

Direct etching of SiO₂ and Al₂O₃ by 900-keV gold ions

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1 Introduction

The use of high-energy ion beams in micromachining has grown significantly in the last two decades thanks to the development of focusing systems capable of delivering beam spot sizes down to the nanometer scale. In general, such ion-based techniques¹ employ protons of a few MeV to irradiate resist materials² deposited over a substrate in order to produce microstructures. Due to the nature of the proton-matter interaction, setups consisting of energy-tunable accelerators and vacuum chambers with manipulators attached to the target holder allow a maskless manufacturing of complex microstructures with variable depth and nonprismatic shapes.³ Metallic structures of hundreds of nanometers can be obtained by coupling the proton beam writing technique with subsequent electroplating of the structure.⁴ In addition to advanced technological applications for microelectromechanical systems (MEMS) like nanowire production,⁵ simple grid patterns and scaffold microstructures have found general acceptance in other fields like biology, where they have been extensively used in studies of cell migration and mobility.^{6,7}

Another way to produce simple patterns in a wide variety of materials is through the use of an ion-sputtering technique, where a swift ion beam impinges on the material covered with a mask that ultimately will generate the same pattern of the mask in the material itself. Although any ion

Abstract. We report the direct etching of Al₂O₃ and SiO₂ using 900-keV Au⁺ ions. 2000-mesh Cu grids were employed as masks using two different configurations: (1) the Cu mesh was placed on top of each insulator separately and independent irradiations were performed, and (2) the Al₂O₃ and SiO₂ substrates were positioned in an edge-to-edge configuration with a single Cu grid providing a common mask to both insulators. Scanning electron microscopy (SEM) analysis revealed quite different patterns resulting from the two irradiation configurations. While the irradiation using individual masks resulted in mirror-image patterns of the Cu mask in the substrates, the use of a common mask led to single line structures approximately normal to the edges of the substrates. The role of charge buildup and sputtering in relation to relative dielectric properties of the substrates and close proximity of the samples during irradiation is discussed. © 2009 Society of Photo-Optical Instrumentation Engineers. [DOI: 10.1117/1.3082185]

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could be used, heavy ions are more desirable due to higher sputtering yields, which significantly decreases the time needed to generate such patterns. Direct sputter etching has the advantage of producing simple patterns with relatively large areas in a single step, eliminating the requirement for subsequent chemical etching of the substrate, and the depth of the structures can be easily controlled with the ion fluence.

While direct sputter etching on metals has been proved to be straightforward,⁸ the same is not true for insulating materials. Indeed, the use of charged particle beams leads to interesting effects due to the charge buildup process in the samples, which eventually generates local potentials of the order of kilovolts.⁹ Secondary electrons emitted in the process are subjected to high accelerations, giving rise to a substantial bremsstrahlung radiation. Also, other effects such as ion migration¹⁰ and residual activity¹¹ have been reported in the literature. The charge buildup in insulators can be substantially reduced by coating the samples with a layer of conducting material such as amorphous carbon¹² and gold.^{13,14} The suppression is not complete, however, since such a layer does not prevent the charging of deeper portions of the substrate.¹² Moreover, the conducting layer has to have an optimum thickness, since too-thin layers may lead to an increase of the charge buildup.^{13,14}

Among several insulators, quartz (SiO₂) and sapphire (Al₂O₃) are the most popular ones due to their relative high dielectric constant and wide range of applications like microelectronics and optical waveguides.¹⁵ In this work, the feasibility of generating simple microstructures on those insulators through the use of direct sputtering with high-energy heavy ions is discussed.

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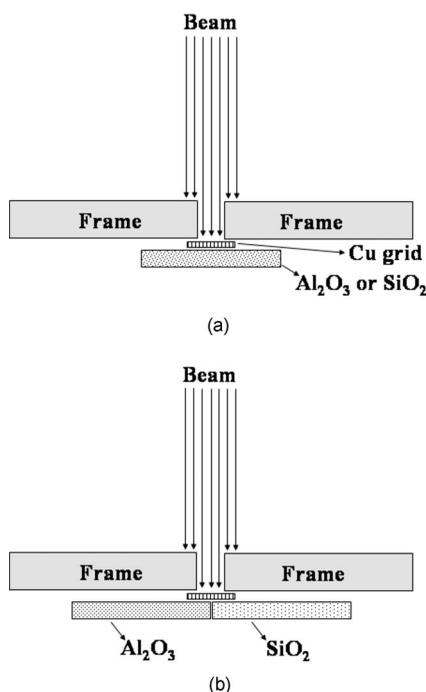


Fig. 1 Scheme of the experimental setup used for the irradiation of Al₂O₃ and SiO₂ with 1-MeV Au⁺ ions. The beam was collimated by a 1-mm-thick aluminum frame before reaching the 2000-mesh Cu grid. The top panel (1a) depicts the C_{1a} configuration, where the dielectrics were irradiated in independent measurements, while the bottom panel (1b) depicts the C_{1b} configuration, where both dielectrics were irradiated simultaneously. See text for further explanation.

