EDFAAO (2019) 4:4-12 1537-0755/$19.00 ©ASM International®

POST-FIB CLEANING OF TEM SPECIMENS FROM 14 NM AND OTHER FINFETS BY CONCENTRATED ARGON ION MILLING

C.S. Bonifacio,[1] M.J. Campin,[1] K. McIlwrath,[2] and P.E. Fischione[1]
[1]E.A. Fischione Instruments Inc., Export, Pennsylvania
[2]JEOL USA, Peabody, Massachusetts
cs_bonifacio@fischione.com

INTRODUCTION

Today's semiconductor devices, specifically fin field-effect transistors (FinFETs), are highly complex due to their multigate transistors and 3D gate structure design. Advanced devices are at the 10 and 7 nm nodes and are currently in production.[1] At the 10 nm node, the source-drain channel or fins are 25% taller and 25% more closely spaced than those in 14 nm node technologies.[2] The industry predicts that the FinFET design will persist up to the 5 nm node;[3] beyond that, the gate-all-around FETs[3,4,5] in the form of vertical nanowire, stacked, and complementary FETs are being considered. These future FETs are more intricate and have smaller features, which will consequently make smaller defects have a larger impact and harder to find.

Metrology and physical failure analysis are already challenging due to the high aspect ratio and complexity of the FinFET structure. To accurately measure the structure of these devices, transmission electron microscopy (TEM) is indispensable due to the resolution it provides. TEM characterization is part of the workflow in semiconductor process development and integration, as well as failure analysis for critical dimension (CD) measurements. Therefore, TEM is crucial for the development and production of advanced semiconductor devices given the decreasing device size.

Specimen thickness of 20 nm or less is required to characterize the 3D structures of the 14 nm node FinFET gate oxide in the TEM.[6] Consequently, fast and reproducible TEM specimen preparation is essential. TEM specimens are usually prepared using a focused ion beam (FIB) tool due to the site specificity and accuracy of specimen thinning and extraction that it provides.[7,8] However, Ga^+ milling causes artifacts such as surface amorphization (post-FIB Si specimens with amorphous layers measured

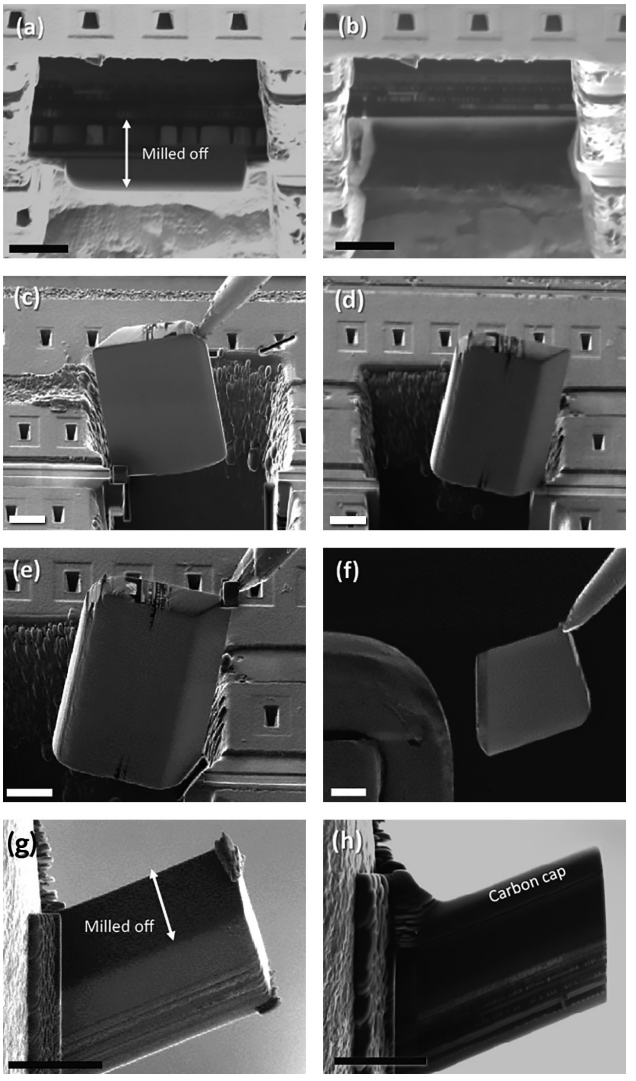

Fig. 1 **Inverted FIB preparation of a full stack deprocessed device modified by additional steps a, b, g, and h; a-b are before and after milling the top metal layers; g-h are before and after milling part of the Si substrate with trimmings during bulk milling and subsequent carbon cap deposition. Scale bars represent 5 µm.**

as 20-30 nm[9] and 2.5 nm[7] thick using 30 kV and 5 kV Ga FIB energy, respectively) and ion-implanted layers, which subsequently limits analytical and high-resolution electron microscopy. This article presents concentrated, small spot (<1 µm), low energy (<1 kV) Ar^+ milling as a post-FIB cleaning step for reproducible specimen preparation of advanced devices with specimen thicknesses of less than 20 nm.

