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ABSTRACT

Focused ion beam (FIB) sample preparation for electron microscopy often requires large volumes of materials to be removed. Prior efforts to increase the rate of bulk material removal were mainly focused on increasing the primary ion beam current. Enhanced yield of etching at glancing ion beam incidence is known but has not found widespread use in practical applications. In this study, etching at glancing ion beam incidence was explored for its advantages in increasing the rate of bulk material removal. Anomalous enhancement of material removal at glancing angles of ion beam incidence was observed with single-raster etching in along-the-slope direction with toward-FIB direction of raster propagation. Material removal was inhibited in an away-from-FIB direction of raster propagation. The effects of glancing angles and ion doses on depth of cut and volume of removed materials were also recorded. We demonstrated that the combination of single-raster FIB etching at glancing incidence in along-the-slope direction with toward-FIB raster propagation and a "staircase" type of etching strategy holds promise for reducing the processing time for bulk material removal in FIB sample preparation applications.

I. INTRODUCTION

Advances in focused ion beam (FIB) instrumentation and control software have transformed the preparation of electron microscopy samples by FIB (Refs. 1–3) into a routine procedure. Preparing SEM cross sections and TEM lamellae by FIB often require large-scale material removal, which remains a time-consuming process. Efforts to reduce FIB processing time and increase the material removal rate have mainly revolved around increasing primary ion beam currents[4] and improving current density and profile[5] of primary ion beam, ultimately leading to emergence of plasma FIB technology.[6]

Significant progress in modeling of FIB sputtering was made over the recent years,[7–15] and effects of beam residence have been considered. Directional effects of ion beam translation over the sample surface at normal incidence were also reported,[16,17] but the associated phenomena are yet to be fully explored and directional effects of ion beam translation on FIB etching at glancing incidence[18,19] are not yet completely understood.

The general dependence of ion beam sputtering yield on the angle of incidence is well known. Specifically, the yield increases with beam incidence diverging from normal 90°, reaching a maximum at glancing angles around 15°–8° and followed by a sharp decline as the glancing angle is further reduced.[20,21] Dependency of FIB etching at glancing incidence[22] on the direction of beam translation over the sample was previously reported from experiments conducted on a dual-beam instrument with a vertical orientation of SEM and tilted FIB. Single-pass etching by ion beam at a glancing angle of 38° to the planar sample surface produced deeper cuts in down-slope and along-the-slope directions. Notably, largest apparent volume of materials was removed by beam propagating in along-the-slope direction. Etching in the up-slope direction produced the shallowest cut with the least apparent volume of removed materials.

Conventions for along-the-slope, down-slope, and up-slope directions of ion beam translation over the sample in dual-beam instrument[22] with a tilted FIB are shown in Fig. 1(a). For the purpose of further discussion, we modified the nomenclature to be independent of instrument design and applicable to any orientation of the ion beam column. Distinctions for translation of glancing-incidence beam over the sample that are agnostic of instrument design are further referred to as "away-from-FIB" column (formerly up-slope), "toward-FIB" column (formerly down-slope), and along-the-slope. Proposed nomenclature depicted in Fig. 1(b) for an instrument with vertical FIB orientation and applicable to any sample-beam system with glancing incidence.

