



This open access document is posted as a preprint in the Beilstein Archives at <https://doi.org/10.3762/bxiv.2024.67.v1> and is considered to be an early communication for feedback before peer review. Before citing this document, please check if a final, peer-reviewed version has been published.

This document is not formatted, has not undergone copyediting or typesetting, and may contain errors, unsubstantiated scientific claims or preliminary data.

Preprint Title Performance optimization of microwave-coupled plasma-based ultra-low energy ECR ion source for silicon nano-structuring and application

Authors Joy Mukherjee, Safiul A. Mullick, Tanmoy Basu and Tapobrata Som

Publication Date 22 Nov. 2024

Article Type Full Research Paper

ORCID® IDs Joy Mukherjee - <https://orcid.org/0000-0002-5387-2617>; Safiul A. Mullick - <https://orcid.org/0000-0002-0985-8914>



License and Terms: This document is copyright 2024 the Author(s); licensee Beilstein-Institut.

This is an open access work under the terms of the Creative Commons Attribution License (<https://creativecommons.org/licenses/by/4.0>). Please note that the reuse, redistribution and reproduction in particular requires that the author(s) and source are credited and that individual graphics may be subject to special legal provisions.

The license is subject to the Beilstein Archives terms and conditions: <https://www.beilstein-archives.org/xiv/terms>.

The definitive version of this work can be found at <https://doi.org/10.3762/bxiv.2024.67.v1>

1 **Performance optimization of microwave-coupled plasma-based ultra-low**
2 **energy ECR ion source for silicon nano-structuring and application**

3 Joy Mukherjee¹, Safiul Alam Mullick², Tanmoy Basu³, Tapobrata Som^{1,4*}

4 ¹SUNAG Laboratory, Institute of Physics, HBNI, Sachivalaya Marg, Bhubaneswar – 751 005,
5 India

6 ²Rabindra Mahavidyalaya, University of Burdwan, Hooghly, West Bengal, 712 401

7 ³Centre for Quantum Engineering, Research and Education, TCG Centers for Research and
8 Education in Science and Technology, Kolkata, West Bengal, 700 091, India

9 ⁴Homi Bhabha National Institute, Training School Complex, Anushaktinagar, Mumbai – 400
10 085, India

11

12 **Abstract:**

13 This literature presents a comprehensive optimization of key parameters crucial for generating
14 ion beams in a microwave-coupled plasma-based ultra-low energy Electron Cyclotron
15 Resonance (ECR) ion source, generally used for nano-structuring on solid surfaces. The
16 investigation focuses on developing, accelerating, and extracting Ar⁺ ions from a magnetron
17 (microwave) coupled plasma cup utilizing three-grid ion extraction composed of molybdenum.
18 The study systematically examines the dependence of ion beam current on critical parameters,
19 such as gas pressure, magnetron power, extraction voltage, and ion energies. Additionally, the
20 influence of extraction voltage on beam current is investigated for different ion energies. The
21 variation of beam current as a function of ion energy is explored under constant magnetron
22 current and extraction voltage at various conditions. The Gaussian nature of the beam profile
23 is scrutinized and elucidated within the context of grid extraction-based ion sources. Plasma
24 physics principles are employed to interpret the observed variations in ion current density

25 (beam current) with various parameters. The corresponding ion-induced nanopatterning on
26 silicon, using the optimized beam current, is explored in detail. Furthermore, the research
27 delves into the temporal evolution of the surface topography of silicon followed by off-normal
28 incidences (60° and 72.5°) is Ar-ion extracted at 450 eV Ar-ions. The changes in the optical
29 property, resulting from nano-patterned surfaces, investigated using UV-VIS spectroscopy, is
30 correlated the with dimension of nano patterning. This manuscript highlights the potential
31 applications arising from these findings, emphasizing the transformative impact of low energy
32 inert ion induced nano-patterning technologies.

33 Keywords: Ultra-low energy ECR-based ion source, Optimization of ion current, Surface
34 topography, TEM, UV-VIS spectroscopy

35 *Corresponding author email: tsomiop@gmail.com

36 **Introduction:**

37 The ion source serves as a fundamental component in numerous scientific and industrial
38 applications, playing a crucial role in generating charged particles (ions). Following ion
39 production, various systems harness these energetic ions for diverse purposes, spanning
40 material science, high-energy physics, medical applications, and agricultural science[1–5].
41 Presently, energetic ions find application in various surface treatments such as nano-patterning,
42 sputter etching, controlled defect formation, and more[6,7]. Particularly, ultra-low energy ion
43 beam proves exceptionally valuable for the precise modification of 2D layers [8], ion-induced
44 nano-patterning on semiconductor surfaces[9]. Over the past few decades, ion-induced nano-
45 patterning and nanoscale functionalizations have garnered significant interest, owing to their
46 broad applications in DNA origami [10], tuning wettability [11], electrical and magnetic
47 anisotropy [12,13], isolated dot formation [14], nanoscale plasmonic array[15], field emission
48 [16], etc. Thus, the ion sources generate enormous possibilities for material modifications both

49 physically and chemically. Further, diversity exists in energetic ion production mechanisms.
50 The fundamental process of producing ions is the collision of atoms with ions or electrons
51 which may be either elastic or inelastic. In elastic collisions, the internal energy of the colliding
52 particles does not change. Ionization, stripping, electron capture, and excitation of atoms due
53 to collisions are examples of inelastic collisions. The free electrons colliding with atoms also
54 produce ions. Electrons in the gas are heated by the inductively coupled method and then
55 acquire enough energy to generate plasma. Due to several drawbacks in such Townsend
56 discharge, these sources are not used now a days. In recent days, compact broad-beam ion
57 sources are widely used in scientific laboratories to generate ions. Depending upon the
58 mechanism of production of various ions using gaseous plasma, the ion sources can be
59 classified in three ways; direct current (dc) operated ion sources, radio frequency (microwave)
60 ion sources, and microwave ion sources. In the past few decades, DC ion sources were
61 commonly used in the above activities [17–19]. These DC ion sources consist of a hot cathode
62 or filament, which is not especially useful in cases of reactive gas discharge; hence, their
63 lifetime is limited [20,21]. Moreover, the beam current produced by those ion sources is not
64 suitable for modern-day applications. In material science as well as surface science
65 applications, the ion source should be mobile and adaptable to the vacuum system, having a
66 longer lifetime. Further, the ion source should produce a relatively high beam current (i.e.,
67 capable of forming a high density of plasma) with lower maintenance. To address this
68 challenge, Electron Cyclotron Resonance (ECR) based ion sources were developed [22,23].
69 ECR ion source is one of the most preferred ion sources for the easy production of ions for
70 different energies and charge states. Since the discharge is maintained in the quartz cup via a
71 strong electric field generated in the cavity, the ECR-based ion sources equipped with
72 microwave cavities neither contain any filament nor any type of electrode. The high plasma
73 density within a quartz cup is confined by solenoid magnets surrounding it, creating a multi-

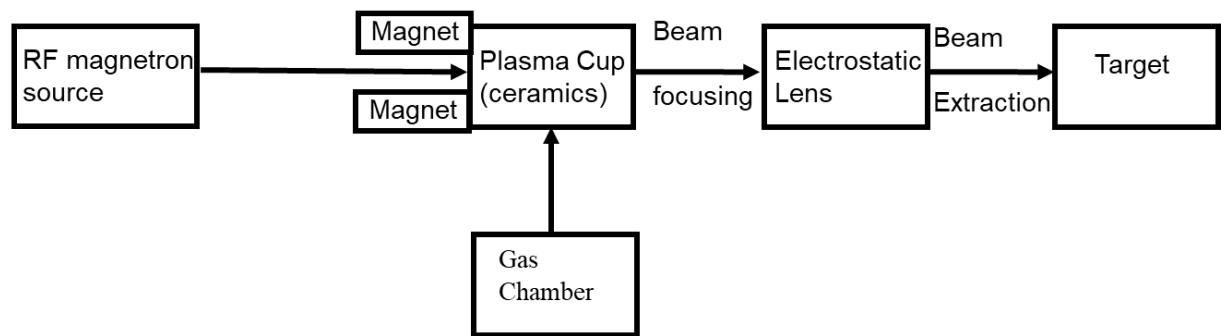
74 cusp magnetic field. However, careful attention is required for the microwave coupling to the
75 plasma cup to minimize microwave-reflected power. Mechanical adjustments to the resonator
76 length and waveguide are made to ensure minimal reflection of microwave power.
77 Additionally, maintaining the necessary magnetic field strength is crucial for sustaining the
78 plasma. The ion source's compact design is user-friendly and capable of producing a high beam
79 current density for a single or multi-grid extraction system. As a result, the extracted beam
80 current is influenced by magnetron power, plasma pressure, and extraction voltage.
81 Furthermore, the beam current varies with different ion energies.