"...TEM IS CRUCIAL FOR THE DEVELOPMENT AND PRODUCTION OF ADVANCED SEMICONDUCTOR DEVICES GIVEN THE DECREASING DEVICE SIZE."

MATERIALS AND METHODS

FIB SPECIMEN PREPARATION

A TEM cross section specimen was created from a depackaged Intel Broadwell M-core processor with 14 nm FinFET structure. The FIB was operated at 30 kV following the inverted FIB preparation described by Alvis et al.[10] However, the flip stage was omitted and additional steps were added to create a curtain-free specimen. Figure 1 shows the modified inverted FIB preparation necessary to target the FinFET structure. Using this method, removal of the top metal layers is essential because of the differential milling rates of the layers, which are the source of curtaining artifacts. The added steps were performed to maintain the specimen integrity and to remove trimming artifacts that were Ga-rich post-FIB milling. Subsequently, conventional FIB final thinning steps (30 and 5 kV) were performed. The experimental conditions of the modified inverted FIB preparation are summarized in Table 1.

With the FinFET structure identified as the region of interest, low kV imaging (2 kV) of the front and back of the TEM specimen was performed during the final FIB polishing steps. Specimen thickness of 50 to 80 nm was achieved after the 5 kV FIB polishing step.

POST FIB ARGON ION MILLING

A low-energy (50 eV to 2.0 keV), concentrated argon ion milling system was used for final polishing and thinning of specimens to thickness of less than 20 nm. Similar to the FIB, the system includes a LaB_6 electron source and electron detectors (secondary electron detector [SED] and scanning transmission electron microscope [STEM] detector) that provide in situ imaging during ion milling. The FIB specimens were mounted on a specimen holder compatible with the ion mill and TEM, which enabled TEM characterization in between milling steps.

Before ion milling of the FinFET specimen, the number of alternating layers of fin and intermetallic layers was identified by tilting the specimen to +27° and imaging in STEM mode in the TEM. Subsequently, ion milling of the side of the specimen without the fin structure, which was identified during low kV FIB imaging, was performed at

Table 1 Experimental conditions for the modified inverted FIB preparation shown in Fig. 1. All steps were performed at 30 kV accelerating voltage in the FIB

Figure	Description	Stage tilt	Stage rotation
1a	Lamella after bulk milling in typical TEM lamella preparation*	7°	0°
1b	1. Milling of top metal layers 2. Typical J or U cut and attachment to the nano manipulator* 3. Lift out the lamella*	7°	0°
1c	1. Rotate nano manipulator to 180° (not shown) 2. Specimen reattached to the bulk	52°	0°
1d	Preparation to orient the lamella with the Si substrate and metal layers at the top and bottom, respectively, by rotating the stage to 180°	52°	180°
1e	1. Reattach the nano manipulator to the lamella 2. Cut the lamella free from the bulk specimen 3. Lift out the lamella and then rotate the nano manipulator to 180°	52°	180°
1f	Attach the lamella to the grid*	52°	0°
1g	Mill of Si substrate with the trimmings from the bulk milling	52°	0°
1h	Carbon or platinum cap deposition	52°	0°

*Steps are not described because they are from the manufacturer's recommended TEM lamella preparation procedure, which is described in the FIB system documentation.

decreasing energies—from 700 to 500 eV at 10°. Table 2 summarizes the post-FIB cleaning steps performed by Ar^+ milling. Iterative milling and TEM imaging were initially implemented at 700 eV to determine the layers removed, i.e., quantification of milling rates per layer.

ELECTRON MICROSCOPY IMAGING AND ANALYSIS

TEM and STEM imaging were performed using a TEM operated at 300 kV. The thickness of the specimens was determined through energy-filtered TEM (EFTEM) imaging using electron energy loss spectroscopy (EELS) with the spectrometer attached to the TEM. Atomic resolution STEM images were acquired using an aberration-corrected TEM (from JEOL USA) operated at 200 kV. The elemental distribution of the specimen was verified using energy dispersive x-ray spectroscopy (EDS) with the spectrometer attached to the aberration-corrected TEM microscope.