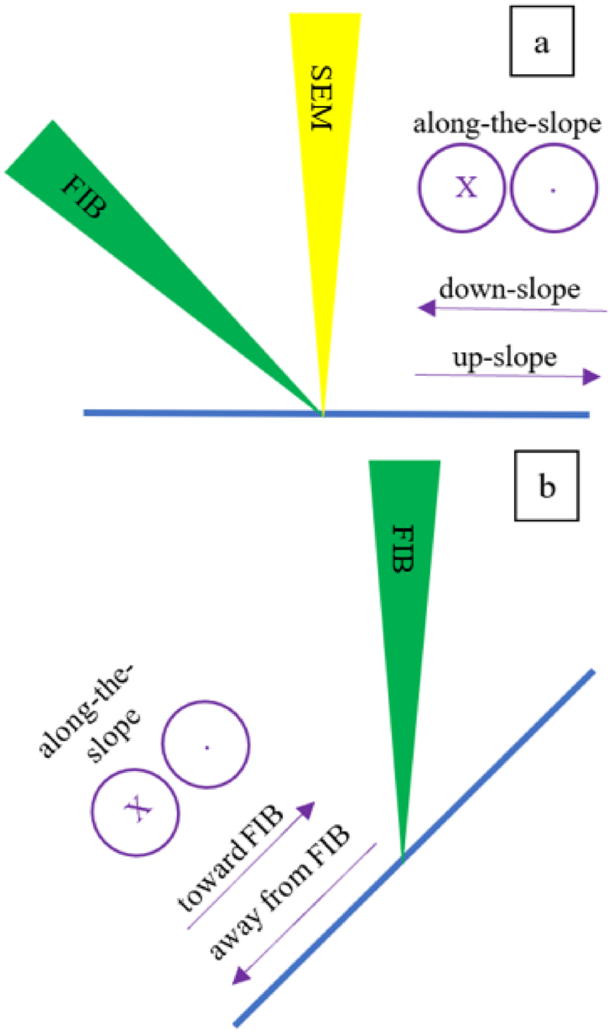

FIG. 1. Naming convention for directions of ion beam translation over the sample at glancing incidence (a) in FIB/SEM with a tilted ion beam column as described in Ref. 22, renamed to dissociate from instrument design and applied to vertical FIB orientation (b). Ion beam, electron beam, and directions of beam translation are labeled.

In the present study, ion beam was translated over the sample at normal and glancing incidences in a classical serpentine pattern. For glancing-incidence experiments, lines of the raster were oriented in along-the-slope direction. All etching experiments were done in a single-raster mode, where the entire dose of ion beam exposure is delivered in a single raster. We examined the influence of the raster propagation direction on depth of FIB cut and volume of removed materials. At glancing incidence, we observed etching by a single-raster pattern propagating in away-from-FIB direction severely inhibited, while removal of materials by a raster propagating in toward-FIB direction exhibited strong enhancement. These findings were qualitatively contextualized within bounds of our current understanding of ion-solid interactions. Finally, we introduce the concept of combining single-raster FIB etching at glancing incidence in along-the-slope direction with toward-FIB propagation and a stepped-dose "staircase" etching strategy. Preliminary results indicate that such combination shows promise for reducing the process time of bulk material removal in FIB cross sectioning, TEM lamella preparation, and site preparation for FIB tomography investigations.

II. EXPERIMENT

Rectangular, single-raster patterns were etched in a silicon wafer sample with a Micrion 2500 FIB by a 50 kV Ga^+ beam over a range of angles of ion beam incidence. The beam current operated at around 5.7 nA and the patterned areas were on the order of tens of micrometers. Figure 2 shows the series of cuts made with a constant dose of 5 nC/μm^2 over a range of glancing angles from 90° to 50° by along-the-slope single-raster propagating in both toward-FIB and away-from FIB-directions.

The effect of doses on material removal at glancing incidence was studied by etching rectangular patterns by along-the-slope single-raster propagating in toward-FIB direction with glancing 30°, 45°, 60°, and normal 90° incidence and ion doses from 1 to 10 nC/μm^2, as indicated in Fig. 3.

Sidewall profiles of cavities produced by all etching experiments were imaged at a 45° stage tilt with a 90° sample rotation. Depth measurements with correction for foreshortening due to a stage tilt were made on visible sidewalls of the cavities, and sidewall areas were calculated from pixel counts on the sidewall image.

III. RESULTS AND DISCUSSION

The effects of the raster propagation direction on removal of materials by along-the-slope single-raster etching at a glancing incidence are evident in Fig. 2, where the sidewall profiles of the cavities etched in toward-FIB direction are significantly deeper than etched by raster propagating away-from-FIB.

This apparent difference in material removal was attributed to two factors. The first is the effect of an increasing area exposed to etching by each consecutive line of ion beam raster propagating in toward-the-FIB direction. As illustrated by Fig. 4, each consecutive line of raster will increase depth of the cavity, thus increasing area exposed to grazing impact of the ion beam on the next line of the raster. As raster propagates, it exposes a larger and larger area to a grazing beam translating along the surface of the exposed face.

