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DIRECT SINGLE ION MACHINING OF NANOPORES

D.M. Follstaedt, A.K. Norman, P. Rossi, B.L. Doyle

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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D.M. Follstaedt, A.K. Norman, P. Rossi, B.L. Doyle
Radiation Solid Interactions Department
Physical and Chemical Sciences Center

Sandia National Laboratories
PO Box 5800
Albuquerque, NM 87185-1056

Abstract

The irradiation of thin insulating films by high-energy ions (374 MeV Au⁺²⁵ or 241 MeV I⁺¹⁹) was used to attempt to form nanometer-size pores through the films spontaneously. Such ions deposit a large amount of energy into the target materials (~20 keV/nm), which significantly disrupts their atomic lattice and sputters material from the surfaces, and might produce nanopores for appropriate ion-material combinations. Transmission electron microscopy was used to examine the resulting ion tracks. Tracks were found in the crystalline oxides quartz, sapphire, and mica. Sapphire and mica showed ion tracks that are likely amorphous and exhibit pits 5 nm in diameter on the surface at the ion entrance and exit points. This suggests that nanopores might form in mica if the film thickness is less than ~10 nm. Tracks in quartz showed strain in the matrix around them. Tracks were not found in the amorphous thin films examined: 20 nm-SiN_x, deposited SiO_x, fused quartz (amorphous SiO₂), formvar and 3 nm-C. Other promising materials for nanopore formation were identified, including thin Au and SnO₂ layers.

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Executive Summary

The formation of nanometer-size pores, or “nanopores”, through thin materials is important because it can advance both scientific investigations and the development of new devices. For instance, threading DNA through a pore while monitoring current or resistivity across the membrane could provide a means to determine the sequence of nucleotides and thereby identify genes. Positioning a bio-molecular cell-wall pore on a nanopore through a rigid, inert film could be used to study its properties. A membrane with uniform, nanometer-scale pores could be used to selectively pass molecular species of interest, and thus would be useful for sensor devices.

Nanopores are currently available through plastic membranes; they are produced by high-energy ion irradiation to produce tracks with broken molecular bonds through the plastic followed by chemical etching that occurs rapidly along the track. This method yields pores only as small as 10 nm, and chemical etching may produce irregular diameters. Other investigators are producing smaller pores by first forming holes 10's of nanometers across and then filling them in to obtain pores as small as 2 nm by continuously monitoring the opening. However, such pores must be individually tailored and monitored (with an electron microscope) while they are being closed-in to the desired diameter.

Producing small nanopores more effectively motivated our investigations. High-energy ions (100's of MeV) are known to deposit much energy into a material along their track. When the energy exceeds 10-20 keV/nm, the material is modified to produce a discernable track. In addition, ions with such energies produce increased sputtering of the surface. ***It was our intent to determine if high-energy ions could spontaneously produce nanopores along their path without subsequent processing such as chemical etching.*** Ion tracks are generally <10 nm in diameter and are regular in shape; these properties would be expected for nanopores if they are produced. Further, it is possible with present ion detection methods to produce a membrane with one and only one pore, making additional investigations possible.

Indeed, molecular dynamics simulations of high energy ions passing through solids indicate that an open track is temporarily produced, but closes as the target atoms reform the solid. After consulting world experts on track formation, we identified insulators such as oxides as likely to retain the opened pathway. Accordingly, we investigated high-energy Au (374 MeV) and I (241 MeV) ions passing through crystalline and amorphous oxides, as well as readily available thin membranes of SiN_x and carbon materials.

We used transmission electron microscopy (TEM) to search for ion tracks, and found them in thin sapphire, quartz and mica. Sapphire showed pits on the surface at the ion entrance and exit positions, with a track line connecting the pits that may have lower density. We were able to thin mica to 18 nm and to detect 5 nm pits at the ion entrance and exit points on both surfaces; this observation was the closest that we came to producing nanopores directly with ion beams. The tracks in quartz showed that the material immediately around them was strained, but we could not identify surface pits. We were unable to detect tracks in the amorphous solids fused quartz, SiO_n, SiN_x, formvar layers and amorphous carbon. Energy

deposition and track formation are not expected to depend on crystallinity. However, it may be that the contrast between a track and an amorphous matrix is too low.