RESULTS AND DISCUSSION

DETERMINATION OF MILLING RATES

Figure 2 shows the STEM images of the specimen before and after Ar^+ milling. The observed metal (M) and fin (F) layers before milling, specifically metal/fin/metal/fin/metal/fin (labeled as M/F/M/F/M/F in Fig. 2a), were used to estimate the initial and final specimen thickness from which the milling rates were derived. The F layer comprised the gate oxide over the fin, which is 20 nm (based on the reported gate length for FinFET structures in the 14 nm node[12]). The repetitive layers of F/M/F are two gate oxide layers with an intermetallic in between. This is identified as the gate pitch and is measured as 70 nm for the 14 nm node FinFETs.[12] Given these values, the initial specimen thickness is approximately 140 nm, based on the observed M/F/M/F/M/F in Fig. 2a.

The removal of the metal (M) and fin (F) layers after the 700 eV step was observed with M/F/M/F remaining (Fig. 2b). Similarly, milling from the same side of the specimen at 500 eV resulted in the removal of one M and one F layer (Fig. 2c). With the observed single F and multiple M layers remaining after Ar^+ milling (Fig. 2c), the specimen thickness can be estimated to be equivalent to a single gate length of about 20 nm. The specimen thickness was later quantified using EELS measurements.

(continued on page 8)

Table 2 Ar^+ milling parameters of the FinFET specimen in Fig. 2

Parameter	Step 1 (Fig. 2b)	Step 2 (Fig. 2c)	Step 3 (not shown)
Energy [eV]	700	500	500
Beam current [pA]	100	100	100
Raster area [μm^2]	10 x 10	10 x 10	10 x 10
Specimen angle [°]	+10	+10	-8
Milling time [mins]	15	20	5

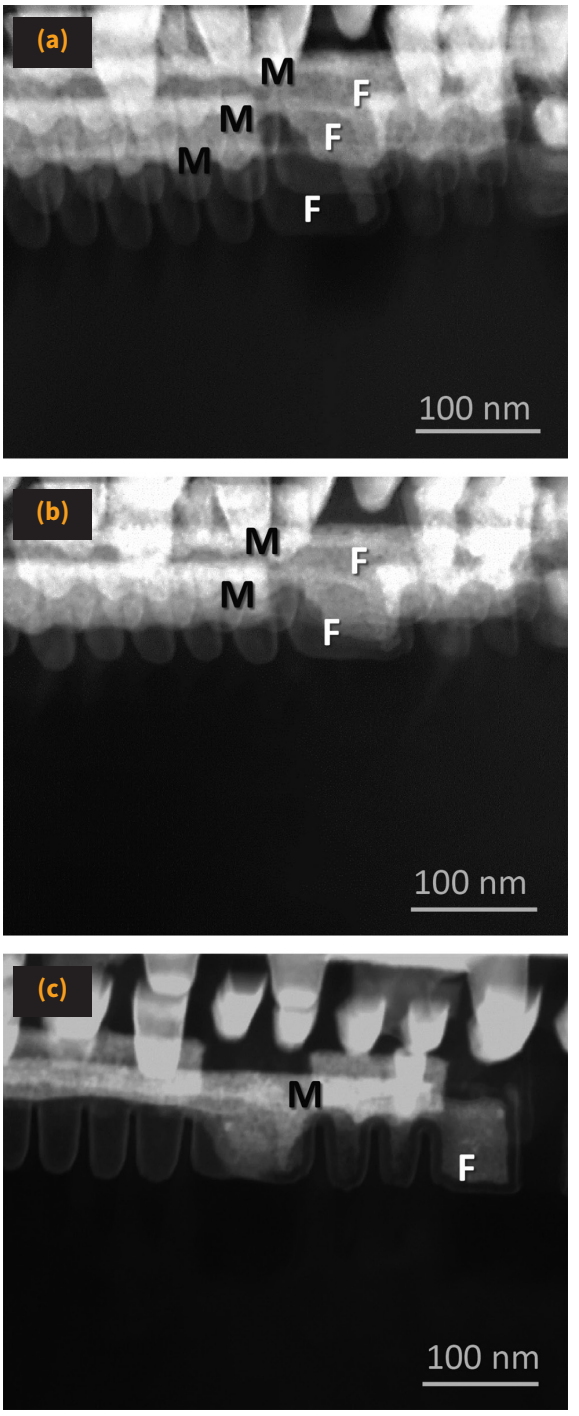

Fig. 2 Dark field STEM images of the specimen tilted +27° before Ar^+ milling (a), after 700 eV Ar^+ milling (b), and after 500 eV Ar^+ milling (c). Specimen layers are identified as metal (M) and fin (F). Figure reproduced from Reference 11.