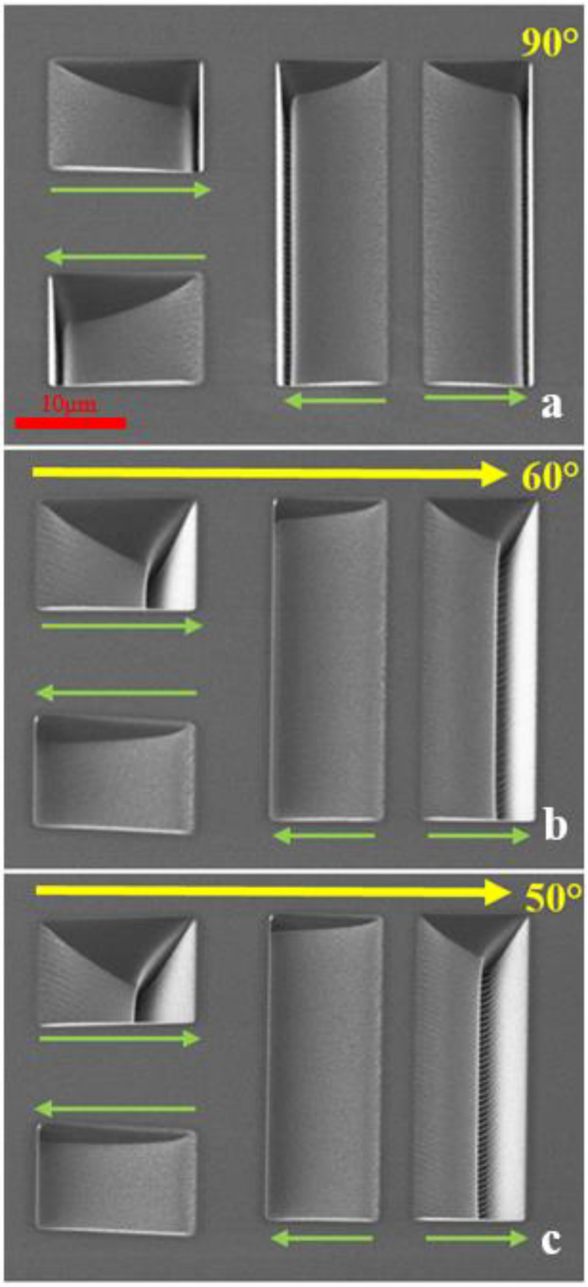

FIG. 2. Side profiles of rectangular cavities etched by along-the-slope single-raster at normal (a) 90° and over range of glancing incidences, (b) 60°, and (c) 50°. Toward-FIB direction is indicated by a long wider arrow, yellow in online version. Directions of raster propagation are shown by short narrower arrows, green in online version. All images are taken at a 45° stage tilt.

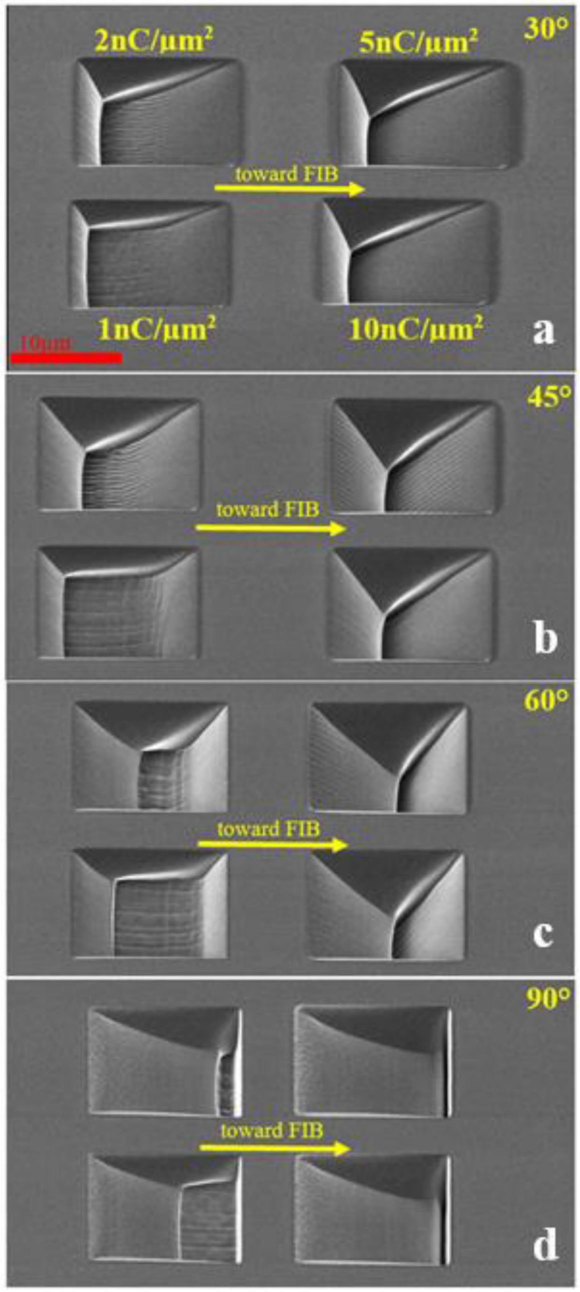

FIG. 3. Side profiles of rectangular cavities etched by along-the-slope single-raster propagating in toward-FIB direction at glancing (a) 30°, (b) 45°, (c) 60°, and normal (d) 90° incidence for a range of ion doses. All images are taken at a 45° stage tilt.