82 This article focuses on optimizing the beam current generated by a cost-effective
83 microwave-based Electron Cyclotron Resonance (ECR) ion source and subsequent
84 development of nanoscale patterns on the surface of silicon. The relationship between the beam
85 current and various parameters is extensively examined and elucidated. Experimental
86 parameters, spanning from plasma generation to ion beam extraction, are systematically
87 optimized for the study of low-energy Ar-ion-induced nanostructures on silicon. The
88 dependence of the extracted ion beam on plasma pressure, magnetron power, and extraction
89 grid voltage is documented for different ion energies. Additionally, the manuscript establishes
90 the relationship between ion beam current and the ion energy. Irradiation of p-type single
91 crystal Si (100) surface at off-normal angles (60° and 72.5°) by a 450 eV Ar-ion results in the
92 well-defined formation of nanoscale ripple patterns. The prominence of ripple structures
93 increases with prolonged irradiation time, while bombardment at 72.5° with the same ion beam
94 parameters leads to the coarsening of nanostructures. Cross-sectional transmission electron
95 microscopy (TEM) measurements confirm the formation of nanostructure as observed from
96 atomic force microscopy images (AFM). The thickness of the amorphous thin layer is well
97 agreement with Monte Carlo Simulation (SRIM)[24]. The article further investigates and
98 explains the optical response (by UV-VIS spectrophotometer) of the nano-patterned surfaces

99 with the dimensions of nano-patterning (i.e., wavelength and rms roughness). The potential
100 applications of such nano-patterned silicon surfaces are enlightened. This article underscores
101 the versatility of an optimized broad-beam ultra-low energy ion source, specifically in the
102 context of optimization of inert Ar-ion and subsequent ion-induced silicon nano-patterning.

103

104 **Description of the ion source:**

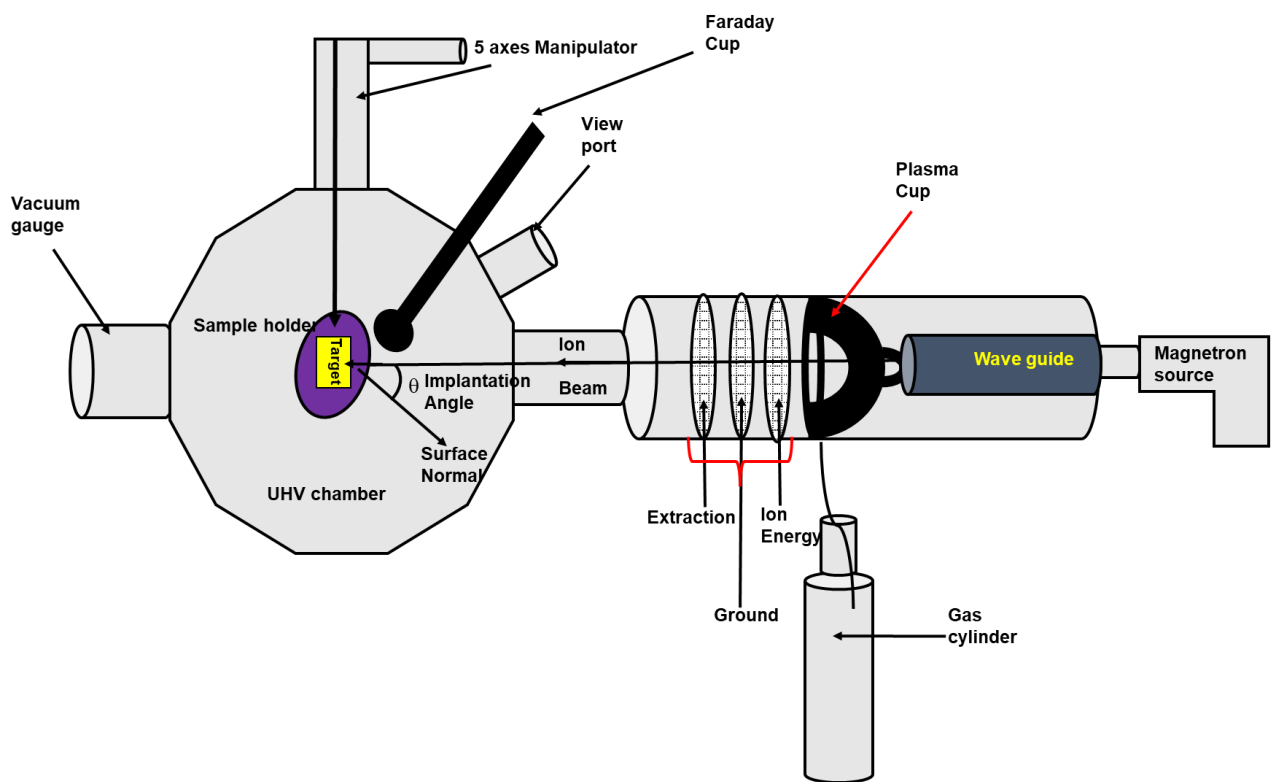


105

106 **Figure 1:** Block diagram consisting of the component of high vacuum plasma ion source
107 equipped with UHV chamber.

108 Figure 1 illustrates the block diagram of the magnetron-coupled ultra-low energy
109 Electron Cyclotron Resonance (ECR) ion source to provide a comprehensive visualization of
110 the entire setup. The schematic representation in Figure. 1 elucidates the process of extracting
111 an ultra-low energy ion beam. An microwave source (magnetron) is connected to the ceramic
112 plasma cup via a waveguide. The gas inlet system facilitates the filling of the plasma cup with
113 gas through a capillary tube. The intense electric field generated by the microwave source
114 (magnetron) induces gas breakdown (discharge), leading to the formation of a highly intense
115 plasma. The produced plasma is confined and sustained by a permanent magnet positioned near
116 the ceramic plasma cup (made of Al_2O_3). For the extraction and focusing of the beam, a gridded
117 electrostatic lens, commonly referred to as an Einzel lens, is employed. The shape and size of
118 the beam are contingent on the extraction voltage applied at the grid and the corresponding ion

119 energy. The directed beam impacts the silicon target kept in an ultra-high vacuum (UHV)
 120 within the target chamber. A faraday cup, connected to a multimeter, measures the beam
 121 current, and the corresponding ion fluence is expressed in terms of irradiation time.
 122 The sample holder, located in a UHV chamber, is connected to a 5-axes (x, y, z, θ, φ)
 123 manipulator system, offering movement and rotation in all possible directions. The sample is
 124 transferred to the ion source using a load-lock system. A later discussion provides a cross-
 125 sectional view of the system.



126
 127 **Figure 2:** Cross-sectional (schematic) view of microwave coupled ultra-low energy ion beam
 128 system.