POST-FIB CLEANING OF TEM SPECIMENS

(continued from page 6)

At the 700 eV step, one metal and one fin layer were removed in 15 minutes; the milling rates of the metal and fin layer at 700 eV can be estimated as 10 minutes and 5 minutes, respectively. It is expected that longer milling times at 500 eV are required and, in this case, a 20-minute milling time was required to remove one M and one F layer.

ITERATIVE ION MILLING

The concentrated beam of argon ions was rastered and directed toward the leading edge of the specimen, which was the Si substrate (Fig. 3). The change in SED (Figs. 3a and 3c) and STEM (Figs. 3b and d) image contrast after Ar^+ milling of the Si substrate indicates the reduction in specimen thickness.

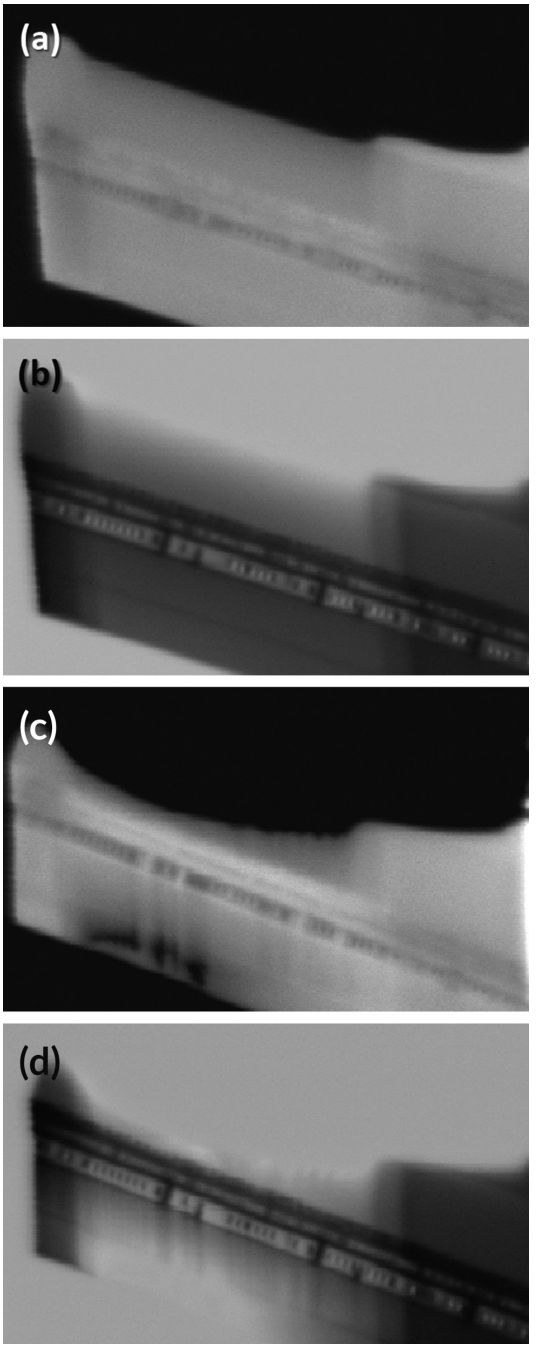

Fig. 3 **SED and STEM images acquired using the Ar^+ milling system before (a-b) and after Ar^+ milling (c-d) showing specimen thickness reduction.**

TEM images acquired between milling steps at decreasing energies show the transition from the epitaxial source/drain (S/D) after 700 eV to the metal gate structure of the FinFET after 500 eV milling (Fig. 4a). Disappearance of the W intermetallic layer and SiGe S/D from 700 to 500 eV (Fig. 4b) indicates controlled milling, which enables targeting of specific integrated circuit features.

HIGH RESOLUTION IMAGING AND ANALYSIS

Figure 5 shows atomic resolution dark field STEM images of the PMOS area (top) and the NMOS area (bottom) of the device after Ga^+ milling and after Ar^+ milling. These images show electron-transparent specimens of differing specimen quality. Low magnification images after Ga^+ milling show that the FinFET was covered with particles (Figs. 5a and c) while the Ar^+ milled specimen was of significantly better quality (Figs. 5b and d).