For the ions and materials we used, it appears that films less than ~10 nm thick would be needed to produce nanopores. It may be possible to cleave mica to such a small thickness and thereby connect the pits on either surface to form a pore. Recent investigations elsewhere indicate that other materials, like SnO₂ and Au, absorb greater energy from high-energy ions; SnO₂ particles exhibited nanopores. Thus our hypothesis that nanopores could be formed directly through a membrane remains viable, and we believe worthy of further pursuit.

DIRECT SINGLE ION MACHINING OF NANOPORES

Introduction

The formation of nanopores is being studied by several laboratories [1-3]. The controlled formation of nanopores with tailored sizes would offer the opportunity both to advance scientific investigations and to develop important new devices. Well defined nanopores could be used to selectively pass one molecular species through a membrane and exclude others. Investigations in genetics and bio-molecules would benefit from a method to sequence deoxy-ribonucleic acid (DNA) molecular chains readily, and threading the DNA through a pore while monitoring current or resistivity across the membrane could provide a means to determine the sequence of nucleotides and thereby identify genes. The passing of DNA through a pore between the two sides of an ionic cell has already been shown to be detectable with the ionic current [4]. Additionally, a bio-molecular cell-wall pore could be positioned on a nanopore through a rigid, inert film as a platform to study its properties or to study molecules passing through it. A membrane with uniform, nanometer-diameter pores could be used to selectively pass molecular species of interest, and thus would be useful for sensor devices [5].

Nanopores are currently produced in plastic membranes and are commercially available [6]. High-energy ion irradiation is first used to break molecular bonds in the plastic along the ion path. Subsequent chemical etching is much more rapid along the path than in unaltered plastic, leading to a pore along the track. But this method yields pores only as small as 10 nm; in addition, the chemical etch may produce irregular pore sizes. Other investigators are producing smaller pores by first producing larger holes 10's of nanometers across; the holes are then filled by using electron or ion beams to deposit material locally, producing pores as small as 2 nm [1-3]. However, the pores must be individually tailored, requiring continuous monitoring of the opening with an electron microscope until they are reduced to the desired diameter.

High-energy ions (a few MeV/amu) transfer large amounts of energy to the electrons in the material they are passing through. The excited state of material along the ion path has been termed a "Coulomb explosion" where ionized atoms are repelled radially outward from the center of the track [7]. This energized track has also been described as a "thermal spike" in which the hot center cools by heat flowing radially outward [8, 9]. Recent atomistic simulations indicate that if a Coulomb explosion is initiated, it generally results in a subsequent thermal spike [10]. Such simulations show that a few picoseconds after the ion passes, the atoms are pushed out of the track leaving a near void along the path as in Fig. 1. Unfortunately, the displaced atoms return to this region and the material heals, closing the track.

Several investigations indicate that disordered tracks a few nanometers across are formed if the ions deposit energies exceeding 10-20 keV/nm to the target electrons [8,11]. Further, ions depositing such high energies also induce electronic sputtering on the incident surface and can produce craters [12-14]. Such cratering could promote pore formation in a

sufficiently thin specimen, especially if a second crater forms at the ion's exit position. Such observations lead us to ask if the right combination of ion and target could retain the open track and surface craters and produce a nanopore through a membrane directly, without subsequent chemical etching.

We consulted with experts from around the world on ion-track formation, and concluded that insulating materials such as oxides appeared likely to retain the open pore, reasoning that if coulomb repulsion displaced the atoms then an insulator would maintain their ionized state and resulting displacement longer.