129 The cross-sectional view of the high vacuum microwave based ECR ion source mentioned
 130 above is shown in Figure 2. The entire system consists of a magnetron-coupled ion source, a
 131 UHV target chamber with a cylindrical cavity and load lock, a 5-axes manipulator (PREVAC
 132 Technologies), and a gas inlet system. This type of magnetron-coupled ion source was first
 133 developed by Anton *et.al* [23]. The ion source is fitted in the cylindrical cavity of the UHV

134 target chamber. The inner diameter of the plasma cup is around 52 mm, where the plasma is
135 generated. The cup is surrounded by water-cooled magnets made up of neodymium-iron-boron,
136 which produces a multi-cusp field to confine the plasma. The 2.45 GHz magnetron-based
137 microwave source is attached to the backside of the ion source, as shown in Figure 2. The
138 dimension of the cylindrical resonator (waveguide) is chosen in such a way that it can produce
139 maximum beam current. To generate plasma in the ceramic cup (known as plasma cup), a gas
140 is inserted into it through a capillary tube attached to the gas chamber. A pressure of 10^{-4} mbar
141 is maintained for sustaining plasma by adjusting a needle valve attached to the gas chamber.
142 The entire length of the ion source is around 130 cm. The extraction of ion beams is
143 accomplished by a three-grid ion optics system, as seen in Figure 2. Wide-ranging extraction
144 voltage is applied to the grid to enable the extraction of an intense beam with different
145 diameters. The circular perfection of the beam shape is evident from observations on the front
146 plate attached to the ultra-high vacuum (UHV) chamber. In this configuration, the beam
147 current, specific to a given ion energy, can be finely adjusted based on magnetron power,
148 working pressure, and extraction voltage. Consequently, optimizing the dependence of beam
149 current on these parameters is a worthwhile pursuit, driven by the need to comprehend the
150 underlying scientific principles. Additionally, the ion current (beam current) is influenced by
151 the extracted ion energy and the position of the target. Hence, a comprehensive investigation
152 into the intricate relationship between ion current and the mentioned parameters emerges as a
153 compelling topic in the current scientific context.

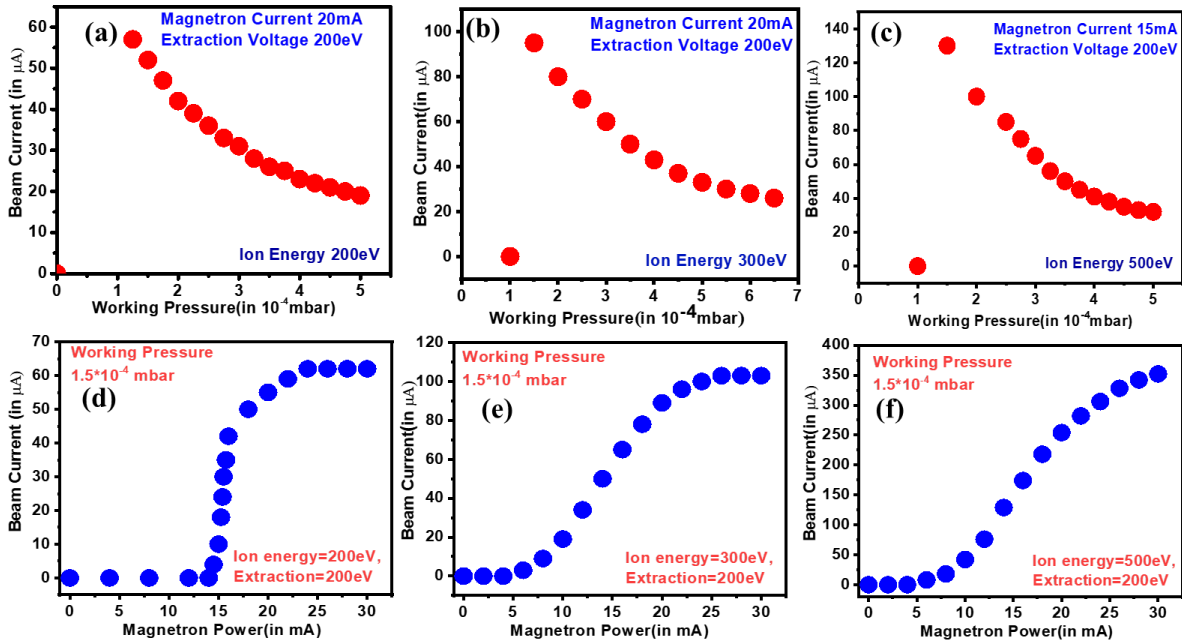
154

155

156

157

158 **Optimization of ion current on various plasma parameters:**



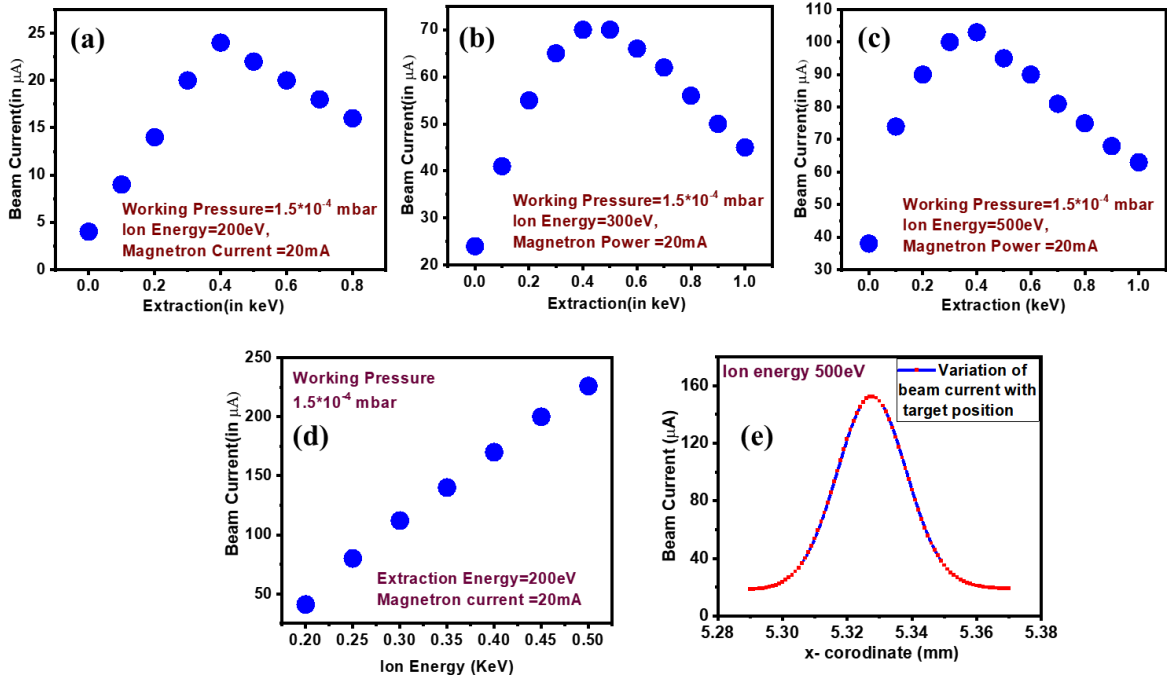
159

160 **Figure 3:** Variation of beam current with (a)–(c) plasma pressure, (d)–(f) microwave
 161 (magnetron) power for different ion energy.