Higher magnification images at the fin from the PMOS region show that the Si atoms on the fin in Fig. 6a are unclear due to the bright haze over the surface, which

(continued on page 10)

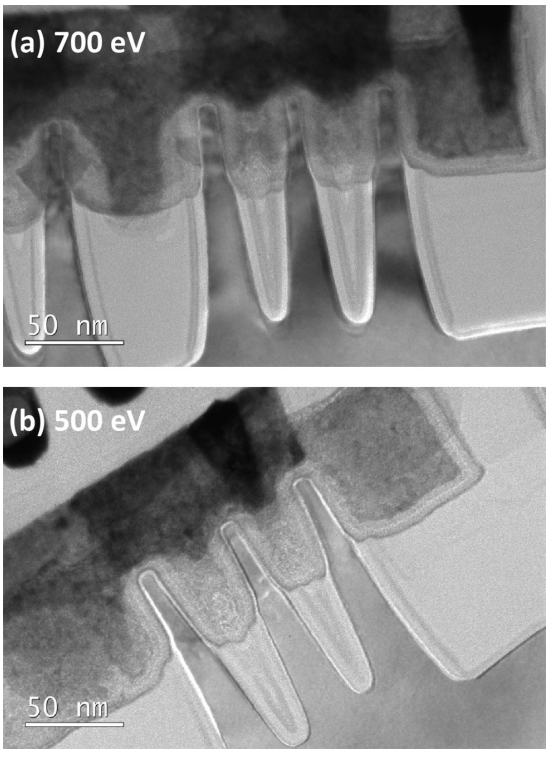

Fig. 4 **TEM images after 700 eV (a) and 500 eV (b) milling show the ability to precisely control the progression of milling through the fin structure. Figure reproduced from Reference 11.**

POST-FIB CLEANING OF TEM SPECIMENS *(continued from page 8)*

may be FIB-induced damage. The fast Fourier transform acquired from the Si in the fin (Fig. 6a, inset) shows a diffused halo in the background, which typically originates from an amorphous material; therefore, the specimen has an amorphous surface following Ga^+ milling.

Conversely, the Ar^+ milled specimen in Fig. 6b has an amorphous-free surface (Fig. 6b, inset) and clearly shows individual atoms of Si on the fin and amorphous high-k and work-function material above the fin. Further, no alteration of the FinFET specimen layers after Ar^+ milling was observed based on the acquired EDS elemental maps (Fig. 7). In effect, thin layers such as the HfO_2 from the FinFET were easily resolved.

The thickness of the Ar^+ milled specimen used for the atomic resolution imaging was determined using EELS. The EFTEM thickness map is a relative-thickness calculated map based on the ratio of the zero-loss map (not shown) and the unfiltered image (Fig. 8a) using the log-ratio method.[13] The relative thickness map is in units of t/λ, where t is the specimen thickness and λ is the inelastic mean free path of the primary beam electrons through a material at a given accelerating voltage. Figures 8a and b show an unfiltered image and an associated EFTEM thickness map of the Ar^+ milled specimen.

Based on the EFTEM map shown in Fig. 8b, dark blue areas are equivalent to $t/\lambda = 0.25$ and are the thinner areas—specifically the Si substrate and at the FinFET structure. The green areas are equivalent to $t/\lambda = 0.50$ and are identified as the metal contact region. The thickness values calculated from the relative t/λ values, which are based on a value of λ for Si with 300 keV primary electrons, are summarized in Table 3. The area of the fins was $t/\lambda = 0.11$, which is 19.2 nm, while the Si substrate was $t/\lambda = 0.07$, which is 12.2 nm. The resulting thickness using targeted Ar^+ milling surpasses the specimen thickness requirement of 20 to 30 nm for imaging 14 nm FinFET structures.[6] Further, the 19.2 nm measured thickness at the fin is close to the estimated specimen thickness of 20 nm, based on the STEM image in Fig. 2c.

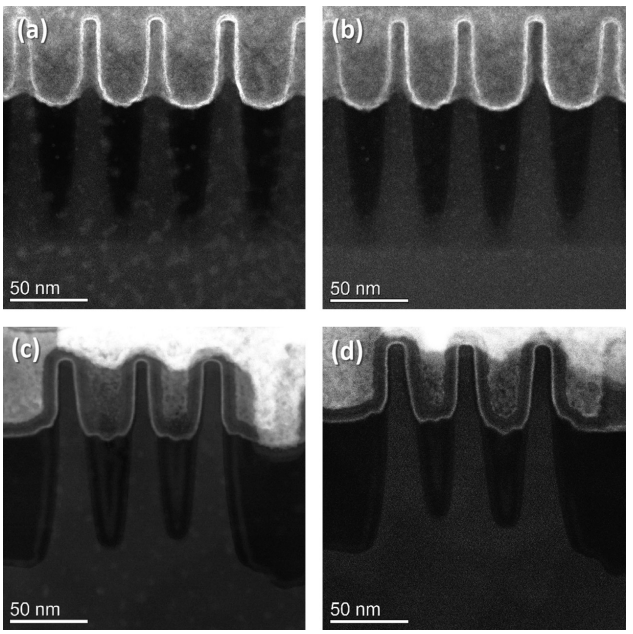

Fig. 5 **HAADF-STEM images of PMOS (top) and NMOS (bottom) regions of a FinFET specimen Ga^+ milled at 30 kV and then 5 kV (a and c) followed by Ar^+ ion milling at 700 eV, 500 eV, and then 300 eV (b and d).**