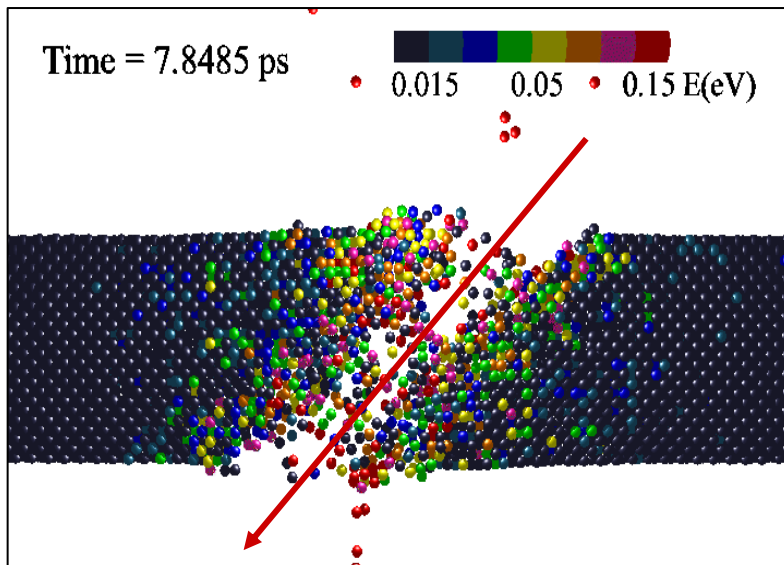


Figure 1. Molecular dynamics simulation showing the disrupted, open track produced by a high energy ion approximately 8 ps after the ion passed through the film, whose atoms are modeled with Leonard-Jones potentials. Courtesy Eduardo Bringa, Lawrence Livermore National Laboratory.
See: <http://dirac.ms.virginia.edu/~emb3t/craters/craters.html>

In the work described below, we used transmission electron microscopy (TEM) to investigate ion tracks in oxides, SiN_x , organic films and thin carbon films. Nanopores were not achieved with this approach, although some results suggest we might be close to forming them. We discuss the ion tracks that were observed and note the materials in which tracks were not detected. Then we consider our results and others in the recent literature to identify promising target materials in which ions might form nanopores directly.

Methods Used

A radio-frequency quadrupole (RFQ) booster was recently added to the tandem ion accelerator in Org. 01111 to obtain ions with energies of 1.9 MeV/amu. This allows the irradiation of target materials with 241 MeV I^{+19} and 374 MeV Au^{+25} . At these energies, the ions have stopping powers of 14 and 20 keV/nm, respectively, in SiO_2 [15] as needed to produce intense ionization along the track and electronic sputtering at the surface. When the RFQ was properly configured and aligned with the ion beam from the tandem, fluxes of $5 \times 10^3/cm^2$ -s were achieved. Day-long irradiations then produced fluences of $\sim 10^8$ ions/cm² at normal incidence.

Commercially available thin films (amorphous 20 nm- SiN_x , SiO_x , formvar, and 3nm-C), pre-thinned specimens mechanically polished and ion-milled for TEM examination (sapphire, quartz, glass), and thin mica layers formed by cleaving were irradiated. The materials were usually examined with TEM at ambient temperature and 200 kV. In materials with tracks not exhibiting strong diffraction contrast, the TEM is under focused by typically -6 to -8 μm and areas $\sim 1 \mu m \times 1 \mu m$ are searched using a CCD camera. At typical fluences, tracks are seen in about one-third of such areas. Underfocusing produces Fresnel contrast at voids and low-density areas, but this contrast is so weak that digital imaging and display at maximum contrast are required to detect ion tracks. To distinguish tracks from occasional irregular pits in the surface, the specimen is tilted 35-40° so that elongated tracks through the film are seen in projection aligned along the known tilt direction. In some specimens the tracks have strong diffraction contrast and can be seen directly on the TEM's viewing screen, making searching easier. Specimen damage from the TEM's electron beam occurred with continued imaging and limited our obtaining high magnification images. The damage rate was not noticeably reduced by imaging at 120 kV, nor by cooling specimens to -140°C.

Results

Sapphire (hexagonal Al_2O_3)

Irradiation with 374 MeV Au ions produced tracks in pre-thinned sapphire specimens as seen in Fig. 2a. The characteristic contrast features in under-focused images are bright areas on either side of the specimen with associated dark areas located beside them, away from the center of the overall feature. In such under-focused images, taken under kinematic conditions (no diffraction contrast), bright areas are areas of low density or voids; at the surface we identify them as pits. The dark areas are thought to indicate material piled-up on the surface around the pits as observed in both experiments and simulations with other materials [13,14]. The contrast reversed appropriately when the image is taken with over-focused conditions, and the separation of the surface features increased in the projected image as expected with increased specimen tilt angle. As seen in Fig. 1, the contrast of the craters on one side of the specimen appears less pronounced; this difference is thought to be due to prethinning with Ar ions that likely amorphized a thin surface layer on one side of the

specimen and perhaps thus reduced the contrast. Such tracks were also observed in a specimen irradiated with I^{+19} .

The higher magnification image in Fig. 2b shows a 6 nm-wide surface crater on one side and a track line passing through the material; the second crater on the other side has such low contrast that it is not readily identified. The track also appears brighter than the matrix, indicating that it may also have lower density; however this contrast could be influenced by residual diffraction contrast in the matrix if the track is amorphous as expected from other work [8, 11].

