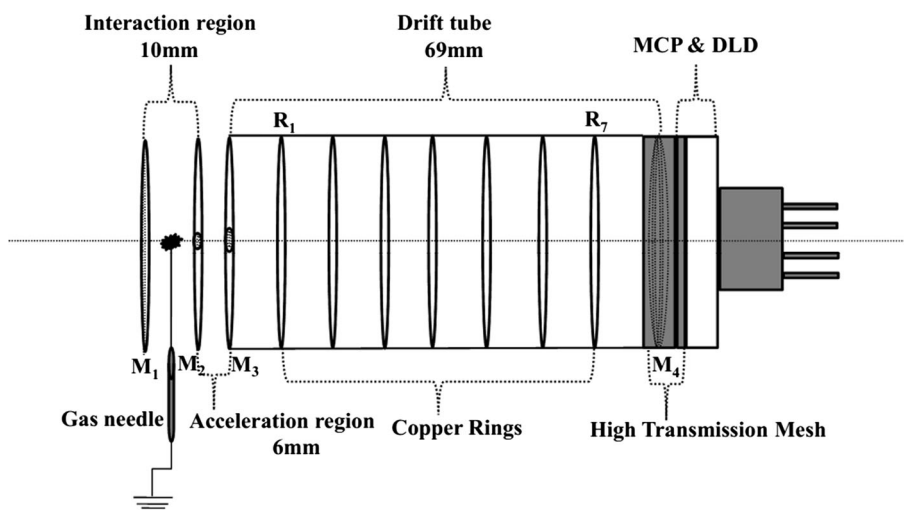
2.1. Spectrometers

2.1.1. TOF mass spectrometer

The ion mass spectrometer is constructed in a double-field configuration of Wiley–McLaren type TOF spectrometer [25]. Different charge states of target ions can be separated on the basis of their times of flight over a certain path, because the time of flight of an ion of mass m and charge state q is proportional to the square root of (m/q) . Ions are finally detected by a position sensitive dual rimless MCP of 40 mm diameter coupled with a delay line anode detector (DLD) assembly [26]. It has an interaction zone enclosed between two extraction electrodes M_1 and M_2 (both covered with a 65% transmission SS-mesh in order to maintain uniformity of the electric field) placed toward CMA and TOF entrance sides respectively; the separation between these electrodes is 10 mm. The acceleration region is enclosed between ion extraction electrode M_2 and the drift tube entrance electrode M_3 . The drift tube contains electrically connected 7 rings (R_1 – R_7) to provide a constant potential gradient in the tube. All rings and electrodes (M_1 – M_4) have outer and inner diameters of 76 and 56 mm and thickness of 0.24 mm respectively. They are made of copper and 304 SS respectively. The length of extraction, acceleration and drift regions is chosen to be 5, 6 and 69 mm, respectively as shown in Fig. 2.

A 50% transmission efficiency mesh (M_4) just before the MCP is used to avoid the penetration of high electric field of MCP into the drift tube. The electric field applied on the extraction electrodes is chosen according to the aim of the experiment. Assuming the point source for secondary ion formation and applying the extraction field 40 V/cm in the interaction region, all singly charged argon ions with kinetic energy of up to 25 eV are found to reach the MCP in focusing condition. This is true for any ionic species as the divergence of the ions in the considered extraction field