Material removal from the exposed face benefits from a higher yield of FIB spattering at grazing incidence,[8] while the long dwell times of single-raster etching facilitate complete removal of materials from the entire face exposed to each consecutive line of the raster. This consideration is supported by Fig. 3 observation of cavities etched with doses of 1 and 2 nC/μm^2 by along-the-slope single-raster pattern propagating in toward-FIB direction. As the area of the face exposed for FIB etching by each consecutive line

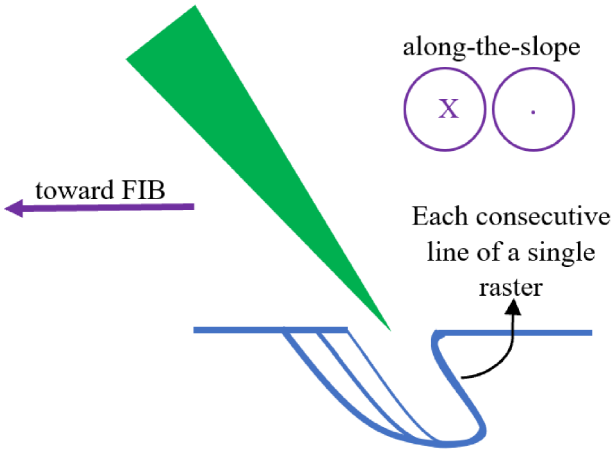

FIG. 4. Increased area of face exposed to ion beam etching at grazing incidence by each consecutive line of raster propagating in toward-FIB direction.

was increasing with propagation of the raster, depth of the etched cavity ceased increasing at the point where duration of the beam exposure (dwell) became insufficient to remove materials from the entire depth of the exposed face. It is also supported by the fact that multiple-raster etching with short dwell times exhibits only minimal, if any at all, enhancement of etching by along-the-slope raster with toward-FIB propagation.[23]

The second factor contributing to enhancement of sputtering by raster propagating in toward-FIB direction and inhibition in away-from-FIB direction is attributed to redeposition. The distribution of materials ejected by FIB sputtering has been approximated by a cosine function.[21,24] It has also been shown that for increased angles of incidence, a larger portion of this distribution is directed toward the sample.[21] The distribution of sputtering byproducts directly benefits removal of the material ejected from face exposed

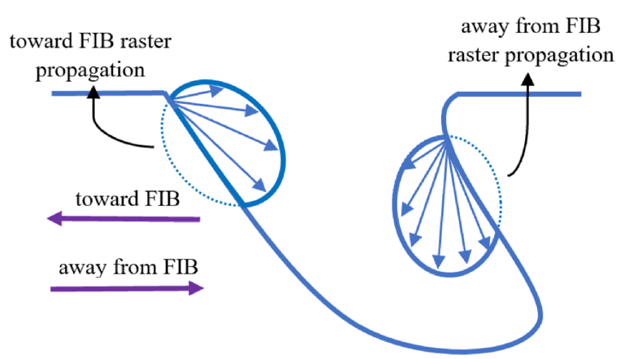

FIG. 5. Ejection profiles for the material sputtered by along-the-slope raster propagating in toward-FIB and away-from-FIB directions. The material sputtered from the face produced by raster propagating in toward-FIB direction is likely to escape. The material sputtered from the face produced by raster propagating in away-from-FIB direction is likely to be redeposited within the cavity.