162 The variation of beam current with plasma pressure and magnetron (microwave) power for
 163 different ion energies are investigated and presented in Figure 3. Figure 3(a)-(c) demonstrates
 164 that the beam current decays almost exponentially with the increase in plasma pressure. The
 165 ion current is maximum at plasma pressure 1.5×10^{-4} mbar irrespective of ion energies. This
 166 indicates that the minimum base pressure required for generating plasma is 1.5×10^{-4} mbar. It
 167 is also evident that for the same plasma pressure, the beam current is maximum for the highest
 168 ion energy. The gas pressure inside the plasma cup is directly proportional to the number of
 169 gas molecules present. At low plasma pressure, the mean free path of gas molecules is larger
 170 due to the lesser density of gas molecules, which allows the produced ions to traverse a longer
 171 distance without collision. This increases the ionization efficiency, and hence, with fewer no
 172 of collisions, the probability for recombination of the ions is very low. Consequently, a large
 173 number of ions are extracted, intensifying the beam current. The entire phenomenon can be

174 summarised through the equation, $\lambda = (\sigma \cdot n)^{-1}$, where λ is the mean free path of the ions, σ is the
175 cross section for recombination, and n is the density of the ions inside the plasma [25–27]. The
176 mean free path of the ions, determined by the recombination cross section and density of
177 plasma, plays a key role in quantifying the ion current.

178 Further, the conversion of the gas to plasma is governed by a magnetron (microwave)
179 source and therefore, the ion current or plasma density must depend on magnetron power. To
180 understand that, the variation of ion current with microwave power is recorded at different
181 plasma pressures and ion energies, as presented in Figure 3 (d)- (f). In general, the plasma
182 density (n) depends on the microwave frequency (ω) as $n = \frac{E_{RF}\omega^2}{\epsilon}$ where E_{RF} is the microwave
183 power (energy) and ϵ is the minimum energy required for ion-electron pair generation [25]. The
184 magnitude of ϵ is different for different gases. A non-zero magnitude of ϵ signifies the
185 minimum energy needed to generate plasma, commonly referred to as ionization energy, which
186 is supplied by magnetron power. It is evident from the above Figure (3(d)-(e)) that up to the
187 microwave power equal to a critical value, the formation of plasma is forbidden, resulting in a
188 zero-beam current. With the increase of magnetron power beyond the magnitude ϵ , the beam
189 current increases almost linearly with the input magnetron power, since the plasma density (n)
190 is directly proportional to the microwave power (E_{RF}). The beam current reaches saturation at
191 a specific microwave power level, which varies based on the ion energy. At sufficiently high
192 microwave power, the plasma density (beam current) is high, and the rate of generation and
193 recombination of the ion-electron pair is equal, resulting in a saturation of beam current as
194 observed in Figure. 3(d)-(f). Further, the cut off power also depends on ion energy. At
195 sufficiently low ion energy, the microwave power required for generating plasma is high. With
196 higher ion energies microwave power required for ion-electron pair generation is also less
197 which is obvious from the above discussion.



198

199 **Figure 4:** Variation of (a)-(c) beam current with ion extraction voltage at different ion
 200 energy;(d) beam current with ion energy; (e) beam current with the target position.

201 The extraction of developed Ar-ion beam was governed by a combination of three-grid
 202 (concave) ion optics system [28–30]. The beam current and the beam profile depend on the
 203 potential applied at the grid and the target position. The change in beam current with the ion
 204 extraction voltage recorded for different ion energies is presented in Figure. 4 (a)-(c). Initially,
 205 the beam current increases linearly with the applied extraction voltage since more ions are
 206 extracted with a higher extraction voltage. Irrespective of ion energy, the beam current is a
 207 maximum extraction voltage of 200 eV. With further increases in extraction voltage, the beam
 208 current decreases rapidly. This is governed by two major phenomena, firstly, the space charge
 209 effect, i.e., a high extraction voltage, creates an electrostatic field that repels the subsequent
 210 ions, causing the beam to spread out; therefore, the current density reduces. Secondly, during
 211 the extraction of the ions, the application of a high extraction voltage leads to the collision of
 212 the ions with residual gas molecules, which also causes a significant decrease in beam current

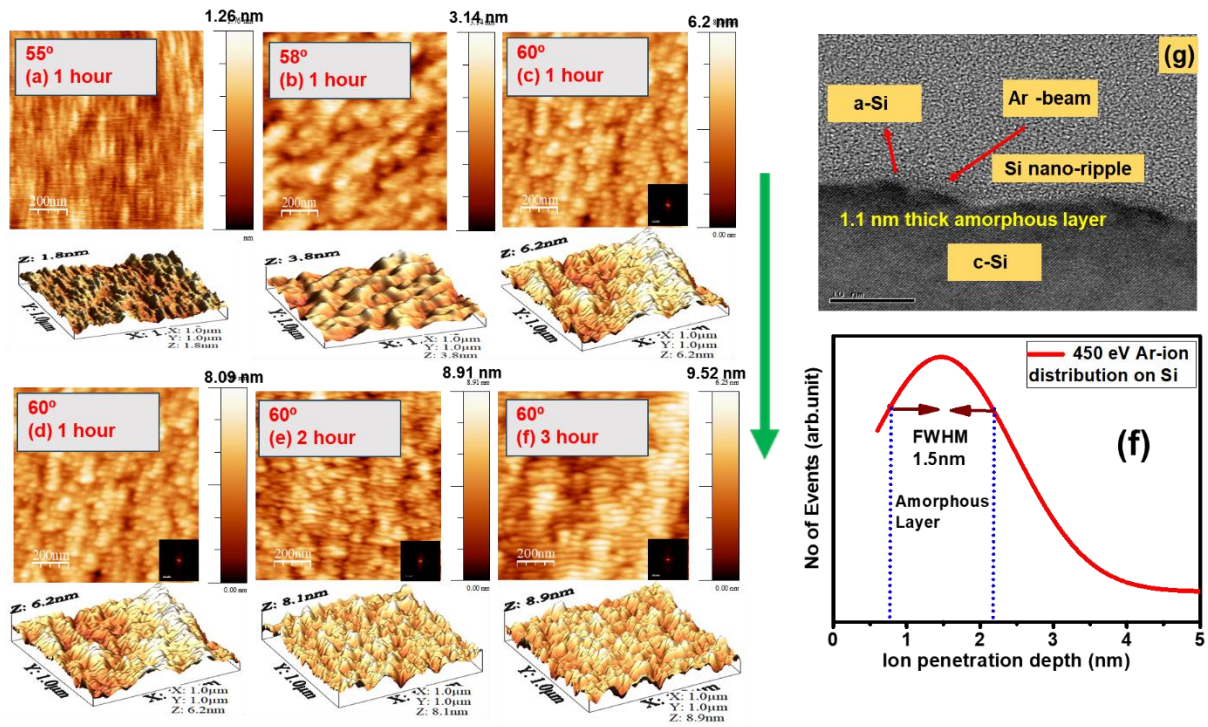
213 due to ion-electron recombination. Generally, the extraction voltage is kept fixed to maintain
214 the shape of the beam, essential for uniform irradiation of samples.

215 The dependence of beam current upon ion energy is also investigated in Figure 4(d). It is clear
216 from Figure 4(d) that the beam current increases almost linearly with the increase in ion energy
217 at a fixed extraction voltage and microwave power. When the applied extraction voltage is 200
218 eV and the ion energy is less than 200 eV, then the number of ions that can overcome the barrier
219 of 200 eV is less, causing a low beam current. With the increase in ion energy, the ions achieve
220 sufficient energy to overcome the barrier of 200 eV; hence, the beam current increases. On the
221 other hand, for a particular ion energy, lowering the extraction voltage also results to a lowering
222 of beam current as observed from the above Figure 4 (a)-(c). Therefore, to maintain a proper
223 beam shape and adequate beam current, the extraction voltage and ion energy are to be
224 precisely optimized. Further, the variation of beam current with the target position, known as
225 the beam profile, is also presented in Figure 4(e). The beam profile is Gaussian in nature for
226 concave grid (molybdenum) beam extraction optics. Such a beam profile precisely ensures the
227 target position is for maximum beam current.