Fig. 2 Schematic of a double-field time of flight mass spectrometer



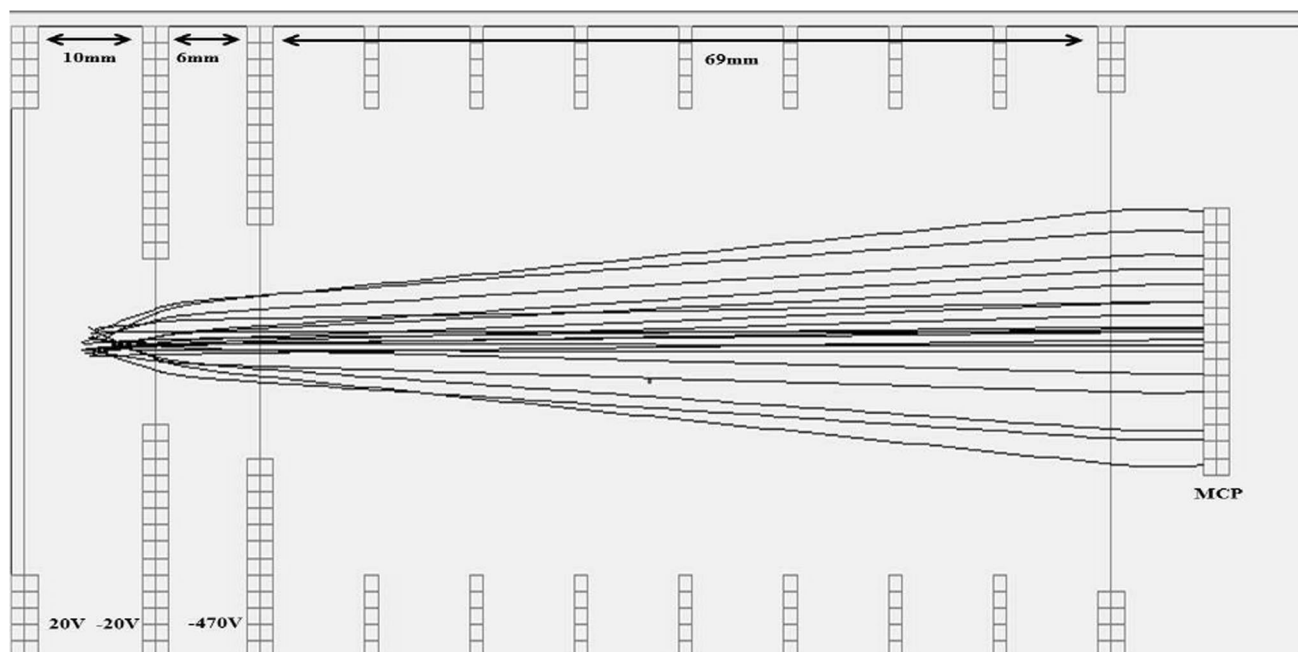


Fig. 3 Ion trajectories of Ar^+ ions with 25 eV initial kinetic energy

is approximately proportional to the square root of the kinetic energy of the ion to its charge. For a range of extraction voltages used here, the ion trajectory simulations were performed using the SIMION 8.0 code [27]. As an example, the simulated trajectories for singly charged argon ions with kinetic energy of up to 25 eV are shown in Fig. 3. Therefore, the extraction fields between 40 and 60 V/cm are found to be moderately good to achieve reasonable momentum resolution and ion collection efficiency for the considered target atoms. The typical values of mass resolution, time dispersion of the ions and the capability of measuring the kinetic energy distribution of the ions are determined to be 4.7, 2.5% and 25 eV respectively.

2.1.2. Electron spectrometer

The ejected electrons of characteristic and non-characteristic energies are produced in the interaction region due to ionization of the target atoms by impact of electrons. The energy analysis of the ejected electrons is done by a single-pass cylindrical mirror analyser (CMA) which is placed coaxially opposite to the TOF mass spectrometer. In order to detect the genuine electrons ejected from the collision volume efficiently, the CMA (Model PHI 15-110A) with high luminosity and high energy resolution is employed in the present setup. The CMA has an angular acceptance of $\pm 7^\circ$ around the mean acceptance angle of about 43° . Its geometrical solid angle $\Delta\Omega$ is determined to be about 8.4% of 4π . Three apertures with an opening of 2.0, 0.9 and

