WSi_2/Si Multilayer Sectioning by Reactive Ion Etching for Multilayer Laue Lens Fabrication

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Introduction

Reactive ion etching (RIE) has been employed in a wide range of fields such as semiconductor fabrication, MEMS (microelectromechanical systems), and refractive x-ray optics with a large investment put towards the development of deep RIE. Due to the intrinsic differing chemistries related to reactivity, ion bombardment, and passivation of materials, the development of recipes for new materials or material systems can require intense effort and resources. For silicon in particular, methods have been developed to provide reliable anisotropic profiles with good dimensional control and high aspect ratios[1,2,3], high etch rates, and excellent material to mask etch selectivity.

A multilayer Laue lens[4] is an x-ray focusing optic, which is produced by depositing many layers of two materials with differing electron density in a particular stacking sequence where the each layer in the stack satisfies the Fresnel zone plate law. When this stack is sectioned to allow side-illumination with radiation, the diffracted exiting radiation will constructively interfere at the focal point. Since the first MLLs were developed at Argonne in the USA in 2006[4], there have been published reports of MLL development efforts in Japan[5], and, very recently, also in Germany[6]. The traditional technique for sectioning multilayer Laue lens (MLL) involves mechanical sectioning and polishing[7], which is labor intensive and can induce delamination or structure damage and thereby reduce yield. If a non-mechanical technique can be used to section MLL, it may be possible to greatly shorten the fabrication cycle, create more usable optics from the same amount of deposition substrate, and perhaps develop more advanced structures to provide greater stability or flexibility. Plasma etching of high aspect-ratio multilayer structures will also expand the scope for other types of optics fabrication (such as gratings, zone plates, and so-on). However, well-performing reactive ion etching recipes have been developed for only a small number of materials, and even less recipes exist for concurrent etching of more than one element so a fully material specific process needs to be developed. In this paper, sectioning of WSi_2/Si multilayers for MLL fabrication using fluorinated gases is investigated. The main goals were to demonstrate the feasibility of this technique, achievement of high anisotropy, adequate sidewall roughness control and high etching rates. We note that this development for MLL sidewalls should be distinguished from work on improving aspect ratios in traditional Fresnel zone plates. Aspect ratios for MLL sidewalls are not similarly constrained.

Experiment

The multilayers were prepared by DC magnetron sputtering under high vacuum onto Si (100) wafers. Layers were deposited by raster-scanning the substrate over stationary targets with figured apertures at well-defined velocities using a rotary deposition system in the NSLS-II deposition laboratory using a method that has been previously reported[8]. Sputtering targets with dimensions of 3 inch dia. and 0.25” thick were comprised of boron-doped silicon and hot-pressed WSi_2. Although the structure of an MLL necessarily contains a layer thickness depth-gradient, which satisfies the Fresnel zone plate law, the multilayer structures utilized for this particular study do not follow a regular stacking sequence and

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were designed for a different purpose[9]. Initial tests utilized very simple periodic multilayer structures, and the final performance reported here used a binary pseudorandom stacking sequence with thickness variation between 3 to 100nm and a total growth thickness of between 1 and 6.4 microns.

After multilayer growth, the samples were spin-coated with Shipley S-1827 photoresist, soft-baked, and then patterned with a photomask to generate trenches with widths varying from 5 to 50 μm. In some cases the photoresist was used as the RIE resist; in others a chromium layer was coated over the multilayers and processed with Transene wet-etch to act as the RIE resist hard-mask. All of the RIE was performed with an Oxford Plasmalab 100 system at the Center for Nanoscale Materials (CNM) at Argonne National Laboratory (ANL). The system has a chamber dedicated for reactive ion etching (RIE) and the second chamber combining RIE and Inductively Coupled Plasma (ICP) etching. CF_4 and SF_6 were used as reactive gases, combined with oxygen. Gas flows were kept in a range of 0 to 50 sccm and pressures in a range of 10 to 25 mTorr. The system has controllable cathode temperature from -110 to 400^0C. After RIE, the photoresist mask was stripped prior to imaging. The etched structures were investigated by surface profilometry using a Dektak 150 to obtain a general indication of the etching uniformity and profiles. Scanning Electron Microscopy (SEM) on the etched samples was performed using the FEI-SEM Nova NanoLab microscope at the CNM and the Hitachi S-4800 microscope at the Center for Functional Nanomaterials (CFN) at Brookhaven National Laboratory (BNL).