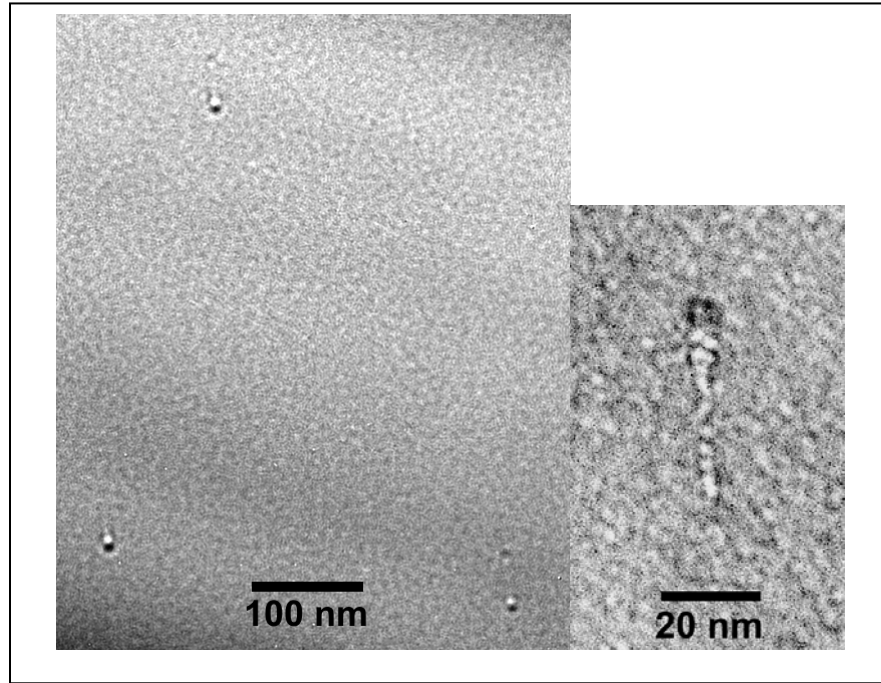


Figure 2. a) (Left) Three tracks of 374 MeV Au in sapphire, imaged with $9\ \mu\text{m}$ under focus. b) (Right) Enlarged image of a similar track.

α -Quartz (hexagonal SiO_2)

Tracks in a prethinned specimen of quartz were readily detected with good contrast that is related to diffraction of the matrix, as seen in Fig. 3. In Fig. 3a the three tracks are taken near the edge of a diffraction contour where the matrix becomes strongly diffracting. Dark contrast is seen on either side of the track line; it oscillates along the line and appears symmetric between the two sides. An enlargement on one track, taken “end-on” without any specimen tilt, is shown in the inset. Four dark centers are seen around the periphery of the track; they appear to be the end-on view of the dark contrasts on either side of the trackline in the larger image with tilting. Note that the overall width of the features is 11 nm.

In Fig. 3b, the specimen was tilted slightly to move the contour of strong diffraction in the matrix (appears dark) over the three tracks. The contour is disrupted by the tracks which appear lighter than the matrix, suggesting, diffraction is less intense in the tracks. This suggests that the crystal structure has been disrupted along the track, which may be amorphous as seen in other work [8, 16]. In Fig. 3c, a track is seen near another contour in which a different set of lattice planes are diffracting. The oscillatory contrast on either side of the track is again seen, but this time the two sides are asymmetric. The diffraction contrasts seen in Figs. 3a-c suggest that detailed analysis using conditions where lattice planes with different symmetries are diffracting might reveal the crystallographic nature of the strain fields and possible lattice displacements associated with them. This contrast may be similar to that seen in GeS [16]. Note that in Fig. 3c the oscillatory contrast vanishes in the center 9 nm of the track; this appears consistent with the track center being amorphous.