OUTLOOK AND CONCLUSION

Although this article examines 14 nm FinFET technology, it is also applicable to the 10 and 7 nm FinFET technologies currently in production. Milling rates for 700 eV post-FIB clean-up of the fin structure with the gate oxide and metal layers can be estimated (see Table 4) using the reported gate pitch and gate length for both the 14 and 10 nm Intel FinFET devices.

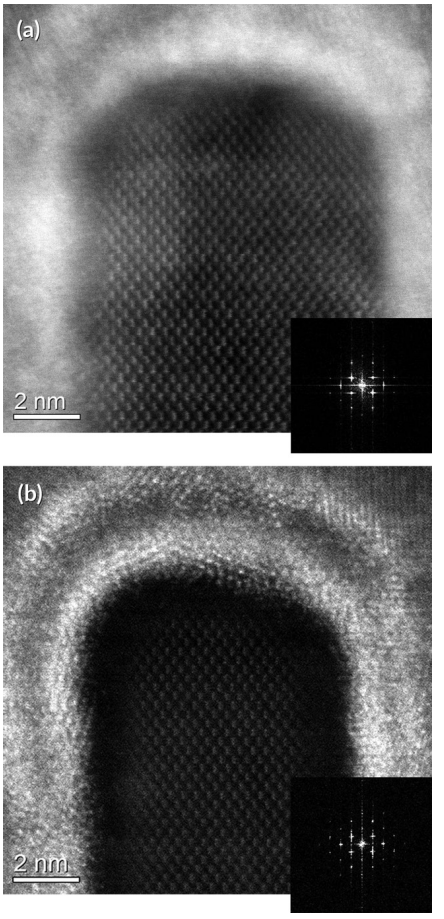

Fig. 6 **HAADF-STEM image of the fin in the PMOS region from Fig. 5 after Ga^+ milling (a) and after sequential Ga^+ and Ar^+ milling (b). Insets in (a) and (b) are fast Fourier transforms derived from the Si in the fin. Figure reproduced from Reference 11.**

The decreasing gate pitch of future FinFET technologies will make targeting the fin structure challenging. Targeted milling is necessary—from FIB preparation to post-FIB clean-up using Ar^+ milling. Based on the results described here, FIB preparation of three to five fin structures followed by iterative Ar^+ milling to target one fin

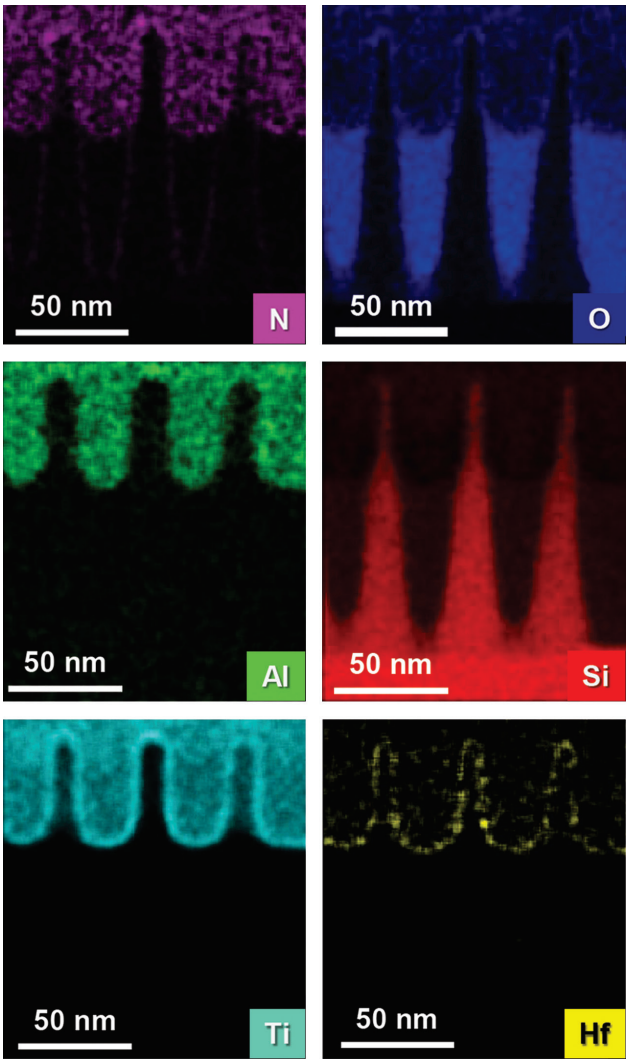

Fig. 7 EDS elemental maps of the PMOS area of the FinFET specimen after Ar^+ milling.

