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Charge equilibration and irradiation damage threshold for MeV ions in polyimide

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ABSTRACT

Polyimide foils were irradiated with MeV ions below charge state equilibrium and etched in sodium hypochlorite solution to measure the material depth below which ion tracks occurred. It was observed that no etchable ion tracks were formed in the first few nanometres of the foil as the electronic stopping power was below the damage threshold. The damage threshold was crossed at a certain depth in the foil and intermittent tracks began to form. The measurement of depth for crossing the damage threshold in polyimide enabled verification of the previously theoretical models for the evolution of nonequilibrium charge variation of electronic stopping power during charge state equilibration of energetic ions and explicitly demonstrated that the equilibration process plays a role in ion-induced nanostructure formation.

1. Introduction

Understanding of the interaction of energetic ions with solids is important for fundamental studies and applications in ion beam material analysis and modification. The electronic stopping power ($S_e = dE/dx$), defined as the energy loss in inelastic collisions per unit length traversed by the ion, is one key parameter in ion-matter interactions, which is reported to be dependent on the charge (q) of the ion [1,2]. The ion entering the solid can either lose or capture electrons depending on the ion velocity and charge state. One of the two processes, i.e., electron capture or loss by the projectile ion, dominates until the ion reaches the mean equilibrium charge (q_{eq}). q_{eq} is defined as the charge at which the electron loss and capture rates by the projectile have reached an equilibrium. The probability of electron loss by the projectile increases with the projectile velocity (v_p), resulting in a higher q_{eq} for higher v_p. For ions with $q \neq q_{eq}$, the distance traversed before reaching the equilibrium can vary from a few nanometres for MeV ions to a few microns for GeV ions [3]. The S_e in the initial layers of the target, before q_{eq} is reached, is different from the S_e in the bulk [4–8]. Thus, studying the evolution of charge state and characteristics of charge state equilibration is significant for predicting material modification or nanostructuring by ion irradiation on or near the surface of a material.

Swift heavy ion (SHI) irradiation is a versatile tool for material modification and nanostructuring, allowing precise damage tuning due to the possibility of controlling irradiation parameters such as ion energy, fluence and irradiation angle, and thus energy deposition rate and distribution in the target material. Understanding the charge evolution along the individual trajectories will allow better control over nanostructuring. Many experimental and theoretical studies have been performed on the charge response of SHI during material modifications and nanostructuring [9–13]. Among many other applications, SHI irradiation and subsequent etching is a well-established method for fabricating solid-state nanoscale pores and channels in polymers [14–19]. The damage threshold for the formation of etchable ion tracks in polymers is commonly reported in S_e. Polyimide (PI) is considered a suitable candidate for studying the obtained nanopores because of its excellent chemical and mechanical properties and stable current signals in pores [20]. SHI irradiation has been reported to form intermittent tracks for S_e above 1.85 keV/nm and continuous tracks above 5 keV/nm [21]. A detailed study of etchable ion track formation in PI foils using MeV ions was performed in [22], reporting a velocity-dependent threshold S_e for intermittent and continuous tracks in the MeV energy regime. However, the effect of the evolution of ion charge states on the etchable ion tracks in PI is not discussed in previous works. As S_e is dependent on q, which varies in the initial part of the interaction where $q \neq q_{eq}$, the ion charge evolution needs to be taken into account. The chemically etched ion tracks can be studied to understand the charge exchange process and thus charge evolution of ions in solid targets [23,24].

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In the present work, the evolution of the charge state in the case of MeV ions is investigated by etching the irradiated PI foils for a set of different times below one hour. Projectile ions with q_{in} such that S_e at the surface is below the damage threshold for intermittent tracks (S_{et}) can be used to observe the crossing of S_{et} at a certain depth in the material by removing the first few layers of material at the well-known bulk etch rate (V_B). The equilibration length (d), defined as the length over which the difference between actual q and q_{eq} reduces by a factor 1/e, is observed to be on the order of a few nanometres for MeV ions [5]. PI etched with sodium hypochlorite (NaOCl) solution of pH 10 at 50 °C is reported to have V_B of ~ 10 nm/h [19,22], making the etching times of less than an hour enough to reach d in the foil. The theoretical value of d is calculated based on the fit of the previous data on charge equilibration, done in [5], which is then used to predict the depth (x_{th}) for the crossing of the damage thresholds reported in [22]. These theoretically predicted values are compared with the experimentally observed values of x_{th} for different projectile ions.