228 **Nano-structuring on Si surface by 450 eV Ar-ion bombardment:**

229 Subsequently, after a detailed optimization of the ultra-low energy Ar-ion beam, the surface
230 topography of Si (investigated by AFM) after the off-normal bombardment of 450 eV Ar-
231 ion at different incidence angles and time is investigated. Figure 5 represents the surface
232 morphology of Si surface after 450 eV Ar-ion bombardment at different incidence angles.
233 The arrow on the right-hand side indicates the direction of the ion beam concerning the
234 surface normal. The irradiation of silicon surface with 450 eV Ar-ion at an angle of 55°
235 leads to no development of surface morphology, and presented in Figure 5(a). However, at
236 an ion incidence angle of 58°, the evolution of surface morphology starts, although no
237 prominent ripple structure is observed (Figure 5(b)). On the other hand, the bombardment

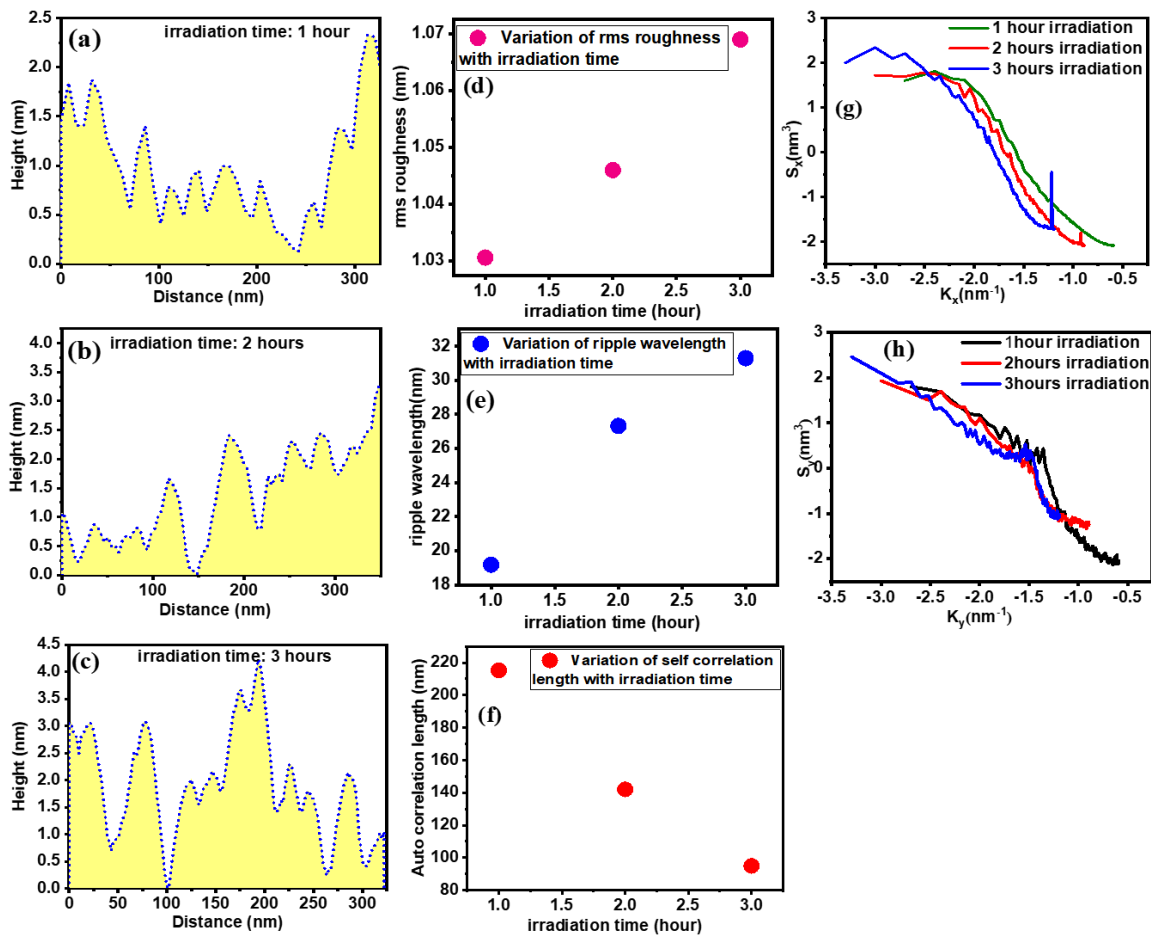
238 of 450 eV Ar-ion on the Si surface for an hour at an angle of 60° concerning surface normal
 239 leads to the formation of a well-defined nanoscale ripple pattern as observed in Figure 5(c).
 240 The growth of the ripple becomes more prominent with the increase in bombardment time.
 241 The amplitude of the ripple grows larger with longer bombardments of Ar-ions.



242
 243 **Figure 5:** AFM image (2D and 3D) of the evolution of surface morphology by 450 eV Ar-ion
 244 bombarded at different incidence angles and time. The arrow indicates the ion beam direction.

245 To visualize the growth of the ripple, the 3D AFM images are presented along with the
 246 2D images. It is clear from the height scale associated with the images, that the ripple height
 247 increases with bombardment time. The first Fourier transform (FFT) image of nano-patterned
 248 surface is inset at the right lowest corner of each image. In the present case, the fluence is
 249 replaced by irradiation time. The quality and the growth of the nano-structures are
 250 quantitatively discussed in Figure 6, where a detailed variation of ripple wavelength, rms
 251 roughness, and the power spectral density is discussed.

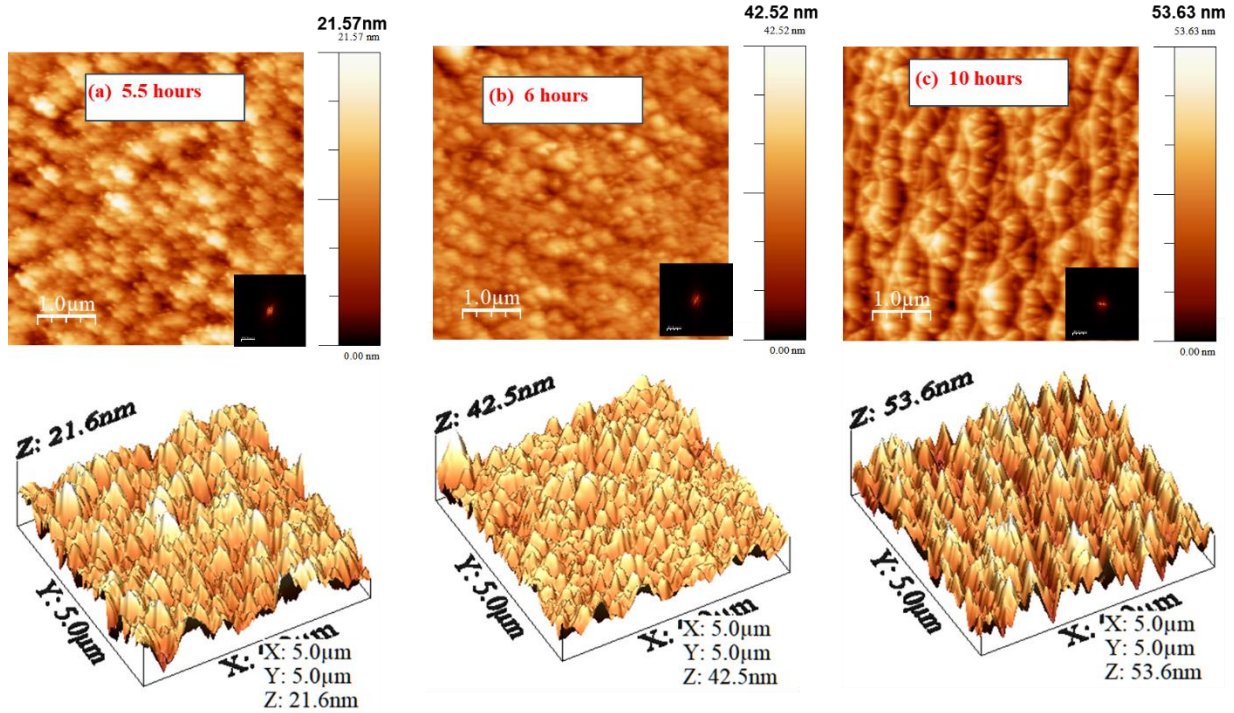
252 Figure 5(g) represents the cross-sectional transmission electron microscopy (TEM) image of
 253 450 eV Ar-ion bombarded Si surface at an angle of 60° with a time of 3 hours. The presence of
 254 Ar-ion-induced surface corrugation in terms of ripple-like nanostructure is evidenced in Figure.
 255 5(g). Although the amplitude of the ripples is not sufficiently large, the observed ripple
 256 wavelength of around 31 nm from the TEM image, is consistent with that of AFM data
 257 (presented in Figure 6(e)). However, in addition to the ripple-like nanostructure, an ultra-thin
 258 amorphous layer formation occurs due to Ar-ion bombardment. The thickness of the
 259 amorphous layer is around 1.5 nm, which is consistent with the penetration depth of the Ar-
 260 ions (1.2 nm), estimated by Monte Carlo Simulation (Fig 5(f)) [24]. Therefore, the
 261 topographical image is consistent with the cross-sectional image, indicating a clear signature
 262 of ripple-like nano-structure formation.