0.4 mm are available in the CMA in front of the mouth of channel electron multiplier (CEM) detector. As per specifications of the manufacturer, the energy resolutions corresponding to these apertures are 1.2, 0.6 and 0.3% respectively. The working distance between the target and the CMA is 6–7 mm. Since the type of experiments we are dealing with, the reaction zone is always immersed in an electric field for extracting the ions, we have constraint to choose an electric field of such value which neither appreciably affects the incident electron beam direction in the reaction zone nor the shape and resolution of the energy spectrum of the detected electrons in the CMA. The CMA was calibrated by the argon peak occurring at energy of 203.5 eV due to $L_{23}\text{--}M_{23} M_{23} (^1D_2)$ Auger transition in collisions of 3.5 keV electrons with argon gaseous target [28]. The measured electron energy resolution $\Delta E/E$ of the CMA is determined to be 2.5% with 2.0 mm exit aperture of the CMA. This resolution is mainly controlled by the size of interaction volume which in the present case is estimated to be $1\text{ mm} \times 1\text{ mm} \times 1\text{ mm}$. With such an intrinsic energy resolution, the presence of a dc field (40 V/cm) in the interaction region neither worsens the energy resolution of the electron energy spectra measured by the CMA nor deflects the electron beam direction in the interaction region. From an auxiliary experiment, it has been verified that with and without the applied static field, the electron spectrum remains identical in relation to its shape, resolution and energy distributions. The diameter of the electron beam at the scattering centre is about 1.0 mm.

This corresponds to an indetermination of 4.0 eV in the Auger electron energy when the above static electric field (40 V/cm) is applied to the interaction region.

2.2. Data acquisition system and associated electronics

The data acquisition system employed in our setup is shown in Fig. 4. It is based on the time of flight (t) and the position (x, y) measurement of each ion produced in the collision. In order to measure (x, y, t), all the charged particles are detected in coincidence with the ejected electrons produced in each collision event.

In the present experiment, six signals are produced; an ion signal from the MCP, an electron signal from the CMA and four signals from the delay line anode. Signals of the MCP and DLD40 are decoupled from dc voltages on the wire using the RC decoupling circuits and special transformer circuits (FT12-TP). The five ion signals are fed to a differential amplifier (ATR19) for amplification; the amplified signals are constant fraction discriminated from noise by adjusting the threshold. The electron signal

from the CMA is also processed through an amplifier (ORTEC-FTA820) and CFD (Tennelec-TC454) through delay generator (TC410A). The amplified ion signal from the MCP triggers as a common start pulse for five channels of a time to digital converter (TDC8HP). The first four channels of the TDC8HP are stopped by delay line signals, resulting in information about the ion position. The last channel of the TDC8HP is stopped by the delayed electron signal from the CMA, resulting in the time of flight of the ion. The TDC8HP has a time resolution of 25 ps, maximum range of 419 μ s and typical dead time between multiple hits on one channel <5 ns. The digitized data obtained from the TDC are stored in a list-mode for each event on a hard disk. The CoboldPC [26] software is used for data acquisition and for off-line analysis. To study the coincidence events between the formed ions and detected electrons, the signal from the ion detector MCP is taken as **START** while the signal from CMA is taken as **STOP**. Both the signals are then fed to TDC8HP for recording the coincidence time of flight (TOF) mass spectra.