Results and discussion

In order to achieve highly anisotropic etch of the materials of interest (Si and WSi_2) by RIE we studied the effects of the gas species mixture (chemistry), sample temperature, gas pressure, plasma power, and mask material.

CF_4/O_2 etching

Initial tests were performed using a CF_4/O_2 gas mixture due to reports in the literature[10,11,12] which indicate, separately, that this particular gas combination has successfully etched both Si and WSi_2. CF_4 provides fluorine radicals to perform the chemical etching of both Si and WSi_2, and oxygen acts as a passivation agent on the sidewalls, which reduces the lateral etch rate[13]. Highly anisotropic etching of the multilayer was obtained, as shown of Figure 1. The anisotropy can be controlled by finding a good balance between the physical (ion bombardment) and chemical etching. This balance is achieved by using an oxygen mixture of 9% and a Cr hard mask.

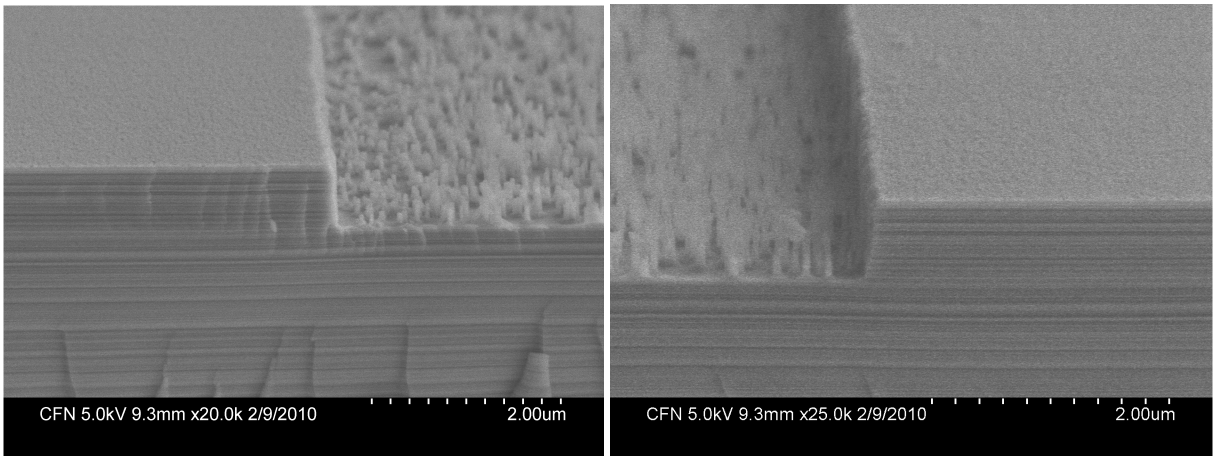
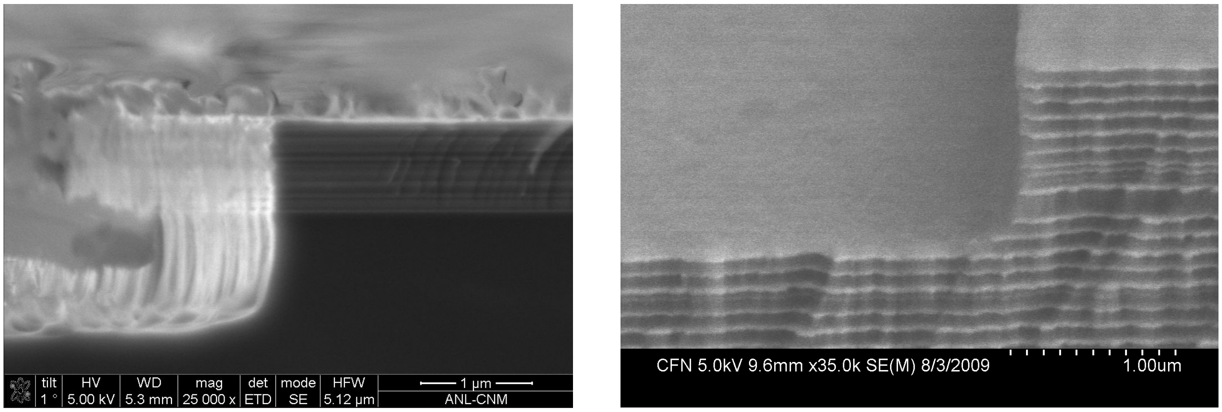