263

264 **Figure 6:** Variation of (a) – (c) surface height modulation of nano-patterned silicon surface;
265 (d) ripple wavelength and (e) rms roughness with irradiation time. Variation of power spectral
266 density of nanopatterned silicon surface on (g) parallel and (h) perpendicular direction.

267 Figure 6 (a)-(c) indicate the variation of the surface profile of the nano-patterned AFM
268 images shown in Figure 5. The height profile is the direct evidence of variation of ripple
269 amplitude with irradiation time. The increase in ripple height with irradiation time is shown in
270 Figure 6 (a)-(c). Further, the fluctuation in ripple height or amplitude generally termed rms
271 roughness, is also investigated in Figure 6(d). It is clear from Figure 6(d), that rms roughness
272 increases linearly with the irradiation time (fluence). Further, the ordering of the nanostructure
273 with the bombardment time, examined in terms of ripple wavelength, is presented in Figure
274 6(e). From Figure 6(e) initially, the ripple wavelength increases as the bombardment time
275 increases from one hour to two hours. With a further increment in irradiation time, the change
276 of ripple wavelength is negligible, i.e., a saturation of ripple wavelength is observed. The
277 degree of similarity between two spatial morphologies is generally quantified by auto-
278 correlation length or self-correlation length, as presented in Figure 6(f). It is clear from Figure
279 6(f), that the auto-correlation length decreases with bombardment time. This indicates a more
280 ordered ripple structure is found to develop with higher irradiation time. To understand the
281 growth of the ripple structure, the power spectral density factor along the parallel and
282 perpendicular direction of the developed ripple is presented in Figure 6 (g)-(h). The prominent
283 peak present in Figure 6(g) indicates, the development of ripple structure along the x direction
284 (parallel) with a particular wavevector (k_x). Besides, the absence of a ripple wavevector in
285 perpendicular mode is evidenced in Figure 6(h). Therefore, 450 eV Ar-ion bombardment on
286 Si, leads to well-defined parallel mode ripple formation at off-normal incidence.



287

288 **Figures 7:** AFM images (2D and 3D) of the evolution of nano-structure on Si surface with
 289 time under 450 eV Ar-ion bombardment at an angle of 72.5°.

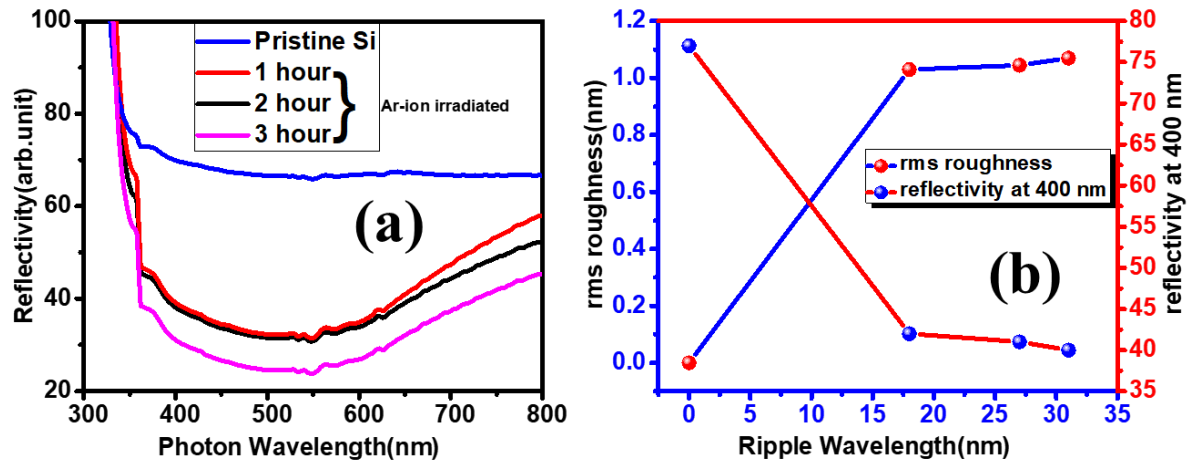
290 Figure 7 illustrates the surface topography after 450 eV Ar-ion bombarded the silicon surface
 291 at an angle of 72.5° with different bombardment time. Corresponding 3D AFM images are also
 292 presented along with 2D surface topography. Generally, the transformation of well-defined
 293 nano ripples into nano-facets occurs at this near-grazing angular region[31]. In the present case,
 294 although no prominent nano-facet formation is observed, a clear signature of transformation of
 295 ripple structure towards nano-facets is seen. At sufficiently large bombardment time (10 hours),
 296 nano-facets like structures with larger dimensions are developed, although the facets are not
 297 well organized. It is also evident that the rms roughness is also increasing with bombardment
 298 time.

299 Surface nano-structuring by energetic ion bombardment is a consequence of ion-beam-
 300 induced off-normal (60° and 72.5°) sputtering of surface atoms and their consecutive
 301 redistribution [9,32,33]. During ion bombardment, the unequal radius of curvature of the surface

302 ensures unequal deposition of energy at different points on the surface, which results in unequal
303 sputtering at those points. This generates surface instability, and consequently, the surface
304 atoms are redistributed to stabilize the surface. These two effects jointly trigger nano-pattern
305 formation on the surface. A first theoretical model was proposed by Bradley and Harper [34],
306 based on curvature-dependent sputtering of surface and near-surface atoms. Later, Carter and
307 Vishnyakov introduced the concept of redistribution of surface atoms [35]. In present days,
308 several experiments have been carried out to understand other factors that contribute to nano-
309 pattern formation, such as preferential and differential sputtering, the role of surface and beam
310 impurities, the effect of chemical compound formation, and compound ion irradiation. In cases
311 of ultra-low energy ion bombardment, the rate of sputtering is lower compared to medium-
312 energy ion bombardment; therefore, in this case, mass redistribution of the surface atoms plays
313 a key role. Being inert, the reaction between Ar-ion and Si atoms is forbidden, ensuring the
314 absence of the chemical aspect of pattern formation. However, the native silicon oxide layer
315 partially sputters out with the bombardment of Ar-ion. In the present case, the sputtering of
316 elemental Si atoms takes place along with the Si atoms in oxide form. This is also a key factor
317 in generating surface instability. The surface morphology largely varies due to different
318 amounts of near-surface mass transport by the surface-confined ion-enhanced viscous
319 flow[36]. Here in the present case, up to an ion incidence angle of 58° , the surface remains
320 unstable under 450 eV Ar-ion bombardment. Due to such sputtering, a well-defined ripple
321 formation is found after an hour of 450 eV Ar-ion bombardment. With the increase in
322 bombardment time, Si atoms in both elemental and oxide form sputter more, and a well-ordered
323 ripple is obtained. However, due to the presence of the ripple, the surface becomes anisotropic.
324 The consequence of such an anisotropic nature of the surface is investigated and discussed in
325 the upcoming section.