2 Experiment

The experiments were carried out at the Louisiana Accelerator Center where a 1.7-megavolt tandem (NEC 5SDH-2) Pelletron accelerator delivered 900-keV Au⁺ ions with typical currents of 1.0 μ A and beam spot size of about 0.1 cm². Inside the implant chamber an electrically isolated Faraday cage, biased to +45 volts, surrounded the isolated target holder so that summing the charges collected on the target holder and the Faraday cage provided an accurate determination of the ion fluence. The irradiation fluences were in the range of 10¹⁷/cm², and all irradiations were performed at room temperature, 24 °C.

The targets consisted of commercially available polished sapphire and quartz 2.5-cm-diam disks with thicknesses of 1.1 mm and 2.3 mm, respectively. Copper Scanning electron microscopy (SEM) calibration grids (2000 mesh) with 3.05 mm diameter and 18 μ m thick and having grid bars 5 μ m wide were used as masks to generate periodic square patterns of 7.5 \times 7.5 μ m². The grids were attached with epoxy to cover 2-mm-diam apertures in a 2 mm \times 5 cm \times 10 cm aluminum sheet, and the substrates were secured using SEM carbon tape immediately behind these grids for the irradiations.

Two independent configurations were employed for the irradiations. In the first configuration (hereafter, referred to as C_{1a}), sapphire or quartz was each irradiated individually with Au⁺ ions [Fig. 1(a)] by placing the Cu grid mask at the center of each disk. In the second configuration (C_{1b}), the sapphire and quartz disks were placed edge-to-edge, and a

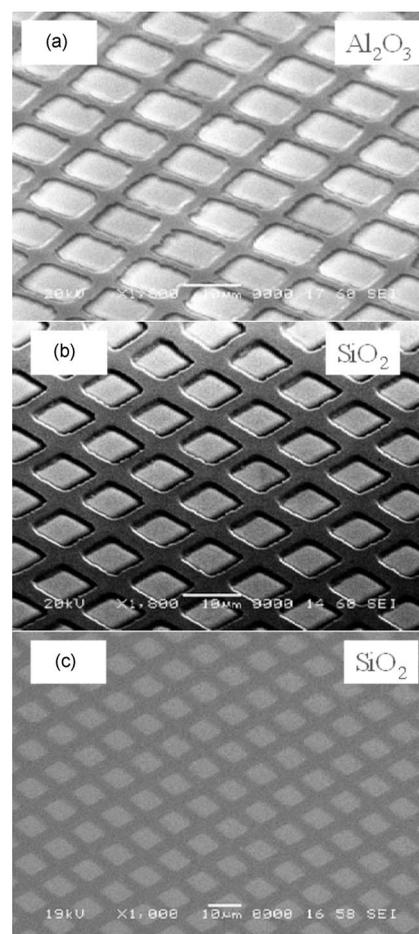


Fig. 2 SEM images obtained at a tilt angle of 60 deg after Au⁺ sputter etching of Al₂O₃ (a) and SiO₂ (b) in the C_{1a} configuration with an ion fluence of 8 \times 10¹⁷ ions/cm². The bottom panel (c) shows the structure obtained for SiO₂ at 2 \times 10¹⁷ ions/cm².

single Cu grid placed across the intersection of the edges was used as a mask for both [Fig. 1(b)], thus allowing a simultaneous irradiation for both substrates. The Cu grid masks were used to allow the fabrication of an easily observable microstructure and were attached to a grounded aluminum sheet placed immediately in front of the substrates. After the irradiations, the integrity of the Cu grids was checked with an optical microscope, and the targets were then analyzed using an SEM model JEOL 6460-LV. The total area able to be irradiated in a single exposure without scanning is approximately 25 mm²; however, if raster scanning and appropriate masks are utilized, areas as large as 500 mm² could be etched in a single exposure.