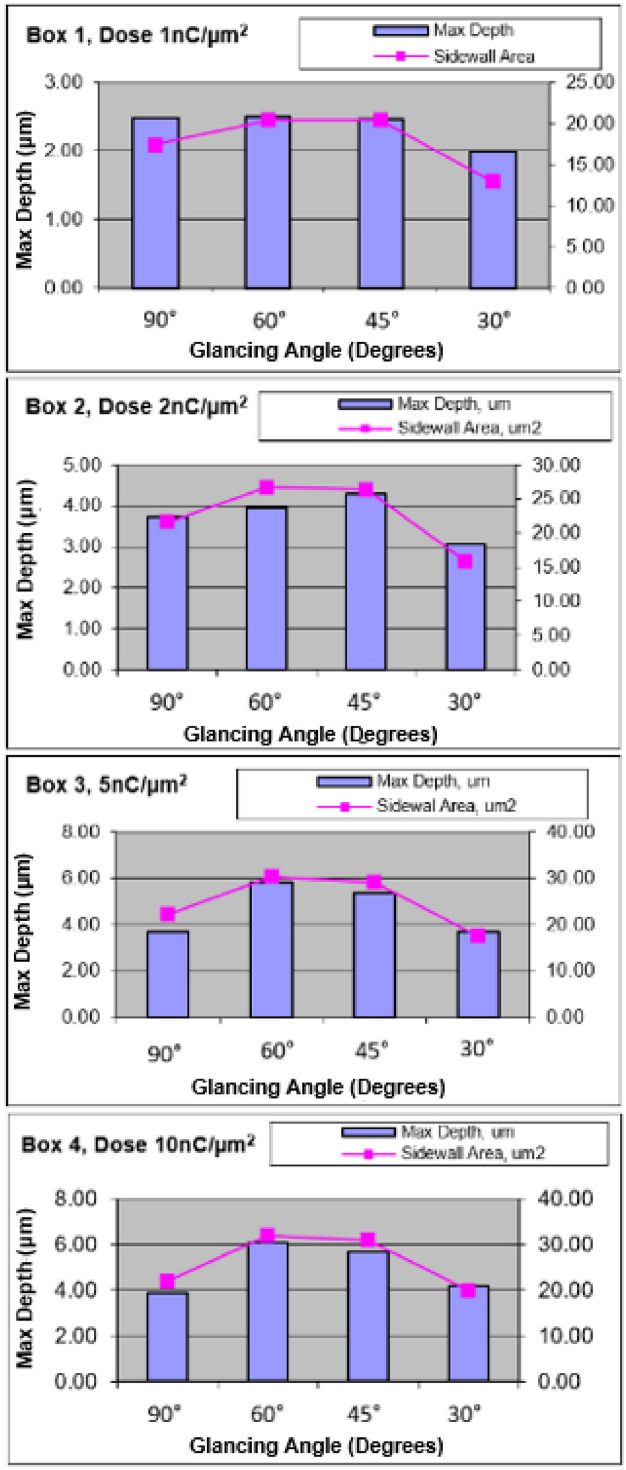

FIG. 6. Max depth of the cavity and the sidewall area of trenches etched by along-the-slope single-raster propagating in toward-FIB direction as a function of glancing angle.

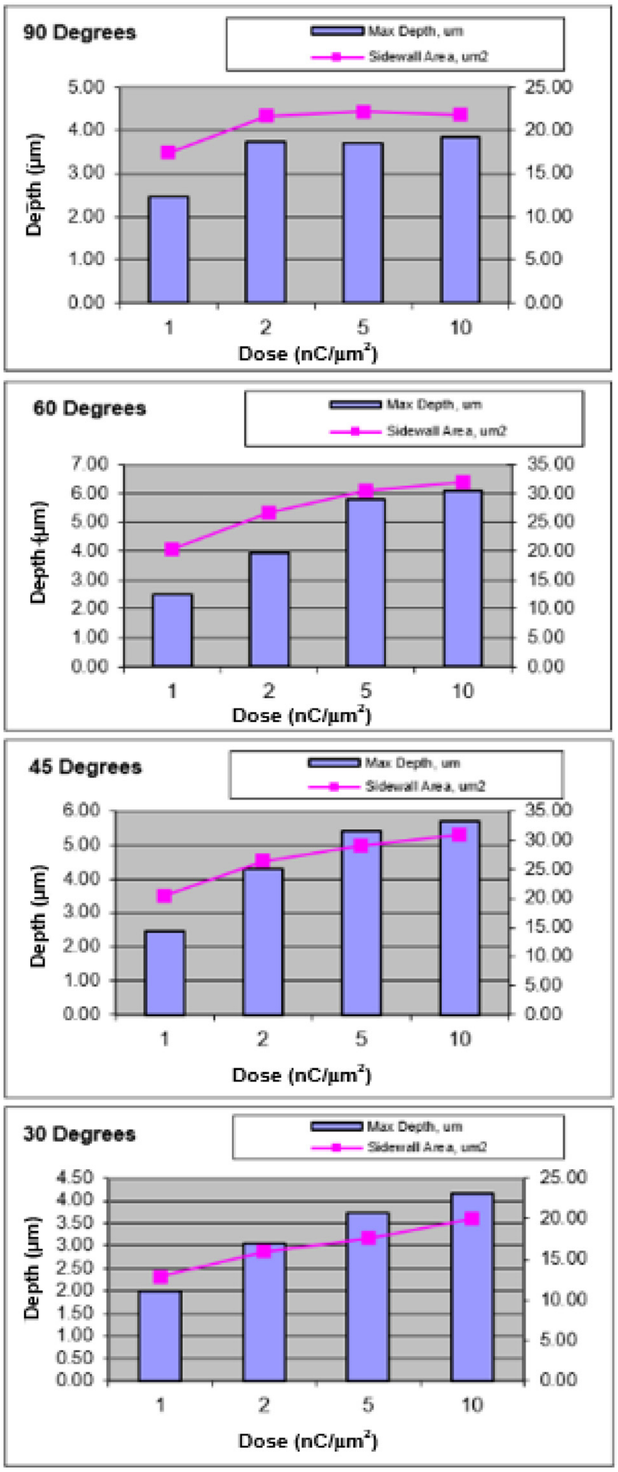

FIG. 7. Max depth of the cavity and the sidewall area of trenches etched by along-the-slope single-raster propagating in toward-FIB direction as a function of the ion dose for normal incidence and as a function of the glancing angle.