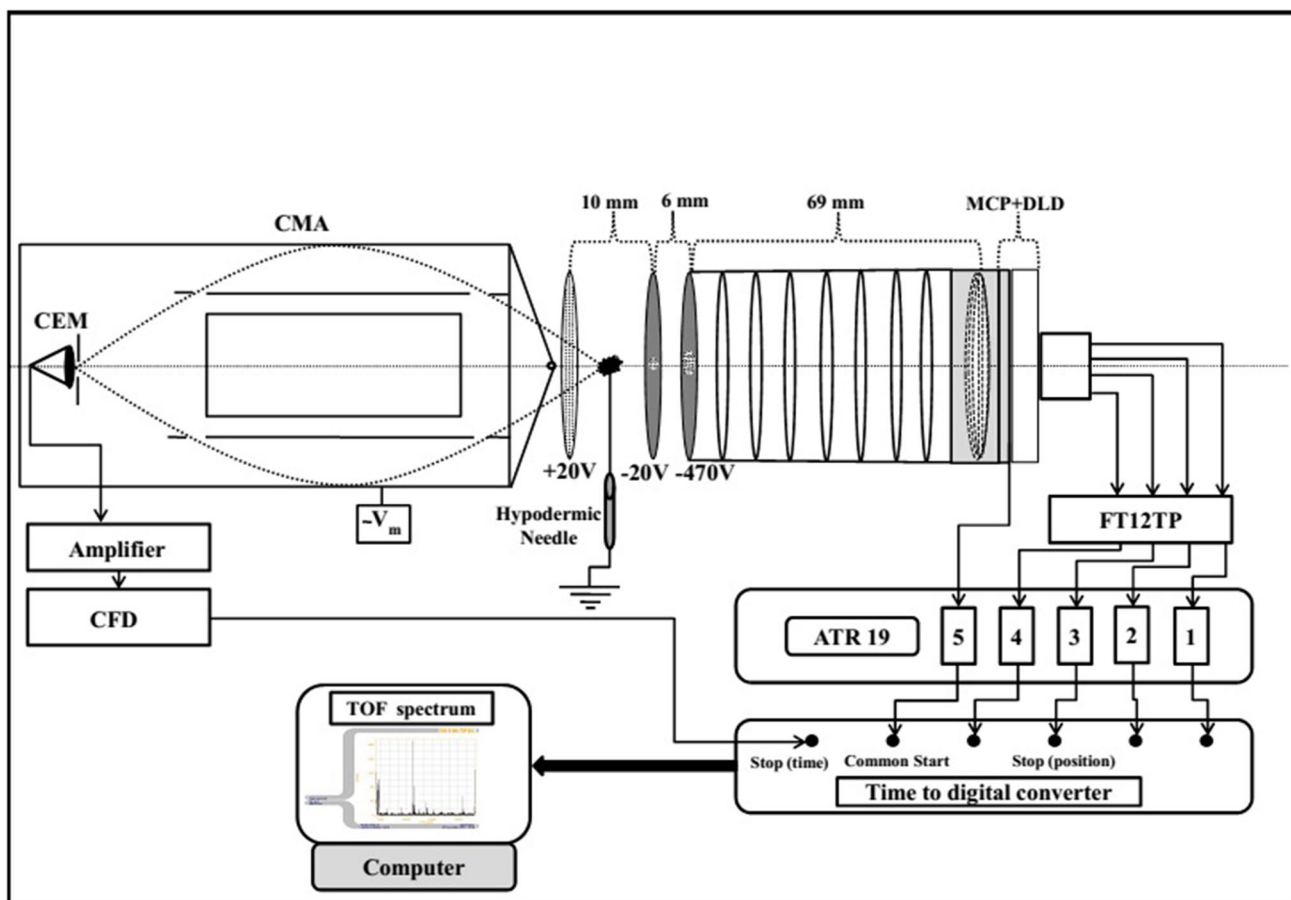


Fig. 4 Electron-ion coincidence measuring system using a double-field TOF mass spectrometer and a single-pass cylindrical mirror analyser (CMA)

3. Results and discussion

3.1. Electron energy spectrum of argon

Energy spectrum of electrons of argon produced in the energy range of 120–250 eV due to impact of 3.5 keV electrons with argon atoms in presence of a dc field of 40 V/cm applied in the interaction region is shown in Fig. 5. A group of multiple peaks (group I) with highest intensity around 203.5 eV arises from the $L_{23}\text{--}M_{23} M_{23} (^1D_2)$ Auger transitions. The structure around 192 eV forms the group II due to $L_3\text{--}M_1 M_{23} (^1P_1, ^3P_{2,1,0})$ transitions and the structure around 178 eV (group III) corresponds to $L_{23}\text{--}M_1 M_1 (^1S_0)$ weak transitions. Following Werme et al. [28], group I of the Auger transition lines is found to consist of four major diagram lines, namely, $L_{23}\text{--}M_{23} M_{23} (^1D_2)$ at 203.5 eV, $L_3\text{--}M_{23} M_{23} (^1S_0)$ at 201.1 eV, $L_3\text{--}M_{23} M_{23} (^3P_{2,1,0})$ at 205.2 eV and $L_2\text{--}M_{23} M_{23} (^1D_2)$ at 205.6 eV. The broad peak arising due to these transitions is not resolved in the spectrum on account of insufficient resolution of the present electron spectrometer. The group II comprises of two unresolved Auger diagram lines, namely, $L_3\text{--}M_1 M_2 (^3P_{2,1,0})$ at 191.0 eV and $L_3\text{--}M_1 M_{23} (^1P_1)$ at 189.0 eV. The group III contains unresolved weak lines of Auger transitions, namely, $L_3\text{--}M_1 M_1 (^1S_0)$ at 177.9 eV, $L_3\text{--}M_1 M_{23} (^1P_1)$ at 179.0 eV and $L_3\text{--}M_1 M_{23} (^3P_{0,1,2})$ at 179.5 eV.