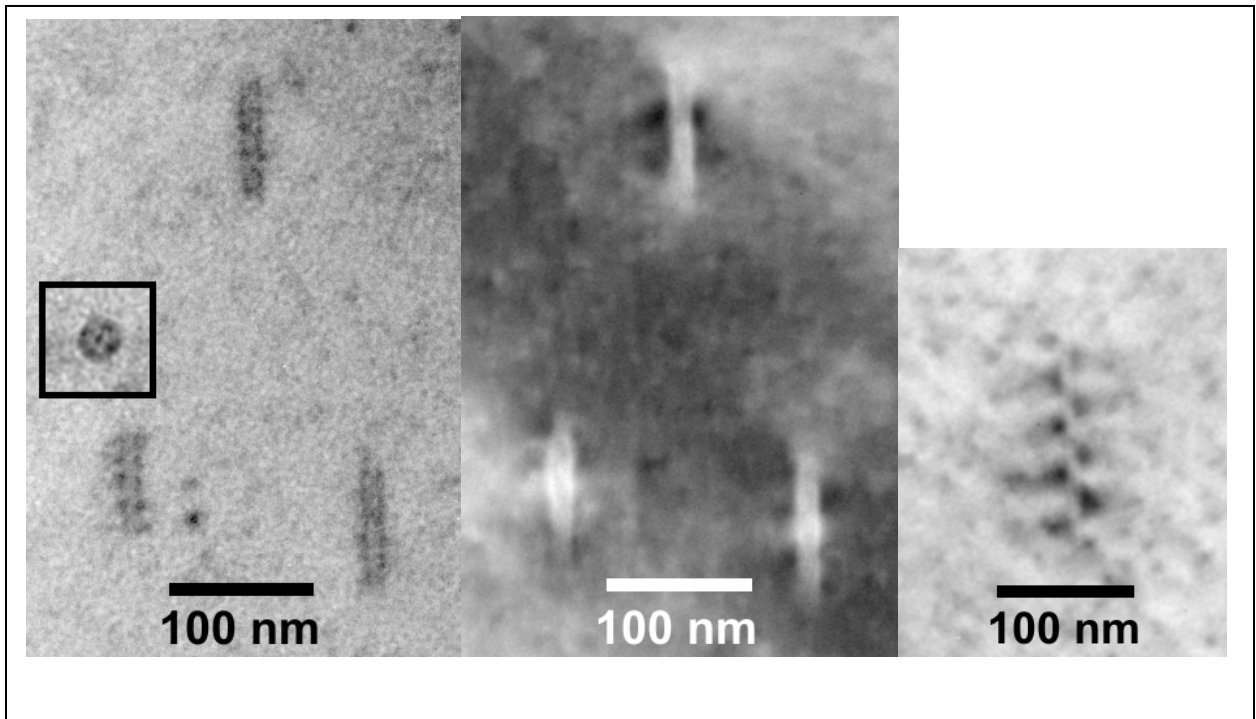


Figure 3. Tracks of 374 MeV Au in quartz, tilted 35°. a) (Left) Image of tracks near a diffraction contour, showing symmetrical diffraction contrast on either side, oscillating with depth in specimen. Insert: Track viewed end-on, showing four dark spots about the center. b) (Center) The same tracks disrupt the contrast of a diffraction contour passing through them. c) (Right) Track near a different contour, showing asymmetric oscillating contrast.

Muscovite Mica (monoclinic, $K_2O \cdot 3Al_2O_3 \cdot 6SiO_2 \cdot 2H_2O$)

Thin specimens of mica were made by gluing a 200-mesh TEM grid to the surface and peeling a layer away with the grid. In thicker areas, the tracks exhibited diffraction contrast and evidence of a non-diffracting amorphous core as in Fig. 4a. Other areas were also examined with the thinnest being 15-20 nm thick. In these areas, kinematic conditions were easily achieved and craters were detected in under-focused images with Fresnel contrast as in Fig. 4b. The contrast in this tilted image is similar to that for alumina; the mottled background is due to electron beam damage in the TEM. The crater width is 4.5 nm and the specimen thickness 18 nm. If crater depths were comparable to their widths, then a nanopore through the film might be achieved if the thickness were less than 10 nm.