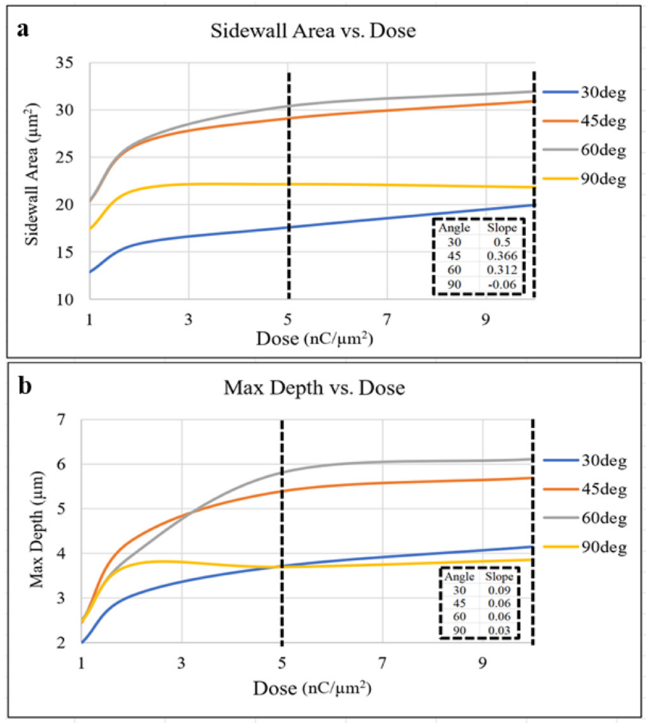

FIG. 8. Measured sidewall area (a) and the maximum depth (b) for normal and glancing-incidence angles as a function of dose. The slope calculated for a linear fit applied to the portion of the graph indicated by dashed lines in the dose range of 5 and 10 nC/um^2.

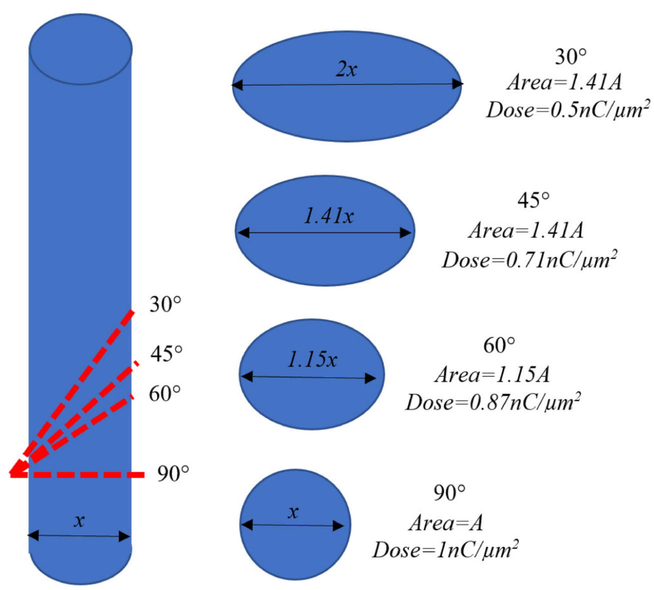

FIG. 9. Projection of ion beam spot projection on the sample and geometrically approximated major axis dimensions as a function of the glancing angle.

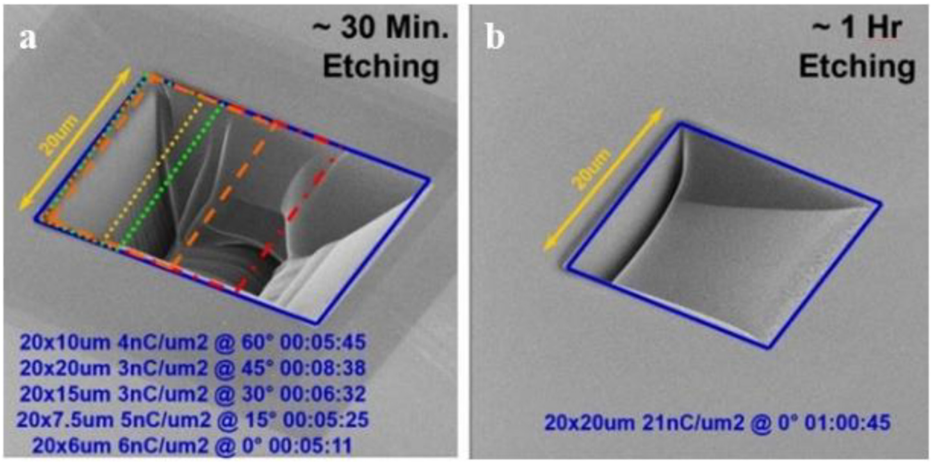

FIG. 10. Results of staircase milling with a varied angle of incidence and a dose of single-raster patterns with a reduced area propagating in toward-FIB direction (a) compared to single-raster milling at a normal angle of incidence (b) with an overall dose equivalent to the last step of the staircase.

by each raster propagating in toward-FIB direction (Fig. 5). The material sputtered from the face exposed by along-the-slope raster propagating in away-from-FIB direction is mostly redeposited, resulting in apparent inhibition of the etching process.