2. Experimental

Commercially available Kapton® 30EN polyimide foils with a thickness of 7.5 µm (from DuPont) were irradiated in high vacuum at room temperature under normal incidence by ^{16}O and ^{59}Co ions with energies ranging from 2 to 17 MeV, an initial charge state between 1+ and 9+ and fluences of (1.0–5.0) × 10^{10} ions/cm^2. All the irradiations were performed using the 5 MV pelletron tandem accelerator at Uppsala University [25]. The complete list of projectile ions used to irradiate the PI foils in the present work is given in Table 1. The q_{eq} was calculated using the formula for solid-state targets from reference [10], mean projected range (R_p) and S_e at q_{eq} were calculated using SRIM-2013 [26] and S_{et} was estimated from the threshold S_e for the formation of intermittent tracks in the MeV energy regime [22]. Calculations of the rest of the parameters in Table 1 are discussed in the Results and Discussion section below. The wet chemical etching was performed using a sodium hypochlorite (NaOCl) solution of pH = 10 kept at a temperature of (50 ± 1)°C, as described in the reference [22]. Zeiss LEO 1530 and Zeiss LEO 1550 Scanning Electron Microscopy (SEM) at an acceleration voltage of 10 kV was used for the surface characterization of the irradiated PI foils. The foils were coated with a thin layer of Au-Pd using a Polaron SC7640 sputter coater to prevent the charging of the foils during SEM measurements. SEM images were processed using ImageJ [27].

3. Results and discussion

The scaling of S_e with charge (q) is given by:

$$\left(\frac{dE}{dx}\right)_q = \left(\frac{q}{q_{eq}}\right)^k \left(\frac{dE}{dx}\right)_{eq} \tag{3.1}$$

Convolution approximation for Swift Particles (CasP) [28], a program for numerical calculations of the mean electronic energy transfer, assumes the value of the k to be less than 2. In literature, the value of k varies from 1.5 to 2 [5,8]. However, in this paper k is assumed to be 2, i.e., q^2-scaling of the S_e, as assumed by various authors in literature [5,7,29,30]. In the case of 2 MeV Co^{1+}, $q_{in} = 0.22q_{eq}$ and thus, S_e on the surface of the foil is 0.04 keV/nm which is lower than the reported S_{et} of 0.5 ± 0.1 keV/nm for intermittent tracks [22]. Thus, the depth for S_{et} crossing (x_{th}) is expected to be reached when the mean q state of the projectile ions reaches 3.3 ± 0.4 (calculated using Eq. (3.1)). Fig. 1 shows the SEM images for PI foils irradiated with 2 MeV Co^{1+} and etched for (a) 15, (b) 20, (c) 25 and (d) 30 min. No pore openings are observed on the surface of the foil etched for 20 min or less but for the foils etched for 25 min or more a few nanometres wide pore openings in the form of etched-out ion tracks are observed on the surface. Converting the etching times of 20 and 25 min into the corresponding depth in PI foil, by using V_B of 10.3 ± 0.4 nm/h [22], we infer that the x_{th} is in the range of 3.4–4.3 nm. The pore production efficiency, i.e., the ratio of the number of pore openings observed on the surface per unit area to the number of incidence ions per unit area, is 0.04 ± 0.01 and 0.1 ± 0.04 for foils etched for 25 and 30 min respectively. As the energy deposition is a stochastic process and we are very close to the threshold for the formation of intermittent tracks, a low efficiency is expected. An uncertainty of 25 % in scan area, 10 % in ion current and 10 % in counting the pore openings is assumed. 3–5 different regions on the surface of PI foils, of area ranging from 1 to 6 µm^2, are analysed for calculating the number of pore openings on the surface.