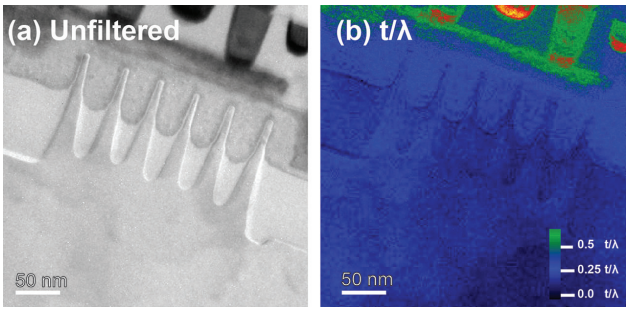

Fig. 8 Unfiltered image (a) and EELS thickness map (b) of the Ar^+ milled specimen shown in Fig. 7b. The color is based on the t/λ scale. Figure reproduced from Reference 11.

Table 3 Derived specimen thickness, t, given the measured t/λ and mean-free path (MFP) value of 174.298 nm[14] for crystalline Si at 300 kV accelerating voltage

	t/λ	Thickness, t [nm]
Si fin	0.11	19.2
Si substrate	0.07	12.2

Table 4 Comparison of milling rates using the concentrated Ar^+ milling system of Intel FinFET technologies at 700 eV and 10° specimen tilt

	14 nm node	10 nm node
Fin layer	4.0 nm/min.	3.6 nm/min.
Metal layer	3.0 nm/min.	1.8 nm/min.

Gate length, $_{14\text{ nm node}}$ = 20 nm[12] Gate pitch,$_{14\text{ nm node}}$ = 70 nm[2]

Gate length, $_{10\text{ nm node}}$ = 18 nm[15] Gate pitch,$_{10\text{ nm node}}$ = 54 nm[2]

structure results in a specimen thickness of less than the gate length of the device, i.e., <20 nm for the 14 nm node and <18 nm for the 10 nm node. Therefore, specimen preparation that results in a TEM specimen thickness of 12 to 19 nm is adequate to prepare a 10 nm FinFET.

The post-FIB cleaning methodology discussed here can be easily integrated into the current FIB preparation and microscopy workflow for failure analysis. Consequently, the turnaround time for high quality TEM imaging and analysis is significantly improved, which is crucial to support FAB production and/or development of new, smaller node geometries. Preparation of 14 nm FinFET TEM specimens was demonstrated, which resulted in high quality specimens for analytical and high-resolution electron microscopy analysis. Controlled and targeted Ar^+ milling using a concentrated beam can be applied not only for thinning TEM specimens, but also for targeting defects by iterative milling. Reproducible specimen preparation with unmatched quality and exceptional specimen thickness of less than 20 nm for imaging and analysis of FinFET structures is possible.

REFERENCES

1. M. Lapedus: Semiconductor Engineering, [Online: 2018]. Available: https://semiengineering.com/nodes-vs-node-lets/.
2. M. Bohr: "Leading at the Edge: Intel Technology and Manufacturing," Technology and Manufacturing Day, San Francisco, March 2017.
3. M. Lapedus: Semiconductor Engineering [Online: 2018]. Available: https://semiengineering.com/transistor-options-beyond-3nm/.