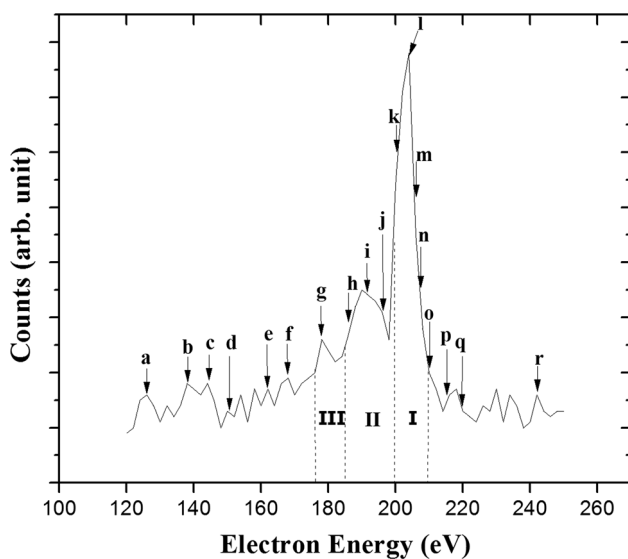


Fig. 5 Energy spectrum of electrons of argon produced in energy range of 120–250 eV under the impact of 3.5 keV electrons with argon atoms in presence of 40 V/cm electric field applied in the interaction region. Arrows below alphabetical symbols denote the energy positions where the coincidence measurements were performed. The dotted vertical lines separate the three main groups I, II and III of the Auger transition lines of considerable intensities (see, text)

3.2. Ion coincidence spectra with energy selected electrons

The formation of doubly charged argon ions following decay of 2p-hole in argon by impact of 3.5 keV electrons indicated by the $L_{23}\text{--}M_{23} M_{23} (^1D_2)$ Auger transition at 203.5 eV provides the characteristic of the target atom under consideration. The argon ion coincidence TOF spectra have been recorded with energy selected Auger electrons at around 203 eV with an effective energy resolution of about 5 eV of the CMA. The observed ion coincidence TOF spectrum is displayed in Fig. 6. This ion coincidence spectrum shows clearly the formation of Ar^{2+} peak indicating an occurrence of a normal Auger transition with no ions of other charge states.

Furthermore, the argon ion coincidence TOF spectra have been recorded under impact of 3.5 keV electrons with argon atoms for a whole series of 18-pass energies of electron spectrometer at an effective energy resolution of about 5 eV. The observed ion coincidence TOF spectra are displayed in Fig. 7.

The selected electron energies are indicated on the right hand side of each panel in order of the kinetic energy of the detected electrons. These spectra contain several charge states of argon ions which indicate that the charge number of argon ions increases with the decrease in electron energy. This also means that the charge number of ions increases with increase in final state energy. The results obtained in the present work indicate that not only the normal Auger and auto-ionization processes but also several other processes (CK, DDA, resonant Auger transitions, and shake processes) followed by Auger cascades with multiple spectator vacancies are responsible to produce multiply charged argon ions formed in coincidence with different energy selected electrons due to decay of L-shell hole states of argon atom. As shown in Fig. 5, the most