3 Results and Discussion

Figure 2 shows SEM images obtained for sapphire [Fig. 2(a)] and quartz [Fig. 2(b)] after irradiation with Au⁺ ions with a fluence of 8 \times 10¹⁷ ions/cm² according to the experimental configuration C_{1a}. As can be seen, the grid patterns were fully reproduced in the substrates, just as those obtained from previous Al, Si, Cu, and Ag,⁸ thus confirming the feasibility of obtaining large area patterns from direct ion sputtering. In Fig. 2(c), bottom panel, the results obtained with a fluence of 2 \times 10¹⁷ ions/cm² for quartz are

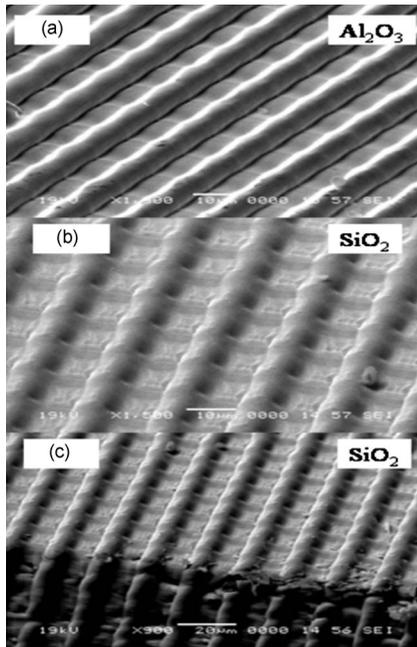


Fig. 3 SEM images obtained at a tilt angle of 60 deg after Au+ etching of Al₂O₃ (a) and SiO₂ (b) in the C_{1b} configuration. The bottom panel (c) shows the structures produced in the edge of the SiO₂.

shown. The much shallower structures are a direct consequence of the lower ion fluences employed in these experiments.

Figure 3 shows the results obtained for both substrates when sharing a single Cu grid during irradiation according to the scheme shown in Fig. 1(b), corresponding to the configuration C_{1b}. The patterns generated for both dielectrics, shown in Figs. 3(a) and 3(b) for sapphire and quartz, respectively, do not reproduce the Cu grid pattern. Instead, parallel channels were created whose orientation follows the direction of the contact point between the sapphire and quartz and extended to the edges of the substrates [see Fig. 3(c)].

No direct measurements of the depth of the structures were performed; however, from the SEM images in Figs. 2 and 3, the depth of the structures is estimated to be 0.5 to 1.0 μm for the center-etched structures and 2 to 3 μm for the edge-etched structures. Based on the estimated depth of the microstructures and with a recorded etch time of 3 h, the etch rate is approximately 5 nm/min for the center-etched structures and 15 nm/min for the edge-etched structures. It is believed that this etch rate can be increased considerably by using a higher current density.

Several factors might be considered in order to understand the results shown in Fig. 3. First of all, the discharge of electrical charge in the sample can be understood as a two-step process,^{9,11} with the first step occurring between the ion end-of-range position (see Table 1) and the surface of the substrate. The second discharge step takes place along the surface of the substrate where the charge flows to the aluminum mounting plate, which is at ground potential. For configuration C_{1a}, the discharge appears to be directed to the nearest copper grid bar without any preferential di-

Table 1 Some physical properties of Al₂O₃ and SiO₂. The energy loss, the ranges, and the sputtering yields were obtained through the SRIM code.¹⁶

Dielectric	Energy loss (eV/10 ⁻¹⁰ m)	Range (10 ⁻¹⁰ m)	Sputtering yield (atoms/ion)
Al ₂ O ₃	773	1458±272	O: 7.9 Al: 3.98
SiO ₂	443	2510±470	O: 4.52 Si: 1.35

rection. For configuration C_{1b}, however, it is possible that a potential difference between the insulators builds up. Indeed, although sapphire and quartz have dielectric strengths of the same order of magnitude (\sim hundreds of KV/cm), even small relative differences can lead to distinct charge buildup processes. Since this is a dynamic system, it is expected that the discharge mechanism for both insulators happens at different times, leading to a potential difference between them. The electric field would have a stronger influence in the second step of the discharge process, diverting charge carriers to a specific direction, which could influence the ion etching process. Another factor is that the insulators have different sputtering yields (see Table 1), including different yields for the constituents of the compound material. For quartz, the ratio between the oxygen and silicon sputtering yields is 3.3, while for sapphire, the ratio between the oxygen and the aluminum sputtering yields is 2.0. On average, there is a surplus of both silicon and aluminum during the sputtering process, which could change the charge balance in both insulators and therefore have an influence on the final etching process.