To study the evolution of the non-equilibrium charge state we assume a simple model, proposed by Bohr and Lindhard [31], such that q approaches q_{eq} as follows:

$$q = q_{eq} + (q_{in} - q_{eq})e^{-x/d} \tag{3.2}$$

where x is the depth in the foil and d is the equilibration length. For 2 MeV Co^{1+}, d is theoretically estimated to be 2.9 nm from the fit to previously reported data on charge equilibration, done in [5] assuming k = 2 in Eq. (3.1). From the values of S_{et} and q_{eq}, mean q at S_{et} is calculated using Eq. (3.1) and further, the depth corresponding to the mean q at S_{et} is calculated using Eq. (3.2). Taking the propagated error in S_{et} into account, the theoretical value of x_{th} is estimated to be in the range of 2.5–4.2 nm. This estimated value of x_{th} concurs with the experimentally observed value of x_{th}. Using the expression of q in Eq. (3.2) and k = 2, Eq. (3.1) becomes:

$$\left(\frac{dE}{dx}\right)_q = \left(1 + 2\left(\frac{q_{in}}{q_{eq}} - 1\right)e^{-x/d} + \left(\frac{q_{in}}{q_{eq}} - 1\right)^2 e^{-2x/d}\right)\left(\frac{dE}{dx}\right)_{eq} \tag{3.3}$$

In this paper, we use ions with $q_{in} < q_{eq}$, making (q_{in}/q_{eq} −1) negative, which gives S_e at any given depth (x) as a sum of the constant and the difference between two exponential functions. Fig. 2 shows the evolution of S_e as a function of etching time and depth in the PI foil for (a) 2 MeV Co^{1+}, (b) 17 MeV Co^{3+} and (c) 13 MeV O^{3+} based on the Eq. (3.3).

Table 1
List of the projectile ions used for irradiating 7.5 µm thick polyimide (PI) foils with their kinetic energy (E_K), mean projected range (R_p), mean equilibrium charge state (q_{eq}), electronic stopping power (S_e) at q_{eq}, initial charge (q_{in}), electronic stopping power (S_e) at q_{in}, threshold electronic stopping power for intermittent tracks (S_{et}), theoretically calculated equilibration length (d), experimentally observed and theoretically estimated depth for crossing the damage threshold for the formation of intermittent ion tracks in the PI foils (x_{th}).

Ion	E_K (MeV)	R_p (µm)	q_{eq}	S_e at q_{eq} (keV/nm)	q_{in}	S_e at q_{in} (keV/nm)	S_{et} (keV/nm)	Experimentally observed x_{th} (nm)	Theoretical d (nm)	Theoretically estimated x_{th} (nm)
^{16}O	2	2.9	3.5	1.15	1+	0.09	0.5 ± 0.1	< 2.8	4.5	(2.5 – 4.2)
	3.85	4.3	4.4	1.46	2+	0.30	0.5 ± 0.1	(0.9 – 1.7)	5.6	(0.7 – 1.5)
	6.5	6.1	5.2	1.54	1+	0.06	0.6 ± 0.1	(3.4 – 4.3)	6.6	(4.2 – 6.0)
					4+	0.93		≤ 3.4		On surface
	13	10.4	6.4	1.4	3+	0.31	1.0 ± 0.1	(3.4 – 5.2)	4.5	(4.5 – 7.0)
^{59}Co	2	2.1	4.6	0.88	1+	0.04	0.5 ± 0.1	(3.4 – 4.3)	2.9	(2.5 – 4.3)
	8	5.3	8.5	2.84	2+	0.16	0.5 ± 0.1	(0.9 – 1.7)	4.6	(0.9 – 1.6)
					6+	1.40		< 0.9		On surface
	17	7.8	11.3	4.43	3+	0.31	0.6 ± 0.1	(0.5 – 0.9)	5.9	(0.6 – 1.2)
					9+	2.81		< 0.5		On surface