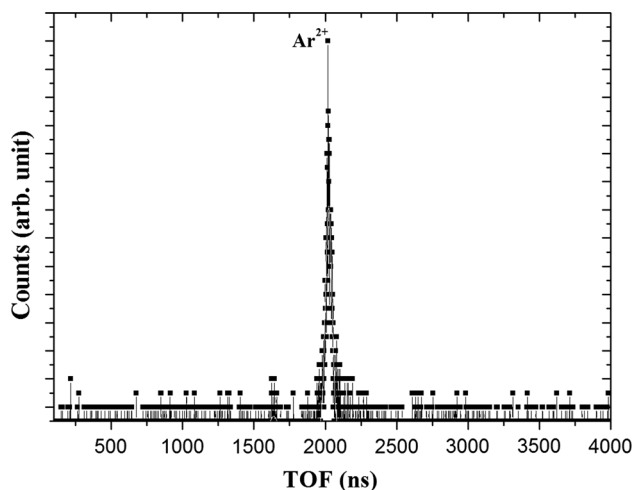


Fig. 6 A raw data showing a TOF Ar^{2+} ion coincidence peak measured with Auger electrons of energy 203 eV

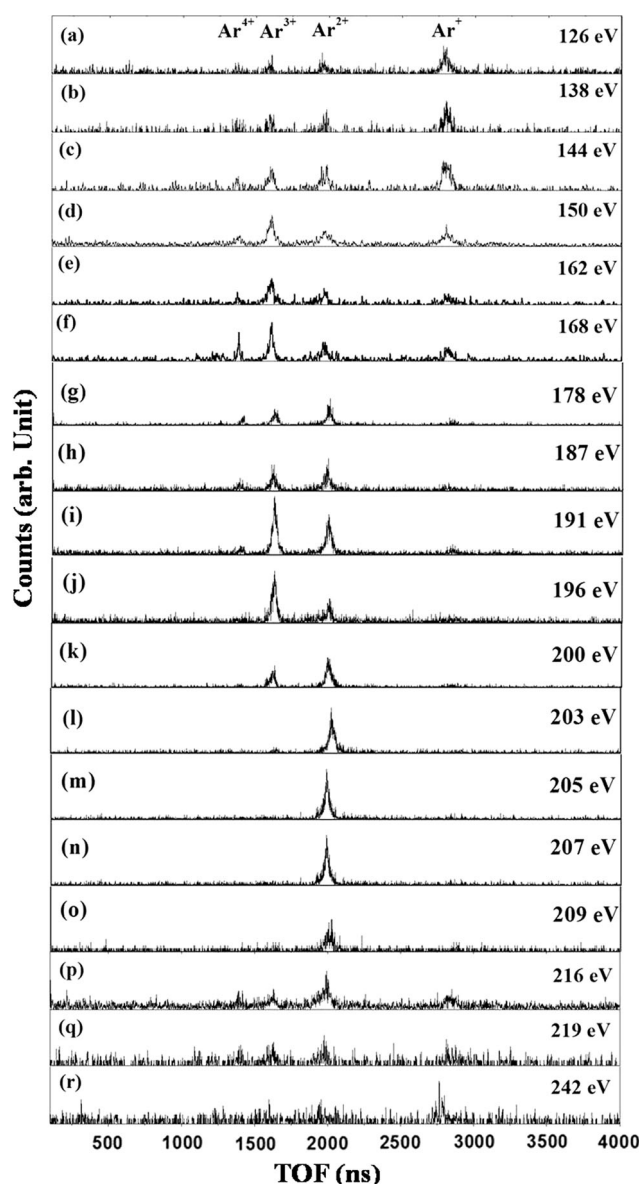


Fig. 7 TOF spectra of multiply charged argon ions observed in coincidence with 18-energy selected electrons shown in panels (a)–(r). The energy values listed on the right hand side denote those of electrons selected with the electron energy analyser. The data collection time of each spectrum ranged from 25 to 30 h

probable decay process in the initial step is the Auger electron emission of $L_{23}\text{--}M_{23} M_{23} ({}^1S_0, {}^1D_2, {}^3P_{2,1,0})$ in the energy range 200–210 eV. The second most probable one is that of the $L_{23}\text{--}M_1 M_{23} ({}^3P_{2,1,0}, {}^1P_1)$ in the energy range of 185–200 eV. The third probable decay process corresponds to the Auger satellite transitions in the electron emission range of 170–185 eV. Thus, it can be concluded that a significantly high fraction of the finally formed multiply charged argon ions are produced through the pathway into the highest state between all energetically possible states from the Auger final states.