326

327 Application of nano-patterned Si surface:



328

329 **Figure 8:** (a) UV-VIS-IR spectra of pristine and nano-patterned surfaces, (b) variation of rms
330 roughness and reflectivity with ripple wavelength.

331 The optical response of pristine and Ar-ion-induced nano-patterned silicon surfaces are
332 investigated through UV-VIS reflectivity & presented in Figure 8. Figure 8(a) depicts the
333 change in reflectivity of the silicon surface due to the presence of nano-pattern. With the
334 increase of bombardment time, reflectivity decreases drastically. Further the change in
335 reflectivity in UV region with respect to rms roughness and ripple wavelength is also
336 investigated in Fig 8(b). It is clear from the Fig 8(b), that the reflectivity decreases with the
337 increase in ripple wavelength. Further, variation of the rms roughness of the Ar-ion irradiated
338 nano-patterned Si surfaces is nominal. In general, the presence of nano-patterns on the surface
339 enhances the reflection of UV-VIS light, due to light trapping by multiple reflections [37–39].
340 The presence of a well-defined ripple pattern, formed due to Ar-ion bombardment (for three
341 hours), leads to the development of a well-defined nanopattern on the silicon surface and hence
342 the reflectivity is minimal compared to the other two surfaces. The change in reflectivity
343 depends on the change in the electronic structure as well as surface topography of the material.
344 Such a change in electronic structure can contain several factors, like the change in chemical

345 nature or impurity incorporation on the surface, and amorphization of the surface. Ar-being
346 inert causes no chemical modification of the silicon surface, along with the absence of the
347 trapped Ar-ion on silicon surface (concluded from TEM image in Fig 5(g)), particularly in this
348 lower energy regime. Therefore, in the present case, the amorphization due to ion beam
349 sputtering, the amorphization of the silicon surface, and the nanostructure formation on the
350 surface, together change the electronic density of the material, causing a lowering in
351 reflectivity. The tailoring of the reflectivity by developing nano-structure is widely applicable
352 for anti-reflective (ARC) coating and photovoltaic device applications [40,41].

353 The formation of nano-structure on the silicon surface by inert ion bombardment is a
354 consequence of ion-induced instability on the surface by the interplay between sputtering and
355 mass redistribution of surface atoms [42,43]. During ion bombardment, the sputtering of the
356 native silicon oxide layer along with the bulk silicon takes place. The rate of sputtering of
357 silicon oxide and the elemental silicon is different which initiates the instability at the initial
358 time of bombardment. Further, the instability is enhanced by differences in the sputtering yield
359 of silicon in native oxide and elemental form. These two effects are jointly responsible for
360 developing the nano-pattern on the surface. With the increasing bombardment time, the rate of
361 sputtering of Si in elemental and compound form increases, and a well-defined periodic
362 structure is observed. Further, during sputter erosion, the native oxide layer is mostly removed,
363 forming a silicon ripple structure. Further, the exposure of the nano-patterned silicon surface
364 to air during optical measurement ensures the formation of non-uniform silicon oxide on the
365 nano-patterned silicon surface. Due to such preferential spatial formation of silicon oxide, the
366 change in reflectivity is triggered. Therefore, nano-patterned silicon surfaces can be an
367 alternative for memory devices.

368

369

370 **Conclusion:**

371 In this comprehensive study, the intricacies of an ultra-low energy magnetron-based Electron
372 Cyclotron Resonance (ECR) ion source are studied systematically by exploring optimal
373 parameters to achieve stable and intense beam currents. The cost-effectiveness and versatility
374 of this ion source make it particularly noteworthy, offering a practical solution for generating
375 reasonable beam currents. Notably, the ion source operates within an ultra-high vacuum
376 environment, rendering it valuable for both implantation and deposition processes. Our
377 meticulous investigation into the ECR-based ultra-low energy ion source lays the groundwork
378 for ion beam-induced nano-structuring and layer-wise material modification, affording precise
379 control over ion penetration depth and fluence. The manuscript emphasizes an intriguing
380 alternative perspective by highlighting the in-depth optimization of low energy ion source and
381 inert ion-induced nano-patterning as a viable approach for ARC coating. Additionally, the
382 manuscript underscores the potential of nano-patterned silicon surfaces as an alternative
383 material for tailoring reflectivity, particularly for Anti-Reflective Coating (ARC) applications.
384 This study not only advances our understanding of ECR-based ion sources but also opens
385 avenues for innovative applications in nanotechnology and materials science.

386 **Acknowledgement:**

387 The authors thank the Department of Atomic Energy, Govt. of India for financial support.

388 **References:**

- 389 (1) Bhowmick, S.; Mukherjee, J.; Satpati, B.; Karmakar, P. *Appl Surf Sci* **2022**, 578.
390 doi:10.1016/j.apsusc.2021.152079
- 391 (2) Barofsky, D. F. *Mass Spectrometric Analyses in Agriculture and Natural Product*
392 *Research*; 1999; Vol. 29

- 393 (3) Gambino, N.; Myalsky, S.; Adler, L.; De Franco, A.; Ecker, F.; Guidoboni, G.; Kurfürst,
394 C.; Penescu, L.; Pivi, M.; Schmitzer, C.; Strasik, I.; Wastl, A. Impact of Ion Source
395 Stability for a Medical Accelerator. In *Journal of Instrumentation*; Institute of Physics
396 Publishing, 2019; Vol. 14. doi:10.1088/1748-0221/14/05/C05017
- 397 (4) Gammino, S. Ion Sources for Medical Applications. In *CERN Yellow Reports: School*
398 *Proceedings*; CERN, 2017; Vol. 1, pp 59–70. doi:10.23730/CYRSP-2017-001.59
- 399 (5) Satoh, K.; Oono, Y. Studies on Application of Ion Beam Breeding to Industrial
400 Microorganisms at Tiara. *Quantum Beam Science*. MDPI June 1, 2019.
401 doi:10.3390/qubs3020011
- 402 (6) Mukherjee, J.; Bhowmik, D.; Mukherjee, M.; Satpati, B.; Karmakar, P. *J Appl Phys*
403 **2020**, *127*. doi:10.1063/1.5144960
- 404 (7) Mukherjee, J.; Bhowmik, D.; Bhattacharyya, G.; Satpati, B.; Karmakar, P. *Journal of*
405 *Physics Condensed Matter* **2022**, *34*. doi:10.1088/1361-648X/ac4937
- 406 (8) De Lima, L. H.; Cun, H. Y.; Hemmi, A.; Kälin, T.; Greber, T. Note: An Ion Source for
407 Alkali Metal Implantation beneath Graphene and Hexagonal Boron Nitride Monolayers
408 on Transition Metals. In *Review of Scientific Instruments*; 2013; Vol. 84.
409 doi:10.1063/1.4848936
- 410 (9) Norris, S. A.; Aziz, M. J. Ion-Induced Nanopatterning of Silicon: Toward a Predictive
411 Model. *Applied Physics Reviews*. American Institute of Physics Inc. March 1, 2019.
412 doi:10.1063/1.5043438
- 413 (10) Teshome, B.; Facsko, S.; Keller, A. *Nanoscale* **2014**, *6*, 1790–1796.
414 doi:10.1039/c3nr04627c