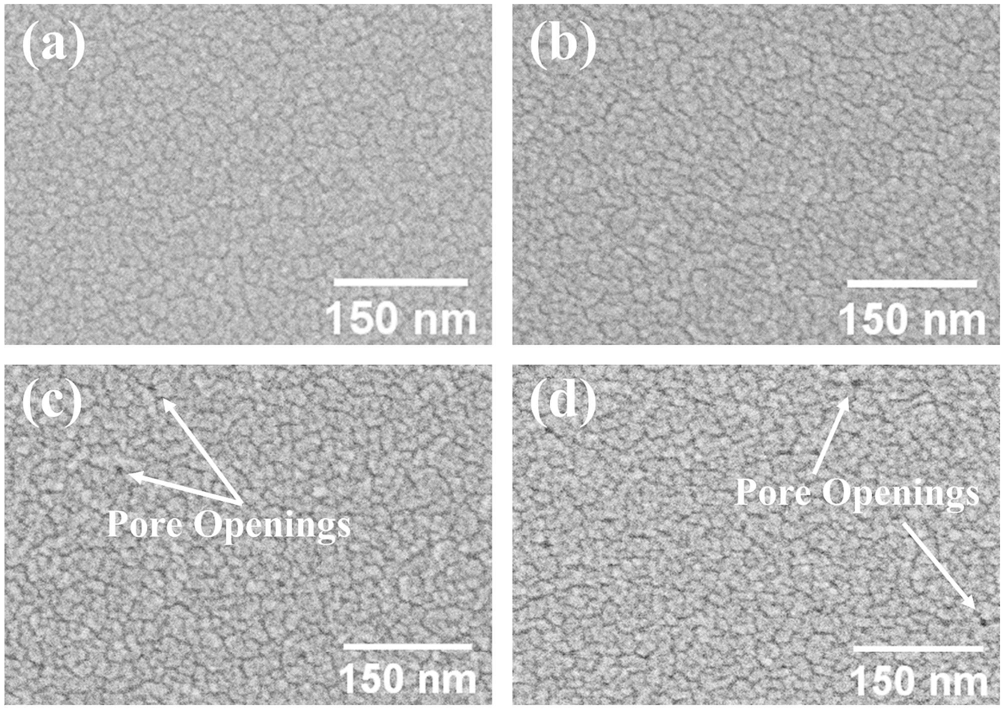

Fig. 1. SEM images of the polyimide (PI) foils irradiated with 2 MeV Co^{1+} and etched for (a) 15, (b) 20, (c) 25 and (d) 30 min in NaOCl solution kept at 50 °C and pH = 10. All the foils were irradiated with a fluence of 2×10^{10} ions/cm^2.

The depth is calculated from the etching time and V_B of 10.3 nm/h reported in [22]. The dashed red vertical lines indicate depths above which $S_e < S_{et}$ (pore openings not observed) and dashed blue vertical lines indicate depths below which $S_e > S_{et}$ (pore openings observed). In the case of dashed blue lines, where pore openings are observed, the pore production efficiency is also plotted in Fig. 2. As the ions traverse in the foil, the S_e increases: first the S_{et} is reached where the intermittent ion tracks start to form and then, as the value of S_e increases above S_{et}, the fraction of the etchable part in the intermittent ion tracks increases resulting in an increase in the efficiency of the pore production.