The observed effect, from Fig. 3, of the glancing angle on the depth of cavities etched by along-the-slope raster propagating in toward-FIB direction with ion doses of 1, 2, 5, and 10 nC/μm^2 is presented quantitatively in Fig. 6. The effect of doses for a normal incidence of 90° and glancing angles of 60°, 45°, and 30° is presented in Fig. 7. The increase in the depth of the cavity with an increase in the dose was observed for etching at all glancing angles, although to a different extent. Depth of cavities produced by normal incidence, however, leveled off within the range of doses between 2 and 10 nC/μm^2. This apparent limitation of depth for cavities produced at normal incidence is attributed to restricted ejection of the sputtered material from the cavity. A small increase in depth and the sidewall area of the cavity for a dosage increase from 5 to 10 nC/μm^2 indicates that saturation of the maximum depth was most likely just outside the experimental dose range. Figure 7 indicates that glancing angles of 60°–45° facilitate the most efficient material removal for a given dose.

Although etching at a glancing angle of 30° is expected to have enhanced sputtering yield[9] and the geometry favoring ejection of the material, it appears to perform similarly to a normal incidence. Further details of etching by along-the-slope single-raster with toward-FIB propagation at normal and glancing incidence is presented in Fig. 8. A linear fit applied to the dashed area of graphs and the respective slopes is calculated to indicate the rate of the increase in the depth and volume of the removed material. It is apparent that although the overall depth and the sidewall area of cavities etched at a glancing angle of 30° are lower than at 45° or 60°, the rate of their increase as a function of dose is higher.

A possible explanation for the observed behavior of etching at a glancing angle of 30° is associated with a reduced density of ion flux within a focused ion beam spot projected on the sample, as illustrated by Fig. 9. The enlarged area of the spot projected on the sample surface by a given focused ion beam current results in a reduced current density, which adversely affects the material removal rate. However, the rate of etching at a glancing angle of 30° increases faster than with the other tested angles, as might be expected from sputtering yield and geometry considerations.

To demonstrate the practical utility of the single-raster etching in along-the-slope direction with toward-FIB propagation, a test of bulk material removal was performed in combination with a common FIB technique known as a "staircase pattern." With a simple staircase pattern, historically used with multiple-raster milling, the dose of ion beam exposure is incrementally increased toward the desired location of cross section or TEM lamella.[25] We used the same strategy but also increased the glancing angle for each consecutive step of the staircase pattern. With such a combined technique, the widest and lowest-dose "step" of the staircase is cut at a smallest (most grazing) angle of ion beam incidence, while the narrowest and highest-dose "step" of final cross section is normal to the sample surface. The face of the resulting cut is orthogonal to the sample surface, as expected for postpreparation imaging of cross sections and/or TEM lamella lift-out. The incidence angles and the corresponding width of steps are indicated in Fig. 10, along with times for etching each step and the entire pattern. Such combination of a staircase etching strategy with ion beam incidence at a varied glancing angle not only removed much larger volume of materials, but accomplished it in about half of the time needed for delivery of the dose equivalent to last step one-step single-raster cut at orthogonal incidence.

IV. CONCLUSION

An anomalous enhancement of material removal by along-the-slope single-raster etching at glancing beam incidence was observed with toward-FIB direction of raster propagation. Raster propagation in away-from-FIB direction resulted in inhibition of material removal. The observed enhancement was attributed to an increase in the area of the cross-sectional face exposed to grazing ion beam sputtering by each consecutive line of the raster, with material removal facilitated by long dwell times of the single-raster pattern and geometry favorable for ejection of sputtered materials in the case of toward-FIB raster propagation. The material ejected by along-the-slope raster with toward-FIB propagation is mostly directed away from the cavity, resulting in enhanced etching. The material ejected by along-the-slope raster propagating in the away-from-FIB direction is mostly projected toward the sample and, therefore, redeposited within the cavity, resulting in apparent inhibition of the etching. For a range of evaluated ion doses, the highest enhancement of material removal was observed for glancing angles within the range of 60°–45°. The observed relationship between the ion dose and the depth of etch for a range of incident angles is suggesting a limit for the maximum achievable depth of cut for each incident angle. A practical application of the observed phenomena was illustrated via proposed combination of etching by along-the-slope single-raster with toward the FIB propagation at a varied incident angle with a "staircase" etching strategy. Apparent enhancement of the material removal with the proposed strategy in comparison with classical etching at normal incidence shows promise for increasing efficiency and, therefore, reducing time for bulk material removal in practical applications of FIB cross sectioning, TEM lamella preparation, and site preparation for FIB tomography investigations.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