Figure 1. SEM cross-section micrographs of etched multilayers using a CF_4/O_2 chemistry. An RF power of 100W was used for 30 minutes at a sample temperature of 20^oC. The sidewall profile is smooth but the bottom of the trenches is very rough.

The etch rates varied between 22nm and 37nm per minute, which is too slow to be reasonably used to etch tens of microns thick of WSi_2/Si MLLs useful for x-ray focusing. In addition, the etch selectivity between the multilayer and the photoresist mask is about 1:1 for all variations of CF_4/O_2. Using a chromium hard mask produced better results, but the selectivity is still not adequate (1:2) for etching of thick multilayers.

SF_6/O_2 etching

The advantage that SF_6 brings over CF_4 is that more fluorine radicals are available to react with the multilayer materials. When etching Si only, SF_6 has been shown in other studies to realize a faster etching rate[13] when compared to CF_4. The initial experimental conditions used for this gas were chosen to be very similar to those employed for the CF_4/O_2 chemistry. Figure 2 illustrates typical cross-section profiles of a multilayer etched by RIE using a mixture of SF_6/O_2 (81% and 9% respectively) at a pressure of 10 mTorr using an RF power of 100W at room temperature (left) and at -30^{0}C (right).



Etch at room temperature

Etch at -30°C

Figure 2. SEM cross-section micrographs of etched multilayers at two different temperatures using a SF_6/O_2 chemistry.

Sidewall profile anisotropy as good as 92° ± 2° has been obtained. The etch rate has been enhanced, as expected, to 100nm per minute. However, when deep etching is attempted, the profile dramatically changes as can be seen in Figure 3.

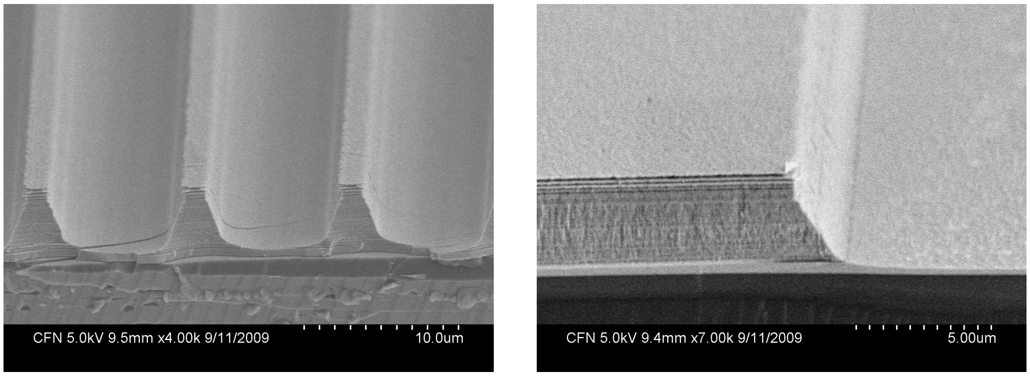


Figure3. SEM cross-section micrographs of etched multilayers using a SF_6/O_2 chemistry. RF power was 100W, SF_6/O_2 mix at 9% O_2, pressure of 10 mTorr. The picture on the right shows the general shape obtain for the feature etched. The picture on the left is a zoom-in on one of the sidewall of the etch feature.