4. H. Mertens, et al.: *IEEE International Electron Devices Meeting (IEDM),* 2016, p. 19.7.1-4.
5. *IMEC Magazine,* [Online: 2017]. Available:. https://www.imec-int.com/en/imec-magazine/imec-magazine-september-2017/the-vertical-nanowire-fet-enabler-of-highly-dense-srams.
6. H. Feng, G.R. Low, P.K. Tan, Y.Z. Zhao, H.H. Yap, M.K. Dawood, Y. Zhou, A.Y. Du, C.Q. Chen, H. Tan, Y.M. Huang, D.D. Wang, J. Lam, and Z.H. Mai: "Investigation of Protection Layer Materials for Ex-situ 'Lift-Out' TEM Sample Preparation with FIB for 14 nm FinFET," *Proc Int. Symp. Test. Fail. Anal. (ISTFA),* 2014, p. 478-482.
7. J. Mayer, L.A. Giannuzzi, T. Kamino, and J. Michael: "TEM Sample Preparation and FIB-Induced Damage," *MRS Bulletin*, 2007, *32*, p. 400-407.
8. L.A. Giannuzzi and F.A. Stevie, "A Review of Focused Ion Beam Milling Techniques for TEM Specimen Preparation," *Micron*, 1999, *30*, p. 197-204.
9. N.I. Kato, "Reducing Focused Ion Beam Damage to Transmission Electron Microscopy Samples," *Journal of Electron Microscopy,* 2004, *53*(5), p. 451-458.
10. R. Alvis, J. Blackwood, S.H. Lee, and M. Bray, "High-Throughput, Site-Specific Sample Prep of Ultra-Thin TEM Lamella for Process Metrology and Failure Analysis," *Proc Int. Symp. Test. Fail. Anal. (ISTFA)* 2012, p. 391-398.
11. C.S. Bonifacio, P. Nowakowski, M.J. Campin, M.L. Ray, and P.E. Fischione: "Low Energy Ar Ion Milling of FIB TEM Specimens from 14 nm and Future FinFET Technologies," *Proc Int. Symp. Test. Fail. Anal. (ISTFA),* 2018, p. 241-246.
12. 14 nm Lithography Process, [Online:2018]., Available: https://en.wikichip.org/wiki/14_nm_lithography_process.
13. R.F. Egerton: *Electron Energy-loss Spectroscopy in the Electron Microscope,* 3rd edition, Springer, New York, 2011.
14. K. Iakoubovskii, K. Mitsuishi, Y. Nakayama, and K. Furuya: "Thickness Measurements with Electron Energy Loss Spectroscopy," *Microscopy Research and Technique,* 2008, *71*, p. 626-631.
15. S. Jones: "Intel versus GlobalFoundries at the Leading Edge," IEDM 2017, [Online: 2018], Available: https://www.semiwiki.com/forum/content/7191-iedm-2017-intel-versus-globalfoundries-leading-edge.html.

ABOUT THE AUTHORS

Cecile Bonifacio has more than 15 years of experience in electron microscopy sample preparation, imaging, and analysis. She has authored more than 50 publications, presented at various meetings, and taught at Lehigh University's annual Microscopy School. In her early career, Bonifacio was a technician for Hitachi Global Storage Technology in San Jose, Calif., and a failure analysis technician for Micron Technology in Virginia. She earned advanced degrees in chemical engineering: M.S. at San Jose State University and Ph.D. at University of California, Davis. Bonifacio gained expertise in TEM, STEM, and in situ microscopy during her doctoral studies and later in her postdoctoral work at University of Pittsburgh. She joined E.A. Fischione Instruments Inc. as an applications scientist in 2016 and now focuses on TEM application development and support.

Michael Campin has more than 15 years of experience in electron microscopy, materials characterization, and physical failure analysis of microelectronic devices. He received a B.S. in astronomy and physics from the University of Arizona and a Ph.D. in physics from New Mexico State University, with a focus on microstructural characterization of corrosion of thin metal films. Much of his doctoral thesis work was conducted while working at the Physical and Chemical Science Center at Sandia National Laboratories in New Mexico. Campin also worked at SEMATECH as a senior engineer/electron microscopist and at Advanced Micro Devices as a member of the technical staff in the device analysis laboratory before joining E.A. Fischione Instruments Inc. as an applications scientist in 2016.

Kevin McIlwrath joined the JEOL TEM applications group in 2010 as a TEM/STEM applications scientist and has been in the industry for more than 24 years. McIlwrath has an extensive background in both materials and biological applications, has co-authored more than 20 papers, given talks at the various meetings in North and South America, and taught the TEM/STEM Tomography section of Lehigh University's Analytical Electron Microscopy course. McIlwrath supports customer training and demonstrations of JEOL's TEM/STEM product line and has personal experience in TEM manufacturing. He concentrates on TEM/STEM applications development in both the materials and biological sciences, with a focus on Cs corrected applications, TEM/STEM, and analytical electron tomography.

Paul Fischione has played a significant role in electron microscopy for more than 30 years. He is the CEO of E.A. Fischione Instruments Inc., a U.S.-based company that designs, manufactures, and distributes advanced microscopy devices for the global scientific community. Products include TEM and SEM sample preparation devices, imaging detectors, and TEM tomography specimen holders. Fischione has firmly grounded new product development in emerging technologies, which is underscored by the number of the company's U.S. and international patents granted during his leadership. Fischione was named a fellow of the Microscopy Society of America in 2017 and currently serves as treasurer of the International Federation of Societies for Microscopy.