Table 1 gives the complete list of the theoretically estimated and experimentally observed x_{th} for different projectile ions. It is observed that for ions with energy less than 1 MeV/u and $0.2q_{eq} < q_{in} < 0.5q_{eq}$, the experimentally observed x_{th} lies within the theoretically estimated range of x_{th}. This confirms that the simple model of charge evolution in Eq. (3.2) and q^2-scaling of the S_e in Eq. (3.1) can be used to predict the charge evolution in these cases. If $k \neq 2$ in Eq. (3.1), then d will also be different from the one calculated in this paper as it is calculated under the assumption that $k = 2$. For a lower value of k, we expect a longer d, leading to a similar range of the x_{th}. However, the fact that for projectiles with $q_{in} < q_{eq}$, the damage threshold for intermittent tracks is crossed at a certain depth in the foil yields a measurable effect, is still valid. In the case of a few projectiles, used in the present work, with $q_{in} > 0.5\ q_{eq}$ the S_e calculated on the surface is above S_{et}. For 8 MeV Co^{6+} and 17 MeV Co^{9+}, the surface openings are observed very close to the surface and at depths less than that for the ions with the same mass and energy but comparatively lower q_{in} (i.e., 8 MeV Co^{2+} and 17 MeV Co^{3+} respectively). In contrast, for 6.5 MeV O^{4+} (S_e at surface = 0.93 keV/nm > S_{et}), no nanopore openings are observed on the surface even after 20 min of etching (3.4 nm deep inside the foil). One of the plausible explanations for not observing the surface openings, in this case, can be that the S_{et} is the threshold for intermittent tracks and hence, either the etchable parts of the intermittent tracks start deeper in the foil or the etchable fraction of the intermittent tracks near the threshold in this case was so low that only a negligible number of pores were formed.

4. Summary and concluding remarks

We have demonstrated that the initial charge state of the ion and charge equilibration process affect the formation of nanostructures. The polyimide foils irradiated with MeV ions below charge state equilibrium were etched for a set of different times and analysed to measure the depth below which ion tracks occurred in the foil. The ions with $q_{in} < q_{eq}$ and S_e at q_{in} less than the damage threshold for intermittent tracks (S_{et}) are observed to cross S_{et} at a depth of a few nanometres. These observations were used to study the equilibration process by comparing them with the previously reported theoretical models of charge evolution and q^2 scaling of S_e with charge. The experimentally observed depth for S_{et} crossing (x_{th}) was observed to lie within the theoretically estimated range of x_{th}.

Ion irradiation is considered a suitable method for nanoporous membrane production because the unique control over the energy, fluence and angle of irradiation allows precise tuning of nanostructure formation. Although ion irradiation is a stochastic process, the area of damage and amount of damage can be controlled by using masks [32] and tuning the ion parameters. Depending on the depth of the damage required or the thickness of the foils used for nanopores fabrication, MeV ion irradiation is a viable option for producing nanoscale pores or channels. However, as demonstrated in this paper, the charge state equilibration cannot be neglected and needs to be considered while planning the nanopore fabrication. The MeV ions have equilibration lengths (d) of a few nanometres whereas SHI have relatively larger values of d (on the order of a few microns for GeV ions). However, the problem of non-equilibrium charge may be fixed by pre-equilibrating the charge state by using a stripper foil, for example, a carbon foil. On the other hand, the dependence of S_e on the charge gives us another parameter to tune the nanostructure production in the materials using ion beams, for example, controlling the geometry of the nanostructured produced as the radius of ion track depends on S_e [33] and depth of nanostructure production in the material.