Figure 3 shows that the profile changed during the etching. One possibility is that the ions arriving on the sidewall surface no longer contain sufficient energy to continue the material etch. Experiments have been repeated 3 times with different layer arrangements (thicknesses) and all have resulted in the same type of profile with a sudden change occurring around the same depth of 1 micron. In order to resolve this question, an etch process involving SF_6/O_2 inductively coupled plasma (ICP) has been implemented to enhance the ion bombardment of the surface. The best results with this gas mixture were achieved with a 9% SF_6/O_2 chemistry at 10 mTorr and an applied RF power of 10W with an ICP power of 350W at room temperature.

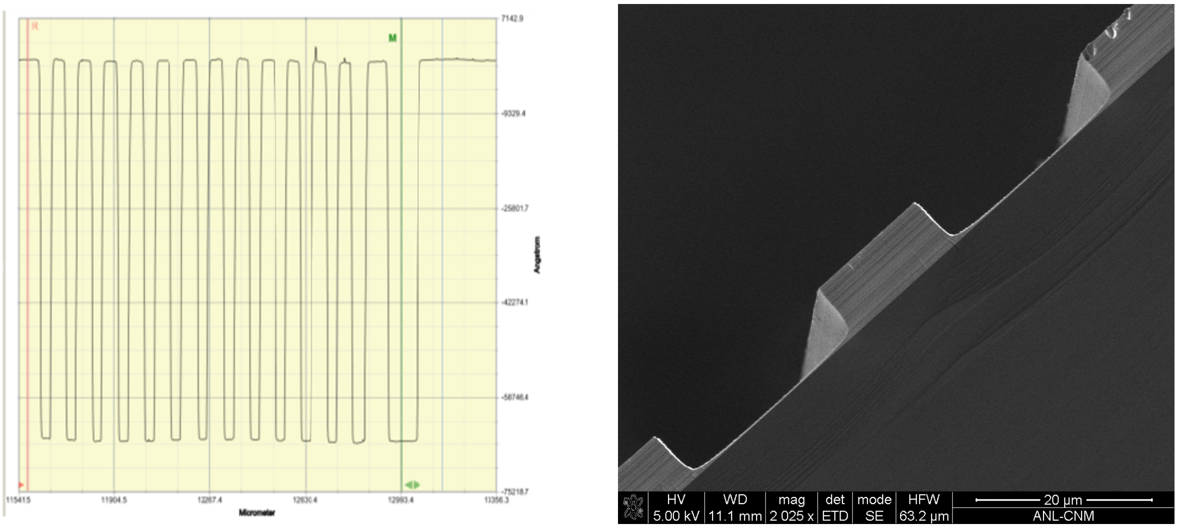


Figure 4. Step-height measurement and corresponding SEM cross-section micrograph of a 6.4um etched multilayer

As can been seen on Figure 4, a highly anisotropic profile ($92^o \pm 2^o$) with very smooth sidewalls has been obtained through the entire depth of the multilayer. By increasing the ion energy, the etch rate has also been enhanced to 325nm per minute.

Conclusion

Sectioning of multilayers consisting of WSi_2/Si using a RIE technique with fluorine-based chemistry has been investigated and optimized. The vertical anisotropy is increased by high fluorine content within the plasma. Higher etching rates are achieved with SF_6, as compared to CF_4. The use of ICP combined with RIE provides higher ion energy, and thus both a higher etching rate and deeper anisotropic etching is obtained. Sidewalls of the etched structures have adequate surface roughness for use as transmission optics when etched with either the CF_4/O_2 or SF_6/O_2 chemistry.