- 415 (11) Bhowmik, D.; Karmakar, P. *Surf Coat Technol* **2020**, 385.
416 doi:10.1016/j.surfcoat.2020.125369
- 417 (12) Mollick, S. A.; Singh, R.; Kumar, M.; Bhattacharyya, S.; Som, T. *Nanotechnology* **2018**,
418 29. doi:10.1088/1361-6528/aaaa74
- 419 (13) Parida, B. K.; Kundu, A.; Hazra, K. S.; Sarkar, S. *Appl Phys A Mater Sci Process* **2021**,
420 127. doi:10.1007/s00339-021-05117-0
- 421 (14) Bhowmick, S.; Mukherjee, J.; Satpati, B.; Karmakar, P. *Appl Surf Sci* **2022**, 578.
422 doi:10.1016/j.apsusc.2021.152079
- 423 (15) Kasani, S.; Curtin, K.; Wu, N. A Review of 2D and 3D Plasmonic Nanostructure Array
424 Patterns: Fabrication, Light Management and Sensing Applications. *Nanophotonics*. De
425 Gruyter 2019. doi:10.1515/nanoph-2019-0158
- 426 (16) Saini, M.; Singh, R.; Sooraj, K. P.; Basu, T.; Roy, A.; Satpati, B.; Srivastava, S. K.;
427 Ranjan, M.; Som, T. *J Mater Chem C Mater* **2020**, 8, 16880–16895.
428 doi:10.1039/d0tc03862h
- 429 (17) Abdel Rahman, M. M.; Abdel Salam, F. W.; Soliman, B. A. Improved Treatment of
430 Home-Made Glow Discharge Ion Source. In *Journal of Physics: Conference Series*;
431 Institute of Physics, 2022; Vol. 2304. doi:10.1088/1742-6596/2304/1/012011
- 432 (18) Publications, T. J. G.; Gaus, A. D.; Htwe, W. T.; Brand, J. A.; Gay, T. J.; Schulz, M.;
433 Gay, T. J. *DigitalCommons@University of Nebraska-Lincoln*
434 *DigitalCommons@University of Nebraska-Lincoln Energy Spread and Ion Current*
435 *Measurements of Several Ion Energy Spread and Ion Current Measurements of Several*
436 *Ion Sources Sources Energy Spread and Ion Current Measurements of Several Ion*
437 *Sources*; 1994

- 438 (19) Schmor, P. W. *A REVIEW OF POLARIZED ION SOURCES*; 1996
- 439 (20) Angra, S. K.; Kumar, P.; Dongaonkar, R. R.; Bajpai, R. P. *Unstable Plasma*
440 *Characteristics in Mirror Field Electron Cyclotron Resonance Microwave Ion Source*;
441 2000; Vol. 54
- 442 (21) Asmussen, J. *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films*
443 **1989**, 7, 883–893. doi:10.1116/1.575815
- 444 (22) Schmidt, A. A.; Offermann, J.; Anton, R. *Thin Solid Films* **1996**, 281–282, 105–107.
445 doi:10.1016/0040-6090(96)08586-0
- 446 (23) Anton, R.; Wiegner, T.; Naumann, W.; Liebmann, M.; Klein, C.; Bradley, C. *Review of*
447 *Scientific Instruments* **2000**, 71, 1177–1180. doi:10.1063/1.1150420
- 448 (24) Ziegler, J. F.; Ziegler, M. D.; Biersack, J. P. *Nucl Instrum Methods Phys Res B* **2010**,
449 268, 1818–1823. doi:10.1016/j.nimb.2010.02.091
- 450 (25) Ohtsu, Y. Physics of High-Density Radio Frequency Capacitively Coupled Plasma with
451 Various Electrodes and Its Applications. In *Plasma Science and Technology - Basic*
452 *Fundamentals and Modern Applications*; IntechOpen, 2019.
453 doi:10.5772/intechopen.78387
- 454 (26) Chu, P. K.; Qin, S.; Chan, C.; Cheung, N. W.; Ko, P. K. *IEEE Transactions on Plasma*
455 *Science* **1998**, 26, 79–84. doi:10.1109/27.659535
- 456 (27) Ghosh, S. N.; Dhungel, S. K.; Yoo, J.; Gowtham, M.; Yi, J.; Bora, D. *Study of High-*
457 *Density Helicon-Plasma Generation and Measurement of the Plasma Parameters by*
458 *Using a Frequency-Compensated Langmuir Probe*; 2006; Vol. 48
- 459 (28) Yang, Y. R.; Fu, S. H.; Ding, Z. F. *AIP Adv* **2022**, 12. doi:10.1063/5.0082813

- 460 (29) Fournier, P.; Lisi, N.; Meyer, C.; Scrivens Cern, R.; Division, P. S.; Ostroumov, P.
461 *Experimental Characterisation of Gridded Electrostatic Lens (GEL) Low Energy Beam*
462 *Transport (LEBT) for the Laser Ion Source (LIS) and Effect of a Wire Grid on the*
463 *Extraction Electrode*; 1999
- 464 (30) Dudin, S. V.; Rafalskyi, D. V. *European Physical Journal D* **2011**, *65*, 475–479.
465 doi:10.1140/epjd/e2011-20402-y
- 466 (31) Deka, A.; Barman, P.; Mukhopadhyay, M. K.; Bhattacharyya, S. R. *Surfaces and*
467 *Interfaces* **2021**, *25*. doi:10.1016/j.surfin.2021.101242
- 468 (32) Jain, I. P.; Agarwal, G. Ion Beam Induced Surface and Interface Engineering. *Surface*
469 *Science Reports*. Elsevier B.V. 2011, pp 77–172. doi:10.1016/j.surfrep.2010.11.001
- 470 (33) Datta, D. P.; Garg, S. K.; Basu, T.; Satpati, B.; Hofsäss, H.; Kanjilal, D.; Som, T. *Appl*
471 *Surf Sci* **2016**, *360*, 131–142. doi:10.1016/j.apsusc.2015.10.133
- 472 (34) Bradley, R. M.; Harper, J. M. E. *Journal of Vacuum Science & Technology A: Vacuum,*
473 *Surfaces, and Films* **1988**, *6*, 2390–2395. doi:10.1116/1.575561
- 474 (35) Carter, G.; Vishnyakov, V. *Roughening and Ripple Instabilities on Ion-Bombarded Si*;
475 1996
- 476 (36) Vorathamrong, S.; Panyakeow, S.; Ratanathamphan, S.; Praserttham, P. *AIP Adv*
477 **2019**, *9*. doi:10.1063/1.5084344
- 478 (37) Mennucci, C.; Muhammad, M. H.; Hameed, M. F. O.; Mohamed, S. A.; Abdelkhalik,
479 M. S.; Obayya, S. S. A.; Buatier de Mongeot, F. *Appl Surf Sci* **2018**, *446*, 74–82.
480 doi:10.1016/j.apsusc.2018.02.186
- 481 (38) Garnett, E.; Yang, P. *Nano Lett* **2010**, *10*, 1082–1087. doi:10.1021/nl100161z

- 482 (39) Amalathas, A. P.; Alkaisi, M. M. Nanostructures for Light Trapping in Thin Film Solar
483 Cells. *Micromachines*. MDPI AG September 1, 2019. doi:10.3390/mi10090619
- 484 (40) Mousavi, B. K.; Mousavu, A. K.; Busani, T.; Zadeh, M. H.; Brueck, S. R. J. *Journal of*
485 *Applied Mathematics and Physics* **2019**, *07*, 3083–3100.
486 doi:10.4236/jamp.2019.712217
- 487 (41) Zang, K.; Jiang, X.; Huo, Y.; Ding, X.; Morea, M.; Chen, X.; Lu, C. Y.; Ma, J.; Zhou,
488 M.; Xia, Z.; Yu, Z.; Kamins, T. I.; Zhang, Q.; Harris, J. S. *Nat Commun* **2017**, *8*.
489 doi:10.1038/s41467-017-00733-y
- 490 (42) Madi, C. S.; Anzenberg, E.; Ludwig, K. F.; Aziz, M. J. *Phys Rev Lett* **2011**, *106*.
491 doi:10.1103/PhysRevLett.106.066101
- 492 (43) Chan, W. L.; Chason, E. *J Appl Phys* **2007**, *101*. doi:10.1063/1.2749198
- 493
- 494