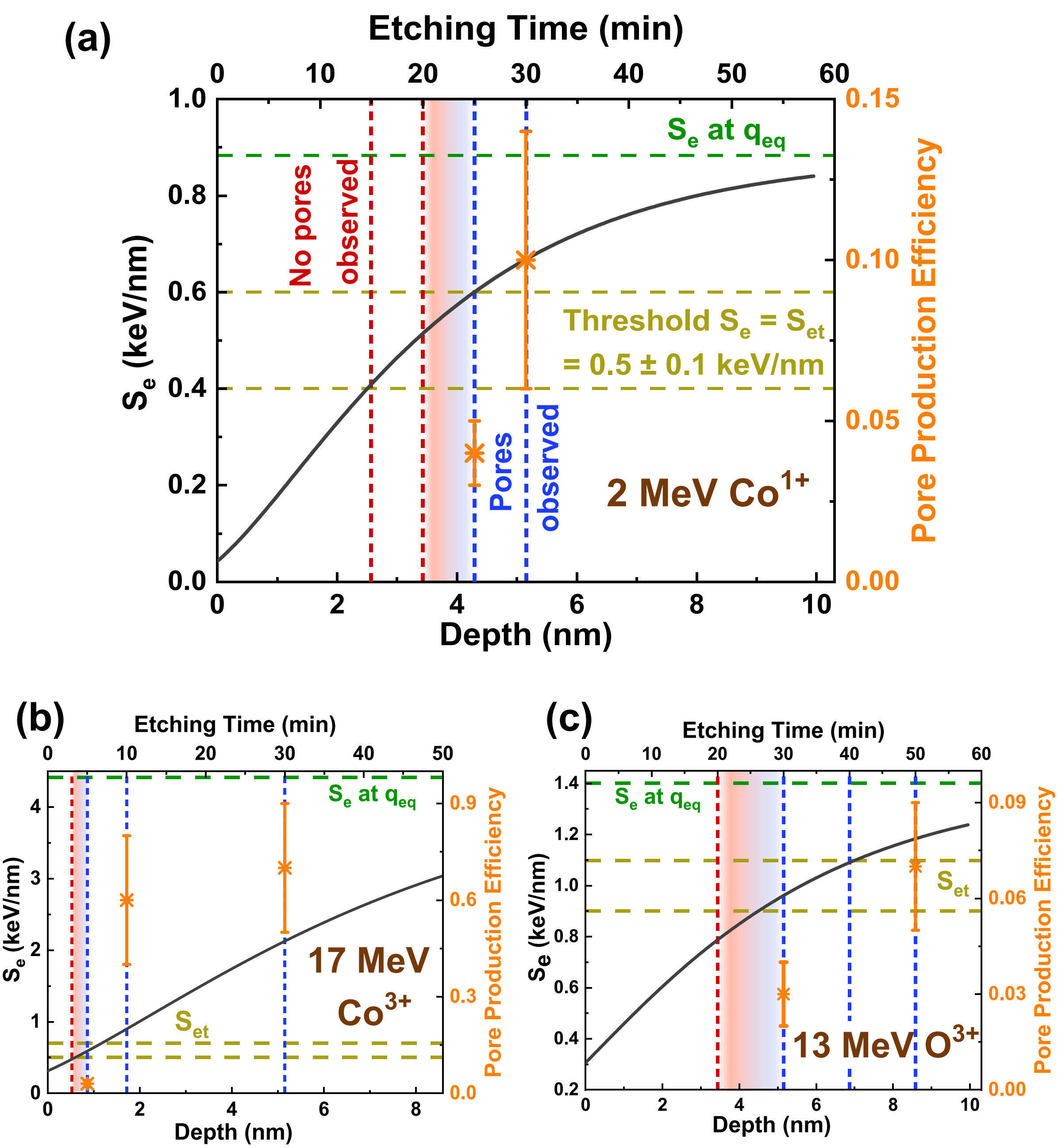

Fig. 2. The evolution of S_e for (a) 2 MeV Co^{1+}, (b) 17 MeV Co^{1+} and (c) 13 MeV O^{1+} in polyimide (PI), based on Eq. (3.3). The etching times are converted into the corresponding depth in PI foil, by using V_B of 10.3 nm/h [22]. The dashed vertical lines correspond to the etching times after which the PI foils were analysed using SEM: red lines indicate depths (or etching times) where no pore openings were observed and blue lines indicate depths (or etching times) where pore openings were observed. The shaded region gives the experimentally observed range of depth where the damage threshold is crossed (x_{th}). The orange crossed points give the efficiency of the pore production as observed on the surface of the foil.

CRediT authorship contribution statement

Rajdeep Kaur: Conceptualization, Data curation, Visualization, Investigation, Validation, Formal analysis, Software, Writing – original draft, Writing – review & editing. **Daniel Primetzhofer:** Conceptualization, Validation, Supervision, Resources, Funding acquisition, Writing – review & editing. **Petter Ström:** Conceptualization, Validation, Supervision, Resources, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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