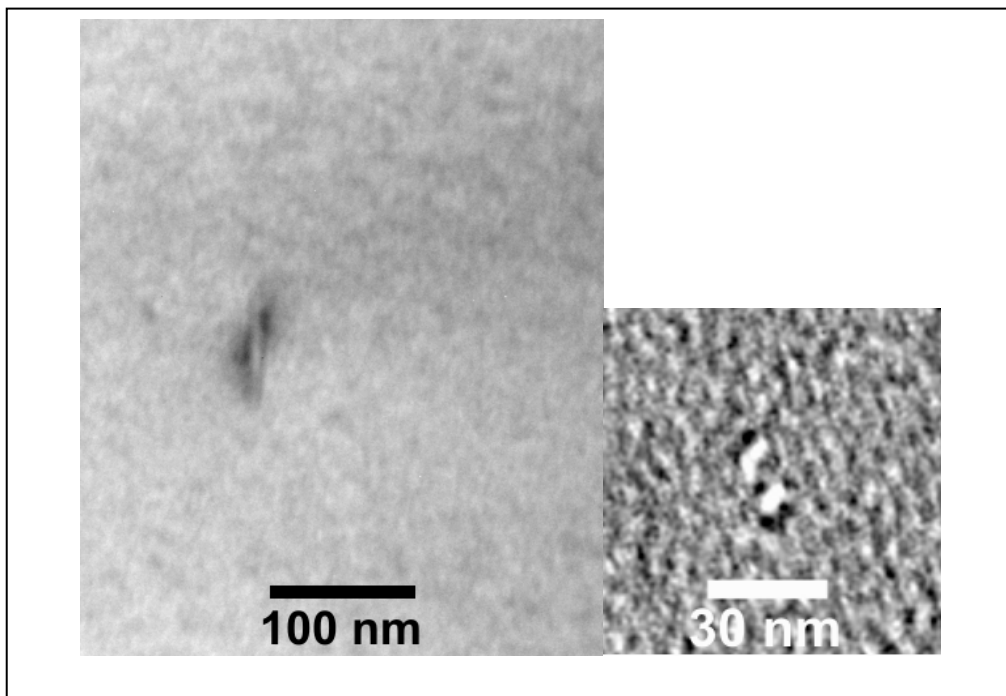


Figure 4. a) (Left) Track in mica irradiated with 374 MeV Au, showing diffraction contrast near a diffraction contour. b) (Right) Track in mica imaged with kinematic conditions and 2.5 μm under focused showing 4.5 nm-wide craters in a film 18 nm thick.

Other Thin Films

Several other thin films were also irradiated and examined with TEM: a 20 nm- SiN_x film, a pre-thinned specimen of fused quartz (SiO_2), a SiO_x film and a 3 nm-C film (advertised thickness). We have been unable to identify tracks in these materials due to the high-energy Au ions; all the films are also amorphous. We suggest that tracks

may not exhibit good contrast in an amorphous matrix; for instance, the density change between the track line and the amorphous matrix may be less than for a crystalline matrix, and the contrast therefore weaker. Although the C film is expected to be thin enough to form a nanopore by connecting craters on either side, it is plausible that at room temperature, surface diffusion could readily fill in a nanometer-scale hole and render the track undetectable.

Conclusion

Of the materials examined, thinned sapphire and cleaved mica show the most promise toward forming nanopores. The electronic energy losses for 374 MeV Au ions for these materials are calculated [15] to be 35 keV/nm and 26 keV/nm, respectively; these values are somewhat higher than those for quartz. Both showed similar surface craters, suggesting that if the films were sufficiently thin, less than ~10 nm, pores might be created. Such thicknesses appear more readily attainable by cleaving mica than by mechanical polishing and thinning by ion milling. In addition, the weak atomic bonding between the cleavage planes of mica may also be helping to promote the formation of craters.

Areas For Future Work

In addition to further work aimed at cleaving thinner mica films, recent publications in the literature indicate other materials appear promising for nanopore formation. Low energy (200 keV) Xe ions have been found to form holes in Au layers [17]. The holes are relatively large (~10 nm) and are not uniform, perhaps due to the stochastic nature of transfer of energy from the Xe ion to the Au nuclei (instead of to the electrons) at this energy. Moreover, the ion impacts examined were at high ion fluence and the craters are only 10's of nanometers apart; adjacent ion impacts may thus have distorted the holes. Notably, Au is calculated to have a very high electronic energy loss for our 374 MeV Au ions, 65 keV/nm, which is favorable for nanopore formation.

Nanopores have been formed by ~1 GeV Pb ions in the sub-micron particles composing SnO₂ powder [18]. It should thus be possible to produce nanopores in thin films of deposited SnO₂, or the transparent film used in microelectronics, indium-tin oxide or "I-T-O". The nanopores formed were evaluated with a numerical code treating the thermal spike model that examined phase changes due to rapid heating of material along the ion path. The pore radius could be accounted for with the calculated radius for vaporized material along the path. This code could thus be used to guide future choices of thin-film materials for irradiation, with selections based on how readily the material can be vaporized as well as the amount of energy calculated to be absorbed from the ions by electrons in the thin film [15].

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