3.3. Relative correlation probability

We have also studied the relative correlation probabilities of Ar^{n+} ions ($n = 1\text{--}4$) as a function of energy of ejected electrons. In this study, we have determined the probability $P(nl^{-1}, n+)$ for the decay of an inner-shell hole state nl^{-1} (initial state) into different ionic charge states $n+$ (final state). Because of the fact that electron impact ionization processes give rise to a continuous distributions in the electron spectra, for example, direct double electron ionization or direct double Auger decay, the correlation probabilities $p(\epsilon, n+)$ measured at a kinetic energy ϵ of a specific ejected electron line, in general, deviate from the decay probabilities $P(nl^{-1} \rightarrow n+)$ of the corresponding core hole state nl^{-1} . Thus, $p(\epsilon, n+)$ provides information on the decomposition of electron spectrum that correlates the electron impact ionization processes ending up in a certain final ionic charge state. In order to obtain the correlation probabilities $p(\epsilon, n+)$ from the ion-coincidence TOF spectra for each coincidence measurements at kinetic energy ϵ , the sum $\sum_{n=1} p(\epsilon, n+)$ is normalised to 1. As a result, we obtain the values of $p(\epsilon, n+)$ for the given final charge state $n+$ at each electron energy ϵ . Such results have been obtained and displayed in Fig. 8 for argon ions with charge states 1+ to 4+.

The above results on the correlation probability for production of argon ions with charge states 1+ to 4+ show clearly that the maximum probability of 100% is found for production of Ar^{2+} ions correlated to ejected Auger electrons with energy in the range of 203–209 eV; in this region, the most intense diagram lines of Auger transitions are found to occur. The satellite Auger lines are shown to arise in the electron energy range of 178–200 eV. The argon ions of multiply charged states are found to arise in low and high electron energy ranges due to other decay processes, for example, auto-ionization, direct double Auger transitions, Coster–Kronig followed by Auger cascades, post collision interaction and shake-up and shake-off processes.

4. Conclusions

We have developed an instrument for studying the dynamics of core-hole decay processes in atoms and molecules excited by energetic charged particles and photons. The apparatus consists of a double-field TOF mass spectrometer coupled with a time- and position sensitive dual MCP detector, a single-pass CMA and a continuous source of mono-energetic beam of electrons. The instrument is capable of yielding quantitative information about the fragmentation dynamics of multiply charged molecules specifying the involved precursor molecular ionic state and