Highly anisotropic etching of a 6.4-micron-thick WSi_2/Si multilayer has been achieved by using a combined RIE/ICP process with a SF_6/O_2 gas mixture. The vertical etching is sustained by the physical etching via ion bombardment for high fluorine content plasmas, while lateral etching is limited by the oxygen reaction with the materials.

Acknowledgements

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[1] Ranganathan, N., Lee,D. Y., Ebin, L., Balasubramanian, N., Prasad, K., Pey, K. L., "The development of a tapered silicon micro-micromachining process for 3D microsystems packaging", J. Micromech. Microeng. 18, 115028-1-8 (2008).

[2] de Boer, M. J., Gardeniers, J. G. E., Jansen, H. V., Smulders, E., Gilde, M.-J., Roelofs, G., Sasserath, J. N., Elwenspoek, M., "Guidelines for Etching Silicon MEMS Structures Using Fluorine High-Density Plasmas at Cryogenic Temperatures", J. Micromech.Syst. 11 (4), 385-401 (2002)

[3] Wells, T., El-Gomati, M., Wood, J., Johnson, S., "Low temperature reactive ion etching of silicon with SF6/O2 plasmas", 9th Int. Vacuum Microelectronics Conf., 349-353 (1996)

[4] Kang, H.C., Maser, J., Stephenson, G.B., Liu, C., Conley, R., Macrander, A.T., Vogt, S., "Nanometer Linear Focusing of Hard X Rays by a Multilayer Laue Lens", Phys. Rev. Lett. 96, 127401-1-4 (2006)

[5] Koyama, T., Ichimaru, S., Tsuji, T., Takano, H., Kagoshima, Y., Ohchi, T., Takenaka, H., "Optical Properties of MoSi2/Si Multilayer Laue Lens as Nanometer X-ray Focusing Device", Appl. Phys. Express 1, 117003-1-3 (2008)

[6] Liese, T., Radisch, V., Krebs, H-U, "Fabrication of multilayer Laue lenses by a combination of pulsed laser deposition and focused ion beam", Rev. Sci. Instrum. 81, 073710-1-4 (2010)

[7] Kang, H.C., Stephenson, G.B., Liu, C., Conley, R., Khachatryan, R., Wieczorek, M., Macrander, A.T., Yan, H., Maser, J., Hiller, J., Koritala, R., "Sectioning of multilayers to make a multilayer Laue lens", Rev. Sci. Instr. 78, 046103-1-3 (2007)

[8] Conley, R., Liu, C., Kewish, C.M., Macrander, A.T., Morawe, C., "Multilayer growth in the APS rotary deposition system", Proc. SPIE 6705, 670505 (2007)

[9] Yashchuk, V.V., Anderson, E.H., Barber, S.K., Bouet, N., Cambie, R., Conley, R., McKinney, W.R., Takacs, P.Z., Voronov, D.L., "Calibration of the modulation transfer function of surface profilometers with binary pseudo-random test standards: Expanding the application range", Proc. SPIE 7801 (submitted)

[10] Oehrlein, G.S., Lindstom, L.J., "Competitive reactions of fluorine and oxygen with W, WSi2, and Si surfaces in reactive ion etching using CF4/O2", Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films 7 (3), 1035-1041 (1989)

[11] Abe, H., Yoneda, M., Fujiwara, N., "Developments of Plasma Etching Technology for Fabricating Semiconductor Devices", Jap. J. of Appl. Phys. 47 (3) , 1435-1455 (2008)

[12] Lee, Y. H., Chen, M.-M., Ahn, K.Y., Bright, A.A., "Reactive etching mechanism of tungsten silicide in CF4-O2 plasma", Thin Solid Films 118 (2), 149-154 (1984)

[13] Buyanova, I.A., Henrya, A., Monemara, B., Lindströmb, J.L., Oehrlein, G.S., "Photoluminescence characterization of SF6-O2 plasma etching of silicon", Materials Science and Engineering B36, 100-103 (1996)