REFERENCES

[1] R. Langford and A. Petford-Long, J. Vac. Sci. Technol. A **19**, 2186 (2001).
[2] M. Vasile, Z. Niu, R. Nassar, W. Zhang, and S. Liu, J. Vac. Sci. Technol. B **15**, 2350 (1997).
[3] T. Ishitani and T. Yaguchi, Microsc. Res. Tech. **35**, 320 (1996).
[4] I. Schulmeyer, T. Volkenandt, F. Perez-Willard, M. Kienle, R. Hill, and J. Marshman, see https://www.zeiss.com/content/dam/smt/downloads/products_and_solutions/prosesscontroll/en_wp_focused_ion_beam_column.pdf for "ZEISS Focused Ion Beam Column: Enabling Precision and Long-Term Stability for Cutting Edge Crossbeam Applications," March 2017; accessed May 2020.
[5] ThermoFisher Scientific, see https://www.fei.com/document/Helios-G4-UX-materials-science-datasheet/#gsc.tab=0 for "Helios G4 UX DualBeam for Materials Science Datasheet," 2019; accessed May 2020.
[6] ThermoFisher Scientific, see https://www.thermofisher.com/us/en/home/electron-microscopy/products/dualbeam-fib-sem-microscopes/helios-g4-pfib-dualbeam.html#documents for "Helios G4 PFIB DualBeam," 2020; accessed May 2020.
[7] K. P. Muller, U. Weigmann, and H. Burgause, Microelectron. Eng. **5**, 481 (1986).
[8] C. Ebm and G. Hobler, *Materials Research Society Symposium*, Boston, January 2009 (Material Research Society, Pittsburgh, PA, 2009), Vol. 1181.
[9] W. Boxleitner and G. Hobler, Nucl. Instrum. Meth. B **180**, 125 (2001).
[10] A. Svinstov, S. Zatisev, G. Lalev, S. Dimov, V. Velkova, and H. Hirshy, Microelectron. Eng. **86**, 544 (2009).
[11] C. Ebm, G. Hobler, S. Waid, and H. D. Wanzenboeck, J. Vac. Sci. Technol. B **29**, 011031 (2011).
[12] S. Lindsey and G. Hobler, Nucl. Instrum. Meth. B **282**, 12 (2012).
[13] H. B. Kim, G. Hobler, A. Stelger, A. Lugstein, and E. Bertagnolli, Nanotechnology **18**, 245303 (2007).
[14] H. B. Kim, G. Hobler, A. Stelger, A. Lugstein, and E. Bertagnolli, Opt. Express **15**, 9444 (2007).
[15] S. Lindsey, S. Waid, G. Hobler, H. D. Wanzenboeck, and E. Bertagnolli, Nucl. Instrum. Meth. B **341**, 77 (2014).
[16] H. S. Yoon, C. S. Kim, H. T. Lee, and S. H. Ahn, Vacuum **143**, 40 (2017).
[17] C. Scheffler, R. Livengood, H. Prakasam, M. Phaneuf, and K. Lagarec, *ISTFA 2016*, Fort Worth, TX, November 6–10, 2016 (ASM International, Cleveland, OH, 2016).
[18] V. Ray, *FIB User Group Meeting at ISTFA 2013*, San Jose, CA, November 3–7, 2013).
[19] J. Hazekamp, S. Doherty, A. Elsaesser, C. Barnes, B. O'Hagan, G. McKerr, and C. Howard, J. Microsc. **242**, 104 (2010).
[20] X. Xu, A. D. Ratta, J. Sosonkina, and J. Melgailnis, J. Vac. Sci. Technol. B **10**, 2675 (1992).
[21] T. Ishitani, K. Umemura, T. Ohnishi, T. Yaguchi, and T. Kamino, J. Electron. Microsc. **53**, 443 (2004).
[22] L. A. Giannuzzi, P. Anzalone, and D. Phifer, *NSTI-Nanotech*, May 8–12, 2005, Anaheim, CA, NSTI Tech Connect Briefs, Vol. 2 (NSTI, Austin, TX, 2005).
[23] O. Zhao, V. Ray, W. Chiou, K. Zaitsev, and A. Zaitsev, *FIBSEM User Group Meeting*, Laurel, MD, February 25, 2014, see https://www.researchgate.net/publication/342520928_Glancing_Angle_of_Incidence_Method_for_Bulk_Material_Removal_in_Cross-sectioning_and_TEM_Sample_Preparation_by_Ion_Beam.
[24] H. B. Kim, G. Hobler, A. Lugstein, and E. Bertagnolli, J. Micromech. Microeng. **17**, 1178 (2007).
[25] L. Giannuzi and F. Stevie, Micron **30**, 197 (1992).