It is possible that the edge effect could be mitigated significantly by sputter coating the substrate with gold prior to sputter etching with high-energy Au ions, since this step would minimize the potential differences caused by charge buildup. Although determining the root cause of the channeling anisotropy in the sputtering effect for the edge-irradiated substrates was not the primary focus of this work, future experiments that include applying a conductive coating to the substrates prior to irradiation should help to elucidate the phenomenon.

4 Concluding Remarks

In this work, it was demonstrated that simple pattern structures can be created on insulators through direct sputter etching using high-energy heavy ions. The depth of the microstructures can be achieved by choosing the correct ion energy and fluences.

The insulating properties of dielectrics can significantly affect the final etching result, and in this work, it was specifically shown that for at least the particular geometry used here, parameters like dielectric strength and sputtering yield may play an important role in the final microstructures obtained through the sputter etching process.

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References

1. S. V. Springham, T. Osipowicz, J. L. Sanchez, L. H. Gan, and F. Watt, "Micromachining using deep ion beam lithography," *Nucl. Instrum. Methods Phys. Res. B* **130**, 155–159 (1997).
2. J. A. van Kan, J. L. Sanchez, B. Xu, T. Osipowicz, and F. Watt, "Resist materials for proton micromachining," *Nucl. Instrum. Methods Phys. Res. B* **158**, 179–184 (1999).
3. F. Watt, J. A. van Kan, and T. Osipowicz, "Three-dimensional micro-fabrication using maskless irradiation with MeV ion beams: proton-beam micromachining," *MRS Bull.*, 33–38 (2000).
4. K. Ansari, J. A. van Kan, A. A. Bettioli, and F. Watt, "Fabrication of high aspect ratio 100 nm metallic stamps for nanoimprint lithography using proton beam writing," *Appl. Phys. Lett.* **85**(3), 476–478 (2004).
5. J. A. van Kan, F. Zhang, S. Y. Chiam, T. Osipowicz, and F. Watt, "Proton beam writing: a platform technology for nanowire production," *Microsyst. Technol.* (2008; online).
6. J. Mai, C. Sun, S. Li, and X. Zhang, "A microfabricated platform probing cytoskeleton dynamics using multidirectional topographical cues," *Biomed. Microdevices* **9**, 523–531 (2007).
7. F. Zhang, F. Sun, J. A. van Kan, P. G. Shao, Z. Zheng, R. W. Ge, and F. Watt, "Measurement of cell motility on proton beam micromachined 3D scaffolds," *Nucl. Instrum. Methods Phys. Res. B* **231**, 413–418 (2005).
8. G. A. Glass, J. F. Dias, A. D. Dymnikov, and B. Rout, "900 keV gold ion sputter etching of silicon and metals," *Nucl. Instrum. Methods Phys. Res. B* **266**, 3330–3331 (2008).
9. J. F. Dias, A. Bulla, and M. L. Yoneama, "Charging effects in thick insulating samples," *Nucl. Instrum. Methods Phys. Res. B* **189**, 72–76 (2002).
10. J. Cazaux, "Some considerations on the electric field induced in insulators by electron bombardment," *J. Appl. Phys.* **59**, 1418–1430 (1986).
11. S. M. Shubeita, C. E. I. Dos Santos, J. L. R. Filho, R. Giulian, L. Meira, P. R. Silva, L. Amaral, J. F. Dias, and M. L. Yoneama, "Residual activity induced by ion bombardment on insulating samples," *Nucl. Instrum. Methods Phys. Res. B* **240**, 297–302 (2005).
12. M. A. Stevens-Kalceff and K. J. Levick, "The assessment of microscopic charging effects induced by focused electron and ion beam irradiation of dielectrics," *Microsc. Res. Tech.* **70**, 195–204 (2007).
13. J. Kim, W. Hong, H. J. Woo, and C. H. Eum, "Charge buildup effect during ion beam irradiation of an insulator and its suppression by deposition of a thin metal film," *J. Korean Phys. Soc.* **43**, 582–584 (2003).
14. P. Trouslard, S. Pellegrino, and L. Beck, "Charging effect during ion beam analysis: comparison between aluminum and glass substrates," *Nucl. Instrum. Methods Phys. Res. B* **240**, 381–385 (2005).
15. A. L. Stepanov, C. Marques, E. Alaves, R. C. da Silva, M. R. Silva, R. A. Ganeev, A. I. Ryasnyansky, and T. Usmanov, "Ion synthesis and optical properties of gold nanoparticles in an Al₂O₃ matrix," *Tech. Phys.* **51**, 1474–1481 (2006).
16. J. F. Ziegler, M. D. Ziegler, and J. P. Biersack, "Stopping and Range of Ions in Matter," www.srim.org, 2006.

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