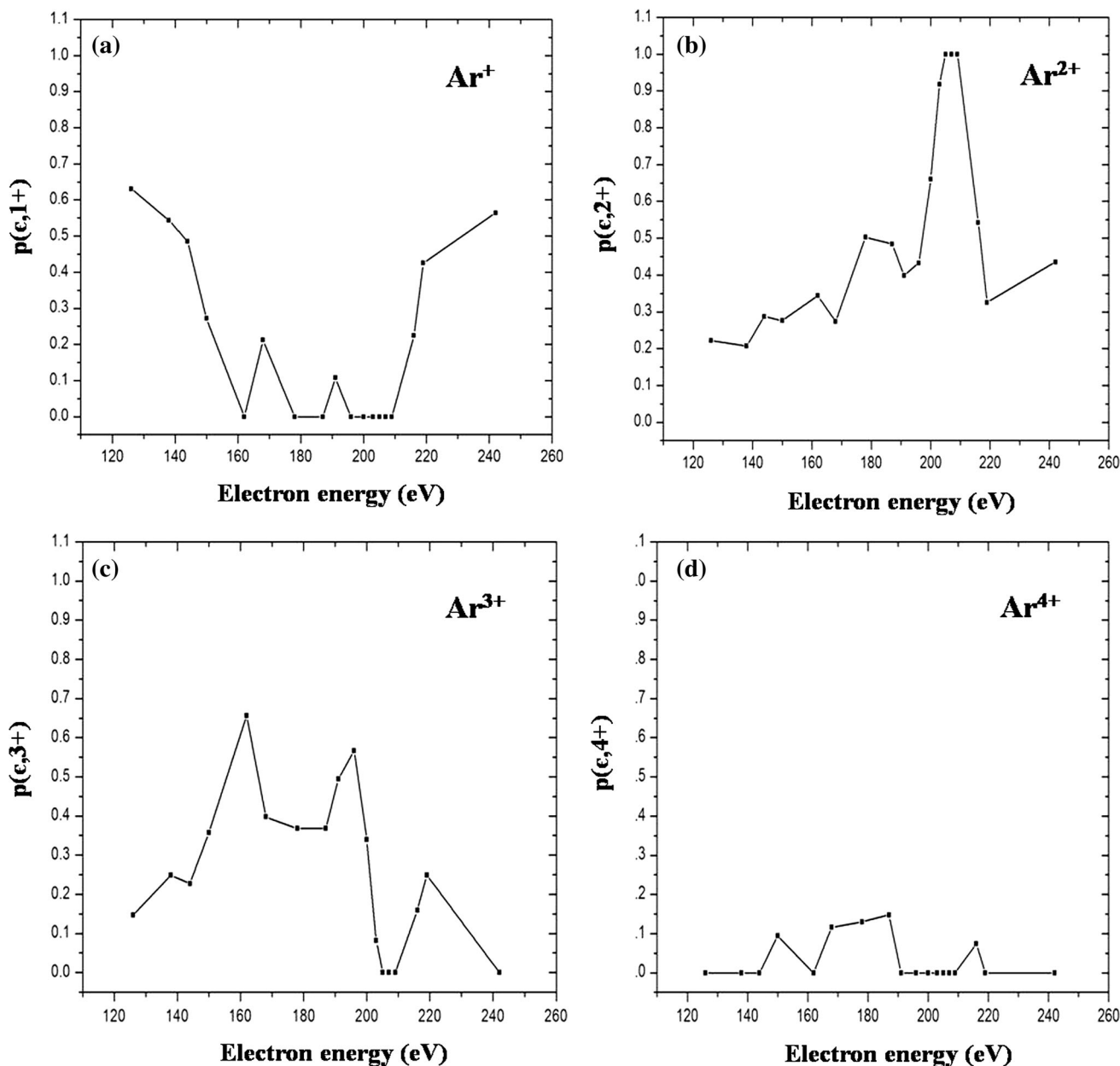


Fig. 8 Relative correlation probability $p(\epsilon, n+)$ for production of argon ions with final charge states 1+ to 4+ as a function of electron energy ϵ . (a) Ar^{1+} , (b) Ar^{2+} , (c) Ar^{3+} and (d) Ar^{4+}

the relative correlation probabilities of producing different charge states of an atom by using the energy selected electron-ion coincidence technique. Such technique has been used here to demonstrate the capability of the apparatus by obtaining some typical results as an example for L-shell excitation of argon atoms by impact of 3.5 keV electrons. The relative correlation probabilities for production of argon ions of final charge states 1+ to 4+ observed in coincidence with 18-energy selected electrons show that the maximum probability of 100% corresponds to Ar^{2+} ions produced in the electron energy range of

203–2019 eV via normal Auger transitions. The satellite Auger lines are shown to arise in the electron energy range of 178–200 eV. The argon ions of multiply charged state are found to arise in low and high electron energy ranges due to other decay processes, for example, auto-ionization, direct double Auger transitions, Coster–Kronig followed by Auger cascades, post collision interaction and shake-up and shake-off processes. The same technique is equally applicable for studies of multiply charged molecular ions as well. Such scientific studies on small organic molecules are planned for the next investigations.

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